

CHEM 232: Quantitative Analysis Laboratory Manual

Spring 2016

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0 Introduction

0.1 Syllabus for CHEM232 Laboratory

0.1.1 Course Specific Policies

1. Attendance

The only legitimate excuses to miss a lab is participation in a university sanctioned trip (in which case you need to provide written documentation ahead of time). Attendance is not mandatory, but **STRONGLY** recommended. There is no extra lab time for you make up missed lab sessions. You have to complete your laboratory experiments in the allotted time.

Also there are no make ups for quizzes and the final exam.

2. Mobile Communication Devices Policy Mobile communication devices are prohibited in the laboratory during quizzes and exams. If I see you using a mobile communication device during an exam or quiz, I will assume that you are cheating, and you will receive a zero on the assignment, and the WCU Academic Integrity Policy reprinted below will be followed.

0.1.2 Grades

Your laboratory reports are due one week after you have completed your measurements and other laboratory procedures or on the Due Date listed on the course website. The reports are due at the beginning of the lab period. Late reports will not be graded. Git 'R' Done!

1. Other Grade Adjustments Cleaning your glassware and lab station after you have finished an experiment is just as important as the experiments that you do. However you must complete the cleaning of glassware and your workstation before the end of class. If you leave your glassware out or put it away dirty or leave your lab station dirty, as determined by your instructor, you will be penalized 10 % from your highest lab grade for every violation.

If you are late in leaving the lab because you did not manage your time well, you will be penalized 4 % of your highest lab grade for ever 10 minutes after the end of the period.

In experiments where you are issued a student specific unknown, you will be given enough of this sample to perform the experiment plus one additional trial. If you need more of this unknown you will have to purchase it with your grade. You will loose 5% of the maximum grade for that particular experiment for each additional unknown that you receive.

2. Grade Determination for Laboratory Course Each laboratory experiment is worth 50 points divided into several categories.

Lab number	Graded item	points	comments
Lab 1	accuracy	NA	
	precision	20	
	quiz	20	
	notebook	10	
Lab 2	accuracy	10	
	precision	20	10 for each cation
	quiz	10	
	notebook	10	
Lab 3	accuracy	15	
	precision	15	
	quiz	10	
	notebook	10	
Lab 4	accuracy	15	
	precision	15	
	quiz	10	
	notebook	10	
Lab 5	accuracy	15	
	precision	15	
	quiz	10	
	notebook	10	

Quantitative Analysis emphasizes getting the correct results with a high degree of certainty, so a significant portion of your lab grade will be determined from your results. Some experiments require some spreadsheet work. You will submit your experimental results in spreadsheet form.

After each due date, a lab calculation quiz will be given. The quiz will last about 30 min. In this quiz you will be given a set of data and asked to calculate a result from the lab experiment that you have finished. You will only be allowed to use a calculator, pencil, and your laboratory notebook.

Your grade that is submitted to the lecture instructor is calculated in the following formula:

$$\text{Lab Score} = \frac{\text{Total Achieved Points}}{\text{Total Possible Points}} \times 50\%$$

Graded Items	possible points
Experiment 1	50
Experiment 2	50
Experiment 3	50
Experiment 4	50
Experiment 5	50
Final Exam	100
Total Possible Points =>	350

Your grades for this laboratory will be based upon your reported results and the final exam.

0.1.3 Laboratory Notebook

Your laboratory notebook will be collected on the due date for each experiment.

The notebook should only be recorded in blue or black non-eraseable ink.

The laboratory notebook is a chronological record of your work. So there should be no skipped spaces or pages. All unfinished space at the end of a page should be neatly crossed out. If you have to amend data or information to a previously recorded page. You should add the correction or information on the current page (current = the day you are writing the information), and indicate on the previous page that additional info is located on another page. This amendment should be in a different color pen (blue or black), initialed and dated. If a single datum is changed on the same date as it was recorded, a line through the erroneous datum accompanied by the corrected value suffices. Where whole pages of data must be discarded a comment must accompany the crossed out section justifying the abandonment of those data. Do not use correction fluid, liquid paper, white-out or any product that covers up original entries.

Efficiency in the laboratory is only possible from experience (of which you probably don't have yet and are gaining in this course) and preparation. You will need to prepare for the lab before you come to lab. Some of the experiments require extensive planning, so this part should be done **before** you come to lab.

Your laboratory notebook should contain the following sections.

Table 1: Notebook sections		
Sections	When completed	Comments
Table of Contents entry	after each day's work	
Purpose	prelab	
Introduction and theory	prelab	
Proposed Procedures	prelab	
Equipment Needed	prelab	
Samples and Reagents	prelab	
Flowchart of work	prelab	
Results	in lab	may include printed/attached info
Calculations	in lab and after lab	may include printed/attached info
Discussions	in lab and after lab	
Conclusions	after lab	maybe not apply to every experiment
Questions	after lab	maybe not apply to every experiment
Summary of Results	after lab	

1. Every Page should include

- Date
- title
- From (where experiment this page is continued from)
- to (where the next page for this experiment continues, but not filled out until next page is started)
- page number

2. Introduction (Complete before you come to lab) The purpose of this section is to show that you have digested the background information necessary (in this laboratory course this includes the lecture and textbook material) and that you can relate that material to the experiment, which you will perform. The introduction must contain some general theory of the particular technique and relate it to the specific experiment. An abstract of a discussion of theory is not sufficient nor is an account of the experiment itself. The introduction should be a synthesis of the two. Be sure to include a description of any chemistry involved along with pertinent chemical equations and any equations that allowed the measured quantity to be converted to the value or result of interest. If any instruments are used, a description of how they work is important. The introduction should be well-organized and divided into subsections with subheadings. The reader should be able to tell by looking at these headings how the material has been organized and what the writer feels is important to the subject.
3. Experimental (Complete before you come to lab and during lab) Since the lab manual is provided, it can be referenced. In this case, it is not necessary to rewrite the procedure, and therefore only changes in the procedure need to be described. Block diagrams of any apparatus must be included in this section. A block diagram is a diagrammatic drawing showing the basic components and their relationship to each other. Do not give a representational drawing (a sketch) of the apparatus.
 - (a) Data, Results, and Discussion In this section the results of the experiment are presented in tables and figures and explained in discussion. The tables and figures should be placed in close proximity to the text discussion of them. The discussion should be divided into topics with each topic having a subheading.
 - (b) Data When appropriate, data should be recorded in a tabular format. See Tables for information about creating tables.
 - (c) Tables Each table must be labeled (e.g., Table p17a [page 17 ath table you can leave the "p" off if you want]) and have a caption which describes concisely the content of the table (e.g., Conditions Employed for the Determination of Iron by Flame Atomic Absorption Spectrometry or calibration data (mass and temperature) for 25 mL pipet labeled p25k). Any further information necessary to an understanding of the table must be footnoted at the bottom of the table using appropriate superscripts and asterisks. All columns in a table must have headings and units indicated according to IUPAC standards.

Some tables can be created on a computer (such as summary results tables) and taped into your lab notebook. These tables must obey the same rules for table formatting and labeling as handwritten tables.
 - (d) Figures Figures must be labeled (e.g., Figure p17a [figure on page 17 ath figure]) and have a caption which indicates something about the nature of the data (e.g., Figure p17a: Calibration curve for the spectrophotometric determination of iron from data experiment 6 starting on data 2012-02-14 page 23 of this notebook). A caption such as "Absorbance versus Concentration" or "Lab 6 figure" is not appropriate. Also, the caption should indicate the page and location for the data from which it was derived. The axes must be labeled with units indicated using IUPAC standards.

Only when more than one data set is being plotted on a single graph is it necessary to use a legend. (1 set of data = no legend)

Grid marks are useful for reading data off of a graph, but you must have both horizontal and vertical directions for this usefulness to be realized. Graphs must be neatly constructed on a computer, and indicate the trend shown by the data points without connecting the markers. The only data types in which it is acceptable to connect the data markers with either smooth curves or straight lines is data with so many points that the data markers are touching adjacent markers on the graph. In this case use, no markers and use a spline interpolation to present the data as a smooth curve. Also for data that is used to indicate a trend or is fitted with a theoretical function, overlay the theoretical function with the fitted data. (This situation requires a legend: one for the data and one for the theoretical function.)

To insert a figure into your laboratory notebook, print it, cut it out and paste or tape it in your notebook. Indicate near the figure, the location and name of the computer file used to create the figure.

Use error bars in graphs of data whenever you have an uncertainty for a given point. Use the caption to describe the nature of the error bars, *e.g.* Figure p17a: Calibration curve for the spectrophotometric determination of iron from data experiment 6 starting on data 2012-02-14 page 23 of this notebook. Concentration axis error bars represent the magnitude uncertainty propagated from the lab equipment used. Absorbance axis error bars represent the standard deviation of 3 trials.

4. Discussion and Questions A discussion must include whatever is necessary to elucidate the material in the tables and figures to the reader. It is the link between these "illustrations" and the experiment itself. If the particular experiment asks questions, these are answered here. Make sure that the questions are restated, labeled and answered in proper sentences.
5. Data Analysis and Error analysis If appropriate, one of the subsections of the Results and Discussion section should be a statistical analysis where the value of the results is evaluated in terms of accuracy and/or precision. If the Standard Reference Material based value or literature value is known, a numerical accuracy should be calculated. A discussion of the factors contributing to the deviation from the true value is also appropriate for this section.

A confidence interval and level is always necessary when reporting results. If several trials were made, obtain a standard deviation and a t-distribution confidence interval to represent your uncertainty. If the results are from a function fitted set of data, report your uncertainty as a Standard Error of Fit (See Appendix for this calculation.).

What not to say in a Statistical Analysis. Don't say "This experiment went pretty well," or "The results were not so good." You will know how to calculate a numerical value for precision and also for accuracy if the "True" value is known. You can say exactly and in a quantitative fashion how good the results are. Remember in presenting data you have analyzed statistically give the mean, uncertainty, and number of measurements.

6. Conclusion or Summary of Results A brief set of statements summarizing the main points of the experiment should make up the conclusion along with a summary of the final results. In an analytical experiment, the conclusion is likely to be numerical. These numbers should be restated with their uncertainty and number of determinations.

0.1.4 Final Exam

Your Laboratory Notebook will be evaluated after every experiment is due. Also there will be a laboratory final examination in which you will be given a set of questions that you must answer from your laboratory notebooks.

0.1.5 Safety

This material will be covered at the beginning of lab, but in brief, eye protection, proper clothing and foot wear are required at all times during the performance of laboratory experiments. If you don't come to lab with proper personal safety equipment, you will be dismissed from the laboratory until you are prepared. A safety presentation will be given at the beginning of every new experiment, if you miss this presentation (because you are late) you will not be allowed to perform this experiment.

You must also complete and pass the safety quiz located on Blackboard. You can not attend lab until you pass the quiz.

0.1.6 Due Dates

Generally, all lab materials are due one week after the completion of experimentation. The exceptions to this general rule are final deadlines for work submission. These dates exist, because some people need a hard deadline to accomplish their tasks. The dates will be listed on the course website. Go there, retrieve them, and write them in the table below.

Laboratory Experiment	Due Date Week
1	TBA
2	TBA
3	TBA
4	TBA
5	TBA

0.1.7 Advice for Good Performance

1. Come to lab prepared. This means that you should read and **understand** the experimental procedures that you are going to perform in lab that day. It also means that you should perform or setup as many calculations for the day **before** you come to lab. How is this possible you might ask? Use a computer to setup calculations, so that you just have to fill in missing values (*e.g.* actual concentrations, formula masses, actual glassware volumes, etc.), and the calculations are quickly computed. Spreadsheets, such as LibreOffice Calc, MS Excel and Gnumeric, are extremely good at this task. This is all part of the prelab portion of the experiment. You can also simply setup the calculations on paper or in your notebook or program you calculator to perform these calculations.
2. Perform experiments in parallel. For example, if experiment A's procedure says to boil water for 1 hour, then let it cool to room temperature before adding reagent snifferpopjuice₇, and you wait around the lab for 2 hours (1 hour of boiling and 1 hour of cool down time) without doing anything else, then you have wasted 2 hours of your 3 hour work week. So what should you do? After setting the water to boil, you should work on experiment B. After 1 hour of boiling, set the water to cooling, and continue with experiment B or C or whatever.

