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**Dep. Web:** <http://www.concord.edu/physci/node/2>

**Course Title:** Instrumental Analysis Laboratory

**Course CRN # and Section, Credit Hours:** CHEM 358 20250 01 01

**Semester Taught (including year):** Spring 2019

**Room Number (if applicable):** Science 400, 406 & 407

**Course Time (if applicable):** M 2:00 PM – 4:50 PM (Changes will be announced in advance)

**Course Management System:** Moodle

**Hardware/Software Needed:** Microsoft Word, Excel, Calculator

**Prerequisites:** CHEM 351, Physics 102 or 202 (pre or co-requisite)

**Co-requisite:** CHEM 352

**Text requirements:** Robinson, Frame & Frame, *Undergraduate Instrumental Analysis*, 7<sup>th</sup> Ed.

Computer access, computers in Math lab and library have all programs needed

Registration on Moodle at [moodle.concord.edu/](http://moodle.concord.edu/)

Handouts will be supplied as needed - you are responsible for printing & bringing to lab

Lab notebook, safety goggles or glasses, sharpie permanent marker, and flash drive

### **Course Description/Rationale:**

We will study and perform experiments in analytical chemistry involving spectroscopy, separations, and electrochemistry. Groups will be assigned and will change frequently. Chemistry 358 is a laboratory course covering many diverse instrumental methods of analysis. The course is designed to familiarize you with the instrumentation most commonly used in quantitative and qualitative chemical analysis. By the end of the semester, you will have gained sufficient analytical background to solve a wide variety of chemical problems, developed some analytical and critical thinking skills, and learned to effectively communicate the results of your completed work. To

realize these goals, you will perform a set of experiments that expose you to a wide variety of chemical instrumentation.

### **Concord University Educational Goals:**

**Skills:** Proficiency in interpreting data, integrating information, formulating ideas, thinking critically, and communicating with others, as demonstrated by the following competencies:

1. An ability to employ appropriate observational, logical, analytical, computational, creative, and critical thinking skills within and across academic disciplines; and to apply these skills in problem solving.
2. An ability to analyze, synthesize, and integrate elements, information and ideas.
3. An ability to evaluate elements, information, and ideas on the basis of appropriate criteria.

**Knowledge:** Familiarity with principles underlying academic discourse in various fields, as demonstrated by the following capabilities:

1. An awareness of the fundamental characteristics and properties of the physical universe.

**National Standards:** American Chemical Society Exam

### **Learning Outcomes:**

As a result of taking the course, the student should be able to:

- Demonstrate proper use of instrumentation
- Demonstrate proper application of fundamental principles in instrumental analytical chemistry
- Illustrate proper propagation of error for mathematical calculations
- Interpret experimental results and draw reasonable conclusions
- Distinguish among a variety of instrumentation and their applications
- Assess the reliability of results and recognize sources of error for each instrument
- Assess application of an instrument for identification of an unknown
- Demonstrate proper use of mathematical formulas and analytical tests for data analysis
- Demonstrate appropriate use of microsoft excel for data analysis
- Illustrate proper written skills and citations
- Distinguish among information provided by various instrumentation
- Choose appropriate instrumentation to the a research question
- Prepare viable scientific proposals based on literature review

### **Course Requirements:**

The following assignments will be used to determine the student's grade in the class.

- **Pre-laboratory Exercises:** Pre-lab exercises are designed to familiarize the student with the lab exercise and stimulate thinking about how the experiment will be designed and executed. These assignments provided in the laboratory manual and must be completed prior to the beginning of YOUR lab session each week and a copy submitted on the Moodle page for the course on the day of the experiment. These pre-lab assignments will be graded. A student will not be allowed to enter the laboratory without completing the pre-lab assignment.
- **Lab Notebook:** Each student will keep a laboratory notebook, which should be a primary record of your experimental procedures, results, data analysis and discussions. A detailed description of how to keep a laboratory notebook is provided in your laboratory manual. The notebook must be initialed by the instructor prior to leaving each lab. The notebooks will contribute to 3% grade for the Laboratory Evaluation. Refer to the **Grading** Section.

- **Evaluation:** You will be assessed each week on your: preparedness, ability to work safely, technique, knowledge and overall citizenship in the lab.
- **Writing Assignments:** Throughout the semester, each group will write one laboratory report for each completed laboratory exercises. The writing assignment will be assigned as formal, memo, or informal reports by the professor. Each member of the group is expected to contribute to these assignments. Upon submitting each assignment, each member of the group will sign a statement that both members contributed equally to the assignment and that both members approve the submitted assignment. Each assignment will be critiqued and graded. Through these assignments, you will learn how to effectively write a scientific paper. A detailed description of how to write a laboratory report is provided in your laboratory manual. Students must demonstrate appropriate use and presentation of tables, graphs, and equations. The use of Equation Editor for writing equations and mathematical formulas in Microsoft Word is highly recommended.
- **Laboratory Activities:** Data analysis for the laboratory activities will include the compiled data from all student groups performing the assigned experiment. Students will be assigned various portions or all portions of any given experiment on the discretion of the professor. It is your responsibility to perform the experiments to the best of your ability. The students must provide detailed explanation of errors and uncertainties based on their datasets with appropriate significant figures.
- **Project:** Four weeks of lab will consist of an extended project. Students will be assigned to a group. Students should adhere to the timelines for submitting initial proposals after discussion with the professor. All written proposals are submitted on Moodle page. You will complete the experiments for this project during three weeks of lab. Each of these weeks will have pre-labs that require you to plan for that week's activities. During the fourth week, your group will complete an oral presentation. Your group will also write a report for the multi-week project. The faculty members will be invited to your presentation and contribute to the overall evaluation. You are recommended to work with your professor on IEP ideas. Students is provided an option to drop two lowest laboratory grades if they choose to present a poster on their IEP on Pre-professional Day at Concord University (Scheduled for the week before Thanksgiving break). Students must indicate their request for poster presentation three weeks in advance of the Pre-professional Day.
- **Grading:**

**All writing assignments will be assigned 100 points each which will include the pre-laboratory assignments unless otherwise stated.** The final grade of the course will be determined by the grades received on the lab reports, the independent experiment project, pre-testing of an experiment and consequent presentation to the class, attendance, evaluation of your performance in the laboratory. **All** experiments must be completed and **all** lab reports must be submitted by Friday of finals week for any credit. If you are missing more than two lab report at this time you will not pass the course. A comprehensive laboratory examination might be announced in mid-April based on your progress in the course.

