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# **Laboratory Manual**

For

### Introductory Chemistry

**Second Eddition** 

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**Prepared By** 

Hamdallah A. Hudali Bassam M. Sweileh Kamal I. Abu Dari Malak I. Qadri

Department of Chemistry
Faculty of Science
University of Jordan
2008



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### **Preface**

The study of chemistry and chemistry labs are required for chemistry students and for students in other fields because of the strong interactions between chemistry and other subjects. These interactions come from the fact that chemistry is the study of matter itself, where as in other disciplines, particular aspects of matter or its applications are studied. This lab manual includes experiments that are selected to teach the students the various standard techniques used by scientists in chemistry and in most other fields of science. In addition, most of the experiments are chosen to demonstrate the topics covered in course lectures.

In order to enjoy the work in the laboratory you should suitably prepare yourself for lab work; otherwise you will spend most of the time wondering what is going on. Therefore, the best way to prepare your self to the lab is to study the whole experiment, and to review the theoretical basis of the experiment. This will help you to understand what you are going to do in the lab.

In each experiment there is a report that you are going to complete in the lab, and each lab report is preceded by a set of Pre Laboratory Questions that you are asked to complete before coming to the lab.

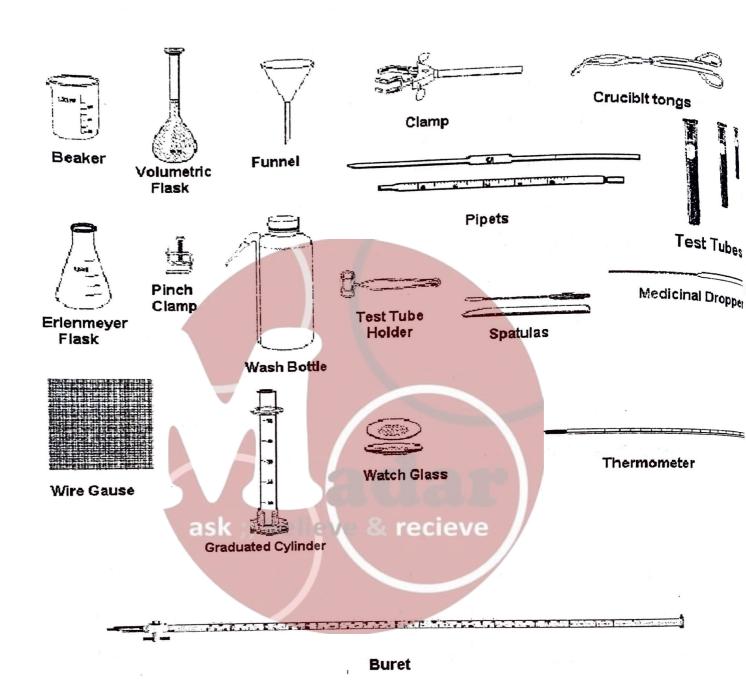
Finally, your lab instructor will guide you how to prepare well for the lab work.

#### **Authors**

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# Common Glassware and Equipment



### **Laboratory Safety Rules**

### **Objectives**

You will be able to learn various safety rules and locate and learn how to use some safety equipments in your laboratory.

- 1. Safety goggles must be worn <u>at all times</u> when working with chemicals or near others doing so.
- 2. Food, drinks, tobacco products, and gum are not allowed in the labs at any time.
- 3. Long hair and loose clothing must be confined or tied back.
- 4. Visitors are not allowed in the labs. Only instructors, stockroom personnel, and students registered in the section are allowed.
- 5. Know the locations of fire alarms, fire extinguishers, chemical fume hoods, safety showers, and emergency eye washes.
- 6. Notify your instructor immediately of <u>any</u> injury, spill, fire or explosion.

  Also notify someone whenever you leave the lab during the lab session except to go to the stockroom.
- 7. Keep your lab space clean and organized. Books, backpacks, jackets, etc. are not allowed in areas where chemicals are used; place them in the appropriate storage locations.
- 8. Whenever your skin (hands, arms, face, ...) comes into contact with laboratory chemicals, wash it quickly and thoroughly with soap and warm water.

- 9. Never leave an ongoing experiment unattended.
- 10. Do not taste anything. If instructed to smell a chemical, do so by carefully fanning the top of test tube or bottle so that a little of the vapor is directed towards your nose.
- 11. Never remove shared chemicals from their original locations, others will need them.
- 12. Wash your hands and arms with soap and water before you leave the lab even if you have been wearing gloves.
- 13. Read labels carefully.
- 14. Label all containers to avoid errors.
- 15. Try to dispense only what will be needed. Do not waste chemicals. Dispose of excess chemicals in appropriate waste containers.
- 16. Know appropriate procedures for cleaning up spills. Clean up small spills immediately. Report all spills (other than just water) to the instructor and request instructor assistance with spills larger than a few milliliters.
- 17. Notify your lab instructor and follow appropriate procedures if there is a mercury spill due to a broken mercury thermometer.
- 18. Dispose of all waste in an appropriate manner. Read waste container labels carefully. Many chemicals need to be disposed of in special containers found in the fume hoods. Never put solids in the sink.
- 19. Do not use flames in the labs unless specifically instructed to do so.

### Treat every chemical as if it were hazardous!

#### **Safety Equipment**

Inspect your lab for the following safety equipment

- 1. First Aid kit
- 2. Fume Hoods
- 3. Safety showers and eye wash stations
- 4. Fire extinguishers
- 5. Fire alarm

#### **QUESTIONS**

- 1. Whom should you call in case of an emergency in the lab?
  - A. Instructor
  - B. Nobody
  - C. A friend
  - D. Anybody
- 2. Why is smoking not permitted in the lab?
  - A. It is inconvenient
  - B. It is bad for your health
  - C. The lab could be blown up
  - D. All the above.
- 3. What should be worn in a laboratory at all times to decrease the likelihood of eye injury?

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- A. Corrective lenses
- B. Safety Glasses
- C. A mask
- D. Nothing
- 4. What should be done if a chemical gets in the eye?
  - A. Notify the Instructor; then return to the experiment.
  - B. Use the eye-wash fountain; then return to the experiment
  - C. Use the eye-wash fountain; then notify the instructor
  - D. Nothing, unless the chemical causes discomfort

- 5. Why is it not advisable to wear contact lenses in the lab?
  - A. Lenses could accidentally fall out of the eye
  - A. Lenses could accidentally lall out of the lenses and the B. Chemical vapor could become trapped between the lenses and the
  - C. Chemical vapor could literally react with the lenses
  - D. All of the above
- 6. What precautions are needed with long hair and beards?
  - A. Keep long hair tied back/keep hair away from flames
  - B. Must be shampooed
  - C. No long hair and/or beards allowed in the lab
  - D. No precautions are necessary
- 7. Eating and drinking is not permitted in the lab because:
  - A. There would not be enough time to finish the experiment
  - B. The storeroom serves terrible appetizers
  - C. You could be poisoned
  - D. The lab would become quite messy with this type of activity

#### YOU MUST BRING THE FOLLOWING ITEMS TO YOUR LABORATORY BEFORE THE FIRST LAB SESSION recieve

1. Note book.

- 2. Safety glasses
- 3. A calculator
- 4. Matches
- 5. Lab coat
- 6. Soap solution
- 7. Sponge
- 8. Towel

### **Agreement**

Sign and date this as a reminder of procedures you will be practicing during chemistry lab class. Keep this for future reference.

I have read and agree to follow all of the safety rules set forth in this contract. I realize that I must obey these rules to insure my own safety, and that of my fellow students and instructors. I will cooperate to the fullest extent with my instructor and fellow students to maintain a safe lab environment. I will also closely follow the oral and written instructions provided by the instructor. I am aware that any violation of this safety contract that results in unsafe conduct in the laboratory or misbehavior on my part may result in being removed from the laboratory.

Date:		
ent		
Student Name:		Reg. No
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Student Signature		
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# **Laboratory Techniques**

# 1. Cleaning Glassware and the Lab Bench

Cleanliness is very important to chemist. Clean glassware immediately after use. Inspect the glassware for cracks or other physical defects.

Clean glassware. Clean all glassware with soap and tap water. Use brushes of appropriate stiffness and size. Rinse the glassware first with tap water and then once or twice with small amounts of deionized water.

Dry glassware. Invert clean glassware such as beakers and flasks on a paper towel to dry. Do not dry calibrated or heavy glassware (graduated cylinders, volumetric flasks, or bottles) in an oven. If the glassware is cleaned as described, but is needed for immediate use, rinse the glassware with small amounts of the solvent or solution being used in the experiment and discard.

Clean lab area. At the end of the laboratory period, wipe clean the surface of the lab bench where you worked with a damp towel.

### 2. Laboratory Balances:

A balance is used to measure the mass of a chemical or a small piece of laboratory equipment. Some various types and models of laboratory balances are shown in Figure (1). Select the balance that provides the sensitivity listed in the procedure of the experiment. In using and caring for a balance, follow these guidelines:

- a) Handle with care; balances are expensive.
- b) Do not attempt to be a handyman. If the balance is not operating properly, ask help from your instructor.
- c) Check the level of the balance; see the instructor for assistance.
- d) Use a beaker, weighing paper, watch glass, or some other container to measure the mass of laboratory chemicals. Do not place laboratory chemicals directly on the pan.
- e) Do not drop anything on the pan.
- f) After the measurements, return the balance to the zero reading. Clean up any spillage of chemicals on the balance or in the balance

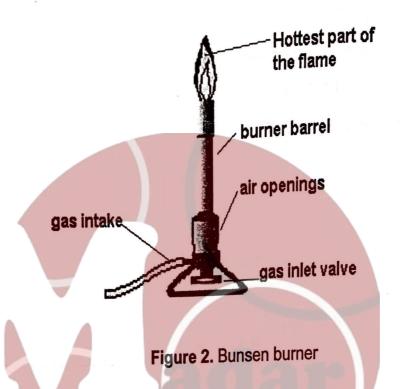


**Figure 1.** Some laboratory balances: (i) balance with accuracy of  $\pm$  0.001 g (ii) balance with accuracy of  $\pm$  0.01 g (iii) balance with accuracy of  $\pm$  0.1 g.

### 3. Bunsen Burner

### Lighting the burner:

 Close the gas inlet valve at the base of the burner and open the main gas valve at the outlet. Close the air openings at the base of the burner (Figure 2).



- 2. Bring a lighted match up the outside of the burner tube and then open slightly the gas control valve until the escaping gas at the top of the burner barrel ignites. Once the yellow luminous flame appears, open slightly the air control at the base of the burner barrel. Open the gas control at the base of the burner until a nonluminous, pale blue flame that has two or more distinct cones appears. Further opening of the air control valve produces a slight hissing sound characteristic of a burner's hottest flame. The addition of too much air may blow out the flame. When the best adjustment is reached, two distinct cones are visible. Obtain an instructor's approval of your well-adjusted flame.
- 3. If the flame goes out, immediately turn off the main gas valve at the outlet and repeat the procedure for lighting a burner.

### 4. Graduated cylinders.

Graduated cylinders (Figure 3a) are used to measure small volumes of liquids and solution for experiments.

Check your glassware: what volumes do your graduated cylinders measure? What is the smallest volume increment marked on the graduated cylinder?

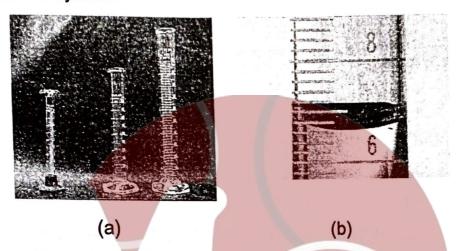


Figure 3. (a) Graduated cylinders and (b) liquid meniscus.

Half fill a graduated cylinder with deionized water. Notice the shape of the meniscus of the water in the cylinder. Sketch a vertical cross section of the meniscus on a sheet of paper. Describe how to read the volume of the water in the cylinder, i.e. how is the eye to be positioned relative to the surface of the water? How precisely can you estimate the volume of water in each graduated cylinder?

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### 5. Pipets

The pipet that you are going to use in this manual is called  $M_{Oh}$  pipet. This type of pipets is graduated so that you can deliver any  $v_{Olume}$  (usually to the tenth of a millimeter) up to its maximum volume.  $M_{Oh}$  pipets are commonly restricted to 5-mL and 10-mL volumes.



### Use of a Pipet

- 1. Prepare a pipet. Clean the pipet with a soap solution; rinse with several portions of tap water and then with deionized water. No water droplets should adhere to the inner wall of the pipet. Transfer the liquid or solution that you intend to pipet from the reagent bottle to a beaker. Do not insert the pipet directly into the reagent bottle. Dry the pipet tip with a clean, dust-free towel or tissue. Rinse through the pipet tip into a waste beaker. Using suction from a collapsed pipet bulb (caution: never use your mouth), draw several 2-3 ml volumes into the pipet as rinse (Fig. 4A). Roll each rinse around in the pipet so that the solution washes the entire surface of the inner wall. Deliver each rinse through the pipet tip into a waste beaker.
- 2. **Fill and operate the pipet.** To fill the pipet, place the tip well below the surface of the solution in the beaker. Then using the collapsed rubber bulb, draw the solution into the pipet until its level is 2-3 cm above the "mark" (Figure 4A). Remove the bulb and quickly cover the top of the pipet with your forefinger, not your thumb (Fig. 4B).

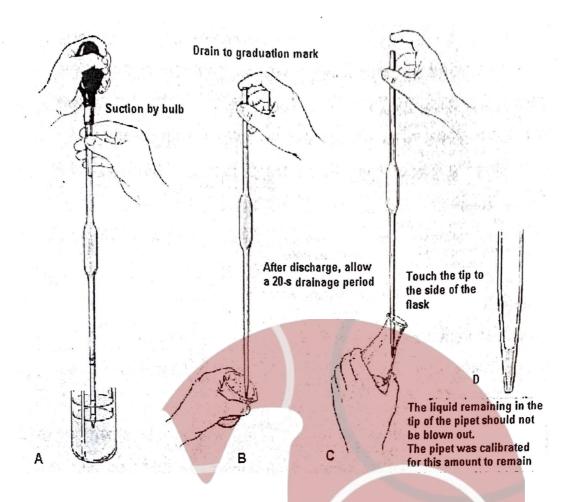


Figure 4. Proper use of the pipet

- 3. Deliver the solution. Remove the tip from the solution, dry the tip with a dust-free towel, and holding the pipet in a vertical position over a waste beaker, control the delivery of the excess solution from the pipet with the forefinger until the bottom of the meniscus is at the mark. Remove any drop suspended from the pipet tip by touching it to the wall of the waste beaker. Again wipe off the tip with a clean, dust-free towel or tissue, and deliver the solution to the receiving vessel (Fig. 4C); keep the tip above the level of the liquid and against the wall of the receiving vessel. Do not blow or shake out the last bit of solution that remains in the tip; this liquid has been included in the calibration of the pipet.
- 4. Clean the pipet. Once the use of the pipet is complete, rinse the pipet several times with deionized water. Roll each rinse to flush the inner wall of the pipet and drain through the tip.

### 6. Burets

When samples of various sizes must be dispensed or measured precisely, a buret may be used. The buret consists of a narrow calibrated glass tube, fitted at the bottom with a valve for controlling the flow of liquid. The valve is more commonly called a stopcock. (See Fig. 5).

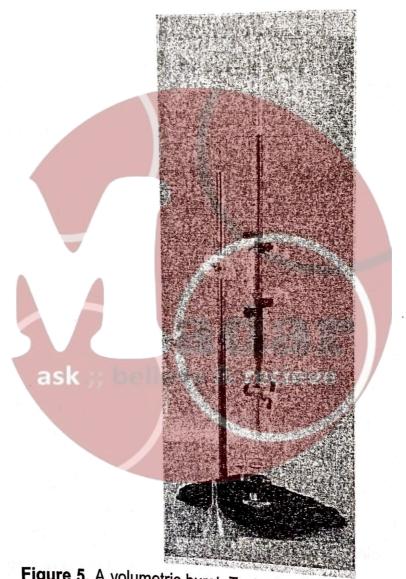


Figure 5. A volumetric buret. Typically, 50-mL burets are used in chemistry laboratories.

Like a pipet, a buret must be cleaned before use. The precision -permitted in reading a buret is 0.02 mL, but if the buret is not completely clean, this level of precision is not attainable. Clean the buret with soap and water, using a special long-handed buret brush

to scrub the interior of the glass. Then rinse the buret with tap water. Do not attempt to admit water directly to the buret from the cold water tap. Fill a beaker with tap water, and pour from the beaker into the buret. Finally, rinse the buret several times with distilled water.

Before use, the buret should be rinsed with several small portions of the solution to be used in the buret. The buret should be tilted and rotated during the rinsings, to make sure that all rinsed water is washed from it. Discard the rinsings.

After use, the burets should again be rinsed with distilled water. Many of the reagent solutions used in burets may attack the glass of the buret if they are not removed. This would destroy the calibration. To speed up the cleaning of a buret in future experiments, the buret may be left filled with distilled water during storage between experiments (if your locker is large enough to permit this).

A common mistake made by beginning students is to fill the buret with the reagent solution to be dispensed to exactly the 0.00 mark. This is not necessary or desirable in most experiments, and waste time. The buret should be filled to a level that is comfortable for you to read (based on your height). The precise initial liquid level reading of the buret should be taken before the solution is dispensed and again after the liquid is dispensed. The readings should be made to the nearest 0.02 mL. The volume of liquid dispensed is then obtained by simple subtraction of the two volume readings.

### 1 Techniques and Measurements

### **Objectives**

You will be able to properly select and operate a balance, use a pipet and to determine the density of a pure liquid and solid.

#### Introduction

The use of laboratory equipment requires a knowledge and understanding of the scope and limits of the equipment that is being used. For example, a bathroom scale (balance) might be "good enough" for collecting data on the average mass of a student in a class of students, but would be useless in determining the average mass of an ant in a colony of ants. Once the appropriate laboratory apparatus has been selected for the experiment, the next step is to use it properly so that best data can be obtained.

The laboratory balance is perhaps the most common and most often used piece of laboratory apparatus. Balances are of different sizes, makes, and sensitivities. The selection of the appropriate balance for mass measurement depends upon the degree of accuracy and precision required from the analysis. Read carefully Technique (2) in the section Techniques and Measurements.

