CHEMISTRY 122L

Separations and Measurements Laboratory
Spring, 2000
Tu/Th 1-5pm
Dr. Bradford D. Pendley
411 Kennedy Hall: 843-3959

GOALS: The goals that you should work towards in this course are;

1. To learn to make observations and assess their importance;
2. To learn to perform several techniques commonly used in chemical analysis;
3. To learn to keep a scientific notebook and to communicate the results of a scientific investigation in a clear manner.

LAB MANUAL: The experiments you will explore come from a variety of sources. You are provided with a lab manual that details the procedures for each experiment.

EVALUATION: Your grade in this course is based on your performance on several laboratory assignments designed to assess your mastery of the three goals listed above. For each experiment, you will be evaluated on whether or not you made certain critical observations, your ability to perform certain measurements, and your analysis and presentation of your findings. Since we will not have discussed the theory behind each experiment prior to your doing it, you may have to consult other resources (such as your textbook and books found in Burrow Library) to explain the observations you made. All experiments except the first one will count either 100 or 200 points (this is noted on the schedule on page 5). The first report will be a practice report and will not count in the determination of your grade. The total number of points attained on the reports determines your final grade.

Be certain to check for spelling and grammatical errors in your typewritten report. Attention to such detail will influence your grade on each report.

Grade	Total points
A- / A	900-1000
B- / B / B+	800-899
C- / C / C+	700-799
D- / D / D+	550-699
F	below 550

POLICIES: My expectation is that you will attend all labs unless directed otherwise. For each laboratory experiment, you are to keep a written record of your procedure, observations, data and calculations in your laboratory notebook. Unless otherwise specified, you are required to turn in the notebook along with a typewritten report no later than the beginning of the lab period one week after the scheduled end of the experiment. Unless there are extenuating circumstances, reports turned in after this time but before two weeks after the scheduled end of the experiment will have 10% of the total available points deducted. Reports will not be accepted after this time and zero points will be recorded for the assignment.

LABORATORY NOTEBOOK FORMAT:

Title/Date:
Procedure: The procedure includes all experimental details related to conducting the experiment.

Data/Observations:	All measurements and observations made during the experiment should be recorded directly in your notebook immediately after they are made. Please label all data and observations clearly.
Calculations:	Include all calculations in this section and identify what you are calculating.

TYPEWRITTEN REPORT FORMAT (for experiments 1-3):

Title/Date/Name/Pledge:	
Introduction:	State what you are studying or determining and why this is important. State the principle(s) behind the analysis and give equations for all pertinent chemical reactions.
Procedure:	You may cite the procedure in the lab manual.
Results/Discussion:	Present all tables and graphs of data. Compare results with expected values. Note differences. State all important observations. Explain all important observations (include equations if applicable).
References:	(if appropriate)

TYPEWRITTEN REPORT FORMAT (for experiments 4-6):

Title/Date/Name/Pledge:	
Introduction:	In narrative form, briefly describe what you are studying or determining and why this is important. In addition, explain the principle(s) behind the analysis and give equations for all pertinent chemical reactions.
Procedure:	You may cite the procedure in the lab manual.
Results/Discussion:	This section should contain an organized (i.e., tables, graphs) presentation of the results of your experiment, a comparison with expected values and a discussion of any significant difference. In addition, you should state and explain all important observations.
References:	(if appropriate)

Use this checklist to help you write your report.

In the report,

1. Do you have a cover page, introduction, procedure, results/discussion section and references?
2. In the introduction have you,

   - stated what you are studying and why?
   - explained the principle(s) behind the analysis and described any pertinent chemistry?
3. In the results and discussion section have you,

   - stated the results of your investigation and compared those results with accepted values?
   - stated and explained all important observations made during your investigation?
4. Have you properly referenced all appropriate material?
5. Do you have page numbers, are graphs needed and included, and have you proofread the report?