Laboratory Evaluation	10%
Writing Assignments	60%
IEP Project	
Proposal	5%
Oral Presentation	10%
<u>Formal Report</u>	<u>15%</u>
 Total of	 100%

### Grading Scale:

≥	≥	≥	≥	≥
<b>90 – 100 %</b>	<b>80 - 89%</b>	<b>70 - 79%</b>	<b>60 – 69 %</b>	<b>60% - Below</b>
<hr/>	<hr/>	<hr/>	<hr/>	<hr/>
<b>A</b>	<b>B</b>	<b>C</b>	<b>D</b>	<b>F</b>

### Course Policies:

- Absence Policy:**  
All make-up laboratories will be scheduled on a case-to-case basis following the original assigned date for the experiment. Your lowest laboratory grade is dropped and it could count towards an absence. Generally speaking, only documented University conflicts and severe illness or family emergencies as documented by the Dean of Students will be acceptable excuses for missing a test or exam. This is a laboratory course with 3-hour meeting per week. You are expected to attend all laboratory sessions throughout the entire semester unless specific permission otherwise is given. Missing two or more lab periods is grounds for a failing grade in the class.
- Electronic Assignment Submission:**  
Some assignments require on-line submission via Moodle. On-line assignments must be completed in a timely fashion. An excuse for "technological difficulties" (internet was down, etc.) is not acceptable.
- Late Assignments:**  
Assignments that are not submitted at the specified times will be considered late. There will be a 10% deduction for every day late.
- Moodle Policy:**  
Many important materials will be available on the course website. **You are responsible for all content on this website and it is recommended that you access this site at least once a week to get this information!** The website is [moodle.concord.edu](http://moodle.concord.edu) and content for this lab will be posted on the **Chemistry 358** page (enrollment password: 358). Check this site periodically for any special announcements from the instructor. It is your responsibility to refer to Moodle for announcements: reminders of homework assignments due, help sessions, and tests, etc. Refer to the complete policy on: <https://moodle.org/mod/page/view.php?id=8148>

You should also find the following resources on the Moodle

- Copy of our Syllabus
  - Homework Information
  - Laboratory Activities
  - Supplementary Information
  - Gradebook: Laboratory course overall grade
- **Help Sessions Policy:**

Students are encouraged to attend office hours and request appointments for review and assistance with material in this course. Please request an appointment at least 24 hours in advance to assure availability of the instructor.
  - **Safety:**

All appropriate safety guidelines will be adhered to strictly. Safety goggles must be worn at all times in the laboratory. In addition, the student must dress appropriately. Shoes that entirely cover your feet and clothing that entirely covers your torso and legs must be worn. Shoes with high heels, shorts, dresses/skirts, and long, flowing clothing will not be permitted in the lab. No exceptions will be permitted. Consult with the instructor if you have any questions. Students who do not follow safety instructions may have points deducted, be asked to leave without the possibility of make up or be dropped from the course. Students will be required to fill in an accident report if it is advised by the instructor. Any other minor incidents will require a completion of an incident report.
  - **Electronic Device Policy:**

All cell phones must be turned off during pre-lab and lab. The instructor reserves the right to make point deductions from your grade for using a cell phone during class. Always bring your computer or secure access to one as it is required to complete data analyses for some laboratory activities during the laboratory periods.
  - **Laboratory Manual:**

This document and other handouts comprise the laboratory manual for Chemistry 358. They describe course requirements and policies, along with the instructions for the individual experiments. You **must** read all of the sections thoroughly and then carefully read the appropriate laboratory chapter and carry out the pre-lab exercises **before** performing each experiment. At the start of each lab period, your prelab assignment will be checked and you may be quizzed on the prelab questions to ascertain your readiness to perform the experiment. There is a penalty for being unprepared for lab! *You must bring a print out of the lab procedure to lab with you everyday! Writings are only to be carried in your laboratory notebooks and other alternatives will not be allowed in the lab. You will be dismissed from the lab period otherwise.* Laboratory experiments will be uploaded on Moodle 24 - 48 hours prior to lab period. You are expected to be checking your Moodle periodically.

## Course Timeline (Note: Schedule is subject to change with advanced notice)

Schedule: You are highly recommended to read thoroughly including book chapters, prepare for performing the experiment in a timely manner, complete the prelab assignment, and submit the appropriate report) after the completion of the experiment on the Moodle page for the course. You have limited time to complete your assigned activity. Lack of proper preparation to complete the experiment will lead to incompleteness of the laboratory activity. Your instructor will assist guide you when necessary. You will be required to troubleshoot and build your own understanding of the proper use of the instrument. The second laboratory period will be assigned for the completion of the experiment. **Materials Safety Data Sheets (MSDS) must be submitted with proper waste disposal procedure for each laboratory with the prelab-assignment.**

