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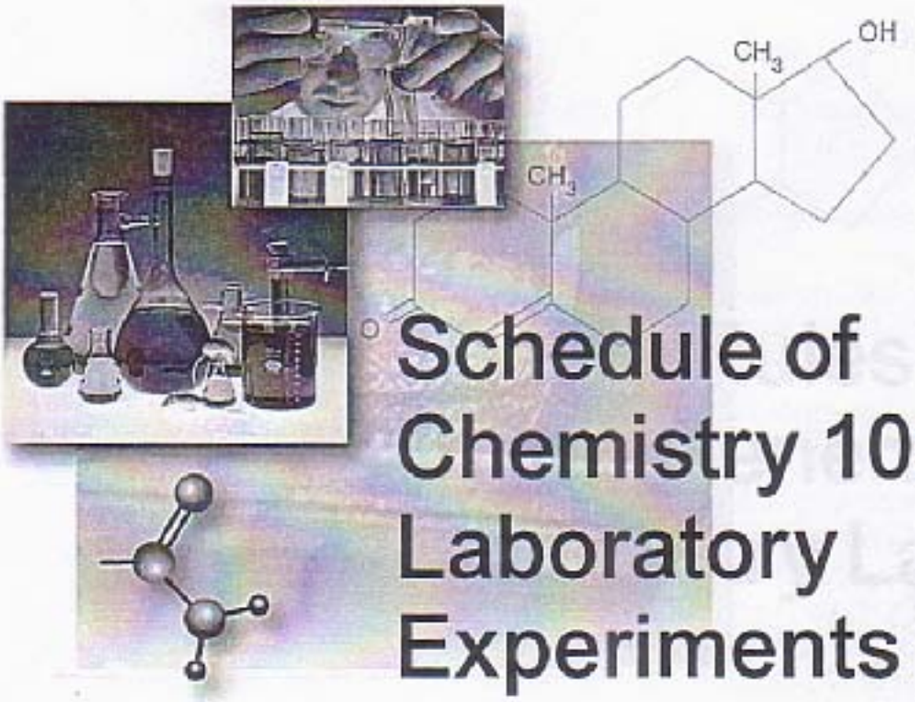
the prentice hall custom laboratory program for chemistry

Scott Berlant

City College of New York

Chemistry 103



Schedule of Chemistry 103-1 Laboratory Experiments

Wk 1:	Lab group formation, material collection, safety discussion. Experiment on the density of solids.
Wk 2:	Introduction to graphing techniques. Manually and computer generated graphs of experimental density data.
Wk 3:	Lecture problem set.
Wk 4:	Discussion on the experimental method for the determination of the formula of an unknown salt. Job problem set and demonstration of filtration technique.
Wk 5:	Determination of the formula of a salt.
Wk 6&7:	Standardization of a sodium hydroxide solution using oxalic acid dehydrate and potassium hydrogen phthalate.
Wk 8:	Acid-base titration of a KHP unknown.
Wk 9&10:	Calorimetry; Enthalpy of neutralization of ammonia. Enthalpy of solution of ammonium salts.
Wk 11:	Introduction to spectroscopy, Discussion and problem set.
Wk 12:	Spectrophotometric determination of iron in vitamin tablets; part 1 standard curve for Fe^{3+}.
Wk 13:	Part 2 determination of the iron content in an unknown tablet.
Wk 14:	PC model assignment.

Grading Scheme

Density/graphing	15%
Formula	15%
Titration	25%
Calorimetry	15%
Spectroscopy	20%
PC Model	10%

Attendance is mandatory! 2 or more unexcused absences from the lab will lead to your being dropped from the course.

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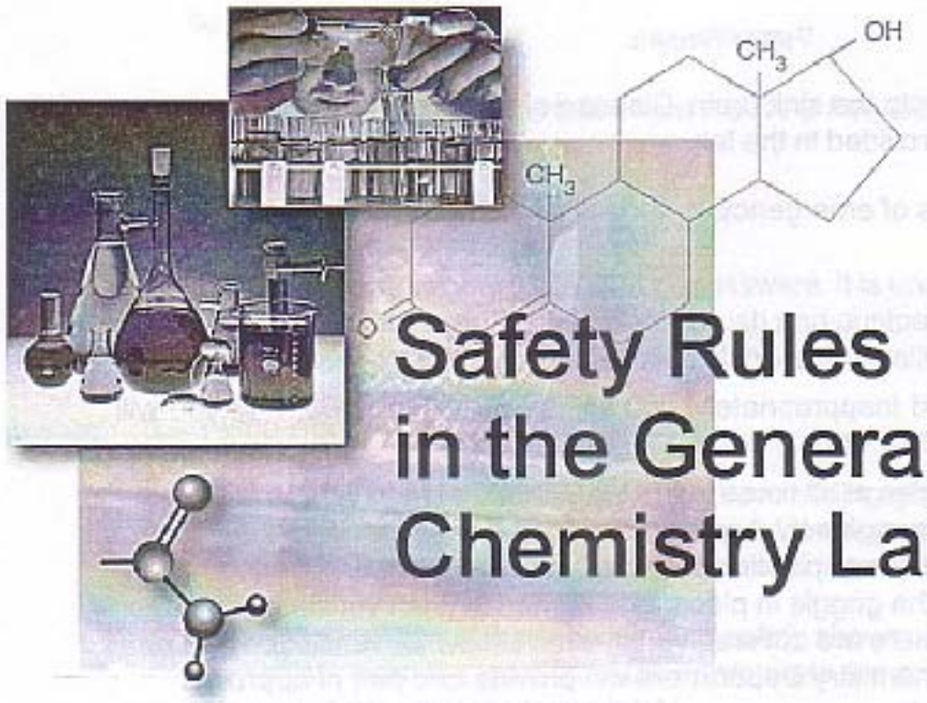

Safety Rules in the General Chemistry Laboratory

Table of Contents

- Introduction to safety rules
- Dress code
- Safety equipment in the lab
- What to do in case of an accident, injury or illness.

Introduction to Safety Rules

The City College Chemistry Department wants you to learn chemistry safely. That means that certain rules of operation in the lab must be followed. Please note: your laboratory instructor will reduce your grade or ask you to leave the lab if the safety rules are not followed!

1. You should bring no food or drink into the laboratory, to avoid possible contamination.
2. Keep your hands away from your face, while working in the lab, and do not put anything in your mouth.
3. Wash your hands as often as possible, especially before leaving the lab.
4. Keep your workstation neat and clean.
5. No horseplay, practical jokes or tricks, which may lead to loss of concentration or worse to serious injury.
6. Be well prepared before you come to the lab.

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7. Do not pour chemicals into the sink drain. Dispose of all hazardous waste in the appropriate container provided in the lab.

8. Know what to do in case of emergency.

Dress Code

If you come to the lab dressed inappropriately, you will be asked to leave, and you will receive a grade of "0" for the day's work.

1. Wear splash-proof goggles at all times in the lab. State and federal law requires the use of splash-proof goggles by anyone working in a chemical laboratory. The goggles must have a rubber or plastic seal, which fits snugly against your face, an elastic strap that holds the goggle in place, and baffled indirect ventilation. Indirect ventilation means that there are covered vents, which allow for ventilation and no chemical splash. The Chemistry Department will provide one pair of approved goggles for each student.

2. Do not wear contact lenses!

   Contact lenses pose several dangers. First, contact lenses can absorb and react with chemical fumes. No goggles can protect you from fumes! Second, if you were to get liquid or solid chemical into your eye, the chemical would get under the contact lens. A contact lens would hamper efforts to wash the chemical out of your eye, thus making the damage much more severe.

3. Tie back long hair.

   Long hair can accidentally fall into flames or chemicals. Many hair sprays, gels, mousse, etc. are flammable. Think about this! Loose long hair can block your vision, which may lead to an accident.

4. Do not wear clothing which is loose enough to knock over containers on the bench or dip into flames or chemicals.

5. Wear clothing (shirt, blouse, or dress) which covers and protects your full front and back, including shoulders, and upper arms.

6. Wear clothing (pants, long skirt or dress), which covers and protects your body all the way down to and including your ankles.

7. Wear shoes which cover and protect your feet completely.

Safety Equipment

1. First Aid

   If injury occurs, a student will normally be escorted to the Student Health Services Office (J-15) to receive treatment from the health care professional there, or can

wait in the lab for an EMS team from the NYC Fire Department if the injury is severe.

2. Brush and Dust Pan

   In the Chemistry Lab we use a lot of glassware. It is unsafe to pick up broken glass with your hands. Instead, use a table brush and dustpan to collect the broken glass. The broken glass should be disposed of in the specially marked box in the lab.

3. Fume Hoods

   These are large metal cabinets, which have sliding glass doors in front. Fume hoods are used to protect you from harmful fume gases and odors. Use the fume hood when working with concentrated ammonium hydroxide or evaporating liquids.

4. Know the location of the eyewash and safety shower in the lab. Also, on the first day of lab, learn the location of the appropriate fire exit. (See the evacuation route placard at the entrance to the lab.)

5. Organic vapors can flare up when brought into contact with a hot surface. Avoid using alcohol/and or acetone, which are very flammable, in the presence of a hot plate or flame.

6. Accidents happen! Even with the greatest care, something will sometimes go wrong.

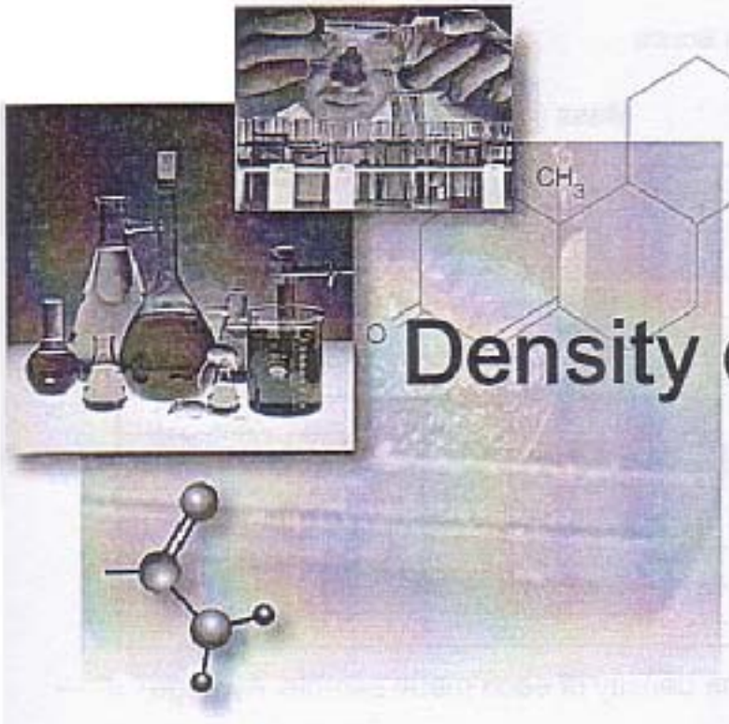
   a. If you get a corrosive chemical on your skin, go to the large sink and wash it off with lots of water. If this is not sufficient, and you still feel a stinging sensation, ask your lab instructor for the acid neutralizing powder. What ever you do, don't try to neutralize a strong acid with a strong base on your skin, or vice versa—This turns a mere accident into a major disaster.

   b. Corrosive chemicals that you will use in the lab include: Nitric Acid, Sulfuric Acid, Hydrochloric Acid, Ammonium Hydroxide, and Sodium Hydroxide.

   c. If there is a fire, the lab instructor will alert you to evacuate the lab room in an orderly manner and proceed to the appropriate exit. If your clothing is on fire go to the safety shower at once to extinguish the flame.

   d. For serious injury or illness in the lab

      1. Use the lab phone to call security at #7777.
      2. Call NYC EMS at #9-911.
      3. If the lab phone is not available proceed to call at the security phone located adjacent to the bank of elevators.



Density of Solids

Name Lab Section

The density of a substance is defined as its mass divided by its volume. In this experiment, we will determine the density of a metal of known composition and one of unknown composition. For each metal, we will measure the density directly and graphically and compare the two methods.

Obtain six aluminum samples and six samples of one metal of unknown composition. Measure the masses of each sample with a top loading balance and record the masses in the data table. Determine the volume of each metal sample by the water displacement method: fill a 25 ml graduated cylinder with 10.0 ml of water, carefully submerge the metal in the water by tilting the graduate cylinder and sliding the metal down the side, and measure the water volume again. The difference in the water volume corresponds to the volume of the metal sample. Record the volume of each sample to one decimal place in the data table.

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DENSITY OF SOLIDS

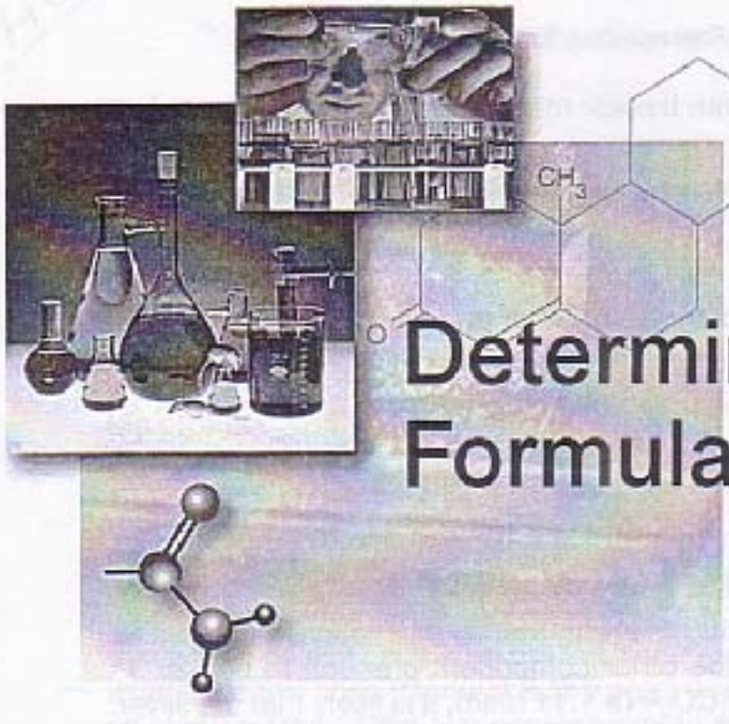
Sample #	Metal	Mass (g)	Volume (cm^3)
1		6.772	~~15.8 Tod~~
2		13.297	11.6 – 16 mL
3		15.119	13.0 – 18.4
4		17.459	13.0 – 19.8
5		21.295	13.6 – 21.4

A. Divide the mass by the volume to get the density of each metal sample. Average the density values for each metal.

B. To identify the relationship between the mass and volume of a given metal, draw a graph, plotting the mass of each aluminum sample on the y-axis and its volume on the x-axis. Measure the slope of the line. (See page A-6 in the Appendix of the Radel and Navidi textbook.)