3. General Laboratory Facilities and Guidelines: The laboratory is equipped with chemical work benches which have water, gas, air and vacuum outlets. There is one ventilated fume hood in which you can safely perform chemical procedures which liberate noxious gases and fumes. Top-loading balances for three decimal-place accuracy and four decimal-place accuracy analytical balances are located around the room.

(a) Reagents:

- The chemicals for the analyses will be found on the reagent table at the front of the laboratory.
 - Never carry reagents bottles away from the table. Bring beakers, flasks or graduated cylinders to the table.
 - Take only the quantity of reagent specified in the directions. Measure the quantities as carefully as possible. Carefully read the labels on the bottles before you take a chemical.
 - Never dip a scupula, stirring rod or medicine dropper into a reagent bottle. Transfer a small quantity of reagent to a beaker take the required amount from there. **Failure to obey this RULE will result in immediate dismissal from the lab!** This means that you will be behind in completing the experiments.
 - Always replace the lid on reagent bottles after using them.
 - Never return reagents to reagent bottles after you have removed them. Dangerous and/or annoying contamination may result. This is particularly important in an analytical laboratory where the purity of substances is so crucial. Give any excess to other students or discard.
 - If you spill reagents, clean them up immediately. If necessary rinse well with water.
 - When diluting concentrated acids, always add the acid to the water.
- (b) Care of the laboratory: Keep your bench area and reagent table clean and tidy at all times. At the end of the period, clean the apparatus and place it where it belongs in the room. Clean with a wet paper towel and dry your workstation.
- (c) Books and Clothing and Chairs: For safety reasons, keep the aisles clear so that your instructor and others can walk around the room. Therefore, store bags and extra stuff under the work benches. Keep chairs tucked under the work benches, when you are not sitting down. You should stand while performing all experiments in order to prevent spillage and splashes away from your face.

0.1.8 Types of Balances:

During the course you will be asked to make a variety of weighings. It is important for you to realize with what sort of accuracy these weighings should be made. Depending on the desired accuracy you should use the proper balance to make your weighings. There are two types of balances available to you in this course:

1. Top-loading balances. These will weigh to an accuracy of ± 1 mg and are suitable for most weighings of amounts that are specified to only two or three significant figures. These balances are next to the reagents at the front of the room.

2. Analytical balances These will weigh to an accuracy of ± 0.1 mg and must be used whenever you desire four or more significant figure accuracy. This will be the case when you weigh out samples of an unknown, primary standards or when taking crucibles to constant weight. These balances are around the edge of the room.

- (a) Various Types of Weighing: When mass amounts are specified in chemical procedures the following terms are commonly used:

- "Weigh out about 2 g of "

This statement means that you are required to weigh an amount of approximately two grams. The accuracy to which this mass amount needs to be known is not high and the top-loading balance will suffice.

- "Accurately weigh out about 0.2 g of"

This statement means that you should, with the aid of the analytical balance, weigh out an amount that is close to 0.2 g, but you must know the exact amount to an accuracy of ± 0.1 mg. Note that this does not mean that you must weigh out exactly 0.2000 g. An amount between 0.1900 g and 0.2100 g is perfectly acceptable. However, you must know the exact amount to the nearest tenth of a milligram. When weighing out triplicate samples it is not necessary that all three weights be exactly the same, indeed, it is poor procedure to attempt to do so.

- (b) Rules for Analytical Balances: The following rules summarize those procedures which must be followed in order to obtain accurate and reliable mass measurements with a single-pan analytical balance. Adherence to these rules will, at the same time, prevent damage to the balance.

For examples, of proper use of an analytical balance, read chapter 2 in your Harris text book, and watch the video linked from the course website.

- i. Close the balance door, while weighing an object, in order to prevent air currents from disturbing the reading. When finished, the operator should close the balance door to prevent dust and dirt from entering the balance.
- ii. Only glass, ceramic, metal or plastic objects and containers should be placed in direct contact with the balance pan.
- iii. Do not handle objects to be weighed with bare hands. Moisture, grease and dirt on your fingers will affect the weight of the objects.
- iv. To be weighed accurately, all objects must be at room temperature. A warm object sets up convection currents inside the balance enclosure, which will make an object appear lighter than it really is. Also, warm air inside the enclosure is less dense than the air that it displaces

and this also leads to a negative determinate error.

- i. Never weigh chemicals directly in contact with the balance pan. Use containers such as beakers, flasks and weighing bottles. **Failure to obey this RULE will result in immediate dismissal from the lab!**

This means that you will be behind in completing the experiments.

- i. All objects and materials that have recently been removed from a desiccator will absorb moisture and thereby gain weight. It is therefore good practice to record

weights after identical time intervals. For example if you are taking crucibles to constant weight. Always

record the weight of the crucible exactly 5 seconds after having placed the crucible on the balance pan. Using this technique it is possible to minimize the random effect of moisture absorption.

- i. The use of weighing paper must be strictly avoided when using an analytical balance.

Failure to obey this RULE will result in immediate dismissal from the lab!
This means that you will be behind in completing the experiments.

- i. Do not spill chemicals inside the balance enclosure. If a spill occurs, clean it up immediately. We have a brush for this purpose.

0.1.9 Laboratory Inventory

Each student will get a buret, 3 pipets and a bin of glassware.

1. Glassware bin Inventory

glassware	Notes
3 125 - 250 mL Erlenmeyer Flasks	
3 rubber stoppers that fit your flasks	
glass stir rod	
thermometer	
scupula	
pipet bulb with nylon tip	
long neck glass funnel	
4 50 mL volumetric flasks	
1 100 mL volumetric flask	
1 200-250 mL volumetric flask	
1 500 mL volumetric flask	
1 25 mL graduated cylinder	

2. Other glassware

pipets
1 25 mL
1 10 mL
1 5 mL
burets
1 50 mL

0.1.10 University Policies

1. Academic Integrity Policy and Reporting Process

This policy addresses academic integrity violations of undergraduate and graduate students. Graduate students should read inside the parenthesis below to identify the appropriate entities in charge of that step of the process.

Students, faculty, staff, and administrators of Western Carolina University (WCU) strive to achieve the highest standards of scholarship and integrity. Any violation of the Academic Integrity Policy is a serious offense because it threatens the quality of scholarship and undermines the integrity of the community. While academic in scope, any violation of this policy is by nature, a violation of the Code of Student Conduct and will follow the same conduct process (see Article VII.B.1.a.). If the charge occurs close to the end of an academic semester or term or in the event of the reasonable need of either party for additional time to gather information timelines may be extended at the discretion of the Department of Student Community Ethics (DSCE).

I. General

This policy addresses academic integrity violations of undergraduate and graduate students. Students, faculty, staff, and administrators of Western Carolina University (WCU) strive to achieve the highest standards of scholarship and integrity. Any violation of this policy is a serious offense because it threatens the quality of scholarship and undermines the integrity of the community.

Instructors have the right to determine the appropriate academic sanctions for violations of the Academic Integrity Policy within their courses, up to and including a final grade of “F” in the course in which the violation occurs.

II. Definitions

- 1 Cheating – Using, or attempting to use, unauthorized materials, information, or study aids in any academic exercise.
- 2 Fabrication – Creating and/or falsifying information or citation in any academic exercise.
- 3 Plagiarism – Representing the words or ideas of someone else as one’s own in any academic exercise.
- 4 Facilitation – Helping or attempting to help someone to commit a violation of the Academic Integrity Policy in any academic exercise (e.g. allowing another person to copy information during an examination).

III. Undergraduate and Graduate Academic Integrity Process

1. Within five (5) business days of the instructor’s knowledge of the alleged violation of the Academic Integrity Policy, s/he will inform his/her department head (or associate Dean of the graduate school when applicable) in writing of the allegation and proposed sanction(s).
2. Within ten (10) business days of the instructor’s knowledge of the alleged violation of the Academic Integrity Policy, the instructor will inform the student of the allegation, including the proposed sanction(s), in writing. In the written notification, the instructor will inform the student of his/her right to request a meeting with the instructor. During the meeting, the instructor shall complete the Academic

Integrity Violation Faculty Resolution Form. If the student does not request a meeting with the instructor within five (5) business days of receipt of the written allegation(s), the student shall be deemed to have mutually resolved the matter and shall be bound to the sanction(s) outlined by the instructor in the written allegation. If the student does not request a meeting, the alleged violation of the Academic Integrity Policy shall not be subject to further review and/or appeal.

3. Within five (5) business days of meeting with the instructor, the student shall either appeal the decision to the department head or mutually resolve the matter by accepting the allegation and proposed sanction(s). No action by the student within five (5) business days of the meeting with the instructor shall constitute a mutual resolution and waiver of the student's rights to appeal pursuant to the Academic Integrity Policy. If the student does not respond within five (5) business days of meeting with the instructor, the alleged violation of the Academic Integrity Policy shall not be subject to further review and/or appeal.
4. Within five (5) business days of receiving a student's appeal, the department head must schedule a meeting with the student. The instructor may be present during the meeting. During the meeting, the department head shall complete the Academic Integrity Violation Department Head Resolution Form. Only information submitted during the meeting with the student, or in the meeting between the instructor and the student, may be considered by the department head. The evidentiary standard for making a decision shall be preponderance of the evidence. The department head may agree or disagree with the allegation(s) of the instructor. The department head may also approve, overturn, or modify the sanction(s) proposed by the instructor. If the student does not attend the scheduled meeting with the department head, the matter will be heard in absentia and shall not be subject to further review and/or appeal.
5. Within five (5) business days of meeting with the department head, the student shall either appeal the decision to an Academic Integrity Board or mutually resolve the matter by accepting the allegation and proposed sanction(s). The student must submit an appeal to the academic Dean listed on the Academic Integrity Violation Department Head Resolution Form. No action by the student within five (5) business days of the meeting with the department head shall constitute a mutual resolution and waiver of the student's rights to appeal pursuant to the Academic Integrity Policy. If the student does not respond within five (5) business days of meeting with the department head, the alleged violation of the Academic Integrity Policy shall not be subject to further review and/or appeal.
6. Within seven (7) business days of receiving a student's appeal, the appropriate academic Dean must schedule an Academic Integrity Board hearing with the student. The Academic Integrity Board shall consist of a minimum of two (2) currently enrolled students and/or faculty members (with a minimum of one faculty member). A faculty member will serve as chair of the board. The instructor may be present during the hearing. Only information submitted during the hearing, or in the meetings between the instructor/department head and the student, may be considered by the hearing board. The evidentiary standard for making a decision shall be preponderance of the evidence. The hearing board may agree or disagree with the allegation(s) of the instructor. The hearing board may also approve, overturn, or

modify the sanction(s) proposed by the instructor and/or department head. If the student does not attend the scheduled hearing, the matter will be heard in absentia and shall not be subject to further review and/or appeal. Within ten (10) business days of the hearing, the appropriate academic Dean shall review pertinent records and send the student written notification of the decision of the Academic Integrity Board.

7. Within five (5) business days of receiving written notification of the decision of the Academic Integrity Board the student may accept the findings and sanctions of the board or submit an appeal to the designated academic Dean. No action by the student within five (5) business days of the meeting with the department head shall constitute a mutual resolution and waiver of the student's rights to appeal pursuant to the Academic Integrity Policy. If the student does not respond within five (5) business days of meeting with the Academic Integrity Board, the alleged violation of the Academic Integrity Policy shall not be subject to further review and/or appeal.
8. If the student elects to file an appeal of the decision of the Academic Integrity Board, she must submit a written appeal within five (5) business days of receiving written notification of the decision of the Academic Integrity Board to the designated academic Dean. An appeal to an academic Dean must be limited to the following grounds; 1) a violation or due process or 2) a material deviation from Substantive and Procedural Standards by the UNC Board of Governors (as set forth in the UNC Manual 700.4.1).
9. If an appeal is heard by an academic Dean, s/he shall review pertinent records within ten (10) business days of receiving a valid appeal. The academic Dean may agree or disagree with the allegation(s) of the instructor. The academic Dean may also approve, overturn, or modify the sanction(s) proposed by the instructor, department head, and or Academic Integrity Board. Within five (5) days of making a decision, the academic Dean shall provide the student with a written decision. The decision of the academic Dean shall be final.
10. The student must remain enrolled in the course related to the case, and may not be permitted to withdraw from the course related to the case, until all hearing timelines, notifications, and/or appeals have been completed.
11. Upon resolution of each level of the case (no matter the outcome), the instructor, department head, and academic Dean must provide the Department of Student Community Ethics with all materials and documents related to the case (i.e. course syllabus, materials in violation of the Academic Integrity Policy, Instructor Resolution Form, Department Head Resolution Form, Academic Integrity Board decision letter, academic Dean decision letter, etc...). The Department of Student Community Ethics shall serve as the repository for all records associated with allegations and violations associated with the Academic Integrity Policy.