Timeline	Topics
Week 1 January 14	<i>Introduction, Check-In and Safety Regulations</i> <i>Module I – Understanding Analytical Analysis with Coins and Statistics</i>
Week 2 January 21	<i>No Lecture January 21 – Holiday</i>
Week 3 January 28	<i>Module I – Designing Linear Response with Beer’s Law via UV-Vis Spectroscopy</i>
Week 4 February 4	<i>Module I – Investigating Rate Laws with Molar Absorptivity for the Kinetics of Bleaching via UV-Vis Spectroscopy</i>
Week 5 February 11	<i>Module II – Spectroscopy II</i> <i>Identification of Molecular Compounds via Infrared and Raman Spectroscopy</i>
Week 6 February 18	<i>Module II – Spectroscopy II</i> <i>Identification of Molecular Compounds via Fluorescence Spectroscopy</i>
Week 7 February 25	<i>Module III – Electrochemistry</i> <i>Investigations electrochemical properties using Cyclic Voltammetry via PAR3000A</i> <i>Three page proposal/Literature review is due with proper citations*</i>
Week 8 March 4	<i>Module III – Electrochemistry</i> <i>Investigating the Molarities of Ions using Potentiometric Titrations via ORP electrodes</i>
Week 9 March 11	<i>Spring Break</i>
Week 10 March 18	<i>Module IV – Separation</i> <i>Separation of Molecular Compounds via GC and Identification using GC/MS</i>
Week 11 March 25	<i>Module IV – Separation</i> <i>Separation of Molecular Compounds via HPLC</i>
Week 12 April 1	<i>Module V – Identification techniques</i> <i>Determination of Structural Properties using NMR – Visitation with Virginia Tech</i>
Week 13 April 8	<i>Investigation of Experimental Parameters (IEP Project)</i>
Week 14 April 15	<i>Investigation of Experimental Parameters (IEP Project)</i>
Week 15 April 22	<i>Investigation of Experimental Parameters (IEP Project)</i>
Week 16 April 29	<i>Presentation of IEP Projects and Check-Out</i>

### \*Assigned Independent Projects for Spring 2019

Design a setup for electrogravimetric analyses for copper and nickel ions using a voltmeter and amperemeter  
Investigating active compounds in aspirin via fluorometry and UV-Vis spectroscopy  
Investigate the impact of coatings on corrosive rate of aluminum metal (AA2024??)  
Investigate active ingredients in e-cigarettes using GC-MS

## Safety Precautions

In a laboratory emphasizing the use of instrumentation, it is easy to forget that potential dangers still exist. For example, some of the experiments require the use of harmful chemicals while others require the use of flames. Therefore, **anyone present** in the chemistry laboratory must comply with the following safety rules:

1. Safety goggles must be worn at all times and a lab coat (or apron) is recommended.
2. No open-toed shoes are allowed.
3. No eating, drinking or smoking is allowed in the laboratory.
4. Note the locations of the fire extinguishers, safety showers, and eye washes before starting the laboratory work.
5. Wear gloves when handling harmful organic solvents or solutions containing heavy metals.
6. Dispose of glass in the square, cardboard containers designated "glass only".
7. **No student is allowed in the lab alone without specific permission from the instructor on the day you are in lab. You will be asked to arrange with your classmates to be present in the lab to assure safety.**
8. All containers must have labels designating their contents. *Separation of organic, inorganic, halogenated acids, and aqueous solutions is a must!*
9. When the lab period is complete for the day, the lab must be clean. All equipment should be put away, and all glassware washed, dried and the labels removed. (Points may be taken away from lab report scores for infractions of the keeping lab clean and safe rule!)

## Waste Disposal

Chemical waste must be disposed of properly. Multiple experiments might be carried out simultaneously in this class, check the waste bottle labels every time you add waste and do not accidentally mix waste from different experiments! Never put anything down the sink without specific permission from the instructor!

## Alternative Rotation Schedule

If necessary, you might be provided a rotation schedule for your experiments based on the enrollment. Because you cannot all use the same instruments on the same days you will be performing most of the experiments in a rotation. You will work in groups of two or three students and must follow the designated sequence of experiments on the day assigned for your slot and with your assigned lab partner.

## IMPORTANT DATES AND DEADLINES (some dates are tentative)

April ? <sup>th</sup>	Radford ACS Meeting (Wednesday)
April ? <sup>th</sup>	Virginia Tech Field Trip (Saturday)
April ? <sup>th</sup>	Concord Research Day – Poster Presentation (Thursday)
May 1 <sup>th</sup>	Last day to turn in lab reports by 11:30 AM

**Several trips will be announced in advance and they are expected to occur in April. You will be responsible for your own transportation so I recommend you plan in advance.**

## Lab Notebooks

You are required to purchase and maintain a lab notebook. All data and observations must be recorded in the lab notebook in ink at the time the experiment is performed, and must be signed

**prior to leaving the laboratory.** You are strongly encouraged to organize data tables in your notebooks.

It is essential that you adequately maintain your notebook and associated data in an appropriate manner. Below are general expectations for how this is to be done:

- A. You should have only ONE NOTEBOOK in use for this course throughout the semester. It must be BOUND and in the event that you use more than one in a semester, they should be labeled in sequential number throughout the semester. Entries upon all work related to this lab course, even on different experiments, are to be made in a chronological order on sequential pages. Keep a RUNNING TABLE OF CONTENTS at the front of the notebook as you go. DO NOT keep separate notebooks for different experiments or projects.
- B. In addition to documenting actual lab work, appropriate entries into your notebook include library searches, thoughts on experiments and/or results, notes from conversations about this work with faculty or lab partners, and notes from relevant class lectures or other sources.

