The City College of New York
Department of Chemistry

Chemistry 10301 Laboratory Manual

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Grading Scheme

	Total	**Week 1**	**Week 2**	**Week 3**
Density/Graphing	**15%**	**7%**	**8%**	**-**
Empiric Formula of a Salt	**15%**	**5%**	**10%**	**-**
Titration	**25%**	**4%**	**4%**	**17%**
Calorimetry	**15%**	**7%**	**8%**	
Spectroscopy	**23%**	**3%**	**8%**	**12%**
PC Model	**7%**	**7%**	**-**	**-**

Safety Rules in General Chemistry Labs

Work in a chemical laboratory has its specifics and requirements. The rules that apply to every place where chemical work is carried out are drawn from the long history of scientific achievements and failures of the chemical guild in centuries of experience. The general safety rules are abided by in every chemical laboratory and are not a subject of choice or questioning.
If you break any of safety rules, your instructor will ask you to leave the laboratory area.

Do not eat or drink in a chemical laboratory
Before entering the lab, be sure you have finished with your snack and put any food and drink (bottles, cans, food containers, candy) safely in your bag or backpack.

Dress appropriately
Do not wear sandals or contact lenses and leave your best clothes at home. Long pants are preferable to shorts or short skirts. Even if you are not clumsy, someone else in the lab may be. Protect yourself and avoid ruining your clothes.
Tie back long hair. You don't want your long hair to fall accidentally into chemicals or flames.

Wear safety goggles at all times when in the laboratory
You will be provided with safety goggles and you must wear them every time you have a lab class.

Know what you are working with
In each laboratory there will be Materials Safety Data Sheets folder. It contains description of all chemicals used in the course of the lab work throughout the semester. You are encouraged to read about chemicals you use before you start working with them.

Don't taste or sniff chemicals
Even the safest chemical compounds may be dangerous. You don't know how pure they are and what they may be contaminated with.
Our goal is to minimize the probability of an accident. That is why you will be working with diluted water solutions and relatively safe solids. If a drop of any solution drops on your skin, don't panic. Just go to the sink and wash the place with water.

Listen to the instructions of your lab instructor
Do not improvise. Your instructor will show you the most efficient and safe way of carrying out your lab experiment. Be prepared for the lab class, know what to expect. Do your homework. That will help you work safely.

Don not casually dispose of chemicals down the drain
Some chemicals can be washed down the drain, but others will be collected in a designated chemical waste bottle. You will receive instructions on how to dispose of chemicals – follow them carefully. If a chemical can go down the drain, be sure to wash it away, rather than risk an unwanted chemical reaction between chemical "leftovers" later.

Know where safety equipment is
Each laboratory is equipped with eye washing station and fire extinguisher. A very common hazard is broken glass. Always use brush and dust pan to collect the pieces.

Use of Laboratory Equipment

For most of you, General Chemistry labs will be the first encounter with experimental chemistry. You will be instructed on how to handle every piece of equipment and glassware. All experiments are described step by step in this manual and while preparing for the lab classes, you can obtain a general idea about what will be the new items you will be working with and how they are used. **Electronic balances** are very commonly used in laboratory practice. There are different kinds of electronic balances. Almost every week during Chemistry 10301 lab classes you will be using simple top loading balances. Here are a few rules:

- Balances are the most expensive pieces of equipment in the General Chemistry lab area; be gentle when using them;
- Each balance has a maximum weight it can measure. If on the display you see any symbol different from digits, quickly remove the sample you are weighing. Most likely, you have overloaded the instrument.
- Never weigh wet glassware. Balances are very sensitive and they can register even the evaporation of water, therefore preventing you from obtaining the exact weight.
- When finished with weighing your sample, make sure you remove the container from the weighing pan. Never leave the balance loaded.
- Make sure the substance you are weighing goes to the container you are using. Don't spill anything on the weighing pan – that will give you an error that usually multiplies with the calculation. Whenever possible, weigh your solids by difference (you will be instructed how to do that).

Different **Glassware** will be used almost every week. Handle the items with care; don't play with them. They can easily break and hurt you. Additionally, some of the items are very expensive and you may be asked to pay for them (the lab fee you have probably paid covers only a small fraction of the price). Wait for the instructor to demonstrate how every piece of glassware is handled.

Working with Experimental Data

The whole idea of carrying out an experiment is to produce a set of data which then can be used to prove something or calculate a target value.
Each means of measurement (instrument or glassware) has its limit of precision. It can only provide you with a value with certain number of digits and decimal places. You will work with the numbers to calculate your final result. It is very important to determine correctly the number of decimal places or significant figures you can retain. Your grade will depend on that to a certain extent.

In general, when we add or subtract numbers, we talk of decimal places. The rule is: we retain the number of decimal places, corresponding to those of the least precise number. For example, in the following example combining different amount of a salt, using a calculator, you will get an answer with three digits after the decimal point:

2.87 g + 231.9 g – 35.476g = 199.294g

But according to the rule of significant figures, the correct number will be 199.3, because the least precise value is 231.9 (only one decimal place). Therefore, your final result should be rounded off to one decimal place too.

When multiplying or dividing, the value with the least number of significant figures determines the final result. For example – when we calculate density, we divide the mass of a specimen by its volume:

35.207 g/8.5 ml = 4.1 g/ml

In this case we are allowed to use only 2 significant figures for the density, because the volume (8.5ml) only has two significant figures.

Zero is a significant figure only when it doesn't determine the decimal place. For example:

20.5ml; 3.400ml – here zeroes are significant, unlike 0.004m, where we only have one significant figure.

Presenting Data Graphically

Many times data that are collected from an experiment are presented in a graph in order to be easier comprehended by the viewer. Many of the experiments in this lab course will require plotting a graph in order to obtain data, needed for the further calculations. When drawing a graph, attention should be paid to the following:

1. Graph should have a title.
2. Axes should be labeled, including the units.
3. The scale of axes should guarantee maximum resolution. The graph should fill the page. An axis doesn't necessarily start with zero.
4. Data points are marked with little circles or crosses, but never with a single dot. They should be clearly visible.
5. If any calculations are shown, they shouldn't obstruct the graphing part.

Density of Solids 1

Calculating of the Average Density by Measuring of Mass and Volume of Samples

The density of an object is one of its most fundamental and useful characteristics. It is independent of the quantity of material measured since it is the ratio of the mass of an object to its volume. The density of an object can be determined by a variety of methods. In this experiment we will determine the density of a metal of known composition and a specimen of unknown composition. For each sample we will measure the density directly and graphically and compare the two methods.

What You Will Use for the First Time

1. Measuring Cylinder.

These are used if reasonable accuracy is important. Cylinders for different volumes are graduated differently. What is common for all of them and also for any type of graduated glassware is that you determine volume by registering position of the bottom of the meniscus. When doing that, you need to make sure that you maintain a proper eye level. Holding the cylinder too high or too low will change the reading you obtain. Fig. 1 shows the proper position of the eye for a correct reading.

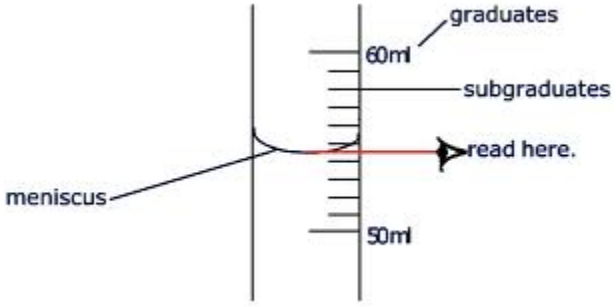

Fig. 1

In this experiment we will use 25mL cylinders. In Fig. 2, you can see how your cylinders are graduated. What is the volume of the liquid on Fig. 2?

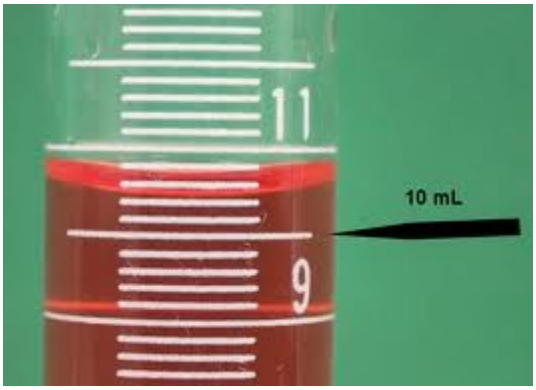

Fig. 2

2. Laboratory Balance.

You will use top loading electronic balances. Your instructor will show you how to use them properly.

Procedure

You will work with 6 aluminum samples and 6 samples of unknown material. In order to calculate density, you will need to find out the mass and volume of each sample.
Weigh each specimen carefully and record the data in the data table.
Determine the volume of each sample by the water displacement method:

1. Fill the 25mL cylinder with enough water to cover the object you are measuring the volume of (usually you need about 10mL water).
2. Read the exact volume of water.
3. Tilt the cylinder and carefully submerge the sample by sliding it down the side (you don't want to splash water out or to break the cylinder).
4. Read the volume of water with the submerged sample.
5. Find the volume of the specimen by calculating the difference in the two volumes. Record the number in the proper place in the data table.
6. Repeat steps 1 to 5 for all 12 samples.

Now you can calculate the density for each of your samples. If you have worked correctly, you should get close numbers. Calculate the average density for aluminum and unknown material and record them in the data table.

Data Sheet
Density of Solids 1

Name:_____ **Section:**_____

Aluminum				Unknown			
#	Mass	Volume	Density	#	Mass	Volume	Density
1				1			
2				2			
3				3			
4				4			
5				5			
6				6			
Average Density				Average Density			

Calculations:

Density of Solids 2

Manual and Computer Graphing of the Density Data

Now you will work with the data for density you obtained last week. To determine density graphically, you will need to draw a graph using the numbers for volume and mass of each sample. You will draw graphs both by hand and on a computer, using simple graphing software.

What You Will Use for the First Time

CBL Graphing Software

When you start the program, you will see a Data Table window, Graph window and a Text window.

1. To enter your data in the data table, just type in the proper numbers for the x- and y-axes. Use the arrow keys or the Enter key to move to another box.
2. In your data table, your initial boxes are labeled **X** and **Y**. To re-label X axis properly, double click in **X** box in the Data Table. A window will open that allows you to enter **New Name**, **New Units**, **decimal places** and **significant figures**. You can label the Y-axis in a similar way, by double clicking in Y box.
3. To draw the "best fit" linear regression line, first you need to highlight both the data table and graph window. You can do that by clicking a little under the first point in the graph window and dragging the mouse to just above the last point.
4. In the top menu click on **Analyze**.
5. In the pull down menu click on **Regression**. The "best fit" line appears, together with a regression window. In the regression window "M" stands for slope and B – for Y-axis intercept. COR is the correlation coefficient.
6. To remove connecting lines, you need the graph window highlighted. In the top menu click on **Graph**. In the pull down menu **Connecting Lines** is checked. Click on **Connecting Lines** to uncheck.
7. To print – go to **File**; then click **Print.** A small window appears – choose **Print the Whole Screen**.