IV. Academic Integrity Board

The Academic Integrity Board shall consist of a minimum of two (2) currently enrolled students and/or faculty members (with a minimum of one faculty member). A faculty member will serve as chair of the board. Students and faculty members serving on boards for each college will be selected by each college Dean. The Department of Student Community Ethics will train all board members prior to their service on a

hearing board. Each academic Dean will convene hearing boards as necessary, and will determine a faculty member to serve as chair prior to a hearing.

V. Sanctions

The instructor, department head, Academic Integrity Board, and/or academic Dean may impose academic sanctions permitted by the institution (not to exceed receiving a grade of “F” for the course). The instructor, department head, Academic Integrity Board, and/or academic Dean may not permanently remove the student from the course or suspend/expel the student from a program or the University. Student behavior of the magnitude to warrant consideration for permanent removal from the course or suspension/expulsion from a program or the University must be referred to the Department of Student Community Ethics.

VI. Habitual Violations of the Academic Integrity Policy

Upon receipt of materials associated with violations of the Academic Integrity Policy, the Department of Student Community Ethics will determine if a student has previous violations of University policies. Students with a prior record of violations, or who commits a gross and/or egregious violation of the Academic Integrity Policy, will be referred to the Department of Student Community Ethics for consideration of being subject to hearing proceedings as a habitual violator. Students with three or more violations of the Academic Integrity Policy will automatically be subject to hearing proceedings as a habitual violator. Students in this category are subject to course-related sanctions imposed by the instructor, department head, Academic Integrity Board, and/or academic Dean and University-level sanctions imposed by the Department of Student Community Ethics for habitual violations of University policies. Additional information is available on the Student Success website under Student Community Ethics.

2. Inclement weather policy:

The University does not, as a matter of general practice, close its operations or cancel classes in Cullowhee because of bad weather. Many Western students commute from different distances and directions and weather conditions for those students may vary greatly from conditions on the Cullowhee campus. Students are advised to check road conditions in their areas and determine whether it is reasonable for them to drive to campus. *The University expects students to make every effort to attend class but not to jeopardize their safety by driving during dangerous conditions.* Faculty members will accommodate those students who are unable to attend class because of hazardous weather conditions.

1 Introduction to and Calibration of Laboratory Glassware

1.1 Equipment and Materials needed

Equipment	Comment
25 mL pipet	
10 mL pipet	
5 mL pipet	
pipet holder	
pipet bulb	
DI water	
3 Erlenmeyer flasks	
3 stoppers	

1.2 Purpose

- To learn how to use some laboratory equipment
- To learn about calibration of laboratory equipment
- To learn about accuracy and precision
- To learn and practice recording information in your laboratory notebook

1.3 Background and Prelab

Before you come to class you should read Chapter 2 in your Harris textbook. You should also watch the lab equipment videos listed on the class website.

1.4 Lab Skills

Your goal with each of these exercises is to learn to properly use some of your lab equipment. For this experiment, you will learn to use an analytical balance and volumetric pipets.

Since your grade for this experiment will depend heavily on your notebook entry (You must do a good job in your notebook to get a good grade.), take your time and record your data correctly. Specifically treat each subsection as a separate experiment. This means that it should contain a brief sentence or two about why you are performing the experiment. Also each data table needs to be explained. Specifically, you should write a sentence or two describing what data is in the table, and what (including the label) equipment was used to record it.

In the example laboratory notebook on the course website, is an example the calculations for this lab exercise, including summary results page. Use this example as a template for this and the remaining lab exercises this semester.

1.4.1 Using a Volumetric Pipet

The first thing that you should do is to label each of your pipets. Each pipet should be the following label:

Last, First
R or T

S or F yyyy

An example for Thursday's lab Lucy Manwich's 25 mL pipet is
Manwich, Lucy
R S2015

We will use the calibrations that you generate during this part of the laboratory exercise for the remainder of the experiments this semester that require these pipets. You should read section 2-6 in your Harris textbook and watch the video before you come to lab.

Obtain a sample of DI water and let it come to room temperature. Before you begin pipeting, record the temperature of the water.

Accurately, measure the mass of 3 small erlenmeyer flasks and stoppers (this is the tare of the flask) and record this mass in tabular form. Keep each stopper with its flask. If there are not 3 flasks and stoppers, then get some from the glassware cabinets. Remember to wear gloves when handling equipment that needs to be accurately weighed; fingerprints have mass.

Select a volumetric pipet 25 mL in volume, record its label, type and tolerances in your laboratory notebook. If it does not have a label tell your instructor, and he or she will assign it one. Note if it does not have a tolerance listed on it, then assume that it is a Class A pipet and look up its tolerance in Table 2-4 of your Harris textbook.

By following the instructions on page 38 of your Harris textbook, pipet using a volumetric transfer pipet the appropriate amount of DI water into your tared flask. Note that all pipets used in this laboratory are TD types. Re-measure the mass of the flask and stopper using the same balance and record the mass in tabular form in your laboratory notebook. Remember to record the units in your table. An example table is provided here.

Table 2: Mass and volume measurements of DI water (all mass measurements have an uncertainty of ± 0.0001 and unit of grams)

Trial	Temperature of water /°C	pipet volume label (± 0.03) /mL	mass of flask	mass of flask and DI water	mass of water	estimated volume of water delivered /mL
A		25	25.1082	50.1038	25.1234	
B		25	25.1087	50.1038	25.1234	
C		25	25.1080	50.1038	25.1234	
					mean →	
					STD →	

Note: for the last column, during the lab period assume that the density of water is 1.0 g/mL. This is not correct but it will allow you to correctly determine the standard deviation of the volumes.

Calculate the corrected volume at 20° C of DI water that you actually delivered taking into account the air and water temperature, density, buoyancy correction, and mass of water delivered into your tared flask using the instructions on page 42-43 and Table 2-7 of your Harris textbook. Determine the mean calculated volume derived from these mass measurements and standard deviation of each sample using equations 4-1 and 4-2 from the textbook by Harris. Record this as part of your laboratory notebook.

Repeat this volume delivery until three consecutively delivered volumes have a standard deviation of less than the tolerance of your pipet. This is your chance to practice using a pipet without the consequence of earning a poor grade; so learn well.

Repeat this procedure for a 10 mL and a 5 mL pipet. (This means that you will need a new table for each pipet.)

1.5 Clean up

Put all glassware and other labware where you found it. Put waste in the waste containers in the near the fume hood.

1.6 Reporting your work

Remember to have a section in you lab notebook Labeled Summary Results. In this section, include all of you corrected volumes and uncertainties of the glassware that you have calibrated in this laboratory exercise.

1.6.1 Use the spreadsheet to report your work.

When reporting the mean and standard deviation of the volumes of your pipets, only use the three consecutive measurements that were below the tolerance of the glassware. Submit your filled out reporting spreadsheet via blackboard.

1.6.2 Write the questions and the answers in your lab notebook.

1. Your mass of water is converted to the true volume using data from Table 2-7 of Harris. What are the three corrections that are embodied in those values?
2. When the glass of a pipet expands due to an increase in temperature does the diameter of the bore increase or decrease?
3. Most volumetric glassware is calibrated at what temperature?
4. What do the letters T.D. and T.C. that are found on various types of volumetric glassware, signify?
5. Before pipetting you DI water, you were supposed to let the water come to room temperature. Why? How can you tell if your water has come to room temperature?
6. What is the error caused by not drying the Erlenmeyer flask, but re-taring the wet Erlenmeyer flask for each new pipet addition.

2 The Determination of Magnesium by Volumetric Analysis

2.1 Background and Prelab

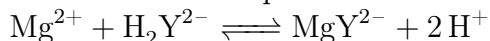
Watch the appropriate videos on titration linked to at the course web page. Read section 2-4 in the Harris text book.

- Set up a calculation for estimating the amount of EDTA that you will need for standardizing the EDTA in Section 2.4.2

2.2 Introduction:

Many metal ions react with electron pair donors to form coordination compounds or metal-ligand complexes. The formation of a particular class of coordination compounds, called chelates, are especially well suited for quantitative methods. A chelate is formed when a metal ion coordinates with two (or more) donor groups of a single ligand. Tertiary amine compounds such as ethylenediaminetetraacetic acid (EDTA) are widely used for the formation of chelates. Complexometric titrations with EDTA have been reported for the analysis of nearly all metal ions.

Because EDTA has four acidic protons, the formation of metal-ion/EDTA complexes is dependent upon the pH. For the titration of Mg^{2+} , the solution is buffered to a pH of 10 so that complex formation will be quantitative. The reaction of Mg^{2+} with EDTA may be expressed as:



The structure of EDTA and the magnesium-EDTA complex (without the hydrogen atoms) is shown below:

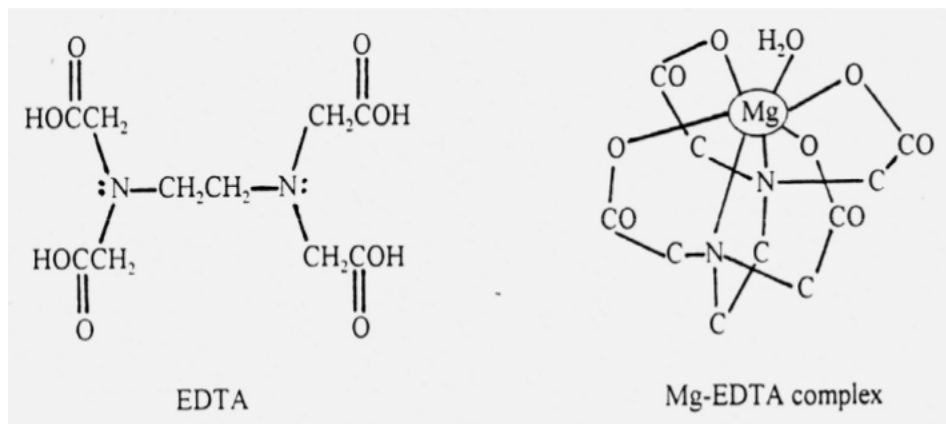
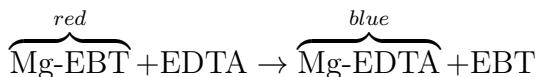


Figure 1: EDTA and EDTA-Mg complex

The endpoint of the titration is determined by the addition of Eriochrome Black T (EBT), which forms a colored chelate with Mg^{2+} and undergoes a color change when the Mg^{2+} is released to form a chelate with EDTA.

The reaction for the titration is essentially



While it is possible to achieve relatively good results by titration with EDTA prepared directly from the solid, better results can be obtained when the EDTA is standardized against a solution containing a known amount of metal ion. You will be provided with a standard solution of Zn^{2+} , which you will use to standardize your EDTA solution.

2.3 Reagents and Apparatus

- EDTA ($\text{Na}_2\text{H}_2\text{Y} \cdot 2\text{H}_2\text{O}$) (HINT: Get the FW from the bottle)
- pH 10 buffer (This has been prepared for you) (2000 mL has been prepared by dissolving 140.0 g of NH_4Cl in 650 mL of deionized water, adding 1140 mL of conc. ammonia and diluting to 2000 mL)
- Eriochrome Black T (this has been prepared for you.)