C. On the same sheet of graph paper, plot the mass and volume of the samples of the metal of unknown composition and measure the slope. What are the similarities and differences in the two graphs?

D. For each metal, compare the average density determined in part A with the slope of the appropriate line. Under what conditions does the slope of each line equal the density of the metal?



Determination of the Formula of a Salt

Objective: To determine the empirical formula for a salt

Prior Reading. Appendix 1. Use of Volumetric Glassware

Introduction: When a new compound is synthesized, one of the first pieces of information needed is its empirical formula. There are several methods of obtaining formulas, depending on the type of compound.

This experiment introduces a straightforward technique for determining the empirical formula of simple, relatively insoluble salts. The method involved mixing known amounts of the salt's cation (positive ion) and anion (negative ion) in varying ratios, and determining which ratio yields the greatest quantity of the salt, this ratio being the formula ratio. As an example, suppose that we wish to determine the empirical formula of silver carbonate.

Solutions of silver nitrate (providing a source of silver ions, Ag^+) and sodium carbonate (a source of carbonate ions, CO_3^{2-} are prepared. Varying quantities of these two solutions are mixed and the precipitate (y axis) is plotted as a function of the quantity of reagents used (x axis). The most precipitate is produced when the amounts of reagents mixed are in the same ratio as in the salt's empirical formula.

For convenience, the concentration of the cation and anion solutions mixed should be the same. The total volume of the two solutions used must be constant for all trials (i.e., as more cation solution is used less anion solution is used).

This process may be understood in terms of the silver carbonate example above. Suppose that the concentration of each of the two solutions is 0.100M. M is the symbol for molarity, moles of dissolved material per liter of solution. In six different beakers, we place 1.0, 3.0, 5.0, 7.0, 9.0 and 11.0 mL of the $AgNO_3$ solution mixing with, respectively, 11.0, 9.0, 7.0, 5.0, 3.0 and 1.0 mL of the Na_2CO_3 solution. Note that the total volume is 12.0 mL in each case.

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The number of moles of each reagent is a given beaker may be easily determined using the relationship

volume x molarity = moles

liter x mole/liter = moles

Thus, we can calculate that the total number of moles of silver and carbonate ions is constant in each mixture:

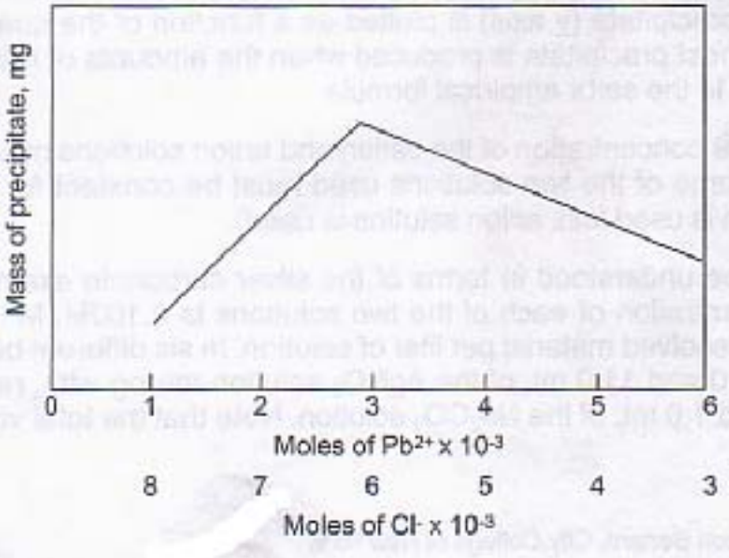
$$12.0 \text{ mL of solution} \times \frac{0.1 \text{ mol of ion}}{\text{liter}} \times \frac{1 \text{ L}}{1000 \text{ mL}} = 1.2 \times 10^{-3} \text{ mol of ion}$$

The amount of Ag^+ increases in a regular fashion from 1.0×10^{-4} to 1.1×10^{-3} mol and the amount of CO_3^{2-} decreases in the reverse order.

This method relies on the concept of the limiting reagent, that is, that reagent present in insufficient amount for complete reaction of the other component present. In beaker 1, with 1.0×10^{-4} mol Ag^+ and 1.1×10^{-3} moles of CO_3^{2-} (a 1:11 ratio), it is likely that the silver ion is the limiting reagent. In the second beaker, the ratio is 1:3 and silver ion is probably still limiting. In successive beakers the mole ratio of silver ion to carbonate ion is 5:7, 7:5, 3:1 and 11:1. With increasing amounts of silver ion from one mixture to the next, we would expect that more precipitate would be collected until the point is reached where silver ion is on longer limiting (remember that the amount of carbonate ion is decreasing.) From that point on, carbonate ion is limiting. Since the amount of carbonate ion decreases, we would expect less and less precipitate to be collected in the remaining beakers. Specific instructions for this type of plot can be found in Pre-Lab Question 1.

A typical plot is shown in Figure 1. Note that the total number of moles of ions is constant at 9×10^{-3} mol, and that the maximum on the graph occurs at 3×10^{-3} mol of Pb^{2+} and 6×10^{-3} mol of Cl^-. Since the amount of Cl^- is twice that of Pb^{2+}, we can conclude that the empirical formula for lead chloride is $PbCl_2$.

In this experiment, the formula of the salt resulting from mixing an unknown cation and oxalic acid dihydrate ($H_2C_2O_4 2H_2O$) will be determined. The oxalic acid is a source of oxalate ion $C_2O_4^{2-}$



Pre-Laboratory Question

The following data were collected for the silver carbonate experiment described earlier:

Beaker No.	1	2	3	4	5	6
Moles Ag^+	$1x10^{-4}$	$3x10^{-4}$	$5x10^{-4}$	$7x10^{-4}$	$9x10^{-4}$	$1.1x10^{-3}$
Moles CO_3^{2-}	$1.1x10^{-3}$	$9x10^{-4}$	$7x10^{-4}$	$5x10^{-4}$	$3x10^{-4}$	$1x10^{-4}$
Mg product	13.0	39.8	65.5	92.3	80.0	26.8

graph axes

Construct a plot using this data and determine the formula of the product. The most accurate method of determining the correct ratio is to plot the moles of Ag^+ and CO_3^{2-} on the x axis, and mass of product on the y axis. To simplify determination of the ratio at any given point, it usually helps to have two x axis scales, moles of one ion increasing to the right, moles of the other ion increasing to the left. The data should generate two straight lines, one with a positive slope on the left and one with a negative slope on the right. The point where the two lines intersect is the point where the ratio of the two ions must be the stoichiometric ratio of the two components in the molecular formula. If both ions are represented on the x axis, this ratio may be read directly from the graph (see Figure 1).

Experimental Procedure

Number six 50 mL beakers from 1 to 6 with a wax pencil. In two additional 100 mL beakers, obtain 60 mL each of 0.100 M unknown cation and 0.100 M $H_2C_2O_4 2H_2O$ solutions. Preheat a hot plate. Obtain two burets to dispense each of the solutions above.

NOTE: Each solution should have its own buret, and it should be pre-rinsed with a small amount of the solution.

In the six different beakers mix 2.00, 6.00, 10.00, 14.00, 18.00 and 22.00 ml (+0.01 mL) of 0.100M unknown cation solution with respectively, 22.00, 18.00, 14.00, 10.00, 6.00 and 2.00 mL of 0.100M $H_2C_2O_4 2H_2O$ solution. Mix the solution thoroughly by swirling or stirring. Heat each of the beakers for 5 minutes on the hot plate. Allow beakers to cool to room temperature.

Weigh and label six weighting boats, each containing a piece of 5.5 cm diameter circle of filter paper. (see Figure 2)

Assemble a suction filtration apparatus and water trap as shown in Figure 2. Swirl beaker 1 to suspend the precipitate and filter the contents using a Buchner funnel. Use a spatula, and 2–3 mL of distilled water from a wash bottle as a rinse to ensure complete transfer. Wash the filter cake with an additional 2–3mL of distilled water. Discard the aqueous filtrate and reassemble the filter. Wash the solid product twice with acetone (2-3 mL each).

Fig.2

NOTE: Acetone is extremely flammable. Keep it away from all flames and hot plates.

Draw air through the filter for 2 minutes to aid drying of the filter cake. Discard the acetone washes in a designated container. Transfer the solid on the paper to the weighing boat.

Use your spatula to transfer any remaining solid to the weighing boat. Repeat the filtering process for each of the five remaining mixtures. When each product is completely dry, weigh to the nearest mg. Dispose of the solid product in an appropriate waste container. Record your data on the Data Sheet.

Data Sheet:

Moles cation	Moles oxalate	Weight of product

Construct a plot (see Figure1) on the supplied graph paper. Determine the formula of the product.

Thermochemistry and Enthalpy of Reaction

PURPOSE

1. To measure the heat released by the neutralization of a strong base with a strong acid.
2. To use this value to calculate the concentration of a NaOH solution.
3. To measure the enthalpy of formation, ΔH_f, of magnesium oxide.

I. INTRODUCTION

A. Thermochemistry

Thermochemistry is the study of the energy changes that accompany chemical reactions, phase changes (such as melting) or the dilution of solutions. When these processes occur at constant volume there is a change in the *internal energy*, ΔE, of the system:

$$\text{Reactants} + \Delta E \rightarrow \text{Products} \qquad (1)$$

Since energy is conserved (*i.e.*, it must be the same on both sides of the arrow in Eq. 1), the change in the internal energy of the system can be written:

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$$\Delta E = E_{products} - E_{reactants} \quad (2)$$

Often ΔE manifests itself as a heat flow into or out of the environment of the system under study. When the internal energy of the products is less than that of the reactants, ΔE as defined by Eq. 2 is negative and heat is evolved during the process. Such a process with a negative ΔE is called *exothermic*. Conversely, if the process absorbs heat, the internal energy change of the system is positive, and the internal energy of the products is greater than that of the reactants. This process is called *endothermic*.

In this experiment and many other laboratory situations, it is more convenient to carry out reactions at constant pressure rather than at constant volume. When the system is open and thus not contained in a constant volume, it can expand or contract against constant atmospheric pressure. When a system expands, it does work on its surroundings. When it contracts, the surroundings do work on the system. In order for expansion to take place, the system must convert heat into work, both of which are forms of energy. When contraction takes place, work is converted into heat.

In the case of constant pressure, the heat change in the system is called the *enthalpy* change, ΔH. When the only work is a volume change against constant pressure, the relationship between the change in enthalpy (ΔH) and internal energy (ΔE) is as follows:

$$\Delta H = \Delta E + P\Delta V \quad (3)$$

Here, P is the pressure and ΔV is the change in volume. For the case in which the system expands and does work on its surroundings, the ΔV term is positive and ΔH is larger than ΔE. In the case where ΔV is negligible or zero (such as chemical reactions in solution), ΔH approximately equals ΔE.

B. Thermochemical Equations and Hess's Law

Thermochemical equations are chemical equations plus the numerical value (magnitude and sign) of the enthalpy change for the reaction proceeding in the forward direction as written. They can be manipulated in the same way as algebraic equations where the arrow replaces the equal sign. This generalization is known as Hess's Law, and it is

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very useful in ascertaining the thermochemical properties of a system for which direct measurement is difficult or impossible. From the definition of ΔE and ΔH (Eqs. 1, 2 and 3 above), the sign of ΔE, $P\Delta V$, and ΔH is changed when a thermochemical equation is reversed. For example:

$$H_2\,(g) + \tfrac{1}{2}O_2\,(g) \rightarrow H_2O\,(l) \qquad \Delta H = -285.8\ kJ/mole \qquad (4)$$

And for the reverse process:

$$H_2O\,(l) \rightarrow H_2\,(g) + \tfrac{1}{2}O_2\,(g) \qquad \Delta H = +285.8\ kJ/mole \qquad (5)$$

This means that the heat *evolved* in forming one mole of liquid water (Eq. 4) is equal to the heat *absorbed* in decomposing one mole of liquid water (Eq. 5).

One use of Hess's Law can be seen in the following example. It is not practical to try to directly measure the heat evolved when carbon burns to carbon monoxide because an uncertain mixture of carbon monoxide and carbon dioxide is produced. However, carbon can be burned completely to carbon dioxide. The heat of reaction has been measured for graphite at 25°C as follows:

$$C\,(s) + O_2\,(g) \rightarrow CO_2\,(g) \qquad \Delta H = -393.51\ kJ/mole\ (exothermic) \qquad (6)$$

The heat evolved when carbon monoxide burns completely to carbon dioxide has also been measured at 25°C and is as follows:

$$CO\,(g) + \tfrac{1}{2}O_2\,(g) \rightarrow CO_2\,(g) \qquad \Delta H = -283.01\ kJ/mole\ (exothermic) \qquad (7)$$

By manipulating these equations, we can calculate the heat evolved in the reaction of interest at 25°C:

$$\begin{array}{ll} C(s) + O_2(g) \rightarrow CO_2(g) & \Delta H = -393.51\ kJ/mole \\ + \ CO_2(g) \rightarrow CO(g) + \tfrac{1}{2}O_2(g) & \Delta H = 283.01\ kJ/mole \\ \hline C(s) + \tfrac{1}{2}O_2(g) \rightarrow CO(g) & \Delta H = -110.50\ kJ/mole \end{array}$$

Notice that the second reaction is reversed; therefore, the sign of its ΔH is changed. In particular, 283.01 kJ of heat is absorbed when one mole of carbon dioxide is decomposed into one mole of carbon monoxide plus half a mole of oxygen.