Procedure

Discuss with your instructor which data will be plotted on the x- and y-axes and how you can calculate the density from a hand graph.

Draw separate graphs for aluminum and unknown material on the graph paper provided using a ruler and a pencil.
Don’t forget that a scientific graph must contain certain features (see **Graphs** section in the beginning of the manual).
When you use CBL Graph, the computer will do all calculations for you. You only will have to write what your instructor requires in the text window and then print the graph out.

Determination of the Formula of an Unknown Salt 1

Discussion on the Limited Reagent Concept and Practice with Laboratory Equipment

This laboratory experiment introduces a technique for determining the empirical formula of simple, relatively insoluble salts. It is based on the concept of the limiting reagent. In order to carry out the experiment successfully, you need to have firm knowledge of what a mole of a substance is and the relation of moles to volume and concentration (molarity).

Each chemical compound always consists of the same elements that bond with each other, always in the same relative ratios. For instance a molecule of water is composed of 2 Hydrogen atoms and 1 Oxygen atom. The ratio is: H : O = 2 : 1. If this ratio is somehow changed, the result will be a compound different from water.

Let's assume we can place different amounts of H and O atoms in six separate vessels and they can bond together forming water. If we keep the total number of atoms in a vessel constant (let's say 11), it will be possible to count how many H_2O molecules can be formed:

Vessel #	1	2	3	4	5	6
H atoms	**2**	**4**	**6**	**8**	**10**	**11**
O atoms	*9*	*7*	*5*	*3*	*1*	*0*
Total atoms	11	11	11	11	11	11
H_2O molecules	**1**	**2**	**3**	**3**	**1**	**0**

In vessel 1 we only have 2 H atoms but 9 O atoms. This results in only one water molecule because only one of all 9 O atoms consumes the 2 available H atoms. The remaining 8 Oxygen atoms cannot produce water (because of lack of Hydrogen). In this case, Hydrogen is the limiting reagent. Its amount limits the number of produced water molecules to only one. With gradual increase in the number of H atoms in each of the following vessels and decreasing O atoms, the yield of water molecules gradually goes up until we get to vessel 4. Here only 3 water molecules are formed, because only 3 O atoms

are available. They connect to 6 H atoms and there are 2 H atoms unused. Here and until the end of our "experiment" Oxygen is the limiting reagent.
If we plot a graph with number of H atoms on x–axis and number of created H_2O molecules on y–axis and then draw the "best fit" lines, we will be able to calculate the X-value, corresponding to the point of intersection. At that point, the x- and y-axes values for both legs of the graph are the same. From regression equations:

0.5x = -x+11

That means that at the point of the highest yield, we have x = 7.33 H atoms consumed. Since it is a given that we maintain a constant number of 11 atoms for each H and O combination, the corresponding number of O atoms at the intersection point will be

11 – 7.33 = 3.67 O atoms.

The ratio H/O will be 7.33/3.67 = 2.00/1.00, which will represent the empiric formula of water – H_2O.

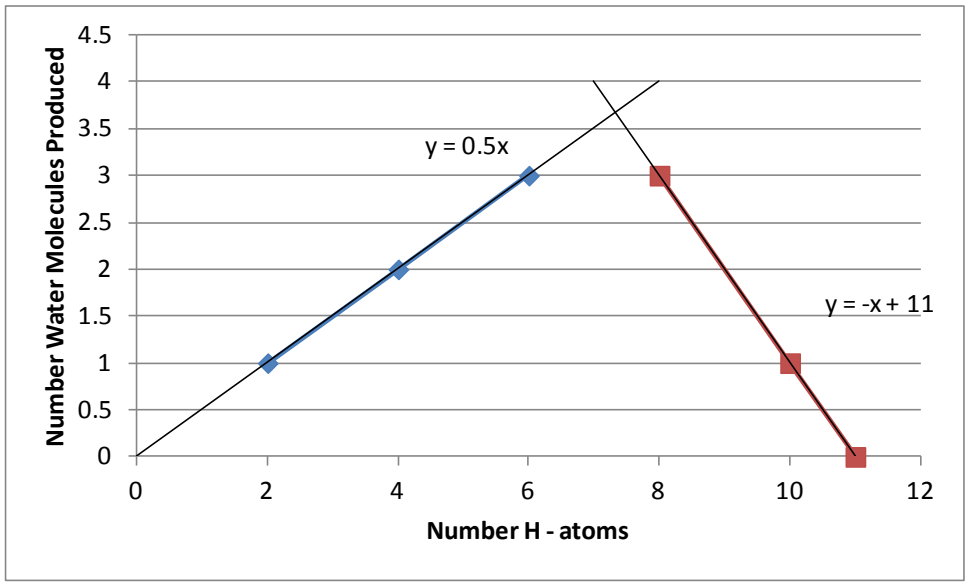

Fig. 1

The data in the pre-laboratory question and in the actual experiment will look very similarly. The only difference is that the number of atoms (on x-axis) will be replaced by moles of a reagent and number of molecules (on y-axis), with grams of product.

What You Will Use for the First Time

1. Beaker

Beakers (Fig. 2) are pieces of glassware used as containers and for approximate measurement of volume. They come in different sizes and some are graduated while others not. The approximate volume of the beaker is always marked.

Fig.2

2. Buret

Burets are calibrated to be very accurate. You will be using 50mL burets and will be able to read volume with up to 4 significant figures.
A buret is graduated in a very similar way to a measuring cylinder and is usually used to dispense accurate volumes of a certain reagent.
Usually, burets are fixed in a clamp, attached to a buret stand.

Fig. 3

You need to take your time when using a buret. Before zeroing it, make sure you open the stopcock fully and keep it open until all air trapped in the tip is released. It comes out as small bubbles and usually takes about 1mL of the reagent in the buret to push it out. Then you can adjust the zero.
When using a buret you need to be patient. You should be able to measure a single drop by gradually turning the stopcock. Sometimes, dispensing an extra drop will give you an error sufficient to affect your efforts in a negative way.
Your instructor will show you how to use a buret.

3 Vacuum (Suction) Filtration

Suction filtration (Fig. 4) is used to separate insoluble precipitate from the liquid. The set up consists of a vacuum filtration flask with a special type of funnel and rubber tubing attached to it. A proper type and size filter is placed in the funnel.
After turning on the vacuum and wetting the filter with your wash bottle, the mixture you want separated is carefully pored in the center of the funnel. At the same time, a vacuum is created in the flask through the hose. Liquid is drawn into the flask and solid remains on the filter in the funnel. When the whole amount of the mixture you want to separate is transferred to the filtration funnel, you need to rinse the container several times with water (in our case) to make sure that the entire contents of the beaker are transferred to the filter. Transfer washes to the funnel too. Then just wait for a minute for the vacuum to remove as much moisture as possible. Now you can apply a little ethanol or acetone to help dry of the “cake” (the solid left on the filter).
To remove the filter from the funnel, first stop the vacuum pump, then take the funnel off and with your spatula, carefully lift up the filter with the cake on it and place it where it belongs. Make sure you scrape all leftover solid from the bottom of the funnel and place it with the filter.
Filtration cannot be executed properly if the filtration flask is full. Check the level of liquid in the flask frequently and when it is close to the level where the vacuum hose is attached to the flask, discard the contents of the flask.

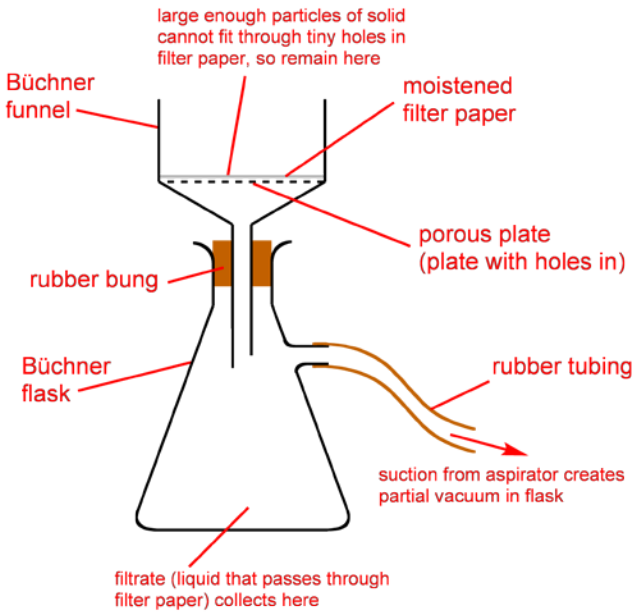

Fig. 4

Pre-Laboratory Question

Solutions with known concentration of silver nitrate (providing a source of silver ions, Ag^+) and sodium carbonate (a source of carbonate ions, CO_3^{2-}) were prepared. Varying quantities of these two solutions are mixed and the following data were collected.

Beaker #	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-}	$1.1x10^{-3}$	$9x10^{-4}$	$7x10^{-4}$	$5x10^{-4}$	$3x10^{-4}$	$1x10^{-4}$
Product, mg	13.0	39.8	65.5	92.3	80.0	26.8

Construct a plot using this data and determine the empiric formula of the product.

Procedure

A. Your instructor will explain how to use a buret. Then you will have a short training in dispensing different amounts of water and reading the volumes. Reading the buret incorrectly is the main source of error in this experiment, so you must be sure that you are able to read the buret correctly before you begin the experiment. .

B. To practice using a buret and suction filtration, you will need to determine the amount of salt in a sample of sea sand.

1. Weigh about 0.5 g of sea sand in a weigh dish. Write down the combined weight of the weighing boat and the sand.
2. Place the filter in another weighing dish and weigh them together. Write down the weight.
3. In a beaker dispense the required (ask your instructor) amount of water.
4. Transfer the sand from the weighing boat into the beaker containing the water.
5. Weigh the empty weighing boat and record the weight.
6. Find the difference between the initial weight of the weighing dish with sand and the empty one. Now you know the exact amount of sand you have in the beaker.
7. Place the filter from the second dish you weighed in the filtration funnel.
8. Turn on the suction filtration and separate the sand from the water. Make sure you have transferred everything from the beaker onto the filter. Apply some ethanol to help the filter dry.
9. Carefully take the dry filter with the sand out using your spatula and place it back in the weighing dish. If there is sand left in the funnel, use the spatula to transfer it to the weighing boat.