(100 mL of solution has been prepared by dissolving 0.5 g of the Eriochrome Black T in a solution containing 4.5 g of hydroxylamine hydrochloride in 95% ethanol.)

- pHydrion paper
- 50 mL of Standard Zn solution: (This has been prepared for you.)

An accurate mass (about 1.3g) of pure zinc has been dissolved in a small volume (~ 15 mL) of 6M HCl. The dissolved zinc was quantitatively transferred to a 2 L volumetric flask and diluted to the mark. This ~ 0.01 M (you need to find the exact concentration, by reading the bottle's label!!) solution serves to standardize the EDTA solution. Remember that the results from your magnesium determination depend on the accurate determination of the concentration of this solution. Each student will be dispensed ~ 50 mL of this solution for each titration.

- 25 mL unknown magnesium solution

2.4 Procedure

First read the section on Techniques below. Create a table at the beginning of this experiment, in which you list the calibrated volumes of your three pipet $\pm$ std. Use these volumes in your calculations.

2.4.1 PART A – Preparation of Standard 0.01 M EDTA Solution (note: this is the formal concentration of EDTA)

Weigh about 1.9 g of the disodium EDTA salt ($\text{Na}_2\text{H}_2\text{Y} \cdot 2\text{H}_2\text{O}$) (what balance should you use?) into a 0.5 liter volumetric flask, dissolve and dilute to the mark with deionized water and mix well. Calculate the approximate concentration of EDTA based upon you mass and volume measurements in this part and label it as **approximate**.

2.4.2 PART B – Standardization of the EDTA Solution

Pipet exactly 4 or 5 mL of standard Zn solution into each of your three Erlenmeyer flasks. This means that you have to perform this careful titration at least 3 times). To each Erlenmeyer flask, add 1 to 2 mL of pH 10 buffer, 20 mL of water, stir, and add a few drops of the Eriochrome Black T indicator solution. It is critical to add only enough indicator to produce a light wine-red color.

From you approximate concentration of EDTA that you calculated in Part A and the concentration of Zn solution, estimate the volume of EDTA that you will need. Perform a rough titration to confirm this estimation.

Perform careful titrations with your EDTA solution until the color changes from pink to a clear blue. Record the total volume of EDTA required to reach the end point to the nearest 0.01 mL. If all experimental parameters are the same, the standard deviation of your three careful titration should agree to within the tolerance of the buret. If they are worse you can perform several additional titrations to improve your standard deviation (N measurements can be > 3).

Use these results to determine the molar concentration of the EDTA solution for use in the titration of your unknown Mg solution. Calculate the average and standard deviation of the concentrations EDTA using these titrations. Use Grubbs test for any outliers in your determined EDTA concentrations. Your mean and uncertainty must be represent at least 3 titrations.

For quality control and assurance, your mean concentration should be similar to your approximate EDTA concentration that you calculated from Part A. If it is not then you have a problem, fix it.

2.4.3 PART C: Determination of Unknown Magnesium

Record the unknown label. Your unknown for this experiment is a solution. When you obtain your unknown, you need to quantitatively transfer exactly 10 (or 9 depending on your medium sized calibrated pipet) mL of it to a 250 mL volumetric flask and dilute it to the mark with DI water, resulting in the **prepared** or **diluted** unknown solution. You are to report the results of the unknown that you received (don't forget to back calculate the dilution). Transfer exactly 20 mL (or 25 mL depending upon your largest calibrated pipet) of the **prepared** unknown solution into your Erlenmeyer flasks. Add approximately 1-2 mL of pH 10 buffer and 20 of distilled, deionized water to each Erlenmeyer flask. (Knowing the amount of DI water added that the previous step is not critical.) Add a few drops of Eriochrome Black T indicator – it is crucial that you only add enough indicator to produce a light, pink color. Titrate one solution with your standardized EDTA solution roughly to estimate the end point of the titration. This is an estimation titration only and not to be used in your calculations of the concentration of the unknown. Carefully, titrate the remaining 3 solutions of unknown with your standardized EDTA solution to a clear blue color. Report your results of each carefully titrated unknown in molarity of magnesium in your received unknown sample. Also include a mean concentration determined from your 3 careful titrations and report the 95% confidence interval for your uncertainty of the determined concentration.

2.5 Information, Hints and Help for Mg Determination

2.5.1 Preparation:

- To prepare the standard EDTA solution used in this experiment, you must first be aware that the structure of the EDTA listed in your text and lab manual is not the compound you will be weighing out to make the solution. The salt you will use is the disodium salt of EDTA. The formula weight can be obtained from the bottle.
- Since you are going to standardize the EDTA solution with a standard Zn solution, you need not dry the EDTA.
- After making your EDTA solution, if you wish to keep some for an additional lab period, store it in a plastic bottle, not a volumetric flask.

- The Zn solution used for standardization may be prepared for you in advance. Consult your lab instructor for this information. Be sure to note the actual concentration of the Zn solution, as it will not be exactly 0.01 M.

2.5.2 Technique:

- Eriochrome Black T exists as a pink complex when Mg^{+2} is present in solution at $\text{pH} = 10$. When the EDTA has chelated all the Mg^{+2} present in the solution, the indicator (free and uncomplexed to Mg^{+2}) will be robin's egg blue. This color change marks the endpoint. Addition of only very small quantities of the indicator are needed. Do your best to keep the intensity of the indicator color relatively weak and consistent from sample-to-sample.
- It is best to test the indicator color change before trying the titration. Try it either with tap water or a dilute Mg^{+2} solution. Using a sample with a small amount of Mg^{+2} and performing a low volume sample titration will help you understand the endpoint changes.
- Whenever you are performing a titration, you can speed up the entire process by first performing an estimation titration. This procedure involves performing the titration in 1 mL increments until the end point is reached. This end point volume should be an estimate of the volume needed to perform a more careful titration. Once you have this estimation, during your careful titrations you can add most of the volume quickly, until you are about 2 mL away from the estimated end point volume. Then you can perform the remainder of the titration very carefully by using 1 drop at a time near the end point. When you are very near the end point, use less than a drop at a time. To do this, carefully suspend a fraction of a drop from the buret tip and touch it to the inside wall of the flask. Carefully tilt the flask so that the bulk solution overtakes the droplet and then swirl the flask to mix the solution.
- When titrating ensure that the solution is well mixed after each buret addition.

2.5.3 Calculations and Data Evaluation

If the results of one of your titrations looks like an outlier, test it with Grubbs test. If you can statistically (95% confidence level) reject one titration, repeat it to get 3 good titrations.

2.6 Reporting your work.

Use the spreadsheet reporting tab to report your work. In Excel do not put units into the same cell as the numbers. Write the following questions with answers in your laboratory notebook.

1. What is the indicator used in this experiment?
2. How else might you determine the end point of this titration (you will have to read your textbook for this one.)?
3. What was the Zinc used for in the experiment?
4. What advantage does using Zn in this experiment have over other techniques utilizing EDTA titrations that might be used?
5. Why do you only use a few drops of Eriochrome Black T?

6. Describe the error that adding 25 mL instead of 20 mL of water to your unknown sample of Mg in the Erlenmeyer flask, before titrating.

3 Method Development: Stoichiometry Ratio Determination of a Dye-Metal Ion Complex

3.1 Equipment and Materials needed

Item	notes
SpectroVis spectrometer	
iron salt	Ferrous ammonium sulfate
dye	1,10-phenanthroline
	hydroquinone
computer	
2 cuvettes	
beaker for waste	

3.2 Purpose

- To learn how to use a visible absorbance spectrometer
- To learn how to use Beer's Law
- To learn how to fit linear data
- To learn about Job's method of Continuous Variation

3.3 Background: Method of Continuous Variations¹ (Job's Method)

Complexation reactions of the form



are based on the reaction of a metal cation (M) and a ligand (L). These reactions are widely used in analytical chemistry. Absorption spectroscopy is a powerful tool for exploring these complexation reactions. In this experiment, a general approach to studying the composition of complexes is used to demonstrate the necessity of carefully evaluating the properties of a particular chemical system in order to select the best method for determining the composition (metal to ligand ratio) of a complex by absorption measurements.

In this method, metal cation and ligand solutions with identical concentrations are mixed in different amounts such that the total volume of the mixture solutions and the total moles of reactants in each mixture is constant. This procedure causes the mole ratio of reactants to be varied across the set of mixture solutions. The absorbance of each solution is then measured and plotted vs. the mole fraction of one of the reactants (M or L). For example, the mole fraction of the metal is

$$\frac{Mole_M}{Mole_M + Mole_L} \quad (2)$$

where $Mole_M$ is the moles of the metal cation in the solution and $Mole_L$ is the moles of the ligand in the solution.

A plot of this type is reproduced above in Figure 2. Assuming the complex absorbs more than the reactants, a maximum occurs at a volume ratio $\frac{Mole_M}{Mole_L}$ corresponding to the combining ratio

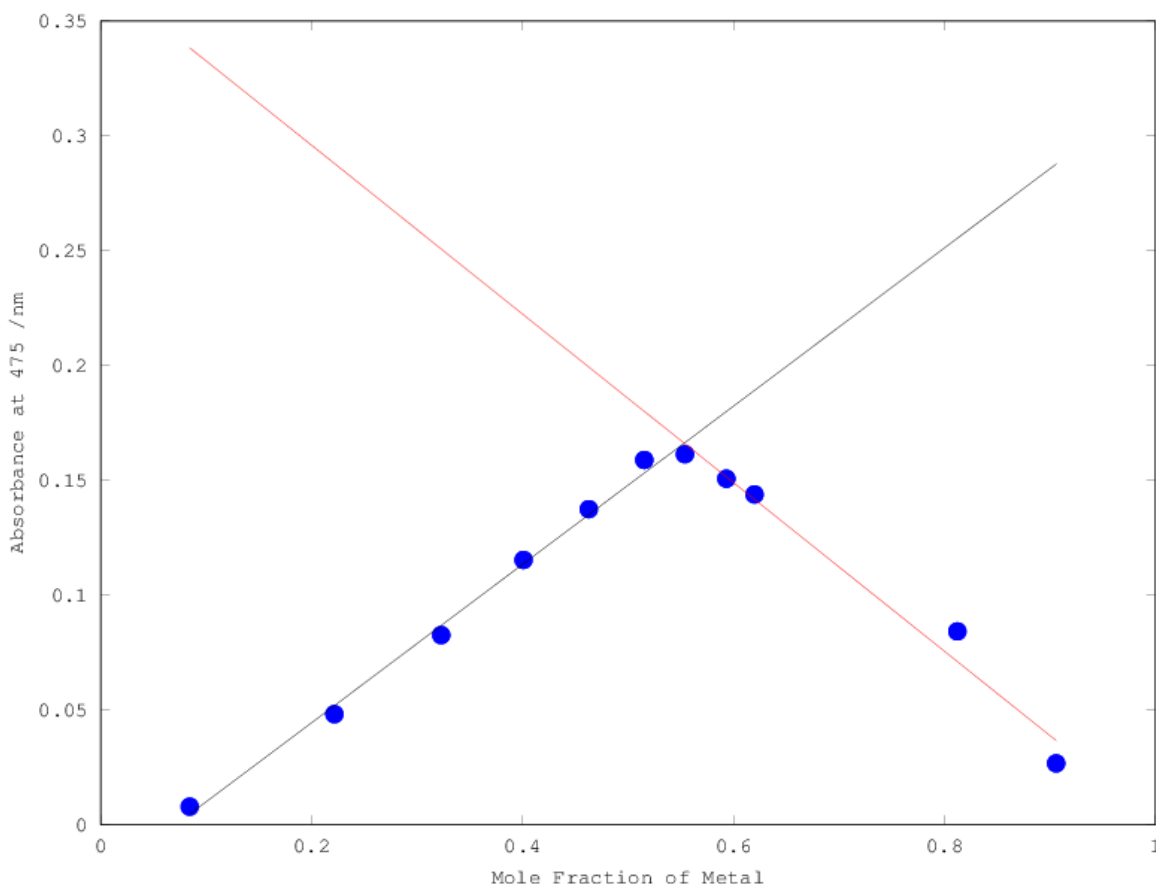


Figure 2: Continuous-variation plot for a 1:2 complex, ML_2 .

of cation and ligand in the complex. At other volume ratios, one of the reactants is a limiting reagent. In the plot in Figure 2, this maximum occurs at $\frac{Mole_M}{Mole_L} = \frac{0.5}{0.5}$, suggesting a complex of the formula ML .

