GENERAL CHEMISTRY LABORATORY I MANUAL

Fall Semester

Contents

LABORATORY EQUIPMENTS

BEAKER (BEHER)

Beakers are containers which can be used for carrying out reactions, heating solutions, and for water baths.

They are for containing and transferring liquids, not for measurements.

ERLENMAYER FLASK (ERLEN)

Erlenmayer flasks are conical shaped flasks, which are made of Pyrex, are safe for heating solutions, recrystallizations and chemical storage.

Because of its conical shape they are best for using with equipments with stopcocks such as separatory funnels and burettes.

They are also, like beakers, for containing and transferring liquids not for making measurements.

GRADUATED CYLINDER (MEZÜR)

Graduated cylinders are used for measuring the volumes of liquids from a few milliliters to many liters.

It is important to choose the graduated cylinder according to the amount of liquid to be measured for more accurate measurements. Always read meniscus point for graduated cylinders, pipettes, volumetric flasks and burettes.

What is Meniscus Point and how to read it?

Meniscus point occurs when liquid molecules adheres themselves onto the walls of the glassware. This phenomenon is known as adhesion.

Always read the value right at the bottom of the meniscus

24 mL not 25 mL
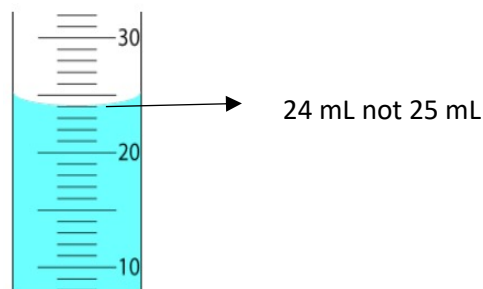
VOLUMETRIC FLASK (BALONJOJE)

Volumetric flasks are used for measuring very precise and accurate amount of a liquid and is used for such when the amount is too much for pipette or burette.

They are also used for solution preparation.

PIPETTE (PİPET)

Pipette is a glass tube used for the delivery of a measured quantity of liquids. There are two kind of pipettes:

a) Graduated (Measuring) Pipette: has many graduated marks, much like a graduated cylinder that can deliver moderately accurate volumes (left picture)

b) Volumetric (Transfer) Pipette: a kind with a bulbous middle section that has a single mark for the quantitative delivery of a single volume of liquid each
time (middle and right picture)

SUCTION DEVICE (PUAR)

A suction device or rubber bulb is a device to place on top of pipettes for generation of vacuum to transfer known volume of liquid, usually from one container to another.

<u>Use of a suction device with a pipette:</u>

Before using the suction device deflate the air inside by pressing button A. Then connect the suction device on to a pipette. Press the button S for suction. Measure desired amount of liquid with pipette by taking meniscus point into account. While emptying use button E
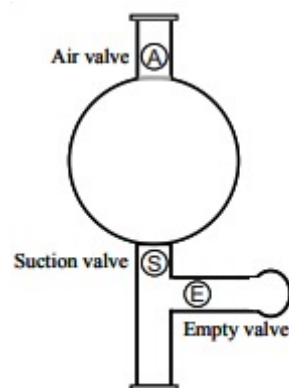
PASTEUR PIPETTE (PASTÖR PİPETİ)

Pasteur pipette (or medicine dropper) is a plastic or glass pipette used to transfer small amounts of liquids, but are not graduated or calibrated for any particular volume.

STAND (STAND)

A metal rod attached to a heavy metal base. The heavy base keeps the stand stable, and the vertical metal rod allows for easy height adjustment of the iron ring/clamp.

BURETTE (BÜRET)

Burette is a vertical cylindircal piece of laboratory glassware with a volumetric graduation on its full length and a stopcock on the bottom. It is used to dispense known amounts of a liquid reagent in a titration experiment.

BURETTE CLAMP (BÜRET KISKACI)

Burette clamp is used to fix the burette onto the stand.

RING SUPPORT (HALKA)

An iron ring/clamp that holds glassware such as a funnels and separatory funnels, and it can be attached to an iron stand.

FUNNEL (HUNİ)

Funnels are used for pouring liquids from one container to another. In addition, with the aid of filterr paper, they can be used as separation devices to separate liquids from solids. It is fixed by a ring support on a stand.

FILTER PAPER (SÜZGEÇ KAĞIDI)

Filter paper is used to separate solid particles from liquids. They can have different size with different pore size.

Solids that remain on filter paper can later be dried on a watch glass or in an oven.

WATCH GLASS (SAAT CAMI)

Watch glass is used to allow crystals to dry after they have been filtered. They can be used as an evaporating surface or to cover a beaker that can be heated to very high temperatures.

SEPARATORY FUNNEL (AYIRMA HUNİSİ)

Separatory funnel is used in liquid-liquid extractions to separate the components of a mixture between two immiscible solvent phases of different densities.
It is fixed on a stand by a ring support.

OVEN (ETÜV)

An oven is an enclosed chamber in which heat is produced to dry chemicals or laboratory equipments.

EVAPORATING DISH (BUHARLAŞTIRMA KROZESİ)

The evaporating dishes are made of porcelain or ceramic material to heat and evaporate solutions to dryness.

ROUND-BOTTOM FLASK (BALON)

Round bottom flasks are used for heating or boiling of a liquid, in distillation procedures and to carry out chemical reactions. Their two or three-necked versions are also available and usually more suitable for carrying out reactions.

TEST TUBE (TEST TÜP)

Test tubes are used as containers for solids and liquids to perform quick tests for properties such as solubility, effect of heat, etc. They can also be used as centrifuge tubes when a separation of solid and liquid is necessary.

TEST TUBE RACK (TÜPLÜK)

The test tube racks provide places to hold the test tubes vertical so that chemicals are not spilled out.

TEST TUBE BRUSH (TÜP YIKAMA FIRÇASI)

Test tube brush is a long and narrow equipment to clean the inside of glassware particularly test tubes.

THERMOMETER (TERMOMETRE)

A thermometer is a device used to measure temperatures or temperature changes.

SPATULA – SPATÜL

Spatula or spoons are hand tools used to weigh solids in balance.

BALANCE (HASSAS TERAZİ)

A balance is a scale for weighing; to obtain mass of various objects.

STIRRING ROD (BAGET)

A stirring rod is made of solid glass and used to stir liquids in

flasks or beakers.

MAGNETIC BAR (MANYETİK BAR)

Magnetic bars employ rotating magnetic field to mix a chemical mixture
or a reaction continuously once they are placed in.
For a magnetic bar to work a stirrer must be placed below or a heater
with a stirrer feature.

HEATER (ISITICI)

A heater is a device that heats water or other solutions to a desired
temperature.

They usually have a stirrer feature to use with magnetic bars while
carrying out a reaction.

BUNSEN BURNER (BEK ALEVİ)

Bunsen burner is a used for heating when no flammable material is present. The burner can be regulated by changing the air and gas mixture.

WASH BOTTLE (PİSET)

Washbottles usually contains distilled water or acetone and makes for a convenient method to wash out lab glasswares.

STOPPER (TIPA)

Stoppers are used to close flasks and test tubes to protect from the environment.

FUME HOODS (ÇEKER OCAK)

The fume hoods protect laboratory workers from fumes and potentially dangerous chemicals reactions by continuously vacuuming air out of the lab and providing a glass shield.

DISPOSAL OF CHEMICALS and CLEANING-UP THE GLASSWARE

If material solid, use solid waste. If it is liquid, determine whether the substance is organic or inorganic then use the related waste.

Use acetone to dissolve organic material. For both organic and inorganic materials, they require distilled water to be cleaned up properly. Use brushes to clean glassware if necessary.

1 Measurements and Density

INTRODUCTION

EVALUATION OF EXPERIMENTAL DATA

One of the most generally accepted axioms in chemistry is that, despite all of the advances in theory during that past fifty years, chemistry is still an experimental science. The vast majority of chemical publications today are experimentally based with advances being made continually that further our already vast reservoir of information and knowledge. The chemist-scientist of today and the future, therefore, needs to have a thorough grounding in experimental techniques, in how to acquire data and, in how to evaluate the data collected. Only with knowledge of how to evaluate collected data can any significance be placed on experimental measurements. Accuracy and precision are two essential concepts in the evaluation of data.

ACCURACY

The agreement of experimental measurements with the accepted value of a quantity is measured in terms of the error. Error is the difference between the value of a quantity as measured and the accepted value of the same quantity.

$$\text{error} = \text{measured value} - \text{accepted value}$$

When the error in a measurement is put on a relative basis it becomes more useful and is known as the relative error. Relative error is defined as the error divided by the accepted value and multiplied by 100 %. This is usually known as % error:

$$\% \text{ error} = \frac{\text{error}}{\text{accepted value}} \times 100\%$$

In making measurements it is important to recognize and, if possible, estimate sources of error. Experimental error may be classified as either systematic (determinate) or random (indeterminate).

In systematic errors the cause usually is detectable and can be corrected. This type of error causes measurements to be consistently higher or lower than the actual value. These include errors present in the system itself, errors present in the measuring device, personal error(bias), and gross errors, such as incorrect recording of data or miscalculation.

Random errors are more related to experimental uncertainty than to accuracy. Their sources cannot be identified and are beyond our control. They also tend to fluctuate in a random fashion about a measured value.

One of our main tasks in designing and performing experiments is to reduce or eliminate the effects of systematic error. In this way, only the cumulative effect of the random errors remains, and this may be estimated in terms of precision.

PRECISION

The key to significance in experimental measurements is repetition. Only with repeated measurements of density, concentration, or other quantities can the experimenter have some confidence in the significance of measurements. Only if a measured quantity can be reproduced repeatedly can the experimenter have that confidence. Precision is a quantitative measure of the reproducibility of experimental measurements. It is how well repeated measurements of the same quantity agree with one another. Precision is frequently measured in terms of the average deviation, which is determined by the following process:

1. From series of measurements (three or more) determine the average value.
2. For each measurement determine its deviation from the average value.
3. Determine the average of the deviations without regard to sign.

In order to make the measurements of precision more useful, the average deviations are put on a percentage basis by determining the relative average deviation. This is the average deviation divided by the average value and multiplied by 100 %.

$$\text{relative average deviation} = \frac{\text{average deviation}}{\text{average of measurements}} \text{ x } 100\%$$

Example: In the determination of concentration of an unknown acid by titration with standard base, four measurements were made: 0.1025 M, 0.1018 M, 0.1020 M, and 0.1024 M.
  a) Compute the average value and the average deviation.
  b) Assuming the accepted value of the unknown acid molarity is 0.1014 M, determine relative error of the experiment.

Solution:

  a) The average value is computed by summing the four measurements and dividing by four. This yields an average value of 0.1022 M.

$$\frac{0.1025 \text{ M} + 0.1018 \text{ M} + 0.1020 \text{ M} + 0.1024 \text{ M}}{4} = 0.1022 \text{ M}$$

The individual deviations of each measurement from the average value are 0.0003 (0.1025), 0.0004 (0.1018), 0.0002 (0.1020), and 0.0002 (0.1024).

$$|0.1022-0.1025| = 0.0003$$
$$|0.1022-0.1018| = 0.0004$$
$$|0.1022-0.1020| = 0.0002$$
$$|0.1022-0.1024| = 0.0002$$

The sum of the four deviations, 0.0011, divided by four yields the average deviation, 0.00028.

$$0.0003+0.0004+0.0002+0.0002 = 0.0011$$

$$\frac{0.0011}{4} = 0.00028$$

Since this deviation represents an uncertainty in the measurements, the molarity of the unknown acid is not precisely 0.1022 M, but ranges from 0.1019 M to 0.1025 M and should, therefore, be reported with the average deviation included, that is, 0.1022 ± 0.0003 M.

$$\text{relative average deviation} = \frac{\text{average deviation}}{\text{average of measurements}} \text{ x } 100\%$$

In the example, the relative average deviation is

$$\frac{0.00028}{0.1022} \text{ x } 100\% = 0.27\%$$

A relative average deviation of 0.27% or better (that is, smaller) is a typical expected precision value for an acid-base titration. In general, however, the precision of an experiment varies with the technique and/or apparatus used. Number of variables built into the method or design of the experiment can affect its precision.

b) Since the error is the difference between the measured value and accepted value, in this case it is

$$0.1022 - 0.1014 = +0.0008.$$

From this error, the % error is calculated as

$$\% \text{ error} = \frac{+0.0008}{0.1014} \text{ x } 100\% = +0.8\%$$

The only significance of the sign of the error (+ or -) is that the measured value is greater or smaller than the accepted value.

In the example above, the % error, which measures the accuracy of the experiment, is larger than the precision. This is an indication of the existence of systematic error and can be corrected. If all systematic errors have been eliminated, the accuracy should be comparable to the precision of the experiment which measures random error. Thus, the accuracy of the experiment is related to the precision (random error) but is also related to possible systematic error.

In a method tested over a long period of time, the results (average value) should not only agree very well with one another (have good precision), but also agree very closely with the true or accepted value (have high accuracy).

PROPAGATION OF ERRORS AND DEVIATIONS

Often the final result of an experiment will be a combination of the measurements of several different items. The errors (or deviations) in all of these measurements are combined to produce the error (or deviation) of the result. If two measurements are added or subtracted, the error of the result is the sum of the absolute errors of the two measurements. If two measurements are multiplied or divided, the error of the result is the sum of the relative errors of the two measurements. Identical rules apply to the propagation of deviations.

Example: The density of a solution was determined by first weighing a clean, dry, stoppered flask four times. A mass of 26.413 ± 0.005 g was obtained. 10.00 ± 0.02 ml of a solution was added with a pipette and the stoppered flask was again weighed four times, obtaining mass of 37.126 ± 0.003 g. Determine the density of the solution and its relative, average, and absolute deviations.

Solution: The mass of the solution is found by substracting the mass of beaker from mass of beaker after weighing with solution inside

(mass of beaker + solution) - (mass of beaker) = mass of solution

37.126 g – 26.413 g = 10.713 g.

The absolute deviation in this mass is found by adding the deviations in both mass of beaker and mass of beaker with solution inside.

0.005 g + 0.003 g = 0.008 g

The relative average deviation in mass is found by dividing absolute deviation to mass of solution and multiplying it by 100%

0.008 g/10.713 g x 100% = 0.07 %.

The relative average deviation in the volume is found by dividing average deviation to average volume and multiplying it by 100 %

$$(0.02 \text{ ml}/10.00 \text{ ml}) \text{ x}100 \% = 0.2 \%.$$

The density is, therefore, is obtained by dividing the mass of solution to the volume

$$10.713 \text{ g}/10.00 \text{ mL} = 1.071 \text{ g/mL}$$

The relative average deviation is found by addition of the deviations in both mass and volume measurements

$$0.07\% + 0.2\% = 0.3\%.$$

The absolute deviation of the density is

$$(0.3\% /100\%)(1.071 \text{ g/mL}) = 0.003 \text{ g/mL}.$$

Thus, we would report 1.071 ± 0.003 g/mL as the density.

SIGNIFICANT FIGURES

Associated with the evaluation of experimental data is an understanding of the extent to which the numbers in measured quantities are significant. For example, the mass of an object can be measured on two different balances, one a top-leading balance sensitive to the nearest 0.001 g and another a triple-beam balance sensitive to the nearest 0.1 g. These balances have a different uncertainty and precision.

	Top-loading Balance	Triple Beam Balance
Quantity	54.236 g	54.2 g
Uncertainty	$\pm$ .002 g	$\pm$ 0.1 g
Measured mass	54.236 ± 0.002 g	54.2 ± 0.1 g
Precision	high (2 parts in 54.236)	low (1 part in 542)

On the top loading balance with high precision, five significant figures are available. On the triple-beam balance only three figures are significant. Thus, significant figures are the numbers about which we have some knowledge.

If no information is available regarding the uncertainty of the measuring device, one may assume that all recorded figures are significant with an uncertainty of about one unit in the last digit. Zeros are significant if they are part of the measured quantity, but not if they are used to locate the decimal place. Thus, 62.070 has five significant figures while 0.0070 has only two (the first three zeros only locate the decimal place).

In calculations involving measured quantities with different numbers of significant figures, the result must be evaluated carefully with respect to the number of digits retained.

In addition, or subtraction, the number of digits retained is based on the least precise quantity. For instance, consider the following summation of masses.

$$
\begin{array}{r}
125.206 \text{ g} \\
20.4 \text{ g} \\
+ \quad 3.58 \text{ g} \\
\hline
149.186 \text{ g}
\end{array}
$$

Here the result should be rounded to 149.2 g since the least precise mass is known only to the first decimal place.