Your notebook should, above all things, be COMPLETE and, of course, legible. Every experiment that you perform should have:

1. a title and brief description of the objective of the experiment
2. all experimental details (or reference by notebook number and page number to a detailed description of these details that has been entered in an earlier notebook) and diagrams of your set-up (or reference by notebook number and page number to a diagram of this set-up in an earlier notebook) that are not in the lab manual
3. data acquisition parameters, including instrument make and model, method details, etc.
4. **filenames and disk name/number on which file can be found**
5. notes on observations
6. tabulated or numerical results or a brief qualitative description of what the results look like

[NOTE: In most cases, these tables can be set up PRIOR to your performing the lab experiment so that you can just enter your data into the existing table in your notebook during the lab experiment. Your ability to construct these tables before the lab is an excellent indication that you truly understand what you are about to do in a given lab experiment.]

7. a brief conclusion
8. thoughts for further experiments
9. your signature or initials and date at the bottom of each page
10. and the signature of your instructor with a date on the bottom of the last page of each section in your notebook that describes the work you did in lab on a given day

- C. Separate entries should be made for data work-up, referring back by page number to the original experiment. Resulting filenames of worked-up data should be tabulated along with original raw data filename.
- D. Hard copies of data (e.g. strip chart recorder output, etc.) should be labeled with a title, a brief description of the sample/experiment, and appropriate experimental parameters (e.g. scan speed, voltage range, etc.). **You should initial and date every piece of hard copy data,** and the parameters under which the data were acquired should be both in your notebook and labeled on every piece of hard copy data. The original hard copy data can be pasted, taped or stapled into your bound laboratory notebook or kept in an organized fashion in an accompanying

organized and appropriately labeled three-ring binder. You should include photocopies of your raw data in your lab reports. **NEVER ATTACH YOUR ORIGINAL RAW DATA TO YOUR LAB REPORT – IT SHOULD ALWAYS REMAIN IN YOUR NOTEBOOK.**

Maintaining your notebook and data in this fashion will provide an excellent start to good laboratory practice that you can carry on into your future career.

## **Pre-Lab Assignment**

Most experiments will have pre-lab assignments. You should also thoroughly read the experiment before coming to lab and must bring a print out of the procedure with you to lab. The purpose of this is to give you an opportunity to be as prepared as possible before you attempt the experiment. The pre-lab assignment will be checked **at the start of the lab period**. In some cases, this will involve a short questioning period and if you meet a satisfactory level of competence, you will be allowed to proceed. If it is determined that you are not adequately prepared, you will be allowed 20 minutes to try to prepare. If again, you have not adequately completed the pre-lab, then you will forfeit the opportunity to perform the lab. You will still be required to write up the lab, with a provided “dry lab” data set, including all data analysis and answers to questions. The maximum grade that this lab report can earn is 50% of the normal maximum. This policy has been put into place as a result of unprepared students who either endanger themselves or the instrument, or take too long to get through the experiment. **Being prepared for lab is to your own benefit!**

## **Lab Report Policy**

### **1. Description of Reports**

There are two different types of lab reports in this course, **Lab Memo** and **Formal Report**. Descriptions follow for which sections are required for each type of report. The Formal report is expected to be much longer and more thoughtfully interpreted.

### **2. Word Processing Policy**

Reports must be generated using computer software, no part of a lab report should be hand written except for notations on attached data (i.e. functional group assignments written on an IR spectra).

### **3. Data**

You will take all hardcopies of data acquired during the lab with you. Remember that instrumental make, model and parameters, unknown numbers, and hard copy such as chart recordings should all be included.

### **4. Due Dates and Late Report Policy**

Memo and informal reports are due **SEVEN DAYS** from the scheduled completion time for the experiment and must be uploaded to Moodle. If an experiment was scheduled for completion on Monday, Sept. 5th for example, it is due Monday, Sept. 12th.

Formal reports are due **FOURTEEN DAYS** from the scheduled completion time. **Late Reports will be heavily penalized.** A report turned in one lab period after the due date will receive a 20% penalty. The maximum grade a lab report turned in one week late can receive is 50%, but they must be turned in anyway to be evaluated as a “minimally acceptable report” in order to pass the course. You are strongly encouraged to avoid falling behind on your lab reports! A summary of the policy follows.

## Laboratory Report Grading Policy

Memo and informal reports are due one week and Formal lab reports are due 2 weeks from the day you are scheduled to complete the experiment.

It is understood and expected that some experiments will not be completely successful; this does not negate the need to turn in a report. Your group might be required to schedule additional time with the instructor to complete your data collection if necessary. Include any data that you have and give your best explanation of what went wrong and what might be done differently if the lab were to be repeated for better results. If you do not include sufficient detail in your report your grade will be penalized.

Every effort will be made to grade lab reports in a timely manner, however graded lab reports will not be returned until the entire class rotation has finished with the experiment.

A minimally acceptable lab report must be turned in for each lab in order for the student to pass the course with at least a "D."

### Report Formats

Memo reports are worth 1000 points each and Formal reports are worth 100 – 200 points each. The following is a **general** breakdown for both types of reports, and a description of what is expected in each section of the report.

#### A) Memo Reports (100 total pts each)

Short reports are generally in response to a specific request from a supervisor or a client. There are several different types and each has its own objectives and styles, appropriate for the particular situation. The investigative type, which analyzes data and seeks to answer what or how much of something is contained in a sample will be used in this laboratory course. To limit length, all short reports must be clear and concise.

A typical short report is divided into sections, e.g. for this class **Purpose, Findings, and Data & Results**, along with possible **Appendixes**. Section headings are aligned with the left-hand margin in a memo and may be simply capitalized and/or given bold or italicized font. The Purpose and Findings together should be one to two pages for most experiments. Recognize that the format itself is also flexible; however, all the critical elements or information need to be included.