One can schematically represent the differences in enthalpy of these chemicals in the following enthalpy level diagram:

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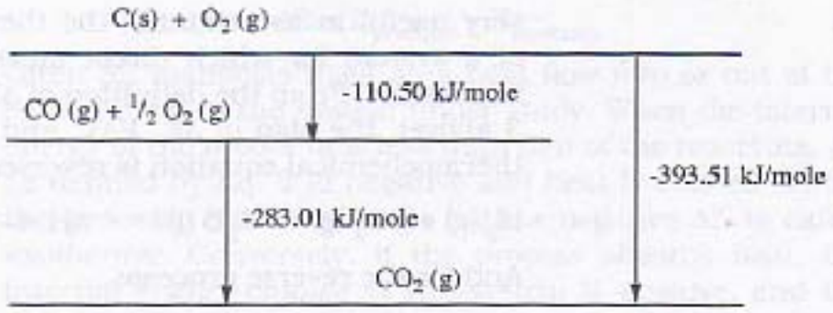

Figure 1

It is important to note that compounds must be in the same physical state (gas, liquid or solid) in order to add or subtract thermochemical equations in which they occur. For example, the enthalpy of water vapor is greater than that of liquid water by 43.93 kJ/mole:

$$H_2O\ (l) \rightarrow H_2O\ (g) \qquad \Delta H = +43.93\ kJ/mole \qquad (8)$$

Stated differently, it requires 43.93 kJ of energy is required to convert one mole of liquid water to water vapor.

C. Heat Capacity and Specific Heat

When heat (Q) is added to a system, the temperature of the system increases in direct proportion to the amount of heat added. Thus:

$$Q \propto \Delta T \text{ or}$$
$$Q = C\Delta T \qquad (9)$$

The proportionality constant, C, between the amount of heat added to a system and its consequent temperature change is called the *heat capacity*. It is the amount of heat required to raise the temperature of the system one degree Celsius (units: Joules/degree). The specific heat (C_s) is the energy (heat) required to raise one gram of a substance one degree Celsius. Remember that when working with liquids, the mass (in grams) is the product of the volume (ml) and the density (grams/ml).

Rearranging the Eq. 9 gives

$$C = \frac{Q}{\Delta T} = C_s \bullet m$$

$$\left(\frac{\text{Joules}}{^\circ C}\right) \qquad \left[\left(\frac{\text{Joules}}{^\circ C \bullet g}\right) \bullet (\text{grams})\right]$$

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Tables of specific-heat data are available in the literature. For example, the specific heat of air is 1.046 J/(g•deg), water is 4.184 J/(g•deg), alcohol is 2.427 J/(g•deg), and copper is 0.3766 J/(g•deg).

A knowledge of the specific heat allows one to calculate the amount of heat absorbed or released by a system by simply measuring the change in its temperature. For example, we can calculate the heat released when 20 ml of water (density = 1.00 g/ml) is cooled 20°C as follows:

$$Q = (20\text{ ml})(1.00\text{ g/ml})\left(\frac{4.184\text{ J}}{\text{g}\bullet\text{°C}}\right)(-20\text{°C}) = -1,673 \qquad (11)$$

The negative sign in Eq. 11 results from the *decrease* in the temperature *of the substance* or from the *increase* in the temperature of its environment.

Let's consider another example. If, as in this experiment, a reaction between two species takes place in water solution (the environment) and the reaction releases heat (Q_{rxn}), and the heat is absorbed by the solution (Q_{soln}), then Q_{rxn} will be negative and Q_{soln} will be positive.

In this experiment, you will obtain the Q_{soln} by measuring the temperature change for a system (solution of solvent plus solute). The specific heat (C_s) and density (ρ) of the system will be assumed to be the same as a pure water solution of the same volume. The heat capacity (C) is calculated by Eq. 10.

For a given reaction, the heat flow into the system is the same magnitude as the heat flow out of the reaction, but the sign is opposite.

$$Q_{soln} = -Q_{rxn} \qquad (12)$$

The value of Q_{rxn} can be used to determine either the concentration of the limiting reagent if the heat of reaction (Eq. 13) is known or the heat of reaction if the moles of limiting reagent are known (Eq. 14) You will do both calculations in this experiment.

$$\text{moles of limiting reagent} = \frac{Q_{rxn}\text{ (Joules)}}{\Delta H_{rxn}\text{ (Joules/moles)}} \qquad (13)$$

$$\Delta H_{rxn} = \frac{Q_{rxn}\text{ (Joules)}}{\text{moles of limiting reagent (moles)}} \qquad (14)$$

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D. Heat of Formation

The heat involved in the formation of a compound in its standard state from its elements in their standard states is referred to as the standard heat of formation of the compound, ΔH_f°. The superscript indicates that all materials are in their standard state. In the standard state, the compound or element is in the stable state 25°C and 1 atm pressure and the formation reaction occurred at 25°C and 1 atm of pressure. The enthalpy change for the formation reaction of the compound in its standard state from its elements in their standard states at some other temperature and/or pressure is called the heat of formation, ΔH_f. For example, the heat of formation of magnesium oxide is the enthalpy change associated with the formation of MgO from the elements, Mg(s) and O_2(g) at temperature T and pressure P:

$$Mg\,(s) + \tfrac{1}{2}O_2\,(g) \rightarrow MgO\,(s) \qquad \Delta H_f = ? \qquad (15)$$

The common unit for the heat of formation is kJ/mole of product formed. Solid magnesium, gaseous oxygen, and solid magnesium oxide represent standard states for these materials. If heat is liberated in the formation of a compound, ΔH_f is negative.

E. Outline of the Method

In this experiment you will measure the heat evolved and the enthalpy change of several exothermic chemical reactions. Each reaction is carried out in solution in an insulated container (to minimize the heat loss to the room). The insulated container used to measure the heat evolved or absorbed during a reaction is called a *calorimeter.*

In actual practice when an exothermic reaction occurs in a solution inside a calorimeter, some heat is absorbed by the calorimeter itself (increasing the calorimeter's temperature) and some heat radiates into the room. However, most of the heat liberated increases the temperature of the reaction solution. In this experiment we will assume that all of the heat is absorbed by the solution, since this is by far the largest fraction. We will further assume that the specific heat of the solution is the same as that of pure water. This is also a reasonable assumption because the solutions are dilute (*i.e.*, mostly water). The specific heat of water is 4.184 J/(g • degree). So if we have 80.0 ml of solution, then for

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each 1.0 °C rise in its temperature that we observe, 334.7 J of heat must have been absorbed. We must therefore measure the temperature increase of the solution that occurs as a result of the reaction being studied in order to measure the heat evolved.

One problem in measuring a temperature change is that calorimeters are never perfect and some heat is exchanged with the environment. Thus, for exothermic reactions, one will always observe a smaller temperature increase in the solution in the calorimeter than one should. By plotting a cooling curve (temperature readings as a function of time, as shown in Figure 2) of the solution and extrapolating it to zero time (the start of the reaction), one can correct for the temperature loss due to cooling. This must be done for each determination.

Scale the graph to fill as much of the graph paper as possible. As in Figure 2 below, the vertical axis (the temperature axis) should not have T = 0 °C at the origin, but rather a temperature slightly below the initial temperature of your experiment. This scaling will ensure that you get the best resolution and the most significant figures from your plot.

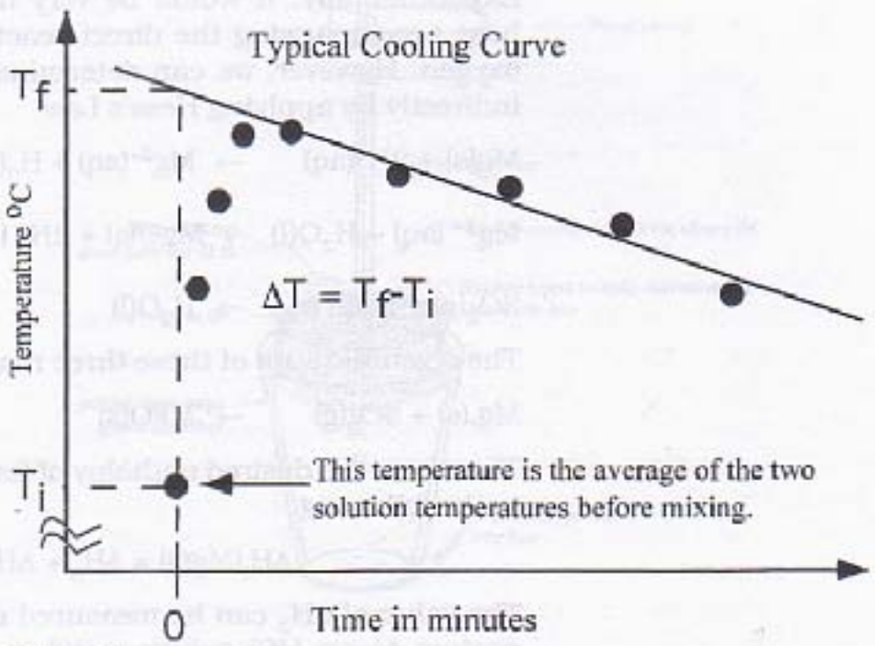

Figure 2.

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F. Heats of Neutralization

The first measurement of the heat evolved in a reaction is for the neutralization of NaOH with HCl. The amount of heat liberated depends on the amount of product that is formed. From your experimental result and the known enthalpy for the formation of H_2O, the concentration of OH^- will be calculated. Neutralization of a strong acid with a strong base can be represented by the net ionic equation:

$$H^+ + OH^- \rightarrow H_2O \qquad \Delta H = -55.81 \text{ kJ/mole} \quad (16)$$

The equation also shows that *55.81 kJ of heat* are released (negative sign) by the reaction for each mole of H_2O formed. In practice, the total amount of heat evolved depends on the amount of acid and base, but not on the type of acid and base used, provided they are both strong electrolytes (*i.e.*, completely ionized). For example, the amount of heat liberated when 50 ml of 2 M $HClO_4$ reacts with a strong base and when 50 ml of 2 M HCl reacts with a strong base is the same because each contains the same amount of available H^+.

In this experiment, the H^+ is always in excess, causing OH^- to be the limiting reagent.

G. Heat of Formation of MgO

Experimentally, it would be very difficult to measure the heat accompanying the direct reaction of magnesium and oxygen. However, we can determine this heat of formation indirectly by applying Hess's Law:

$$Mg(s) + 2H^+(aq) \rightarrow Mg^{2+}(aq) + H_2(g) \qquad \Delta H_3 \quad (17)$$

$$Mg^{2+}(aq) + H_2O(l) \rightarrow MgO(s) + 2H^+(aq) \qquad \Delta H_4 \quad (18)$$

$$\tfrac{1}{2}O_2(g) + H_2(g) \rightarrow H_2O(l) \qquad \Delta H_5 \quad (19)$$

The algebraic sum of these three reactions is as follows:

$$Mg(s) + \tfrac{1}{2}O_2(g) \rightarrow MgO(s) \qquad \Delta H_f(MgO) \quad (20)$$

Therefore, the desired enthalpy of formation of MgO is equal to the following:

$$\Delta H_f(MgO) = \Delta H_3 + \Delta H_4 + \Delta H_5 \quad (21)$$

The value of ΔH_3 can be measured directly by adding magnesium to an HCl solution and measuring the solution's

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change in temperature. The enthalpy change for the reaction as written in Eq. 18 is simply the negative of the heat liberated when MgO is dissolved in hydrochloric acid (that is, reverse the direction of the reaction and change the sign of ΔH). The value of ΔH_5 for Eq. 19, the enthalpy of formation of one mole of water, can be found from a reference table to be –285.85 kJ/mole.

II. PROCEDURE

A. Construction of the Calorimeter

Assemble the calorimeter according to Figure 3. Take care with the special thermometer. It is calibrated to 0.1°C and is rather expensive. The thermometer should be held in the buret holder with the aid of a rubber band *and* a buret clamp. Hold your hand underneath the thermometer when you first insert it into the buret clamp until you are assured that it is tightly held in place. When inserted in the calorimeter, the line on the thermometer should be ~ 1 cm above the top of the lid (measure with your ruler) so that the

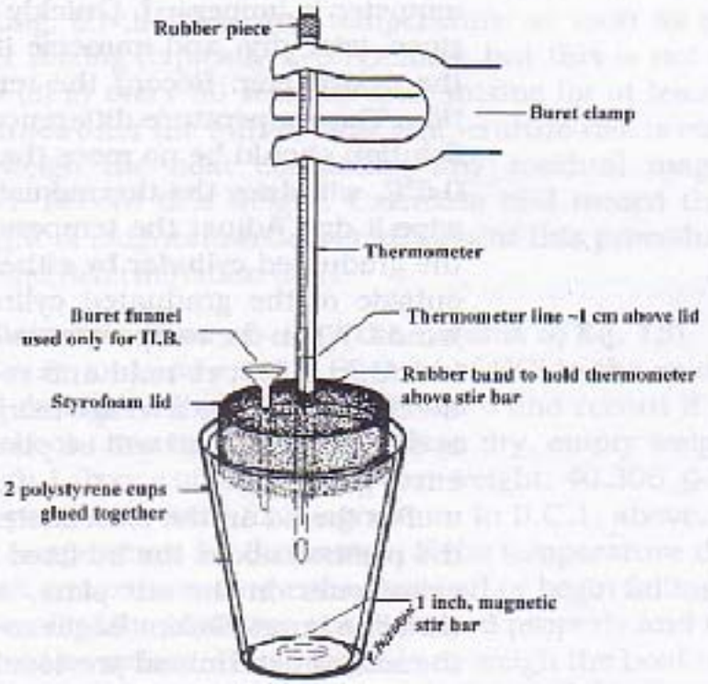

Figure 3. Calorimeter

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thermometer is not hit by the magnetic stir bar as it rotates. Place a second rubber band around the thermometer immediately above the lid to fix this position. Lift the lid and find the stir plate setting for slow stirring, before the solutions are added.