In multiplication and division, the number of significant figures retained in a result is equal to the number in the least reliably known factor in the computation. For example, in the determination of the density of an object, the measured mass is 54.723 g while the measured volume is 16.7 mL. The density of the object is 54.723 g/16.7 mL = 3.28 g/mL. Note that only three significant figures can be retained.

When a number is rounded, the last figure retained is increased by one unit if the one dropped is more than five and decreased by one unit if the one dropped is less than five.

Examples:

        a) 3.276 → 3.28   **b)** 149.74 → 149.7   **c)** 5.45 → 5.5

As we have emphasized, chemistry is very much an experimental science in which careful and accurate measurements are the very essence of meaningful experimentation. It is therefore, essential for the beginner student of chemistry to learn how scientific measurements are carried out properly using common measuring instruments. Further, it is equally important

for the student to acquire an appreciation of the significance of measurements and to apply learned technique to a common specific experiment.

In this experiment you will become familiar with how mass and volume measurements are carried out and how an evaluation of the measurements is reflected in the number of significant figures recorded. These mass and volume measurements will then be used to determine the density of (a) a metal bar (b) a salt solution by two different methods. Finally, the results of the density measurements will be evaluated with respect to their precision and accuracy.

The density of an object is one of its most fundamental useful characteristics. As an intensive property it is independent of the quantity of material measured since it is the ratio of the mass of an object to its volume.

The density of an object can be determined by a variety of methods. In this experiment you will practice using a balance to measure mass. In addition, you will learn how to measure volume using a graduated cylinder and a pipet and learn how to calibrate the pipet. A comparison of the results allows for the calculation of the relative average deviation which is a measure of the precision of the experiment. Also, in the case of the metal bar, the results of measuring the density of the bar may be compared with the accepted density value for the bar. Thereby the relative error (a measure of accuracy) for the density of the bar may be determined. The sections in the introduction of this laboratory manual pertaining to precision, accuracy, significant figures and laboratory notebook should be studied carefully before performing this experiment.

SAFETY PRECAUTIONS

Take special care in inserting the bar into the graduated cylinder. **Do not drip it in! The glass cylinder may break.**

Pipetting should always be done using a suction device. **Never suction by mouth**.

NOTE:
Record all measurements in your laboratory notebook (data sheet) in ink. The proper use of a sensitive balance is critical to useful mass measurements. Also, pipetting is a very useful, accurate, and common method for transferring exact volumes of liquids. Therefore, the instructor should demonstrate good balance and pipet techniques to the class at the beginning of the laboratory period. Please note that when a portion of the experiment contains the instruction ''Repeat...twice;'' each portion is to be performed all the way through three times: initially and two repetitions.

MATERIALS	CHEMICALS

MATERIALS

50 mL beaker
25 mL pipette
25 mL graduated cylinder
Suction Device
Balance
Metal Bar
Ruler

CHEMICALS

Distilled water
Salt Solution

PROCEDURE

PART I: MEASUREMENTS

A. Mass Measurements

After balance instruction, you will be assigned to select a balance for use during the experiment.

1. Zero the balance.
2. Measure the mass of a clean dry 50.00-ml beaker to the nearest ±0.001 g.
3. Record, in ink, your observation directly into the lab notebook/data sheet.
4. Remove the beaker from the pan. Again, zero the balance.
5. Weigh the same beaker as before (step 2) and record the result.
6. Repeat the steps 4 and 5 one more time.
7. From the three measurements, calculate the average mass of the beaker.

B. Volume Measurements

Use of a pipet: In order to accurately measure a liquid volume using a pipette, we must consider several things. Most volumetric pipets are designed to deliver rather than contained the specified volume. Thus, a small amount of liquid remains in the tip of the pipet after transfer of liquid. This kind of pipet is marked with the letters **''TD''** somewhere on the barrel of the calibration line. Also, for purposed of safety, **never pipet by mouth**; that is, **never use your mouth to draw liquid into the pipet**. **Always use a suction device/bulb** (Figure 1).
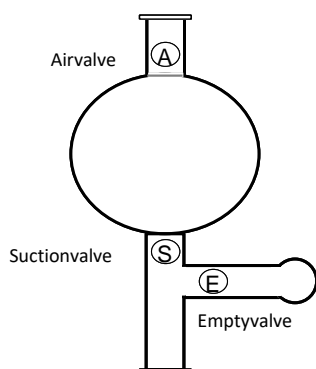
A: Air valve expels air from bulb.

S: Suction valve draws solution into the pipet.

E: Empty valve drains solution from the pipet.

Figure 1. Suction device/bulb.

Use a clean, and dry, 20.00 or 25.00-ml pipet. Rinse the pipette several times with small portions of the liquid to be transferred. To measure the desired volume, a volume of liquid greater than that to be measured is needed in order to keep the pipette tip under the liquid surface while filling.

Before using the suction device, deflate the air inside by pressing button A. Then connect the suction device on to pipette. Immerse the tip of the pipette inside the liquid, but not touch the bottom of the container (a chipped tip causes error) and hold it vertically. Press the button S for suction. Measure desired amount of liquid with pipette by taking meniscus point into account. While emptying use button E.

1. Measure the temperature in the laboratory. Your instructor will provide you with the density of water at this temperature.
2. Use the same 50.00-ml beaker from Section A for determining the mass of each liquid of water. Rather than weighing the empty beaker, the average mass of the beaker determined in Section A will be used as the mass of the dry beaker.
3. Measure 20.00 or 25.00-ml of water (depending on the size of pipette available) into the 50.00-ml beaker.
4. Record the volume of water measured with the pipette to the appropriate number of significant figures.
5. Record the number of significant figures in the volume measurement.
6. Weigh the beaker and water to the nearest mg (±0.001g).
7. Calculate the mass of water in the beaker.
8. Use the mass and density of water to determine the volume of water measured.
9. Repeat steps 3-8 using a 25.00-ml graduated cylinder and 50-ml beaker instead of the pipet to measure the 20.00 or 25.00 ml of water.

PART II: DENSITY

A. Density of A Metal Bar

Mass Measurement of A Metal Bar

NOTE: Use the same metal bar for all trials.

1. Zero your balance. Weigh a metal bar on a balance to the nearest mg (±0.001g) and record its value. Repeat step 1 twice. Do not allow the first measurement that you obtain to influence subsequent measurements that you take. Each time you weigh, make sure you zero the balance before proceeding.

Determine the volume of the metal bar by each of the following methods, making at least 3 measurements by each method. Do not allow the first measurement to influence the

subsequent measurements as your data will then be less significant for the purpose of measuring the precision of this experiment.

Volume Measurements of A Metal Bar

Method I: Graduated Cylinder

1. Insert the bar into a graduated cylinder wit enough water so that the bar is immersed. Note and record as precisely as possible the initial water level, and the water level after the bar is immersed. Read the lowest point of the meniscus in determining the water level and estimate the volume to one digit beyond the smallest scale division.
2. Discard the water and repeat this measurement twice with a different initial volume of water.
3. Calculate the average density of the bar.

Method II: Ruler

1. Measure the dimensions of the bar with a measuring stick ruled in centimeters and record the measured values.
2. Repeat these measurements twice.
3. Calculate the volume of the bar from these dimensions. Note that the bar is cylindrical in shape, therefore, the formula for the volume of a cylinder should be used:

$V = \pi r^2 h.$
4. Calculate the average density of the bar.

For each method, determine the relative error of your result comparing it with the accepted value as provided by your instructor. Which one of the two methods is more accurate? Explain.

B. Density of a salt solution

Method I: Pipette

1. Use the same 50.00-ml beaker from Section A for determining the mass of water. Rather than weighing the empty beaker, the average mass of the beaker determined in Section A will be used as the mass of the dry beaker.
2. With a clean 20.00 or 25.00 ml volumetric pipet, pipet the salt solution into the beaker and reweigh. Repeat this measurement twice. Calculate the average density of the salt solution.

Method II: Graduated Cylinder

1. Use the same 50.00-ml beaker from Section A for determining the mass of water. Rather than weighing the empty beaker, the average mass of the beaker determined in Section A will be used as the mass of the dry beaker.

2. Take 10.00 ml of salt solution using a clean empty dry 10.00-ml graduated cylinder, record the volume as precisely as possible. And then put it into the beaker (use the same 50.00-ml beaker from Section A) and reweigh. Repeat this measurement twice. Calculate the average density of the salt solution.

For each method, determine the relative average deviation of your results. Which method is more precise? Explain.

DISPOSAL
Salt solutions: Do one of the following, as indicated by your instructor.
Recycle: return the salt solution to its original container.
Treatment/Disposal: Dilute the salt solution 1:10 with tap water and flush down the sink with running water.
Disposal: Put the salt solution in a labeled waste bottle.

Name_____Department_____Group_____

DATA&RESULTS

PART I: MEASUREMENTS

A. Mass Measurements

	Trial 1	Trial 2	Trial 3
Mass of 50 mL beaker, g	_____	_____	_____
Average mass of beaker, g		_____	

B. Volume Measurements

	Transfer pipet	Graduated cylinder	Beaker
Volume of water (direct reading)	_____	_____	_____
Number of significant figures for volume	_____	_____	_____
Mass of beaker + water, g	_____	_____	_____
Mass of dry beaker, g (average from Part A)	_____	_____	_____
Mass of water, g	_____	_____	_____
Volume of water calculated from mass, ml	_____	_____	_____

Name_____Department_____Group_____

PART II: DENSITY

A. Density of a metal bar

	Trial 1	Trial 2	Trial 3
Mass of bar, g	_____	_____	_____
Average mass of bar, g		_____	
Accepted density of metal bar, g/cm^3		6.70 g/cm^3	

Method I Volume (with graduated cylinder)

	Trial 1	Trial 2	Trial 3
Final level of water, ml	_____	_____	_____
Initial level of water, ml	_____	_____	_____
Volume of bar, cm^3	_____	_____	_____
Density of bar, g/cm^3	_____	_____	_____
Average density of metal bar, g/cm^3		_____	
Relative Error		_____	

Method II Volume (with measuring stick)

	Trial 1	Trial 2	Trial 3
Diameter, cm	_____	_____	_____
Height, cm	_____	_____	_____
Volume of bar, cm^3	_____	_____	_____
Density of bar, g/cm^3	_____	_____	_____
Average density of metal bar, g/cm^3		_____	
Relative Error		_____	

Name_____Department_____Group_____

B. Density of a salt solution

Method I (with pipet)

	Trial 1	Trial 2	Trial 3
Mass of beaker, g (from Part A)	_____	_____	_____
Mass of beaker + solution, g	_____	_____	_____
Mass of solution, g	_____	_____	_____
Volume of solution, ml	_____	_____	_____
Density of salt solution, g/ml	_____	_____	_____
Average density of salt solution, g/ml		_____	
Relative Average Deviation		_____	

Method II (with graduated cylinder)

	Trial 1	Trial 2	Trial 3
Mass of beaker, g (from Part A)	_____	_____	_____
Mass of beaker + solution, g	_____	_____	_____
Mass of solution, g	_____	_____	_____
Volume of solution, ml	_____	_____	_____
Density of salt solution, g/ml	_____	_____	_____
Average density of salt solution, g/ml		_____	
Relative Average Deviation		_____	

Name_____Department_____Group_____

CALCULATIONS

PART I: MEASUREMENTS

A. Mass Measurements

Average mass of beaker, g:

B. Volume Measurements

	Transfer pipet	Graduated cylinder	Beaker
Volume of water calculated from mass, ml			

Which laboratory equipment (transfer pipet, graduated cylinder, beaker) is more accurate based on your calculations?

Name_____Department_____Group_____

PART II: DENSITY

A. Density of metal bar

Average mass of bar, g:

Method I Volume (with graduated cylinder)

Average density of metal bar, g/cm^3

Method II Volume (with measuring stick)

	Trial 1	Trial 2	Trial 3
Volume of bar, cm^3			
Density of bar, g/cm^3			

Name_____Department_____Group____

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	Trial 1	Trial 2	Trial 3
Average density of metal bar, g/cm^3			
Relative error			

Which method is more accurate based on your calculations?

Name_____Department_____Group_____

B. Density of a salt solution

With pipet:

	Trial 1	Trial 2	Trial 3
Density of salt solution, g/ml			
Average density of salt solution, g/ml			
Relative average deviation			

Name_____Department_____Group____

With graduated cylinder:

	Trial 1	Trial 2	Trial 3
Density of salt solution, g/ml			
Average density of salt solution, g/ml			
Relative average deviation			

Which method is more precise based on your calculations?

Name_____Department_____Group_____

REPORT QUESTIONS

1. From your data, calculate the volume occupied by 100.0 g of:

a) salt solution

b) metal bar

2. From your answers to question 1 above, which occupies the larger volume, the metal bar or the salt solution? Explain your answer in the context of the densities of solids and liquids, in general.

3. Define the terms, precision and accuracy in such a way as to distinguish them. Which of these terms can be measured for the metal bar?

4. Are your results for the metal bar, more precise or more accurate? Explain.

5. From your data for the salt solution, evaluate the two methods in terms of their precision. Which method should lead to greater precision? Which method is more precise? Explain.

Name_____Department_____Group____

PRELAB QUESTIONS

The following data were obtained in order to determine the density of cylindrical metal bar:

	Trial 1	Trial 2	Trial 3
Height (cm)	6.50	6.45	6.44
Diameter (cm)	1.25	1.26	1.22
Mass (g)	46.683	46.332	47.014

In the following calculations on this data, show the formula used, the substituted numbers and the result.

1. Calculate the average density of the metal bar.

2. Calculate the percent relative average deviation of the measurements.

3. If the accepted value for the density of the bar is 6.70 g/cm^3, what is the % relative error?

4. Are these measurements more precise or more accurate? Explain.

5. What is the purpose of repetition in measurements?

2 The Stoichiometry of a Reaction

INTRODUCTION

The object of this experiment is to determine the mol ratio with which two substances combine chemically. Such information is helpful in determining the stoichiometry of the reaction, that is, the balanced chemical equation and the information that can be derived from it.

Stoichiometry is the measurement of quantitative relationships in chemical formulas and equations. Mostly in chemical reaction two or more reactants (reagents) are placed. The reagent that is totally consumed before other reactants at that time reaction stops defined as limiting reagent. Limiting reagent is the reactant, which is totally consumed before other reactants. The limiting reactant limits theamount of product that can be formed since the consumption of the reactant stops the reaction. The reactant that is remained once the limiting reagent is consumed is called as excess reagent

The principle underlying this experiment is based on maintaining a constant quantity of reaction A while varying the quantity of reaction B in series of experiments. The mass of an insoluble product C is then used as an indicator of the effect of varying the quantity of B. When sufficient B has been added to react completely with the constant of A there will be no further increase in C and the mole ratio of A to B can be deduced.