3.3.1 Stock Solutions

Prepare enough of your stock solutions to complete the entire experiment.

- Ferrous ammonium sulfate ($(NH_4)_2Fe(SO_4)_2$), $\sim 7.0 \times 10^{-4}$ M (note 1: Adjust this concentration to be close to the actual 1,10-phenanthroline concentration. note 2: check the bottle for the actual formula weight.)
- 1,10-phenanthroline (FW = 198.23), $\sim 7.0 \times 10^{-4}$ M (This has been prepared for you. Check the bottle for the actual concentration.)
- pH 4.0 buffer
- hydroquinone, 5 % (w/v = g/mL) (This has been prepared for you.)

3.3.2 Measurement Solution Preparation and Measurement

All of your measurement solutions need to be made in the same size volumetric flasks. This procedure describes using a 50 mL flask. We don't necessarily have enough of these for the entire

lab, so adjust your solution preparations to match the glassware that you have.

The software for our spectrometers uses the word calibrate instead of reference or blank. Be mindful of these differences.

1. **Prepare the stock solutions.** Make the phenanthroline solutions first. ^a
2. **Prepare the buffer** Use the pH 4.0 tablets as per the instructions on the bottle.
3. **Absorbance Spectra of Stock Solutions.** Record the absorbance spectrum of each of your stock solutions vs. a distilled water reference.
4. **Method of Continuous Variations solutions** Using 2 burets (one with the 1,10-phenanthroline stock solution and one with your ferrous ammonium sulfate stock solution) transfer 0, 1, 2, 3, 4, 5, 6, 7, 8, 9, and 10 mL of the standard Fe^{2+} solution to separate 50 mL volumetric flasks. Add 5 mL of the buffer solution followed by 1 mL of the hydroquinone solution. To each flask, respectively, add 10, 9, 8, 7, 6, 5, 4, 3, 2, 1, and 0 mL of the 1,10-phenanthroline stock solution. Dilute to the mark with distilled water, and mix. It is not necessary to use these exact amounts of iron and phenanthroline, what is important is that the volumes are close and are well known.
5. Prepare a spectroscopic blank containing no iron or phenanthroline by adding 5 mL of the pH 4.0 buffer and 1 mL of the hydroquinone solution to a 50 mL volumetric flask and diluting to the mark with distilled water.
6. **Measurement of Spectra.** After ten minutes, record an absorbance spectrum of the 5 mL Fe^{2+} - 5 mL 1,10-phenanthroline solution (vs. your spectroscopic blank). Measure the absorbance of each solution in triplicate.

3.3.3 Data Analysis

1. Determine the wavelength of maximum absorbance of the complex by visually comparing the stock solutions and the 5:5 mixture spectra. Find a wavelength for the complex that has a maximum absorbance while possessing minimum overlap with any absorption bands due to the chemical species present in the stock solutions. Use this wavelength and its corresponding absorbance values for all further calculations.
2. The procedure below will produce a Job's plot with linear portions that intersect. For the plot, associate each point with either the left or right linear portions and perform two least-squares calculations to estimate the linear equations ($y = mx + b$) that represent the left and right linear portions of the plot. Slopes, intercepts, and values of R^2 should be tabulated for each of these least-squares calculations. The intersections of the lines can be found mathematically by finding the point on the x-axis where the lines have the same y-value (i.e., setting the equations equal to each other and solving for x). All plots should have error bars based on the 95% confidence intervals of the triplicate absorbance measurements.
3. Plot absorbance vs. mole fraction of Fe^{2+} . Extrapolate the linear portions of the curve and locate the intersection by use of the mathematical procedure described above.
4. Postulate the formula for the complex and draw its structure.

^aThis has been done for you

3.4 Clean up

Dispose of all waste appropriately.

3.5 Reporting your work

Use the spreadsheet reporting tab to report your work. Write the following questions and their answers in your laboratory notebook.

1. What would be the effect your determination stoichiometric ration, if you included point A in to the best of fit line C?

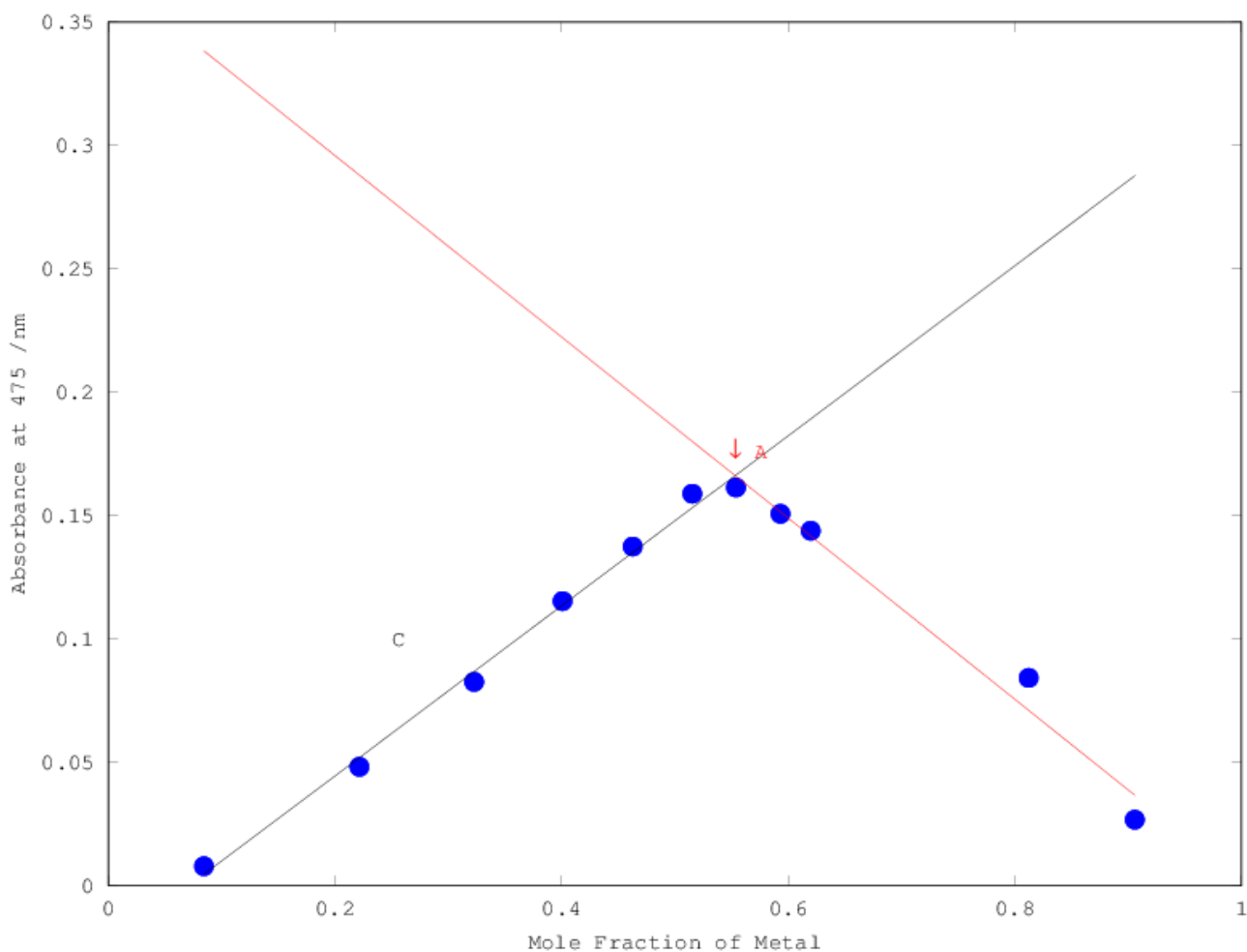


Figure 3: Job's Plot of and fitted data

1. Why do you "calibrate" the spectra using a blank instead of DI water. What is the effect on the determined stoichiometric ratio? Why?

4 Determination of the Identity, Concentration and Hydration Number of a Weak Base

4.1 Introduction and Background

In this experiment you will use a pH electrode to follow the course of an acid-base titration. You will observe how pH changes slowly during most of the reaction and rapidly near the equivalence point. You will create a Gran plot of the titration curve to determine the equivalence point of the titration and equilibrium constant of the conjugate acid. You will use the equilibrium constant to determine the identity of the weak base. From this ID and the equivalence point information of the titration you will predict the hydration number of the weak base. Section 10-5 (specifically pages 217-218) of the textbook provides background for this experiment.

Whenever solids are precipitated from aqueous solutions and depending upon the conditions of the precipitation, they can contain water molecules that are incorporated into the crystal structure. Evidence of this situation can be found on the chemical names on labels of chemicals purchased from chemical manufacturers and the existence of the formula weight (or formula mass) of these substances often abbreviated FW or FM. In a material in which water molecules are part of the crystal structure, the substance is called a hydrate, *e.g.* sodium phosphate hexahydrate ($\text{Na}_3\text{PO}_4 \cdot 6\text{H}_2\text{O}$), which has 6 water molecules per molecule of sodium phosphate (hydration number is 6). One of your goal of this experiment is to use the titration data with the mass of unknown base to determine the hydration number.

4.1.1 Gran Plot

Read pages 217-218 in the textbook. For now you can ignore the symbols γ ; we will cover this material later in the semester. Your experiment will be the titration of a weak base by a strong acid. That means that your Gran plot will look like the following (eqn 10-6 in Harris):

$$V_a 10^{pH} = \frac{1}{K_a}(V_e - V_a) \quad (3)$$

where V_a is the volume of the acid, K_a is the equilibrium constant of the acid dissociation chemical reaction, V_e is the equivalence volume.

4.2 Equipment and Materials needed

- Standardized 0.1 M HCl (This has been prepared for you.)
- Bromocresol green indicator: See Table 10-6 in the textbook for additional information.
- pH calibration buffers: pH 7 and pH 4.
- Unknown base: stored in a desiccator
- computer with LoggerLite installed
- pH probe
- LabPro ADC

- powerblock for LoggerPro
- power splitter for all of the electronics
- buret, clam and stand
- becker for titrations
- a stir plat with stir bar

4.3 Procedure

4.3.1 Weigh out accurately the Instructor specified amount of your unknown base.

Dissolve it in distilled water in a 250 mL volumetric flask. Dilute to the mark and mix well. This solution is your unknown solution.

4.3.2 ph meter/probe calibration

Following instructions for your particular pH meter, calibrate a meter and glass electrode, using buffers with pH values near 7 and 4. Rinse the electrodes well with distilled water and blot them dry with a tissue before immersing in any new solution.

4.3.3 Rough titration

The first titration is intended to be rough, so that you will know the approximate end point in the careful titration. For the rough titration, pipet 25.0 mL of unknown solution into a 125 mL flask. Add 3 drops of bromocresol green to your 125 mL flask indicator and titrate with standardized 0.1 M HCl to the green end point. Add 0.5 mL of titrant at a time so that you can estimate the equivalence volume to within 0.5 mL. Near the end point, the indicator temporarily changes color as titrant is added. If you recognize this, you can slow down the rate of addition and estimate the end point to within a few drops.

4.3.4 Careful titration setup

Now comes the careful titration. Pipet 25.0 mL of unknown solution into a 250 mL beaker containing a magnetic stirring bar. Position the beaker on top of a magnetic stir plate. Clamp the electrode(s) in the liquid so that the stirring bar will not strike the electrode. Allow the electrode to equilibrate for 1 min with stirring and record the pH.

4.3.5 Careful titration

Add 1 drop of indicator and begin the titration. The equivalence volume will be very near the volume determined in Step 4.3.4. Add about 1.5 mL aliquots of titrant and record the exact volume, the pH, and the color 30 s after each addition. When you are within 2 mL of the equivalence point (as determined in STEP 4.3.4), add titrant in 2-drop increments. When you are within 1 mL of the equivalence point, add titrant in 1-drop increments. Continue with 1-drop increments until you are 0.5 mL past the equivalence point. The equivalence point has the most rapid change in pH. Add five more 1.5 mL aliquots of titrant and record the pH after each.