References

During the course, you may want additional information to help you understand a concept or interpret your data. I encourage you to come to me and ask questions and/or consult written material (i.e., books and/or journals) to better understand those concepts or to learn more about the subject. **However, you may NOT consult laboratory reports written by former Chemistry 122 students.**

Any time that you use either the *ideas* or *writings* of another person in your report, you must acknowledge that you are not the originator of that work. This is a professional courtesy, and failure to

give proper credit is plagiarism. In this course, I recommend that you use the following format to properly cite another author's work.

Journal publication:

1) First author last name, FI, MI; next author *Journal name abbreviation* **year of publication**, *volume number*, inclusive page numbers.

For example,

1) Robertson, R.T.; Pendley, B. D. *J. Electroanal. Chem.* **1994**, *374*, 173-177.

Book:

1) First author last name, FI, MI; next author *Title of book*, edition; publisher: place of publication, year of publication; chapter (or pages).

For example,
1) Harris, D. C. *Quantitative Chemical Analysis*, 5th ed.; Freeman: New York, 1999; chapter 1.

Laboratory Manual:

1) Pendley, B. D. Chemistry 122 lab manual. 2000.

Notes:

1. The number of the reference (superscripted) should be listed after the cited material in your text. For example, if you consulted chapter 13 in Harris to help you explain some of your observations on the second lab experiment, and this happened to be the first reference you used, then you would place [1] immediately following that explanation in your discussion. If, at a later point in your discussion, you again refer to Harris, refer to that reference as [1] again.

2. You should cite the procedure for the laboratory experiments by citing the laboratory manual.

SAMPLE
LABORATORY NOTEBOOK DOCUMENTATION

The determination of the hardness of water by EDTA titration 1/27/00

I weighed some dry $Na_2H_2EDTA \cdot 2H_2O$ and dissolved it in 1.000 liter of deionized water.

weight of $Na_2H_2EDTA \cdot 2H_2O$: 0.3107g

observation: The EDTA dissolved readily to form a clear, colorless solution.

A 25.00mL portion of Evian water was pipeted into a 250mL Erlenmeyer flask and 3mL of the pH 10 buffer solution along with 3-4 drops of Eriochrome Black T indicator was added. The sample was titrated until the color changed from wine-red to blue.

observation: The bottled water remained clear and colorless after addition of pH 10 buffer and had a characteristic ammonia smell. After addition of the Eriochrome Black T indicator, the solution turned a clear wine-red.

initial buret reading:	0.05mL	wine-red
final buret reading:	20.15mL	blue
volume of EDTA added:	20.10mL	
initial buret reading:	0.10mL	wine-red

final buret reading:	20.15mL	blue
volume of EDTA added:	20.05mL	
initial buret reading:	0.00mL	wine-red
final buret reading:	20.24mL	blue, overshot endpoint
volume of EDTA added:	20.24mL	
initial buret reading:	0.20mL	wine-red
final buret reading:	20.24mL	blue
volume of EDTA added:	20.04mL	

A blank was performed using 25.00mL of deionized water.

observation: The blank remained clear and colorless after addition of the pH 10 buffer solution and had an ammonia smell. After addition of the indicator, the solution turned a clear wine-red.

initial buret reading:	31.10mL	wine-red
final buret reading:	31.16mL	blue
volume of EDTA added:	0.06mL	

To determine the concentration of calcium in the water, a 25.00mL portion of bottled water was pipeted into a 250mL Erlenmeyer flask and 30 drops of 50% NaOH was added to the solution and swirled for two minutes to precipitate the $Mg(OH)_2$. About 0.2g of solid hydroxynaphthol blue indicator was added to the flask and the sample was titrated with the EDTA solution. The titrated solution was allowed to stand and was occasionally swirled to redissolve any $Ca(OH)_2$ that may have precipitated. It was then titrated to a blue endpoint if necessary.

observation: Upon addition of the 50% NaOH solution to the water, the solution remained clear and colorless. After addition of the indicator, the solution turned a clear, pale wine-red.