10. Weigh the dish containing the filter with the sand. The difference between the registered weight and the weight of the empty weighing boat with the filter will give you the amount of sand without the salt (salt is dissolved in the water and separated from the sand.
11. Now you can find what percent of your sample of sea sand is the salt.

C. Pre-Laboratory Question.

1. Plot the moles of Ag^+ on the x-axis and mg of product on the y-axis. The data should generate two straight lines: one with a positive slope on the left and one with negative one on the right.
2. Draw separate "best fit" lines for both straight lines.
3. The point where the two lines intersect should correspond to the stoichiometric ratio of the two ions.
4. You can either calculate the x-axis value of that point using the information in your regression boxes, or draw a perpendicular line from the cross point to the x-axis.
5. Once you know the number of moles of Ag, corresponding to the intersection point, you can easily find the number of moles of CO_3 (anion), since the total number of moles is constant.
6. Now you can find the ratio of Ag to CO_3 and simplify the fraction - that should result in the empiric formula you were looking for.

Plotting Data for Pre-Laboratory Question in Excel 2007 and Excel 2010

1. Enter data in a spreadsheet – Moles Ag in one column (x axis) and Mg product in another (y axis). Name columns. Now you have your data table.
2. Select all data in the data table and go to **Insert → Scatter → Scatter with only Markers**.
3. Now you see all points on your graph. You need to choose which points will form the ascending part of the graph (that with positive slope), and which – descending part (with negative slope). For the problem set it is obvious that first 4 points are on a straight line, defining the positive slope line. So, first we work with the first 4 lines in your data table. Discuss with your instructor possible data, resulting from the real experiment you will carry out next time.
4. Right click anywhere in the plot area → **Select Data** → Highlight first 4 lines in your Data Table → **OK**. Now you see only first 4 points we mentioned above.

5. Right click on any point → **Add Trendline**. In the pop up window choose: "**Linear**"; "**Forward"** = 2; "**Backward"** = 1; Check "**Display equation on chart**". Close the Trendline window. Now you have your "best fit line" for the first leg of the graph.
6. We need to add the rest of the points to the graph. Right click anywhere in the plot area → **Select Data** → In the pop up window click on "**Add**" → Fill in the boxes: **Name** – "Series 2"; **Series X values** – enter the last 2 X values from your data table separated with a coma; **Series Y values** – enter corresponding last 2 Y values → **OK** for both pop up windows.
7. You should be able to see the last two points on the graph. Repeat point **5** when working with the negative slope line. In the Trendline window switch numbers for "**Forward**" and "**Backward**".
8. Label your axis (don't forget units) and enter a title on your chart.
9. Calculate X axis value of two regression lines intersection point.
10. Find the ratio and the Empiric formula.

Data Sheet
Determination of the Formula of a Salt 1

Name:_____ **Section:**_____

Initial mass of the sea sand sample	Mass of the sand without salt	Percent salt in the sea sand sample

Calculations:

Determination of the Formula of an Unknown Salt 2

You must be very organized and accurate in everything you do during this experiment. It will take the entire 2 hours of your laboratory session. If you make a mistake you will not have an option to start over.
Look at the equipment on your work station. When leaving, you will be required to make sure that everything is clean and in a similar order.
The outcome of the experiment will look similarly to the data in the pre-laboratory question. The only difference is that now you will produce your data. That is why you shouldn't expect the points on the graph to form perfectly straight lines. There will be deviations, but the more accurately you work, the smaller the deviations will be.
You will find your reagents in two 150mL beakers. They are labeled and both of them are with concentration 0.100 Moles/L. The Unknown solution is a source of cations and Oxalic Acid – source of anions. You should not allow the two compounds to mix before you dispense the necessary amounts in the numbered 50mL beakers. To prevent mixing, you will use two burets. The plastic one (with a black stopcock) is for Oxalic Acid. The glass buret (blue or yellow stopcock) is for the Unknown.
When using the burets, make sure you have expelled the air from the tip and properly zeroed the buret. Dispense exactly the required amounts. After mixed, the reagents will start reacting immediately and the solutions will turn yellow. That must happen only in the small numbered beakers.
To accelerate the reaction, you will heat the beakers. Be careful with the hot plates.
A precipitate will be formed as a product of the reaction. You will separate the mixture and dry the precipitate using suction filtration. The less of the product you lose during your work, the better final number you will attain. In order to do so: use burets properly; don't allow the liquid in the small beakers to boil when heating it; when filtering, slowly pour the beaker's contents onto the filter; rinse the beaker three times and add the after-rinsing-water to the funnel; don't forget to use some ethanol to speed up drying; scrape the yellow product from the funnel after removing the filter and place it in the weighing dish.

Procedure

1. Turn on the hot plate and adjust the thermostat to about 170 on the digital and somewhere in the middle of the range on the analog ones.
2. Number the 6 weighing boats 1 to 6 and place a filter in each of them. Weigh each weighing boat and record the numbers. They all will have different masses.

3. Invert the glass buret so the tip is facing down, and close the stopcock.
4. Fill in and zero buret from the beaker labeled “Unknown.”
5. Dispense the following volume of Unknown (in mL) in the numbered small beakers:

Beaker #	**1**	**2**	**3**	**4**	**5**	**6**
Volume of Unknown dispensed	**2.00**	**6.00**	**10.00**	**14.00**	**18.00**	**22.00**

6. Now work with the plastic, black stopcock buret and dispense the following amounts of Oxalic acid in the small beakers, so the volume in each beaker is 24 mL:

Beaker #	**1**	**2**	**3**	**4**	**5**	**6**
Volume of Oxalic Acid dispensed	**22.00**	**18.00**	**14.00**	**10.00**	**6.00**	**2.00**

7. Place all 50mL beakers on the hot plate. By now, it should have heated up enough. It usually takes 5 – 7 minutes for the reaction to be completed. Your instructor will help you determine when to take the beakers off the hot plate.
8. While the solutions are on the hot plate, clean both burets, rinse them with your wash bottle, and invert them with the tip up and the stopcock open.
9. When you remove the solutions from the hot plate, leave them on the cool bench top for a couple of minutes to cool down. Turn off the hot plate. Do not unplug it.
10. Take the filter from weighing dish #1 and place it in the suction filtration funnel. Carefully filter the solution from beaker #1 and apply a small amount of ethanol. When reasonably dry, place the filter with the cake on it back in dish #1. Proceed

in the same fashion with the other weighing dishes, making sure that the numbers on the weighing boats match the numbers on the 50mL beakers.

11. After finishing the filtration, weigh all dishes with the filters and solid in them.
12. Find the mass of the product by subtracting the weight of the dishes without product from their weight with the solid inside. Record the data in the table.
13. Discard the filter paper with the cake in the provided container. The empty weighing boats can be discarded with the regular garbage.
14. Calculate the number of moles of Unknown and Oxalic Acid using the known concentration of the initial solutions (0.100 M) and the volumes in each beaker. Record the data in the table.
15. Draw a graph, similar to one in the pre-laboratory question. Print it out.
16. Show your calculations on the graph sheet.
17. Determine the ratio Unknown/C_2O_4.

Data Sheet

Determination of the Empirical Formula of a Salt

Name _____ **Section** _____

Your computer generated plot must be attached to this worksheet. The calculations of your x-axis value must be shown.

Beaker #	Moles Cation	Moles Oxalate	Weight of the weighing boat with the filter paper	Weight of the weighing boat with the filter paper and product	Weight of Product
1					
2					
3					
4					
5					
6					

Calculations:

Acid Base Titrations 1

Standardizing of 0.1 Molar Sodium Hydroxide Solution with Oxalic Acid

Titration is a widely used analytical method for quantitative determinations. It is among the most accurate of all analytical procedures. Although automatic titrators are commercially available, still manual titration is very common in the wet chemistry labs. Titration is a chemical reaction with known stoichiometry between diluted solutions of the reactants of interest. Usually one of the solutions is placed in a buret and the other in the vessel the titration will be carried out in. The reactant in the buret is called titrant. The substance titrated is the other reactant. The compound with unknown concentration (analyte) can be both a titrant and a substance titrated. Titration is controlled by gradually increasing the amount of titrant to the equivalence point (end point) as indicated by the color change of a chemical indicator (or response of an instrument).
In order to make a quantitative determination, one of the reagents needs to be standardized. That means that its exact concentration should be known. At the end point, the amount of the standard solution necessary to reach the equivalence point can be related to the amount of targeted reagent (analyte).
There are different kinds of titration. The simplest one is acid-base titration. For example if we titrate HCl with a standard solution of NaOH, we can calculate the amount of HCl present, because at the end point the number of moles of NaOH is equal to that of HCl. The most common indicator for acid-base titrations is Phenolphthalein. It changes its color from colorless to pink when the medium transforms from acidic to basic. Other common indicators are Methyl orange and Methyl red, which are orange or red, respectively, in an acidic medium and yellow in a basic medium.

If titrant A reacts with a substance titrated B, forming end products C and D:

$$\mathbf{A + B = C + D}$$

at the endpoint of titration (when reaction is completed and the color of solution has changed) **Moles A = Moles B**. This is true if the ratio in the balanced equation between A and B is 1/1. If, for instance, the balanced equation is:

$$\mathbf{2A + B = 2C + D,}$$

at the endpoint, **Moles A/Moles B = 2/1** (ratio of their coefficients). Accordingly, we can conclude: **Moles A = 2Moles B**. Using this simple equation, you can easily calculate the concentration of one of the reactants if the concentration of the other reagent is known. Concentration of analyte can be determined only with the accuracy we know the concentration of the standard solution. Most chemical compounds usually employed in titration methods either absorb moisture from the air, or are unstable enough and they cannot be accurately weighed out. This prevents us from directly producing a solution with precisely known concentration.

The problem is solved by preparing of solution with an approximate concentration and then standardizing it against a so called Primary standard.

A Primary standard is a highly purified, atmospheric stable compound with reasonably large molecular mass and modest cost. Very limited amount of primary standards are available commercially.

Oxalic acid ($H_2C_2O_4$) and Potassium hydrogen phthalate ($KHC_8H_4O_4$, also known as KHP) are commonly used primary standards for standardizing of bases. Oxalic acid is diprotic and reacts with 2 moles of sodium or potassium hydroxide:

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

KHP is monoprotic:

$$\mathbf{KHC_8H_4O_4(aq) + NaOH(aq) \rightarrow H2O + KNaC_8H_4O_4(aq)}$$

Usually sodium or potassium hydroxide are titrated with a solution of one of these primary standards and their concentration is accurately determined. This way, they are standardized and can be used for titrating of an acid solution with unknown concentration.

In this experiment, each group will be provided a 1 L bottle with sodium hydroxide. In the first two weeks, you will standardize the base in your bottles. In the third week, you will work with an unknown sample using the standardized NaOH from your bottles. The more accurately you work in the first two weeks, the better you will master the titration technique, and the better chances you have to correctly determine the amount of the targeted compound in the unknown sample during the third week's session.