B. Heat of Neutralization for 1.00 M HCl with a NaOH Unknown

In the following, your 25 ml graduate cylinder will be used for the 1.00 M HCl and your 100 ml graduate cylinder for your unknown NaOH solution. Carefully measure 20.0 ml of the HCl solution with your 25 ml graduate cylinder and add it to the calorimeter. Add a second 20.0 ml aliquot of HCl solution for a total of 40.0 ml of HCl solution in the calorimeter. Place the lid in position, but leave the thermometer out. Carefully measure exactly 40.0 ml of your NaOH unknown solution using your 100 ml graduate cylinder. Allow it to stand near the calorimeter for 3 or 4 minutes. Measure and record the temperature of the NaOH solution by immersing the thermometer bulb just below the level of the solution, waiting about a minute, and then reading the temperature to the nearest 0.1°C while the thermometer is immersed. Quickly withdraw the thermometer, rinse, wipe dry, and immerse it in the acid solution inside the calorimeter. Record the temperature of the acid solution. The temperature difference between the base and acid solution should be no more than 0.5°C. If it is greater than 0.5°C, withdraw the thermometer from the calorimeter and wipe it dry. Adjust the temperature of the base solution in the graduated cylinder by either running cold water on the outside of the graduated cylinder or warming with your hands. When the temperatures of the solutions are adjusted to within 0.5°C, re-read and re-record the temperatures of the solutions. (The average temperature of the two solutions is defined as T_i, and will be placed at t = 0 on your cooling-curve graph.)

Put the lid on the calorimeter, insert the thermometer to the position above the lid fixed by the rubber band, set the calorimeter on the stir plate, and clamp the thermometer with the buret clamp. Begin to stir the acid solution using the setting determined previously. Then add the NaOH solution by pouring it through the funnel in the second hole of the cap (Figure 3). Record (1) the time of mixing, (2) the time

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and temperature as soon as possible after mixing (typically 2-5 seconds, but this is not at t=0), and (3) at every 30 seconds after mixing for at least 4 or 5 minutes after the initial, large temperature rise is complete. Clean and *dry* the calorimeter and repeat the neutralization temperature change measurement, for a total of two runs.

Record your unknown number in your notebook.

C. Heat of Formation of MgO

1. Determination of ΔH_3 (Eq. 17)

This procedure is essentially the same as that used in Part II.B. Measure and add exactly 60.0 ml of HCl to the calorimeter and determine its initial temperature and record the temperature. (This temperature is T_i and will be placed at t=0 on your graph.) Accurately weigh a clean, dry, empty weigh boat. Then weigh out 0.18xx g of granulated magnesium (atomic weight: 24.305g) in this weigh boat. Record this weight to four decimal places. Begin stirring the acid. Then add the magnesium to the calorimeter by lifting and replacing the lid (**not through the funnel**). Add the solid to the center of the cup, avoiding contact of the solid with the walls (where it can stick). Record (1) the time of mixing, (2) the time and temperature as soon as possible after mixing (typically 2–5 seconds, but this is not at t=0), and (3) at every 30 seconds after mixing for at least 4 or 5 minutes after the initial, large temperature rise is complete. Re-weigh the boat containing any residual magnesium solid. Record this weight. Calculate and record the exact weight of magnesium delivered. Repeat this procedure for a second determination of ΔT.

2. Determination of $-\Delta H_4$ (the reverse of Eq. 18)

Measure and add exactly 60.0 ml of HCl to the calorimeter and determine its initial temperature and record it in your notebook. Accurately weigh a clean dry, empty weigh boat. Weigh 1.0xxx g of MgO (molecular weight: 40.305 g). Repeat the procedure used for magnesium in II.C.1. above. Record the temperature for 5 minutes. If the temperature does not reach a maximum and either level off or begin falling in this time, all of the MgO was not dissolved properly and the trial should be redone. Don't forget to re-weigh the boat containing any residual MgO. Calculate and record the exact weight of MgO delivered. Repeat this procedure for a second determination of ΔT.

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DATA GUIDE

This page summarizes the minimum key data that must be recorded in your lab notebook. All other observations and data also should be recorded in your notebook.

II.B. Heat of Neutralization for 1.00 M HCl with a NaOH Unknown

1. For each of the two runs of the reaction of HCl and NaOH (runs 1 and 2):
   a) Temperature of the NaOH before mixing.
   b) Temperature of the HCl before mixing.
   c) Time and temperature for each reading after mixing.
   d) Unknown number.

II.C. Heat of Formation of MgO

1. For each run of the reaction of HCl and Mg (runs 3 and 4):
   a) Temperature of HCl before mixing.
   b) Weight of the empty weigh boat.
   c) Weights of Mg + boat.
   d) Time and temperature for each reading after mixing.
   e) Weight of boat containing any residual Mg.
2. For each run of the reaction of HCl and MgO (runs 5 and 6):
   a) Temperature of HCl before mixing.
   b) Weight of the empty weigh boat.
   c) Weights of MgO + boat.
   d) Time and temperature for each reading after mixing.
   e) Weight of boat containing any residual MgO

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EXPERIMENT • THERMOCHEMISTRY AND ENTHALPY OF REACTION

(two sides)

Name (PRINT) _____
(Last) *(First)*

The time-temperature plots for all 6 determinations noted in the two Tables that follow must be *stapled* to this report. Each plot must be labeled by its determination # (to correspond to the calculated results below) and the chemical equation for the reaction under study. Clearly label on each graph T_i and T_f. Write on each graph "$\Delta T = T_f - T_i =$ " and "heat capacity = ".

II.B. Heat of Neutralization for 1.00 M HCl with a NaOH Unknown

Unknown number _____

Determination	1	2
1. ΔT (observed)		
2. C (calculated)		
3. Amount of heat absorbed by your solutions, Q_{soln}		
4. Number of moles of OH^- reacted by neutralization		
5. Molarity of the original NaOH solution, M		
6. Average molarity of the NaOH, $\overline{M}$		
7. Deviation from average, d		
8. Average deviation, $\overline{d}$		
9. Report $\overline{M} \pm \overline{d}$	±	

10. Show your calculations of 2), 3), 4) and 5) for determination #1 below: (Copy calculations from your notebook.)

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II.C. Heat of Formation of MgO

Determination	3	4	5	6
	HCl + Mg		HCl + MgO	
11. ΔT observed				
12. C (Calculated)				
13. Q_{soln}				
14. Number of moles of solid used				
15. ΔH_{rxn}				
16. Average ΔH_{rxn} in kJ/mole				

17. Calculate the heat of formation of MgO using your average values of ΔH_{rxn} (line 16) in the space below. Include the manipulations of the balanced (thermo)chemical equations and the enthalpy changes (ΔH). Circle your answer for ΔH_f.

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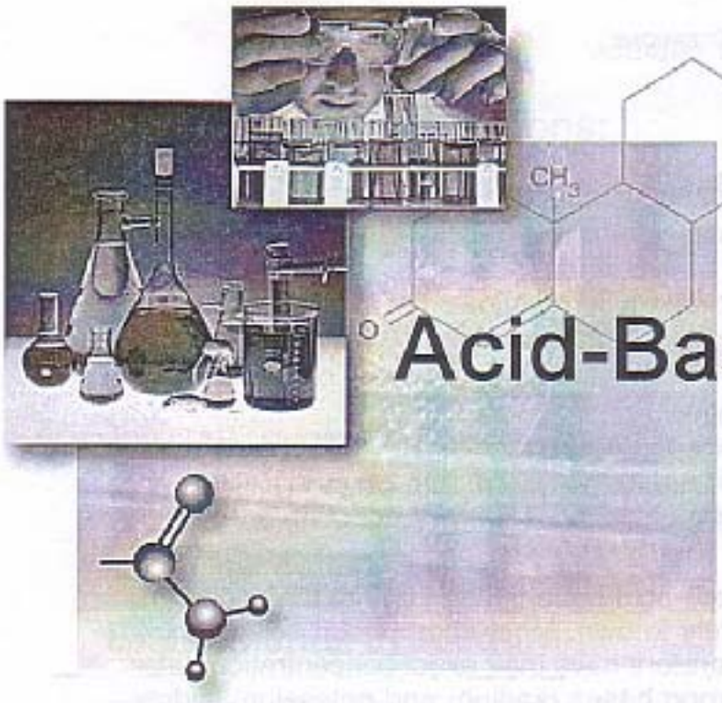

Acid-Base Titrations

Objectives:

- To perform an acid – base titration
- To standardize a base solution of unknown concentration
- To analyze a potassium hydrogen phthalate unknown

Introduction

There are many circumstances where one wishes to know the amount of a particular chemical (analyte) in a solution or mixture. This can be accomplished using an instrument such as a visible spectrophotometer; or can be done on the benchtop using a technique known as titration.

In a titration, a buret is filled with a solution of known concentration that will react with the analyte in question. Often, the reaction is arranged to result in the formation of a colored product, the onset of color indicating the end of the titration. In some cases, the colored product is formed by the reaction of the buret solution and the analyte. In other cases, a small quantity of an indicator is added, which will change color at the desired reaction endpoint. In still other cases, the analyte is colored to begin with, and the end of the reaction is determined by the disappearance of that color.

The simplest type of titration is an acid-base titration. Here, an acid of unknown concentration is titrated with a base of known concentration (or vice versa). The endpoint is determined by the color change of an added indicator. The standard indicator solution for this type of titration is phenolphthalein (pronounced "fee-nol-thay-leen"), which undergoes a color change from colorless in acid solution to pink in base solution. Only a few drops of phenolphthalein are needed. Some common indicators are listed in the Table.

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Common Indicators

Indicator Name	Acid Color	Base Color
Methyl orange	red	yellow
Methyl red	red	yellow
Litmus	red	blue
Phenolphthalein	colorless	pink

Standardization of Acids and Bases

One of the difficulties in performing an accurate acid-base titration is that the concentration of the titrating solution must be accurately known. Most commercial acids vary to some degree from batch to batch and must therefore have their exact concentration determined before they are used. The common strong bases (sodium and potassium hydroxide) are also problematic, as the solids are hygroscopic (absorb moisture from the air) and are nearly impossible to weigh accurately. It is therefore difficult to prepare a solution of exact concentration of these bases. This being the case, how does one prepare a solution of known concentration of any acid or base?

The simplest way is to standardize an acid or base solution using an acid or base which is solid, nonhygroscopic, and available in high purity. Solid acids or bases are, of course, easy to weigh accurately, and if they are nonhygroscopic, one need not worry about absorbed water. There are several common organic acids and bases that are used for this purpose. Oxalic acid ($H_2C_2O_4$) is a common primary standard which has two acidic hydrogens (diprotic), and consequently reacts with two moles of sodium or potassium hydroxide:

$$H_2C_2O_4(aq) + 2\ NaOH(aq) \rightarrow 2\ H_2O + Na_2C_2O_4(aq)$$

Potassium hydrogen phthalate ($KHC_8H_4O_4$, KHP) is also commonly used for this purpose. It is monoprotic and therefore reacts with one mole of sodium or potassium hydroxide:

$$KHC_8H_4O_4(aq) + H_2O\ NaOH(aq) \rightarrow H_2O + KNaC_8H_4O_4(aq)$$

Sodium or potassium hydroxide is usually standardized by titration with a solution of one of these primary standards, and their concentration can thereby be accurately determined. Any acid solution can then be titrated with the now standardized sodium or potassium hydroxide solution, and its concentration determined a well. Since the primary standard is used to standardize all other solutions, it is imperative that an exact mass of the material and an exact volume of solution be used.

Pre-Laboratory Questions:

1. Why are NaOH and HCl not used as primary standards for acid-base titrations? What properties should a primary standard possess?

2. When 1.05 mL of a solution of unknown concentration of NaOH is titrated with standardized 0.100M oxalic acid ($H_2C_2O_4$), 295 microliters of oxalic acid are needed to reach the endpoint. What is the molarity of the unknown NaOH?

3. The NaOH solution in Question 2 is used to standardize an unknown solution of HCl. It requires 134 microliters of the NaOH solution to titrate 1.00 mL of the HCl to the endpoint. What is the molarity of the HCl?

Experimental Procedure

See the Appendix at the end of the manual for instructions on the use of volumetric glassware.

Weigh out about 1.2 g of oxalic acid dihydrate, $H_2C_2O_4 \cdot 2H_2O$, to the nearest 1 mg in a weighing boat, and record the value on the data sheet. Place the solid in a 100 mL volumetric flask, and fill to the mark with distilled water. (Your instructor will demonstrate the technique of quantitative transfer.) Shake the flask to dissolve all of the acid. Calculate the molarity of this solution and record the value in the data sheet. This solution will serve as your primary standard. For more accurate results, more than one standard solution should be used and the results compared. Obtain 400 mL of approximately 0.1M NaOH solution to be standardized; this solution will be used in several subsequent experiments and should be kept in a tightly stoppered polyethylene bottle.

Obtain a 50 mL buret and rinse it twice with the sodium hydroxide solution, making sure that the solution wets the entire inner surface. Refill the buret with the sodium hydroxide solution and note the volume on the data sheet. Pipet exactly 20.00 mL of the oxalic acid solution into a 125 mL Erlenmeyer flask, add about 20 mL of distilled water, and add 2-3 drops of phenolphthalein solution. Place a white piece of paper under the flask for color contrast.