initial buret reading:	0.10mL	wine-red, very pale
final buret reading:	10.14mL	pale blue, let stand 2 minutes, still blue
volume of EDTA added:	10.04mL	
initial buret reading:	0.00mL	wine-red, very pale
final buret reading:	10.10mL	pale blue, let stand 2 minutes, turned pink
initial buret reading:	10.10mL	
final buret reading:	10.15mL	pale blue, stayed blue
volume of EDTA added:	10.15mL	
initial buret reading:	0.06mL	wine-red, very pale
final buret reading:	10.14mL	pale blue, let stand 2 minutes, still blue
volume of EDTA added:	10.08mL	
initial buret reading:	1.00mL	wine-red, very pale
final buret reading:	11.14mL	pale blue, let stand 2 minutes, still blue
volume of EDTA added:	10.04mL	

A blank was performed using 25.00mL of deionized water.

observation: The blank remained clear and colorless after addition of the 50% NaOH solution. After addition of the indicator, the solution turned a clear pale wine-red.

initial buret reading:	41.10mL	wine-red, very pale
final buret reading:	41.14mL	pale blue
volume of EDTA added:	0.04mL	

Calculations:

Molarity of the EDTA solution:

$$0.3107 \text{ g EDTA} \times \frac{\text{a mol EDTA}}{\text{b grams EDTA}} = \text{mol EDTA}$$

$$\text{M (EDTA)} = \frac{\text{mol EDTA}}{1.000\text{L}} = \text{answer}$$

(moles of Ca^{2+} + moles of Mg^{2+}):

Titration 1:

$$(0.02010\text{L EDTA sol'n} - .00006\text{L}) \times \text{M EDTA sol'n} \times \frac{\text{a mol EDTA}}{\text{b mol } Ca^{2+} + Mg^{2+}} = \text{mol } Ca^{2+} + Mg^{2+}$$

Repeat for other titrations and other calculations.

CHEMISTRY 122L

Spring, 2000

Day	date	Laboratory
Th	1/13	Safety; orientation; check in
Tu	1/18	Relationship between a penny's mass and its minting
Th	1/20	Relationship between a penny's mass and its minting
Tu	1/25	Analysis of Ca^{2+} and Mg^{2+} in bottled water
Th	1/27	Analysis of Ca^{2+} and Mg^{2+} in bottled water (**100 pts**)
Tu	2/1	Acid/base chemistry
Th	2/3	Acid/base chemistry
Tu	2/8	Acid/base chemistry
Th	2/10	Acid/base chemistry (**200 pts**)
Tu	2/15	Determination of Fe in vitamins
Th	2/17	Determination of Fe in vitamins (**100 pts**)
Tu	2/22	Analysis of Fe in pigments
Th	2/24	Analysis of Fe in pigments
Tu	2/29	Analysis of Fe in pigments
Th	3/2	Analysis of Fe in pigments
Tu	3/14	Analysis of Fe in pigments

Th	3/16	Analysis of Fe in pigments (**200 pts**)
Tu	3/21	Gas chromatography (all)
Th	3/23	Caffeine in beverages / Analysis of alcohols
Tu	3/28	Caffeine in beverages / Analysis of alcohols (**100 pts**)
Th	3/30	Analysis of alcohols / Caffeine in beverages
Tu	4/4	Analysis of alcohols / Caffeine in beverages
Th	4/6	Analysis of alcohols / Caffeine in beverages (**100 pts**)
Tu	4/11	Team challenges
Th	4/13	Team challenges
Tu	4/18	Team challenges
Tu	4/25	Team challenges (**200 pts**)
Th	4/27	Check out

CHEMISTRY 122L

Separations and Measurements Laboratory

Introduction

Science is that human endeavor that seeks to understand how and why the universe works. The general approach that scientists use in trying to answer these questions is to first define and ask a specific question about the workings of a piece of the universe. Almost always the scientist has some knowledge about the particular area that is of interest to her or him and with this knowledge, the scientist makes a hypothesis about how or why something works the way it does, and then designs some experiment to test the hypothesis. The experiment is performed while the scientist carefully observes and records the events of the experiment, and from these records, the scientist can compare the predicted results with those observed and support or refute the original hypothesis.