Pre-Laboratory Questions

1. What is Potassium Hydrogen Phthalate? For what is it used?

2. A solution of sodium hydroxide is standardized against potassium hydrogen phthalate. From the following data, calculate the molarity of NaOH solution.
   Mass of KHP used: 0.4536 g
   Buret reading before titration: 0.23 ml
   Buret reading at the end point: 31.26 ml

3. How many liters of the standardized NaOH in question 2 will be needed to neutralize a water solution with 0.1453 g Oxalic acid dissolved?

What You Will Use for the First Time

1. Volumetric Flask

Volumetric flasks are calibrated to contain a specified volume; they are used to prepare solutions of accurately known concentration. They come in different sizes – from several milliliters to liters. These flasks have a specific shape (Fig. 1)

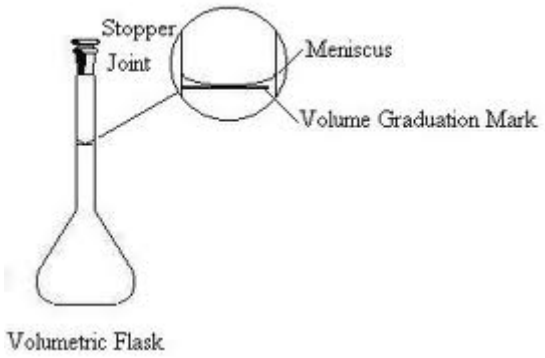

Fig. 1

On the narrow part of the volumetric flask, there is a mark that shows the level to which flask should be filled to in order to contain the specified volume. Flasks of the same volume may differ a little in height, but they are always calibrated to contain equal volumes. When we fill a volumetric flask, we should hold it at eye level, so the mark is seen as a line and the meniscus lies on the mark.

2. Erlenmeyer flask (Fig. 2)

Used for different purposes, mainly for the titrating and heating of liquids. Erlenmeyer flaks are graduated to indicate the approximate volume.

Fig. 2

3. Pipets

A pipet resembles a buret (Fig. 3). It is graduated in a similar way and is read similarly. There are different sizes and types of pipets.
Pipets are mainly used for transferring accurately measured volumes of liquid reagents. To use a pipet, one always needs a sort of pipet pump that helps with drawing the liquid in the pipet.

Fig. 3

4. Pipet pumps (Fig. 4)

Usually, pipet pumps consist of body, plunger, thumbwheel, and chuck. Some may have a fast release lever. Depending on the volume of pipets they can fill, pipet pumps come in different sizes. They are very simple to use – just depress the plunger, insert the proper pipet into the chuck and start rotating the thumbwheel clockwise. Liquid rises in the pipet. When required amount is reached, stop. Release the liquid by turning the thumbwheel in the opposite direction.

Fig. 4

Procedure

1. Write your names and section on the provided label and stick it to the bottle with NaOH. You will be using the same bottle during the three weeks this experiment will last.
2. Weigh about 1.2 g of oxalic acid dihydrate to the nearest 1 mg (don't forget to zero the balance after placing the empty weighing boat on the pan) and record the value on the data sheet.
3. Transfer the solid into a 100 mL volumetric flask. Rinse the transfer funnel several times while on the flask to make sure the entire sample is washed down.
4. Fill the volumetric flask to the mark with deionized water. Make sure the meniscus is exactly on the mark (not a little bellow or a little above). Shake the flask at least 10 times to make sure all solid is dissolved.
5. Invert the buret so the tip is facing down. Close the stopcock. Fill the buret with sodium hydroxide, expel the air from the tip, and zero the buret.
6. Using a pipet and a pipet pump, transfer 20 mL of the solution from the volumetric flask to an Erlenmeyer flask.
7. Add a couple drops of phenolphthalein and titrate the solution in the Erlenmeyer flask until the first drop that turns the color of the solution pink. (Your instructor will demonstrate how a titration is carried out).
8. Read the volume on the buret and write the number in the data sheet.
9. Carry out two more titrations in a similar way. Register the volumes of NaOH used to reach the endpoint and write them on the data sheet. You can measure different volumes of sample from the volumetric flask and this way titrate different amounts of your analyte.
10. Calculate the molarity of NaOH for each trial. The numbers should agree to ±0.004. Calculate the average molarity.

Acid Base Titrations 2

Standardizing of 0.1 Molar Solution of Sodium Hydroxide with KHP

In this week's experiment you will standardize your sodium hydroxide with the other primary standard mentioned - KHP. You already know that 1 mole of NaOH reacts with 1 mole of KHP. The ratio 1/1 between reagents simplifies calculations. You will also titrate your sample directly after weighing. This way you will avoid transferring and diluting the solution, which makes this procedure more reliable.
Weighing will be done by difference. That means that first you weigh out the container with KHP, then take out a certain amount and weigh it again. The difference between the two weights will give you the amount KHP you have taken out of the container. This is easy to accomplish with the modern electronic balances, because they can do the subtraction for you. Your instructor will show you this technique. It is advantageous, because you avoid using scoops, transfers of your sample and errors from spillages on the weighing pan. Weighing by difference will be used also when you will be working with the unknown sample next week.

Procedure

1. Weigh by difference about 0.5 g KHP. Place the solid in the Erlenmeyer flask you will be using for the titration. Record the mass of the sample on the work sheet.
2. Add about 20 – 25 mL water. By swirling, dissolve the KHP crystals. Make sure all solid is dissolved before starting to titrate the solution. The solution should be absolutely clear.
3. Add a couple of drops of indicator and titrate. You must be able to stop the titration after the single drop of NaOH that changes the color of your solution to pink.
4. Read the volume of sodium hydroxide used and record it in the data table.
5. Do at least two more titrations. Make sure that each of you practices the technique, because you will have to work on separate unknown samples (one per student, not one per pair).
6. Calculate the average molarity.

Acid Base Titrations 3

Titration of a KHP Unknown Sample

In the last two weeks you determined the exact molarity of the sodium hydroxide in your bottle. Now you can use these results in order to find the content of KHP in your unknown sample. The Unknown sample consists of unknown amounts NaCl and KHP. Only the KHP will react with the NaOH when you titrate it. That means that you will work exactly the same way as in the last laboratory session.
Since you know the molarity of your titrant and its volume at the endpoint, you can easily calculate number of moles of NaOH. Given that the molar mass of KHP is 204.2 g/mol, you can find the mass of your unknown (KHP) in the titrated sample and from there – its percentage in the weighed out amount.
The unknown is a fine powder, which is very hygroscopic (draws moisture from the air, which changes the mass of the sample). That is why you need to keep it tightly closed after weighing your amount to titrate. Make sure to wash down the powder that has stuck to the walls of your Erlenmeyer flask and then dissolve completely.
Use your sample sparingly, because you will only have a limited amount of it (enough for about 10 titrations) and will not be allowed another sample.
Carry out at least two titrations before calculating the results. If time allows, you may have to do 4 or more titrations in order to reach a correct number (if you work accurately enough).

Procedure

1. **Write the number of your unknown on your data sheet!**
2. Weigh out about 0.5 g of your unknown sample. (If you happen to weigh a little more, don't worry. That will not be a mistake. Don't throw it away; titrate it – it will only need more NaOH to reach the endpoint).
3. Wash down the powder stuck to the walls of the flask with water. Make sure you have enough water to dissolve the solid in the flask (20 – 25mL). Dissolve the sample fully.
4. Titrate carefully. At the endpoint, record the volume of NaOH in the data table. Do another trial with a similar (not the same) amount of sample.
5. Calculate the percentage of KHP in your unknown sample and show it to your lab instructor. Your lab instructor will tell you if you need more titrations.
6. Carry out additional titrations, if necessary.

ACID-BASE TITRATION DATA SHEET

Name(s): _____ Date: _____

Chemistry 10301 Section: _____

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)_____

ACID-BASE TITRATION DATA SHEET

Name(s): _____ Date: _____

Chemistry 10301 Section: _____

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)_____

ACID-BASE TITRATION DATA SHEET

Name(s): _____ Date: _____

Chemistry 10301 Section: _____

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 _____

Thermochemistry 1

Heat of Neutralization for 1.00M HCl with NaOH Unknown. Calculating of the Concentration of NaOH

Energy transferred between a system and its surroundings as a result of a temperature difference is called **heat**.
Heat capacity is the quantity of heat required to change the temperature of a system by one degree.
The quantity of heat, transferred into or out of a system as it undergoes a chemical or physical change at constant pressure is defined as the **Enthalpy change** – **ΔH –** of the process. Abundance of experimental data on enthalpy changes of different reactions are available in the literature.
Enthalpy is always interpreted as **enthalpy change per mole of reaction**.
For example if the thermochemical equation is:

A + 3B → 2C + D Δ**H = - 156 kJ/mol** (exothermic reaction),

we can write:
-156 kJ/1mol A = -156 kJ/3mol B = -156 kJ/2mol C = -156 kJ/1mol D

The amount of heat, necessary to change the temperature of one gram of a substance by one degree Celsius is called **specific heat capacity**, or more commonly used - **specific heat (C_s)**.
Specific heats of many substances are available in data tables in the scientific literature.

The amount of heat depends on the mass of the system, the temperature change and the specific heat of the substance:

$$Q = m \cdot Cs \cdot \Delta T$$

The SI unit for heat is Joule (J). Since the units for mass (g) and temperature (^oC) are known, we can figure out the unit for the specific heat.

$$Cs = Q/m \cdot \Delta T$$

From here the unit for specific heat will be **J / g^{-1} . $^oC^{-1}$**

Heats of reactions are determined experimentally in a **calorimeter**. That is a device that doesn't allow a significant heat exchange between the reaction medium and its surroundings. Because the calorimeter itself is part of the insulated system, it has its own heat capacity, depending on how much heat it absorbs.
Heat capacity (C) can be calculated as follows:

$$\mathbf{C = C_s \, . \, m}$$

Then, for the quantity of heat we have:

$$\mathbf{Q = m \, . \, C_s \, . \, \Delta T = C \, . \, \Delta T}$$

In this experiment, you will combine certain amounts of 1.00 M HCl and NaOH with unknown concentration. The base will be your limiting reagent. Your goal will be to find the concentration of NaOH using some of above equations.
For the purpose of your calculations, assume that no heat is exchanged with the surroundings and the calorimeter doesn't absorb or evolve heat. That means that the whole heat exchange happens in the solution. When we mix reagents, a chemical reaction occurs. Depending on if it is an exothermic or endothermic reaction, the temperature of the solution will increase or decrease. You can register the temperature change and use it to calculate the heat of reaction. You will determine the initial temperature of both solutions by registering temperature at the end of each minute. After combining the solutions, you will keep reading the thermometer in the same fashion.
One problem is that we need the temperature at the moment of mixing of the reagents, but our thermometer reading occurs a minute after that. Additionally, because of heat losses, the increase or decrease in temperature that we observe will be less than what the chemical reaction produces. To solve these problems, you will plot a cooling curve, similar to that in Fig. 1, and that will allow you to calculate the temperature at the moment of mixing and use it in your calculations.