Most of the glassware in your laboratory has been marked by the manufacturer to indicate the volume contained by the glassware when filled to a certain level. For example, beakers and Erlenmeyer flasks are marked with very approximate volumes which serve merely as a rough guide to the volume of liquid in the container. Graduated cylinders, burets and pipets are used for more precise measurements of volume as needed.

The most common apparatus for routine determination of liquid volumes is the graduated cylinder. Although a graduated cylinder does not permit a precise determination of volume as the pipet or buret, for many applications the precision of graduated cylinder is sufficient.

Pipets are long narrow pieces of glassware that measure precisely the volumes of liquids or solutions. The techniques for filling and dispensing a liquid require practice. Technique (5) describes the correct procedure for the use of pipets in transferring liquids.

In this experiment you will use some of the laboratory equipment  $t_0$  determine the density of a liquid sample and the density of a solid substance. Density is an **intensive property** of all substances. Its measurement is expressed as a ratio of mass to volume:

#### Density = Mass/Volume

For most solid and liquid samples, it is reported in the units of g/ml (or g/cm³). A substance having a large density has a large mass occupying a small volume. Water has a density of 1.0 g/mL at 4 °C. Osmium metal has the greatest density among the elements with a density of 22.6 g/cm³. In this experiment the density of a liquid is determined by measuring the mass of a known volume of the liquid. The density of a water-insoluble solid is determined by measuring its mass and then the volume of water it displaces.

### **Safety Precautions:**

- Wear safety glasses at all times while in the laboratory.
- When using a pipet, use rubber safety bulb to apply the suction force.

  Never use your mouth to apply the suction.

### Apparatus/Reagents Required

Graduated cylinder (50 mL), pipet (10 mL), pipet filler, sodium chloride,

water-soluble liquids, small pieces of solid material that are insoluble in water (pellets, shots, or spheres)

#### **Procedure**

Record your data and observations directly on the data sheet.

Perform two trials for the following determinations if time permits.

### A. Determination of the Density of Pure Liquid

- 1. Clean and wipe dry a 50-mL beaker. Weigh the dry beaker to the nearest 0.01g.
- 2. Obtain about 50 mL of distilled water in a clean 100-mL Erlenmeyer flask. Determine and record the temperature of the water.
- 3. Obtain a 10-mL pipet and a pipet bulb. Clean the pipet with soap and water. Rinse the pipet with tap water and then with small portion of distilled water.
- 4. Pipet 10 mL of the distilled water from the flask into the clean beaker you have weighed. Reweigh the beaker containing the 10 mL of water. Determine the mass of water transferred by the pipet.
- Calculate the density of water. Compare the measured density of water with the value listed in the handbook for the temperature of your experiment.
- 6. Obtain an unknown liquid and record its identification number.
- 7. Determine the density of the unknown liquid, using the method just described above (steps 1-5). In this case, and after cleaning the pipet with water (as listed in step 3), wash the pipet with small portion of the unknown liquid.
- 8. Return the used unknown liquid to the appropriate disposal container.

#### **B. Density of Solutions**

1. Prepare approximately 50 mL of 10% m/v of sodium chloride solution by dissolving about 5 g of NaCl in 50 mL of distilled water (measured

- by a graduated cylinder) in an Erlenmeyer flask. Mass of the solute should be determined up to the nearest 0.01 g.
- 2. Clean and wipe dry a 50-mL beaker. Weigh the dry beaker to the nearest 0.01g.
- 3. Rinse the pipet with small portion of the solution.
- 4. Pipet 10 mL of the salt solution from the flask into the clean beaker you have weighed. Reweigh the beaker containing the 10 mL of salt solution. Determine the mass of salt solution transferred by the pipet.
- 5. Calculate the density of salt solution.

### C. Density of Solids

- 1. Obtain a sample of the solid substance (pellets, shots, or spheres) and record its identification number.
- 2. Weigh an empty 50-mL beaker to the nearest 0.01 g. Add approximately 50 g of the solid material and record the total mass to the nearest 0.01 g.
- 3. Add water to your 50-mL graduated cylinder to approximately the 25mL mark. Record the exact volume of water in the cylinder to the precision permitted by the calibration marks of the cylinder.
- 4. Pour the solid sample into the graduated cylinder, making sure that none of the pieces sticks to the wall of the cylinder above the water level. Shake the cylinder gently to make certain that no air bubbles have been trapped among the pieces of the solid.
- 5. Read the level of the water in the graduated cylinder, again making your determination to the precision permitted by the calibration marks of the cylinder. It is clear that the change in water levels represents the volume of the solid pieces.
- 6. Calculate the density of the unknown solid substance.
- 7. Dry the pieces of the solid with paper towel and return them to your instructor. (Do not discard)

# 1 Techniques and Measurements

Name:	Section
Lab. Instructor	Date
Pre-Laboratory Qu	uestions
density of the metal.  2. An empty beaker weighs 32.4257 liquid is transferred to the beaker. liquid sample was 39.4507 g. Calliquid	g. A 10.00 mL sample of unknown The total mass of the beaker and culate the density of the unknown
	the units of specific gravity?

# 1 Techniques and Measurements

Name:	Section	
Lab. Instructor	Date	

### **Results and Calculation**

### A. Determination of the Density of Pure Liquid

Pure water	Trial (I)	Trial (II)
Mass of beaker	g	g
Mass of beaker + water	g	g
Mass of water	g	g
Volume of water	mL	mL
Temperature of water	°C	°C
Density	g/mL	g/mL
Average density	2000	g/mL
Handbook density ask	elleve & reci	ieve / g/mL
Unknown liquid	The street of	
Unknown Nomber		
Mass of beaker	g	g
Mass of beaker + unknown liquid	g	<b>9</b>
Mass of unknown liquid	g	g
Volume of unknown liquid	mL	mL
Density of unknown liquid	g/mL	g/mL

B. Density of Solutions

Trial (I)	Trial (II)
g	
mL	E S
· g	
g	8
°C	300
g/mL	g/mL
	g mL g g

### C. Density of Solids

	Trial (I)	Trial (II)
Mass of beaker	g	9
Mass of beaker + solid pieces	9	9
Mass of solid pieces	recieve <sup>9</sup>	(
Initial water level in the graduated	mL	m
cylinder Final water level in the graduated	mL	m
cylinder		m
Volume of solid pieces	mL	g/n
Density of the solid substance	g/mL	·

### Questions

1.	What error would be introduced into the density of the metal pellets if you had not shaken the pellets to remove adhering air bubbles? Would
	the density be too high or too low?
2.	The density of silver is 10.5 g/cm <sup>3</sup> and the density of platinum is 21.45 g/cm <sup>3</sup> . If equal masses of silver and platinum were transferred to equal volumes of water in graduated cylinders, which graduated cylinder would have the highest volume reading? Explain.
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### 2 Formula of a Hydrate

#### **Objectives**

You will be able to determine the percentage of water of crystallization and the formula of potassium alum, i.e. the value of "x" in the formula KAI(SO<sub>4</sub>)<sub>2</sub>.xH<sub>2</sub>O, and to determine the percent by mass of water of crystallization in an unknown hydrate.

#### Introduction

A hydrate is a compound that has crystallized from aqueous solution with weakly bound water molecules contained in the crystal. These compounds could be transition metal salts as CuSO<sub>4</sub>.5H<sub>2</sub>O or non-transition metal salt as NaC<sub>2</sub>H<sub>3</sub>O<sub>2</sub>.3H<sub>2</sub>O or even a covalent compound. But normally the name hydrate is used for metal salts. A class of hydrates is the alums, these consist of a double salt with the general formula (M<sup>I</sup>M<sup>III</sup>(SO<sub>4</sub>)<sub>2</sub>.xH<sub>2</sub>O), where M<sup>I</sup> is a univalent cation and M<sup>III</sup> is a trivalent cation. An example is the potassium alum KAI(SO<sub>4</sub>)<sub>2</sub>.12H<sub>2</sub>O.

Many hydrated salts can be transformed to the anhydrous (without water) salt by heat. For example, if a sample of copper sulfate pentahydrate, CuSO<sub>4</sub>.5H<sub>2</sub>O, is heated, the bright blue crystals of the hydrate are converted to the white, powdery, anhydrous salt:

$$\begin{array}{ccc} CuSO_{4}.5H_{2}O_{(s)} & \rightarrow & CuSO_{4(s)} & + & 5H_{2}O_{(g)} \\ & & \text{(blue)} & \text{(white)} \end{array}$$

During heating of copper sulfate pentahydrate, the water of crystallization is clearly seen escaping as steam from the crystals. It is also possible to reconstitute the hydrate of copper sulfate; if water is

added to the white anhydrous salt, the solid will recover the blue color of the hydrated salt.

Not all hydrated salts are converted simply into the anhydrous compound when heated, however. Some hydrated metal salts will decompose upon losing the water of crystallization; subsequently they are converted to the metal oxide if the heating is carried out in air. Most covalent hydrates decompose rather than simply lose water when heated.

In this experiment you will start with a measured quantity of the hydrate, heat it in a porcelain crucible to evaporate all water of crystallization and then measure the mass of the anhydrous salt produced. From the masses of the hydrate, the water lost and the anhydrous salt produced you will be able to calculate the percentage of water of crystallization and the formula of the hydrate.

### **Safety Precautions:**

- Wear safety glasses at all times while in the laboratory.
- All copper, cobalt, nickel, chromium and barium compounds are highly toxic. Wash hands after use.
- When you are heating the hydrated metal salts, they may spatter if heated too strongly. To avoid this, heat the solid with as small a flame as possible at first, then heat strongly to ensure complete loss of water of hydration.
- Dispose of the metal salts as directed by the instructor. Do not wash the salts down the drain, and do not place them in the wastebasket.
- Use crucible tongs to handle the hot crucible and cover. Do not touch the crucible or the ring at any time during the experiment.

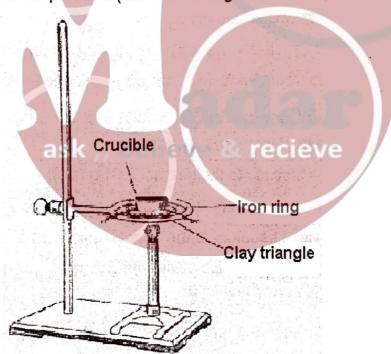
### Apparatus/Reagents Required

Porcelain crucible, crucible tongs, clay triangle, stand with metal ring, potassium alum and an unknown hydrate.

#### **Procedure**

#### A. Potassium Alum

- Obtain a crucible and cover from the store room, examine them for any crack. Wash the crucible, rinse with distilled water and dry it.
- Place the crucible on a clay triangle supported by an iron ring (Figure 2.1) and heat it to redness using the non-luminous flame of the Bunsen burner. Turn off the Bunsen burner and allow the crucible to cool to room temperature (without moving the crucible).



**Figure 2.1.** The correct arrangement of the ring, clay triangle, crucible and the burner.

3. Weigh the crucible to the nearest 0.01 g  $(m_1)$ .

- 4. Place about one gram of potassium alum in the crucible and weigh
- with its content to the nearest  $0.01 g (m_2)$ . with its content to the nearest the Bunsen burner. After the salt has 5. Carefully heat the crucible with the Bunsen burner. After the salt has
- Carefully heat the crucible with hot non-luminous flame expanded and frothed, heat the crucible with hot non-luminous flame for five minutes.

  6. Allow the crucible to cool to room temperature and then weigh it (m<sub>3)</sub>
- 7. Heat the crucible again for two minutes, allow it to cool to room
- temperature and weigh it again. If the mass obtained is different from  $m_3$  by 0.04 g or more, heating, cooling, and weighing should  $b_0$ repeated

### **B. Unknown Hydrate**

- 1. Obtain from the store room an unknown hydrate; write down its identification number on your lab report. Wash the crucible, rinse with distilled water and dry it.
- 2. Place the crucible on a clay triangle supported by an iron ring and heat it to redness using the non-luminous flame of the Bunsen burner. Turn off the Bunsen burner and without moving the crucible allow it to cool to room temperature.
- 3. Weigh the crucible to the nearest 0.01 g (m<sub>4</sub>).
- 4. Place about one gram of the unknown hydrate in the crucible and weigh it with its content to the nearest 0.01 g (m<sub>5</sub>).
- 5. Carefully heat the crucible with the Bunsen burner, note any change in the color or shape of the crystals. When no more water vapor in observed, heat the crucible with hot non-luminous flame for five minutes.
- 6. Allow the crucible to cool to room temperature and then weigh it  $(m_6)$ .
- 7. Heat the crucible again for two minutes, allow it to cool to room temperature and weigh it again. If the mass obtained is different from (m<sub>6</sub>) by 0.04 g or more, heating, cooling, and weighing should be

# 2 Formula of a Hydrate

Nam	ne: Section
Lab.	Instructor Date
	Pre-Laboratory Questions
1.	What are alums? Give examples other than potassium alum.
2.	What are the hydrates? Give few examples of metal salt hydrates.
	ask :: helleve & recieve
3.	Potassium chromic alum has the formula: KCr(SO <sub>4</sub> ) <sub>2</sub> .xH <sub>2</sub> O. A sample
	of 1.12 g of this alum was heated in a crucible to get a constant mass.
	The mass of the anhydrous salt produced (KCr(SO <sub>4</sub> ) <sub>2</sub> ) was 0.64 g.
	Calculate the value "x" in the formula of the alum.

# 2 Formula of a Hydrate

Name:	Section	
Lab. Instructor	Date	

### **Results and Calculations**

### A. Potassium Alum:

Mass of empty crucible (m <sub>1</sub> )	g
Mass of crucible and the alum (m <sub>2</sub> )	g
Mass of crucible and anhydrous salt (m₃)	g
Mass of alum (m <sub>2</sub> -m <sub>1</sub> )	g
Mass of anhydrous salt (m <sub>3</sub> -m <sub>1</sub> )	g
Mass of water lost upon heating (m <sub>2</sub> -m <sub>3</sub> )	g
Number of moles of water lost upon heating	mol
Number of moles of anhydrous salt ( KAI(SO <sub>4</sub> ) <sub>2</sub> )	mol
Percentage of water of crystallization, by mass	%
The value "x" in the formula, (number of moles of	
water of crystallization / number of moles of	
anhydrous salt)	

### B. Unknown Hydrate:

Unknown number: -----

Mass of empty crucible (m₄)	
Mass of crucible and the hydrate (m₅)	, <u></u> • , (4.3) .d • .g
Mass of crucible and anhydrous salt (m <sub>6</sub> )	9
Mass of anhydrous salt (m <sub>6</sub> −m <sub>4</sub> )	9
Mass of water lost upon heating (m₅–m₅)	9
Percentage of water of crystallization, by mass	i wygme is ami %



#### **QUESTIONS**

<ol> <li>What is the effect on the calculated value of "x" if the dehydration alum is not complete</li> </ol>		
2.	A student heated 1.16 g of hydra	ated sodium sulfate in a crucible to get
		is the formula of the anhydrous salt?
	(Show your work)	
		e Ve & recieve

## 3 The Empirical Formula of an Oxide

#### **Objectives**

You will be able to determine the empirical formula for magnesium oxide from the masses of magnesium and oxygen that react to form magnesium oxide.

#### Introduction

Magnesium metal is a moderately reactive element. At room temperature, magnesium reacts very slowly with atmospheric oxygen and can be kept for long periods of time without appreciable oxide buildup. At elevated temperatures, however, magnesium will ignite in an excess of oxygen gas. The burning is accompanied by an intensely white flame to produce magnesium oxide. Because of the brightness of its flame, magnesium was used in photographic flashbulbs and is still used in flares.

In this experiment, you will heat a known mass of magnesium in a closed crucible and expose it gradually to the air. Because air contains both oxygen and nitrogen gases, magnesium will react with both of them, and gradually turning from shiny metal to grayish-white powder. Under these conditions of burning, a portion of this powder is magnesium nitride, Mg<sub>3</sub>N<sub>2</sub>. Although the amount of molecular nitrogen in the air is approximately four times that of molecular oxygen, more oxide than nitride is formed when an active metal is burned in air. The reason is the superior reactivity of molecular oxygen. Water will convert the magnesium nitride in the mixture to magnesium hydroxide [Mg(OH)<sub>2</sub>] with the liberation of

ammonia (NH $_3$ ). Heat will cause conversion of the hydroxide to the oxide with the loss of gaseous water.

$$Mg_3N_2 + 6H_2O \rightarrow 3Mg(OH)_2 + 2NH_3$$
  
 $Mg(OH)_2 + heat \rightarrow MgO + H_2O$ 

The ammonia produced by this reaction can be detected by its odor, which is released upon heating the mixture.

Because the product consists of magnesium oxide only after this treatment, you can determine the mass of oxygen that is present in the oxide from its mass and the original mass of magnesium. From the masses of O and Mg, and by applying the laws of conservation of mass and the mole concept, you can determine the empirical formula of this oxide.

#### **Safety Precautions:**

- Use crucible tongs to handle the hot crucible and cover.
- Avoid burning your fingers. Do not touch the crucible or the iron ring at any time during this experiment.

#### ask ;; believe & recieve

#### **Apparatus/Reagents Required**

Porcelain crucible and cover, crucible tongs, clay triangle, magnesium ribbon (or turnings), litmus paper.

#### **Procedure**

- 1. Record all data and observations directly in your notebook in ink.
- 2. Ask your laboratory instructor about discarding the magnesium oxide that you will prepare in this experiment.

- 3. Obtain a crucible and cover and examine for any defects. The crucible and cover are extremely fragile and expensive. Use caution in handling them. Wash the crucible and its cover, rinse and dry them.
- 4. Obtain about 0.2 g of magnesium ribbon (or magnesium turnings). If it is not bright, clean the surface with sandpaper.
- 5. Place the covered crucible in a clay triangle on an iron ring that is attached to a ring stand. Adjust the height of the ring so that the bottom of the crucible will be in the hottest part of a properly adjusted laboratory burner. The correct arrangement of the equipment, crucible, and burner is shown in Figure 3.1.