For example, the stoichiometry of the cadmium nitrate-sodium sulfide system was investigated using this technique. With a constant volume (6.00 mL) of 1.00 M $Cd(NO_3)_2$ solution [6.00 mmol $Cd(NO_3)_2$], the quantity of insoluble product was plotted against the number of millimoles of Na_2S (Figure 2-1). The mass of precipitate formed for each volüme of the Na_2S solution is given in Table 2-1. The two straight lines in Figure 2-1 intersect at 0.85 g of precipitate, which corresponds to 6.00 mmol Na_2S. Since 6.00 mmol of $Cd(NO_3)_2$ was used, the mole ratio of Na_2S to $Cd(NO_3)_2$ is 6.00 to 6.00 or 1:1. This suggests that the precipitate could be CdS or $NaNO_3$. Since nitrates are generally soluble and sulfides are generally insoluble, CdS is indicated as the precipitate. The equation for the reaction can then be written.
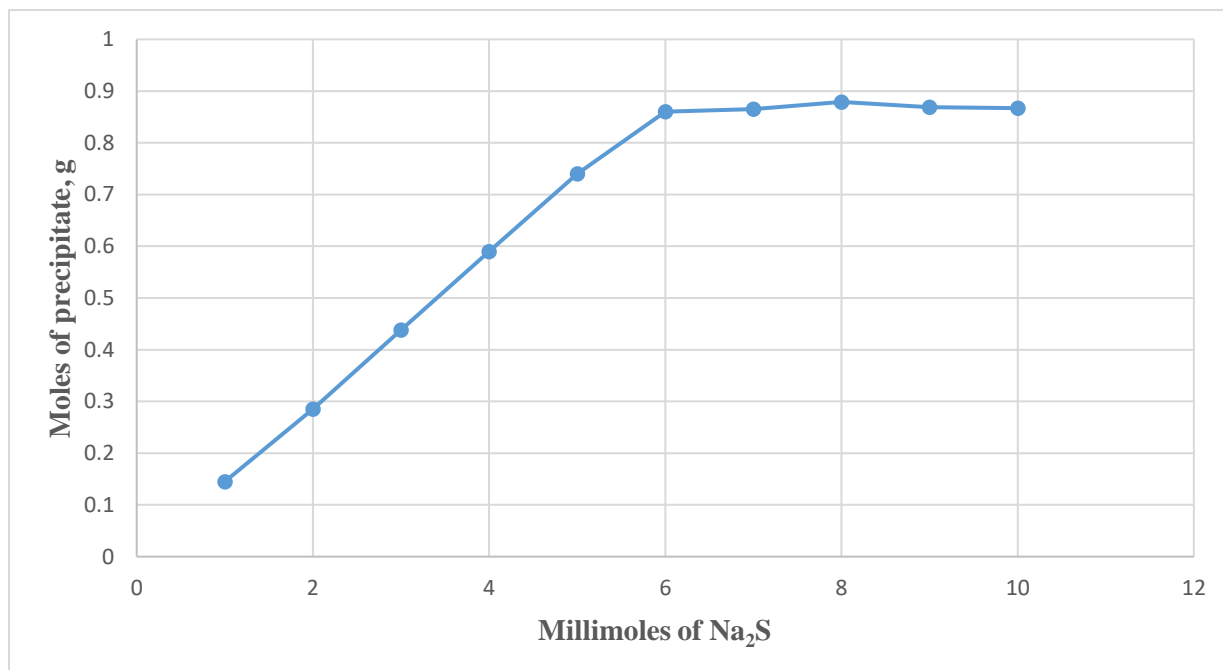
$$Na_2S \text{ (aq)} + Cd(NO_3)_2 \text{ (aq)} \rightarrow CdS \text{ (s)} + 2 NaNO_3 \text{ (aq)}$$
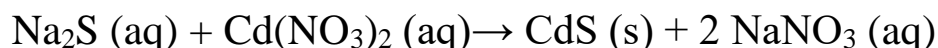
Figure 2.1. Plot of Mass of Precipitate vs. Millimoles of Na$_2$S

Table 2.1. Data for reaction of 1.00 M Na$_2$S with 1.00 M Cd(NO$_3$)$_2$

Assignment number	1,00 M Na$_2$S		1,00 M Cd(NO$_3$)$_2$		Precipitate, g
	Volume, mL	Number of mmol	Volume, mL	Number of mmol	
1,00	1,00	1,00	6,00	6,00	0,145
2,00	2,00	2,00	6,00	6,00	0,285
3,00	3,00	3,00	6,00	6,00	0,438
4,00	4,00	4,00	6,00	6,00	0,59
5,00	5,00	5,00	6,00	6,00	0,74
6,00	6,00	6,00	6,00	6,00	0,86
7,00	7,00	7,00	6,00	6,00	0,865
8,00	8,00	8,00	6,00	6,00	0,879
9,00	9,00	9,00	6,00	6,00	0,869
10,00	10,00	10,00	6,00	6,00	0,867

To confirm the identity of the precipitate, one computes the masses of NaNO$_3$ and of CdS that would be formed from 6.00 mmol Na$_2$S.

$$6.00 \text{ mmol Na}_2\text{S} \times \frac{2 \text{ mmol NaNO}_3}{1 \text{ mmol Na}_2\text{S}} \times \frac{0.085 \text{ g NaNO}_3}{1 \text{ mmol NaNO}_3} = 1.02 \text{ g NaNO}_3$$

$$6.00 \text{ mmol Na}_2\text{S} \times \frac{1 \text{ mmol CdS}}{1 \text{ mmol Na}_2\text{S}} \times \frac{0.145 \text{ g CdS}}{1 \text{ mmol CdS}} = 0.870 \text{ g CdS}$$

Since the constant mass of precipitate averages 0.868 g, which is much closer to the 0.870 mass values of CdS than to the 1.02 mass value of $NaNO_3$, the identity of the precipitate as CdS is confirmed.

Forming Precipitates

When a solid precipitate forms from a supersaturated solution, the particle size may be so small that some of the particles pass through the filter paper. This is the case with the bright yellow precipitate formed in this experiment. To create larger particles of solid, nitric acid is added to the reaction mixture and the mixture is gently heated. Nitric acid neutralizes the excess charge on the surface of the precipitate particles.

This excess charge consists of extra anions or cations on the surface of each particle. For example, when CdS (s) forms from a solution with excess Cd^{2+} (aq) ion, the particles of solid are positively charged. Since like charges repel, the solid particles cannot coagulate into larger particles until the excess positive charge is neutralized. Heating the reaction mixture dissolves the smaller particles of precipitate. This Works because most solids are more soluble at high temperatures and because dissolving occurs at the surface of a particle. The smaller particles have a relatively larger surface area than larger ones. When the heated solution is allowed to cool, the dissolved solid re-precipitates on the larger particles that remain. Thus, the net result of this technique, known as digestion, is to replace the small particles with large ones.

SAFETY PRECAUTIONS

Be particularly careful in handling chemicals. Wipe up all spills promptly with a damp sponge or towel. If any chemicals get on your hands or clothing, wash immediately with soap and running water. Heat the reaction mixture on a hot plate in the hood with glass door closed as much as possible. Watch the reaction mixture constantly while it is being heated and stir it continually to minimize spattering. Do not look down into the beaker while it is being heated. Be very careful when handling hot beakers; use either beaker tongs or a carefully folded towel.
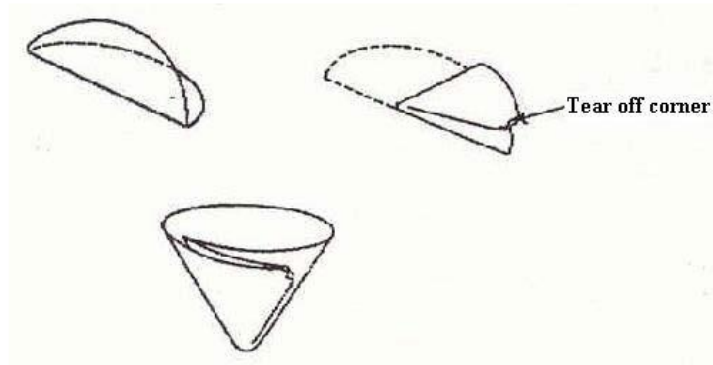
PROCEDURE

Assignment number	1	2	3	4	5	6	7	8	9	10	11	12
Volume of 0.100 M Pb(NO3)2, mL	6,00	8,00	10,00	10,50	11,00	11,50	12,00	12,50	13,00	14,00	16,00	17,00
Volume of 0.100 M K2CrO$_4$, mL	12,00	12,00	12,00	12,00	12,00	12,00	12,00	12,00	12,00	12,00	12,00	12,00

In this experiment the stoichiometry of the lead (II) nitrate - potassium chromate system will be investigated. Note that the volume of 0.100 M K_2CrO_4 (*potassium chromate*) is held constant at 12.00 mL, while the volume of 0.100 M $Pb(NO_3)_2$ (*lead II nitrate*) is varied in each assignment.

Using a burette, dispense 12.00 mL of 0.100 M K_2CrO_4 solution into a clean, dry, small (100- or 150-mL) beaker. From a second burette, dispense the assigned volume of 0.100 M $Pb(NO_3)_2$ solution, slowly pour the 12.00 mL of the K_2CrO_4 solution into the beaker containing the $Pb(NO_3)_2$ solution. Then rinse the K_2CrO_4 beaker with 10 mL of a 5% nitric acid solution and add this rinse solution to the beaker. Heat the beaker on a hot plate in the hood for 2-3 min. At this point, most of the precipitate will have settled to the bottom of the beaker. *Watch the reaction mixture carefully while it is on the hot plate.* It will have a tendency to spatter. Stir the contents of the beaker to minimize spattering

Your instructor will demonstrate the correct filtration technique. Determine the mass of a piece of filter paper to the nearest milligram. Fold the paper and insert it into a funnel and moisten with distilled water. Carefully pres the top of the filter paper to the funnel such that a slight suction is created. Use the glass stirring rod to direct the liquid, and pour the liquid from the beaker onto the filter paper in the funnel. Next, wash the precipitate in the beaker with 10 mL of cold distilled water and quantitatively transfer the precipitate to the filter paper. Use cold distilled water from your wash bottle to rinse the remainder of the precipitate out the funnel and place it on a paper towel in your locker to dry until the next laboratory period. Be very careful at this stage, as wet filter paper has a tendency to tear.

Your instructor will demonstrate the correct filtration technique. Determine the mass of a piece of filter paper to the nearest milligram. Fold the paper and insert it into a funnel and

moisten with distilled water. Carefully pres the top of the filter paper to the funnel such that a slight suction is created. Use the glass stirring rod to direct the liquid, and pour the liquid from the beaker onto the filter paper in the funnel. Next, wash the precipitate in the beaker with 10 mL of cold distilled water and quantitatively transfer the precipitate to the filter paper. Use cold distilled water from your wash bottle to rinse the remainder of the precipitate out the funnel and place it on a paper towel in your locker to dry until the next laboratory period. Be very careful at this stage, as wet filter paper has a tendency to tear.

Now repeat the above procedure for a second determination using the same assigned volume of $Pb(NO_3)_2$. If time permits, do a third determination. In each determination the filter paper should be marked (with a pencil) before weighing so it can be correctly identified after dying.

During the next laboratory period weigh the filter paper with the solid precipitate to the nearest milligram. Subtract the mass of the filter paper to find the mass of the precipitate. The experimental results for each trial you performed will give one data point which will be used with the class data to determine the stoichiometry of the reaction. Record your values for volume of $Pb(NO_3)_2$, millimoles of $Pb(NO_3)_2$, and mass of precipitation in a table with the class data.

Disposal:

Solid lead (II) chromate: Dispose solid and filter paper in a waste bottle labeled for this compound.

Unused solutions and filtrate solutions: Pour into the same waste bottle labeled for this purpose. These wastes will be treated later to precipitate all the lead (II) and chromate ions for solid waste disposal.

Name_____Department_____Group_____

DATA
Fill in the blanks with doing suitable calculations

Mass of filter paper, g: 0,833

Assignment Number	Volume of 0.100 M K_2CrO_4, mL	Number of millimoles of K_2CrO_4	Volume of 0.100 M $Pb(NO_3)_2$, mL	Number of millimoles of $Pb(NO_3)_2$	Mass of precipitate and filter paper, g	Mass of precipitate, g	Mole ratio of K_2CrO_4 to $Pb(NO_3)_2$
1	12,00		6,00		1,01425		
2	12,00		8,00		1,18925		
3	12,00		10,00		1,3805		
4	12,00		10,50		1,5705		
5	12,00		11,00		1,758		
6	12,00		11,50		1,908		
7	12,00		12,00		1,91425		
8	12,00		12,50		1,93175		
9	12,00		13,00		1,91925		
10	12,00		14,00		1,91675		
11	12,00		16,00		1,94425		
12	12,00		17,00		1,92175		

Name_____**Department**_____**Group**_____

Graph

  Using datas, plot the mass of precipitate on the ordinate and the amount in millimoles of 0.100 M $Pb(NO_3)_2$ on the abscissa. Select the scale units on the ordinate to range from 0 to 0.1 g more than the maximum mass of precipitate reported. Select the scale units for the abscissa to range from 0 to 2.00 mmol of $Pb(NO_3)_2$.

  Draw the best straight-line curve through the plotted points on the left-hand side of the graph. Draw a second best straight-line curve through the plotted points on the right-hand side of the graph. The point of intersection of the two straight lines is the stoichiometric point for the system. The **stoichiometric point** represents the number of millimoles of $Pb(NO_3)_2$ that react with 1.20 mmol of K_2CrO_4.

Data at the stoichiometric point
millimoles of Pb(NO3)2, mmol _____
mass of precipitate, g _____

CALCULATIONS AND QUESTIONS

1. Calculate the amount in millimoles of K_2CrO_4 in 12 mL of 0.100 M K_2CrO_4 solution.

[(Number of millimoles) = (volume, mL) x (molarity).]

2. Calculate the amount in millimoles of $Pb(NO_3)_2$ in your assigned volume of 0.100 M $Pb(NO_3)_2$solution.

3. Write the balanced molecular equation for the reaction between K_2CrO_4 (aq) and $Pb(NO_3)_2$(aq).

4. Based on your answers to above questions, which reactant is the limiting reagent in the reaction for your assigned volume of $Pb(NO_3)_2$? How many millimoles of each product will be produced? Show your sample calculations.

5. Which product of the reaction is likely to be the one that precipitated? Why? Confirm your answer with a calculation based on the mass of precipitate. (Hint: Find the stoichiometric point from graph. Consider your graph while answering this question.)

6. How would the following modifications of the experimental procedure used in this experiment affect the mass of precipitate obtained? State whether the mass would be more, less or unchanged. Assume that other aspects of the procedure are performed as specified.

a) The mixture is not heated on the hot plate at all.

b) The reactants are mixed at 40°C rather than room temperature.

c) The nitric acid solution is not added to the reaction mixture.

d) The precipitate is not washed with distilled water after filtering.

e) The student who performed assignment number 3 combined incorrectly 10.00 mL of 0.100 M K_2CrO_4 with 12 mL of $Pb(NO_3)_2$ rather than combination specified in the procedure.

Name⎯⎯⎯⎯⎯⎯⎯⎯⎯⎯⎯⎯⎯⎯⎯Department⎯⎯⎯⎯⎯⎯⎯⎯⎯⎯⎯⎯Group⎯⎯⎯

PRELAB QUESTIONS

1. What are the chemical formulas of the reagents mixed in this experiment?

2. Why is it necessary to warm the reaction mixture?

3. Why should more than one trial be run for each assignment?

4. When is the filter paper weighed?

5. What is meant by the **"stoichiometric point"** of a reaction?

3 Titration of Acids and Bases

INTRODUCTION

This experiment is in one that will already be familiar to many students. For several reasons, however, it might be worthwhile to repeat it. First, it gives one a chance to improve and perfect experimental technique, especially for the degree of accuracy and precision required in this exercise. Second, it can serve to reinforce one's knowledge of stoichiometry as well as to provide the self-confidence that comes with doing a thing well and with understanding.

Titration is the process of mixing measured volumes of reacting solutions in such a manner that one can determine when chemically equivalent amounts of reactants are present. The purpose of titration is to determine the concentration of solution of unknown strength. The concentration of one of the solutions, expressed as molarity, in this case, must be known. After titration, the molarity of the other solution can be calculated.

The equivalence point of a titration is the point at which equivalent amounts of reactants have been mixed. In order to determine the equivalence, point one usually adds as a visual indicator to the solution to be titrated. Such an indicator, if properly selected, undergoes a sharp color change at (or very near) the equivalence point. The point of the titration at which the indicator changes color is known as the end point. If the indicator is chosen correctly, the end point will occur at the same instant when the equivalence point has been reached.

In acid-base titrations, phenolphthalein often is used as the indicator (Figure 3-1). Phenolphthalein is colorless in acidic solution. At a pH of about 8.3, it undergoes a sharp change to a pink color as one adds a base. At a pH of about 10, it is red.