4.4 Data Analysis

4.4.1 Gran plot setup

Construct a Gran plot according to the instructions on page 218 in your textbook. Mark on your graph where the indicator color change(s) was (were) observed.

4.4.2 Fit the data

Fit the Gran plot $\sim 10\%$ - 20% from the equivalence point. You have to subjectively decide where this is. From this fit determine the equilibrium constant. From the equilibrium constant and the table below of possible unknowns, determine the identity of your unknown.

Table 3: Unknown weak acid choices (Remember you titrated the conjugate base of these)

alanine	3-nitrobenzoic acid
bromoacetic acid	phosphoric acid
2-aminophenol	iodoacetic acid
acetic acid	hypobromous acid
4-aminobenzenesulfonic acid	imidazole
arsenious acid	3-nitrophenol
aminobenzene	2-naphthoic acid
hydroxyacetic acid	valine
chloroacetic acid	piperidine
dimethylamine	glycine
benzoic acid	4-methylaniline
cyanoacetic acid	propanoic acid
hydrogen sulfide	hydroxbenzene
2,4-dinitrophenol	ethylamine

4.4.3 Molar mass calculation of the weak base

From the ID of your weak acid, determine the molar mass of its conjugate weak base by summing the atomic masses. If your acid is represented by HA, then your weak base is the sodium salt of its conjugate base, or NaA. Remember to account for the sodium atoms in your molar mass.

4.4.4 Determination of equivalence point

Go back to your Gran plot and its fit from Step 4.4.1 and determine the equivalence volume of the titration. From this value and the other experimental parameters determine the concentration of the unknown solution.

4.4.5 FW and Hydration number

From the equivalence volume and the mass of unknown, calculate the formula weight of the unknown. The difference between the formula weight and the molar mass as determined by summing the atomic masses in the formula are due to error in the measurement and the water molecules hydrating the unknown base. Determine the hydration number.

4.5 Reporting your work

Write the following questions and their corresponding answers into your laboratory notebooks.

1. In the Gran plot fitting, which side of the equivalence point do you fit? Explain how you know.

4.5.1 Section 1 Graphics

- Properly labeled graph of the titration data. Volume of 0.100 M HCl vs pH.
- Properly labeled Gran plot. The graph should have x and y axis labels, the Gran plot data, the linearly fit of the Gran plot, a legend, x and y grid lines (major and minor), also a horizontal line at $y = 0$.

4.5.2 Section 2 Results

- unknown acid
- pK_a of the unknown acid
- equivalence volume in mL
- Gran plot predicted FW
- Gran plot predicted hydration number (rounded to the nearest 0.01)
- Gran plot predicted hydration number (rounded to the nearest integer)

5 Application: Determination of Iron Ion Concentration using Dye-Metal Ion Complex and Spectrophotometry

5.1 Equipment and Materials needed

Item	notes
SpectroVis spectrometer	
iron salt	
dye (o-phenanthroline)	
computer	
2 cuvettes	
beaker for waste	
10 mL solution of unknown iron concentration	

5.2 Prelab

Read the Vernier manual for using the spectrometer. Bring a copy to lab for reference.

5.3 Purpose

- To learn how to use a visible absorbance spectrometer
- To learn how to use Beer's Law
- To learn how to fit linear data

5.4 Background

In this experiment you will determine the concentration of total iron (Fe^{+2} and Fe^{+3}) in an aqueous solution of unknown concentration. For high concentrations of iron, this is simple because iron(III) has its own absorbance, but for lower concentrations < 0.00001 M, the absorptivity of iron(III) and a 1 cm sample path length typically used for laboratory spectroscopy gives spectral responses that are below the limit of detection for solution electronic spectroscopy. Previously you determined the stoichiometric ratio of an iron and o-phenanthroline complex that improves the sensitivity of visible absorbance spectroscopy for the detection iron.

In this experiment you will use a visible spectrometer to measure the absorbance of a series of calibration solutions. You will also measure the absorbance of a solution of unknown iron concentration. From these measurements and the results from the previous lab experiments, you will determine the concentration of iron in an unknown solution.

5.5 Procedure Part 1

5.5.1 Solutions Preparation

1. pH 4 Buffer Make this solution with the tablets following the directions on the bottle. You will need enough to buffer all of your solutions.
2. Stock hydroxylamine Prepare a stock ~ 0.09 M hydroquinone solution with water as the solvent. (This has been done for you.)

3. Stock o-phenanthroline Prepare a stock ~ 0.0012 M o-phenanthroline solution with 10 % v/v ethanol/water solution as the solvent. (This has been done for you.)
4. Stock Iron solution Accurately, prepare a stock solution of iron that is ~ 0.007 M from reagent grade $\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2$.
5. Calibration solutions Prepare 5 calibration solutions (knowns, or standards) from your stock iron solution ranging from 70×10^{-6} M to 7×10^{-6} M Fe^{+2} of the same pH. Each 100 mL of iron solution should also contain 3.00 mL of your stock o-phenanthroline solution and 2.00 mL of your stock reducing agent and 10 mL of acetate buffer. You don't really need to make 100 mL of each of these solutions. Ask yourself, how much do I really need. Save your environment.
6. Check Standard Prepare 2 two additional solutions in the same concentration range as your calibration standards (above). These concentrations need to be different than the calibration solutions.
7. Blank Calibration Solution Prepare a blank calibration solution that contains hydroquinone and o-phenanthroline at the same pH as your other calibration solutions.
8. Unknown solution Get an unknown solution aliquot from your instructor and add 5 mL (or 4 mL depending on you calibrated pipet) of it to a 50 mL volumetric flask. Treat this sample the same as your calibration standards.

5.5.2 Spectroscopic Measurement of Dye-Metal Complexes

Read the Vernier manual for using the spectrometer. Measure a background spectrum of your blank using visible spectrometer according to the instrument's operational instructions (Experiment -> Calibrate). Measure the absorbance spectrum of each solution. Save the whole spectrum.

5.5.3 Data Analysis

Using the calibration solutions' (knowns) absorbance values and the method of least squares, find the best of fit linear function for the dye-iron complex. Perform a separate validation using the check standards for calibration to ensure the quality of the calibration. Report the part-per-thousands error (ppt error) for each check standard solution.

$$\text{ppt Error} = \frac{\text{known concentration} - \text{Beer's law determined concentration}}{\text{known concentration}} \times 1000$$

From this linear function and the absorbance of the solution of unknown concentration, predict the concentration of the unknown solution. Determine and report the standard error of the predicted concentration.

5.6 Clean up

Dispose of all waste appropriately.

5.7 Reporting your work

Use the spreadsheet to report your work.

Write the following questions and their answers in your laboratory notebook.

1. Why is hydroquinone used in this procedure?
2. Over what range of iron concentrations does the iron-phenanthroline complex obey the Beer-Lambert law?
3. Tea contains a significant amount of tannic acid. Given this fact explain why a cup of tea made with DI water does not show the characteristic dark brown color of tea made with ordinary water.
4. List some characteristics of a good complexing agent for colorimetric analyses.

6 Characterization of BBQ Sauce

6.1 Equipment and Materials needed

Item	notes
SpectroVis spectrometer	
computer with loggerLite installed	
power block for Labpro	
Labpro	
pH probe	
conductivity probe	
2 cuvettes	
beaker for waste	
BBQ sauce sample	
50 mL flask	
NaOH	
KHP	

6.2 Prelab

Read the Vernier manual for using the spectrometer, and bring a copy to lab for reference. Bring ~ 50 mL of BBQ sauce to lab, this must be from a restaurant (not a grocery store). Also bring a photo of the restaurant, location (GPS coordinates) of the restaurant, and the name of the restaurant.

Item	Data	comments
Name of Restaurant		
GPS location of Restaurant		latitude, longitude
Photo of outside of Restaurant		upload this to the wiki
Date of BBQ sauce sampling		yyyymmdd
County/Parish		
State/Province		
Country		
Restaurant's name of sauce		This is only an example
Amount sampled (mL)		
Taste descriptors		use your own words
Visible color of Sauce		
Your opinion of the sauce		1-5 (1 bad, 5 excellent)

If you don't have a GPS mapping device you can type in the address of the Restaurant at <http://www.gpsvisualizer.com/geocode> to get its GPS coordinates

6.2.1 Example:

Item	Data	comments
Name of Restaurant	Haywood Smoke House	
GPS location of Restaurant	35.4797249, -83.0064928	latitude, longitude
Photo of outside of Restaurant		upload this to the wiki
Date of BBQ sauce sampling	20160229	yyyymmdd
County/Parish	Haywood	
State/Province	NC	
Country	USA	
Restaurant's name of sauce	Smokey Man Juice	This is only an example
Amount sampled (mL)	50.0	
Taste descriptors	smokey,sweet, chocolaty	use your own words
Visible color of Sauce	red/orange	
Your opinion of the sauce	5	1-5 (1 bad,5 excellent)

6.3 Learning Outcomes

-
-
-

6.4 Background and Introduction

In this laboratory, we will export the fun and exciting world of food science and food analysis. BBQ sauce is that stuff that you, your mom, or the restaurant puts on meat to make it BBQ. I am just kidding. I don't really know what makes a BBQ sauce a BBQ sauce; that is what we are trying to figure out herein. We are going to characterize several BBQ sauces in several ways and hopefully be able to define what is a BBQ sauce.

So the parameters that we will characterize are listed in the table below:

Parameter	Analyte	measurement technique	Why it might be important
Conductivity	electrolytes	conductivity	total salty flavor
pH	hydronium ion	pH probe	sour flavor intensity
total acidity	weak acids	NaOH titration	sour flavors
color	colored molecules	visible spectroscopy	tomato's have color
redox potential	??	redox probe	state of rot

6.5 Procedure: Solutions Preparation

6.5.1 Standardization of NaOH solution.

Follow the procedure at the end of chapter 10 (page 235) in Harris to standardize your NaOH solution. You need to determine the volume of NaOH that you will need for this entire set of procedures and make that much. If you run out you will have to re-standardize it.

6.5.2 Blank Calibration Solution

Prepare a blank calibration solution that contains DI water.

6.5.3 Unknown Solution

Add 10 mL of your BBQ sauce sample using a graduated cylinder (note the volume as best as you can.) to a 100 mL volumetric flask, dilute to the mark and mix well. This solution will be referred to as your Unknown Solution.

6.6 Procedure: Measurements

6.6.1 Total Acidity titration

Rinse and dry the probe before you store it, or change solutions.

1. Calibrate your pH probe with pH 4 and 7 buffers.
2. Add your standardized NaOH solution to your buret.
3. Pipet 10 mL of your Unknown Solution to a clean beaker. Measure the pH of this solution and record the value. Add ~ 25 mL of DI water to the beaker.
4. Setup the measurement apparatus with the beaker containing a magnetic stir bar and a pH probe on top of a magnetic stir plate under your buret. Make sure that the stir bar does not hit the pH probe. Set the stir bar to spinning slow enough mix, but not to splash.
5. First perform a rough titration of your unknown solution, by adding 1 mL of your standardized NaOH solution then measuring/recording the pH of the solution 30 seconds after each titration step. Add your strong base until the pH measurements are above 7.0 **and** the pH values have leveled off. From this titration you should be able to estimate the equivalence volume (s) (V_e). Remove the stir bar, pour your waste into your waste beaker, and wash the breaker.
6. Now perform at least 3 careful titrations of your Unknown Solution. Pipet 10 mL of your Unknown Solution into a clean beaker. Measure the pH of this solution and record the value. Add ~ 25 mL of DI water to the beaker. Setup the beaker with pH probe and stir bar. Titrate the solution with standardized NaOH. Until you are within 4 mL of the equivalence point(s), add the base in ~ 1.5 mL aliquots, recording the volume and pH 30 s after each addition. Thereafter, use 0.4 mL aliquots until you are within 1 mL of the equivalence point(s). After that, add base 1 drop at a time until you have passed the estimated equivalence point(s) by a few tenths of a milliliter. (During this part of the titration you should be seeing a large change in pH.) Then continue adding 1 mL aliquots until the pH is above 7 and the pH values have leveled off.