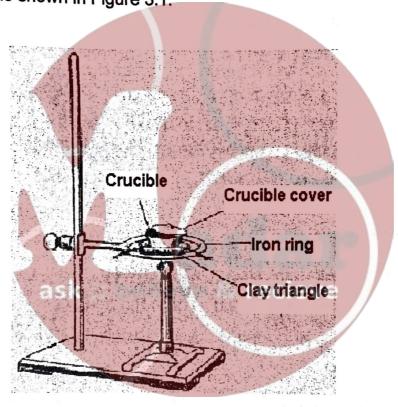


Figure 3.1. The correct arrangement of the the ring, clay triangle, crucible and its cover, and the burner.

6. Heat the covered crucible for about 5 min. The bottom of the crucible should attain a red-hot glow during this time. Remove the burner and allow the crucible to cool (5-10 min). When the covered crucible is cool, transfer the crucible to the pan of a balance using crucible tongs while holding the wire gauze (or the watch glass) under the crucible.

- 7. Obtain and record the mass of crucible to the nearest 0.01 g.
- 8. Return the crucible and cover to the clay triangle. Reheat in the full heat of the burner flame for 5 min. Allow the crucible/cover to  $co_{0}$ completely to room temperature. Reweigh the crucible after it has cooled. If the mass this time differs from the earlier mass by more than 0.02 g, reheat the crucible for an additional 5 min and reweigh when cool. Continue the heating/weighing until the mass of the crucible and cover constant to within 0.02 g. Record the average value of these masses.
- 9. Fold the magnesium ribbon to fit completely inside the crucible. Obtain the mass, and record it again.
- 10. Return the crucible to the clay triangle and adjust the cover to be slightly open to allow air to enter the crucible. Do not open the cover too far, because doing so will allow the metal to enflame. The metal should glow brightly without flames. Flames from the metal must be avoided because they will carry part of the solid oxide out of the crucible.
- 11. With a very small flame, begin heating the crucible gently (If the crucible begins to smoke when heated, immediately cover the magnesium completely and remove the heat for 2-3 min). Next heat the crucible until no metal is evident and no glow occurs when the cover is lifted.
- 12. Allow the covered crucible and its content to cool. The contents should be white or slightly gray.
- 13. Remove the cover and place it on the wire gauze. Add about 10 drops of distilled water, using a medicine dropper, directly on the contents. The smell of ammonia may be evident at this point.
- 14. Return the crucible and its cover to the clay triangle and heat the crucible first with a small flame to drive off the water that has been added. As the water is driven off, hold a piece of moistened red litmus paper (with a forceps) in the stream of steam being expelled from the

- crucible. Record your observations. When it is certain that all the water has been driven off, heat the crucible strongly for 8-10 min to convert the hydroxide to the oxide.
- 15. Allow the covered crucible to cool completely to room temperature.

  Obtain the mass of the crucible (without cover) and record it.
- 16. Heat the covered crucible strongly again for about 3 min. Obtain and record the mass after the crucible has cooled. Continue the heating/weighing until the mass of the crucible is constant to within 0.02 g. Record the average value of these masses.
- 17. Clean the crucible and its cover carefully.
- 18. Calculate the mass of magnesium that was taken, the mass of magnesium oxide formed, and the mass percentage of magnesium in magnesium oxide.



## 3 The Empirical Formula of an Oxide

Na	me: Section
_a	b. Instructor Date
	<b>Pre-Laboratory Questions</b>
1.	What is the mole and molar mass? What are the molar masses of magnesium and atomic oxygen?
2.	When 0.192 g of phosphorus is burned, 0.341 g of a white oxide is
	obtained. (a) Write a balanced chemical equation for the reaction of
	phosphorus with molecular oxygen based on this empirical formula. (b)
	Determine the empirical formula of the oxide.
	ask ;; believe & recieve
	······································

## 3 The Empirical Formula of an Oxide

Name:	Section
Lab. Instructor	Date

#### **Results and Calculations**

Mass of empty crucible (after first heating)	g
Mass of empty crucible (after second heating)	g
Final mass of empty crucible	g
Mass of crucible and Mg	g
Mass of Mg	g
Moles of Mg (n1)	mol
Final mass of crucible and Mg-oxide	g
Mass of Mg-oxide produced	ve / g
Mass of oxygen gained	g
Moles of oxygen atoms (n2)	mol
Formula of magnesium oxide (Mg <sub>n1</sub> O <sub>n2</sub> )	
Empirical formula of magnesium oxide	
Mass percent of Mg in the oxide (x <sub>1</sub> ) (experimentally)	%
Mass percent of Mg in the oxide (x2) (calculated for	75
MgO)	%
Percentage error = $[ x_2-x_1 /x_2] \times 100\%$	%
1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1	%

#### QUESTIONS

1.	If water had not been added to your initial product, what error in the
	determined percentage of magnesium would have resulted (that is, if
	part of the product has been magnesium nitride)? Explain.
	part of the product has been magnessarily .
2.	If large amount of magnesium oxide had been lost during the heating
	of the crucible, would this have made the calculated %Mg in the
	product too high or too low? Explain.
	ask : belle & recieve

## 4 Limiting Reactant

#### **Objectives**

You will be able to determine the limiting reactant in the reaction of sodium phosphate and barium chloride solutions from the mass of barium phosphate produced, and you will be able to calculate the original masses of reactants.

#### Introduction

In any chemical reaction, the reaction will continue towards equilibrium or until one of the reactants is completely consumed. Such reactant thus limits the reaction and is referred to as the limiting reactant. Other reactant, or reactants, will present at the end of the reaction in excess. Once the limiting reactant is known, the theoretical and the percentage yields can be calculated.

In this experiment, you will carry out an aqueous reaction between sodium phosphate and barium chloride:

$$2 \text{ Na}_3 \text{PO}_{4(aq)} + 3 \text{ BaCl}_{2(aq)} \xrightarrow{\cdot} \text{Ba}_3(\text{PO}_4)_{2(s)} + 6 \text{ NaCl}_{(aq)}$$

the net ionic equation for the reaction will be:

$$2 PO_4^{3-}_{(aq)} + 3 Ba^{2+}_{(aq)} \rightarrow Ba_3(PO_4)_{2(s)}$$

You will be given a mixture of the two salts:  $Na_3PO_4.12H_2O$  (molar mass = 380.2 g/mol) and  $BaCl_2.2H_2O$  (molar mass = 244.2 g/mol) with unknown composition. You will then mix a weighed amount of the mixture in water to dissolve. Collect the precipitate, dry and weigh. At the same time the student will test the filtrate for the excess reactant whether it is phosphate ion or barium ion. Knowing the limiting reactant and the mass of

the precipitate, you will then calculate the masses of the two salts in  $t_{\mbox{\scriptsize h}_{\mbox{\scriptsize e}}}$  original mixture.

#### **Safety Precautions:**

- Barium salts are toxic, so clean your hands carefully after handling barium salts
- Use a towel in handling the beaker containing hot solution.

#### **Apparatus/Reagents Required**

Digital balance, filter funnel, watch glass, filter paper, 0.5 M Na<sub>3</sub>PO<sub>4</sub> solution, 0.5 M BaCl<sub>2</sub> solution, drying oven.

#### **Procedure**

#### A. Precipitation of Ba<sub>3</sub>(PO<sub>4</sub>)<sub>2</sub>

- 1. Weigh accurately (to the nearest 0.01 g) about 0.70 g of the unknown salt mixture. You can weigh it into a clean dry 250 ml beaker or on a piece of weighing paper and then transfer it into a 250 mL beaker.
- 2. Add about 150 mL of distilled water to the salt mixture. Stir the mixture with a stirring rod for about one minute. Allow the precipitate to settle.
- 3. Cover the beaker with a watch glass and heat the solution gently on a low flame of Bunsen burner, and keep the temperature in the range 80–90 °C for about 20 minutes. Rinse the glass rod used for stirring with distilled water before taking it out of the beaker. Do not allow the solution to boil.

4. Weigh accurately (to the nearest 0.01 g) and Record the mass of a piece of fine porosity filter paper, fold it twice and seal into the filter funnel with small amount of distilled water. Figure (4.1) illustrates how to fold the filter paper.

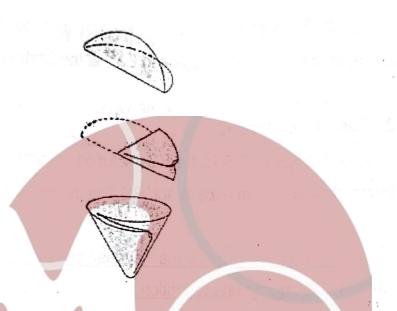


Figure 4.1. Folding the filter paper for gravity filtration.

Decant most of the hot solution through the filter paper. Do not allow the level of the liquid in the funnel to reach the top of the paper. Filter the precipitate and collect any precipitate remaining on the inside walls of the beaker by washing it with hot distilled water. Finally, rinse the precipitate with two 5 mL portions of hot water.

- 5. Label two 100 mL beakers (as I and II) and transfer two 50 mL volumes of the filtrate into each. Keep solutions I and II for part B.
- 6. Wash the precipitate while in the filter funnel with two 25 mL portions of acetone (or ethanol) to remove most of the water. Transfer your filter paper containing the precipitate to a watch glass labled with your name. Place the watch glass in a drying oven at 110 °C for 20 minutes. Cool and weigh the filter paper with the precipitate. You can

heat the precipitate again for 5 minutes for a constant weight. Record the final mass of the paper and the precipitate.

### B. Determination of the Limiting Reactant

#### 1. Test for excess PO<sub>4</sub><sup>3</sup>.

Add 2 drops of 0.5 M BaCl<sub>2</sub> solution to beaker I. If a precipitate forms, then PO<sub>4</sub><sup>3-</sup> is in excess and Ba<sup>2+</sup> is the limiting reactant.

#### 2. Test for excess Ba2+.

Add 2 drops of 0.5 M Na<sub>3</sub>PO<sub>4</sub> solution to beaker II. If a precipitate forms, then Ba<sup>2+</sup> is in excess and PO<sub>4</sub> <sup>3-</sup> is the limiting reactant.

Note: Some cloudiness may appear in both tests but one shows more definite precipitate formation.

ask ;; believe & recieve

# 4 Limiting Reactant

Name:	•
Name:	Section
Lab. Instructor	Date
Pre-Laborato	ry Questions
1. Na <sub>3</sub> PO <sub>4</sub> .12H <sub>2</sub> O and BaCl <sub>2</sub> .2H <sub>2</sub> O	are examples of compounds known as
hydrates. Calculate the molar mass	and Salah Book in the huderted
and anhydrous farms	ses of Na <sub>3</sub> PO <sub>4</sub> and BaCl <sub>2</sub> in the hydrated
and anhydrous forms.	
·····	
	N <sub>2</sub> O and 0.72 g Na <sub>3</sub> PO <sub>4</sub> .12H <sub>2</sub> O was
dissolved in water. Which of the	two reactants is the limiting reactant?
Calculate the mass of the precipi	tate formed, Ba <sub>3</sub> (PO <sub>4</sub> ) <sub>2</sub> .
•••••	
•	

4 Limiting Reactant

Name:	Section
Lab. Instructor	Date
Results and Calculation	ons
A. Precipitation of Ba <sub>3</sub> (PO <sub>4</sub> ) <sub>2</sub> :	
Unknown Number:	
Mass of salt mixture (m <sub>1</sub> )	g
Mass of filter paper (m <sub>2</sub> )	g
Mass of filter paper and Ba <sub>3</sub> (PO <sub>4</sub> ) <sub>2</sub> (m <sub>3</sub> )	g
B. Determination of the Limiting Re	eactant:
Limiting reactant in salt mixture is	
Excess reactant in salt mixture is	
Mass of Ba <sub>3</sub> (PO <sub>4</sub> ) <sub>2</sub> precipitated (m <sub>3</sub> -m <sub>2</sub> )	g
Number of moles of Ba <sub>3</sub> (PO <sub>4</sub> ) <sub>2</sub> precipitated (n <sub>1</sub> )	recieve   mol
(a) If the limiting reactant is BaCl <sub>2</sub> . 2H <sub>2</sub> O:	
Number of moles of BaCl <sub>2</sub> .2H <sub>2</sub> O reacted (n <sub>2</sub> )	mol
Number of moles of Na <sub>3</sub> PO <sub>4</sub> .2H <sub>2</sub> O reacted (n <sub>3</sub>	mol mol
Mass of BaCl <sub>2</sub> .2H <sub>2</sub> O reacted (m <sub>4</sub> )	g
Mass of Na <sub>3</sub> PO <sub>4</sub> reacted ( <b>m</b> <sub>5</sub> )	g
Mass of excess Na <sub>3</sub> PO <sub>4</sub> [ m <sub>1</sub> -(m <sub>4</sub> +m <sub>5</sub> )]	g
Mass percentage of BaCl <sub>2</sub> .2H <sub>2</sub> O	%

## (b) If the limiting reactant is Na<sub>3</sub>PO<sub>4</sub>.12H<sub>2</sub>O:

lom.
mol
9
9
g
%



#### **QUESTIONS**

1.	Calculate the mass of $Ba_3(PO_4)_2$ produced from the reaction of 0.78 g $Na_3PO_4.12H_2O$ with excess $BaCl_2.2H_2O$ . What is the purpose of heating
	the mixture in step 3 for 20 minutes?
	er Cakera
2.	What is the purpose of washing the precipitate with hot water in step 4?
	How would the reported percentage of the excess reactant be affected if the precipitate was not washed in this step?
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•••	
•••	

# 5 Determination of Acetic Acid in Vinegar

#### **Objectives**

You will be able to standardize a solution of NaOH and to determine the mass percent of acetic acid in vinegar.

#### Introduction

Vinegar is a dilute solution of acetic acid (CH<sub>3</sub>COOH) in water. The acid concentration of a known volume of vinegar can be measured by adding just enough basic solution of known concentration to react with it completely.

The experiment is divided into two parts. In Part A, a sodium hydroxide solution is prepared and standardized, that is, its concentration is accurately measured. In Part B this standard NaOH solution is used to measure the acid concentration of vinegar. The calculations in both parts in this experiment are based on the fact that when one substance exactly reacts with another, so that no excess of either remains, the same number of moles of each reactant is used. The principal equation for calculation is:

Number of moles of base = Number of moles of acid

For a solution, the number of moles of a solute (n) is the product of the molarity (in moles per liter) times the volume (in liters) that is:

$$n = M \times V(L)$$

whereas for a pure substance the number of moles (n) can be calculated by dividing mass by molar mass of that substance:

#### n = mass/ molar mass

All volume measurements are made by burets and pipets, which can be reported to the nearest 0.05 mL. The indicator phenolphthalein, which is colorless in acid and pink in base, marks the end point of the reaction. One drop of excess base turns the entire solution faint pink.

The primary standard substance (KHP) has the formula  $KHC_8H_4O_4$ . In this salt, there is only one acidic hydrogen per molecule and would react with the base. The equation for the neutralization reaction is:

#### **Safety Precautions:**

- The primary standard acidic substance potassium hydrogen phthalate (KHP) will be kept stored in an oven to keep moisture from adhering to the crystals. Use tongs or a towel to remove the KHP from the oven.
- Sodium hydroxide is extremely caustic and its dust is very irritating to the respiratory system. Do not handle the pellets with the finger. Wash hands after weighing the pellet. Work in a ventilated area and avoid breathing NaOH dust.
- Use a rubber bulb when pipeting. Never pipet by mouth.
- The unknowns to be used are acidic and may be irritating or damaging to the skin. Avoid contact with skin, and wash after using them.

### Apparatus/Reagents Required

50-mL burets, stand, clamps, 10-mL pipet, pipet bulb, ~0.3 M sodium hydroxide solution, white vinegar, primary "standard" grade potassium hydrogenphthalate (KHP), phenolphthalein indicator solution.

#### **Procedure**

#### A. Standardization of NaOH Solution

- Weigh a sample of potassium hydrogenphthalate (KHP) of about 0.2 g on a piece of paper and place it in a previously weighed 250-mL flask. Weigh the flask and its contents. (Do not waste too much time trying to get exactly 0.2 g as no advantage is gained).
- 2. Add 50 mL of distilled water to the sample in the flask and swirl until the acid is dissolved. Use swirling rather than a stirring rod to prevent loss of sample on the stirring rod.
- 3. Set up the titration apparatus as shown in Figure 5.1.
- 4. Obtain about 100 mL of the NaOH solution (~ 0.3 M) in a dry clean beaker.
- 5. Wash the buret with soap, water, distilled water and then rinse twice with about 10 mL of the NaOH solution to be used. Make sure that the NaOH solution comes in contact with the entire inner surface of the buret. Let the solution run out of the tip. This assures that the standard solution will not be diluted with distilled water adhering to the buret wall.
- 6. Fill the buret, making sure that no bubbles remain in the tip. A quick squirt of solution will usually sweep them out.
- 7. Record the initial buret reading to 0.05 mL.

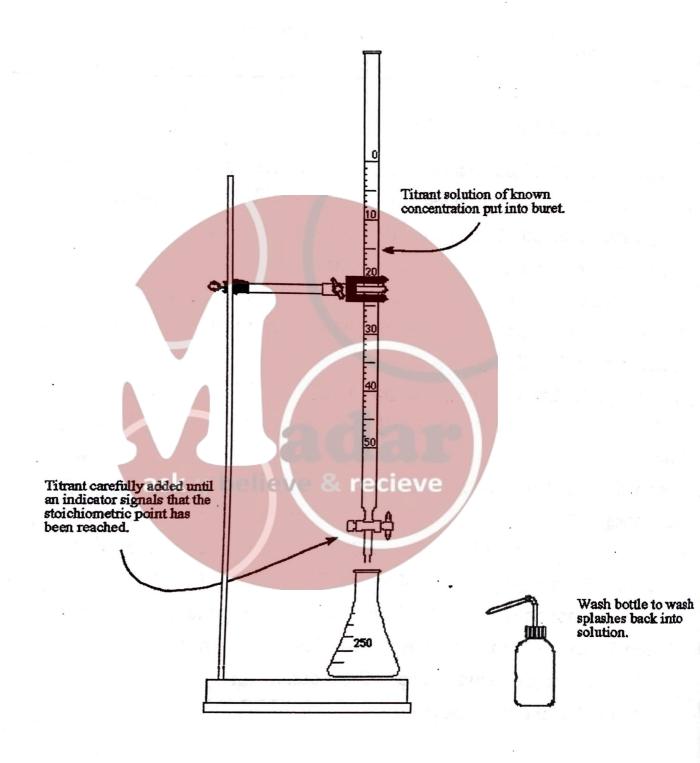


Figure 5.1. Setup for titration; stand, clamp, buret, and flask

- 8. Add 3 drops of phenolphthalein indicator to the KHP sample. Place the flask on a piece of white paper under the buret and allow the NaOH solution to run in slowly with swirling. Pink color will appear where there is a high local base concentration, but it will vanish quickly as the solution is swirled. Near the end point, the addition should be done one drop at a time with swirling between drops.
- 9. As soon as the color shows any tendency to persist, the rate of addition should be slowed. Do not add too much base. At the end point, one drop of base produces a permanent pale pink color. Record the final volume of the base used, estimating to the nearest 0.05 mL.
- 10. Repeat steps 6 through 9 for a second sample.
- 11. Calculate the molarity of the NaOH from each sample. If the two molarities do not agree to within 0.02, run a third sample.