Other visual indicators are available for different pH ranges. In the titration of a strong acid with a strong base, the pH of the solution increases rapidly and has a value of 7 at the equivalence point. Since equivalence points for many acids lie between a pH of 7 to 9, phenolphthalein is an excellent general-purpose acid-base indicator.
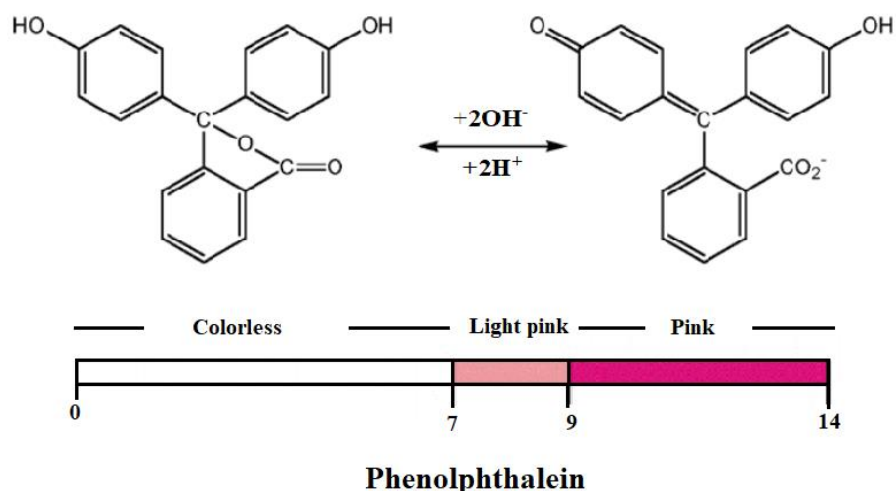
Figure 3.1. Phenolphthalein Behavior Over the pH Scale and Molecular Structure

SAFETY PRECAUTIONS

Both HCl and NaOH are harmful to skin and eyes. If any gets on the skin, wash thoroughly with water.
Wear eye protection! If any gets in your eye, rinse immediately and thoroughly with water.
Never add water to acid. Always add acid to water.

PROCEDURE

Required Lab. Materials

Chemical	Glass
Phenolphthalein solution	Beaker
0.1 M NaOH solution	Burette
0.1M HCI solution	Erlen mayer flask
Distilled water	Clamp
	Stand

A. Standardization of NaOH Soluion

Into a clean 500 mL Florence flask place enough NaOH pellets to provide about 2 g of compound. Add approximately 250 mL of water and mix thoroughly to dissolve the NaOH. Add 150 mL more water and mix thoroughly. Add water to the bottom of the neck of the flask and mix thoroughly. This should yield a homogeneous solution of approximately 0.1 M NaOH. The concentration of your NaOH solution is now known only approximately (to one significant figure). *(You are not supposed to prepare this solution because it is already on your bench)*

You will be supplied with a clean burette and a clean pipet. Rinse your burette with about 5 mL of your 0.1 M NaOH solution. Next, fill the burette to the 0.00 mL mark with this

solution. Make sure the tip of the burette is filled. The level of the liquid in the burette is read at the bottom of the meniscus.

Rinse the pipet twice with about 5 mL of the standard 0.100 M HCl solution. This solution will be available in the laboratory. Transfer 25.00 mL of the standard 0.100 M HCl solution into a clean Erlenmeyer flask. Add 2 or 3 drops of phenolphthalein indicator solution. Titrate this acid solution with your NaOH solution until a faint pink color persists for over 15 sec (See Note). Swirl the solution in the Erlenmeyer flask as you titrate and rinse the walls of the flask with distilled water from a wash bottle prior to the end point. Record the volume (to 0.01 mL) of base used.
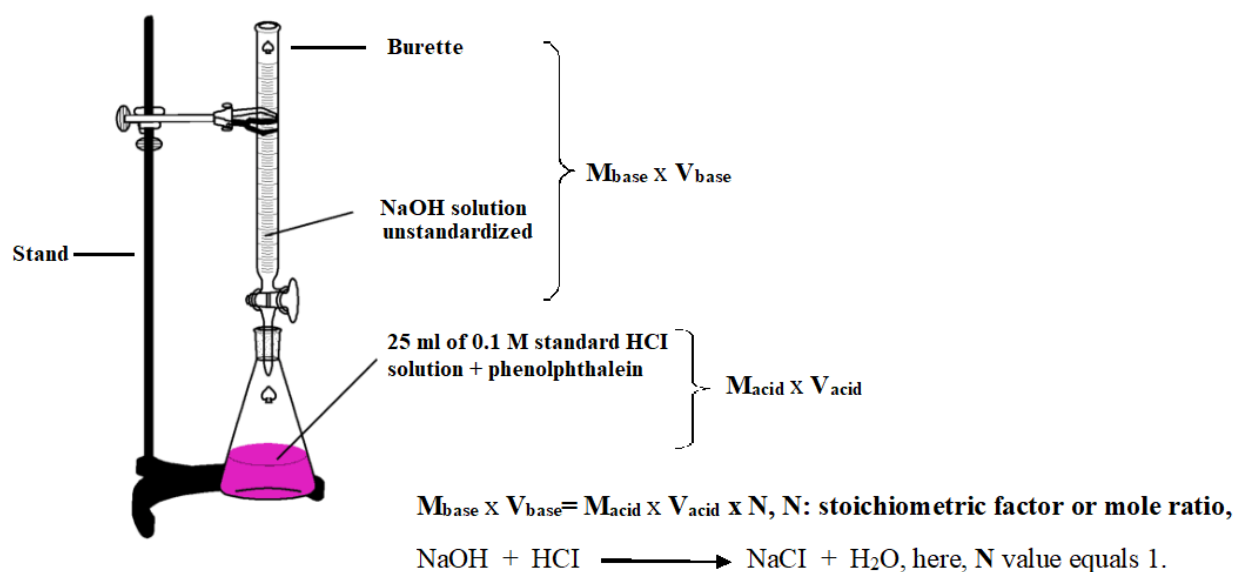

Figure 3.2. Standardization of NaOH solution-Setup

Repeat the titration at least twice using 25.00 mL portions of 0.100 M HCl solution and record your data. The molarities of your base as calculated from the titration a-data should agree to within 1% (relative average deviation) of the average value. If this is not true, repeat the titration with 25.00 mL 0.100 M HCl until two successive trials agree to within 1%.

Note: The color will fade on sitting as the carbon dioxide gas in the air dissolve and reacts with the slight excess of NaOH.

B. Titration of Unknown Acid

In a clean, dry beaker obtain an unknown acid sample from your instructor. This acid is monoprotic. Rinse the pipet that was filled with HCl solution with three 5 mL portions of distilled water and then with three 5 mL of portions of your unknown acid solution. Transfer a 25.00 mL portion of this solution into a clean Erlenmeyer flask. Add 2 or 3 drops of phenolphthalein indicator to each solution. Titrate with the standardized NaOH solution until a faint pink color persists for over 15 sec while the solution is being swirled. Use the above data and the average molarity of NaOH solution determined in Part A, to calculate the molarity of your unknown acid. Repeat the titration until the calculated molarities of the unknown acid are within 1% relative average deviation. Record your data in your lab notebook.

Figure 3.2. Calculation of unknown HCI concentration-Setup

Disposal:

Titration solutions: Since these are virtually neutral salt solutions, they may be flushed down the sink with running water.

Unused HCl and NaOH: Mix unused acid and base in a large beaker. Add phenolphthalein and neutralize with 1 M HCl (the first disappearance of pink color) or NaOH (first appearance of permanent pink color). Flush the resulting salt solution down the sink with running water.

Name_____Department_____Group_____

DATA&RESULTS

Standardization of NaOH Solution

	Trial 1	Trial 2	Trail 3	Trial 4
Molarity of HCl		_____ _____		
Volume of HCl used, mL	_____	_____ _____		_____
Initial buret reading, mL	_____	_____ _____		_____
Final buret reading, mL	_____	_____ _____		_____
Volume of NaOH used, mL	_____	_____ _____		_____
Molarity of NaOH, mmol/L	_____	_____ _____		_____
Average Molarity of NaOH	_____	_____ _____		_____
Relative Average Deviation in M	_____	_____ _____		_____

A. Titration of Unknown Acid

	Trial 1	Trial 2	Trail 3	Trial 4
Molarity of NaOH (avg. from Part A)		_____ _____		
Initial burette reading, mL	_____	_____ _____		_____
Final burette reading, mL	_____	_____ _____		_____
Volume of NaOH used, mL	_____	_____ _____		_____
Volume of Unknown used, mL	_____	_____ _____		_____
Molarity of Unknown, mmol/L	_____	_____ _____		_____
Average Molarity of Unknown	_____	_____ _____		_____
Relative Average Deviation in M	_____	_____ _____		_____

Name_____**Department**_____**Group**_____

CALCULATIONS

The stoichiometry for this titration is calculated from the balanced equation for the chemical reaction.

$$HCl_{(aq)} + NaOH_{(aq)} \rightarrow NaCl_{(aq)} + H_2O \qquad [3.1]$$

Part A:

At the equivalence point, the mmol NaOH added equals the mmol of HCl present. Since mmol = M x mL, it can be said that, at the end point of the titration,

(mL NaOH added)(M NaOH) = (25.00 mL HCl)(0.100 M HCl)   [3.2]

Part B:

By substituting the average value of the molarity of NaOH (Part A) and its end point volume into equation [3.2], we can calculate the molarity of the unknown acid from the relationship

mL NaOH added)(average M NaOH) = (25.00 mL)(M unknown acid)   [3.3]

Name_____**Department**_____**Group**_____

REPORT QUESTIONS

1. Concentrated HCl is approximately 12.1 M and is 37% HCl by mass. What is the density of concentrated HCl in g/mL?

2. An acid solution is titrated to the pink phenolphthalein endpoint. As time passes the pink color fades and the solution becomes colorless again. Explain why this happens.

3. If the volumes of NaOH needed for successive trials in Part A increased by more than 2%, what is likely to be the cause and what should be done about it ?

4. A solution from Part A is titrated but some of the acid sample splashed onto the inside walls of the flask and was not rinsed down prior to or after the endpoint was reached. Will the calculated molarity of the NaOH solution be erroneously high, low or not changed? Explain.

5. During the titration in Part B some NaOH leaked from the burette into the reaction flask but was not noticed by the experimenter who then recorded the volume of NaOH used. Will the calculated molarity of the unknown be erroneously high, low or not changed? Explain.

Name_____**Department**_____**Group**_____

PRELAB QUESTIONS

1. What is a titration? Give a precise definition.

2. How does the equivalence point of a titration differ from the endpoint?

3. Why is phenolphthalein added to the reaction mixture?

4. How long should the indicator color persist at the endpoint of the titration?

5. Why is phenolphthalein a suitable indicator even though it does not change color until a pH of 8.3 is reached?

4 Oxidation – Reduction Titration

INTRODUCTION

In addition to its application to acid-base reactions, titration techniques may be applied to other types of reactions, such as oxidation-reduction. In this experiment, the oxidation of $K_2C_2O_4$ *(potassium oxalate)* by $KMnO_4$ *(potassium permanganate)* will be studied. KMO_4 is a inorganic compound and a strong oxidizing agent, also it is known as permanganate of potash or Condy's crystals. Pure $K_2C_2O_4$ is a solid salt and is a very good primary standard. It will be used first to standardize a $KMnO_4$ titrant, which then will be used to determine the percentage purity of unknown oxalate sample. The reaction is carried out in the presence of dilute sulfuric acid which prevents the formation of the brown, insoluble MnO_2 and ensures that reduction of permanganate proceeds according to equation [4.1]

$$5\ e^- + MnO_4^- + 8H^+ \longrightarrow Mn^{2+} + 4H_2O \qquad [4.1]$$

At the same time oxalate ion $C_2O_4^{2-}$, is oxidized according to equation [4.2]

$$C_2O_4^{2-} \longrightarrow 2\ CO_2 + 2\ e^- \qquad [4.2]$$

The combination of half-reactions [4.1] and [4.2] yields the overall reaction, equation [4.3]

$$5\ C_2O_4^{2-} + 2\ MnO_4^- + 16H^+ \longrightarrow 10CO_2 + 2Mn^{2+} + 8H_2O \qquad [4.3]$$

The permanganate-oxalate reactions system contains a built-in indicator. The end point of the titration is ascertained by noting the presence of the first excess of permanganate ions in the solution being titrated. Thus, when the oxalate has all reacted, the first excess of added permanganate solution will no longer be decolorized via reaction [4.3], and the solution will change from colorless to pink. One drop of excess permanganate solution is sufficient to cause this color change.

At room temperature the rate of the reaction is too slow to give a sharp, accurate end point. Thus, the titration is carried out in the presence of a small quantity of manganese (II) sulfate, $MnSO_4$, which catalyzes the reaction. (Note that Mn^{2+} is also a product of the overall reaction. Such a reaction, in which a product act as a catalyst, is said to be auto-catalyzed) Alternatively, the solution may be heated to 60-70 ^{0}C to speed up the reaction.

Solid $KMnO_4$ usually is contaminated with manganese dioxide, MnO_2, which catalyzes the reduction of the permanganate in aqueous solution according to equation [4.4].

$$4\ MnO_4^- + 2H_2O \longrightarrow 4\ MnO_2 + 3\ O_2 + 4\ OH^- \hspace{3cm} [4.4]$$

Therefore, before the $KMnO_4$ solution can be standardized, any MnO_2 contaminant must be removed. This is done by filtration through glass wool. Filter paper must not be used because $KMnO_4$ is readily reduced by the organic matter. By taking these precautions, however, standard $KMnO_4$ solutions may be stored for months.

The prepared $KMnO_4$ used in this experiment will be pre-filtered, and the H_2SO_4 will have $MnSO_4$ added to it prior to their use in this experiment. All titrations are to be carried out with a relative precision of within ±1%. When two successive trials agree within 1%, one need not perform another trial.

SAFETY PRECAUTIONS

These chemicals can be harmful or toxic. If any spills on skin, clean, rinse and wipe thoroughly.

Avoid ingestion of any of these chemicals and wash your hands before leaving the laboratory.

PROCEDURE

Required Lab. Materials

Chemical	Glass
K2C2O4 (Solid)	Beaker
0.1 M KMnO4 solution	Burette
3M H2SO4 solution	Erlen mayer flask
MnO_2 (solid)	Clamp
Distilled water	

A. Standardization of Prepared Permanganate Solution

Weigh about 0.25 g (to ±0.001 g) of $K_2C_2O_4$ into a 150 mL beaker. Dissolve the $K_2C_2O_4$ in about 50 mL of distilled H_2O and acidify with 10 mL of 3M H_2SO_4, to which some manganese (II) sulfate has been added. You may wish to heat this solution to near boiling before titrating. This has no effect on the titration except to make the reaction go faster. Titrate with the prepared permanganate solution, stirring constantly.

The end point is the first persistence of the permanganate color. Be careful not to add the permanganate solution too rapidly initially, as the color will not clear if this is done. One should add no more than 1 mL of permanganate solution before stopping and waiting until the solution clears. The color change in this experiment is not rapid like that of phenolphthalein. Consequently, titration proceeds more slowly. If your solution turns a permanent muddy brown (MnO_2 formation) rather than pink or clear, you should discard the entire trial and start again.
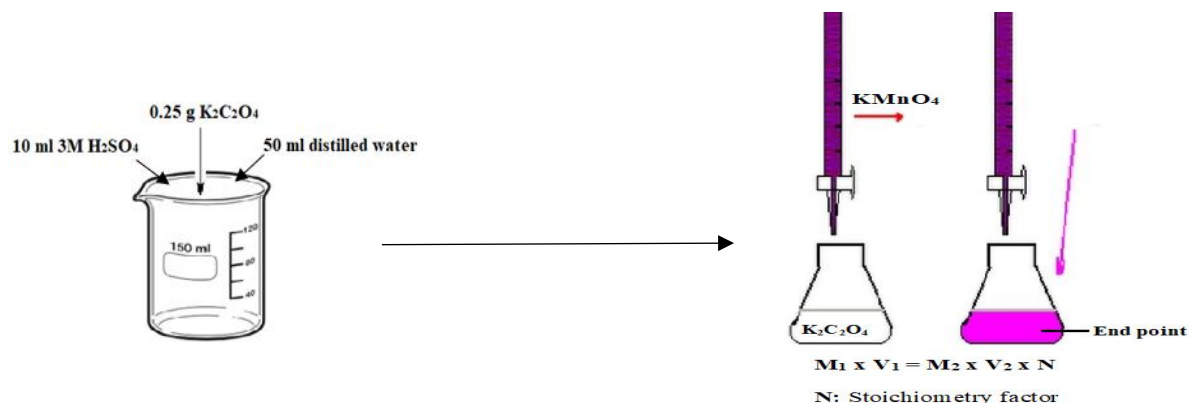
Figure 4.1. Standardization of Prepared Permanganate Solution

Use the mass of pure $K_2C_2O_4$ and the mL of $KMnO_4$ required reaching the end point to calculate the molarity of $KMnO_4$ titrant. In order to determine the relative precision of the titrations, calculate the ratio g $K_2C_2O_4$ / mL $KMnO_4$ for each titration. These ratios should be consistent to within ±1% for two consecutive trials before you go on the next part.