In Fig. 1, reagents were combined at the end of the 11th minute. At that moment, the temperature jumped up and then started slowly cooling down. To calculate ΔT we need to find the difference between the Y-values of point A and point B (note that the points are the joints between the "best fit lines" and the perpendicular at the moment of mixing). Because we know the equations that describe our regression lines and also know that for both point A and point B the X-value is 11 (remember we started the neutralization in the 11th min.), we can easily calculate temperatures at both points of interest.

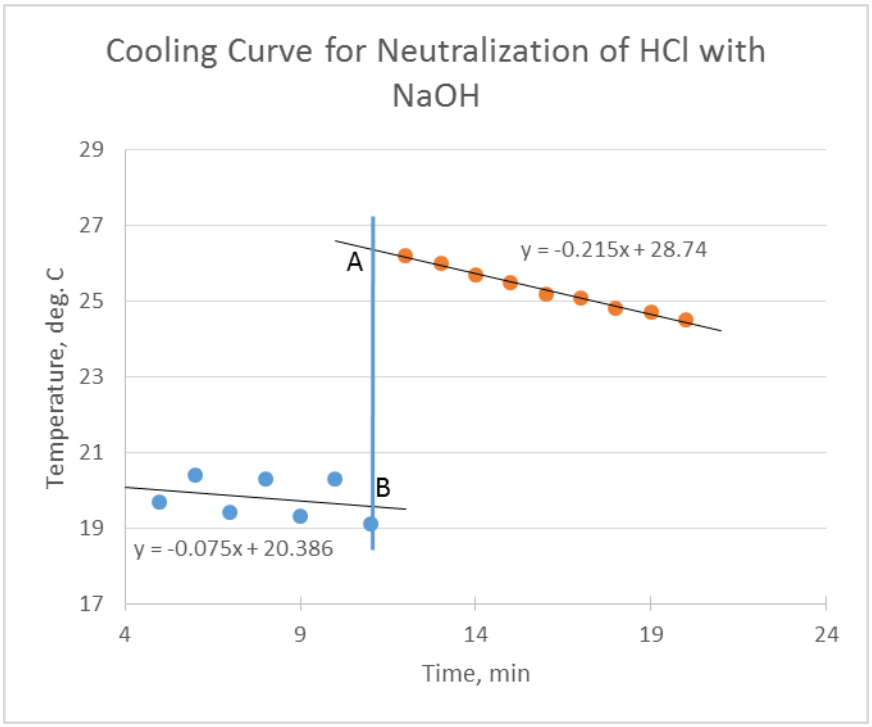

Fig. 1

Another assumption we need to make is that the specific heat of both solutions we use is that of water. That is reasonable, because solutions are diluted. The specific heat of water is **4.184 J/g.ºC**

Now you are prepared to calculate the heat of neutralization between NaOH and HCl. Once you have done that, you will have to further determine the concentration of OH^- groups (concentration of NaOH). To do that you will need to know the enthalpy change of the neutralization of a strong acid with a strong base. That can be represented by:

$H^+ + OH^- \rightarrow H_2O$ **$\Delta H = -55.81$ kJ/mol**

This tells you that the reaction is exothermic (the number is negative) and that for each mole of NaOH, 55.81kJ heat is evolved. Pay attention to the units and compare them to those of specific heat of water. You probably notice that you will need a small conversion.

Now, based on your calculation of the heat of the reaction you carried out, you can calculate the molarity of the NaOH unknown.

What You Will Use for the First Time

1. Magnetic Stirrer

A spinning magnet in the stirrer causes a magnetic stirring rod to rotate in concert with it. Magnetic stirrers are commonly used for mixing of diluted solutions. Special care should be taken when you turn on the stirrer. A very high rotating speed can cause splashing out of the liquid you are stirring. Speed should be increased very slowly until it reaches the desired level.

2. Digital thermometer

The kind you will use is a battery operated device, which allows comfortable reading of temperature on a display with precision 0.2^0C. After measurement, the thermometer needs to be turned off to conserve of the battery.

Procedure

Instead of a real calorimeter, you will use a Styrofoam cup, which will work well for the purpose of our experiment. The cup is placed in a ring to prevent possibly knocking it over when a thermometer is submerged in the vessel.

1. Place the stirring rod in the marked "Acid" styrofoam cup. Carefully measure 40.0 mL 1.00M HCl using a graduated cylinder and transfer it in the cup.
2. Place the cup in the ring above the magnetic stirrer. The ring should be adjusted to a height that allows the bottom of the styrofoam cup not to lie on the stirrer, but to be very close to it. This way, by using the stirrer, you can change position of the stirring rod in the cup.
3. In the 100 mL beaker, measure 40.0mL of NaOH with unknown concentration, using the plastic 50 mL cylinder.
4. Turn on the thermometer and submerge it in the beaker with NaOH.
5. Turn on your timer. It is set to 20 min and will start counting down. **Don't stop it until it signals and the experiment is over in 20 min.**
6. At the end of the first minute, (at the moment the timer shows 19), read the temperature and write the value in the data table. **Don't write numbers in the shaded boxes.**
7. Carefully take out the thermometer, wipe it off with a paper towel and submerge it in the styrofoam cup. At the end of the second minute (timer shows 18) read the temperature and record the number in the data table.
8. After wiping it off again, move the thermometer back to the beaker. At the end of the minute, register the temperature and move thermometer to the cup. Alternate

the position of thermometer between the beaker and the cup in the same fashion until you reach to the 10th minute.

9. During the 10th minute, place the thermometer in the cup. After reading the temperature, mix the NaOH with the HCl in the cup. Carefully turn on the stirrer and make sure the stirring rod doesn't hit the thermometer's stem. You don't need vigorous stirring.
10. Read the temperature at the end of each minute and record the data. When the timer signals, your experiment will be over. You should have 20 readings for temperature (at the end of each one of 20 min).
11. Using the data from the temperature data table, plot the time on x-axis and the temperature on y-axis. Draw a best fit line throughout the points before 11th minute and another best fit line through the points following the 10-th minute.
12. Using the data in the regression boxes, calculate the temperature at the 10th minute for both lines. The difference in both numbers will be your ΔT.
13. Calculate the concentration of your unknown NaOH and writhe the numbers in the data table. Show your calculations on the graph sheet.

Heat of Neutralization for 1.00 HCl with NaOH Unknown

Temperature Data Table

Temperature, deg C Base, Beaker	Time, min	Temperature, deg C Acid, Styrofoam Cup
	1	
	2	
	3	
	4	
	5	
	6	
	7	
	8	
	9	
	10	
Mix Reagents →	11	
	12	
	13	
	14	
	15	
	16	
	17	
	18	
	19	
	20	

Data Sheet

Heat of Neutralization for 1.00 M HCl with a NaOH Unknown

Name: _____ Section: _____

The computer generated time-temperature plot must be stapled to this worksheet and the calculations must be shown bellow the data table.

1	ΔT calculated	
2	Heat capacity (C) calculated	
3	Amount of heat absorbed by your solution (Q_{soln})	
4	Number of moles of OH^- reacted by neutralization	
5	Molarity of the original NaOH solution	

Calculations:

Thermochemistry 2

Heat of Formation of MgO

To avoid differences in interpretation of the values of ΔH, a set of conditions is designated as standard. A substance is in its standard state if it is a pure element or compound at a pressure of 1 bar (10^5 Pa) and usually temperature of 298.15 K (25°C). If reactants in their standard states produce products in standard states, the enthalpy change of that reaction will be called **standard enthalpy of reaction - ΔH^o**.
If **a mole** of a substance in its standard form is produced from the elements in their standard states, the enthalpy change is denoted as **standard enthalpy of formation** of that compound - ΔH_f^o.
A few important features make the enthalpy concept very useful:

- ΔH is directly proportional to the amounts of the reagents in a system (we used that in the previous experiment);
- ΔH changes its sign when the process is reversed;
- ΔH is a state function. As a direct consequence of that is Hess's Law. It postulates that if a process occurs in stages (or if we can theoretically present a process as a sum of several steps), the enthalpy change of the whole process is the sum of enthalpy changes of the individual steps.

These features enable us to calculate the heats of reactions for reactions that are difficult to carry out or the heats of formations of compounds that are difficult to produce in pure form.
For example, if we want to calculate the heat of reaction of ethylene with water to form ethanol

$$\mathbf{C_2H_4(g) + H_2O(l) \rightarrow C_2H_5OH(l)}$$

and we have the following data:

$$\mathbf{C_2H_5OH(l) + 3O_2(g) \rightarrow 2CO_2(g) + 3H_2O(l)} \qquad \mathbf{\Delta H^o = -1367\ kJ/mol} \qquad \mathbf{(1)}$$

$$\mathbf{C_2H_4(g) + 3O_2(g) \rightarrow 2CO_2(g) + 2H_2O(l)} \qquad \mathbf{\Delta H^o = -1411\ kJ/mol} \qquad \mathbf{(2)}$$

we can reverse the first equation (ΔH^o also changes its sign) and then add it to equation (2)

$2CO_2(g) + 3H_2O(l) \rightarrow C_2H_5OH(l) + 3O_2(g)$ $\Delta H^o = +1367$ kJ/mol

$C_2H_4(g) + 3O_2(g) \rightarrow 2CO_2(g) + 2H_2O(l)$ $\Delta H^o = -1411$ kJ/mol

After canceling of $2CO_2$ and $3O_2$ on both sides and $2H_2O$ on the right site, the result will be:

$C_2H_4(g) + H_2O(l) \rightarrow C_2H_5OH(l)$ $\Delta H^o = -44$ kJ/mol

We can use similar approach to calculate the heat of formation of MgO. Since it will be very difficult to directly measure the heat produced by the reaction between Mg and O_2, we need to employ Hess's law. The sum of equations (3), (4) and (5) should result in the direct interaction of Mg with O_2.