Add the sodium hydroxide solution from the buret, fairly rapidly at first, with swirling, until the pink color of the indicator begins to persist. At this point add the NaOH solution dropwise. Swirl after each drop is added. The endpoint is reached when the pink color of phenolphthalein indicator persists for at least 30 seconds. Note the final buret reading on the data sheet. Calculate the concentration of the NaOH solution. Repeat the procedure with 18.00 and 19.00 mL portions of oxalic acid solution, until your molarity values agree to ±0.004.

Analysis of a Potassium Hydrogen Phthalate Unknown

Weigh out 0.7-0.8 g of the sample of impure potassium hydrogen phthalate. Record the weight to the nearest mg on the data sheet, as well as the unknown number. Dissolve the solid in 50 ml of water in a 125 ml Erlenmeyer flask and add 2-3 drops of phenolphthalein. Titrate the impure (unknown) acid with the standard sodium hydroxide. Calculate the percent of potassium hydrogen phthalate in the sample. Repeat the procedure at least two additional times until the precision of your percent acid agrees to ±0.3%.

Calculations

The most common way of expressing the concentration of a solution is molarity (M). To determine the molarity of the oxalic acid solution used in this experiment, one divides the number of moles of oxalic acid in the solution by the volume of the solution (in liters). Keep in mind that oxalic acid is a diprotic acid. Thus, 2 moles of NaOH are required to neutralize completely 1 mole of oxalic acid.

$$H_2C_2O_4\ (aq) + 2\ NaOH\ (aq) \rightarrow 2\ H_2O + Na_2C_2O_4\ (aq)$$

Since the concentration and volume of the acid are known, the number of moles of acid can be easily calculated. Using the 2:1 base: acid stoichiometric ratio, we can determine the number of moles of base added. Since the volume of base added is known, the concentration of base can thereby be established.

Sample Calculation

Suppose that 10.00 mL of 0.09895M oxalic acid were titrated with sodium hydroxide solution. It was found that it took exactly 17.20 mL of the sodium hydroxide to reach the endpoint. What is the concentration of the sodium hydroxide solution? We first must calculate the number of moles of oxalic acid (OA) that were added:

$$\text{moles OA} = 0.0100\text{L OA} \times (0.09895\ \text{moles OA/L}) = 9.90 \times 10^{-4}$$

This is half the number of moles of sodium hydroxide that were added, due to the 2:1 mole ratio of base to acid. The molarity of the sodium hydroxide is therefore calculated from the following:

$$\text{moles NaOH} = 2\ \text{mole NaOH} / 1\ \text{mole OA} \times 9.90 \times 10^{-4}\ \text{moles}$$

$$\text{OA} = 1.98 \times 10^{-3}\ \text{moles}$$

$$M = 1.98 \times 10^{-3}\ \text{moles NaOH} / 0.0172\ \text{L NaOH} = 0.115\ \text{moles/L}$$

name _____ section _____ date _____

Acid-Base Titration Data Sheet

Chemistry 10301

Week 1: Oxalic Acid-Sodium Hydroxide Titration

Mass of Oxalic Acid Dihydrate (g) _____

Moles of Oxalic Acid Dihydrate _____

Moles of Oxalic Acid (in 100ml solution) _____

Moles of Oxalic Acid (in 20ml solution) _____

Moles of NaOH required to react with 20ml Oxalic Acid _____

	Trial 1	Trial 2	Trial 3	Trial 4
Initial Buret Reading (ml)				
Final Buret Reading (ml)				
Volume of NaOH added (ml)				
Molarity of NaOH (mol/l)				

Average Molarity of NaOH (mol/l) _____

Week 2: KHP (known)-Sodium hydroxide Titration

	Trial 1	Trial 2	Trial 3	Trial 4
Mass of KHP (g)				
Moles of KHP				
Moles of NaOH				
Initial Buret Reading (ml)				
Final Buret Reading (ml)				
Volume of NaOH added (ml)				
Molarity of NaOH (mol/l)				

Average Molarity of NaOH (mol/l) _____

name section date

Acid-Base Titration Data Sheet

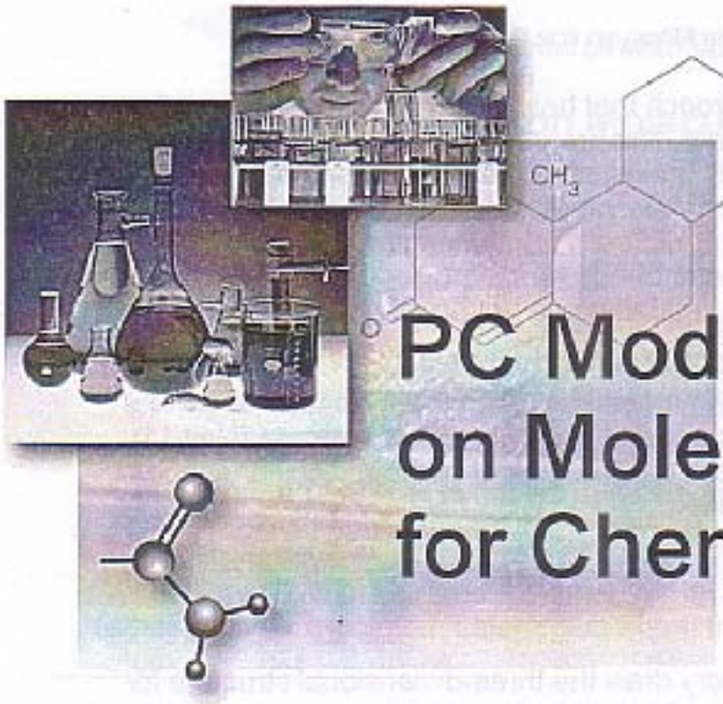
Chemistry 10301

Week 3: KHP (unknown)-Sodium hydroxide Titration

Unknown Number: _____

	Trial 1	Trial 2	Trial 3	Trial 4
Mass of Unknown sample (g)				
Initial Buret Reading (ml)				
Final Buret Reading (ml)				
Volume of NaOH added (ml)				
Moles of NaOH added				
Moles of KHP				
Mass of KHP (g)				
Percent of KHP				

Average Percent of KHP in sample _____



PC Model — A Module on Molecular Modeling for Chemistry 103.1

Objectives

- To generate computer models of several molecular compounds
- To measure bond lengths and bond angles for these compounds
- To determine their molecular shape and predict their polarity

Introduction

The chemical and physical properties of substances depend largely on the shape and the geometry of their molecules. For example, the fact that water is a liquid at room temperature, dissolves many salts and other compounds, is more dense than ice and boils at a high temperature can all be explained on the basis of its bonding and the bent arrangement of the atoms in the molecule.

Using the concepts proposed by Lewis and the principles of VSEPR theory we have learned in class to predict the shape and geometry of molecules. These concepts, although very useful, are empirical, that is, they are not based on rigorous mathematical modeling, and only provide qualitative pictures of molecules and their shapes. For more quantitative predictions of the actual bond lengths, bond angles and properties such as the polarity of a molecule we must make use of mathematical theories and models. There are different types of models that assist us in this purpose. Some are deduced entirely from fundamental properties of the atomic and molecular structure. Others are based on previous measurements and combine these with fundamental principles. We refer to the latter as "semi-empirical". They are limited to the availability of prior measurements and results that we can relate to our new system, but they are often less complex.

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Molecular mechanics is a semi-empirical approach that helps us obtain quantitative information about molecules and molecular structure. The program we will use in this exercise (PC-Model) uses molecular mechanics. In it, previously determined bond lengths and bond angles have been collected for many types of atoms. Then, two concepts are applied when attempting to determine a new structure. 1) Bond lengths and bond angles between two atoms are similar even when these bonds are found in different molecules, and 2) the overall arrangement of the atoms in a molecule will be the one that gives the lowest energy configuration.

In this exercise you will use Lewis and VSEPR theories to predict the shapes of several simple molecules. These have been made in class and in the Workshop exercise (Unit 11). Then, we will use PC-MODEL to obtain a more detailed model of the molecular shapes. The results will be compared and trends identified.

Pre-Laboratory Assignment

1. Using Lewis concepts and VSEPH theory draw the three-dimensional structure for the following molecules: CH_4, CH_3I, NH_3, CO_2 and PCl_5

   NOTE: Some of these molecules were studied in Workshop Unit 11. Use your results from the Workshop

2. Complete the following chart:

Molecule	Shape	Polar or Non-polar

PC Model – A Module on Molecular Modeling for Chem 103.1

Procedure:

To enter the PC Model Program:

- If you have: a) windows 95, Double click on the "Shortcut to PC Win" or for b) windows 3.1, Double click on the PC mod Icon
- Expand the drawing tablet by: clicking on the square at the upper right hand corner for windows 95 or the set of arrows for windows 3.1
- Drag the **Tool Bar** to the left hand side of the tablet (this will leave the tablet with lots of room for you to work).

All of our work with PC Model will begin with a central carbon atom to which one or more other atoms are bonded. Later if you need, that carbon atom can be changed to another atom.

To draw a Carbon Center, click on the **Draw** button and click again in the center of the tablet. A light blue dot will appear at the center of the tablet. We can add hydrogens to the carbon center by clicking once on the **H/AD** button located in the **Tool Bar** at the left of the tablet. A molecule similar to figure 1 should appear on the tablet.

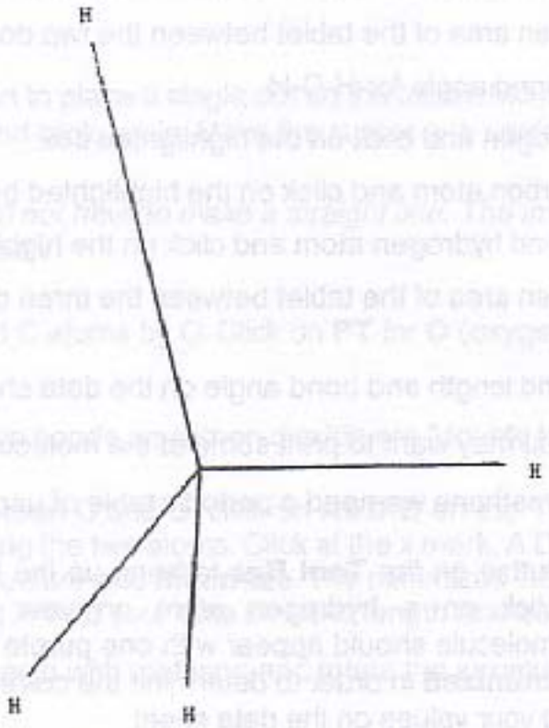

FIGURE 1 PC MODEL METHANE

The tablet is two-dimensional. The CH_4 methane molecule that you have created on the tablet is three-dimensional. We can rotate the molecule to see what it really looks like.

- To rotate the molecule, select **View** from the pull down menu and click on the **Control Panel.** Note: The **Control Panel** shows that all three axis are set to 180 deg. Click the y axis arrow to increase the **Rotate** value to 200 deg. Then exit the control panel.

At this point you should have four hydrogen atoms projecting to the corners of some regular shape. PC model will allow us to find the most stable conformation for the methane molecule.

- Pull down the **Analyze** menu and click on **Minimize**. The computer will perform a computation which will redraw the molecule. This operation will be repeated several times until the "Best" shape is found. When the iterations are finished, you may close the minimizer box. At this point you have found the best shape for the methane molecule.
- To measure the bond length and bond angle for methane click on the **Query** button.

a. To measure bond length for C-H
   1. Point to the hydrogen atom and click on the highlighted box.
   2. Point to the Central Carbon atom and click on the highlighted box.
   3. Click on an open area of the tablet between the two dots.

b. To measure the bond angle for H-C-H
   1. Point to a hydrogen and click on the highlighted box.
   2. Point to the carbon atom and click on the highlighted box.
   3. Point to a second hydrogen atom and click on the highlighted box.
   4. Click on an open area of the tablet between the three dots.

Record your data for bond length and bond angle on the data sheet provided.

If a printer is available you may want to print some of the molecules you have created.

To construct **CH_3I** from methane we need a periodic table of useful elements.

- Click the **PT** button on the **Tool Bar** to bring up the table. Select Iodine I and Click. Then click on a hydrogen atom on your methane molecule. An Iodomethane molecule should appear with one purple atom. This new molecule needs to be **Minimized** in order to determine the correct bond lengths and bond angles. Record your values on the data sheet.

To erase your molecule and clear the tablet:

- Pull down the **Edit** menu. Click on **Erase**. Answer yes to the question.

To draw an **NH_3** molecule

- Click on **PT** and bring up the periodic table of useful elements, if you do not have it already.
- Click on the **Draw** button and place a dot on the center of the screen.
- To replace the carbon atom with a nitrogen atom, click on the N button found on the periodic table. Then click again on the blue dot. Add hydrogens and the lone pair of electrons by clicking on the **H/AD** button.
- To get a complete 3-D view of the molecule use the **Control Panel** to rotate the molecule to 200 degrees on the y axis. **Minimize** the structure and **Query** to find the bond lengths and bond angles. Record your data on the data sheet. **Erase** the structure and clear the tablet.

To draw an **H_2O** molecule

1. Use the same procedure as in the NH_3 example. Replace the Carbon with an oxygen O from the periodic table.
2. The water molecule should have two sets of lone pair electrons. Rotate the structure to 200 deg on the y axis to see the lone pairs.
3. **Minimize** and **Query** your water molecule. Record your data.

To draw a **CO_2** molecule

1. Use the **Draw** button to place a single dot on the tablet. Move the cursor about 1" away from the dot and click again. Move the cursor one again and click.

   Note: the three clicks do not have to make a straight line. The image represents three C atoms bonded in a chain.

2. Replace the two end C atoms by O. Click on **PT** for **O** (oxygen). Click on the two end Carbon atoms.

3. The carbon to oxygen bonds on carbon dioxide are "double bonds".

To draw double bonds between C and O, click on **ADD-B** on the **Tool Bar** and highlight the middle of the bond joining the two atoms. Click at the x mark. A D will appear indicating a change. **Update** your structure and **Minimize**. The minimized CO_2 molecule should be linear in shape. **Query** and record your data for bond length and bond angle.