Most data tables belong in the Data & Results section but anything longer than one page may be included as supporting documentation in an Appendix, in this course that is most likely to be copies of chromatograms or spectra. Text should avoid all usage of first person (we, us, our) second person (you, your) or third person (they). The text should be professional in nature, not conversational. This is a report of findings, so everything should be in the past tense. Do cite your sources using ACS format, even for websites.

#### B) Memo Report Format:

*Your memo should be concise and informative. To achieve this it needs to be organized. Writing a memo is NOT easy! The following are elements generally found in the memo, with each section labeled. The report starts with the heading, followed by the body of the report, and finishes with tables, graphs, and sample calculations.*

TO: (readers' name and job titles)

FROM: (your name and lab partner names)

DATE: (date report submitted)

SUBJECT: (what the memo is about, descriptive name of experiment)

**Purpose** – Very brief description of what analyte you were analyzing and what instrumental technique was used to do the analysis. This is the 'question' you are answering.

**Findings** - A synopsis of the essential information. The client should only have to read the Purpose and Findings sections to obtain the requested information. This is the 'answer' to the question. **Begin the section with the most significant results and conclusions.** Include numerical results with statistical description of uncertainty

whenever possible. Present an interpretation of the data, are the results reasonable? **Always** look at available packaging and on the internet to gain information about the expected results for each experiment and compare the “literature values” when available with your results . Discuss the sources and relative magnitude of any significant errors as well as any suggestions for changes in procedure.

### **Data & Results**

Begin with short procedure; do NOT rewrite the lab manual, just give a brief description of what you did. This section should a concise overview of major points using complete sentences. Assume your reader is a scientist generally familiar with the instrument and technique. Do not include steps that are not crucial to doing the experiment - the fact you used a 250mL beaker for step 2 is not a useful detail in a brief report, the fact that there was difficulty maintaining the temperature of a water bath and it varied from 70-90°C might be significant.

Include tables and/or graphs as appropriate. Graphs and tables are very effective and efficient methods for presenting lots of information or data in a short space. Include tables with raw data as well as the calculated values. The results section should not just be a table of values or listing of numerically crunched numbers. The section should stand on its own and be understandable. Use WORDS to string together numbers that you calculated. Do include sample calculations showing first the formula with all variables, then the formula with your numbers included, and lastly the final calculated answer with units. All numbers reported in the Findings section should be somewhere in the results. This section will be the longest section for most of the memos you write.

After you have completed the memo, look at the heading and closing sections and ask: Have the audiences, source, subject, and relevant reference information been identified so that the recipient(s) immediately know the significance of the letter?

Look at the first one or two paragraphs and ask: Is a concise statement of the client’s request and/or the problem and the objective of the test provided? Are the important conclusions and recommendations briefly stated so that the reader will not have to read further into the report? The reader should be able to get the bottom-line results and recommendations within the first two paragraphs without digging further into the memo itself or the appendixes.

Look at the overall Purpose and Findings sections and ask: Are the bottom-line results given in a clear, concise format? Is the data interpreted for the reader so they know the physical implications of the results? Is error quantified and error sources identified qualitatively? Are the final recommendations clearly stated? Are all questions asked in the lab manual answered?

### **C) Formal Reports (200 total pts each)**

The following is a **general** outline of what needs to be included in the report, but all experiments have a different emphasis and it is acceptable to be somewhat flexible when an experiment does not fit this format well. Informal reports will exclude the introduction section.

**Abstract:** This is a very concise (less than 200 words) statement of the results and how they were obtained. Include the most significant numerical results in this section! This should not be *written* until after the body of the report is completed even though it appears first in the report.

**Introduction:** This is a brief statement of the chemical problem and the technique(s) used to solve it, including a short justification of the analysis in terms of the underlying physical or chemical principles involved. Include background and theory here!

Also in the introduction describe techniques related to the one used in this experiment. By "related to" it is meant any technique that could be used to perform the same or a similar analysis or that gives the same type of information. For example, in an ICP formal report one would discuss other important instrumental forms of elemental analysis and atomic spectroscopy, such as X-ray fluorescence and optical or mass spectrometry. Emphasis should be placed on what each technique has in common with the others -- essentially on how you have decided the issue of whether or not a technique is related to the method in question. What are the unifying aspects of the techniques surveyed? Introduce only as much theory as you need to make clear your discussion of these similarities. Briefly discuss the most important differences between the various techniques only after their similarities have been so established. Why was the method of this lab chosen to solve the given problem, and are any other techniques perhaps more appropriate? This section should be at least one but no more than two or three pages.

**Calculations, Graphs, and Results:** Begin with a brief statement of what experiment was performed and what samples were analyzed. This should be 2-4 sentences and contain enough information that someone reading your report understands the context of the data that follows.

Any tabulated data acquired during the experiment along with photocopies showing your of printouts or any other form of "data" as described previously. **Be sure that the data contained in your report matches the data acquired in the laboratory.** Points will be awarded both for presentation and for experimental completeness. For experiments with a lot of pages of data it may be acceptable to include only a couple of sample pages instead of all data, this is primarily true for the separations experiments. For spectroscopy experiments look for ways to include multiple spectra on the graphs in order to fit lots of data into fewer pages (you do NOT need to include data tables with the entire absorbance spectra data if you have included the data as graphs).

Calculations must be neatly typed. One and only one sample calculation for each important type of calculation should be shown. Computer printouts of statistical results should be attached, if applicable. Graphs should be done on a computer with special attention given to generating a best fit line (when plotting linear calibration data). Graphs should have titles and labeled axes (with units), and if it is necessary to put different data sets on the same graph the data should be clearly distinguishable from set to set.