#### B. Mass percent of acetic acid in vinegar

- Clean your pipet very well and rinse it with distilled water.
   Obtain a sample of vinegar solution (in a dry clean flask) from the store room; write down the sample number on your data sheet.
- 2. Rinse your pipet with small portions of the vinegar solution and discard the solution used for washing.
- 3. Use your 10 ml pipet to measure 10.00 mL of the vinegar solution, transfer it into a clean 250 mL Erlenmeyer flask and add one or two drops of phenolphthalein indicator. The solution should remain colorless.
- 4. Fill the buret with the standard NaOH solution and run some through the stopcock of the buret and make sure that no air bubbles remain. (Your instructor will demonstrate that for you).
- Place the flask under the buret and record the initial reading on the NaOH buret in the data sheet.

- 6. Start adding the base to the acid solution while stirring the acid solution constantly with a rotary motion of the flask. A spot of pink color will appear momentarily when a drop of base is added but it disappears as you stir.
- 7. Titrate each solution with care. If the end point is overshot, discard the whole solution and repeat the titration as described in Part A above.



# 5 Determination of Acetic Acid in Vinegar

Nam	Section
Lab.	Instructor Date
	Pre-Laboratory Questions
1.	Why was the standard NaOH solution not prepared by calculating the amount of solid NaOH needed for 100 mL of solution, weighing it accurately, and making it up to exactly 100 mL of total volume?
	Why not simply rinse the buret with distilled water rather than the solution to be used in it?
	ask ;; belleve & recieve
3.	Why does the volume of water added to potassium hydrogen phthalate not have to be measured carefully?
•••••	

# 5 Determination of Acetic Acid in Vinegar

Name:	Section
Lab. Instructor	Date

#### **Results and Calculations**

#### A. Standardization of NaOH Solution

	Trial (I)	Trial (II)
Mass of flask	g	g
Mass of Flask + KHP	g	g a
Mass of KHP	g	g
Molar mass of KHP	g/mol	g/mol
Moles of KHP ask	; believe mol	ecieve mol
Initial buret reading	mL	mL
Final buret reading	mL	mL
Volume of NaOH	mL	mL
Moles of NaOH	mol	mol
Molarity of NaOH	М	М
Average Molarity of NaOH		М

### B. Mass percent of acetic acid in vinegar.

	Trial (I)		Trial (II)
Volume of vinegar		mL	m(
Initial buret reading		mL	mt.
Final buret reading		mL	mΓ
Volume of NaOH used		mL	mί
Average molarity of NaOH, From P	Part A:		M
Moles of NaOH used		mol	m <sub>0</sub> l
Moles of CH <sub>3</sub> COOH in vinegar Reacted with the NaOH		mol	mol
Molarity of CH₃COOH in vinegar		М	M
Average molarity of acetic acid in vinegar		M	
Molar Mass of acetic acid, CH <sub>3</sub> COOH recieve			g/mol
Mass of CH <sub>3</sub> COOH per liter of vine	gar		g
Mass percent acetic acid in vinegar			%
(assume that vinegar has a density	/ of 1.00 g/mL)		

### **QUESTIONS**

1.	Calculate the percent error would have been in a titration that used
	32.75 mL of a solution if a bubble with a volume of 0.25 mL had
	been swept out of the tip during the titration.
2.	The label on the vinegar bottle used in this experiment claims that
	the vinegar contains 3 % acetic acid by weight. Use your results and
	a density of 1.0 g/mL to investigate this claim.
*	
	ask ;; belleve & recieve

### 6 The Neutralizing Capacity of Antacid Tablets

#### **Objectives**

You will be able to determine the amount of base in commercial antacids and amount of acid that can be neutralized by 1.00 g antacid tablet.

#### Introduction

When you eat or drink too much, your digestive system may generate too much acid, a condition called "heartburn". Heartburn and acid indigestion are primarily due to excess acid in the stomach. There are many preparations sold in our market to relieve these conditions, all of which act by neutralizing the excess acid in the stomach, and some may be more effective than others. In most of these products, other ingredients are added, flavoring agents and substances to soothe the walls and lining of the stomach, binders to make the tablet hold together and so forth. This will lead to low solubility of antacid in water. The active ingredients, however, are there to neutralize the excess acid in the stomach.

Among the most common active ingredients are: Mg(OH)<sub>2</sub> (the active ingredient in most of commercial antacid treatments.), NaHCO<sub>3</sub> and CaCO<sub>3</sub> and in some Al(OH)<sub>3</sub>. You are going to evaluate some of the commercially available antacids by determining the amount of HCl (stomach acid!) that one gram tablet will neutralize.

The reaction between the metal hydroxides and the stomach acid is an acid base reaction very similar to that in the previous experiment:

$$Mg(OH)_{2(s)} + 2HCI_{(aq)}$$
  $\longrightarrow$   $MgCI_{2(aq)} + 2H_2O_{(I)}$   
 $AI(OH)_{3(s)} + 3HCI_{(aq)}$   $\longrightarrow$   $AICI_{3(aq)} + 3H_2O_{(I)}$ 

Metal carbonates, which are the most common active component in antacids, neutralize the stomach acid by forming a salt, water and a gas.

$$CaCO_{3(s)} + 2HCI_{(aq)} \longrightarrow CaCI_{2(aq)} + H_2O_{(l)} + CO_{2(g)}$$

In this simple experiment you will determine the neutralizing capacity of a commercial antacid tablet. To test their capacity to neutralize acid, you will first dissolve an appropriate and measured amount of the sample in a simulated stomach environment. This is a solution containing a known quantity of HCl that will react with all of the antacid and still leave some extra HCl. Then you will determine how much of the original HCl remains by titrating it to neutrality with a standardized solution of NaOH. Simple subtraction will reveal how much of the acid was neutralized by the antacid tablet. This general method of analysis is called back-titration.

If  $n_A$  is the number of moles of HCl that you use for dissolving the antacid sample, and  $n_B$  is the number of moles of NaOH needed to backtitrate the excess HCl, then  $n_{sample}$ , the number of moles neutralized by the sample, is given by:

$$n_{\text{sample}} = n_A - n_B$$

For metal carbonates, boiling is an important element of this experiment. Dissolved carbon dioxide is converted into the weak acid, carbonic acid ( $H_2CO_3$ ) which reacts with sodium hydroxide (NaOH). Although the distilled water is free of most impurities, it does contain dissolved carbon dioxide that must be removed by boiling to give accurate titration results. All of the antacids that you titrate contain carbonates. When you acidify the antacid sample with standard HCI, the carbonates are converted into carbonic acid that must be boiled off in the form of  $CO_2$ .

#### Safety Precautions:

- Hydrochloric acid is corrosive. In the event of skin contact, wash well with water.
- Sodium hydroxide is caustic. In the event of skin contact, wash well with water.
- Follow your instructor directions for the handling and disposal of all chemicals and solutions.

#### Apparatus/Reagents Required

Digital balance, dispenser, stirring rod, 250 mL Erlenmeyer flasks, wash bottle, buret, mortar and pestle, 0.150 M sodium hydroxide solution, 0.150 M hydrochloric acid solution, bromothymol blue indicator, antacid tablets.

#### **Procedure**

- 1. Record all data and observations directly in your notebook in ink.
- 2. Obtain one tablet of the antacid from the store room. Crush the tablet using the mortar and pestle, or by placing it between two clean pieces of paper and pressing by a spatula.
- 3. Divide the crushed tablet in to two roughly equal portions.
- 4. Weigh the crushed portion accurately and transfer it quantitatively to a 250 mL Erlenmeyer flask. In case of using antacid powder, weigh accurately 0.15–0.20 g samples.
- 5. Add exactly 50.0 mL of 0.150 M hydrochloric acid solution to the flask-using dispenser (Figure 6.1) and dissolve the crushed tablet as

completely as possible. Part of the tablet (the starch binder)  $\psi_{||}$  probably not dissolve.



Figure 6.1. A 50-mL dispenser

- 6. Warm gently on a Bunsen burner (use stand, ring and wire gauze) to dissolve the sample and then boil for one or two minutes to get rid of CO<sub>2</sub>. Some components of the samples may remain undissolved, but these will not cause problems. Allow the flask to cool.
- 7. Add 2 drops of bromothymol blue acid-base indicator to the solution. (This acid-base indicator is yellow in an acidic solution and blue in a basic solution.)
- 8. Titrate the yellow acid solution with 0.150 M sodium hydroxide solution until the indicator just turns blue. This color change indicates the end point. (The blue color should remain for at least 30 seconds.)
- 9. Record the exact volume of 0.150 M sodium hydroxide solution required to neutralize the excess acid.
- 10. Repeat the above procedure (steps 4-9) with a second sample of antacid tablet.
- 11. Repeat the procedure if your results (mol of acid consumed per 1.00 g of tablet) are drastically different for the two runs.

# 6 The Neutralizing Capacity of Antacid Tablets

lame:			Section
ab. Inst	tructor		Date
	Pre-Laboratory	/ Ques	tions
1. Wr	rite a balanced equation for th		
		. (8)	
	• *		eact with 0.47 g of NaHCO₃?
		a	
	hy we do not use strong base blet?		
			·····

# 6 The Neutralizing Capacity of Antacid Tablets

Name:	Section		
Lab. Instructor	Date		
Results and Calculations			
Name of antacid tablet:			
Ingredients:			

7- 11	Trial (I)	Trial (II)
Mass of antacid sample	g	g
Volume of 0.150 M HCI solution	mL	mL
Moles of HCl (used to dissolve antacid)	mol	mol
Initial buret reading	mL mL	mL
Final buret reading	mL	mL
Volume of NaOH added	nemeye & rec	ieve mL
Moles of NaOH (used to titrate the excess acid )	mol	mol
Moles of excess HCl	mol	mol
Moles of HCl(needed to neutralize the antacid tablet)	mol	mol Harman
Neutralizing capacity of	mol HCl/g	mol HCl/g
antacid  Average (mol HCl/g antacid)		mol HCl/g antacid

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1.	Assume the concentration of stomach acid is 0.10 and the neutralizing capacity of the acid is 0.013 mol/g, calculate the volume of stomach acid needed to neutralize 1.0 g antacid tablet.
2.	A 0.333 g-sample of antacid was dissolved in 40.00 mL of 0.135 $_{ m M}$ HCl solution, then back-titrated to the end-point with 9.28 mL of a 0.0203 M NaOH solution.
	a) Calculate number of moles of acid in the original 40.00 mL of HCl?
	b) How many moles of base were used in the back titration of excess HCI?
	c) How many moles of excess HCI I?
	d) How many moles of HCl reacted with the antacid sample?

## Vitamin C Analysis

#### **Objectives**

You will be able to determine the amount of vitamin C in a vitamin C tablet or an unknown sample.

#### Introduction

The human body does not synthesize vitamins; therefore, the vitamins we need are gained only from the food we eat. Vitamin C can be obtained from citrus fruits, a variety of fresh fruits and vegetables. It is important to know that storage and processing causes vegetables to lose part of their vitamin C content. Cooking leaches water soluble vitamin C from the vegetables and high temperatures accelerates its degradation by air oxidation. Therefore, in order to maximize the intake of vitamin C, freshly harvested fruits and vegetables should be consumed.

Vitamin C (Fig. 7.1), also called ascorbic acid, is one of the more abundant and easily obtained vitamins. It is a white water-soluble acid that is a powerful reducing agent, which means that it readily undergoes

Figure 7.1. Structural formula of ascorbic acid (vitamin C)

oxidation, even by oxygen of the air. Even though vitamin C is an acid, its reducing properties will be used in this experiment to analyze its concentration in a tablet and in an unknown vitamin C sample. There are concentration in a tablet and in an unknown vitamin C with the selective other acids present in foods that would interfere with the selective other acids present in foods that would interfere with the oxidation of determination of vitamin C. The oxidation half-reaction for the oxidation of vitamin C is:

$$H_{2}C - CH$$
 $H_{2}C - CH$ 
 $H_{3}C - CH$ 
 $H_{4}C - CH$ 
 $H_{2}C - CH$ 
 $H_{5}C - CH$ 

In order to perform a vitamin C analysis, the sample containing vitamin C is dissolved in water and treated with a measured amount of iodate ion,  $IO_3$ , in an acidic solution containing an excess of I. Potassium iodate, a strong oxidizing agent, oxidizes I to  $I_2$ . Since an excess amount of iodide ion I present in solution,  $I_2$  binds to the iodide ion to form a red-brown soluble triiodide ion,  $I_3$ , a milder oxidizing agent than  $IO_3$ :

$$IO_{3\,(aq)}^{-} + 8I_{(aq)}^{-} + 6H_{(aq)}^{+} \rightarrow 3I_{3\,(aq)}^{-} + 3H_{2}O_{(I)}$$

For the analysis, vitamin C from the sample reduces a portion of the known amount of  $l_3$  generated in solution:

$$C_6 H_8 O_{6(aq)} \ + \ I_3^-{}_{(aq)} \ + \ H_2 O_{(I)} \ \rightarrow \ C_6 H_8 O_{7(aq)} \ + \ 3 \ I^-{}_{(aq)} \ + \ 2 \ H^+{}_{(aq)}$$

The remainder of the  $I_3^-$  (the excess) is titrated with a standard thiosulfate,  $S_2O_3^{\ 2^-}$ , solution, producing a colorless solution containing  $I^-$  and  $S_4O_6^{\ 2^-}$  ions:

$$2 S_2 O_3^2_{(aq)}$$
 + excess  $I_{3(aq)} \rightarrow 3 I_{(aq)}^7 + S_4 O_6^2_{(aq)}^7$ 

Therefore, the difference between the  $I_3^-$  generated from reaction of  $IO_3^-$  with  $I^-$  and that titrated as an excess from reaction with  $S_2O_3^{2^-}$  is a measure of the vitamin C content of the sample. The stoichiometric point is detected using starch as an indicator. Just prior to the disappearance of the red-brown triiodide ion,  $I_3^-$ , starch is added; this forms a deep-blue ion,  $[I_3^-$  starch]. The addition of the thiosulfate titrant is continued until the  $[I_3^-$  starch] ion is reduced to  $I^-$ ; the solution appears colorless at the end point.

#### **Apparatus/Reagents Required**

Vitamin C tablet, balance, 250 mL Erlenmeyer flask, 0.50 M H<sub>2</sub>SO<sub>4</sub>, sodium hydrogencarbonate, standard sodium thiosulfate solution, 0.10 M, standard potassium iodate solution, 0.01 M, potassium iodide, starch, and buret.

#### **Safety Precautions:**

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- Remember that vitamin tablets contain binders and other material that may be insoluble in water-do not heat in order to dissolve the tablet.
- The NaHCO<sub>3</sub> reacts in acidic solution to produce CO<sub>2</sub> gas, providing an inert atmosphere above the solution, minimizing the possibility of oxidation of vitamin C by the oxygen of the air.

#### **Procedure**

The sample is provided in the form of a tablet or a powder.

- 1. In case of using tablets from pharmaceutical preparations, read the label on the bottle to determine the approximate mass of vitamin C in each tablet.
- 2. Weigh accurately about 0.10 g of vitamin C.
- 3. Dissolve the sample in a 250 mL Erlenmeyer flask with 40 mL of 0.5  $_{
  m M}$  H $_2$ SO $_4$  and then add about 0.5 g NaHCO $_3$ .
- 4. Add exactly 50.0 mL of the standard 0.01 M KIO<sub>3</sub> solution (using a dispenser) into the sample solution and add about 0.5 g of NaI (or KI).
- 5. Add about 5 mL of 0.5 M H<sub>2</sub>SO<sub>4</sub>, and 0.1 g of NaHCO<sub>3</sub> to the flask.
- 6. Fill a 50 mL buret with a standard 0.1 M Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> solution. Drain the air bubbles from the buret.
- 7. Titrate the excess I<sub>3</sub> in the sample with the standard 0.10 M Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> solution. When the red-brown solution (due to I<sub>3</sub>) changes to a pale yellow color, add 2 mL of starch solution (the solution turns deep blue) While stirring, continue the titration slowly until the blue color disappears. Read and record the final buret reading to the nearest 0.05 mL
- 8. Repeat the analysis to complete two titrations. The result of the titration should be within ± 0.1 mL
- Dispose the remaining KIO<sub>3</sub> in the "waste oxidizing agent" container.
   Dispose the excess Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> solution in the "waste reducing agent" container.

7 Vitamin C Analysis

Nam	ne: Section
Lab.	Instructor Date
	Pre-Laboratory Questions
1.	Explain why cooked fruits and vegetables have lower vitamin content than fresh fruits and vegetables.
	What are the oxidizing agents in this experiment?
	What will be the color change of the starch indicator that indicates the end of the titration in this experiment?
	Vitamin C is an acid (ascorbic acid) and a reducing agent. Which property is utilized for this analysis in this experiment?
	If 39.50 ml of a vegetable juice contains 45% of the recommended daily allowance of vitamin C (equal to 60 mg). How many milliliters of the vegetable juice will provide 100% of the recommended daily allowance?