To draw a **PCl_5** molecule begin with methane and rotate the structure to 200 deg on the y axis.

1. Add an additional atom by clicking on **Draw** and then on the tablet and again on the central carbon atom.

2. Use the **Periodic Table** to substitute **Cl** for all of the hydrogens and the additional carbon atom. Substitute **P_5** for the central carbon atom.

3. **Minimize** the molecule.
4. Rotate the molecule to get a better idea of its shape. Set the control panel as X = 170 deg, y = 200 deg, and z – 250 deg.
5. **Query** and record your data for bond length and bond angles.

Note: not all P-Cl bond lengths are the same.

To draw an **HCHO** molecule begin with methane and rotate the structure to 200 deg on the y axis.

1. **Delete** one hydrogen atom and **Update** the structure.
2. Use the **Periodic Table** to substitute **O** for one hydrogen.
3. Use the Lewis structure to predict the bond order for HCHO.

Compare your prediction to the computer drawn molecule. To add bonds to the structure, click on **Add-B** on the **Tool Bar**. Then click at the intersection joining the two atoms. A **D** will appear indicating a change in bond. **Update** your structure and **Minimize**. **Query** the structure. Predict the shape and record the measured bond lengths and bond angles.

name _____ section _____ date _____

Data Sheet

Molecule	Shape	Bond lengths		Bond angles		Dipole Moment

Questions:

1. What is the effect of substituting one of the H-atoms with I in methane?

2. Compare the bond lengths and the bond angles of CH_4 and NH_3. Explain the observations.

1. Introduction to the Methods of Absorption Spectroscopy

Prelaboratory Assignment: Read about the wave and particle theories of radiation in sections 6.1, 6.2 & 6.3 of your textbook (*Chemistry* by Brown, LeMay, Burstein). Your laboratory instructor will spend no more than one-half hour reviewing this material.

Qualitative Aspects of Absorption Spectroscopy

Many compounds and solutions exhibit color. If **white light** (light that contains photons of all visible wavelengths) is passed through a solution and some of the photons are absorbed, the color of the emerging light is due to the photons that are not absorbed, and is complementary to the color of the absorbed wavelengths (Table 1). The blue color of copper solutions, for example, is due to the absorption of orange photons in the 610 nm range.

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Table 1. Colors associated with the absorption of visible wavelengths.

1 nanometer (nm) = 10^{-9} meter (m)

Absorbed Color	Absorbed Wavelength (nm)	Transmitted Color
Violet	400-450	yellowish-green
Blue	450-480	yellow
Greenish-blue	480-490	orange
Bluish-green	490-500	red
Green	500-550	purplish-red
Yellowish-green	550-570	reddish-purple
Greenish-yellow	570-575	violet
Yellow	575-590	blue
Orange	590-610	greenish-blue
Red	610-720	bluish-green

The intensity of color depends on how much light is absorbed. This, in turn, depends on the nature of the absorbing species, the concentration of the absorbing species, and the thickness of the sample through which the light passes. The more photons absorbed, the more pronounced is the resulting color.

A set of absorbed frequencies, or **absorption spectrum**, is a characteristic of the absorbing substance that can be used as a "fingerprint" for identification. Intensities of absorption can be used to determine concentration. When we recognize something by its characteristic color, such as blue copper sulfate or violet iodine vapor, or when we compare solution concentrations according to their relative depths of color, we are said to be practicing "eyeball spectroscopy"—admittedly not a very exact branch of spectroscopic science.

For obtaining quantitative information, instruments called **spectrometers** or **spectrophotometers** are used to separate the incoming radiation into its component wavelengths and to analyze the intensity of absorption in each wavelength range. Figure 1 is a schematic diagram showing the components of an absorption spectrophotometer.

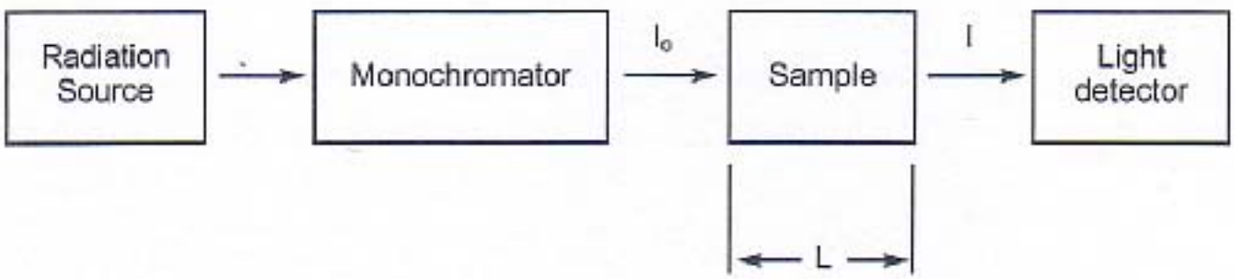


FIGURE 1. COMPONENTS OF AN ABSORPTION SPECTROMETER. LIGHT OF A SPECIFIC WAVELENGTH ENTERS A SAMPLE OF THICKNESS L. L_O IS THE ORIGINAL INTENSITY OF THE LIGHT AND I IS THE INTENSITY AFTER IT LEAVES THE SAMPLE. I IS LESS THAN L_O BECAUSE SOME OF THE LIGHT IS ABSORBED BY THE SAMPLE.

The **radiation source** (light source) emits photons of all wavelengths in the range for which the spectrophotometer is used. An incandescent tungsten lamp (similar to an ordinary light bulb) is generally used for the visible range.

The **monochromator** (wavelength selector) sends light of a single wavelength (or more precisely, a very narrow band of wavelengths) into the sample. It consists of a **dispersing element** such as a prism or diffraction grating to spread out the wavelengths emitted by the source, and a **wavelength selector** to select the wavelength that is to enter the sample.

The sample is in a **sample holder** or **cuvette**, a container that is transparent to the wavelengths used. Cuvettes used for absorption spectroscopy in the visible region are made of good optical glass, are of an exact thickness, and present perfectly flat parallel faces to the light path.

The **light detector** responds to the intensity of the emerging light with an electrical signal proportional to the rate at which is receives photons. Visible light is detected by a phototube ("electric eye"). The phototube is typically an evacuated transparent bulb containing an electrode coated with some metal such as potassium which responds to light by emitting **photoelectrons**. The emitted electrons are attracted to a positively charged plate, thus producing a current that is proportional to the original number of photons.

Quantitative Aspects of Absorption Spectroscopy

It can be shown experimentally that the **absorbance (A)** of a solution is directly proportional to the **concentration (C)** of absorbing material and to the path **length (L)**, that is,

$$A=\varepsilon CL \tag{1}$$

If concentration is expressed in g/ml and path length is given in cm, then the proportionality constant ε (Greek: epsilon) is called the **specific absorptivity**. (Note: In some books ε is called the **specific extinction coefficient**.) Equation 1, known as the **Beer-Lambert law** or simply **Beer's law**, is the fundamental equation of absorption spectroscopy. It shows that the absorbance of light increases with increasing sample concentration and path length.

If I_o is the intensity of a particular wavelength of light entering a solution, and I is the intensity of the emerging light (Figure 1), then the fraction of light passing through the solution is called the **transmittance (T)**, that is,

$$T = I/I_o \tag{2}$$

The **percent transmittance**, or percentage of light transmitted, is simply 100 x T. It can be shown that the absorbance is equal to the negative logarithm of the transmittance, that is,

$$A=\varepsilon CL = -\log(I/I_o) \tag{3}$$

Problems

1. The absorbance is a dimensionless quantity, that is, a quantity without units. What will be the units of ε, the specific absorptivity? Remember that 1 mL = 1 cm^3.
2. How does the percent transmittance of a solution vary with (a) increasing concentration and (b) increasing path length?
3. The absorbance of an iron thiocyanate solution containing 0.00500 mg Fe/mL was reported as 0.4900 at 540 nm.
   a. Calculate the specific absorptivity, including units, of iron thiocyanate on the assumption that a 1.00 cm cuvette was used.
   b. What will be the absorbance if (1) the solution is diluted to twice its original volume and (2) the solution is placed into a 5.00 cm cuvette?
4. Refer to Problem 3.
   a. What percent of light is transmitted by the original iron thiocyanate solution?
   b. What concentration of iron thiocyanate will absorb 50.0% of the entering light?
5. The concentration of yeast t-RNA in an aqueous solution is 10.0 µg/mL. The absorbance is found to be 0.209 when this solution is placed in a 1.00 cm cuvette and 258 nm radiation is passed through it.
   a. Calculate the specific absorptivity, including units, of yeast t-RNA.
   b. What would be the absorbance if the solution is diluted to 5.00µg/mL?
   c. What would be the absorbance if the path length of the original solution is increased to 5.00 cm?
6. The following absorbances were reported for solutions of cobalt(II) ion at 515 nm:

Concentration (g/100mL)	Absorbance
0.4418	0.4134
0.3542	0.3251
0.2181	0.2027
0.0879	0.0820
0.0532	0.0491
0.0351	0.0315

Prepare a graph, using the CBL Graphing Program, plotting concentration along the x-axis and absorbance along the y-axis. Refer to the Appendix to the graphing experiment (Week 2) for entering your data into the CBL Program. Use the instructions to find the slope and y-intercept of your graph for use in Problem 7.

7. A 1.0631 g sample of an unknown compound containing cobalt(II) ion was dissolved in water in a 100 mL volumetric flask. The absorbance of this solution was found to be 0.2195 at 515 nm.
   a. Use your graph from Problem 6 to find the concentration of cobalt(II) ion in the solution.
   b. Calculate the percentage of cobalt in the unknown compound.

Spectrophotometric Determination of Iron in Vitamin Tablets

Purpose: To determine a trace concentration of metal ion in a commercial vitamin pill by Spectrophotometric analysis.

The chemistry of the analysis involves two reaction steps:

1. The Fe^{2+} solution is converted into a highly colored complex with 1,10-phenanthroline

   The $Fe^{2+} + 3(C_{12}H_8N_2) \rightarrow Fe(C_{12}H_8N_2)_3^{2+}$

   lt green no color orange-red

2. The oxidation state of iron is changed from +2 to +3 by the addition of hydroxylamine hydrochloride to the solution to be analyzed.

The colored solution of the complex will absorb a maximum of light at 508 nm and should have a pH of about 3.5 to prevent the precipitation of various iron salts from the solution. Therefore the analysis system employs a saturated sodium acetate buffer to maintain the pH at a viable level.

Equipment needed: 6-100 mL volumetric flasks with covers, 2-10 mL transfer pipets, 1-5 mL transfer pipet, 1-2 mL transfer pipet, 1 funnel, 100 glass beaker, 4 plastic beakers and a wash bottle, 2 cuvets.

Note before you begin work with the solutions provided for you, rinse all of the glassware several times with deionized water. In addition designate and label each of the pipets for a different task. This will help eliminate contamination of solutions and make for a more meaningful experiment. The plastic beakers have been provided so that you can collect the solutions required for the experiment and not pipet from the stock bottles.

Suggested rough quantities of reagents:

sodium acetate 80 mL; all other solutions 35 mL

Use of the Spectrophotometer

A spectrophotometer is an instrument that measures the color intensity of a solution. A typical Spectronic 20 diagram is shown in appendix IV.

There are three things the experimenter must watch if precise measurements are to be obtained (1) that the instrument gives proper readings at each end of the scale, (2) that the solutions measured are not contaminated or diluted, (3) that the outside of the cell is spotlessly clean and dry.

1. Turn on the instrument and adjust the wavelength know to 508 nm. Allow the instrument to warm-up for 15 minutes before taking measurements.

2. Rinse the cuvet 4-5 times with deionized water. Fill it $^1/_2$ full with water. Holding the cell at its top, wipe the entire outside dry with a clean towel. Check the outside for smudges and liquid. If any exist, remove them. From now on handle the cell only at its top.

3. With no cuvet in the instrument set the reading on the meter at exactly 0%T using the left-hand knob on the front of the instrument.

4. Insert the water cuvet into the cell compartment so that the mark on the cuvet lines-up with the mark on the compartment. Close the compartment lid. Using the right-hand knob on the front of the instrument set the reading on the instrument to 100%T (A=0).

5. Repeat steps 3 and 4 until proper readings are obtained at both 0%T and 100%T.

6. Designate one cuvet for measurement of a blank solution and the other for the analyte.

Preparation of the standard Complex solutions for the Calibration Curve.

Using a 10 mL pipet designated for iron, transfer 10.0 mL of the stock iron (II) solution into a 100 mL volumetric flask. The stock solution concentration is 40.0 mg F^{2+}/IL. Add 10 mL of saturated sodium acetate solution, 2 mL of hydroxylamine hydrochloride and finally 3 mL of 1,10 phenanthroline to the same volumetric flask. Dilute to the mark with deionized water and mix well. Label this solution A.

To prepare three additional standards deliver 5.0, 2.0 and 1.0 mL of the stock iron (II) solution into three separate 100 mL volumetric flasks. Add the buffer, reducing agent and phenanthroline as you did to make solution A. Dilute the three solutions to the mark with deionized water and mix well.

Allow at least 15 minutes after adding the reagents before making absorbance measurements. This gives time for the color of the complex to develop fully.

While you are waiting, prepare a blank solution by putting 2 ml of hydroxylamine hydrochloride, 3 mL of 1.10-phenanthroline and 10 mL of sodium acetate into a fifth 100 mL volumetric flask.

Dilute to the mark and mix well.

Measure the absorbance of your prepared complexed iron (II) solutions against the blank at 508 nm. Record the absorbance values on your data sheet.

Preparation of the Calibration Curve Using Beer's Law (A=abC)

Use CBL Graph to plot the absorbance reading on the y axis versus the concentration of iron (II) in mg/L on the x axis. This plot should be linear. We will use the regression routine in the program to find the best straight line going through the four data points. Record the slope of the line (M), the y intercept (b), and the correlation coefficient (cor) on your data sheet. Have your instructor initial your data sheet.