Chemists try to understand how and why atoms and molecules behave in the fashion they do, and to use this knowledge to improve the quality of human life. In designing chemical experiments, the chemist must consider what information is sought about some atom, molecule or collection of both and what is the best way to obtain this information. When scientists want such information, they do what most people do; using their senses, they observe some property or collection of properties of the substance and look for some pattern which leads them to the

information they need to answer the question they asked. However, life is complicated. Many times our senses cannot give us the "data" we need to answer a question, or our senses give us so much information that we cannot make sense out of all of it, or our senses deceive us. Because of these complications, scientists try to develop tests that reliably ascertain the information that our senses cannot provide. And in order to develop these tests, a theoretical understanding of the concepts and principles that relate the measurable or observable quantities to the desired property must be understood. These tests follow one of two strategies. The first is to **separate** the desired component from everything else that interferes with the observation and measurement of the property of interest. The second is to design a test that allows the scientist to observe and **measure** only the desired component's property.

In this course you will learn about several of the major tests that scientists use to study chemical systems, and the principles on which they are based. The laboratory experiments will teach you about these tools and about doing science. Your laboratory experience will not focus on tests that illustrate principles in the lecture, but rather you will construct those principles based on your measurements and observations in the laboratory. Thus, you will be doing science the way scientists do: constructing models of how and why something works based on a critical analysis of observations and measurements made during a carefully planned experiment. It is critical that while you are performing all aspects of your experiment, no matter how trivial, you pay very close attention and be open to observe what occurs. Many times in science "unexpected" results in an experiment occur. Observing and assessing the value of these results is a critical skill you should develop. In the experiments you will do this semester, many planned "unexpected" events will occur during the course of an experiment. Part of what you will be evaluated on will be your ability to make keen observations and measurements and assess their importance and implications.

Experiment 1: Relationship between a penny's mass and its year of minting

In this first laboratory experiment, you will investigate the relationship between the weight of a U.S. penny and the year it was minted. You are to weigh between 10 and 15 pennies to investigate this relationship. I will provide you with the pennies (although you are free to bring your own sample). You may use any of the balances in this lab that you choose, and the manner in which you make the measurement is also your choice. However, you should make a hypothesis about the relationship and then design your measurement to provide you with the appropriate information. For instance, suppose you postulate that the weight of the penny is larger for older pennies than for newer ones because of all the fingerprints and grime that accumulates on the surface. Therefore, you might not wish to clean this grime off the surface and not touch the pennies (and thus put additional finger oils on them) during the weighing.

After you have weighed your pennies you are to draw preliminary conclusions from your results. Was your hypothesis correct? We will then discuss similarities and differences between your results and those of your classmates, as well as different ways to present your results that maximizes the information you wish to convey. Next, you will pool your data and see what conclusions you can draw. And finally, you may propose additional experiments to further clarify your conclusions, and if so, you will be free to pursue these.

Since this is the first laboratory experiment, you will be required to turn in your lab notebook and a report but it will not be "graded." Instead, I will provide you with feedback based on your data, observations, calculations, organization, presentation of results and the conclusions you have drawn from both your and your classmates' data. Based on these results, we will discuss errors, precision, accuracy, and statistical treatment of data.

This experiment is an adaptation of an experiment described by Ricci, R.W.; Ditzler, M.A. *J. Chem. Ed.* **1991**, *68*, 228-231.

Experiment 2: Analysis of Ca^{2+} and Mg^{2+} in bottled water

Natural bottled water has become very popular in the past five years, and numerous companies distribute a variety of products such as mineral, spring, or artesian water. The Food and Drug Administration places specific definitions on these categories, and one of the distinguishing characteristics of these waters in the amount of Ca^{2+} and Mg^{2+} in them.