$Mg(s) + 2H^+(aq) \rightarrow Mg^{2+}(aq) + H_2(g)$ **(3)**

$Mg^{2+}(aq) + H_2O(l) \rightarrow MgO(s) + 2H^+(aq)$ **(4)**

$1/2O_2(g) + H_2(g) \rightarrow H_2O(l)$ **(5)**

The sum of above equations will result in:

$Mg(s) + 1/2O_2(g) \rightarrow MgO(s)$

If we have the values of enthalpy changes of above three reactions (3, 4 and 5), we can calculate the enthalpy of formation of MgO as a sum of those values:

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

We can easily carry out reaction 3, measure the temperature change, and calculate ΔH_3. The best way of obtaining the value for ΔH_4 is to reverse reaction (4), calculate the enthalpy change, and reverse the sign.
The value for ΔH_5 can be found in the literature as the enthalpy of formation of 1 mole of water: -285.85 kJ/mol.

Procedure

A. Determination of ΔH_3

1. Measure and add exactly 60.0 mL of HCl to the Styrofoam cup marked “Acid” with the stirring rod in it.
2. Place a weighing dish on the balance pan and weigh it. Record the weight. Zero the balance with the weighing boat on it. Weigh 0.18xx g of granulated magnesium (atomic weight 24.305g).
3. Turn on the thermometer and submerge it in the cup. Carefully turn on the magnetic stirrer. Turn on your timer. Start registering temperature at the end of each minute. Write the data in the temperature data table (pg. 49).
4. At the end of 7th minute, add the magnesium to the HCl. Keep registering the temperature at the end of each minute until the timer signals. Make sure all of the Mg is dissolved.
5. Weigh the empty weighing boat (after placing the magnesium in the HCl) and find the exact amount of Mg reacting with HCl in the cup.
6. Find ΔT.
7. Calculate ΔH_3.
8. Calculate the heat of formation of MgO.

B. Determination of $-\Delta H_4$

The procedure is exactly the same as with determining of ΔH_3. Measure 60 mL HCl and register the initial temperature for 10 min. Weigh 1.0xx g of MgO (molecular weight 40.305g) and add it to the cup at the end of the 10th minute. Keep reading the temperature until the timer signals. Make sure all MgO is dissolved. Don’t forget to re-weigh the boat containing any residual MgO. Calculate the exact weight of MgO delivered. Calculate ΔT and then $-\Delta H_4$.

Heat of Formation of MgO

Temperature Data Table

	Time, min	Temperature, deg. C
	1	
	2	
	3	
	4	
	5	
	6	
Combine reagents →	7	
	8	
	9	
	10	
	11	
	12	
	13	
	14	
	15	
	16	
	17	
	18	
	19	
	20	

Data Sheet
Heat of Formation of MgO. Reaction of Mg with HCl

Name: _____ **Section:** _____

The computer generated time-temperature plot must be stapled to this worksheet and the calculations must be shown bellow the data table.

1	ΔT calculated	
2	Heat capacity (C), calculated	
3	Heat of calorimeter (Q_{soln})	
4	Number of moles of solid used	
5	Enthalpy of reaction (ΔH_3)	
6	Heat of formation of MgO	

Calculations:

Introduction to the Methods of Absorption Spectroscopy 1

Discussion and Problem Set

Electromagnetic radiation is a form of energy that is transmitted through space. We refer to UV, visible, and IR regions of electromagnetic radiation as light, but in fact the human eye can register only the visible part of the electromagnetic spectrum (visible light).

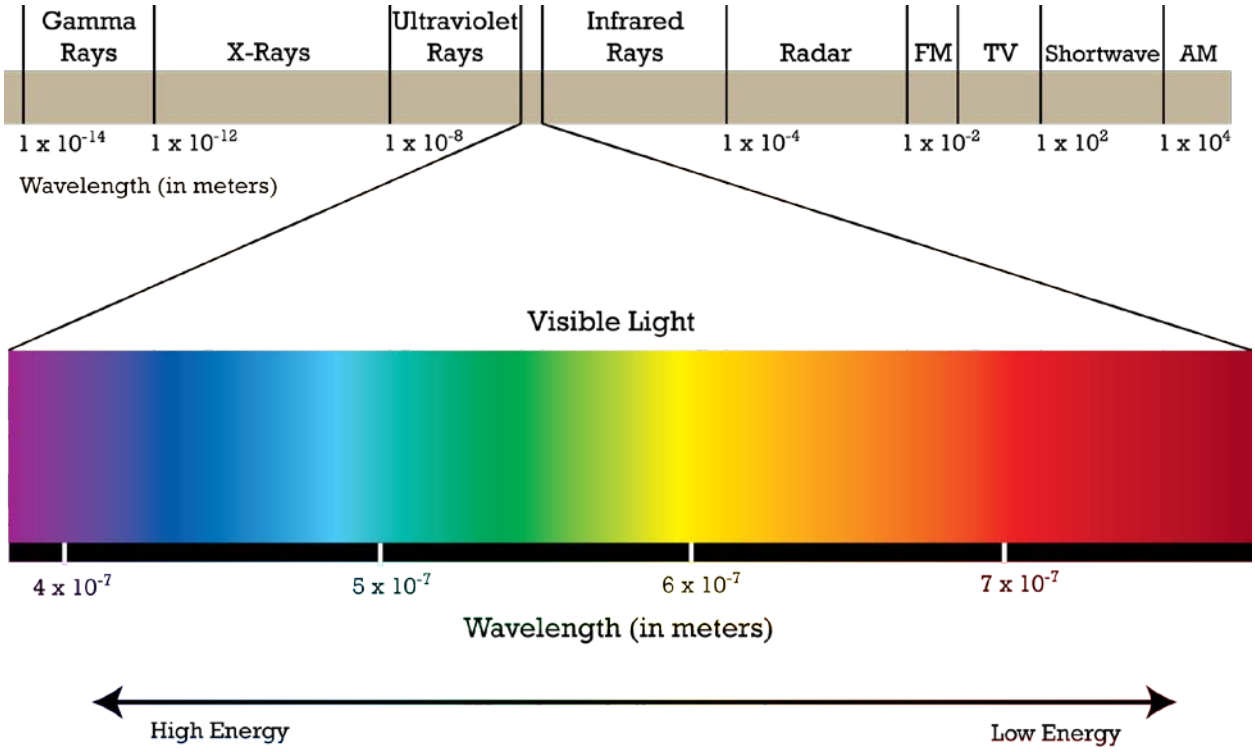

Fig. 1

Spectroscopy is the study of interaction of electromagnetic radiation and matter.

When light with intensity I_0 strikes a sample, part of it will be lost, another part will be absorbed, and a third part, with intensity I ($I < I_0$) will go through the sample. The ratio of I/I_0 is defined as Transmittance:

$$\mathbf{T = I/I_0} \qquad \mathbf{(1)}$$

Commonly used is percent transmittance:

$$\%T = T \cdot 100 \qquad (2)$$

Unfortunately transmittance is not linear with concentration. That makes it difficult to use for quantitative purposes. However, because the relationship between transmittance and concentration is logarithmic, the logarithm of transmittance should be linear. Thus we define the parameter of "absorbance" as the negative logarithm of the transmittance:

$$A = -\log T \qquad (3)$$

Absorbance is a parameter that increases linearly with concentration and is important for quantitative analysis. If transmittance is measured by an instrument, it must be converted to absorbance before plotting it versus concentration.
The exact relationship between absorbance and concentration is known as the Beer-Lambert law or simply as Beer's law:

$$A = \varepsilon CL \qquad (4)$$

Here **A** is **absorbance, ε** is **specific absorptivity** (**absorptivity**) or **the extinction coefficient** – the inherent ability of a chemical species to absorb light – and is constant at a given wavelength, and **L** is the **path length** – the distance the light travels through the measured solution. Usually the analyte solution is placed either in a test tube or a small container called cuvette. L will be the inside diameter of the test tube or the inside length of the cuvette. Usually that size is 1 cm.
Absorbance is dimensionless. That means that the units of specific absorptivity will depend on the units of concentration and those of path length.

The relationship between absorbance and concentration serves as the basis for the quantitative analysis of a great many substances. In practice, the absorbances of a series of solutions of known concentrations are measured and a plot of absorbance versus concentration is prepared (Fig. 1). Such a plot is known as a Beer's Law graph and also represents a calibration curve for the particular system being studied. An unknown solution containing the same absorbing substance may then be analyzed by measuring its absorbance, locating its A value on the calibration curve, and reading the corresponding concentration.

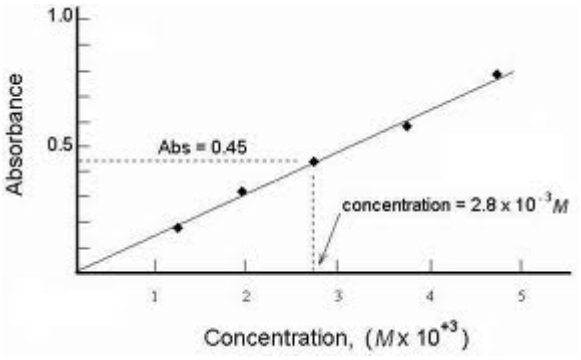

Fig. 2

In Fig. 2, the dotted line shows how the curve is used to find the concentration of an unknown solution with an absorbance of 0.45.

Spectrophotometers are the instruments, used to obtain quantitative information
The essential components of a spectrophotometer are:

Source of light → Wavelength selector → Sample → Detector → Display

As a **source of light** for the visible region, a light bulb with a tungsten filament is used. Such a source is very bright and emits light over the entire visible region. The **wavelength selector** (**monochromator**) is designated to transform the incoming complex white light into a single wave monochromatic light beam. That happens by first dispersing the white light into a spray of rainbow colors and then selecting the proper one for the analysis. This way, the **sample** is struck by light with a known wavelength. Light that passed through the sample is then registered by the **detector** and converted into a digital signal which is read on the **display**.
The **absorption spectrum** is the pattern of absorption over a range of different wavelengths. It is unique for a particular chemical species (it is usually called "fingerprint" of that species). That is why it can be used as a means of qualitative determination. It also shows at what wavelength a sample of analyzed compound has the highest absorption value – the wavelength at which quantitative determinations for that compound should be conducted.
The instrument registers the intensity of light that passes through the sample and then calculates transmittance, using equation (1) or equation (2). After that, transmittance is transformed into a reading for absorbance utilizing equation (3).

When the light from the source strikes the sample, part of it is lost as reflected light absorbed by smudges or drops of liquid on the cuvette by different solutions present in the cuvette. To solve this problem, the instrument needs to be calibrated before conducting measurements on the samples. Also, a single cuvette should be used for all measurements (cuvettes look the same way but in fact there are differences we cannot see, which can affect negatively the measured values). The cuvette must be clean and rinsed multiple times with the solution we will be analyzing before the real sample is measured.

Instruments are calibrated for $I_0 = 0$ and $I_0 = 100\%$ (eq. 1). When $I_0 = 0$, light is physically blocked from striking the detector. To calibrate for $I_0 = 100\%$ a **blank sample** is used. It contains all chemical species that will be present in the samples to be measured except for the analyte species. Such a solution should not display any absorption due to the analyte and the light reaching the detector represents the maximum light that can be registered by detector at any time.