B.   Analysis of Unknown Oxalate

Weigh 0.75 g (to ±0.001 g) of the unknown solid into a 250mL beaker and follow the same procedure as in Part A. Use the mL $KMnO_4$ and its average molarity to calculate the mass and percentage of $K_2C_2O_4$ in the unknown sample. Calculate the value of the same ratio (g unknown/mL $KMnO_4$) as you did in Part A and it should have the same precision as it did there.

Here, $V_{initial} - V_{final} = V_{usedKMO4}$, $M_{unknown} \times V_{usedunknown} = M_{KMnO4} \times V_{usedKMnO4} \times N$

N: Stoichiometric factor

$$5C_2O_4^{-2} + 2MnO_4^{-2} + 16H^+ \longrightarrow 10CO_2 + 2\ Mn^{+2} + 8H_2O$$
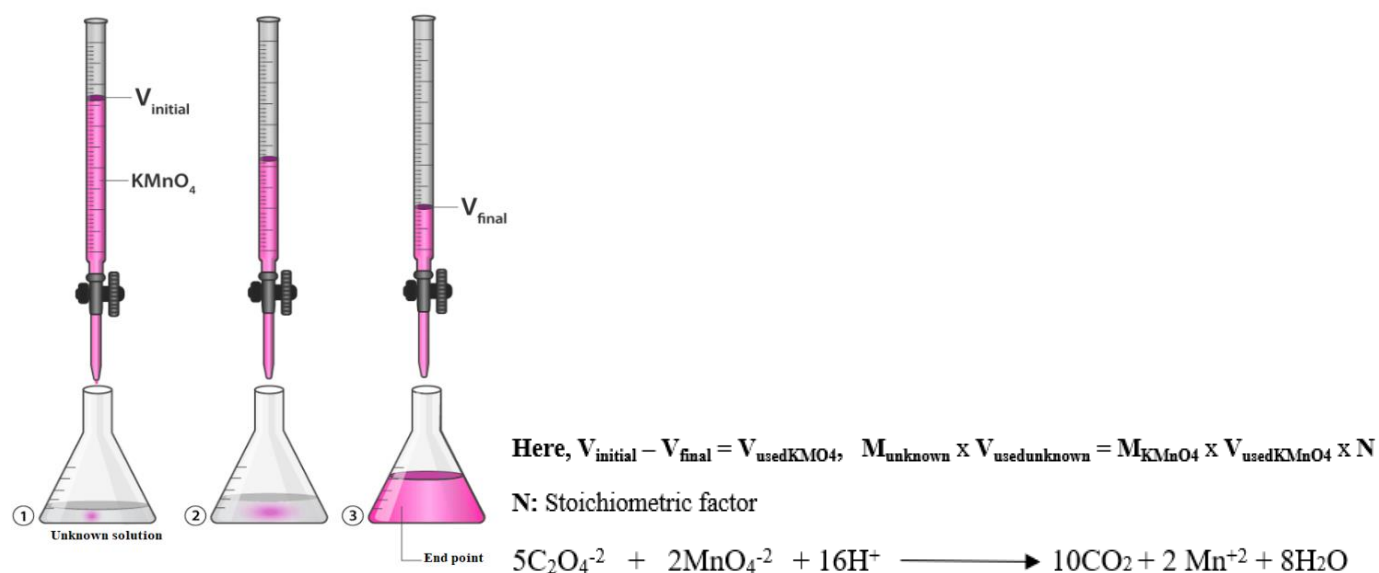
Figure 4.2 Calculation the mass and percentage of $K_2C_2O_4$ in the unknown sample

Disposal:

Titration solutions: Dispose in a waste bottle labeled heavy metals-solutions

Unused KMnO₄: Dispose in a waste bottle labeled waste KMnO$_4$. It will be treated further with reducing agent and safely disposed.

Unused solids: Dispose K$_2$C$_2$O$_4$ $_{(s)}$ and K$_2$C$_2$O$_4$/Na$_2$CO$_3$ in a waste bottle labeled waste solids-inorganic.

INORGANIC WASTE

ORGANIC WASTE

NORMAL WASTE

Name _____ **Department** _____ **Group** _____

DATA&RESULTS

A.   Standardization of Solution

	Trial 1	Trial 2	Trial 3
Mass of $K_2C_2O_4$, g	——————	——————	——————
Volume of $KMnO_4$ solution, ml	——————	——————	——————
Molarity of $KMnO_4$ solution, (mol/L)	——————	——————	——————
Average Molarity, (mol/L)		——————	
Relative Average Deviation		——————	

B.   Analysis of Unknown Oxalate

	Trial 1	Trial 2	Trial 3
Mass of unknown, g	——————	——————	——————
Volume of $KMnO_4$ solution, ml	——————	——————	——————
Mass of $K_2C_2O_4$ in unknown, (g)	——————	——————	——————
Percent of $K_2C_2O_4$ in unknown	——————	——————	——————
Average Percent of $K_2C_2O_4$ in unknown		——————	
Relative Average Deviation		——————	

Name_____Department_____Group_____

CALCULATION 54

A. Standardization of Solution

Molarity of $KMnO_4$ solution, (mol/L)

Average Molarity, (mol/L)

Relative Average Deviation in M

B. Analysis of Unknown Oxalate

Mass of $K_2C_2O_4$ in unknown, (g)

Percent of $K_2C_2O_4$ in unknown

Average Percent of $K_2C_2O_4$ in unknown

Relative Average Deviation

Name_____**Department**_____**Group**_____

REPORT QUESTIONS

1. If the accuracy in this experiment were equal to the experimental precision (% relative average deviation), within what range of values about your average unknown result would be the " true" value lie?

2. a) What is meant by the term autocatalysis?

  b) Give an example of a reaction that is autocatalyzed.

3. Compare the percent relative average deviation about the average molarity in part A to that about the average percent sodium oxalate in part B. Are they similar in magnitude? If not, which is larger? Can you think of a plausible explanation?

4. How would the calculated percent oxalate of the unknown be affected (too large, too small, no effect) by the following analytical errors. Explain your answer.

a) The oxalate used for standardization was dried to remove moisture prior to weighing.

b) The buret was not clean, and the titrant was left on the walls above the observed liquid level.

c) Some unknown sample is spilled and lost after weighing but before titration.

Name_____Department_____Group_____

PRELAB QUESTIONS

1. Write the balanced net ionic equation for the reaction that occurs in the titration of this experiment.

2. In the titration reaction, which substance is

   b) Reduced?   _____

   c) Oxidized?   _____

3. What is the cause of the color change at the end point of this titration?

4. 0.725g of solid unknown requires 25.52 mL of 0.0250 M $KMnO_4$ titrant to reach the end point. Calculate the percent sodium oxalate in the unknown sample.

5. When using the chemicals in this experiment, what precautions should you take, and what should you do before leaving laboratory

5 Quantitative Analysis Based on Gas Properties

INTRODUCTION

The aim of this study is to determine the percentage of $KClO_3$ in a mixture by one of the most important quantitative relationships involving gases- a relationship which depends on the following two facts;

1. One mole of any substance contains the same number of molecules as 1 mole of any other substance. This is implicit from the definition of a mole.

2. Equal volumes of all gases, under identical conditions of temperature and pressure, contain equal number of molecules. This follows from "the kinetic theory of gases," and was first stated about 1811 by *Avogadro*, an Italian chemist.

Under identical conditions of temperature and pressure, 1 mole of all gases occupies equal volumes- this is called *the molar volume of a gas*.

The Molar Volume at Standard Conditions: The Perfect Gas

To evaluate the molar volume in metric units, recall that the symbol O stands for 1 gram-atom, namely 15.9994 g of oxygen and that 1 mole of oxygen gas, O_2, therefore weighs 31.9988 g. In this experiment we shall measure the weight and corresponding volume of a sample of oxygen gas and from this data calculate the volume at standard conditions: then we shall calculate the volume of 32.0 g (1 mole) of oxygen gas.

All real gases deviate more or less from the ideal behavior described above because their molecules do have some slight attraction for one another and do occupy some slight volume themselves. A *perfect gas* is an idealized gas in which the molecules would have no attractive forces and would be mere "points" without significant sizes. The standard molar volume for a perfect gas has been calculated from measurements on real gases at very low pressures to be 22.4136 liters. Most common gases, unless they have a high molecular weight or are measured quite near their boiling point, have molar volumes which do not deviate more than 1 % from this volume.

Calculation of Gas Volumes

The quantity of a gas sample can be measured more easily by volume than by weight. In measuring the volume of a gas, it is also necessary to measure its temperature and pressure.

$$P\ V = k_1 \text{ and } \qquad P\ V = k_2 \qquad \text{or} \qquad V = k_3 T$$

For a given gas sample, these two laws may be combined into one equation, which shows the way in which all three variables - pressure, volume, and absolute temperature- are dependent to one other:

$$PV = kT \qquad \text{or} \qquad \frac{PV}{T} = k \qquad\qquad\qquad (1)$$

Since any two corresponding sets of PV / T measurements will be equal to "k" and to each other, we may write:

$$\frac{P_1 V_1}{T_1} = \frac{P_2 V_2}{T_2} \qquad\qquad\qquad (2)$$

This may be transposed to give

$$V_1 = V_2\, x\, \frac{T_1}{T_2}\, x\, \frac{P_2}{P_1} \qquad\qquad\qquad (3)$$

Note in equations (2) and (3) that if the temperature is constant ($T_1 = T_2$) the inverse proportionality of pressure and volume (Boyle's law) is expressed. Likewise, for constant pressures ($P_1 = P_2$) the direct proportionality of volume and absolute temperature (Charles' law) is expressed. If five of the quantities in equation (2) are known, the sixth can of course be calculated by simple algebraic means.

Many students and teachers prefer to reason out the pressure-volume-temperature relationships rather than blindly to "follow a formula" and to apply corrective factors for pressure (and for temperature) according to whether the pressure change (and the temperature change) will cause an increase (or a decrease) in the volume. Either approach yields the same result.

The General Gas Law Equation

The specific amounts of the same gas or for any different gases, equation (1) may be restated in its most general form:

$$PV = nRT$$

In this *general gas law*, the gas constant R (0.0821 atm.L.mol^{-1}.K^{-1}) has the same value for all gases. In all calculations using this constant, pressure must be expressed in atm, volume in liters, and temperature in Kelvin.

As an example of the application of general gas law where both volume and weight of a gas sample are involved, consider the following. What weight of chlorine gas, Cl_2, would be contained in a 5.00 - liter flask at 20°C and at 600 mmHg pressure? Substituting in equation (4), transposed to give, n, the number of moles, we have;

$$PV = nRT, \quad \text{or} \quad n = \frac{PV}{RT}$$

$$n = \frac{600/760 atm \times 5.00 liters}{0.0821 \frac{liter.atm}{mol.K} \times 293\ K} = 0.164\ mole$$

and, in grams.

$$0.164 mole \times 70.9\ \frac{g}{mole}\ Cl_2 = 11.6\ g$$

Aqueous Vapor Pressure: Dalton's Law of Partial Pressures

When any gas in a closed container is collected over, or exposed to, liquid water, the water evaporates until the opposing rates of evaporation and condensation of water molecules at the liquid surface reach a "balance". These gaseous water molecules contribute their due share to the total gas pressure against the walls of the container. Thus, of all the gas molecules, if 3% are water molecules and 97% are oxygen molecules, then 3% of the total pressure is due to water vapor and 97% of the total pressure is due to oxygen. *Each gas exerts its own pressure regardless of the presence of other gases*. This is Dalton's Law of Partial Pressures. Stated as an equation;

$$P_{total} = P_{H_2O} + P_{O_2}$$

or, transposing
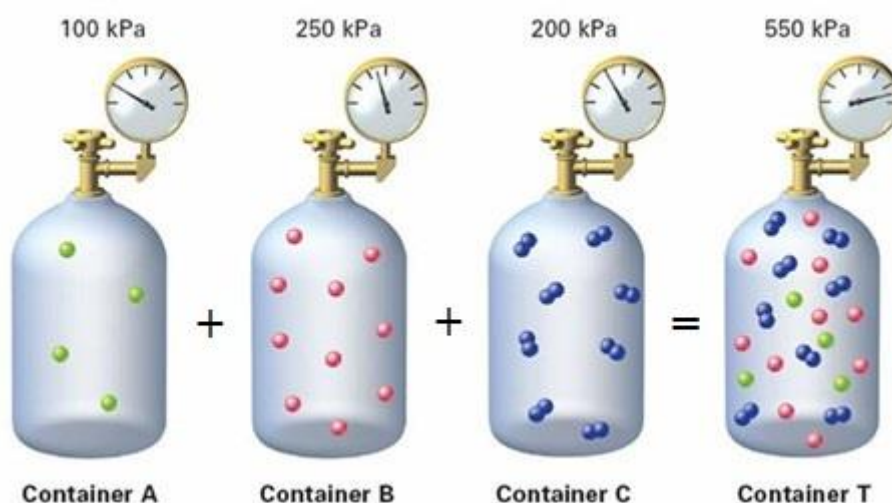
$$P_{O_2} = P_{total} - P_{H_2O_2}$$

Figure 1. The application of Dalton's law of partial pressure, when three gasses mix together in container T, pressure of the mixture equals to the sum of partial pressures of each gas.

For any gas which behaves ideally, Avogadro's Law tells us that the volume is proportional only to the number of moles of the gas, at a constant temperature and pressure. So, all gases have the same molar volume under given conditions. For example, we know that the molar volume of a gas at STP is 22.4 L/mol. This is an experimentally derived number. We can use this number to calculate the constant of proportionality, R, assuming ideal gas behavior.

Temperature (°C)	Pressure (mmHg)	Temperature (°C)	Pressure (mmHg)	Temperature (°C)	Pressure (mmHg)
0.0	4.6	19.5	17.0	27.0	26.7
5.0	6.5	20.0	17.5	28.0	28.3
10.0	9.2	20.5	18.1	29.0	30.0
12.5	10.9	21.0	18.6	30.0	31.8
15.0	12.8	21.5	19.2	35.0	42.2
15.5	13.2	22.0	19.8	40.0	55.3
16.0	13.6	22.5	20.4	50.0	92.5
16.5	14.1	23.0	21.1	60.0	149.4
17.0	14.5	23.5	21.7	70.0	233.7
17.5	15.0	24.0	22.4	80.0	355.1
18.0	15.5	24.5	23.1	90.0	525.8
18.5	16.0	25.0	23.8	95.0	633.9
19.0	16.5	26.0	25.2	100.0	760.0

Table 1. Water vapor pressures in mmHg corresponding to temperature in °C.