Given the variation in the concentration of Ca^{2+} and Mg^{2+} with product, your task in this laboratory experiment will be to determine the concentration of Ca^{2+} and Mg^{2+} in a bottled water of your choice.

A standard method for determining the concentration of Ca^{2+} and Mg^{2+} is by titration with EDTA (ethylenediaminetetraacetic acid), a metal chelating agent that reacts with both of these ions. The procedure you will use is a slightly altered version of the one found in your textbook, Experiment 29-9. The entire procedure is detailed below.

1. Analytically weigh about 0.3g of dry $Na_2H_2EDTA \cdot 2H_2O$ and dissolve it in 1.000L of deionized water. Mix well.

2. Pipet 10.00mL of the bottled water sample into a 250mL Erlenmeyer flask and add about 3mL of pH 10 buffer solution and 3-4 drops of the Eriochrome Black T indicator solution. Titrate the sample with the EDTA solution. The color changes from wine-red to blue at the endpoint. Repeat the titration until your precision is less than 0.2%. Perform a blank using 10.00mL of deionized water instead of the bottled water. **Note**: Depending on the concentration of Ca^{2+} and Mg^{2+} in your bottled water, you may have to dilute the bottled water with deionized water or adjust the amount of bottled water you use in order to use a convenient amount of titrant.

3. For the determination of calcium, pipet 10.00mL of the bottled water into a 250mL Erlenmeyer flask, add 30 drops of 50% NaOH to the solution and swirl for two minutes to precipitate the $Mg(OH)_2$. Add about 0.2g of solid hydroxynaphthol blue to the flask and titrate the sample with the EDTA solution. Let the titrated solution stand and occasionally swirl it to redissolve any $Ca(OH)_2$ that may have precipitated, then titrate to a blue endpoint if necessary. Perform a blank using 10.00mL of deionized water. Repeat the titration until you obtain a precision less than 0.2%.

Experiment 3: Acid-base chemistry

Acid-base reactions are used by scientists in many ways. One common use involves determining the volume of a standardized acid or base needed to completely react with another base or acid of unknown concentration, and then relating this volume to the concentration of the base or acid. Such acid-base titrations commonly employ an acid-base indicator, or a chemical compound that signals the completion of the reaction.

Another use of acid-base reactions involves a similar acid-base titration experiment, but now the course of the reaction is followed by measuring the pH of the solution that is being titrated and not the use of an acid-base indicator. This allows the endpoint of the titration of the base or acid to be determined.

In this experiment, you will first prepare and standardize a solution of hydrochloric acid using an indicator to signal the endpoint of the reaction. After you have accomplished this, you will then use your standardized acid to titrate a known quantity of a pure base, and you will follow the course of the reaction by using a pH electrode and meter to measure the pH of the solution as you titrate. From an analysis of this data, you will determine the compound's equivalence weight.

The procedure for the first part of this experiment can be found in experiment 29-5 of your textbook (p 849). The entire procedure is detailed below.

1. Prepare approximately 1 liter of 0.1M HCl from the 1 M HCl stock solution. Mix well.

2. Standardize the HCl solution in the following manner. Accurately weigh enough dry sodium carbonate to react with about 25mL of the 0.1M HCl solution and place it in an Erlenmeyer flask. The reaction is,

$$2HCl + Na_2CO_3 \rightarrow CO_2 + 2NaCl + H_2O$$

Dissolve the sodium carbonate in about 25mL of deionized water and add a few drops of bromocresol green indicator.

3. Titrate the sample until it just turns from blue to green (If the solution turns yellow, you have added too much HCl). Then boil the solution to expel the dissolved carbon dioxide. The solution should return to a blue color.

4. Continue titrating the sample until you reach the endpoint (green). You should repeat this procedure at least four times to obtain a precision less than 0.1%. You should also perform a blank titration using the same number of drops of bromocresol green as used previously in 50mL of 0.05M NaCl.