6.6.2 Total conductivity

Using a clean conductivity probe, measure and record the conductivity of your Unknown Solution. Rinse and dry the probe before you store it.

6.6.3 Spectroscopic Measurement of your Unknown Solution

Read the Vernier manual for using the spectrometer. Measure a background spectrum of your blank using visible spectrometer according to the instrument's operational instructions (Experiment -> Calibrate). Measure the absorbance spectrum of your Unknown Solution. Save the whole spectrum.

6.6.4 Solution pH

You already did this at the beginning of the titrations, before you added any base. Average your pH measurements.

6.7 Data Analysis

Using the Gran plot technique that you used in the experiment 4, determine the equivalence volumes and pK_{as} of all of the acids in your Unknown Solution. Analyze each measurement separately and average your results. Express your uncertainty in these determinations with a t-distribution based confidence interval.

6.8 Clean up

Dispose of all waste appropriately.

6.9 Reporting your work

Use the spreadsheet to report your work.

Write the following questions and their answers in your laboratory notebook.

1. In the total acidity titration, how can you tell if there is more than one weak acid in your sample?
2. What are the units of conductivity?
3. Can we use the o-phenathroline-iron complex lab to determine the concentration of iron in your BBQ sauce? Why or why not?
4. Can we use the EDTA titration for divalent metals to determine the magnesium or calcium concentration in your BBQ sauce? Why or why not?
5. List some reasons why you might perform more than three careful total acidity titrations.

7 Appendix

7.1 Grading Rubrics

7.1.1 lab points

7.1.2 Notebook

7.1.3 Final Exam Questions

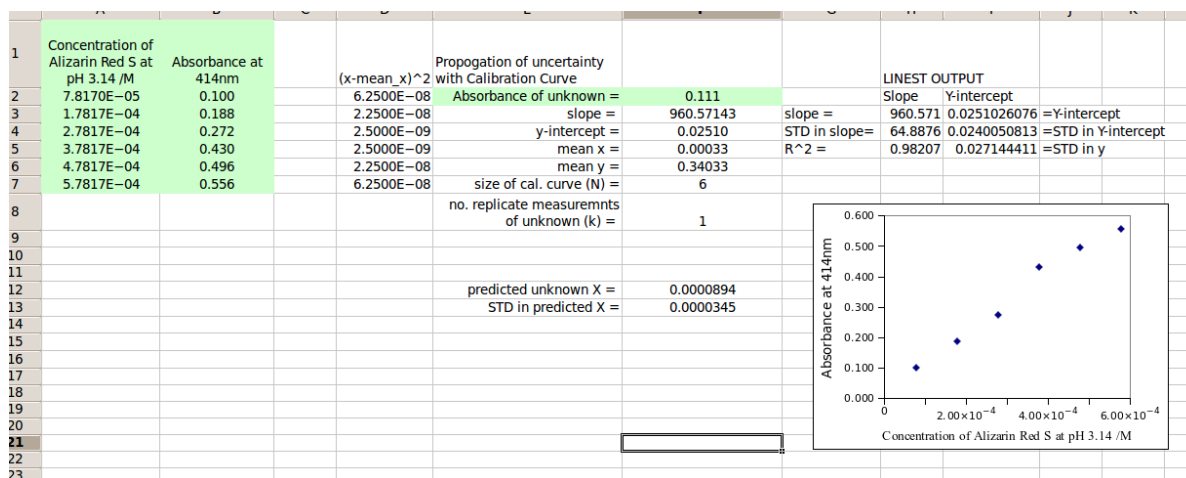
7.2 Grubbs Critical Values

Number of measurements	Critical Value
3	1.1531
4	1.4625
5	1.6714
6	1.8221
7	1.9381
8	2.0317
9	2.1096
10	2.1761
11	2.2339
12	2.2850
13	2.3305
14	2.3717
15	2.4090

7.3 Standard Error of Calibration

7.4 Beer's Law and Classical Least Squares

7.4.1 Spreadsheet setup



see spec let. article for text and stuff.

7.5 Cleaning solutions

7.5.1 ammonium peroxydisulfate in 98% sulfuric acid

add 36 g $(\text{NH}_4)_2\text{S}_2\text{O}_8$ in a loosely stoppered 2.2 L bottle of 98 wt% H_2SO_4

HM Stahr, W Hyde, and L Sigler, "Oxidizing Acid Baths - without Chromate Hazards", Ana. Chem. 1982, 54, 1456A

7.5.2 commercial EOSULF cleaning solution

7.5.3 piranha solution

1:1 (vol:vol) mixture of 30 wt% H_2O_2 plus 98 wt% H_2SO_4

7.6 Reference Data

7.7 Sample of lab notebook

7.7.1 fix TOC for example labnotebook

- State "TODO" from "" [2012-12-13 Thu 14:17]

http://polaris.cs.wcu.edu/~huffman/include/example_labnotebook.pdf

7.8 Sample of scientific procedure from the literature

7.9 Non-linear Hard model based Curve fitting of titration data

7.9.1 Introduction

Titration is used in analytical chemistry in two ways. The first is a very "low tech" method for determining concentration.

The other use of a titration is in the determination of equilibrium constants. From the Henderson-Hasselbalch Equation 4

$$pH = pK_a + \log \frac{[A^-]}{[HA]} \quad (4)$$

it can be seen that when $[A^-] = [HA]$, called the equivalence point, the $pH = pK_a$, as shown in Figure 7.9.1.

In this laboratory exercise you will learn to evaluate titration data in several ways to determine the concentration of an unknown and the equilibrium constants involved in the titration.

7.9.2 Determination of equivalence point

There are several ways to determine the equivalence point of a titration curve. By definition the equivalence point of a titration curve is the place along the curve where the slope is changing at the greatest rate. This point is indicated in Figure 7.9.1 by an arrow. The challenging part of evaluating a titration curve is the determination of the equivalence point or points.

1. Curve fitting Approach This method of evaluating titration data is based on a comparison between the experimental data and theoretical data. You will learn how to obtain experimental data in another laboratory exercise, and you will learn in gross detail how to determine the theoretical data from the book in chapter 10. In brief the theoretical curve (pH vs. Volume) for a titration begins with the systematic treatment of Equilibria (See Harris, Chapter 7). We will explore the derivation of these theoretical titration curves with an example.

- (a) Theory This derivation works for a monoprotic weak acid such as acetic acid. For your work you may have a polyprotic acid. So for the fit you will need to choose the appropriate function to fit your data (a list of functions are on page 228 of your textbook). Titration of a volume V_a of weak acid, denoted HA, with initial concentration of C_a (unknown) with a volume V_b of NaOH of concentration C_b (known).

The charge balance for this mixture is

$$[H^+] + [Na^+] = [A^-] + [OH^-] \quad (5)$$

Assuming that the concentrations are dilute, the two volumes add within the accuracy of measurement ($V_{\text{total}} = V_b + V_a$), from which the concentration of Na^+ is

$$[Na^+] = \frac{C_b V_b}{V_a + V_b} \quad (6)$$

The formal concentration of the weak acid is

$$F_{HA} = [HA] + [A^-] = \frac{C_a V_a}{V_a + V_b} \quad (7)$$

The fractional composition α_{A^-} is defined as

$$\alpha_{A^-} = \frac{[A^-]}{[HA] + [A^-]} = \frac{K_a}{[H^+] + K_a} \quad (8)$$

and is the fraction of a particular species, denoted by the subscript, in the solution and where K_a is the acid dissociation of HA. After a few algebraic rearrangements, we come to the equation

$$[A^-] = \alpha_{A^-} F_{HA} = \frac{\alpha_{A^-} C_a V_a}{V_a + V_b} \quad (9)$$

By substituting Equations 6 and 9 into the charge balance equation we get

$$\Phi = \frac{C_b V_b}{C_a V_a} = \frac{\alpha_{A^-} - \frac{[H^+] - [OH^-]}{C_a}}{1 + \frac{[H^+] - [OH^-]}{C_b}} \quad (10)$$

where Φ is fraction of the titration. We can rearrange Equation 10 to get the useful equation

$$V_b = \frac{\Phi C_a V_a}{C_b} \quad (11)$$

Equation 11 provides a link between the volume of titrant added from the buret and the measured pH. In this exercise, if we generate a theoretical curve from Equation 11 that fits our experimental data, then when the fit is good we know the unknown concentration C_a and equilibrium constant K_a .

- (b) Setting up the Spreadsheet Start your spreadsheet program and read in the experimental data. Put the data in the first two columns and beginning at the 7th or 8th row.
- In cell A1 type **Monoprotic Weak Acid - Strong Base Titration**
- In cell A2 type **Constants**
- In cell A3 type **K_w =**
- In cell A4 type **Experimental Knowns**
- In cell A5 type **C_b = .**
- In Cell A6 type **V_a = .**
- In Cell B3 type **1.00 e-14}** .
- In Cell B5 type the concentration of the solution in the buret
- In Cell B6 type the starting volume of the solution in the beaker beneath the buret.
- In Cell A7 type **Experimental** and in Cell C7 type **Theoretical**
- In Cell A8 type **pH**
- In Cell B8 type **V_b_x**
- In Cell D8 type **V_b_t**
- In Cell E8 type **[H₃O⁺]**
- In Cell F8 type **[OH⁻]**
- In Cell G8 type **alpha A⁻**
- In Cell H8 type **PHI**
- In Cell I8 type **DIF²**
- In Cell I6 type **SEF**
- In Cell E2 type **Experimental Unknowns**
- In Cell E3 type **K_a =**
- In Cell G3 type **C_a =**

Read or type in your experimental pH vs Volume data beginning with columns A8 and B8, respectively. (If you already have the data in another spreadsheet or from Vernier Logger Pro Lite, then you can copy it in to these two columns.) These two columns are your experimental data. At this point your spreadsheet should look like Figure 5, and we are ready to input the experimental data and calculate the theoretical data.

- (c) Calculation of your Theoretical Titration Curve If you have not already done so, type into your spreadsheet the values of C_b (the concentration in the buret), input a guess for C_a in cell H3, and V_a (the volume of the unknown).

Use the definition of pH ($\text{pH} = -\log[\text{H}_3\text{O}^+]$) and K_w ($K_w = [\text{H}_3\text{O}^+][\text{OH}^-]$) to calculate the [H₃O⁺] and [OH⁻] in columns E and F in your spreadsheet. Use Equation 9 to calculate the value for α_{A⁻} in column G of your spreadsheet. Use Equation 10 to calculate PHI in column H of your spreadsheet using cell H3 as an approximate value for C_a. Use Equation 11 to calculate the theoretical volume used during titration labeled V_b_t in your spreadsheet column D.

Copy the pH into the theoretical column and plot the pH vs. V_{b_t} and V_{b_x} on one graph. Plot the Experimental data with data points and the theoretical data with a smooth line. Manually adjust the the values of K_a and C_a until the two curves overlap. Notice what the influence these two parameters have on the theoretical curve.

When these two curves overlap you have a pretty good estimate of the values of K_a and C_a. We will let the computer search for values of K_a and C_a for the best fit. Add a new column after your **PHI** column labeled DIF². In this column use Equation 12 to

calculate the square of the residual.

$$dif^2(i) = (V_{bx}(i) - V_{bt}(i))^2 \quad (12)$$

In Cell I6 type **SEF** and calculate the square root of the sum of the squares of the residuals (DIF) using equation 13.

$$SEF = \sqrt{\frac{\sum (y_x - \hat{y}_t)^2}{N}} \quad (13)$$

Finally, there is a tool in most spreadsheet programs called the **Solver**. It is usually located under the menu item ***Tools**.