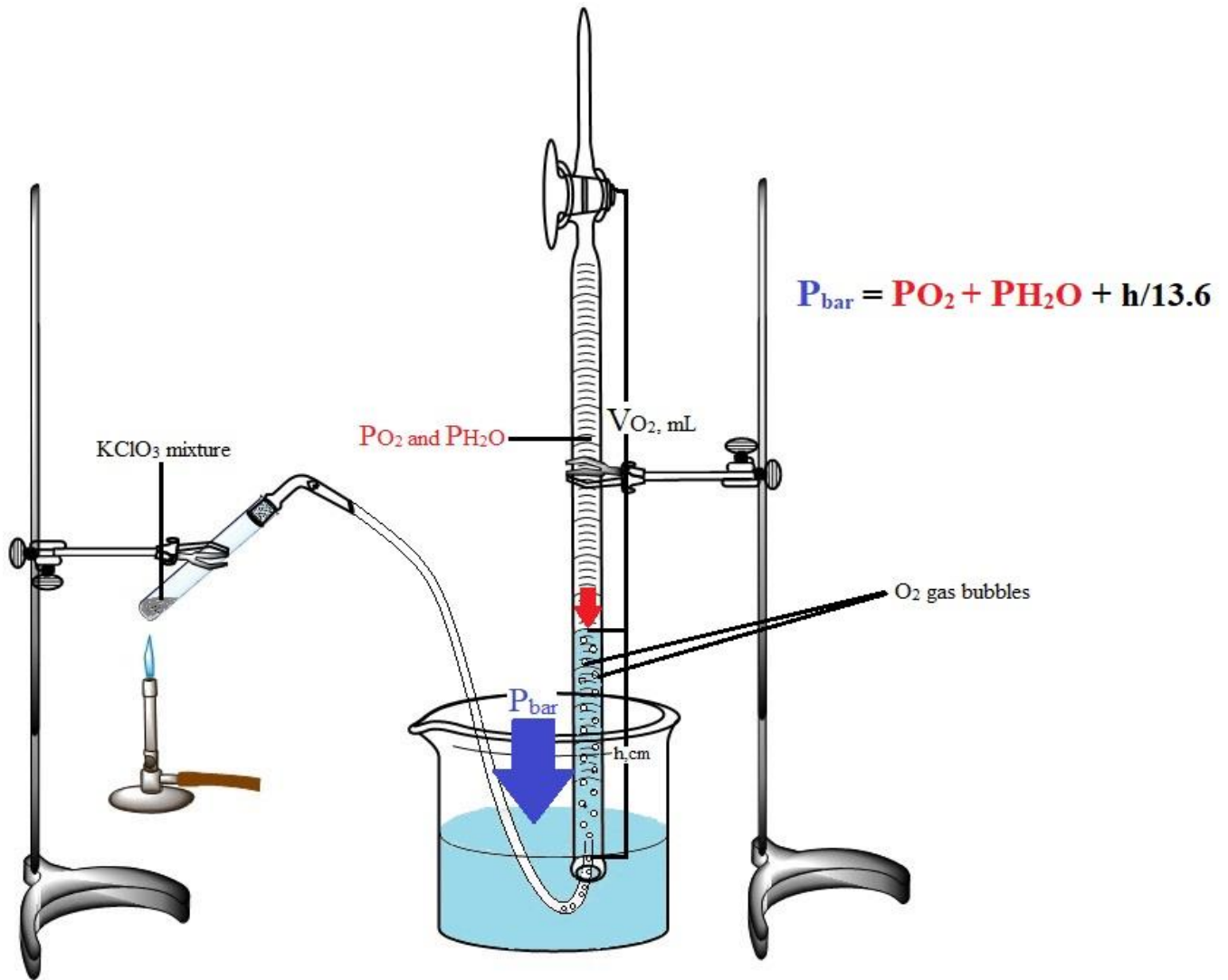## PROCEDURE

Required Chemicals	Required Lab. Materials
$KClO_3$	Burette (or graduated cylinder)
KCl	Clamp
MnO_2	Erlenmeyer flask
Water	Test tube
	Bunsen burner (or a strong lighter)
	Stopper
	Rubber tube

In this experiment O_2 gas will be generated from decomposition of potassium chlorate.

$$2\ KClO_{3(s)} \rightarrow 2\ KCl_{(s)} + 3\ O_{2(g)}$$
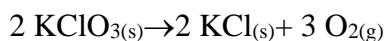
The gas will be collected by displacement of water; so, the volume of gas is determined by how much water is displaced. The pressure can be determined by correcting for the pressure of the water in the flask, and the temperature is essentially room temperature. Thus, the mole of O_2 generated can be calculated by ideal gas law. Mole of $KClO_3$ can be calculated by using stoichiometric ratio in decomposition reaction. Finally, mass and percentage of $KClO_3$ can be determined.

1. Obtain an appropriate amount of unknown mixture containing $KClO_3$, KCl and MnO_2 from your instructor. Record this amount. (MnO_2 acts as a catalyst for the reaction. It is NOT consumed in the reaction, and therefore does not appear in the equation.)

2. Add the $KClO_3$ mixture (~ 0.1 g) to the test tube and save the weight data. (mixture will be prepared by lab assistant and content ratio will be unknown by students)

3. Set up the experiment as shown in the figure 2. (A graduated cylinder or burette will be used)

$$P_{bar} = PO_2 + PH_2O + h/13.6$$

KClO$_3$ mixture

PO$_2$ and PH$_2$O

V$_{O_2}$, mL

O$_2$ gas bubbles

P$_{bar}$

h, cm

4. Heat the tube gently at first since oxygen is driven off quickly as the decomposition of the potassium chlorate begins. Move the burner around to achieve uniform heating. Only heat the bottom of the tube. DO NOT HEAT near the stopper. Ensure that the mixture is ONLY at the bottom of the test tube.
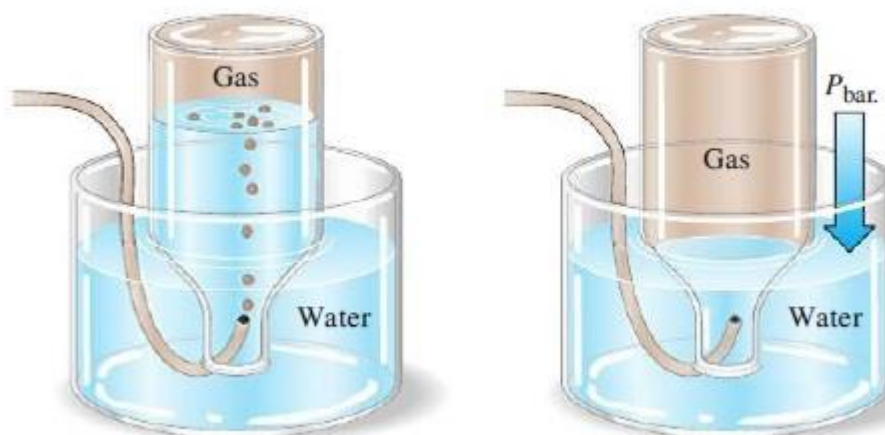
5. As O_2 gas generated by reaction, water will be displaced by accumulation of gas in burette. As the gas flow finishes record the volume (mL) of gas which was accumulated in burette and the height (h=mm) of the water in burette. (d_{Hg}=13,6 g/ml, d_{H2O}=1 g/ml)

6. Obtain appropriate water vapor pressure value from the table 1.

7. Obtain the atmospheric pressure from the barometer.

8. Place all used test tubes, including their contents, in the designated solid waste container in the laboratory. $KClO_3$ must NOT be disposed off in the trash since it can react with combustibles.

Name_____**Department**_____**Group**_____

RESULTS & DATA

Weight of empty tube, g   _____

Weight of tube and contents before heating, g   _____

Barometer reading, atm   _____

Volume of O_2 collected in burette, ml   _____

Temperature, K   _____

Aqueous vapor pressure of water at temperature of gas, atm   _____

Height of the water in burette, mm   _____

Molecular weight of $KClO_3$ 122.55 g/mol

Name **Department** **Group**

	CALCULATIONS	RESULT
Pressure of oxygen alone in the flask (atm)		
Moles of your oxygen (mol)		
Volume of your oxygen at STP (L)		
The molar volume of oxygen (L)		
Percentage error		
Moles of $KClO_3$ in sample (mol)		
Weight of $KClO_3$ in sample (g)		
Percent of $KClO_3$ in sample (%)		

**Name**_____**Department**_____**Group**_____

PRELAB QUESTIONS

1- a) What is the purpose of using MnO_2 in this experiment?

b) Why MnO_2 doesn't affect the stoichiometry of the reaction in the experiment?

2- a) Calculate the ideal gas constant, R, if the molar volume of a gas at STP is 22.4 L/mol. Remember to express your units as L atm/mol K.

b) Calculate R in units of mL torr/mol K.

3- The decomposition of potassium chlorate, $KClO_3$, produces oxygen gas O_2, and a solid product. The solid residue could be potassium hypochlorite, potassium chlorite or potassium chloride.

Solid potassium chlorate $\longrightarrow$ oxygen gas + solid residue
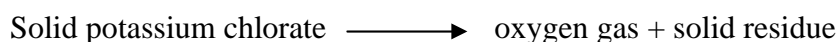
a) Write a balanced equation for the decomposition assuming that the solid residue is potassium hypochlorite.

b) Write a balanced equation for the decomposition assuming that the solid residue is potassium chlorite.

c) Write a balanced equation for the decomposition assuming that the solid residue is potassium chloride.

6 Thermochemistry: The Heat of Reaction

INTRODUCTION

Chemical thermodynamics deals with the energy changes that accompany chemical reactions. Such energy changes contribute to determining the following.

1. How fast a chemical reaction takes place, that is to say, the problem of chemical kinetics.

2. How complete the reaction will be, that is the position of chemical equilibrium.

Thermochemistry concerns energy changes manifested as the enthalpy change of reaction, ΔH. ΔH is the heat given off by the reaction at constant pressure. A reaction in which heat is lost by the reactants to the surroundings has a negative ΔH and is said to be exothermic; one in which heat is absorbed has a positive ΔH and is endothermic.

The general term, enthalpy of reaction, may be classified into more specific categories:

1. The enthalpy of formation is the quantity of heat involved in the formation of 1 mole of the substance in its standard state directly from its constituent elements in their standard states.

2. The enthalpy of combustion is the quantity of heat evolved per mole of a combustible substance, such as carbon or methane, undergoing a reaction with excess oxygen.

3. The enthalpies of solution, vaporization, fusion, and sublimation are concerned with changes in state or solvation of molecules or ions.

4. The enthalpy of neutralization is the heat evolved when 1 mole of water is produced by the reaction of an acid and base.

In this experiment, we shall measure the enthalpies of neutralization of HCl and NaOH solution, the enthalpy of solution of $NaOH_{(s)}$.

Heat measurements are performed by carrying out the reaction in a calorimeter (Figure 6.1), in which the temperature change and mass of solution are measured. The heat of the reaction, q_{rxn}, is the negative of the heat gained by the solution, q_{soln}, plus that gained by the calorimeter, q_{cal}, i.e. $q_{rxn} = -(q_{soln} + q_{cal})$.

q_{soln} is determined from the formula $q_{soln} = mC\Delta t$, where m and Δt are the mass and temperature change, respectively, of the solution.

The specific heat, *C*, is the quantity of heat required to raise the temperature of one gram of a material (in this case, that material is the solution) by one degree Celcius (J g^{-1} °C^{-1}). The specific heat is given for the different parts of this experiment. The product of the units for *m*, *C*, *Δt* yields the unit for heat, the joule, and is the heat of solution, *q$_{soln}$*. After correction is made for the heat absorbed by the calorimeter (Part A of the procedure), *ΔH* is determined by dividing the negative of the corrected *q* by the number of moles of reactant or product (in this experiment, the number of moles of water formed in Part B equals the number of moles of each reactant).

Figure 6.1. Simple Calorimeter for Measuring Heat of Reaction

Determination of Temperature

Three factors make it difficult to determine temperatures quickly in this experiment.

1. The calorimeter is not a perfect insulator and heat "leaks" out of it (causing the temperature to drop by about 0.1 °C/min).

2. It takes at least 1 min for the calorimeter to reach the same temperature as the mixture within it.

3. In Part C it takes about 3 min for the NaOH$_{(s)}$ to dissolve.

All three effects are evident in this set of student data from Part B of the experiment.

Time, min	1	2	3	4	5	6	7	8	9	10	11	12
Temp, °C	16.5	16.6	16.7	mix	22.0	24.0	26.4	28.5	28.0	28.0	27.8	27.7

These data are graphed in Figure 6-2. Notice at times 1, 2, and 3 that the temperature is slowly rising because the temperature of the distilled water is cooler than that of the laboratory. To determine the temperature of the distilled water at the time of mixing, one extrapolates these points forward in time (draws the best straight line through them) and obtains 16.8°C at the time of mixing. From time 5 to time 8 the NaOH dissolves in distilled

water and the temperature rises rapidly to 28.5°C. It then drops back quickly to 28.0°C, probably because it took this last time to heat the calorimeter. Now the temperature begins to fall slowly and regularly (about 0.1°C/min) and one can extrapolate through these data backward in time to find out what the temperature at the time of mixing would have been if the NaOH had dissolved instantaneously and the calorimeter had warmed instantaneously. Observe carefully in Figure 6.2 that data are collected for 8 min after mixing in order to obtain good results. Do not stop collecting data too soon. In fact, it is good idea to plot your data as you collect it. You should make one graph like the one in Figure 6.2 for each set of data you collect.
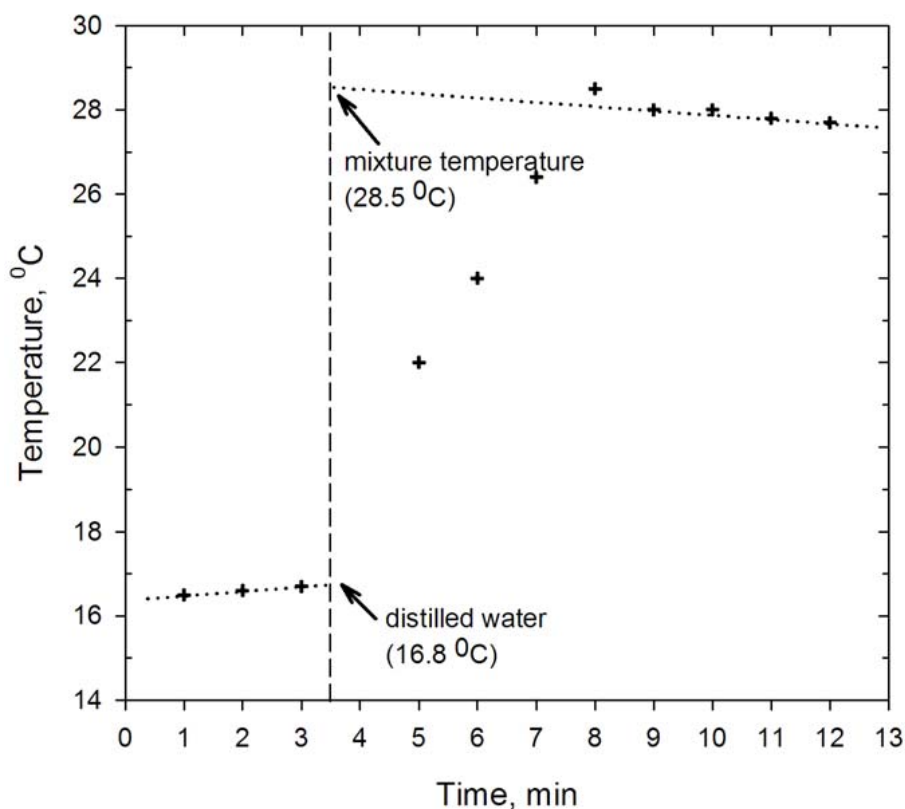
Figure 6.2. Temperature-Time Plot

SAFETY PRECAUTIONS

Be particularly careful when handling the sensitive thermometers used for this experiment. The breaking of a thermometer can occur easily and is a serious problem as mercury can be released into the environment. Mercury spills are one of the safety hazards that chemists fear the most. In addition, a broken thermometer presents the hazard of broken glass.

If you spill any of the solutions (acids or bases) used in this experiment on yourself or your work area, wash thoroughly with running water and tell your instructor immediately.

PROCEDURE

A. The Water Equivalent of the Calorimeter

Prepare two calorimeters, each similar to one illustrated in Figure 6-2, as directed. Compare your two thermometers by immersing them together in water at room temperature for 1 min, and reading the temperature as nearly as possible to nearest 0.1 °C. Be careful to avoid parallax in your readings. Always use the same calorimeter in which the temperature change occurs, and in all subsequent readings apply any necessary correction to the other, so that the readings of both thermometers will correspond.

To correct for the heat lost to the calorimeter, carry out measurement in a manner similar to those to be used later. Place 50.0 mL of distilled water at room temperature in one calorimeter, and 50.0 mL of distilled water that has been heated to 15-20 °C above room temperature in the other. With the lids and thermometers in place, make careful readings (±0.1°C) for each at 1-min intervals for 3 min. At the minute mark, pour the warmer water quickly and as completely as possible in to the calorimeter, and continue the readings for the next 4 min. The temperature of separate and of the mixture can be extrapolated back to the time of mixing by making a graph of each, with temperature as the ordinate and the time as the abscissa (or, where temperature changes only slight, simply by numerical extrapolation of the data.)

Calculate the heat lost by warmer water, and the heat gained by the cooler water. (Water mass x Δt (°C) x specific heat. Assume density of water = 1.0 g/mL, and the specific heat = 4.18 $Jg^{-1}°C^{-1}$). The difference, representing the heat gained by the calorimeter, divided by the corresponding Δt (°C) gives the water equivalent of the calorimeter in J/°C. Repeat this determination as a check, and average your results.

An example will illustrate how the water equivalent may be determined, and later used in calculations.

Example (a) Determination of water equivalent (WE) of the calorimeter.