Problems

1. A sample in a 1cm cuvette gives an absorbance reading of 0.558. If the absorptivity for this sample is 15000 L/(mol.cm), what is the molar concentration?
2. If the transmittance for a sample having all the same characteristics as in previous problem is measured as 72.6%, what is the molar concentration?
3. The transmittance of a solution, measured at 590 nm in a 1.5-cm cuvette, was 76.2%.

a. What is the corresponding absorbance?
b. If the concentration is 0.0802 M, what is the absorptivity of this species at this wave length?

If the absorptivity is 10000 L/(mol.cm), what is the concentration?

4. What is the path length in centimeters when the transmittance is 0.692, the molar absorptivity is 7.39 x 104 L/(mol.cm) and the concentration is 0.0000923 M?
5. A standard iron sample with concentration 26 mg/L gave a transmittance reading of 52.8%. What is the concentration of an unknown iron sample if its transmittance is 61.7%?

6. A series of solutions, containing cobalt (II) ion were prepared and their absorbance measured at 515 nm. The following values were obtained:

Concentration (g/100 ml)	Absorbance
0.8836	**0.8238**
0.7084	**0.6502**
0.4362	**0.4054**
0.1758	**0.1640**
0.1064	**0.0982**
0.0702	**0.0630**

Plot a calibration line Absorbance versus Concentration. From the graph determine the specific absorptivity.
Using the data you obtained from the graph in the previous problem determine the concentration of cobalt (II) in 1.975 g sample of a substance, which was dissolved in water in a 100 mL volumetric flask and then diluted to the mark. For the absorbance of the cobalt solution from the flask a value of 0.405 was obtained. What is the percentage of cobalt in the unknown compound?

Introduction to the Methods of Absorption Spectroscopy 2

Preparation of a Calibration Curve for Iron Using Beer's Law

Using a stock solution of iron, you will carry out an experiment which will result in a set of data that will allow you to plot your calibration curve. If you work precisely, the points on your Absorbance vs Concentration plot should be very close to forming a straight line. Inaccuracies in your work will result in more scattered points and thus less reliable data to be used in the following week.
The chemical reaction that will produce a colored compound proportional to the amount of iron in the solution is:

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

Here iron 2+ ions react with 1,10-phenanthroline to result in an orange colored complex with a wavelength of maximum absorption 508 nm. Before carrying out the reaction we need to make sure that proper pH value (about 3.5) of the solution is maintained (sodium acetate buffer is utilized for that purpose). Usually in the iron solution there is also a certain amount of Fe^{3+} ions. They all have to be reduced to Fe^{2+} (Fe^{3+} ions also form a colored complex with 1,10-phenanthroline, but it absorbs at a different wavelength). The reducing agent we will use is hydroxylamine hydrochloride.
Because of the above, the order in which we combine the solutions should be:

Iron + Sodium Acetate + Hydroxylamine Hydrochloride + 1,10-Phenanthroline

What You Will Use for the First Time

Digital spectrophotometer Spectronic 20D

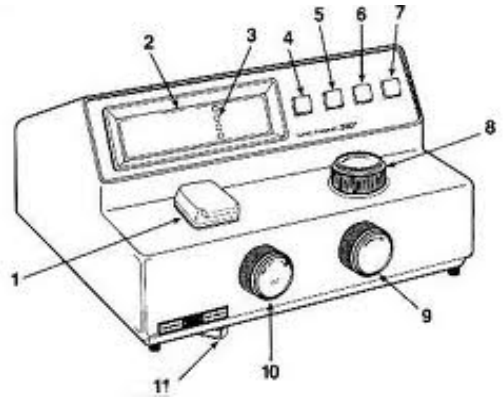

Fig. 1

The instrument is used for measuring the color intensity of a solution.

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
10. Power switch/Zero control
11. Filter level

The Spectronic 20D is turned on by turning the “Power switch/Zero control” knob (10) clockwise. The instrument needs at least 15 minutes to warm up and stabilize the source and detector. Wavelength can be set with the “wavelength control” knob (8). In this experiment, the wavelength will be already set for you at 508 nm. Before doing anything else, you first need to carefully adjust 0% transmittance with “Power switch/Zero control” knob (10). Make sure the sample compartment is empty and the cover is closed. To measure absorbance, a cuvet with the analyzed solution **in a cuvet holder** is inserted into the sample compartment. You always start with a blank solution (which doesn’t contain the targeted element). After inserting the blank in the sample compartment and closing the cover, you need to adjust 100% transmittance or 0 absorbance (using the “Mode” button you can switch from transmittance to absorbance) using the “Transmittance/Absorbance control” knob. Now instrument is ready to measure your colored solutions.

Procedure

On your work station, you will have four different solutions in four labeled plastic vessels. The concentration of the stock iron solution is 40.0 mg Fe/L. Using the pipet pump and each of the four pipets, you will need to measure

amounts of each solution shown in the table (in mL), and transfer it in the volumetric flasks.

Flask #	**1**	**2**	**3**	**4**	**5**
Iron Stock Solution, mL	**10**	**5**	**2**	**1**	**0**
Sodium Acetate, mL	**10**	**10**	**10**	**10**	**10**
Hydroxylamine Hydrochloride, mL	**2**	**2**	**2**	**2**	**2**
Phenanthroline Solution, mL	**3**	**3**	**3**	**3**	**3**

1. Using the 10 mL pipet marked for iron, carefully measure the amounts of stock solution shown in the table and transfer them into 4 different flasks. Be very accurate, because the volumes of iron solution determine the quality of your calibration curve. Note that you will not have iron solution in the fifth flask. It will be your blank solution which you will use to calibrate the spectrophotometer before measuring the absorbance of the colored solutions.
2. With the other 10 mL pipet, (not labeled), measure 10 mL of Sodium Acetate for each of the 5 flasks.
3. Use the smallest size - 2 mL - pipet to measure Hydroxylamine Hydrochloride and transfer it into the volumetric flasks.
4. Measure 3 mL of Phenanthroline with the available 5 mL pipet and place it in each flask.
5. Dilute all 5 flasks with deionized water to the mark. One of them (the blank) should be colorless.
6. Turn on the spectrophotometer. It is important to wait for 15 minutes for the machine to warm up and the color to develop fully in the flasks. If you proceed with measurements earlier, you risk getting lower absorbance numbers and consequently, a wrong calibration line.
7. In 15 minutes, make sure the measuring compartment is empty and its lid closed. Then carefully adjust 0% transmittance with the left hand side knob (10).
8. In order to have consistent results, use only one cuvet for all measurements. Rinse your cuvet 3 times with deionized water. Rinse it once more with the blank solution and only then fill it with the

blank solution for measuring. Insert the cuvet in the provided cuvet holder with its clear side facing the small window on the holder. Insert the cuvet holder with the cuvet in the measuring compartment. Make sure the protected part of the cuvet is facing left.

9. With the right hand side knob, adjust 100% transmittance. Press the mode button (4) once to switch to absorbance. Now, the display should show 0.
10. Take out the cuvet, rinse it three times with deionized water, then once with the solution you will be measuring. Fill the cuvet with the solution you want to measure. Using the cuvet holder, insert the cuvet in the measuring compartment. Read the display. Repeat this for each of the solutions you are measuring.
11. After you finish the measurements, turn off the spectrophotometer. Make sure the measuring compartment is empty.
12. Draw the calibration line using CBL graph and write your results in the data table.

Data Sheet
Introduction to the Methods of Absorption Spectroscopy 2
Calibration Curve for Iron

Name: _____ Section: _____

Your computer generated plot must be attached to this worksheet. The calculation of at least one concentration must be shown.

Flask #	Volume of stock solution containing Iron	Concentration of Iron, mg/L Fe 2^+	Absorbance
1			
2			
3			
4			
5			

Specific absorptivity (ε)_____

Y intercept _____

Correlation coefficient (COR)_____

Equation of the calibration curve: A = _____ C ± _____

Calculations:

Introduction to the Methods of Absorption Spectroscopy 3

Determination of the Iron Content of a Supplement Tablet

Ideally, you should obtain the vitamin tablet, dissolve it, filter the solution, dilute it and then analyze the final solution using a spectrophotometer. However, because dissolution and filtration are time consuming techniques, you will have your vitamin pill dissolved for you in 6M hydrochloric acid in a 50 mL beaker. Solutions may have a different color depending on the vitamin dissolved. As you know, color may affect the values you obtain when you use a spectrophotometer. To prepare your sample, you will dilute the initial solution to a solution with a concentration hundreds of times smaller and this way, color influence on your results will be negligible.
The procedure for this experiment is similar to the procedure from the last week. Please remember that your sample is dissolved in 6M HCl. It will be a very small amount of solution, which makes it easy to handle, but you must still be very careful when you handle your initial solution.

Procedure

1. Obtain a dissolved vitamin pill from your instructor.
2. Stir the solution with a glass rod and transfer it carefully from the beaker to a 100 mL volumetric flask. Rinse the beaker several times with deionized water, and transfer the washes to the volumetric flask.
3. Carefully dilute the solution to the mark. Now, you have your vitamin pill diluted in exactly 100 mL (0.1 L) solution. Shake the flask several times. This will be your flask A.
4. Using the Iron marked pipet, measure exactly 10 ml of solution from flask A and transfer it to another volumetric flask – B1. Transfer another 10 mL from flask A to one more volumetric flask – B2. Dilute flasks B1 and B2 to the marks. Shake them several times to mix the solutions.
5. Prepare your samples by transferring 5 mL solution from flask B1 to an empty flask – C1 and from flask B2 to C2. Flasks C1 and C2 will

be your final samples you will be measuring with a spectrophotometer.

6. Using proper pipets, add 10mL of Sodium Acetate, 2 mL of Hydroxylamine Hydrochloride and 3 mL of Phenanthroline solutions to each of C1 and C2 flasks. Dilute to the marks and shake flasks well.
7. Wait 15 min for the color to develop before measuring. Use the time to clean your work station and rinse the pipets. **Note – you must wait at least 15 minutes**. Otherwise the reading of the spectrophotometer will not represent the real concentration of your solutions.
8. Measure your samples (flasks C1 and C2) and record the results in the data table.
9. Discard the solutions in the provided waste bottle. Rinse your flasks.
10. Calculate the amount of Iron in the vitamin pill.