Determination of the Iron Content of the Vitamin Pill

Place one vitamin pill in a 100 mL beaker. Add 25 mL of 6M HCl and boil gently in the fume hood for about 15 minutes. Add 10 ml deionized water and filter the solution while warm through #40 filter paper directly into a 100mL volumetric flask. Wash the filter paper containing any residue with two or three 5 mL portions of deionized water adding these washings to the mark with deionized water. Label this solution A.

Pipet a 10.0 mL aliquot of A into a clean 100 mL volumetric flask and dilute to the mark. Label this solution B.

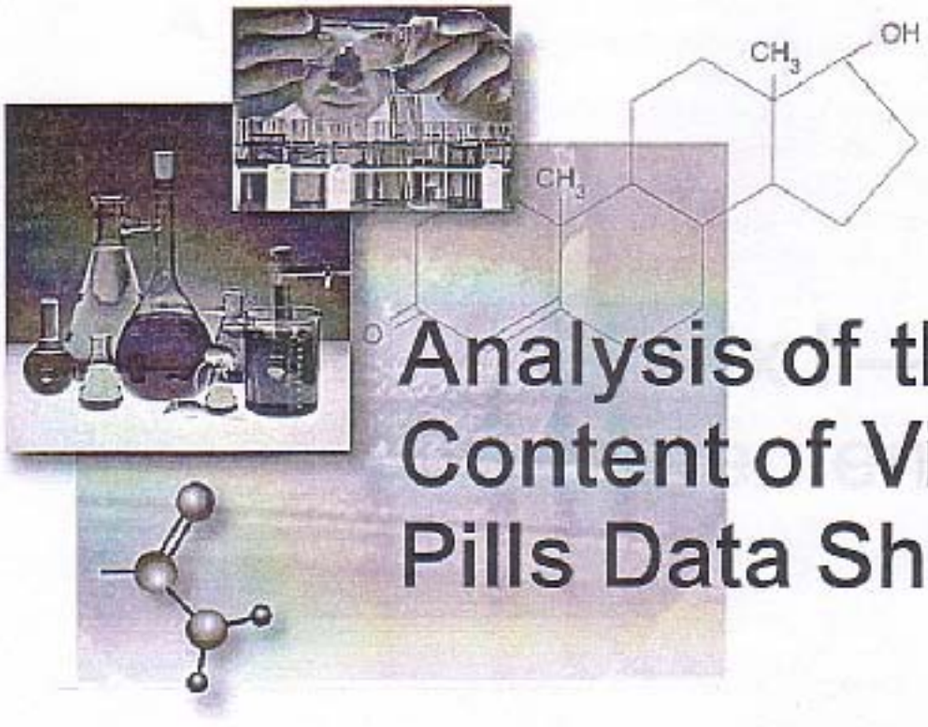
Finally prepare the unknown by pipetting 10.0 mL of solution B into a 100 mL volumetric flask, adding 10 ml saturated sodium acetate, 2 mL hydroxylamine hydrochloride and 3 mL of 1,10-phenanthroline. Dilute to the mark and label as C.

Wait 15 minutes and determine the absorbance of solution C. Record this value on your data sheet.

Discard solutions B and C. Repeat your analysis with a new aliquot from solution A. Precision for this analysis should be to +/-1.5 mg/Fe^{2+} per pill.

Calculation of the Mass of Iron (II) in the Vitamin Tablet.

Using the relationship y=mX + b, calculate the concentration of Fe^{2+} as mg/L for solution C from the absorbance value. Refer to your data sheet to obtain the slope, and y intercept of your calibration curve. Record this value on your data sheet. Calculate the number of mg of Iron (II) per tablet.



Analysis of the Iron Content of Vitamin Pills Data Sheet

I. Calibration Curve Data

Solution #	Concentration, mg/L Fe^{2+}	Absorbance
10.0 mL		
5.0 mL		
2.0 mL		
1.0 mL		

Slope of Best Line _____ Y intercept _____ Correlation Coefficient _____

Instructor's initial _____

II. Determination of the Mass of Iron (II) in the Vitamin Pill

	Trial 1	Trial 2
Concentration of Iron (II) in solution C, mg/L		
Concentration of Iron (II) in solution B, mg/L		
mg Iron (II) per tablet		

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Appendix I — Volume Measurements

You will often need to measure the volumes of liquids in your laboratory work. Several measuring vessels are available and the choice of which to use will depend on the required accuracy of the measurement.

Beakers and Erlenmeyer flasks. Some of these have approximate volume markings that can be used when rough estimates of volume are acceptable.

Graduated cylinders. These are used if reasonable accuracy is important. A 10-mL graduated cylinder has 1-mL graduation marks; these are numbered. Each 1 mL is divided into five small divisions of 0.2 mL each; these are not numbered. The position of the bottom of the liquid meniscus gives the volume reading. In Figure 1 the meniscus is between the 7.6 and 7.8 mL marks; the volume is more than 7.6 mL but less than 7.8 mL. Mentally divide the distance between the marks into smaller divisions and estimate where the meniscus lies along this scale. Because the 0.2-mL marks are so close together you will probably find it possible to divide the distance only into two halves rather than into smaller division such as tenths; that is, you will probably be able to distinguish a distance that is about halfway between the two marks but not a smaller distance. Volumes from about 7.65 mL to 7.75 mL would be indistinguishable and the meniscus would look like it was about halfway between 7.6 mL and 7.8 mL; your reading would therefore be 7.7 mL. Since any true volume from 7.65 mL to 7.75 mL would look the same to you, there is an uncertainty of 0.1 mL in the volume reading of 7.7 mL, as possible error that cannot be eliminated even with practice, for it arises from the limitations of your eyes and of the equipment.

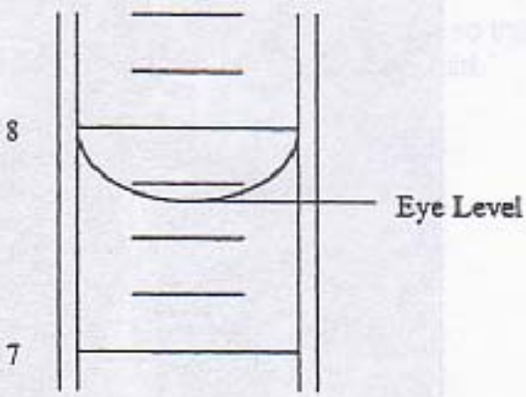

FIGURE 1 MEASURING VOLUME WITH A GRADUATED CYLINDER

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Volumetric Glassware. This term includes pipets, burets and volumetric flasks. These are constructed and calibrated to be much more accurate than graduated cylinders and are the most accurate glassware you will use to measure volumes.

Fast Release Pipette Pump II

To insert pipette:

1. Depress the plunger.
2. Insert the proper pipette into the chuck.

To pipette:

1. Rotate the Thumbwheel counter clockwise, the liquid will rise in the pipette.
2. Stop when the required amount is reached.
3. To release the liquid drop by drop, turn the thumbwheel slowly in the opposite direction.
4. For fast release of the liquid, press the side lever until the level reaches the lowest marking on the pipette.

Burets. A Telfon stopcock never needs to be greased. To Clean the buret makeup a dilute soap solution and coat the inside of the buret. From a beaker rinse out the soap with a large amount of tap water. Finally, rinse the buret with deionized water from a plastic bottle. Clamp the buret in place, close the stopcock, and fill the buret with solution. Open the stopcock and drain solution from the buret until the tip of the buret is filled and does not contain an air bubble. If necessary add more solution so that the buret is filled to just below the 0.00 mark.

Each graduation mark on the buret corresponds to 0.1 mL. Estimate the fractional distance between two marks to obtain a reading to the nearest 0.02 mL. The position of the meniscus in Figure 3 is 18.37 mL. In your readings it is important to avoid errors that result from viewing the meniscus from various angles. Note that the meniscus seems to change position depending on whether you look at it from above or below (again, see Figure 3). To make an accurate reading the eye must be level with the meniscus. To ensure this, look at the buret so that the front of the mL graduation mark nearest the meniscus is superimposed on the back of this same mark and your eyes see only a single line; read the meniscus level from this position.

When you begin to use a buret you might find it difficult or awkward to master the proper technique. However, it will be worthwhile to exert the effort required to use a buret properly. If you are right-handed operate the stopcock with your left hand (see Figure 4A); grasp the stopcock's handle between your thumb on the front of it and your first and second fingers on the back of it. With your right hand, continuously swirl clockwise the flask into which the solution flows. If you are left-handed, operate the stopcock with the thumb and first fingers of your right hand (see Fig. 4b); swirl the flask counterclockwise with your left hand.

Before beginning a titration, touch the buret tip against the inside of a waste beaker to remove the drop of liquid that might be clinging to it.

After running solution out of a buret, wait a minute before making a volume reading so that the liquid remaining on the upper wall has time to drain down into the bulk of the liquid.

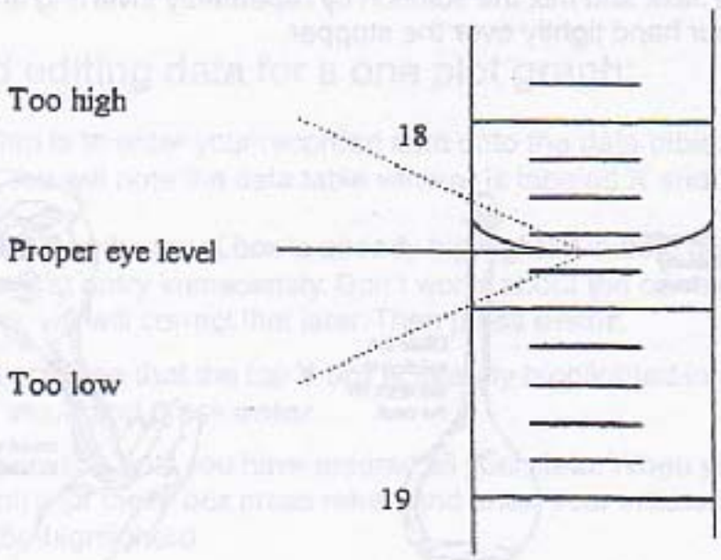


FIGURE 3 READING A BURET

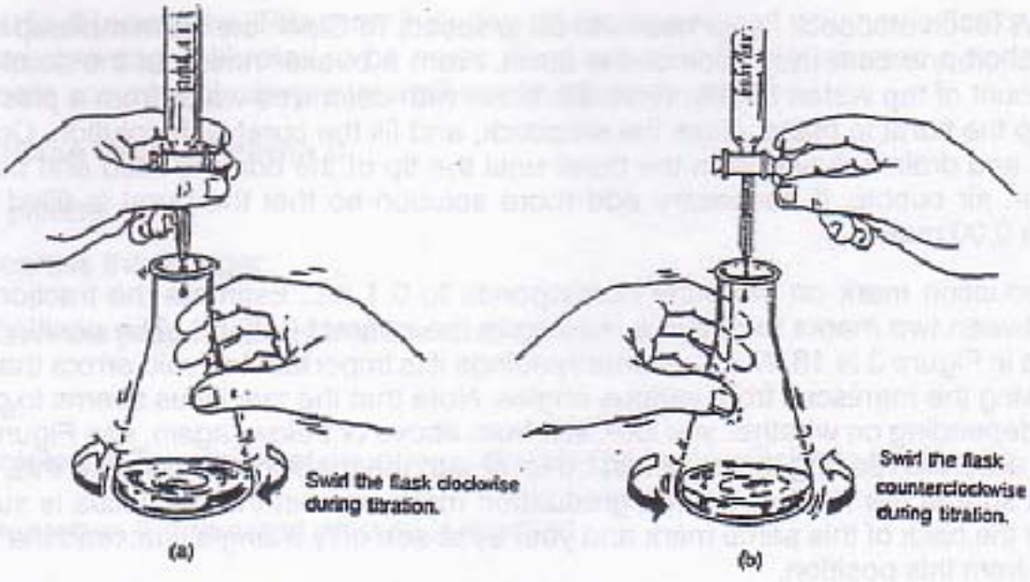


FIGURE 4 PROPER BURET TECHNIQUE

Volumetric flasks. Volumetric flasks (see Figure 5) are calibrated to contain a specified volume; they are used to prepare solutions of accurately known molarity (moles of solute per liter of solution). For example, 0.100 mole of NaCl is put into a 1.00-liter volumetric flask and the flask is filled with water to the calibration mark giving 1.00 liter of solution; the concentration is 0.100M. The solid that is to be dissolved must be very carefully weighted, but it is not necessary to measure the amount of solvent that is used; instead, the final volume of solution is accurately known. After you put the solid into the flask, use a little of the solvent to wash out the container in which the solid was weighed and add the washings to the flask; be sure that none of the solid is lost. Add a little more solvent to the flask and swirl the flask to dissolve as much of the solid as possible before the flask is filled with solvent. Then add more solvent until the bottom of the meniscus just touches the calibration line; it is helpful to use a medicine dropper or wash bottle to add the last few milliliters. Stopper the flask and mix the solution by repeatedly inverting and uprighting the flask while holding your hand tightly over the stopper.