**Discussion:** Included in this discussion should be a thorough, critical analysis of all potentially important determinate and indeterminate errors, some estimate as to their relative magnitude, and a statement of how they contribute to the overall uncertainty of the results. Reference your error propagation calculations as necessary. Tell how you might improve the accuracy and precision of your measurement (excluding gross personal errors, please!), if applicable. You will be evaluated here essentially on your analytical thinking skills, so you will serve yourself well to start thinking about the limitations of your measurement as it is performed.

**Questions:** Some questions may have been answered in the natural course of writing the report

and most can be included in the discussion; if not answer them in detail here so that they are given careful consideration by the grader.

**References:** A bibliography referencing all literature sources used for writing the report, including sources of physical constants, must be presented in standard form (see any ACS journal) in the back of the report. **You must use outside sources and have multiple references for the formal report.**

Finally, part of your grade will be assigned for overall **English proficiency, report presentation**, etc. No attempt will be made to grade these aspects of the report on a point-by-point basis; your grade here is essentially a rating on a scale of one to ten. Give careful attention to your spelling, grammar and writing clarity!

### Graphs and Figures in lab reports

- a) Use a software program such as Excel, graphs cannot be handwritten.
- b) Label graphs with a title that unambiguously explains what is being presented. An example of a title for a graph would be Calcium Atomic Emission Calibration Curve.
- c) **Choose a scale for each axis so that the plot fills the page. It is not necessary to start with zero at the origin.** Zoom in on the part of the scale with meaningful data. (i.e. don't include 0 to 1000nm when all your datapoints are from 400 to 800nm)
- d) Label each axis and include units - e.g., Time (min.) or Temperature (°C).
- e) Always plot the **independent variable on the x-axis** and the **dependent variable on the y-axis**.
- f) If it is necessary to plot more than one set of data on the same axes, use a different mark and a different style or color of line for each data set, and **include a legend that identifies each one**.
- g) The points should be plotted as small as possible with error bars to indicate the associated error of each point.
- h) **DO NOT** "connect the dots" on your graph. The straight line fit should be the results of a linear regression operation; or if nonlinear, a smooth curve should be drawn through the points with a French curve or a flexible curve drawing device.
- i) A least-squares analysis should be done when appropriate and the results reported in the Data and Calculations section of the lab report. You must include more than one significant figure for the least-squares fit.  $0.005x + 0.002$  is not sufficient for quality calculations,  $0.00484x + 0.00211$  with three significant figures should be the minimum unless the data plotted is unusually low in significant figures.

• **Approximate Assignment of Points (Subject to Change)**

<b><u>Lab Reports*</u></b>	<b>60%</b>
Prelab activities and Notebook	10%
Reports	50%
<i>Memo reports</i>	
<i>Formal reports</i>	
<i>Informal reports</i>	
<b><u>Independent Project</u></b>	<b>30%</b>
Proposals and Updates	5%
Poster Presentation	10%
Final Written Report	15%
<b><u>Safety</u></b>	<b>5%</b>
<b><u>Instructor Evaluation</u></b>	<b>5%</b>
<b>Total of</b>	<b>100%</b>

## Instructor Evaluation

The evaluation of your performance in the laboratory has been assigned a percentage of the final grade in the course. The experiments in Chemistry 358 require very thorough preparation before starting the lab period. Without this preparation, completion of the experiment is difficult, and you become a liability both to the working order of the equipment and to the personal safety of the other students working in lab. It is for this reason that student preparation and competence in lab will be the most important factors considered when evaluation points are awarded. The points are broken down as follows:

- a. 20% Safety
- b. 20% Preparedness
- c. 20% Citizenship
- d. 20% Technique
- e. 20% Chemical Knowledge

## Student Conduct and Academic Integrity

I expect everyone to avoid academic dishonesty, which includes, but is not limited to cheating, plagiarizing, fabrication of information, facilitating acts of academic dishonesty by others, having unauthorized possession of examinations, submitting work of another person or work previously used without informing the instructor, or tampering with the academic work of other students. Serious disciplinary action is warranted for any observed violation of academic integrity up to and including a failing grade for the class and formal charges at the university level as described in the Concord University Student Handbook.

The guiding principle of academic integrity is that a student's submitted work must be the **student's own work**. In this course you will sometimes work in groups to collect experimental data and to analyze your results. This can lead to misunderstandings regarding academic integrity. The following is expected:

- \* You will adhere to all rules and regulations regarding safe laboratory practices as detailed in the lab manual, in the "Student Safety Agreement", and by the instructor. Failure to do so may be considered a code of conduct violation and can result in expulsion from lab.
- \* Each and every scheduled lab period, you will actively participate in the performance of experiments and collection of data. Failure to turn in a minimally acceptable lab report for each lab experiment will result in a failing grade.
- \* Lab reports are individual efforts, in cases where you submit one report per lab pair you **both** must contribute significantly to **each** report. You are not allowed to take turns writing reports and suspicion of such may result in a failing grade on the lab report for both partners. You are to submit your own, original work for all assignments. In cases when you work with other students collecting and analyzing data, you must indicate in your report which data are yours and who provided any other results you present. Failure to attribute laboratory results to the person(s) responsible for collecting the data is an academic integrity violation. Likewise, group analysis must be so identified. Submission of any material that is substantially the same as some other written document (e.g. another report, a journal article, a textbook, or a web page) and is not properly attributed constitutes an academic

integrity violation. In no case should you use someone else's data and write it up as your own, even if you have their permission.

Any form of academic dishonesty, including cheating and plagiarism, will not be tolerated in CHEM 358.

Cheating (including plagiarism) will be punished as severely as allowed under University guidelines as described in the Academic Handbook. Having been found guilty of cheating can be as bad as having had a felony conviction when employers look over your college record. It is an extremely serious offense, and students have been expelled from universities (and subsequently denied admission to another university) for cheating on exams or lab reports. Be sure to name all references you use when writing your reports. **This includes web pages!**

Students caught copying a lab experiment, altering data on a lab experiment, turning in photocopies of laboratory data (i.e. graphs, data table, calculations, etc.) not their own or turning in a lab report of other work which they did not do will face consequences ranging from a zero on the report to a failing grade in the lab for the entire semester. This information may be sent to the appropriate university authorities who may take additional action.