Temperature of 50 mL of warm water	37.9 °C
Temperature of 50 mL of cold water	20.9 °C
Temperature after mixing	29.1 °C

Heat lost by warm water	(50 g x 8.8 °C x 4.18 Jg-1°C-1) = 1841 J
Heat gained by cold water	(50 g x 8.2 °C x 4.18 Jg-1°C-1) = 1715 J
Heat lost to calorimeter	1841 J - 1715 J = 126 J

Since:

Heat lost by warm water = heat gained by cold water + heat gained by calorimeter and

Heat gained by calorimeter = WE x Δt (°C) with Δt = 29.1 °C – 20.9 °C = 8.2 °C

The water equivalent of this calorimeter is:

WE = 126 J/8.2 °C = 15 J/°C

(b) Use of this water equivalent in the calculation of the heat of the reaction. During a reaction in this calorimeter, 100 g of solution increases in temperature by 6.5 °C. (Specific heat of solution = 4.00 $Jg^{-1}°C^{-1}$)

Heat gained by the solution

(100 g x 6.5 °C x 4.00 $Jg^{-1}°C^{-1}$)   = 2600 J

Heat gained by the calorimeter

(15 J/°C x 6.5 °C)   = 100 J

Total heat gained by solution and calorimeter   = 2700 J

Heat of the reaction   = -2700 J

B. The Enthalpy of Neutralization of HCl(aq) and NaOH(aq)

Place 50.0 mL of 1.0 M HCl in one calorimeter and 50.0 mL of 1.0 M NaOH in the other calorimeter. With the lids and thermometers in place, read the temperatures (±0.1°C) for 3 min at 1-min intervals; quickly mix the NaOH thoroughly into the HCl solution, and continue the readings for 4 min at 1-min intervals. Extrapolate the temperatures to the time of mixing for each solution as in Part A, and calculate the enthalpy of neutralization per mole of water produced. (The density of the 0.5 M NaCl produced is 1.02 g/mL, and its specific heat is 4.00 J g^{-1} $°C^{-1}$.)

Disposal:

Solutions: Add phenolphthalein and neutralize with 1 M HCl (the first disappearance of pink color) or NaOH (first appearance of permanent pink color). Flush the resulting salt solution down the sink with running water.

C. The Enthalpy of Solution of NaOH (s)

Carefully weigh (±0.01 g) about 2.00 g (0.05 mol) of $NaOH_{(s)}$. (Because of its hygroscopic nature, weigh this by difference, in a stoppered 50 or 125 mL Erlenmeyer flask used as a weighing bottle. Your instructor will tell you the approximate number of $NaOH_{(s)}$ pellets required to assist in estimating the mass needed. Be sure to clean up any spilled $NaOH_{(s)}$. This solid absorbs water from the air and forms a slippery solution that is also corrosive.)

Place 50.0 mL of distilled water in your calorimeter. With the lid and thermometer in place, read temperature (0.1 °C) for 3 min at 1-min intervals; then add the $NaOH_{(s)}$, replace the lid and thermometer, and gently swirl the mixture and stir it carefully with the thermometer to dissolve the NaOH as quickly as possible. At the same time continue the temperature readings at 1-min intervals for at least 9 min total (at least three readings after the maximum temperature reading is attained). Because of the time required for solution and complete mixing, the proper estimate of the temperature for complete solution at the time of mixing is more difficult. Make your best estimate based on extrapolations of temperature before and after mixing as explained in the "Determination of Temperature".

A plot of your data is essential. Calculate the heat of solution per mole $NaOH_{(s)}$ to form a 1.0 M NaOH solution. (Note that you have about 52 g of solution. The specific heat of 1.0 M NaOH solution is 3.90 Jg^{-1} $°C^{-1}$.)

Disposal:

NaOH solution: Add phenolphthalein and neutralize with 6 M H_2SO_4 or HCl (the first disappearance of pink color). Flush the resulting salt solution down the sink with running water.

Name_____Department_____Group_____

DATA & RESULTS

A. The Water Equivalent of the Calorimeter

50.0 mL of water placed in each calorimeter; temperature readings for three 1-min intervals for separate calorimeters, mix on 4th minute, continue readings for 4 min.

Trial 1
Calorimeters

	Warm	Cold
1.	_____	_____
2.	_____	_____
3.	_____	_____
4.	mix	
5.	_____	
6.	_____	
7.	_____	
8.	_____	

B. The Enthalpy of Neutralization of HCl (aq) and NaOH (aq)

50.0 mL each of 1.0 M HCl and 1.0 M NaOH, at room temperature, placed in respective calorimeters; temperature readings as follows:,

Trial 1
Calorimeters

	HCl	NaOH
1.	_____	_____
2.	_____	_____
3.	_____	_____
4.	mix	
5.	_____	
6.	_____	
7.	_____	
8.	_____	

Name_____**Department**_____**Group**_____

C. The Enthalpy of Solution of NaOH (s)

(Record the mass of NaOH (s) in the calculation part.) Temperature readings for 50.0 mL of water placed in a calorimeter, before and after adding NaOH (s), as follows:

Trial 1
Calorimeters
Water

1. _____

2. _____

3. _____

4.   mix

5. _____

6. _____

7. _____

8. _____

9. _____

10. _____

11. _____

12. _____

Name_____**Department**_____**Group**____

CALCULATIONS

Part A) Determination of Water Equivalent (Heat Capacity) of a Calorimeter	Calculations	Results
Density of water (g/mL)		
Mass of cold water (g)		
Mass of warm water (g)		
Mass of total mixture (g)		
Specific heat of the water (J/g°C)		
Temperature of cold water (°C)	from graph by extrapolation	
Temperature of warm water (°C)	from graph by extrapolation	
Temperature of mixture (°C)	from graph by extrapolation	
Temperature change (°C)		
Heat lost by warm water (J)		
Heat gained by cold water (J)		
Heat lost to calorimeter (J)		
Water equivalent (heat capacity) of calorimeter (J/°C)		

Name_____Department_____Group_____

Part B) The Enthalpy of Neutralization of HCl (aq) and NaOH (aq)	Calculations	Results
Density of NaCl solution (g/mL)		
Mass of NaCl solution (g)		
Moles of NaCl (mol)		
Specific heat of NaCl solution (J/g°C)		
Temperature of HCl (aq) (°C)	from graph by extrapolation	
Temperature of NaOH (aq) (°C)	from graph by extrapolation	
Avg.Temperature of mixture ((°C)	from graph by extrapolation	
Temperature change (°C)		
Heat gained by mixed solutions* (J)		
Heat gained by calorimeter (J)		
Total heat gained (J)		
Total heat of the reaction (J) (negative of total heat gained)		
Enthalpy of neutralization (J/mol)		
Is this reaction exothermic or endothermic?		

* For temperature gain, Δt°C, use the temperature of the mixture minus the average temperature of HCl and NaOH. The density of 0.5 M NaCl produced is 1.02 g/mL and its specific heat is 4.00 J/g°C.

Name_____Department_____Group____

Part C) The Enthalpy of Solution of NaOH (s)	Calculations	Results
Mass of NaOH (g)		
Mass of water (g)		
Mass of solution (g)		
Moles of NaOH (mol)		
Specific heat of NaOH solution* (J/g°C)		
Temperature of water (°C)	from graph by extrapolation	
Temperature of mixture (°C)	from graph by extrapolation	
Temperature change (°C)		
Heat gained by solution (J)		
Heat gained by calorimeter (J)		
Total heat gained (J)		
Total heat of the reaction (J) (negative of total heat gained)		
Enthalpy of solution (J/mol)		
Is this reaction exothermic or endothermic?		

* Specific heat of NaOH solution is about 3.90 J/g°C.

Name_____**Department**_____**Group**_____

PRELAB QUESTIONS

1. Describe how the calorimeter is made in this experiment.

2. What is meant by extrapolation?

3. Throughout this experiment, the quantities of chemicals used are approximately the same. How many moles are used in each case?

4. Why is it necessary to measure the water equivalent of the calorimeter in this experiment? How is this value used?

5. What are the two reactions whose enthalpies are used in this experiment? Write the balanced equations for both reactions.

7 Group I: The Soluble Group

INTRODUCTION

Na^+, K^+ and ammonium ion NH_4^+ are the ions included in Group I. Sodium and potassium ions are representative of the ions of the very active alkali metals located in group IA of the periodic table. The salts of these metals are nearly all readily soluble in water. Although ammonium ion is not an alkali metal ion, its salts likewise are nearly all soluble. In fact, NH_4^+ is often compared to K^+ in that many of its properties are quite like those of K^+.

The identification tests for NH_4^+, Na^+ and K^+ must be carried out on separate portions of unknown solution. These ions are unique in this respect. No separation tests are necessary. Their tests are simple and convenient.

$$Na^+_{(aq)} \xrightarrow{\Delta} Na^+_{(g)} \tag{1}$$

$$Na^+_{(g)} + 1e^- \xrightarrow{\Delta} Na_{(g)} \tag{2}$$

$$Na_{(g)} \xrightarrow{\Delta} Na^*_{(g)} \tag{3}$$

$$Na^*_{(g)} \rightarrow Na_{(g)} + h\nu \text{ (589 nm; yellow color)} \tag{4}$$
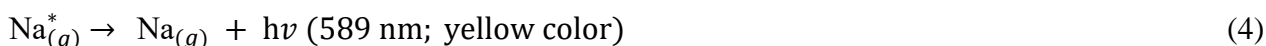
A distinctive feature of the Group 1A metals is their ability to produce colors in the flame. For example, when sodium ion containing solution is introduced in a flame, water in the solution is vaporized and gaseous sodium ion is produced (Eqn. 1). Sequentially gaseous sodium ion is converted to gaseous atom (Eqn. 2). The heat in the flame further excites the ground state sodium (3s1 orbital) atom to exited state (Na*) (3p orbital) (Eqn. 3). Since the ground state is more stable than excited state atom reverts to its ground state electron configuration (Eqn. 4) by emitting characteristics yellow color which corresponds to energy difference between two states. Similar reactions are also occurring in case of K+ ion with its characteristic emission color. Table 1 summarizes the wavelength region corresponding to various colors.

Table 1. The visible spectrum

Color	Wavelength region(nm)
Red	650-750
Orange	595-650
Yellow	580-595
Green	500-560
Blue	435-480
Violet	400-435

PROCEDURE FOR THE ANALYSIS OF AN UNKNOWN SOLUTION FOR NH_4^+, K^+ AND Na^+
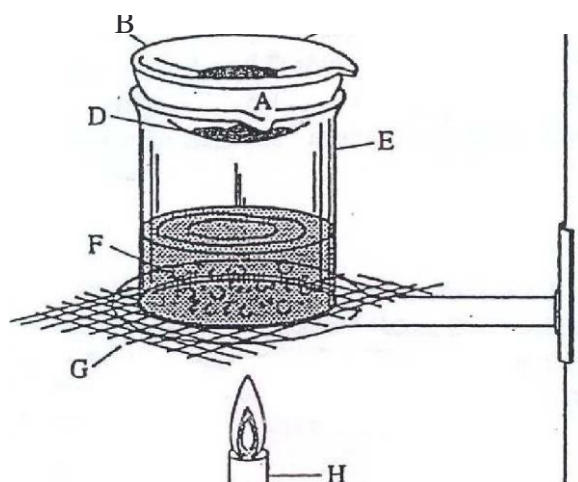
Test for Ammonium Ion

Figure 1: The Test for Ammonium Ion: *A* is red litmus paper moistened and placed on the underside of the watch glass, *B*. *C* is an evaporating dish containing the sample and some sodium hydroxide solution, *D*. *E* is a beaker, *F* is hot water, *G* is a wire screen and *H* is a burner.

$$NH_4^+{}_{(aq)} + OH^-{}_{(aq)} \leftrightharpoons NH_3{}_{(g)} + H_2O \tag{5}$$

Place 3 mL of the solution to be tested in an evaporating dish and add 1 - 2 mL of 6 M NaOH. Quickly cover the dish with a watch glass, on the underside of which is attached a moist strip of red litmus paper. Warm the solution very gently (over hot water, Figure 1, not with a burner flame or a hot plate) to liberate any ammonia present as the gas. (Avoid boiling, which would contaminate the litmus with spray droplets of the NaOH solution.) An even, unspotted blue color indicates the presence of ammonium ion according to reaction described in Eqn. 5. The characteristic odor of ammonia observed soon after the solution is warmed, also serves as a positive test. (Be very cautious in bringing your nostrils close to a hot sodium hydroxide solution, however,

as it might be superheated, and spatter in your face or eyes.)

Test for Sodium and Potassium Ions

Clean a wire of nickel - chromium alloy as shown in Figure 2 until no color is observed when the wire is held in the flame of the bunsen burner. Dip the clean wire in the solution to be tested, and then hold it in the nonluminous flame of the burner. A luminous fluffy yellow - orange coloration in the flame which persist for 30 sec or more indicates the presence of Na+, while K+ ion has violet color in the flame. Care must be taken since this test is extremely sensitive. Traces of sodium ion get into solutions as a result of contacting glassware; similarly contact of the wire with one's fingers will cause some salt to be transferred to the wire; further, nearly all reagents contain a trace of sodium ion. The result is that most solutions give what might be a weakly positive test for Na^+. A comparison with a solution known to contain sodium ion as well as with a solution containing trace amount of Na^+ is very helpful in making decision whether sodium ion is present in the unknown solution.

Look at the flame against a dark background.

Observe the color of the flame when the wire is heated in the edge of the Bunsen flame.

Clean the wire by alternatively dipping in concentrated HCl and heating to incandescence. A colored flame indicates a contamined wire.

Dip the clean wire into the substance which has been moistened with distilled water.

(a)　　　　　　　　　　　　　　　　　　(b)

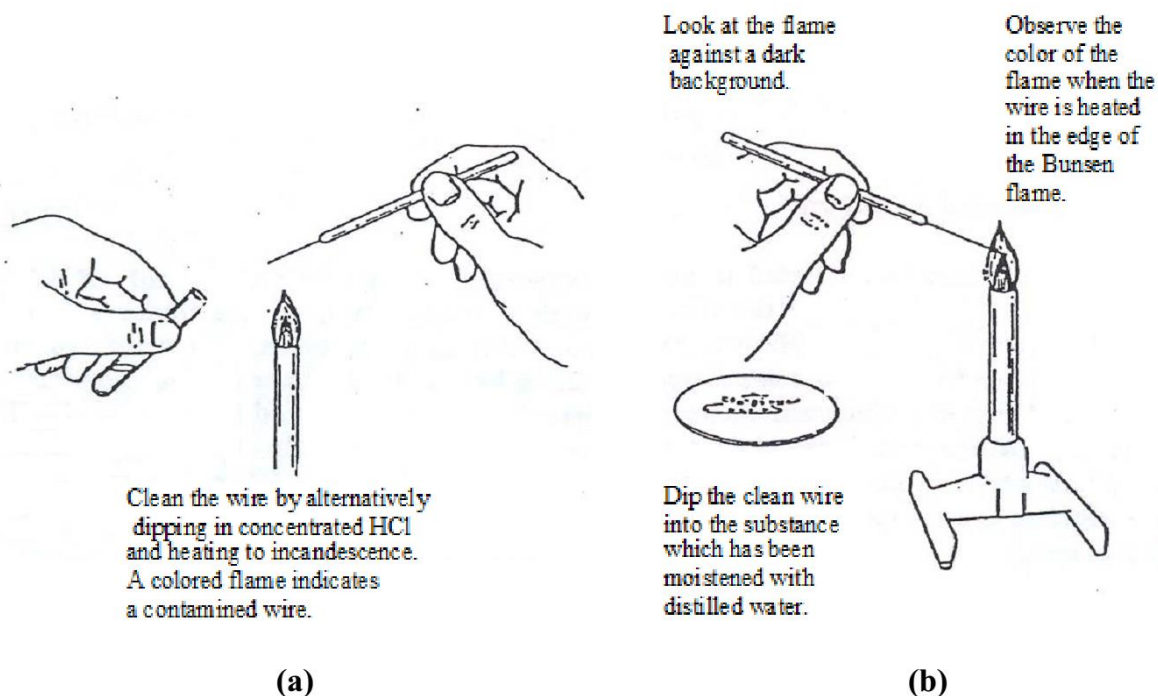**Figure 2.** The Flame Test for Na^+ and K^+ Ions: (a) Cleaning the Wire and (b) Observing the Flame Colorations

Disposal: Aqueous solutions: Dispose in a common waste bottle labeled for *qualitative analysis solutions*

Name _____**Department** _____ **Group____**

DATA&RESULTS

	Flame Test Observations	NH_4^+ Test Observations
NH_4^+		
Na^+		
K^+		
Unknown		

Name_____ **Department** _____**Group**_____

PRELAB QUESTIONS

1. Why are Na+ and NH₄+ ions not separated by precipitation with the addition of an anion?

2. What reacts with the litmus paper to turn it blue in the first test?

3. Why is necessary to heat the ammonium test solution gently without boiling?

4. How should the flame test wire be cleaned before testing for Na+ or K+?

5. When doing the flame test for sodium what should you do if you are not certain that your results indicate the presence of sodium ions?