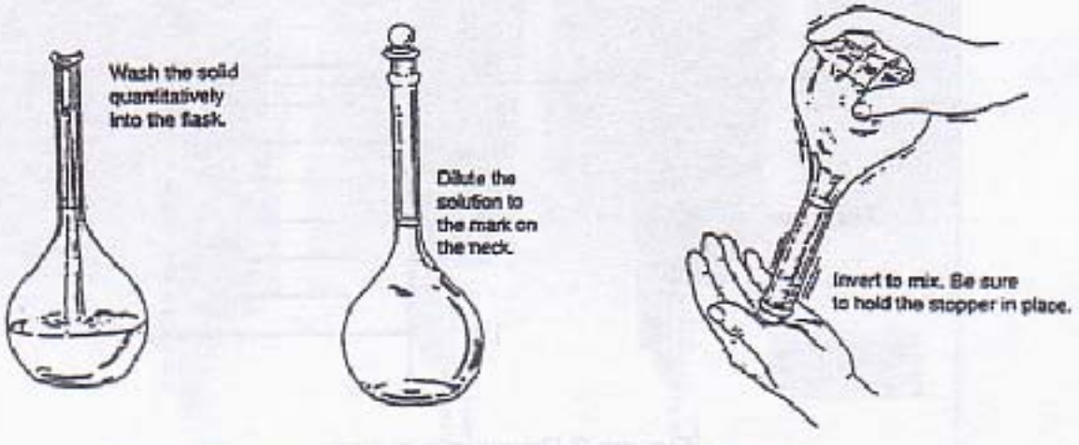
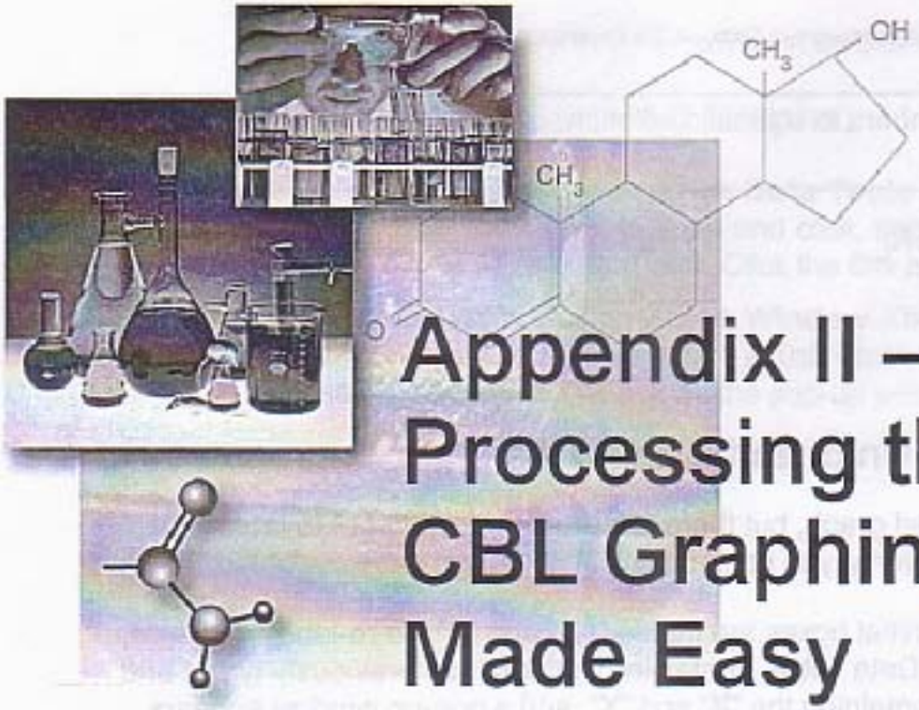


FIGURE 5 MAKING UP A SOLUTION USING A VOLUMETRIC FLASK



Appendix II — Processing the Data— CBL Graphing Made Easy

- The CBL Graphing program is designed to help you gain a basic understanding of simple graphing techniques with the use of a computer based program. This program is designed to help you with more accurate graphing and most importantly time conservation.
- Firstly, double click the CBL Graphing Icon on the monitor to get into the application, then click OK to begin. When the program starts, you will see a Data Table window on the left of the screen and a Graph window to the right of the screen. If you do not see these windows, ask for help from your workshop leader. If you do see windows, begin.

Entering and editing data for a one plot graph:

A. The first step is to enter your recorded data onto the data table of the graphing program. (You will note the data table window is labeled **X** and **Y**.)

B. You will note that the top X box is already highlighted in bold gray. You may begin typing your first entry immediately. Don't worry about the correct number of significant figures, we will correct that later. Then press **enter**.

   - You will now see that the top **Y** box is already highlighted in bold gray. Type your first "**Y**" value and press **enter**.

C. Follow this routine until you have entered all your data. When you have entered the final entry for the **Y** box press return and enter your initials in the final **X** box which will be highlighted.

D. As you enter the "**X**" and "**Y**" values, the program will automatically graph the data for you.

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Note: If you are entering numbers in scientific notation, use the "**E**" or "**e**" key for the exponent.

Ex>	TO ENTER:	TYPE THIS:
	3.5×10^4	3.5 E4 or 3.5 e^4
	-3.5×10^4	-3.5 E4 or 3.6 e^4

Editing the Axis labels, Units and Graph Title:

A. You now have a completed graph, but there is a problem, you forgot to label your Axis on the graph. Don't worry, you can go back and change it.

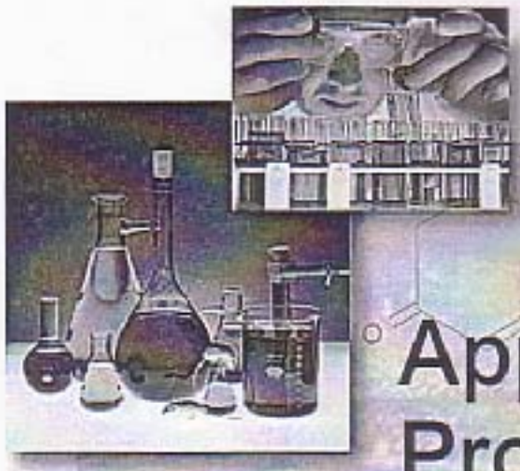
- Remember that your initial boxes are labeled "**X**" and "**Y**". To re-label your Axis, simply go back to the Data Table, containing the data you previously typed and double click the box containing the "**X**" and "**Y**", and a pop-up window appears.

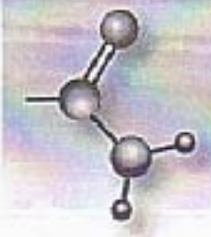
B. To make these changes, click your mouse in "**New Names**". Delete "**X**" or "**Y**" and enter your title in "**New Name**" box. Click in "**New Units**" and enter the appropriate units.

- Go to "**Rounding**" and click on "**decimal places**" box. Delete the number then enter the correct decimal places.
- Click in circle next to "**Significant Figures**". Once the box appears, click in the box, delete the number and enter the correct number of significant figures.
- Now is a good time to look at your changes before you proceed. All of these changes can be done in the same pop-up window. (Do not press **enter/return** until absolutely all changes have been made or you will have to click "**X**" or "**Y**" cell to get the pop-up window again.)
- At this time you may also change your "**point protector**", the symbol that represents your data to which ever symbol you want. (To actually change the color of the point protector and the symbol all changes have to be done when changing the "**Y**" Axis label.)
- Once you are satisfied with the changes, click the box that says **OK**. Your screen will return to the data table and graph with the changes you have just made.
- To change the title of your graph, simply click the title of the graph window. You will see the graph title highlighted. Press "**delete**" and enter the change along with your initials and press **return**. (Be sure at this point to personalize your graph with your initials to avoid confusion before printing!)
- Congratulations, you have made your first graph!

Printing your Data Tables and Graphs:

- To print the data table, click on the **Data Table Window**. The title will be highlighted in blue. Go the **File** menu and click, select **Print**. Choose **Data Table** from the small blue box and click. Click the **OK** box on the pop-up window.
- To print your graph, click on **Graph Window**. The title will be highlighted in blue. Go to **File** menu and Click. Select **Print**. From the blue box choose **Selected Display**. Click on the **OK** box on the pop-up window.





Appendix III — Processing a graph for a "Best-Fit" linear regression line using the CBL Graph Program

This program option causes the "best-fit" linear regression line to be drawn through all or part of the data. The line is calculated using the method of least squares.

1. See appendix 2 for entering and editing data. Stop at the congratulations salutation.

2. To proceed, you may remove the connecting lines on your graph by highlighting the graph window in blue. Go to the word **Graph** at the top of the screen and highlight it. A pull down menu will appear. Drag the mouse down to "**Connecting Lines**" and click. (Notice the lines have disappeared.)

3. Highlight the Data Table Window in blue. Next click on the grey area of the Data Table window above the "X" to automatically highlight all of the X values on the Table. Repeat this operation for the Y values also.

4. Go back to the Graph Window and highlight it in blue. At the top of the screen highlight the word **Analyze** and click on it. A pull down menu will appear. Drag the mouse down to the word "**regression**" and click on it. A small Regression box will appear on the right side of your graph window. The letters in the box indicate: M=slope of the line; B=y intercept; cor=correlation coefficient.

5. Click on the blue portion of the regression box and drag the box towards the upper left on the graph window. This will provide you with an uninterrupted view of your graph and fit line. (Notice two dark lines on either side of your graph window. These lines indicate that you have properly highlighted all of you data points.)

6. Print your results as indicated in appendix II.

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Appendix IV — The Use of Analog Spectronic 20 and Digital Spectronic 20D Spectrophotometers

Sample Measurement-Spectronic 20

A. Turn on the Spectronic 20 by turning the power switch/zero control (knob on the left side of instrument) clockwise. Allow the instrument to warm up at least 15 minutes to stabilize the source and detector.

B. After the warm-up period, set to desired wavelength with the wavelength control knob.

C. Adjust the meter to 0%T with the power switch/zero control (knob on left hand side of the instrument). Make sure the sample compartment is empty and the cover is closed.

D. Fill a clean cuvet with water (or another blank solution) and wipe the cuvet with a towel to remove liquid droplets, dust, and fingerprints. Place the cuvet in the sample compartment and close the lid. Your lab instructor will demonstrate this procedure for the type of cuvet which is used for your experiment. Adjust the meter to 100%T with the transmittance/absorbance control (knob on right side of instrument). Remove the cell from the sample compartment.

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E. Fill another cuvet with the solution(s) you wish to measure and insert the cuvet(s) into the sample compartment. Close the lid as before. Read the appropriate value (%T or A) from the meter.

F. Remove the cuvet from the sample compartment and repeat your measurements for other solutions.

G. When all measurements are completed, turn off the Spectronic 20 by turning the power switch/zero control counterclockwise until it clicks.

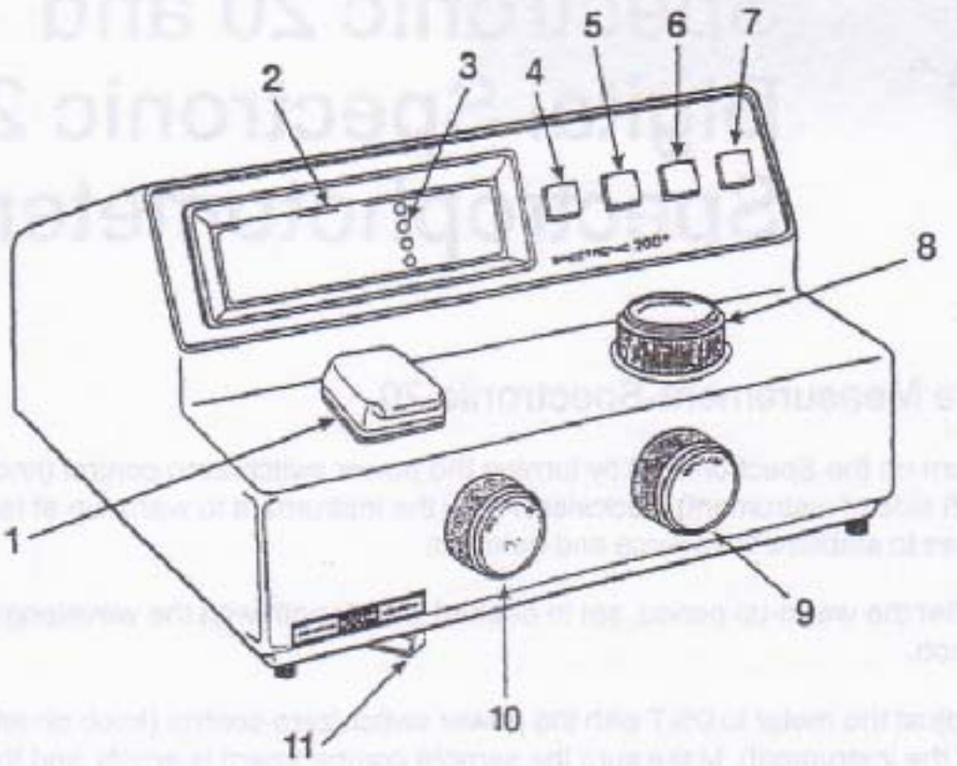

FIGURE 1 SPECTRONIC 20D* DIGITAL SPECTROPHOTOMETER

1. Sample compartment
2. Digital readout
3. Mode indicators
4. Mode selection
5. Decrease
6. Increase
7. Print
8. Wavelength control
9. Transmittance/ Absorbance control (100%T/0A
10. Power switch/Zero Control
11. Filter lever

Figures 1 shows a diagram of the one type of instrument used in our General Chemistry Labs.

Note: To read the meter properly, align the needle with its reflection in the mirror.

Sample Measurement: Spectronic 20 D+

A. Turn on the Spectronic 20D+ by turning the power switch control (knob on left side of instrument) clockwise. Allow the instrument to warm-up for at least 15 minutes to stabilize.

B. After the warm-up period, set the desired wavelength with the wavelength control knob.

C. Set the filter lever to the appropriate position for the selected wavelength.

D. Adjust the display to 0%T with the zero control (knob on the front left side of the instrument). Make sure that the sample compartment is empty and the cover is closed.

E. Set display mode to transmittance or absorbance by pressing the mode control key until the appropriate LED is lit.

F. Fill a clean cuvet with water (or another blank solution) and wipe the cuvet with a towel to remove liquid droplets, dust and fingerprints.

G. Place the cuvet in the cell holder and then into the compartment and close the lid. Your instructor will show you how to place the cuvet into the instrument.

H. Adjust the display to 100%T or 0.0A with the transmittance/absorbance control (knob on the right side of the instrument).

I. Remove the cuvet from the instrument. Fill other cuvets with samples to be measured.

J. Place your samples into the instrument one at a time. Read the appropriate value (%T or A) from the display.