5. Obtain a sample of your base and dry it at 110^{o}C for two hours. Accurately weigh about 0.3g of your pure, dry sample into a beaker and dissolve it in 100mL of deionized water. Titrate your sample with the HCl and follow the reaction using a pH electrode and meter, recording the pH of the solution after every one milliliter of acid has been added. I will demonstrate the use of the pH meter and electrode as well as the experimental setup for this portion of the experiment. Prepare a graph of these data (pH vs. volume of HCl added) and determine the equivalence volume. Then, repeat the titration twice with two new 0.3g portions of your sample. However, for these two trials, when you are within a few milliliters of the equivalence volume, use 0.1-0.2mL instead of 1mL increments so that you will be able to more

accurately determine the equivalence volume. From these data, you will determine your base's equivalence weight.

Experiment 4: Spectrophotometric determination of iron in an iron supplement tablet

In this laboratory experiment, you will determine the amount of iron in a commercial iron supplement tablet. I will provide you with the iron supplement, or you may provide your own sample if you check with me first.

The procedure for this experiment is found in experiment 29-17 of your textbook (pp 863-864). The entire procedure is detailed below.

1. Prepare a **standard** Fe solution by dissolving 0.141g of reagent-grade $Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$ in deionized water in a 500mL volumetric flask containing 0.5mL of concentrated sulfuric acid.

2. Place one Fe tablet in a 100mL beaker and boil gently (in the hood) with 25mL of 6M HCl for about 20 minutes. Filter the solution into a 100mL volumetric flask and rinse the beaker several times (with small portions of deionized water) to complete the quantitative transfer. Dilute to the mark with deionized water. Pipet 5.00mL of this solution into a clean 100mL volumetric flask and dilute to the mark with deionized water. This **diluted Fe tablet solution** will be used in steps 4 and 6.

3. Pipet 10.00mL of the **standard** Fe solution into a beaker and measure the pH with a pH electrode and meter. Add the sodium citrate solution dropwise until you reach a pH of 3.5. Count the drops needed.

4. Pipet 5.00mL of the **diluted Fe tablet solution** into a beaker and measure the pH with a pH electrode and meter. Add the sodium citrate solution dropwise until you reach a pH of 3.5. Count the drops needed.

5. Pipet a 10.00mL aliquot of the **standard** Fe solution into a 100mL volumetric flask and add the same number of drops of citrate solution you used in step 3 to bring the pH of the solution to 3.5. Prepare three more Fe solutions using 5.00, 2.00, and 1.00mL of the **standard** Fe solution and the same proportion of citrate solution (i.e., if you used 30 drops of citrate solution for 10.00mL of Fe, then use 15 drops for 5.00mL of Fe). Pipet 2.00mL of hydroquinone solution and 3.00mL of o-phenanthroline solution into each flask, and dilute to the mark with deionized water. Prepare a blank using no Fe, but all other reagents.

6. Pipet a fresh 5.00mL aliquot of the **diluted Fe tablet solution** into a 100mL volumetric flask and add the necessary amount of citrate solution, 2.00mL of hydroquinone solution and 3.00mL of o-phenanthroline solution, and dilute to the mark with deionized water.

7. Allow the solutions to stand for at least 10 minutes, then measure the transmittance or absorbance of each solution at 508nm using a Spectronic 20.

Experiment 5: A determination of the iron content in a pigment

Venetian Red is a red pigment comprised of iron oxides that has been used by artists since early times. Its color is somewhat variable and depends upon the amount of iron present in the pigment. In this experiment, you will perform a redox titration to determine the amount of iron present in a pigment sample. You should follow the procedure outlined below.

1. You should first prepare and standardize a solution of 0.02M potassium permanganate. The procedure for this is a slightly modified version of the one found in an earlier version of Harris. The procedure is detailed below.

2. Prepare a 0.02M potassium permanganate solution by dissolving 2.9g of $KMnO_4$ in 900mL of deionized water. Store this solution in a dark glass bottle and allow it to stand at least 2 days. If you observe a brown residue in the solution at this time, filter the solution through a sintered glass funnel.