### **Make Up Policy**

In the event that you experience difficulties in completing the experiment (not including instrument difficulties out of your control) and you came to lab sufficiently prepared, you may be allowed to schedule a make up on that instrument if it is not otherwise being used. The make up must be completed in a timely fashion *as the original due date will still apply*.

### **PRE-TESTING EXPERIMENT ASSIGNMENT**

For a selected set of experiments, each of the groups will be assigned to pre-test one experiment before the rest of the class performs it. This will involve making solutions, testing the equipment and procedure, and preparing a brief instruction sheet and presentation to your classmates instructing them how to run the instrument for the given experiment. The pre-testing will be done outside of normal class time and I will assist but not lead in this endeavor. The pre-testing must be completed a full week before the module containing this experiment begins. For some experiments this will take the same amount of time as in a normal lab period but if the pre-testing turns up difficulties it may require substantially more time. Be prepared for this possibility! You will want to read the chapter in *Robinson* on your technique and may find other useful sources on the internet or in other textbooks.

It is expected that you will gain valuable experience in the preparation process required for a smooth experiment and in individual troubleshooting. Some instruments used in this class are used only once a year and require some work before they operate correctly. Some of the software that controls the instruments require specific knowledge to operate correctly. You will prepare a **handout roughly 1-2 pages long** on how to run the experiment, including everything but focusing on describing how the software is operated such as starting the program, specifying the collection parameters, saving the data, and exporting or printing the results. Also include on this handout any warnings needed to operate the instrument safely and without damaging either the instrument or its accessories. (For example, don't put aqueous samples on a salt plate for IR

or you will dissolve your substrate!) You do not need to put together an entire experimental procedure for the experiment, think of this more like a useful "cheat sheet." You will also give a **brief presentation to the class** in the lab on how the instrument works and things to watch out for. **Include in this presentation the basic theory and applications for the instrument as we will often be doing experiments in lab that have not been covered in class yet.**

## THE INDEPENDENT EXPERIMENT PROJECT

### A. Purpose

The Independent Experiment Project is intended as an opportunity for you to design a chemical analysis protocol for a sample of interest using the variety of instrumentation available in the lab. Ideally, the kinds of problems that will be addressed are the same problems encountered by any chemist faced with a practical chemical problem -- sampling, sample preparation, purity of samples, matrix effects, detection limits, and interferences. This project will thus be an opportunity for you to use the knowledge you have gained, both in this course and in the rest of your undergraduate career, to tackle a "real world" chemical analysis. Finally, you will present your work, both in a written report and in a poster presentation, and thus learn valuable "real world" communication skills.

### B. Experiment Design

You will design your own experiment but it must be based on a published method, not simply made up by you. You must choose one of the recommended ideas and utilize primary sources such as the *Journal of Chemical Education* (the last year is available in print in the common area in Science 401). You might consider one of the following three approaches:

- 1- Select two of the provided ideas. You will turn in at least two proposals and will have to discuss ideas with me before receiving approval. You will save time and effort if you discuss your ideas with me throughout the semester and **before** spending time writing up the formal proposals. The experiment should be something that can be carried out in three lab periods. Proposals must be limited to instruments we have available here at Concord and for obvious reasons I must limit how many students use each instrument. (Eight people trying to use the same piece of equipment on the same week is not a good plan for a fun week for anyone!)

### C. The Proposal

Once the various qualitative and quantitative questions about the product to be analyzed have been thoroughly researched, a proposal must be submitted. This proposal will contain a brief description of the analytical problem, a flowchart of how the analysis will be carried out, an outline of the experimental plan, including any sample preparation and standards that are used, a list of all equipment and reagents needed for the analysis and a list of references. The first draft of your written proposal will be reviewed and returned to you for necessary revisions. A written final report as well as an oral presentation or poster is required.

### D. Laboratory Work

The independent experiment will take place near the end of the semester. Before

beginning the lab work, the reagents and glassware must be obtained. A list specifying the quantities of reagents and glassware must be submitted **at least two weeks before** lab work on the project begins. Any special reagents or equipment that are not readily available will have to be ordered, and not all requests will be approved so it is in your best interest to submit requests as soon as possible.

## **E. USEFUL TIPS ON LABORATORY TECHNIQUE**

- \* When using the analytical balance, make sure the pan is clean (and leave it this way), that the balance is level (check bubble level) and that the tare is stable. Weigh your samples by difference. To do this, place your small vial containing the sample on the balance, tare the balance and then tap some sample into a vessel. Reweigh the vial noting the absolute value of the weight. Repeat until you have transferred an amount in the appropriate range for your experiment. Always check that the area is clean when you are done and return chemicals to their storage area, do not leave them next to the balance.
- \* When using glass pipets, first rinse the pipet with the solution to be transferred. Discard this rinsing and check to see that the pipet drains well without solution beading up. If it does not drain cleanly, more thorough cleaning is required. Start with a small amount of soap and water and progress to a stronger glass cleaner or dilute acid if needed. Always pour some of the solution that you wish to transfer into a small beaker first and then pipet out of that. Discard to waste any remaining solution- do not return it to the stock! When pipetting, a slightly moist finger on the top of the pipet combined with a twisting motion of the pipet in the other hand can greatly aid in the control the volume in the pipet as you are releasing volume down to the mark.
- \* When using the Eppendorf pipets, you *may* insert the pipet directly into the stock solution provided that you are using a new tip each time. Adjust the volume carefully on these pipets (which are about \$250.00!). Pull the top out to rotate to adjust. On some models, you must push in a side button to rotate the top to dial in the volume. In either case, *never* force the rotation of the top! When drawing up solution using an Eppendorf pipet, release the top slowly and check the contents of the tip for bubbles. You can expect these pipets to be accurate to about ~1-2%. A good way to calibrate these pipets for extra careful work is to weigh a given volume of water several times delivered to a tared beaker on a balance. Then average the masses (10 readings is a good number) and adjust the mass to volume given the temperature of the water and the density of water at that temperature.
- \* When transferring standard solutions from a volumetric flask to a Nalgene bottle for storage, make sure to rinse the bottle first with some of the standard solution. If the bottle has been recently washed, there may be residual water in it which will slightly dilute your standard.
  - Throughout the various experiments, be conscious of when you need to measure something carefully, and when it is not necessary. The text of the labs often includes directions when guiding you to transfer a certain amount; whether or not to just use a graduated disposable pipet (a very handy thing when an approximate volume is ok!), or a graduated cylinder, or for more careful work a Class A pipet. Knowing when it is worth the time and effort to be super careful and when it is not important is a valuable skill!