## 7 Vitamin C Analysis

Name:	Section
Lab. Instructor	Date

### **Results and Calculations**

	Trial (I)	Trial (II)
Mass of sample	g	g
Volume of 0.010 M KIO₃ added	mL	mL
Moles of IO <sub>3</sub> added	mol	mol
Moles of I <sub>3</sub> generated, total	mol	mol
Initial buret reading	mL	mL
Final buret reading	mL	mL
Volume of 0.10 M Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> added	mL	mL
Molar concentration of Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>	mol/L	mol/L
Moles of S <sub>2</sub> O <sub>3</sub> <sup>2</sup> - added	a recieve mol	e mol
Moles of I <sub>3</sub> <sup>-</sup> reduced by S <sub>2</sub> O <sub>3</sub> <sup>2</sup> -	mol	mol
Moles of I <sub>3</sub> <sup>-</sup> reduced by C <sub>6</sub> H <sub>8</sub> O <sub>6</sub>	mol	mol
Moles of C <sub>6</sub> H <sub>8</sub> O <sub>6</sub> in the sample	, mol	mol
Mass of C <sub>6</sub> H <sub>8</sub> O <sub>6</sub> in the sample	g	g
Mass percent of C <sub>6</sub> H <sub>8</sub> O <sub>6</sub> in the sample	%	%
Average percent of C <sub>6</sub> H <sub>8</sub> O <sub>6</sub> in the sample		%

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wi			10

1. A 25.0 mL volume of 0.010 M KIO <sub>3</sub> , containing an excess of KI, is added to a 0.346 g sample of a lemon solution containing vitamin C. The red brown solution, caused by the presence of excess I <sub>3</sub> , is titrated to a colorless starch end point with 7.70 mL of 0.100 M Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> .
a. Calculate the moles of 13 that are
b. How many moles of I <sub>3</sub> reacted with the 0.100 M Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> in the titration?
uuauon:
c. How many moles of I <sub>3</sub> had reacted with the vitamin C in the lemon sample?
ask, a recieve
d. Calculate the moles and grams of vitamin C in the sample.
e. Calculate the percent (by mass) of vitamin C in the lemon sample.
<ol> <li>If the blue color does not appear when the starch solution is added during the titration, should you continue titrating or discard the sample's Explain.</li> </ol>

## 8 Bleach Analysis

### **Objectives**

You will be able to determine the concentration of hypochlorite ion (CIO ) in bleach solution by redox titration.

#### Introduction

The active ingredient in most chlorine bleaches is sodium hypochlorite, NaOCI, which is an oxidizing agent. It kills germs and also decolorizes many stains and dyes. The strength of bleach solution is rated by a term called available CI<sub>2</sub> or as mass percent of NaCIO that will be determined in this experiment.

Substances (fabrics and stains) have a color due to light absorption. Upon oxidation, stains and most colors disappear. The hypochlorite ion in bleach removes the color of stains by oxidation. That is to say, the hypochlorite ion acts as an oxidizing agent:

$$CIO_{(aq)}^{\dagger} + 2H_{(aq)}^{\dagger} + 2e^{\dagger} \rightarrow CI_{(aq)}^{\dagger} + H_2O_{(l)}^{\dagger}$$

In this experiment, the oxidation-reduction analysis of bleach involves the reaction of the hypochlorite ion as an oxidizing agent, with iodide ion. The iodide ion ( $I^-$ ) is oxidized to  $I_2$ , which forms a triiodie ion ( $I_3$ ) in the presence of excess  $I^-$ .

$$CIO_{(aq)}^{-} + 3I_{(aq)}^{-} + 2H_{(aq)}^{+} \rightarrow I_{3(aq)}^{-} + CI_{(aq)}^{-} + H_{2}O_{(I)}$$

The triiodide ion  $(I_3)$  generated in the reaction, is titrated with a standard sodium thiosulfate  $(Na_2S_2O_3)$  solution until the red-brown color of

 $I_3$  nearly disappears. Then, starch which forms deep blue color with  $I_3$  is added as an indicator to detect the end point

$$I_{3(aq)} + 2 S_2 O_3^{2(aq)} \rightarrow 3 I_{(aq)} + S_4 O_6^{2(aq)}$$

The chemical analysis for hypochlorite ion in a bleach solution is summarized in the net ionic equation:

$$\text{CIO}^{\text{-}}_{(aq)} + 2 \, \text{S}_2 \text{O}_3^{\, 2^{\text{-}}}_{(aq)} + 2 \, \text{H}^{^{\text{+}}}_{\, (aq)} \, o \, \text{CI}^{\text{-}}_{(aq)} + \text{S}_4 \text{O}_6^{\, 2^{\text{-}}}_{(aq)} \, + \, \text{H}_2 \text{O}_{(I)}$$

This equation serves as the basis for determining the strength  $_{\mbox{\scriptsize of}}$  bleaching agent in this experiment.

Since thiosulfate solution might be oxidized by oxygen or by bacterial action, it is necessary to standardize it just before use against a more stable **primary standard**, such as potassium dichromate.

$$Cr_2O_7^{2-}_{(aq)} + 9I_{(aq)}^- + 14H_{(aq)}^+ \rightarrow 2Cr_{(aq)}^{3+} + 3I_{3(aq)}^- + 7H_2O$$

Standardization of sodium thiosulfate involves the reaction of an accurately known amount of the dichromate with excess potassium iodide and then titrating the resulting triiodide  $l_3$ , with the thiosulfate using starch as an indicator.

## **Safety Precautions:**

- Although a common household item, bleach is a dangerous material that causes burns and is toxic.
- Potassium iodide and iodine are toxic.
- Avoid contact with chemicals.
- Wash spills immediately with large amounts of water and Wash your hands after the experiment.

### Apparatus/Reagents Required

Digital balance, dispenser, 250 mL Erlenmeyer flasks, wash bottle, buret, standard potassium dichromate solution ~0.010 M, sodium thiosulfate solution, 6 M hydrochloric acid solution, potassium iodide, starch, dilute commercial bleach solution.

#### **Procedure**

#### A. Standardization of thiosulfate solution

- 1. Weigh out roughly 1.0 g potassium iodide (KI)
- 2. Quickly dissolve the KI in about 25 mL of distilled water in a 250 mL Erlenmeyer flask. Then add about 5 mL of 6M HCI. (If the solution turns yellow discard and begin with a cleaner flask).
- 3. To the above acidified solution add from the dispenser 25.0 mL of 0.010 M K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> solution. Cork the flask and place it in the dark for about 5 minutes to complete the redox reaction. The solution should be yellow to red in color.
- 4. Rinse the buret with 3–5 mL thiosulfate solution and then fill it with thiosulfate solution. Be sure to clear the buret tip and record the buret reading. Titrate the solution in the flask till the solution becomes yellow. At this stage most of the iodine in the flask is titrated, but not all of it.
- 5. As the solution in the flask becomes yellow, add 3 mL of starch indicator. The color of the solution should turn blue. If the color does not turn blue it means that you have already passed the end point in the titration. In such a case you need to repeat the experiment.
- Continue the titration carefully until the blue color is turned to yellow green due to presence of Cr<sup>3+</sup>. Record the final buret reading.
- 7. Repeat steps 1-6 for the second trial.

## B. Analysis of bleach solution

- Weigh out roughly 1.0 g potassium iodide (KI) 1.
- Quickly dissolve the KI in about 25 mL of distilled water in a 250 mL Erlenmeyer flask. Then add about 5 mL of 6 M HCl. (If solution turns 2. yellow discard and begin with a cleaner flask).
- To the above-acidified solution add from the dispenser 25.0 mL of diluted bleach solution. Cork the flask and place it in the dark for about 3. 5 minutes to complete the redox reaction. The solution should be yellow to red in color.
- Fill the buret with thiosulfate solution. Be sure to clear the buret tip 4. and record the initial buret reading. Titrate the solution in the flask. At this stage most of the iodine in the flask is to be titrated, but not all of it, before addition of the starch indicator.
- As the solution in the flask becomes yellow, add 3 mL of starch indicator. The color of the solution should turn blue. If the color does not turn blue it means that you have already passed the end point in the titration. In such a case you need to repeat the experiment.
- Continue the titration carefully until the blue color is turned to colorless. Record the buret reading.
- 7. Repeat steps 1-6 for the second trial.

8 Bleach Analysis

Name:	Section
_ab. Instructor	
Pre-Laboratory	
1. Write balanced equation(s) for the of sodium thiosulfate.	reactions involved in standardization
,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,	
2. In today's chemical analysis of oxidized by the hypochlorite ion?	bleach solution, what substance is
3. What are the formulas of sodium	
ask.,,hadi	
4. Does sodium thiosulfate serve	as an oxidizing agent or as a
reducing agent? What does sodiu	m thiosulfate reduce?
	••••••

## 8 Bleach Analysis

Name:	Section
4.0	Date

## **Results and Calculations**

## A. Standardization of thiosulfate solution

	Trial (I)	Trial (II)
Volume of 0.010 M K <sub>2</sub> Cr <sub>2</sub> O <sub>7</sub> solution	mL	mL
Initial buret reading	mL	mL
Final buret reading	mL	mL
Volume of Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> solution	mL	mL
Number of moles of K <sub>2</sub> Cr <sub>2</sub> O <sub>7</sub>	k ;; believe emol	cieve / mol
Number of moles of Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>	mol	mol
Molarity of Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>	M	M
Average molarity of Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>		М

## B. Analysis of bleach solution

		<b>-1.1</b> (11)
	Trial (I)	Trial (II)
Dilution Factor		
Volume of bleach solution	mL	mL .
Initial buret reading	mL	mL
Final buret reading	mL	mL
Volume of Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> solution	· mL	mL
Number of moles of Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> oxidized	mol	· mol
Number of moles of CIO <sup>-</sup> reduced	mol	mol
Molarity of diluted bleach solution	M	M
Molarity of original bleach solution	& recieve	М
Average molarity of original bleach solution		M
Mass% of NaClO(assume density of bleach solution = 1.00g/mL)		%

## QUESTIONS

1. A 10.0 mL bleach sample is diluted to 100 mL in a volumetric flask. A 25.0 mL of this solution is analyzed according to the procedure in this experiment. If 12.5 mL of 0.30 M Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub> is needed to reach the end point, calculate the mass percent of NaClO in the original sample? (Assume the density of bleach solution is 1.084 g/mL).			
2. A bleach solution that is 6.58% NaClO (density= 1.10 g/mL) was diluted			
to 0.056 M concentration. Calculate dilution factor.			
(0 0.050 M concentration. Calculate dilution factor.			
***************************************			
***************************************			
***************************************			

## 9 Molar Mass of a Volatile Liquid

#### **Objectives**

You will be able to determine the molar mass of a volatile liquid using Dumas method.

#### Introduction

Chemical and physical methods for determining atomic and molecular weights or molar masses have historically been important as a way of analyzing and categorizing new materials. The modern laboratory is generally equipped with instrumentation which makes many of these methods obsolete. However the principles upon which the older methods were based are not insignificant and may from the foundation for the prediction of physical and chemical properties and behaviors of substances.

The classic Dumas\* method for determining the formula weight of a volatile liquid is a case in point. Avogadro proposed, as early as the mid-1800's, that equal volumes of gases measured under identical conditions would contain equal numbers of gas particles. With an established relative atomic mass scale it was possible to describe a constant volume, which would contain a mass of one mole of a gaseous element or compound under fixed conditions, as the molar volume. At STP this volume is 22.4 L for an ideal gas.

<sup>\*</sup>Dumas method: is a classical method for determining molar mass of volatile liquid

An understanding of the various gas laws allows for an application of the various gas laws allows for an application of the various gas laws allows for an application of the various gas laws allows for an application of the various gas laws allows for an application of the various gas laws allows for an application of the various gas laws allows for an application of the various gas laws allows for an application of the various gas laws allows for an application of the various gas laws allows for an application of the various gas laws allows for an application of the various gas laws allows for an application of the various gas laws allows for an application of the various gas laws allows for an application of the various gas laws allows for an application of the various gas laws allows for an application of the various gas laws allows for an application of the various gas laws allows for an application of the various gas laws allows for an application of the various gas laws allows for an application of the various gas laws allows for an application of the various gas laws allows for an application of the various gas laws allows for an application of the various gas laws allows for an application of the various gas laws allows for an application of the various gas laws allows for an application of the various gas laws allows for an application of the various gas laws allows for an application of the various gas laws allows for an application of the various gas laws allows for an application of the various gas laws allows for an application of the various gas laws allows for an application of the various gas laws allows for an application of the various gas laws allows for an application of the various gas laws allows for an application of the various gas laws allows for allows gas laws allows for allows gas laws gas l

Weighing a sample of gas is relatively simple. These two pieces of information (volume and mass) are the minimum requirements for a molar information (volume and mass) are the minimum requirements for a molar information (volume and mass) are the minimum requirements for a molar information (volume and mass) are the minimum requirements for a molar information (volume and mass) are the minimum requirements for a molar mass determination. In the Dumas method a volatile liquid is heated to a known temperature (above its boiling point) and allowed to escape from a known temperature dispointly at the container container through a tiny hole. Once the liquid has vaporized, the container is cooled to room temperature. Gradually the vapor, which remained in the is cooled to room temperature, condenses to a liquid and its mass is container at the higher temperature, condenses to a liquid and its mass is then measured. If the volume of the container is known along with the high temperature, the room pressure can be used (because the system is open to the atmosphere through the orifice) to calculate moles. From the mass and moles, the molar mass can be determined. The vapor is assumed to behave ideally at the temperature and pressure at which it occupies the container.

When the liquid is present, it vaporizes to some extent depending on by its vapor pressure at room temperature. Since the internal pressure is equalized with the room pressure and the number of gas particles remains constant if the temperature and pressure remain constant. Thus, when the mass of container is weighed again, the mass of the condensed liquid is determined by difference. The result is the mass of the condensed liquid which is too small.

From the mass of condensed vapor, the molar mass of unknown liquid can be calculated:

$$PV = \frac{m}{M} RT$$

#### **Safety Precautions:**

- The unknown volatile liquid could be highly flammable.
- Avoid burning your fingers while removing the flask from the water bath.

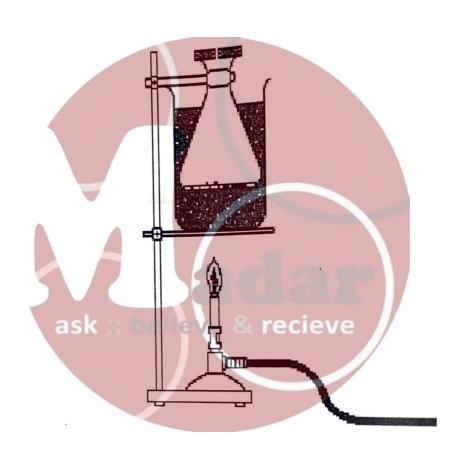
### Apparatus/Reagents Required

Aluminum foil, rubber band, boiling stones, 600 mL beaker, thermometer, 150 mL flask, volatile liquid, digital balance.

#### **Procedure**

- Obtain a 150 mL Erlenmeyer flask and ensure that it is completely dry before starting the experiment. If it is not completely dry, rinse the flask with acetone and leave it to dry.
- 2. Determine the total mass of the dry Erlenmeyer flask, a rubber band, and a square of aluminum foil.
- 3. Add about 7 mL of a volatile liquid into the dry flask.
- 4. Cover the mouth of the flask with the small aluminum foil followed with larger one. Secure it with the rubber band.
- 5. Make a small hole through both aluminum foils.
- 6. Clamp the flask assembly into the beaker so that the flask is as far down as possible in the beaker (Fig. 9.I). Fill the beaker with water as shown in figure 6.1 and then add few pieces of boiling stones to the water in the beaker. Heat at the boiling point of water until liquid is no longer visible in the flask, continue heating for another 5 minutes. Record the boiling point of water to the nearest ±0.5 °C. Also, record the current barometric pressure.

- Remove the flask and allow it cool to room temperature. Dry the outside of the flask and weigh it along with its contents, the aluminum outside of the flask and weigh it along with its contents, the aluminum outside of the flask and weigh it along with its contents, the aluminum outside of the flask by totally filling the flask.
   Measure the volume of the Erlenmeyer flask by totally filling the flask.
- 8. Measure the volume of the English water to a graduated cylinder. If time with water and transferring the water to a graduated cylinder. If time allows, repeat the experiment.



**Figure 9.1.** Setup for the determination of molar mass of a volatile liquid.

## 9 Molar Mass of a Volatile Liquid

Name:	Section
Lab. Instructor	Date
Pre-Laboratory Quest	tions
Dumas method assumes the gas behave when gases behave ideally?	
2. The vapor from an unknown volatile liquid Erlenmeyer flask at 98.5 °C and 745 torr	
0.841 g. a) What is the molar mass of the volatile	e liquid?
ask;; believe	& recieve
b) What is the density of vapor at STP?	<b>}</b>

## 9 Molar Mass of a Volatile Liquid

Name:	Section
Lab. Instructor	Date

#### **Results and Calculations**

	Trial (I)		Tria	I (II)
Boiling point of water		°C	* * *	°C
Atmospheric pressure	n	nmHg		mmHg
Mass of empty flask		g		g
Mass of flask and condensed vapor		g		g
Mass of condensed vapor		g		g
Volume of flask		mL	90	mL
Boiling point of water	pelleve & r	K	ve	K
Atmospheric pressure		atm		atm
Volume of flask		L		L
Gas constant (R)			0.0821	L.atm /mol.K
Molar mass of unknown		g/mc	ol .	g/mol
Average molar mass			1	g/mol

### **QUESTIONS**

•	OLO 11010
	If the outside of the flask is not dried after vaporizing the liquid, will the
	If the outside of the flask is not dried and too low? Explain.  calculated molar mass be too high or too low?
1.	If the outside of the flask is not too low? Explain.
•	he too high of too
	calculated molar mass by
	Outout to the second se
	The state of the s
	***************************************
_	and the superimental data: Mass of condensed vapor =
2.	Consider the following experimental data: Mass of condensed vapor =
	0.395 g at 96 °C and 755 mm Hg occupies 137 mL. What is the molar
	mass of the liquid?
	ask - balla & raciova
	•••••••••••••••••••••••••••••••••••••••

## 10 Thermochemistry and Hess's Law

#### **Objectives**

You will be able to determine the heat capacity of a simple coffeecup calorimeter. The same calorimeter will be used to measure the enthalpy change when solutions of sodium hydroxide (NaOH) and hydrochloric acid (HCI) are mixed. Finally you will apply Hess's law of heat summation to calculate the enthalpy change for the reaction of ammonium chloride (NH<sub>4</sub>CI) and sodium hydroxide (NaOH).