3. Standardize your permanganate solution using sodium oxalate. Analytically weigh a 0.25g sample of dry sodium oxalate into a 250mL Erlenmeyer flask and add 50mL of deionized water and 30mL of 3M sulfuric acid. Add about 90% of the theoretically calculated volume of permanganate necessary for the titration, and let the solution stand until it turns colorless. Then heat it to 55-60^o C and complete the titration by adding the $KMnO_4$ solution until the pink color of permanganate persists. Repeat the process until the permanganate solution is standardized. Perform a blank. The equation for the standardization is;

$$2MnO_4^- + 5H_2C_2O_4 + 6H^+ \rightarrow 2Mn^{2+} + 10CO_2 + 8H_2O$$

purple colorless

4. After you have standardized your potassium permanganate solution, you should analyze a known iron sample to test your ability to perform this procedure. This sample has been dried at 110^o C for two hours. Accurately weigh a 0.35-0.40g portion of the iron sample into a 500mL Erlenmeyer flask, add 10mL of concentrated HCl and gently heat the solution (do not boil) to dissolve the sample. You should cover the flask with a watch glass while heating.

5. Each of the iron (III) samples must be reduced to iron (II) prior to titrating. To do this, add some 0.25M $SnCl_2$ dropwise to the warm dissolved sample solution until the yellow color disappears, then add two more drops excess to ensure complete reduction of the iron (III). Cool

the solution to room temperature and then **rapidly** add 10mL of 5% $HgCl_2$ solution to remove the excess tin (II). A **small** amount of a silky white precipitate (Hg_2Cl_2) should form. Wait 2-3 minutes and then add 25mL of Zimmermann-Reinhardt reagent and 300mL of deionized water to the flask. Titrate immediately until the pink color of permanganate persists. Do not add the $KMnO_4$ solution rapidly at any time. Perform a blank. Subtract the volume of permanganate consumed in the blank from that used in the sample.

6. After you have verified your ability to perform this test using the known iron sample, you may analyze your unknown pigment sample. Dry your pigment sample at 110^o C for two hours. Repeat the procedure described in steps 4 and 5 using your unknown sample until you obtain an acceptable precision (< 0.1%).

Experiment 6: The separation and determination of caffeine in a soft drink by thin layer chromatography

In this experiment, you will explore some of the principles of thin layer chromatography and then apply these to separate and measure the amount of caffeine in a soft drink of your choice.

The procedure for this experiment is described below.

1. Prepare 10mL of a standard caffeine solution (in methanol) with a concentration of 0.5mg/mL.

2. Clean four 200mL beakers and watch glasses. Rinse each beaker with several small portions of a mobile phase (There are four mobile phase compositions of ethyl acetate/methanol: 100:0; 90:10; 80:20; and 70:30). Place a different mobile phase in each beaker along with a piece of filter paper that is wrapped around the inside of the beaker. After the filter paper has been soaked with the mobile phase, ensure that about 0.5cm of the mobile phase covers the bottom of the beaker.

3. Clean several TLC plates by placing a plate in each of the beakers, covering the top with a watch glass, and allowing the mobile phase to move about 80% up the plate. Remove the plate and dry it with the heat gun.

4. Select a soft drink from our vast collection of beverages or bring your own sample. Place a 2µL quantity of the beverage and a 0.5µL quantity of your caffeine standard about 0.5cm apart onto the clean silica gel TLC plate and dry both spots with the heat gun. Develop your plate using different mobile phase compositions of ethyl acetate/methanol. After the plates have been developed, remove them, mark the solvent front, and dry them. Visualize the results using an ultraviolet lamp. CAUTION: When you use the ultraviolet lamp to see the spots on your plate, do not look directly at the lamp or shine it in a friend's eyes. It can cause severe eye

damage. From your results, identify the caffeine spot in your beverage and calculate the retardation factor for every spot. Select a mobile phase composition that facilitates separation of the caffeine from the other beverage components.