8 Gravimetric Analysis

INTROCUTION

The techniques of classical chemical analysis can be divided into two broad categories: gravimetric analysis and volumetric analysis. The purpose of this experiment is to employ the gravimetric method of analysis to determine the concentration of H_2SO_4 in solutions that have been prepared by dilution from a sulfuric acid solution of definite, but unknown, concentration. From the dilutions, the concentration of the unknown H_2SO_4 solution may then be calculated.

There are 2 common types of gravimetric analysis. Both involve changing the phase of the analyte to separate it from the rest of a mixture, resulting in a change in mass.

Volatilization gravimetry involves separating components of our mixture by heating or chemically decomposing the sample. The heating or chemical decomposition separates out any volatile compounds, which results in a change in mass that we can measure.
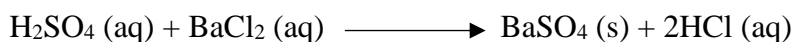
Precipitation gravimetry is an analytical technique that uses a precipitation reaction to separate ions from a solution. The chemical that is added to cause the precipitation is called the precipitant or precipitating agent. The solid precipitate can be separated from the liquid components using filtration, and the mass of the solid can be used along with the balanced chemical equation to calculate the amount or concentration of ionic compounds in solution.

A gravimetric precipitation method must fulfill certain basic requirements in order to provide an accurate analysis.

1. The precipitation reaction should be ***quantitative*** so that only a negligible fraction on the component of interest remains in solution after the precipitate is isolated. A moderate excess of the precipitating agent is commonly employed to help achieve this condition.

2. Other constituents in the sample solution should not interfere with the precipitation of the component of interest.

3. The precipitation should be as free of contaminants as possible.

4. The particles of precipitate should be large enough to filter easily and to facilitate the removal of soluble impurities during washing operations.

5. The precipitate should easily be converted to a form suitable for weighing.

6. In addition to a definite composition, the weighing form should have: stability at the relatively high temperatures required for the drying.

In this experiment, the amount of sulfuric acid present is determined using precipitation gravimetry by producing and weighing $BaSO_4$ (s), by reaction with $BaCl_2$ (aq).

$$H_2SO_4 \text{ (aq)} + BaCl_2 \text{ (aq)} \longrightarrow BaSO_4 \text{ (s)} + 2HCl \text{ (aq)}$$

A precipitate for precipitation gravimetric analysis must be of low solubility, high purity and of known composition if its mass is to accurately reflect the mass of an analyte. Also, the precipitate must be in the form that is easy to separate from the reaction mixture.
$BaSO_4$ is suitable for this purpose, because it is precipitated readily and completely from the solution being analyzed. Furthermore, the resulting precipitate is pure and easily filtered, it is a solid of known and definite composition, and its molar mass is sufficiently large that an appreciable and easily weighed mass of precipitate will be produced.

SAFETY PRECAUTIONS

The original, concentrated H_2SO_4 solution can cause chemical burns if it comes in contact with your skin. Any spills or splashes should be promptly cleaned up so that others in the laboratory are not accidentally exposed. If you come in contact with this solution, you should wash the affected area for at least 5 minutes with running water, inform your laboratory instructor, and seek medical attention. An accidental exposure to concentrated H_2SO_4 will manifest itself after some time as a burning sensation on your skin. If you feel this sensation, seek immediate attention. Delay can lead to a serious skin burn. The dilution of concentrated solutions of acids can be hazardous operation if proper precautions are not taken. The concentrated acid should be added to water, rather than water to the concentrated acid ("Acid to water is the way that you oughter.'' Splattering or spitting often occurs when water is added to a concentrated acid because of the substantial heat of dilution that is generated.). Of course, you should wear goggles throughout this procedure (as well as during the rest of the experiment) and avoid looking down into the reaction mixture, to avoid inadvertent splashes to your hands or face. Barium ion is a heavy metal poison and should be handled with care. Although there is no conclusive evidence that poisoning occurs on skin contact, all spills should be cleaned up

promptly in order to avoid accidental contact. Also, skin exposed to solutions of barium ion should be washed repeatedly with soap and water. Once it is converted to barium sulfate, however, barium ion poses little hazard because of the extreme insolubility of the solid. Thus, after use glassware that has been used to hold $BaCl_2$ (aq) should be rinsed with a small volume of dilute H_2SO_4. This rinse should be disposed of in the manner described for solutions in the Disposal section of Part B.
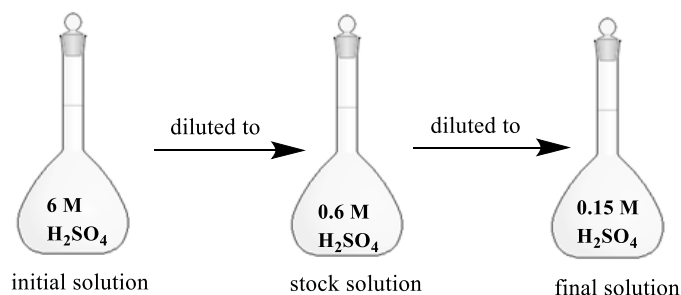
PROCEDURE

The steps involved in procedure are precipitation, digestion, filtration, washing, drying and weighing.

A. Dilution of Concentrated H_2SO_4

1. Obtain 25 ml of 6 M H_2SO_4 in a 100-ml or smaller graduated beaker. Label this as the initial solution. This beaker should be scrupulously clean to avoid contamination. After use, carefully rinse the beaker with water and discard the rinsing (see Disposal section at the end of Part A).

2. Place approximately 75 ml of distilled water in a clean 250-ml Erlenmeyer flask. Pipet 10.00 ml of 6 M H_2SO_4 into the distilled water. (Be sure to rinse the pipet with a small volume of the sulfuric acid solution first. If you are unsure of the proper, safe procedure for pipetting, consult your instructor before beginning. Follow the safety precautions noted above. Swirl the solution well to ensure thorough mixing.

3. Transfer the diluted H_2SO_4 solution to a 100.00-ml volumetric flask. Rinse the Erlenmeyer flask with three small aliquots (less than 5-ml) of distilled water and add each rinse to the solution in the volumetric flask. Do not allow the level of solution in the volumetric flask to go above the calibration mark on the neck of the flask. This rinsing procedure assures that all of the H_2SO_4 is transferred to the volumetric flask and is known as a **quantitative transfer**. Finally, add distilled water to the calibration mark of the volumetric flask. Swirl well to ensure through mixing and uniform concentration throughout the solution. This solution will be referred to as the stock solution (It has a concentration of approximately 0.6 M.). It can be transferred to a stoppered (to prevent spills and

contamination; make sure the stopper is clean and dry), clean (to prevent contamination), and dry (to

avoid dilution) Erlenmeyer flask for storage (and label "Stock Solution"). Rinse the volumetric flask with three 5-ml aliquots of distilled water. Discard the rinse water (Retain this stock solution until you have completed the experiment, so that you can quickly make up additional quantities of the final solution, by following the directions of step 4.).

4.   Pipet 25.00 ml of the stock solution into a 100.00-ml volumetric flask. This flask must be clean but not necessarily dry; you will be adding distilled water to its contents. Add several portions of distilled water, swirling well to mix thoroughly after each addition, to fill the flask to the mark. This **final solution** has a concentration of approximately 0.15 M. It should be stored in a stoppered, clean, and dry 125-ml Erlenmeyer flask (labelled "Final Solution") for further use. (Pipetting from a volumetric flask is awkward and can be hazardous.)

| 6 M H_2SO_4 | diluted to | 0.6 M H_2SO_4 | diluted to | 0.15 M H_2SO_4 |
| initial solution | | stock solution | | final solution |

Disposal:

Excess acid: Add phenolphthalein and neutralize with 1M NaOH (first appearance of permanent pink color). Flush the resulting salt solution down the sink with running water.

B. Gravimetric Determination of Sulfate Ion

1.   Obtain approximately 12 ml of 0.2 M $BaCl_2$(aq) in a clean, dry, stoppered 125-ml Erlenmeyer flask.

2.   Determine the mass of a piece of filter paper to the nearest mg. With pencil (not with ink!) write an identifying mark on the outside edge of the filter paper. Fold the filter paper in quarters, place it in a funnel, and moisten it with distilled water

so that it adheres to the walls of the funnel. Avoid handling the filter paper, so that contamination is minimized.

3.   Using a 10-ml graduated cylinder transfer 5.0 ml of the 0.2 M BaCl$_2$ obtained in step 1 into a 50-ml beaker. This provides a slight excess of barium ion. Warm this solution to near boiling (about 15 minutes on a hot plate). Do not heat to dryness.

4.   While the above solution is heating, pipet 5.00 ml of the **final solution** of H$_2$SO$_4$ into a 50-ml beaker. Add 5 ml of 1.0 M HCl (measured with a graduated cylinder) to this solution. This added acid ensures that large particles of precipitate will form. Heat this solution to near boiling (about 15 minutes on a hot plate). Do not heat to dryness.

5.   While both solutions (steps 3 and 4) are still hot, slowly add the BaCl$_2$ (aq) into the beaker of H$_2$SO$_4$ (aq), with *vigorous stirring*. Add *slowly* to ensure that large particles of precipitate are formed. Rinse the stirring rod with your wash bottle before removing it from the beaker.

6.   To further promote the formation of large particles of precipitate, you may heat the mixture in the beaker to near boiling on a hot plate and keep it at this temperature for at least 30 minutes. Do not heat to dryness. Stir occasionally. Then allow cooling slowly. This digestion procedure dissolves small particles of precipitate, and the BaSO$_4$ that dissolves at a high temperature precipitates out on the remaining particles when the mixture cools.

7.   Use the stirring rod to direct the liquid, and pour the mixture from the beaker onto the filter paper in the funnel. Wash the precipitate from the beaker with repeated 5-ml aliquots of distilled water. Make sure to also wash off the stirring rod so that all solid is quantitatively transferred. (If some of the fine precipitate passes through the filter paper, re-filter the filtrate through the same filter paper. Make sure to quantitatively transfer all of the filtrate to the funnel.) Carefully transfer the wet filter paper from the funnel onto a clean dry paper towel, and place it in your locker to dry until the next laboratory period. Be very careful with this transfer, since wet filter paper may tear.

8.   Repeat steps 2 through 7 for a second determination.

9. Rinse all glassware that has been used for $BaCl_2$ (aq) with small portions of H_2O. Discard these rinsing in the manner indicated for solutions in the Disposal section.

10. During the next laboratory period, determine the mass of filter paper and $BaSO_4$(s) to the nearest mg. Subtract the recorded mass of the filter paper to determine the mass of the precipitate.
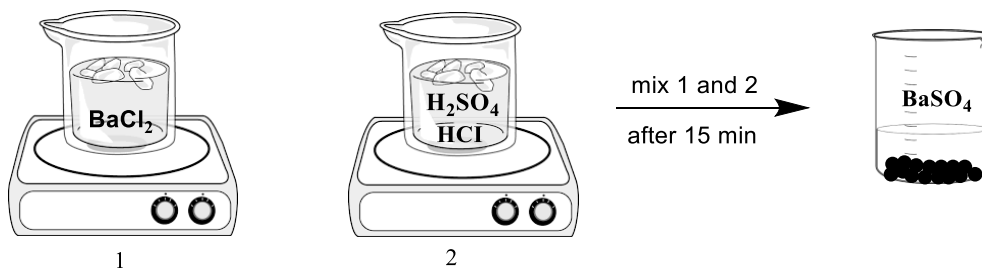
Disposal:

Solid BaSO₄: Put precipitate and filter paper into a waste bottle labeled *heavy metals-solids*.

Filtrate and unused BaCl₂ solution: Add 6 M H_2SO_4 to precipitate all the barium as $BaSO_4$ and filter into a labeled waste bottle. The solid will be disposed in the same manner as that above, and the acid solution will be neutralized and disposed in a safe manner.

Name_____ **Department** _____**Group** ____

DATA & CALCULATIONS

	Trial 1
Mass of filter paper, g	
Mass of (BaSO$_4$ + filter paper), g	
Mass of BaSO$_4$ precipitate, g	
Millimoles of BaSO$_4$	
Millimoles of H$_2$SO$_4$ reacted	
Volume of final solution reacted, ml	
[H$_2$SO$_4$], final solution, M	
[H$_2$SO$_4$], stock solution, M	
[H$_2$SO$_4$], initial solution, M	
Average [H$_2$SO$_4$], initial solution, M	

Name_____ Department _____Group _____

REPORT QUESTIONS

1. There is an inherent flaw in the drying procedure for the gravimetric determination that causes the measured mass of $BaSO_4$ to be erroneously high. What is it and how could the procedure be improved?

2. If the $BaSO_4$ residue was baked in an oven at 100 °C for several hours rather than air drying, what effect would this have on its mass and how would the initial concentration of H_2SO_4 be affected (higher, lower or no change)? Explain.

Name_____ **Department** _____**Group** ____

PRELAB QUESTIONS

1. A 25 ml sample of 6 M H_2SO_4 is diluted by following the procedure in Part A. Calculate the concentration of the ''final solution'' after two dilutions.

2. Assuming the final solution has the concentration calculated in question 1 above, what mass of $BaSO_4$ should be obtained? Show your calculations.

3. Assuming the same final solution concentration calculated in question 1 above, what volume of 0.100 M NaOH will be needed for each titration?

4. What is meant by the term quantitative transfer?

5. Why is it necessary to be very careful when handling H_2SO_4?

Scores of the General Chemistry Laboratory I Experiments

Experiment 1: Measurements and Density

	CHEM 141	CHEM 131
Title Page	5	5
Purpose	5	5
Introduction	10	-
Procedure	5	10
Data & Results	5	10
Calculation	30	30
Questions	20	20
Conclusion	15	15
Tidy	5	5

Experiment 2: The Stoichiometry of a Reaction

	CHEM 141	CHEM 131
Title Page	5	5
Purpose	5	5
Introduction	10	-
Procedure	5	10
Data & Results	10	10
Calculation	30	30
Graph	15	15
Conclusion	15	20
Tidy	5	5

Experiment 3: Titration of Acids and Bases

	CHEM 141	CHEM 131
Title Page	5	5
Purpose	5	5
Introduction	10	-
Procedure	5	10
Data & Results	5	10
Calculation	25	25
Questions	25	25
Conclusion	15	15
Tidy	5	5

Experiment 4: Oxidation – Reduction Titration

	CHEM 141	CHEM 131
Title page	5	5
Purpose	5	5
Introduction	10	-
Procedure	5	5
Data & Results	10	10
Questions	20	20
Calculation	25	30
Conclusion	15	20
Tidy	5	5

Experiment 5: Gas Analysis Based on the Molar Volume

	CHEM 141	CHEM 131
Title Page	5	5
Purpose	5	5
Introduction	10	-
Procedure	5	10
Data & Results	10	10
Calculation	45	45
Conclusion	15	20
Tidy	5	5

Experiment 6: Thermochemistry: The Heat of Reaction

	CHEM 141	CHEM 131
Title Page	5	5
Purpose	5	5
Introduction	15	-
Procedure	5	15
Data & Results	5	10
Calculation	30	30
Graphs	15	15
Conclusion	15	15
Tidy	5	5

Experiment 7: Group I: The Soluble Group

	CHEM 141	CHEM 131
Title Page	5	5
Purpose	10	15
Introduction	15	-
Procedure	15	20
Observation	25	30
Conclusion	25	25
Tidy	5	5

Experiment 8: Gravimetric Analysis

	CHEM 141	CHEM 131
Title page	5	5
Purpose	5	5
Introduction	10	-
Procedure	5	10
Data & Results	10	10
Questions	10	10
Calculation	30	30
Conclusion	20	25
Tidy	5	5