Initiate the solver. If you have trouble finding and using Solver use the help for your spreadsheet program. You should see a dialog box open up (Figure 6). In this dialog box you will make three changes. In the item labeled **Set Target Cell:** type or select on the spreadsheet \$I\$7, or where ever you calculated the value for **SEF**. In the next item labeled **Equal to:** select the **min** radio button. Last in the **By Changing Cells:** text box type or select the spreadsheet cells corresponding to the values of K_a (typically \$F\$3) and C_a (typically \$H\$3). The way the **Solver** works is that it changes the cells for K_a and C_a and looks in the cell for **SEF**. If the **SEF** goes down the **Solver** knows that it changed the values for K_a and C_a in the correct way, and if **SEF** goes up then the values for K_a and C_a where changed in the wrong direction. This is an iterative process, so it will take several attempts to get the fit as good as possible. Before we actually start the solver, we need to change a few options. Select the **Options** button in the dialog box. Make your options dialog box look like the one shown in Figure 7.

Once you have the **Options** dialog correctly setup press the **OK** button. The **Solver Parameters** dialog box should reappear. Press the Solve button. The **Solver** will then iterate through the changes of K_a and C_a and if the value of **SEF** is not low enough, it will give you a new dialog box that gives you the option of continuing or bailing out of the search (Figure 8). Press **Continue** until the **Results** Dialog box appears (Figure 9). Select **Keep Solver Solution** and press **OK**. What you should have left in the spreadsheet is in the cells for K_a and C_a are the values from the **Solver** for K_a and C_a .

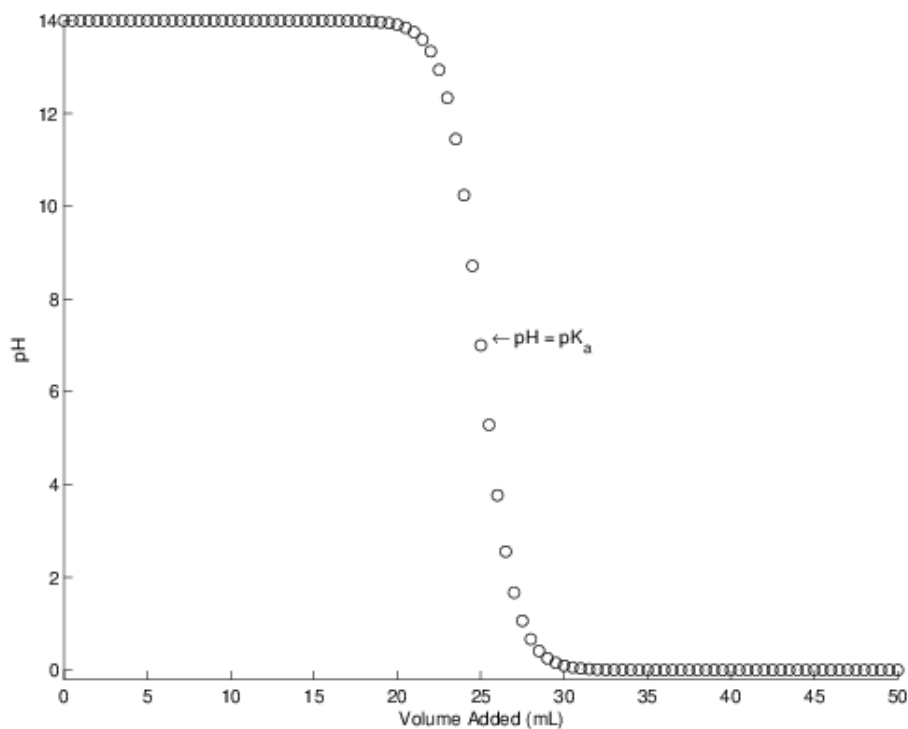


Figure 4: Ideal Titration Curve with an arrow marking the equivalence point

	A	B	C	D	E	F	G	H	I	J	K
1	Monoprotic Acid-Base Titration										
2	Constants				Experimental unknowns						
3	Kw = 1.00E-14				Ka = 1.00E-07			Ca = 0.105			
4	Experimental knowns										
5	Cb = 0.1										
6	Va = 50										
7	Experimental		Theoretical						SRR		
8	pH	Vb_x	pH	Vb_t	[H+]	[OH-]	alpha A-	PHI	RR	0.00E+00	
9	3.5	-0.0463									
10	3.6	0.0149									
11	3.7	0.0769									
12	3.8	0.1429									
13	3.9	0.2164									
14	4	0.3012									
15	4.1	0.4017									
16	4.2	0.5229									
17	4.3	0.6711									
18	4.4	0.8534									
19	4.5	1.0788									

Figure 5: Spread Sheet Setup

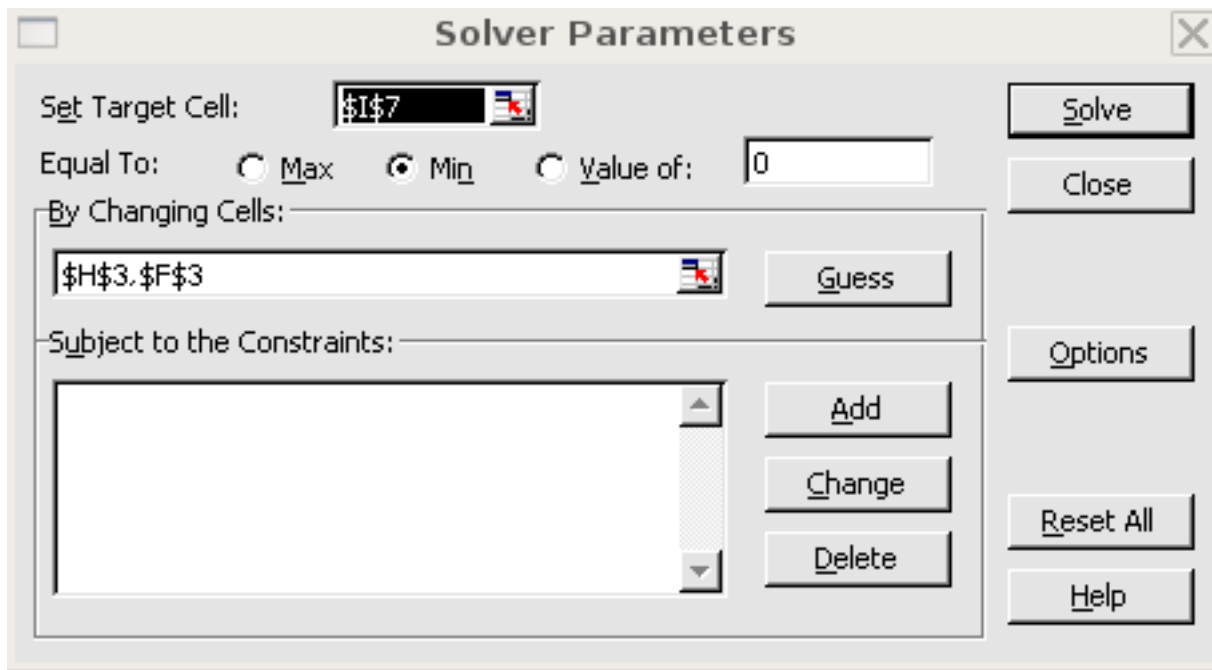


Figure 6: Solver dialog box

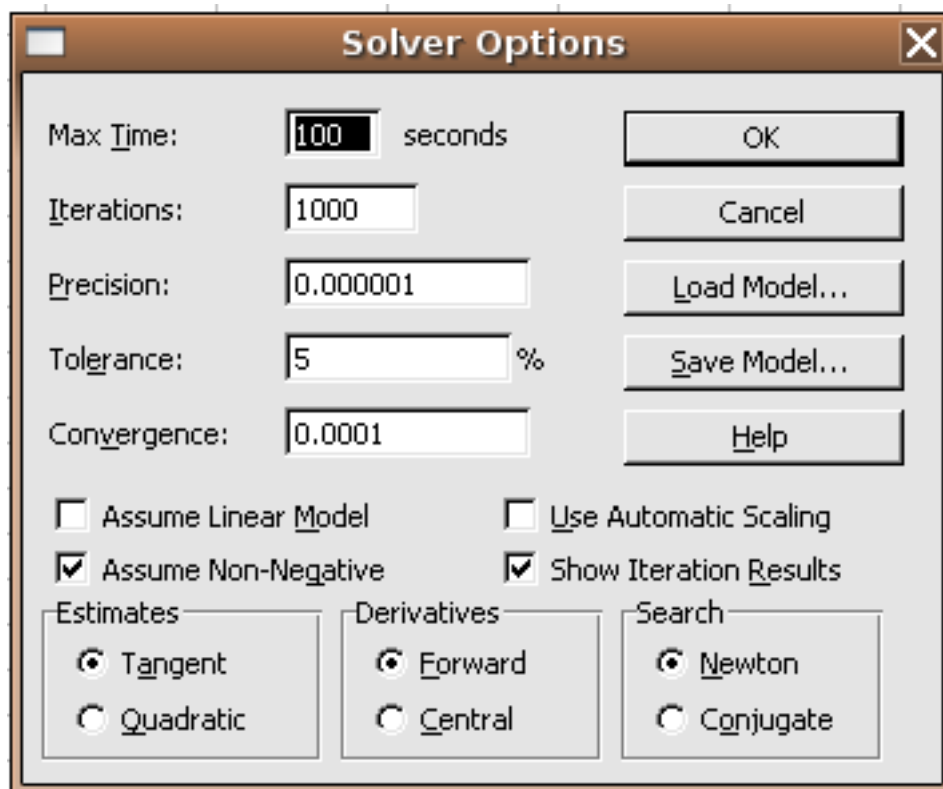


Figure 7: Options dialog box for Solver

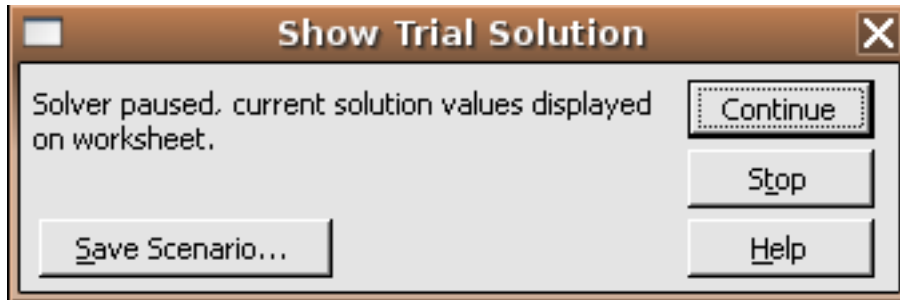


Figure 8: Iteration dialog box for Solver

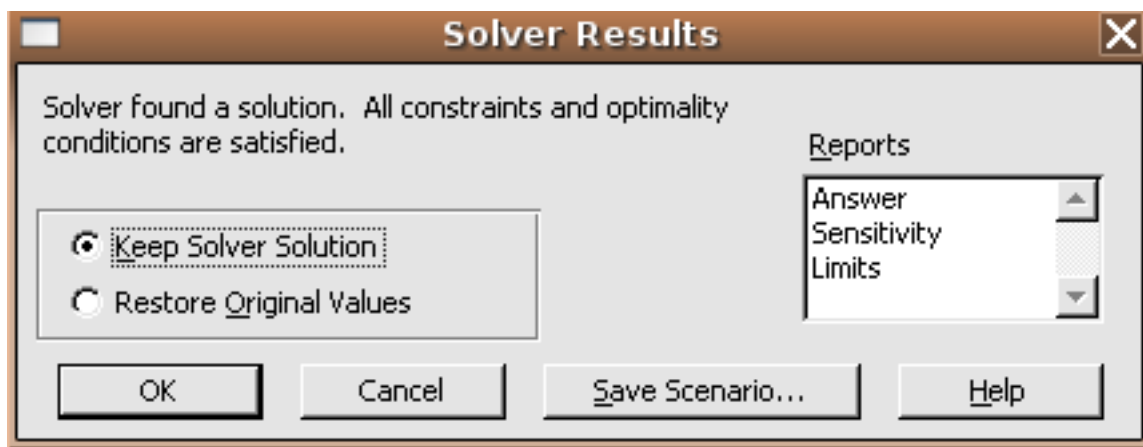


Figure 9: Results dialog box for Solver