5. Each plate has a compound on it that fluoresces when ultraviolet light strikes it. Caffeine does not fluoresce and therefore shows up as a dark spot on a light green background. The darkness of the spot is related to the amount of caffeine present. You can estimate the concentration of caffeine in your sample by adjusting the amount of caffeine standard you place on the plate until the spots from the standard and the sample are the same. You may adjust the amount of caffeine placed on the plate by adjusting the concentration of the standard solution or varying the volume of the spot.

Experiment 7: Separation, identification and determination of alcohols using gas chromatography

In this experiment, you will investigate how temperature influences separation in gas chromatography, and then apply this knowledge to separate the components in a mixture of alcohols. You will identify each component on your chromatogram at three temperatures and then quantify one of the components using the method of standard addition. You will work in teams for this experiment, and only need to turn in a single, typed report for the team.

You will be given a mixture containing three compounds: ethanol; 1-propanol and 1-butanol. Your first task will be to investigate the effect of temperature (of the column) on the separation of these compounds. Inject 0.2-0.4μL of the mixture onto the column at 80, 120 and 160^{o} C, and record the resulting chromatogram.

You must identify which peak on the chromatogram corresponds to which compound at each temperature, and this can be done by injecting 0.2-0.4μL portions of each pure compound into the column and then matching its retention time with that of a peak in your mixture. After you have identified each component, you will confirm each component's identity using a gas chromatograph/mass spectrometer.

You will then quantify one of the alcohols (of my choosing) using the method of standard addition and using one of the other alcohols as an internal standard. I will explain the details of these procedures to you in lab.

Experiment 8: Team Challenges

In this experiment, you will work as part of a team and apply what you have learned in this course to solve real chemical problems.

Projects: The class will be divided into teams that will work on various projects. The idea is that your team is a company that has been asked to solve a problem by another company. Two teams working independently on an assigned project will compete with each other. The projects will be communicated to the manager of each team. No team member may consult any source (book, person, or other information source) unless I give you permission.

Team composition: Each team will have one manager whose responsibilities and authorities are listed below. Each team may have, at the discretion of the manager, an assistant manager. The remainder of the group is composed of staff scientists.

Manager's responsibilities:

1. Serve as the team's liaison between your team and the company.

2. Advise the team of the project and deadline.
3. Organize and direct the team's activities.
4. Prepare or supervise the preparation of the final report.
5. Submit the report.

Manager's authorities:
1. The manager may delegate work.
2. The manager may appoint an assistant manager.
3. The manager's decisions are final.
4. The manager may spend money.
5. The manager may fire a team member for not working. This is a last resort and must have my approval. The person dismissed will receive 100 points for the lab and will be relieved of all further team duties.

Report: The proposal should be a professional document that addresses the question posed to your team. It should be typed and laser printed, free of typographical and grammatical errors. It should be carefully constructed and brief. There is no format. For some projects, it might be appropriate to write a proposal and cover letter (perhaps on your company's stationary). Please include the names of all team members on the report.

Evaluation: Each team member will receive the same grade (unless a person is fired) for the project. The grade is computed as follows:

	100 points for completing the work
up to	50 points - did your team correctly answer the question?
up to	50 points - team proposal and selling your team's work

In addition, each team receives 1 point per every $10 of unused consulting money. Of the two competing teams, the team that answers the question correctly and does the better job promoting itself and/or has the lower cost receives a bonus 10 points.

Consulting: Each team has $100 in their consulting budget that they may use to consult with me on technical matters. I will consult at a rate of $400/hr, with a minimum of $10 for any work done.

Buying supplies: Chemicals and equipment may be obtained from Mr. Goode, our local chemical supplier. To obtain chemicals, you must first consult our catalog and make certain that we sell the desired compound(s). To order either equipment or chemicals, a purchase order must be correctly completed and turned into Mr. Goode. Once turned in, delivery time will be one hour from receipt of the purchase order. However, Mr. Goode will provide Federal Express (15 minutes) service for an additional fee. All fees and prices, along with the group's supply budget will be made known to the manager.