#### Introduction

Chemical and physical changes are always accompanied by a change in energy. Most commonly, this energy change is observed as a flow of heat energy either into or out of the system under study. Heat flows are measured in an instrument called a calorimeter.

The calorimeter you will use in this experiment is made of heat-insulating plastic foam material. It consists of two nested plastic foam coffee cups and a cover, with thermometer and stirring wire inserted through holes punched in the cover. Although the plastic foam material from which your calorimeter is constructed does not conduct heat well, it does still absorb some heat. In addition, a small quantity of heat may be transferred to or from the metal wire used for stirring the calorimeter's contents, or to the glass of the thermometer used to measure temperature changes. Some heat energy may also be lost through the openings for these devices. Therefore, the calorimeter will be *calibrated* (i.e. to find its heat capacity) using a known system before it is used in the determination of the heat flows in unknown systems.

When the temperature of a sample of any substance changes, the quantity of heat, q, involved in the temperature change is given by:

$$q = mass x specific heat x \Delta t$$

or

$$q = m \times S \times \Delta t$$
 ... (1)

Where m is the mass of the substance,  $\Delta t$  is the temperature change, and S is the specific heat of the substance. The specific heat represents the quantity of heat required to raise the temperature of one gram of the substance by one degree Celsius. Specific heats are quoted in units of joules per gram per degree, J/g.°C. To determine the calorimeter constant for the simple coffee-cup apparatus that is to be used in this experiment, we will make use of the conservation of energy principle: Energy cannot be created or destroyed during a process, but can only be transformed from one form to another or transferred from one part of the universe to another. Measured quantity of cold water is placed in the calorimeter to be calibrated and is allowed to come to thermal equilibrium with the calorimeter. Then a measured quantity of warm water is added to the cold water in the calorimeter. Since the energy contained in the hot water is conserved, we can make the following accounting of energy:

The amount of heat absorbed by the calorimeter,  $q_{\text{calorimeter}}$ , can be written as:

$$q_{calorimeter} = C_{calorimeter} \times \Delta t$$
 ... (3)

in which  $\Delta t$  is the temperature change undergone by the calorimeter, and  $C_{calorimeter}$  is the calorimeter constant, which represents the number of joules of heat required to warm the calorimeter by 1  $^{\circ}$ C.

Applying Equations (1) and (3) to account for the energy transferred in the system as given in Equation (2), we get:

$$(m \times S \times \Delta t)_{warm \ water} = [(m \times S \times \Delta t)_{cold \ water} + (C \times \Delta t)_{calorimeter}]$$
 ...(4)

since the specific heat of water is effectively constant over the range of temperatures in this experiment ( $S_{water} = 4.184 \text{ J/g.}^{\circ}\text{C}$ ), determination of the calorimeter constant is possible by making two measurements of mass and two measurements of changes in temperature.

The chemical reactions that will be considered during this experiment are given in the following equations:

$$\begin{array}{ccccccc} \text{NaOH} + & \text{HCI} & \rightarrow & \text{NaCI} + \text{H}_2\text{O} \\ \text{NH}_3 & + & \text{HCI} & \rightarrow & \text{NH}_4\text{CI} \\ \text{NaOH} + & \text{NH}_4\text{CI} & \rightarrow & \text{NaCI} + & \text{NH}_3 + & \text{H}_2\text{O} \end{array}$$

You may have noted that there is a relationship among these equations. If the first two equations are manipulated and then combined by addition, the third equation is generated. This relationship provides the basis for using Hess's law of heat summation. You will find that you can predict the enthalpy change for the third reaction by combining the enthalpy changes for the first and second reaction.

The heat evolved or absorbed during the first two reactions will be measured with a coffee-cup calorimeter (Fig. 10.1). The enthalpy change for the third reaction will be calculated using Hess's law.

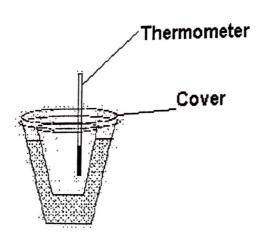


Figure 10.1. Simple calorimeter

The three chemical reactions in this experiment are examples of acid-base reactions. Although you will learn much more about these terms acid-base reactions. Although you will learn to identify the bases and during this course, it will be useful for us at least to identify the bases and the acids in this experiment.

### **Safety Precautions**

- Wear safety glasses at all times while in the laboratory.
- Use towels to protect your hands when handling hot glassware.
- CAUTION: Hydrochloric acid, sodium hydroxide and ammonia can cause chemical burns in addition to ruining your clothing. If you spill one of these solutions on you, wash the contaminated area spill one of these solutions on your laboratory instructor, thoroughly and report the incident to your laboratory instructor. You may require further treatment.

### Apparatus/Reagents Required

Plastic foam coffee cups and covers, thermometer, one-hole papers punch. 1.0 M HCL, 1.0 M NaOH, 1.0 M NH<sub>3</sub>

#### **Procedure**

### A. Determination of the Heat Capacity of Calorimeter

Work with a partner.

- 1. Record all data and observations directly in your notebook in ink.
- 2. Nest two similar-sized plastic foam coffee cups (If thick-walled styrofoam cups are available, you can use only one cup) for use as

- the calorimeter. If the cups have been rinsed with water, dry them out. Obtain a plastic lid that tightly fits the coffee cups. Using the paper punch, make one small hole near the center of the lid for the thermometer.
- 3. Insert the thermometer as indicated in Figure 10.1. Since the density of water over the range of temperatures in this experiment is very nearly 1.00 g/mL, the amount of water to be placed in the calorimeter can be more conveniently measured by volume.
- 4. With a graduated cylinder, place 75.0 ml of **cold** water into the calorimeter. Cover the calorimeter with the plastic lid and insert both the thermometer and stirrer in the apparatus.
- 5. Measure 75.0 mL of water into a clean, dry 250 mL beaker, and heat the water to 60-70 °C. Stir the water with a glass rod occasionally during the heating to ensure that the temperature is as uniform as possible. While the water is heating, monitor the temperature of the cold water in the calorimeter for 2-3 minutes to make certain that it has become constant. Record the temperature of the cold water in the calorimeter to the nearest 0.2 °C.
- 6. When the water being heated has reached 60-70 °C, use tongs or a towel to remove the beaker from the heat source. Allow the beaker to stand on the laboratory bench for 2-3 minutes with stirring the water occasionally during this time period. After the standing period, record the temperature of the hot water to the nearest 0.2 °C.
- 7. Quickly remove the lid from the calorimeter, and pour the hot water into the cold water in the calorimeter. Immediately replace the lid of the calorimeter, stir the water for 30 seconds to mix, and begin monitoring the temperature of the water in the calorimeter.
- 8. Record the *highest* temperature reached by the water in the calorimeter, to the nearest 0.2 °C. From the masses (mass = volume x 1.00 g/mL) of cold and hot water used, and from the two

- temperature changes, use equation (4) to calculate the caloring temperature changes, use equation (4) to calculate the caloring temperature changes, use equation (4) to calculate the caloring temperature changes, use equation (4) to calculate the caloring temperature changes, use equation (4) to calculate the caloring temperature changes, use equation (4) to calculate the caloring temperature changes, use equation (4) to calculate the caloring temperature changes.
- 9. Repeat the experiment to obtain additional value for the calonimeter constant. Use the mean value of the two determinations of the calonimeter constant in Part (B) of this experiment.

### B. Heat of Acid-Base Reaction

- Work, as in Part (A), with a partner.
- Use the coffee-cup calorimeter that you determined its constant in Part (A).
- Obtain 50.0 mL of the 1.0 M solution of HCL in a clean, dry graduated cylinder. Obtain 50.0 mL of the 1.0 M solution of NaOH in another clean, dry graduated cylinder.
- 4. Measure the temperature of each of these solutions, using the same thermometer. However, rinse the thermometer and dry it after the first measurement. The temperatures should agree to within  $\pm$  0.2 °C.
- 5. Record the mean temperature. This is the initial temperature.
- Add carefully the base to the calorimeter and then add the acid.Immediately place the cover on the calorimeter and begin stirring.
- 7. Record the highest temperature reached by the mixture (to the nearest 0.2 °C).
- 8. From the change in temperature undergone by the mixture upon reaction, the total mass (volume) of the combined solutions, and the calorimeter constant (determined in Part (A)), calculate the quantity of heat that flowed from the acid-base reaction into the solution and calorimeter.
- Calculate the number of moles of the acid or base and the number of moles of water produced when 50.0 mL of 1.0 M HCl reacts with 50.0 mL of 1.0 M NaOH.

- $_{10.}$  Calculate  $\Delta H$  in terms of the number of kilojoules of heat energy transferred when one mol of water is formed in the neutralization of aqueous HCI with aqueous NaOH. If time permits, repeat the determination of  $\Delta H$  for the HCI/NaOH reaction using the same acid and base and calculate a mean value for  $\Delta H$  of the reaction.
- 11. Repeat steps (2-8) but using 50.0 mL of 1.0 M ammonia solution instead of 1.0 M NaOH solution.
- $_{12}$ . Calculate the number of moles of the acid or base and the number of moles of NH $_4$ Cl produced when 50.0 mL of 1.0 M HCl reacts with 50.0 mL of 1.0 M NH $_3$ .
- transferred when one mol of NH $_4$ CI is formed in the neutralization of aqueous HCI with aqueous NH $_3$ . If time permits, repeat the determination of  $\Delta H$  for the HCI/NH $_3$  reaction using the same acid and base, and calculate a mean value for  $\Delta H$  of the reaction



## 10 Thermochemistry and Hess's Law

Name:	Section	
Lab. Instructor	Date	
	tory Questions	
	tory Questions	
1. What is the definition of t	ne joule in terms of the basic SI units?	
	·····	
2. In a calorimeter calibration	on experiment, a sampe of 51.682 g of wa	otor
at 55.2 °C is added to	calorimeter containing 50.220 g of water	11C1
23.5 °C. After stirring a	nd waiting for the system to equilibrate,	the
final temperature reac	ned is 36.6 °C. Calculate the calorime	me
constant.	led is 36.6 °C. Calculate the calorimo	eter
7-a-4-a		•
		••
		••
ask	; belleve & recieve	••
		••
		••
3 Give chemical equations	or the reactions that will be a larger	
	or the reactions that will occur during this	ì
Experiment		
••••••••		•••

## 10 Thermochemistry and Hess's Law

Name:	Section
Lab. Instructor	Date

## **Results and Calculations**

## A. Determination of a Calorimeter Constant.

White is		
	Trial (I)	Trial (II)
Mass (or volume) of cold water	g	g
Temperature of cold water	°C	°C
Mass (or volume) of hot water	g	g
Temperature of hot water	°C	°C
Final temperature reached	°C	°C
Increase of cold water temperature (ΔT)	°C	°C
Decrease of hot water temperature (ΔT)	& recie <sup>°</sup> Ce	°C
Heat lost by hot water	J	J
Heat gained by cold water	J	J
Heat gained by calorimeter water	J	J
Calorimeter constant (C)	J/°C	J/°C
Average value of calorimeter constant		J/°C

## B. Heat of Acid/Base Reactions

1. HCI/NaOH	Trial (I)	Trial (II)
	mL	m
Volume of 1.0 M NaOH used	°C	
Initial temperature of NaOH	C	00
Volume of 1.0 M HCl used	mL	ml
Initial temperature of HCI	°C	00
Final temperature reached	°C	00
Total volume of mixture	mL	mL
Total mass of mixture	g	paril 9
Temperature change, Δt	°C	00
Average value of the Calorimeter constant (C)		J/°C
Heat gained by the solution*	J	Fig.
Heat gained by the calorimeter	J	· · · · · · · · · · · · · · · · · · ·
Heat of the reaction	& recieve	J
Moles of NaOH reacted	mol	mol
Moles of HCl reacted		
Moles of water produced	mol	mol
ΔΗ	mol	mol
Average value of ΔH	kJ/mol H₂O	kJ/mol H₂0
Literature value of ΔH		kJ/mol H <sub>2</sub> 0
31 411		- 55.9 kJ/mol H <sub>2</sub> 0

<sup>\*</sup> Assume density of solution = 1.00 g/mL and specific heat of solution =

#### 2. HCI/NH<sub>3</sub>

	Trial (I)	Trial (II)
Volume of 1.0 M NH <sub>3</sub> used	mL	mL
Initial temperature of NH <sub>3</sub>	°C	°C
Volume of 1.0 M HCl used	mL	mL
Initial temperature of HCI	°C	°C
Final temperature reached	°C	°C
Total volume of mixture	mL	mL
Total mass of mixture	9	g
Temperature change, Δt	°C	°C
Average value of the Calorimeter constant (C)		J/°C
Heat gained by the solution*	J	J
Heat gained by the calorimeter	J	J
Heat of the reaction	Jan Company	J
Moles of NH <sub>3</sub> reacted Sk	Me V= & re mol	/e mol
Moles of HCI reacted	mol	mol
Moles of NH₄CI produced	mol	mol
ΔΗ	kJ/mol	kJ/mol
Average value of ΔH		kJ/mol
ΔH for the reaction:		kJ
NaOH + NH₄Cl → NaCl + NH₃ + H₂O		

<sup>\*</sup> Assume density of solution = 1.00 g/mL and specific heat of solution = 4.07 J/g. °C.

#### **Questions**

1.	What effect on the calculated calorimeter constant would be if the calorimeter cup were made of conducting material metal) rather than plastic foam?	(such a
		- 1
••••		· · · · · · · · · · · · · · · · · · ·
2.	Why is water typically used as the heat-absorbing	liquid <sub>ir</sub>
	calorimeter?	
3	Given that:	
	$H_2O_{2(\ell)} \to H_2O_{(\ell)} + \frac{1}{2}O_{2(g)}$ $\Delta H = -96.0 \text{ kJ/mol}$	
	$2H_{2(g)} + O_{2(g)} \rightarrow 2H_2O_{(\ell)} = \Delta H = -571.6 \text{ kJ/mol}$	
Use	e Hess's law to calculate ΔH for the reaction:	
	$H_{2(g)} + O_{2(g)} \rightarrow H_2O_{2(g)}$	
•••••		
••••		
	de more and finding and an arrangement	

# 11 Molar Mass from Freezing Point Depression

#### **Objectives**

You will be able to determine the molar mass of a nonvolatile solute by observing the freezing point depression of a solvent due to solution formation.

#### Introduction

Solutions differ in their properties from pure solvents. Among these properties are vapor pressure, osmotic pressure, freezing point depression and boiling point elevation. These properties are called colligative properties and are affected by the number of moles of solute particles dissolved in the solvent and not due to the kind of solute. As a consequence, colligative properties can be used to determine the number of moles per a certain quantity of solute, and thus the molar mass of a solute. The apparatus you will use will probably resemble that shown in Figure (11.1).

In this experiment, you will determine the freezing-point depression,  $\Delta T_f$  (freezing point of solvent – freezing point of solution). This quantity is proportional to the molal concentration of the solute, m:

$$\Delta T_f = k_f m$$

The molal concentration (m) is defined as the number of moles of solute divided by the mass of solvent in kilograms.

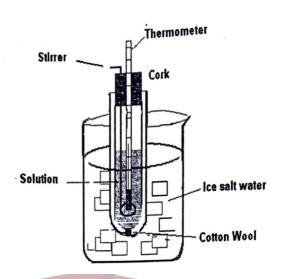


Figure 11.1. Apparatus for freezing point depression

The proportionality constant is called the molal freezing-point depression constant and is characteristic of the solvent. You can calculate the molar mass of a solute from  $\Delta T_f$ ,  $k_f$ , the mass of the solute, and the mass of the solvent. The molal freezing point depression constant for some solvents are reported in Table 11.1.

Table 11.1 the molal freezing point depression constants for some common solvents.

Solvent	k <sub>f</sub> (°C/m)	
Water	1.86	7 12
Benzene	5.07	
Cyclohexane	20.0	

In order to determine  $\Delta T_f$  you will measure the freezing point of pure solvent and the freezing point of a solution of the solute in that solvent.

To evaluate the freezing point of pure solvent, you will cool a sample of this substance and measure the temperature as a function of time. As the solvent is cooled, the temperature of the solvent starts decreasing.

However, as soon as the solvent begins to freeze, the temperature no longer decreases but remains constant. The freezing point is the constant temperature that occurs while solvent is freezing (Fig. 11.2).

The freezing point of a solution (solute in a solvent) can be obtained in a similar way, but there is a difference. The difference can be seen in Figure 11.2. Cooling the solution results in an initial rapid decrease in the temperature until freezing begins. However, with the solution (unlike pure solvent) the temperature does not remain constant until all the solvent has frozen. As the solvent in the solution freezes, the solution becomes more concentrated because less liquid remains. The molal concentration of the solute must increase, and the freezing point must decrease still further. The result is a steadily decreasing freezing temperature. When all the solvent has frozen, the temperature decreases more rapidly.

The freezing point that you need to record for the solution, is the initial freezing point, that is, the point at which only a small fraction of solvent solidifies.

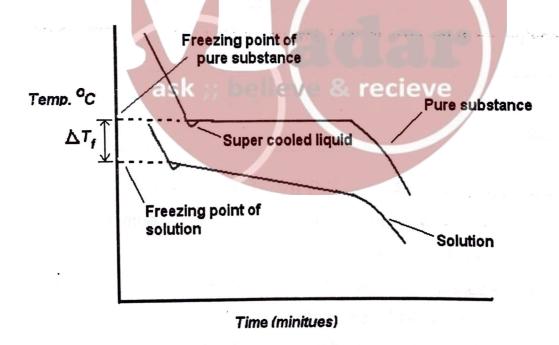


Figure 11.2 Cooling curves for pure solvent and solution.

It is worth noting that the expression:

 $\Delta T_f = k_f m$ 

is valid and holds for dilute solutions of nondissociating and nonassociating solutes. For these solutes, the molar mass may be calculated from  $\Delta T_{f_i}$   $K_{f_i}$ the mass of solute, and the mass of solvent.

## Apparatus/Reagents Required

Freezing point depression apparatus consisting of small tube, larger tube, cork, cotton, a stirring wire. 600-mL Beaker, thermometer. cyclohexane, ice and an unknown solute.