- **Accessibility/Accommodations:**  
 Concord University is committed to responding to the needs of students with disabilities as defined by the Americans with Disabilities Act. Please inform your instructor at the beginning of the class semester if you have a disability and are requesting accommodations. It is your responsibility to self-disclose that you are requesting accommodations. The University and instructor will provide you with a reasonable accommodation. You should register with CU's Disability Services Office, located in the Athens campus Jerry and Jean Beasley Student Center, Bottom Floor, across from the Campus Post Office. The Disability Services Office phone is 304-384-6086 or you can email the Director, Nancy Ellison, at nellison@concord.edu for assistance.
- **Academic Dishonesty**  
 Academic dishonesty is morally unacceptable as well as destructive to the learning and teaching atmosphere. Academic dishonesty includes the giving or receiving of improper help on examinations or assignments, falsifying documents, and plagiarism (the act of stealing and using, as one's own, the ideas or the expression of the ideas of another). Such dishonesty can lead to a variety of penalties — including but not limited to failure of assignment, failure of course, loss of institutional privileges, or dismissal from the University. (See University Catalog Academic Policies and Procedures.)
- **Concord University Honor Code**  
 A Concord University Honor Code was approved by students, staff, faculty, administration, and the CU Board of Governors. The Code states:  
*"As a member of the Concord University Community I will act with honesty and integrity in accordance with our fundamental principles and I will respect myself and others while challenging them to do the same."*  
 The Honor Code is intended to unite the Concord community behind a culture of honesty, integrity, and civility.
- **Class/Online Attendance Policy**  
 Regular class attendance is part of a student's academic obligation at Concord. Irregular attendance may affect academic performance adversely and is detrimental to the atmosphere of a class. (See University Catalog Academic Policies and Procedures.)
- **Emergency Alert System**  
 In an effort to increase safety and security on our campus, Concord University encourages everyone to register for instant text message alerts. Alerts will only be used for security and safety notices. All students, faculty, and staff are eligible to receive text message alerts on their cell phones or email alerts. Please contact the IT Help Desk for further assistance (304-384-5291).
- **Emergency Information**  
 Emergency/courtesy telephones are located at the main entrance of each residence hall and at various other locations on campus. Emergency telephones can be identified by the flashing blue light and will provide the user with a direct link to Public Safety at the press of a button. To report an on-campus emergency, call 304-384-5357 or 911. The Office of Public Safety is located on the bottom floor of the Rahall Technology Center. For further emergency information go to:  
<http://www.concord.edu/administration/office-public-safety>.
- **Inclement Weather Policy**  
 As a general policy, the University will remain in normal operations during adverse weather conditions. In the event of severe weather conditions, the following may occur:
- University Closure  
 No students or employees are to report.

### Classes Cancelled

Students do NOT report BUT employees are expected to report to work at their normal time.

### Operating on an Inclement Weather Delay

Under this schedule, all 8 a.m. classes will start at 10 a.m. Students and faculty will follow the Inclement Weather Schedule. (See <http://www.concord.edu/emergency-alerts> for Athens/Beckley Inclement Weather Schedules.)

*\*Announcements invoking the late schedule or other options referenced above are aired on area radio and television stations and are sent as text and email messages to those enrolled for this service.*

- **Student Conduct**

In classrooms, online, laboratories, and during any activities that are part of course requirements, students are expected to observe reasonable rules of conduct.

- **Sexual Harassment & Assault**

Federal law, Title IX, and Concord University policy prohibits discrimination, harassment, and violence based on sex and gender (Including sexual harassment, sexual assault, domestic/dating violence, stalking, sexual exploitation, and retaliation). If you or someone you know has been harassed or assaulted, you can receive confidential counseling support through the Concord University Counseling Center (304-384-5290). Alleged Violations can be reported non-confidentially to the Concord University Title IX Coordinator at 304-384-6327 or [titleix@concord.edu](mailto:titleix@concord.edu). Reports to Campus Security can be made at (304-384-5357). As an employee at Concord University, I am a mandatory reporter which means I must report any sexual misconduct I am made aware of. This includes verbal or written (such as in an assignment) disclosures of sexual harassment or sexual assault.

- **Technology Services**

Contact the CU Help Desk at extension 5291 from campus or 304-384-5291 off campus. You may also e-mail [cuhelpdesk@concord.edu](mailto:cuhelpdesk@concord.edu).

### **Syllabus Disclaimer**

**"This syllabus is subject to change based on the needs of the class. Please check it regularly. It is your responsibility to remain updated with the materials and announcements made by the instructor during lecture and/or via email."**