Data Sheet
Introduction to the Methods of Absorption Spectroscopy 3
Iron Content of a Vitamin Pill

Name: _____ Section: _____

	Flask 1	Flask 2
Absorbance of solution C		
Concentration of Iron in solution C, mg/L		
Concentration of Iron in solution B, mg/L		
Concentration of Iron in solution A, mg/L		
Content of Iron per tablet, mg		

Equation of the calibration line: _____

Calculations:

Molecular Modeling using PC Model Program

In general, the properties of a substance depend on the size and shape of its molecules together with the length, strength and polarity of its chemical bonds. A small change in the size and shape of a molecule of a drug, for example, can make a big difference in its effectiveness and possible side effects.

Armed with the Lewis theory and the VSEPR theory, we can predict the structure and the shape of a molecule in most cases with reasonable accuracy.

For quantitative predictions, however, the principles of those theories need to be combined with experimental data. That is achieved by mathematical models, which, in turn, are developed into different software.

The program PC Model is based on molecular mechanics – it utilizes accumulated experimental data together with the fundamental principles regarding molecular shape and structure. To calculate an unknown bond length, bond angle, or dipole moment, the model assumes that:

a. bond angles and bond lengths between 2 atoms are similar no matter what structure they are part of;
b. atoms in a molecule are arranged in a way that guarantees the lowest energy configuration of the structure.

Pre-Laboratory Assignment

Based on what you already know about molecular shape and structure, determine the shape of the molecules from the data table for the PC Model experiment (CH_4, CH_3I, NH_3, H_2O, CO_2, PCl_5, HCHO). How many different bond lengths and bond angles you expect for each molecule? Which of the molecules are polar and which nonpolar?

Draw and fill in a table similar to the following one:

Molecule	Shape	Number of different bond lengths expected	Number of different bond angles expected	Polarity

Procedure

To begin drawing your molecule, click on the **Draw** button and then click somewhere about the center of the drawing window. A dot that represents a **carbon atom** will appear. By clicking on **H/AD** button we can add **hydrogen atoms** bonded to the carbon center (Fig. 1). You may have to **rotate** the newly created molecule to be able to see all 4 bonds. To do that go to **View → Control Panel → Rotate.** You can chose the axis to rotate your structure around. When finished, close the **Control Panel**. If the molecule is too large or too small, you can click on the **Update** button. That function will place the structure in the center of the graph window with a proper size.

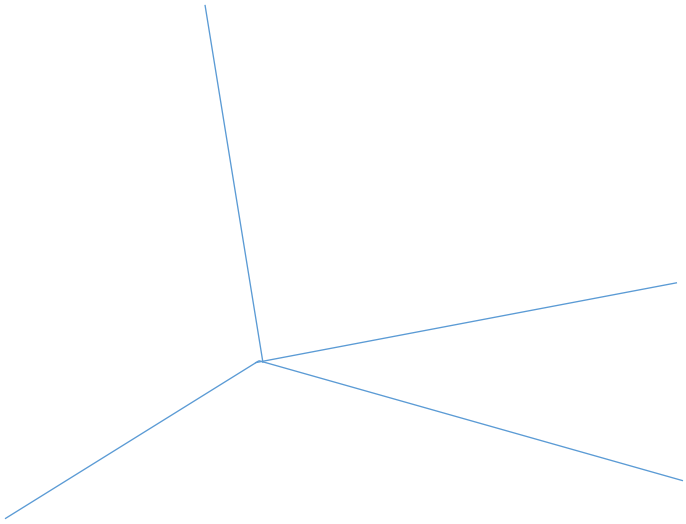

Fig. 1

To start collecting quantitative data for the molecule on your screen, you first click on **Analyze** on the menu and then on **Minimize**. The program executes multiple computations in order to redraw the molecule in its most stable conformation. On the right of your screen the computed **dipole moment** will appear. You need to minimize every new molecule you have created before obtaining quantitative information. Now you are ready to measure bond lengths and bond angles. To begin that click on **Query** (for every new molecule you start measuring by clicking on **Query** button).

To **determine a bond length** you need to tell the software which bond you are measuring by **clicking on the atoms the bond connects**. When you point the cursor to an atom, a small box will appear; click to select the atom. Repeat that for the second atom. To see a value for the bond length, click somewhere aside of the bond between the two atoms.
If we determine the length of bond AB, first we click on atom A, then on atom B and the third click is just to show the value for the length (Fig.2). Record the number in the data table on your worksheet.

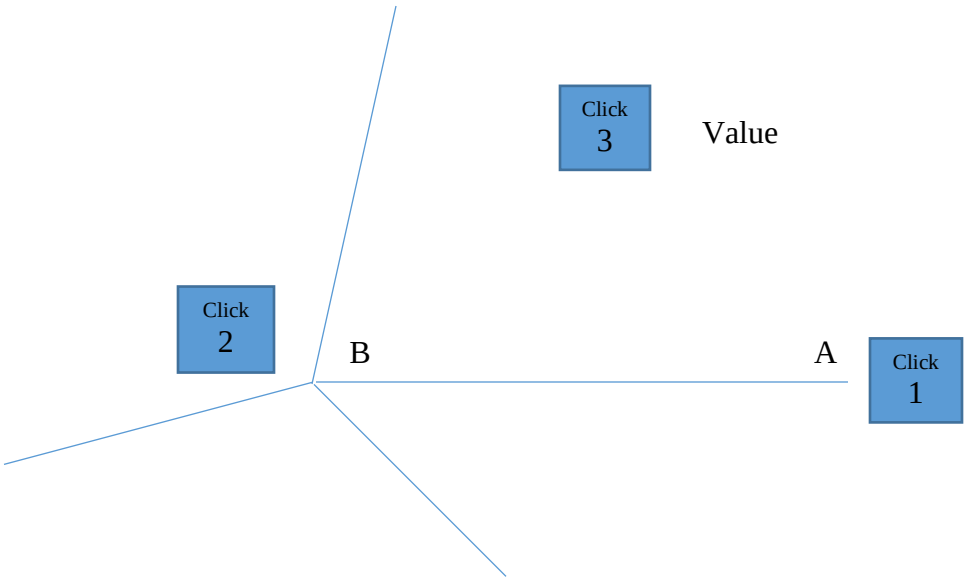

Fig. 2

To **measure a bond angle,** the approach is similar. Because an angle is determined by 3 points (3 atoms), you need to select the angle you are measuring by clicking 3 times.
The fourth click, usually somewhere in the area between the shoulders of the angle, should give you the value of the angle you want to measure. Here are the 4 clicks for angle ABC (Fig. 3):

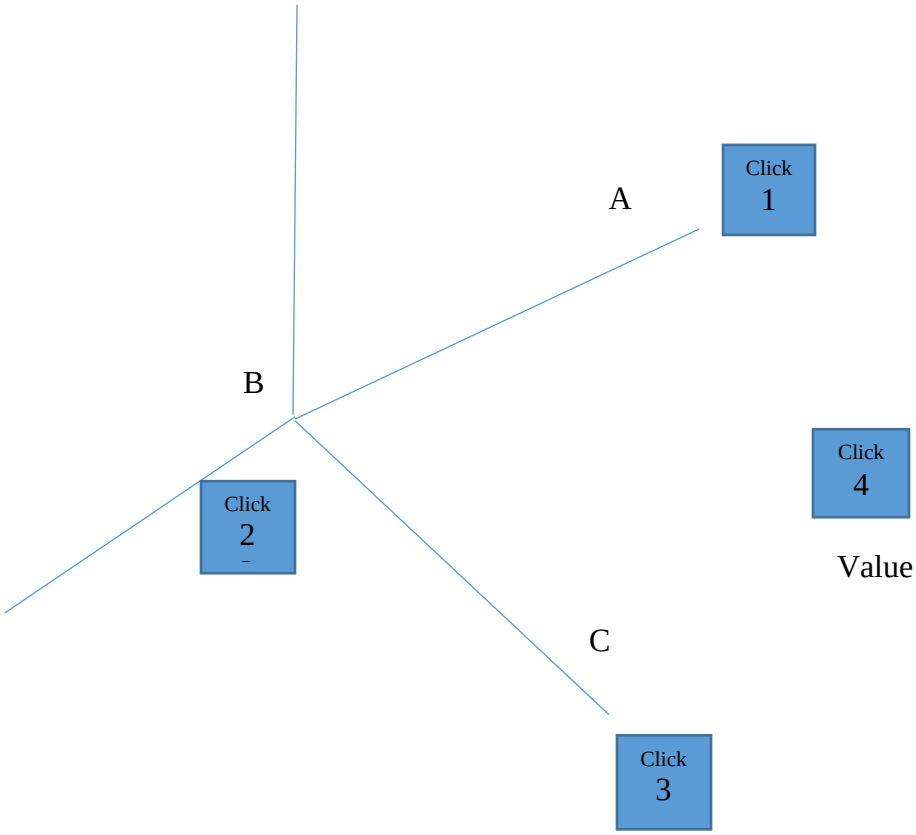

Fig. 3

To **erase a drawing** go to **Edit → Erase**.
To **erase a bond or atom**, click on **Del** on the **Tool Bar** and then on what you need erased.
To draw various molecules you will need to replace carbon or hydrogen atoms with those you need for your structure. To do that simply click on **PT button** on the **Tool Bar**, select the element you need and then click on the atom you need to be replaced.
For example: if you need to CH_3I from CH_4, first draw CH_4, then click consequently on **PT → I → either of H atoms in CH_4**. **Close the PT window**, because it may interfere with the query process. Now that you have **CH_3I** on the screen, you can do the usual steps – rotate, update, minimize, query.
For the **NH_3** and **H_2O** obviously the carbon center should be replaced by N or O respectively before adding of H atoms.

To draw **CO_2** molecule you will need to construct double bonds. To begin place 3 separate carbon centers on the graph window. Replace two of them with **O** atoms, using **PT.** To add additional bonds between carbon and oxygen atoms click on **ADD-B** on the **Tool Bar,** point the cursor to the middle already existing bond (an x should show that the bond is highlighted) and click. A new bond should appear. Repeat that with the other single bond, rotate, update, minimize and query.

To draw **PCl_5**, you will have to substitute **P5** for the central atom and **Cl** for all **H** atoms. To add a fifth **H** atom, click **Draw** and place an **additional carbon center** in the vicinity of your already available structure and then click on the **central atom** to establish the new bond. Replace the new carbon center with another **Cl** atom.

Now you have the knowledge and tools to draw and investigate **HCHO** molecule.

PC Model Data Sheet

Name _____ Section _____

Molecule	#	Bond Lengths	Bond Angles	Shape	Dipole Moment
CH_4	1.			_____	_____
	2.				
	3.				
CH_3I	1.			_____	_____
	2.				
	3.				
NH_3	1.			_____	_____
	2.				
	3.				
H_2O	1.			_____	_____
	2.				
	3.				
CO_2	1.			_____	_____
	2.				
	3.				
PCl_5	1.			_____	_____
	2.				
	3.				
HCHO	1.			_____	_____
	2.				
	3.				