#### **Safety Precautions:**

- Cyclohexane is flammable. No open flames are allowed during this experiment.
- Do not discard cyclohexane into the sink. Obtain directions for discarding cyclohexane and cyclohexane solution that you will use in this experiment from your instructor.

recieve

#### **Procedure**

#### A. Measuring the freezing point of cyclohexane

- Obtain the apparatus required for this experiment. It will be 1. demonstrated by your laboratory instructor.
- Make sure that the small test tube of the apparatus is dry. If not wash 2. it with acetone and allow to dry.
- Weigh the empty dry small test tube to the nearest 0.01 g. 3.
- Measure, using a graduated cylinder, about 15.0 mL of cyclohexane 4. and place into the small tube.

- 5. Assemble the freezing point apparatus and place it in a beaker containing crushed ice and water, as shown in Figure 11.1.
- 6. Stir the cyclohexane gently but constantly observing the temperature at which the cyclohexane starts to solidify.
- 7. Continue the cooling process for 2-3 minutes while observing the temperature (do you observe any change in temperature?).
- 8. Record the temperature at the start of solvent crystal formation (this temperature will remain constant until all of the solvent solidifies). This temperature is the freezing point of the solvent.
  - Remove the small tube and allow the solvent to melt.
  - 10. Repeat steps 5 through 8 for trial (II).
  - 11. Save the cyclohexane for the next part of the experiment.

## B. Measuring the freezing point of the solution

- 1. Obtain two pieces of weighing paper. Mark each one for recognition.
- Weigh accurately about 0.20-0.25 g of the solute on the first paper.
   Record the mass of the solute.
- 3. Remove the stirrer and thermometer from the small tube and then weigh the small tube with cyclohexane. (You can use a small beaker to hold the test tube on the balance pan).
- 4. Transfer the first sample into the test tube that contains the cyclohexane.
- 5. Stir until you obtain a complete solution. All of the solid, including any on the walls of the inner tube, must dissolve before the measurement can begin.
- 6. Place the apparatus in a beaker containing crushed ice and water.
- 7. Stir the cyclohexane gently but constantly, and record the temperature at which the solution starts to solidify (first freezing point).
- Remove the apparatus from the beaker and allow the solution to melt completely.

- Weigh accurately about 0.10 g of the same solute and add it to the cyclohexane solution.
- 10. Repeat steps 5 through 7.
- 11. Record the second freezing point.
- 12. Calculate the molar mass of solute



# 11 Molar Mass from Freezing Point Depression

Name: Section
Lab. Instructor Date
Pre-Laboratory Questions
Define what we mean by the colligative property.
2. A 0.36-g sample of an unknown substance was dissolved in 30.0
mL of cyclohexane. The density of cyclohexane is 0.779 g/mL.
The freezing-point depression was 2.50 °C. Calculate the molar
mass of the unknown substance. [k <sub>f</sub> (cyclohexane) = 20.0 °C/m]

# 11 Molar Mass from Freezing Point Depression

Name:	Section
Lab. Instructor	Date

## **Results and Calculations**

# A. Freezing point of cyclohexane

	Trial (I)	Trial (I	l)
Mass of empty small tube			
	g		g
Freezing point of cyclohexane	00		00
	°C		°C
Average value of freezing point			00
g point			°C

## B. Freezing point of solution

Many of any d	Data
Mass of empty small tube	g
Mass of solute	g
Mass of empty small tube + cyclohexane	
Mass of cyclohexane	9
	9
First freezing point	°C
$(\Delta T_f)_1$	°C
Molar mass of solute	g/mol
Mass of second sample of solute	g
Total mass of solute	g
Second freezing point	°C
$(\Delta T_f)_2$	°C
Molar mass of solute	g/mol
Average molar mass of solute	g/mol

### **QUESTIONS**

<b>Q</b> 1.	What would be the effect of each of the following on the calculated molar mass of the solute?
	a) Some cyclohexane evaporated while the freezing point of pure cyclohexane was being measured.
	b) Some cyclohexane evaporated after the solute was added  c) A foreign solute was already present in the cyclohexane.
	d) The thermometer is not calibrated correctly. It gives a temperature that is 1.5 °C too low at all temperature.

# 12 Solubility Product Constant and Common Ion Effect

#### **Objectives**

You will be able to determine the molar solubility and solubility product constant for  $Ca(OH)_2$  and to determine the molar solubility for  $Ca(OH)_2$  in the presence of excess  $Ca^{2+}$ .

#### Introduction

Salts which have a low solubility in water are called slightly soluble or sparingly soluble salts. In a saturated solution of a slightly soluble salt there is a dynamic equilibrium between the solid salt and the very low concentrations of its ions in solution. Limestone rock appears to be insoluble in its natural environment, however with time limestone dissolves due to its low solubility. The major component of limestone is calcium carbonate, CaCO<sub>3</sub>. Its equilibrium with its ions, Ca<sup>2+</sup> and CO<sub>3</sub><sup>2-</sup>, favors the solid CaCO<sub>3</sub>, thus the equilibrium lies far to the left:

$$CaCO_{3(s)} \iff Ca^{2+}_{(aq)} + CO_3^{2-}_{(aq)}$$

The mass action expression for this system at equilibrium is represented as:

$$K_{sp} = [Ca^{2+}][CO_3^{2-}]$$

where  $K_{sp}$  is called the solubility product constant, and the concentrations,  $[Ca^{2+}]$  and  $[CO_3^{2-}]$  are those of  $Ca^{2+}$  and  $CO_3^{2-}$  at equilibrium. This does not mean that the molar concentrations of  $Ca^{2+}$  and  $CO_3^{2-}$  are always equal, if a solution has higher  $[Ca^{2+}]$ , then its  $[CO_3^{2-}]$  must be necessarily low, it is their product that is constant at same temperature.  $K_{sp}$  for  $CaCO_3$  is  $8.7x10^{-9}$  at  $25^{\circ}C$ ,

 $K_{sp} = [Ca^{2+}][CO_3^{2-}] = 8.7x10^{-9}$ and so, in a saturated solution of CaCO<sub>3</sub> the concentration of the ion<sub>S</sub>, Ca<sup>2+</sup> and CO<sub>3</sub><sup>2-</sup> is  $[Ca^{2+}] = [CO_3^{2-}] = (8.7 \times 10^{-9})^{1/2} = 9.3 \times 10^{-5} \text{ mol/L}$ , and the

molar solubility of CaCO<sub>3</sub> is 9.3x10<sup>-5</sup> mol/L.

When an ion, cation or anion, is added to a saturated solution of CaCO<sub>3</sub>, its equilibrium will shift according to LeChatelier's principle to compensate for the added common ion to favor the precipitation of some solid CaCO<sub>3</sub>, thus reducing its solubility in water. This effect on the equilibrium is called Common Ion Effect.

In this experiment you will determine the molar solubility and the solubility product constant, K<sub>sp</sub>, of Ca(OH)<sub>2</sub> and the molar solubility of Ca(OH)<sub>2</sub> in a saturated solution containing added Ca<sup>2+</sup>. The hydroxide ion, OH, in the saturated solution of Ca(OH)2 is titrated with a standard hydrochloric acid solution to the end or stoichiometric point, using a bromocresol green as an indicator. The stoichiometric point occurs, for this titration, when equal moles of H<sub>3</sub>O<sup>+</sup> and OH<sup>-</sup> are present in the receiving flask. Bromocresol green is an acid-base indicator that is blue in its basic form and yellow in its acidic form.

According to the equation:

$$Ca(OH)_{2(s)} \Leftrightarrow Ca^{2+}_{(aq)} + 2OH_{(aq)}^{-}$$

for each mole of Ca(OH)2 that dissolves, 1 mol Ca2+ and 2 mol OH are produced in solution. Thus by determining the [OH], the [Ca2+], the molar solubility of Ca(OH)<sub>2</sub>, and the K<sub>sp</sub> for Ca(OH)<sub>2</sub> can be calculated.

Since  $[Ca^{2+}] = \frac{1}{2}[OH^{-}]$ Then:

Molar solubility of  $Ca(OH)_2 = [Ca^{2+}] = \frac{1}{2}[OH^{-}]$ 

$$K_{sp} = [Ca^{2+}][OH^{-}]^{2} = \frac{1}{2}[OH^{-}] \times [OH^{-}]^{2}$$

Likewise, the same procedure is used to determine the molar solubility of  $Ca(OH)_2$  with the common ion  $Ca^{2+}$  added to a saturated solution of  $Ca(OH)_2$ .

#### safety Precautions:

Some reagents used in this experiment are corrosive and harmful to the skin, avoid any contact with the skin.

### Apparatus/Reagents Required

Three 150 or 250 mL Erlenmeyer flasks, 25-mL pipet mL, saturated solution of Ca(OH)<sub>2</sub>, standard 0.05 M HCl, saturated solution of Ca(OH)<sub>2</sub> in 0.10 M CaCl<sub>2</sub> solution, bromocresol green indicator.

#### **Procedure**

### A. Molar solubility and $K_{sp}$ for $Ca(OH)_2$ .

- Clean and label two 150 or 250 mL Erlenmeyer flasks. Use a 25-mL pipet to transfer 25.00 mL of saturated solution of Ca(OH)<sub>2</sub> into each flask. Cover each flask with aluminum foil or parafilm to avoid contact with CO<sub>2</sub> from air.
- Prepare 50 mL buret for titration. Rinse the clean buret and tip with 5 mL portions of a standard 0.05 M HCl solution. Fill, read and record the volume of 0.05 M HCl in the buret (initial reading).
- 3. Record the molar concentration of HCl solution.
- 4. Add 2 drops of bromocresol green indicator and titrate the saturated solution of Ca(OH)<sub>2</sub> with the standard HCl solution. Record the volume of HCl solution needed to just turn the blue color of the indicator yellow (final reading).

 Repeat the titration using the second solution, record initial and final volumes of the buret.

# B. Molar solubility for $Ca(OH)_2$ in 0.10 M $CaCl_2$ solution,

Repeat steps 1-4 in part A above by titrating a saturated solution of Ca(OH)<sub>2</sub> in 0.10 M CaCl<sub>2</sub> solution (no need to clean the buret again). Record your data on the data sheet.



# Solubility Product Constant and Common Ion Effect

Name:	ructor		••••••			
Lab.	Pre	-Labo	ratory Q	uestion:	s	•
1. Write					soluble salt ed	quilibria:
<b>a</b> )			† <sub>(aq)</sub> + S <sup>2-</sup> <sub>(aq)</sub>		K <sub>sp</sub> =	
b)	Al <sub>2</sub> S <sub>3(s)</sub>	⇒ 2Al	<sup>3+</sup> (aq) + 3S <sup>2-</sup> (a	q);	K <sub>sp</sub> =	
c)	BaSO <sub>4(s)</sub>	<b>⇒</b> Ba	a <sup>2+</sup> (aq) + SO	4 <sup>2-</sup> (aq);	K <sub>sp</sub> =	
d	) Sr <sub>3</sub> (PO <sub>4</sub> ) <sub>2</sub>	2(s) \( \leftrightarrow \)	3Sr <sup>2+</sup> <sub>(aq)</sub> +	2PO <sub>4</sub> <sup>3-</sup> (aq);	K <sub>sp</sub> =	
	for PbCl <sub>2</sub> = 1	.6x10 <sup>-5</sup> ).	1		g/L) of PbCl <sub>2</sub> ,	
		ask ;;	belleve	& reci	leve	
:			,			
	ulate the mol			in the pres	ence of 0.15 N	1

# 12 Solubility Product Constant and Common Ion Effect

## **Results Calculations**

## A. Molar solubility and $K_{\text{sp}}$ for $\text{Ca}(\text{OH})_2$

Concentration of standard HCI solution used =

mol/L

	Trial (I)	Trial (II)
Initial buret reading	mL	mL
Final buret reading	mL	mL
Volume of standard HCl added	mL	mL
Moles of HCI added	mol	mol
Moles of OH⁻ in saturated Ca(OH)₂ solution	mol & recieve	mol
Volume of saturated Ca(OH) <sub>2</sub> solution titrated	mL	mL
[OH <sup>-</sup> ] at equilibrium	mol/L	mol/L
[Ca <sup>2+</sup> ] at equilibrium	mol/L	mol/L
Molar solubility of Ca(OH) <sub>2</sub>	mol/L	. mol/L
Average molar solubility of Ca(OH) <sub>2</sub>		mol/L
Average solubility of Ca(OH) <sub>2</sub>		g/L
K <sub>sp</sub> of Ca(OH) <sub>2</sub>		

# B. Solubility of Ca(OH)<sub>2</sub> in the Presence of Ca<sup>2+</sup>

Concentration of standard HCl solution used =

mol/L

Concentration of standard 1101 35 m	T - 1 (1)	•
	Trial (I)	Trial (II)
	mL	
Initial buret reading		m(
Final buret reading	mL	mL.
Volume of standard HCl added	mL	mL
- Claric of Standard Horadou		
Moles of HCl added	mol	mol
Moles of OH <sup>-</sup> in saturated Ca(OH) <sub>2</sub> /Ca <sup>2+</sup>	mol	mol
solution		
Volume of saturated Ca(OH) <sub>2</sub> /Ca <sup>2+</sup> solution	mL	mL
titrated		
[OH] at equilibrium	mol/L	mol/L
[Ca²⁺] at equilibrium	mol/L	mol/L
Molar solubility of Ca(OH) <sub>2</sub> in	mol/L	mol/L
Ca(OH) <sub>2</sub> /CaCl <sub>2</sub> solution	ecieve ///	Voiv
Average molar solubility of Ca(OH) <sub>2</sub> in		mol/L
Ca(OH) <sub>2</sub> /CaCl <sub>2</sub> solution		
Average solubility of Ca(OH) <sub>2</sub> in		g/L
Ca(OH) <sub>2</sub> /CaCl <sub>2</sub> solution		

## QUESTIONS

1.	A saturated solution of Ca(OH) <sub>2</sub> is prepared by adding enough $Ca(OH)_{2(s)}$ to distilled pre-boiled water, the solution is then filtered to remove any insoluble amount of Ca(OH) <sub>2</sub> .  a) Why pre-boiled water is used to prepare the saturated solution of $Ca(OH)_2$ ?
	b) If some solid $Ca(OH)_2$ remained after filtration, how would it affects the calculated values of molar solubility of $Ca(OH)_2$ and the $K_{sp}$ for $Ca(OH)_2$ ?
	Ca(C11)2
2.	How did the added CaCl <sub>2</sub> affect the molar solubility of Ca(OH) <sub>2</sub> ?
	Explain.
	ask ;; belleve & recieve
3.	Calculate the molar solubility of Ca(OH) <sub>2</sub> in 0.10 M NaOH solution. Use
	the value of K <sub>sp</sub> obtained in part A.

# 13 Solubility Rules: Solubilities Within a Family

### **Objectives**

You will be able to find the relative solubilities of some compounds of the alkaline earth metals. You will also compare the solubilities of these compounds with similar compounds of lead (a metal in Group IVA).

### Introduction

The periodic table is arranged in such a way that the electron configuration of the elements displays a periodic variation. The same kind of outer configuration occurs within a group. For example, the outer electron configuration of alkaline earth metals (Group IIA) is always ns<sup>2</sup>, no matter what period of the element. The periodicity in the outer electron configuration is responsible for the periodic law: When the elements are arranged by atomic number, their physical and chemical properties vary periodically. The periodic law, however, does not imply that all the properties of the elements within a group will be identical. Trends in properties are usually found instead. In this experiment you will observe the trends in the solubilities within the alkaline earth metals.

The solubility of a compound in a liquid is usually defined as the maximum amount of that compound that will dissolve in 100 mL of liquid, at a fixed temperature. In this experiment you will be interested in the qualitative aspect of solubility. You will find that the qualitative terms soluble and *insoluble* can be used to describe a compound's solubility. There are two ways to determine the solubility of a compound qualitatively.

First, you can take a sample of the the compound and place it in the desired liquid, and observe the solubility. Second, you can synthesize (make) the compound by a chemical reaction in the liquid. If the compound appears as a precipitate, we know that it cannot be very soluble in the liquid. If it doesn't appear, it must be soluble. You will employ the second method in this experiment.

The alkaline earth metals and lead form nitrates, hydroxides, chlorides, bromides, and iodides with general formulas of  $M(NO_3)_{2}$ , M(OH)<sub>2</sub>, MCl<sub>2</sub>, MBr<sub>2</sub>, and Ml<sub>2</sub>, respectively. The following reaction:

$$M(NO_3)_2 + 2NaX \rightarrow MX_2 + 2NaNO_3$$

will be used to determine the qualitative solubilities of the hydroxides, chlorides, bromides, and iodides in water. Each of these is represented by X in the general reaction above. We know that sodium nitrate (NaNO<sub>3</sub>) is very soluble (recall solubility rules: all nitrates are soluble). Thus, if a precipitate appears, it can only be MX2, and this must mean that this compound is not very soluble in water.

The alkaline earth metals and lead also form sulfates, carbonates, oxalates, and chromates with general formula of MSO<sub>4</sub>, MCO<sub>3</sub>, MC<sub>2</sub>O<sub>4</sub>, and MCrO<sub>4</sub>, respectively. These compounds will be prepared by the reaction:

$$M(NO_3)_2 + Na_2Y \rightarrow MY + 2NaNO_3$$
  
Where  $Y = SO_4^{2-}, CO_3^{2-}, C_2O_4^{2-}, CrO_4^{2-}$ 

Where, Y represents sulfate, carbonate, oxalate, and chromate. The solubility's of the MY compounds will be determined in exactly the same way as those of the MX2 compounds.

Because you will be examining only the qualitative aspects of the solubility's of these compounds, you will not observe trends directly. Instead, you will need to infer their presence. The following example should make this process easier.

Consider the solubility's of the chromates shown in Table 13.1. let us pretend for the moment that we do not know these solubility's. What would you find if you try to dissolve 1.0 x 10<sup>-4</sup> mol of each substance in 1-L of water? Note that only the solubility of BaCrO<sub>4</sub> has been exceeded. As a result, all of the MgCrO<sub>4</sub>, CaCrO<sub>4</sub>, and SrCrO<sub>4</sub> would remain in solution, and a precipitate of BaCrO<sub>4</sub> would appear. This experiment would force us to admit the possibility that if a trend exists, the solubility's may decrease as the atomic number of the Group IIA metal increases. As Table 13.1 shows, this is correct.

Table 13.1. Solubilities of Some Metal Chromates (mol/L H<sub>2</sub>O)

Compound	MgCrO <sub>4</sub>	CaCrO <sub>4</sub>	SrCrO <sub>4</sub>	BaCrO <sub>4</sub>
Solubilities	9.9	1.2	5.9 x 10 <sup>-3</sup>	1.1 x 10 <sup>-5</sup>
Observations	soluble	Soluble	soluble	Solid precipitate

#### **Safety Precautions:**

■ Wash your hands thoroughly after using solutions containing lead, barium, or oxalate because they are poisonous.

### Apparatus/Reagents Required

5 small test tubes, 10 mL graduated cylinder, 0.1 M solutions of:  $Mg(NO_3)_2$ ,  $Ca(NO_3)_2$ ,  $Sr(NO_3)_2$ ,  $Ba(NO_3)_2$ ,  $Pb(NO_3)_2$ ,  $Na_2CO_3$ ,  $Na_2C_2O_4$ , 1 M NaOH, 1 M NaCl, unknown salt.

#### **Procedure**

- 1. Obtain 5 small and clean test tubes. Use a 5-mL or a 10-mL graduated cylinder to place 1 mL of distilled water in each of these test tubes. Mark the height of the water in each test tube with a small piece of tap or a marking pencil. Add an additional 1 mL of distilled water to each test tube and mark the new height of the water in each. Pour the water into the sink.
- 2. Obtain directions for discarding the solutions that you will use in this experiment.
- 3. Observe the safety precaution during the remainder of this experiment.
- 4. Mark the test tube with identification numbers (1 through 5).
- 5. Using the lower marks on the test tubes as guides, add 1 ml of 0.1 M Mg(NO<sub>3</sub>)<sub>2</sub> to the first test tube, 1 mL of 0.1 M Ca(NO<sub>3</sub>)<sub>2</sub> to the second,1 mL of 0.1 M Sr(NO<sub>3</sub>)<sub>2</sub> to the third, 1 ml of 0.1 M Ba(NO<sub>3</sub>)<sub>2</sub> to the fourth, and I mL of 0.1 M Pb(NO<sub>3</sub>)<sub>2</sub> to the fifth.
- 6. Using the upper marks as guides, add 1 mL of 1 M NaOH to each test tube.
- 7. Shake each tube gently and wait about 30 seconds. Note the color of all precipitates. If any of the precipitates is white, it may be difficult to see. Be observant! Note the relative amount of the precipitates. If the solubility of a compound is barely exceeded, only a trace amount of a precipitate will appear. Record your observation.

#### Caution: do not use your finger as a stopper.

- 8. Wash the test tubes carefully and rinse them with distilled water.
- Repeat steps 5 through 8 in turn with 1 mL 0.1 M NaCl, 0.1 M NaBr, 0.1 M NaI, 0.1 M Na<sub>2</sub>SO<sub>4</sub>, 0.1 M Na<sub>2</sub>CO<sub>3</sub>, and 0.1 M Na<sub>2</sub>C<sub>2</sub>O<sub>4</sub> instead of the solution of NaOH.
- 10. Repeat steps 5 through 8 with 1 mL of the unknown solution.
- 11. Record your observations.

# Solubility Rules: Solubilities within a Family

Nai Lat	ne:
	Pre-Laboratory Questions
1.	Give the names and symbols of the alkaline earth metals that you will encounter in this experiment.
2.	What is the general electron configuration of the alkaline earth Metals and lead?
	ask :: belle ve & recieve
3.	Compare the formulas of the oxides formed by the alkaline earth metals with those of lead.
4.	Give general formulas for the following compounds with alkaline earth
	metals or lead:  (a) A sulfate
	(b) A carbonate(c) A chromate
	(d) An oxalate

# 13 Solubility Rules: Solubilities within a Family

Name:	Section
Lab. Instructor	Date

### **Results and Calculations**

Record the color and amount (large, small, traces, none) of precipitates.

	Mg(NO <sub>3</sub> ) <sub>2</sub>	Ca(NO <sub>3</sub> ) <sub>2</sub>	Sr(NO <sub>3</sub> ) <sub>2</sub>	Ba(NO <sub>3</sub> ) <sub>2</sub>	Pb(NO <sub>3</sub> ) <sub>2</sub>
NaOH					
NaCl					
NaBr		A			
Nal	as	k ;; belle	& rec	ieve	
Na <sub>2</sub> SO <sub>4</sub>					
Na <sub>2</sub> CO <sub>3</sub>					
Na <sub>2</sub> C <sub>2</sub> O <sub>4</sub>					
Unknown					

### **Questions**

1. What trends in the solubility's of the compounds of the alkaline
a authoritate can be concluded?
earth metals can be consider
111111111111111111111111111111111111111
2. (a) Compare the solubility's of the lead compounds with those of
the alkaline earth metals. How are the solubilities similar, and
the alkaline earth metals. How are this country
how do they differ?
(b) As you have seen, lead can form the same kinds of compounds as
the alkaline earth metals. Nevertheless, the solubility's may differ
markedly. Give a convincing reason.
indikodiy. Give a collinating to see
•

## **Appendices**

## Appendix A: Significant Figures in Calculations

### 1. Numbers

There are two kinds of numbers:

a) Exact Numbers: Defined and Countable numbers

Examples: There are exactly 12 eggs in a dozen. Most people have exactly 10 fingers and 10 toes.

#### b) Measured Quantities:

Example: If I quickly measure the width of a piece of notebook paper, I might get 2.2 cm (2 significant figures). If I am more precise, I might get 2.16 cm (3 significant figures). An even more precise measurement would be 2.156 cm (4 significant figures).

recieve

### 2. Number of Significant Figures

The number of significant figures in a measurement, such as 2.531, is equal to the number of digits that are certain (2, 5, and 3) plus one uncertain digit (1). As we improve the sensitivity of the equipment used to make a measurement, the number of significant figures increases.

#### **Rules for Counting Significant Figures:**

i) All nonzero digits are significant: 1.234 g has 4 significant figures;1.2 g has 2 significant figures.

- ii) Zeroes between nonzero digits are significant: 1002 kg has  $_{\rm 4}$  significant figures; 3.07 mL has 3 significant figures.
- iii) Leading zeros to the left of the first nonzero digit are not significant; such zeroes merely indicate the position of the decimal point. For example, the number 0.001 °C has only 1 significant figure and 0.012 g has 2 significant figures.
- iv) Trailing zeroes to the extreme right of a number in the presence of a decimal point are significant. For example: 0.0230 mL has 3 significant figures, 0.20 g has 2 significant figures.
- v) When a number ends in zeroes that are not to the right of a decimal point, the zeroes are not necessarily significant. For example: 190 could be considered with 2 or 3 significant figures and 50,600 calories could be considered 3, 4, or 5 significant figures.

If you are not sure whether a digit is significant, assume that it isn't. For example, if the directions for an experiment read: "Add the sample to 400 mL of water," assume the volume of water is known to one significant figure.

#### 3. Significant Figures in Calculations

#### a) Addition and Subtraction

In addition and subtraction, round the final result to the least number of decimal places, regardless of the number of significant figures in any measurement. For example:

100. (0 decimals) + 23.643 (3 decimals) = 123.643. This sum should be rounded to 124 (0 decimals).

23.152 (3 decimals) - 10.1 (1 decimal) = 13.052. This sum should be rounded to 13.0 (1 decimal)

### b) Multiplication and Division

In multiplication and division the final result should be rounded to the least number of significant figures of any measurement.

For example:

3.0 (2 significant figures) × 12.60 (4 significant figures) = 37.8000.

The result should be rounded off to 2 significant figures, i.e. 38. In a similar way:

$$\frac{8.33 \times 9.1167}{4.500} = 16.87602467$$

The result should be rounded off to 16.9 (3 significant figures).

#### 

When the answer to a calculation contains too many digits, it must be rounded off.

There are 10 digits that can occur in the last decimal place in a calculation. One way of rounding off involves *underestimating* the answer for five of these digits (0, 1, 2, 3, and 4) and *overestimating* the answer for the other five (5, 6, 7, 8, and 9). This approach to rounding off is summarized as follows. If the digit is smaller than 5, drop this digit and leave the remaining number unchanged. Thus, 1.684 becomes 1.68. If the digit is 5 or larger, drop this digit and add 1 to the preceding digit. Thus, 1.247 becomes 1.25

### 4. Scientific Notation

Chemists often work with numbers that are extremely large  $_{0r}$  extremely small. For example Avogadro's number could be written as:

1 mole = 602,200,000,000,000,000,000 atoms

But it is more convenient to record the number in **scientific notation** which has the form:  $N \times 10^x$ , where N = a number greater than 1 but less than 10 and x = exponent of 10. So the number should be recorded in scientific notation as: **6.022**  $\times$  **10**<sup>23</sup>

#### **Examples:**

If the number (150000000000000) is a measured quantity with 3 significant figures, it will be reported in the scientific notation as:  $1.50 \times 10^{14}$ 

15.0 X 10<sup>13</sup> .(Wrong; the first part is greater than 10)

0.150 X 10<sup>15</sup>..(Wrong; the first part is less than 1)

The number (0.0000050) is reported in scientific notation as: 5.0 X 10<sup>-6</sup>

0.50 X 10<sup>-5</sup> .(Wrong; the first part is less than 1)

50 X 10<sup>-7</sup> (Wrong; the first part is greater than 10)

## Appendix B: Conversion Factors

### Length Conversion Factors

 $_{1 \text{ km}} = 1000 \text{ m}$ 

 $_{1 \text{ m}} = 100 \text{ cm}$ 

 $_{1}$  °A = 1 X  $10^{-10}$  m

1 yard = 91.44 cm

 $_{1}$  foot = 30.48 cm

1 inch = 2.54 cm

#### **Volume Conversion Factors**

 $1 \text{ m}^3 = 1.000 \text{ L}$ 

 $1 L = 1 dm^3 = 1000 mL$ 

#### **Mass Conversion Factors**

1 metric ton = 1000 kg

1 kg = 1000 g

1 kg = 2.2046 lb ask

1 ounce = 28.35 g

1 pound = 453.6 g

#### **Energy Conversion Factors**

1 calorie = 4.184 joules

 $1 J = 1 kg.m^2/s^2$ 

1 eV/molecule = 96.485 kJ/mole

 $1 \text{ kJ/mol} = 83.54 \text{ cm}^{-1}$ 

#### **Temperature Conversion Factors**

 $0 \, ^{\circ}\text{C} = 273.16 \, \text{K} = 32 \, \text{F}$ 

 $100 \, ^{\circ}\text{C} = 373.16 \, \text{K} = 212 \, \text{F}$ 

#### **Pressure Conversion Factors**

1 atm = 76 cm Hg = 760 mm Hg = 760 torr

1 atm = 101.325 kPa

 $1 \text{ atm} = 14.69 \text{ lb/in}^2 \text{ (psi)}$ 

#### **Useful Constants**

R = 8.314 J/mol.K ieve

= 0.08206 L.atm/mol.K

N (Avogadros' Number) =  $6.022 \times 10^{23}$ 

 $c (Speed of light) = 2.9979 \times 10^8 \text{ m/s}$ 

Appendix C: Data for Concentrated Acids And
Bases

Reagent	HCI	HNO₃	H <sub>2</sub> SO <sub>4</sub>	CH₃COOH	NH <sub>3</sub>
Formula Weight	36.46	63.01	98.08	60.05	17.03
Density of Concentrated Reagent	1.19	1.42	1.84	1.05	0.90
Mass Percent %	37.2	70.4	96.0	99.8	29.0
Molarity	12.1	15.9	18.0	17.4	14.8



# Appendix D: Density of Water at Various Temperatures

Temperature (°C)	Density (g/mL)	Temperature (°C)	Density (g/mL)
0	0.99987	22	0.99780
2	0.99997	24	0.99732
4	1.0000	26	0.99681
6	0.99997	28	0.99626
8	0.99988	30	0.99567
10	0.99973	32	0.99505
12	0.99952	34	0.99440
14	0.99927	36	0.99371
16	0.99897	38	0.99299
18	0.99862	40	0.99224
20	0.99823	ada	90

ask ;; belleve & recieve

# Appendix E: Solubility Products, K<sub>sp</sub>, For Ionic Substances at 25°C

Ag <sub>2</sub> CO <sub>3</sub>	8.1 X 10 <sup>-12</sup>	FeS	3.7 X 10 <sup>-19</sup>
Ag <sub>2</sub> CrO <sub>4</sub>	9.0 X 10 <sup>-12</sup>	Hg <sub>2</sub> Cl <sub>2</sub>	1.1 X 10 <sup>-18</sup>
Ag <sub>2</sub> S	1.6 X 10 <sup>-49</sup>	HgS	1.6 X 10 <sup>-54</sup>
Ag <sub>2</sub> SO <sub>4</sub>	1.2 X 10 <sup>-5</sup>	Mg(OH) <sub>2</sub>	8.9 X 10 <sup>-12</sup>
AgBr	5.0 X 10 <sup>-13</sup>	MgCO <sub>3</sub>	1.0 X10 <sup>-15</sup>
AgCI	1.6 X 10 <sup>-10</sup>	MnS	2.3 X 10 <sup>-13</sup>
AgI	1.5 X 10 <sup>-16</sup>	NiS	3 X 10 <sup>-21</sup>
Al(OH) <sub>3</sub>	2.0 X 10 <sup>-32</sup>	Pb(OH) <sub>2</sub>	1.2 X 10 <sup>-15</sup>
Ba(OH) <sub>2</sub>	5.0 X 10 <sup>-3</sup>	PbBr <sub>2</sub>	4.6 X 10 <sup>-6</sup>
BaCO <sub>3</sub>	1.5 X 10 <sup>-9</sup>	PbCl <sub>2</sub>	1.6 X10 <sup>-5</sup>
Ca(OH) <sub>2</sub>	1.3 X 10 <sup>-6</sup>	PbCO <sub>3</sub>	1.5 X 10 <sup>-15</sup>
CaC <sub>2</sub> O <sub>4</sub>	2.3 X 10 <sup>-9</sup>	recPbCrO <sub>4</sub>	2.0 X 10 <sup>-16</sup>
CaCO <sub>3</sub>	8.7 X 10 <sup>-9</sup>	Pbl <sub>2</sub>	4.6 X 10 <sup>-6</sup>
CaSO <sub>4</sub>	6.1 X 10 <sup>-5</sup>	PbS	7 X 10 <sup>-29</sup>
CuS	8.5 X 10 <sup>-45</sup>	Zn(OH) <sub>2</sub>	4.5 X 10 <sup>-17</sup>
Fe(OH) <sub>2</sub>	1.8 X 10 <sup>-15</sup>	ZnCO <sub>3</sub>	2.1 X 10 <sup>-10</sup>
Fe(OH) <sub>3</sub>	4.0 X10 <sup>-38</sup>	ZnS	2.5 X 10 <sup>-22</sup>

# Appendix F: Properties of Substances

## **Properties of substances**

The following table does not list all notable properties of the substances mentioned, additional information about the substance to be used in an experiment should be obtained before use of the substance. Additional information can be obtained from handbook of toxic substances and from posters of hazardous substances distributed in your lab. Corrosiveness and toxicity risks are indicated as L (low), M(moderate), or H (high).

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Name	Flammability	Corrosivity	Toxicity if	Notes	
	combustibility	to skin	ingested	140162	
Acetaldehyde	Yes	M	M	1,4	
Acetic acid	Yes	Н	Н	1,4	
Acetic anhydride	Yes	Н	Н	1,4	
Acetone	Yes	M	Н		
Ammonia	No	M	Н	1,4	
Ammonium Chloride	No	L	Н	1,4	
Ammonium Nitrate	No	M	Н	3	
Barium chloride	No	M	H	-	
Barium nitrate	No	M	H	3	
Bromine	No	Н	H	1	
Calcium carbonate	No	🙎 recieve	// L	2	
Calcium hydroxide	No	M	M		
Chlorine	Yes*	Н	Н	1,4	
Chromium(III) chloride	No	H	Н	1	
Copper(II) sulfate	No	M	M		
Cyclohexane	Yes	L	Н		
Diethyl ether	Yes	M	Н		
Ethanol	Yes	L	Н	5	
Formaldehyde	Yes	M	Н	1,4	
Hydrochloric acid	No	H	Н	1,4	
Hydrogen peroxide (3%)	No	H	M	3	
lodine	No	M	M .		
Iron(III) chloride	No	M	M		
Iron(III) nitrate	No	M	M	3	

		Corrosivity	Toxicity if	
Name	Flammability	to skin	ingested	Notes
· · ·	combustibility	- M	M	
Isopropyl alcohol	Yes	L	M	
Magnesium	Yes	Н	Н	
Mercury(II) chloride	No	Н	Н	
Methanol	Yes	M	Н	
Nickel(II) sulfate	No	H	Н	
Phosphoric acid	No	H	Н	3
Potassium dichromate	No	M	Н	
Potassium ferrocyanide	No	H	Н	
Potassium hydroxide	No		M	
Potassium iodide	No	M	H	
Potassium nitrate	No	M	M	3
Silver nitrate	No	M	H	
Sodium	Yes	H		6
Sodium chloride	No	L		
Sodium Hydroxide	No	Н	Н	
Sodium hypochlorite	No	Н	Н	1,4
Sodium iodide	No	M	M	A
Sodium nitrate	No	Н	Н	3
Sodium sulfate	No	. L	L	7/4
Sodium sulfite	No	M	M	3,4
Sodium thiosulfate	No No	M	M	A
Sulfuric acid	No	ISM H	Н	3,6
Zinc sulfate	No	L	M	

\*Will burn if deliberately ignited, but is not a fire hazard under normal laboratory conditions.

#### Notes:

- 1. This substance is a respiratory irritant. Confine use of the substance to the fume hood.
- 2. This substance vigorously evolves carbon dioxide if acidified.
- 3. This substance is a strong oxidant/ reductant. Although the substance itself may not be flammable, contact with an easily oxidized/reduced material may cause a fire or explosion. Avoid especially contact with any organic substance.
- 4. This substance possesses or may give rise to a stench. Confine use of this substance to the fume hood.
- 5. Ethyl alcohol for use in the laboratory has been denaturated. It is not fit to drink and cannot be made fit to drink.
- 6. This substance reacts vigorously with water and may explode.