

Intensive General Chemistry Laboratory

General Information

GENERAL INFORMATION

CHEM C2507y, Intensive General Chemistry Laboratory, is intended for students who have completed CHEM C2407x, Intensive General Chemistry Lecture, or CHEM C3045x, Intensive Organic Chemistry Lecture for freshmen. The course will provide an introduction to techniques and practices of modern experimental chemistry in a contextual, collaborative learning environment.

COURSE WEB SITE: <http://www.columbia.edu/itc/chemistry/c2507>

DIRECTORY

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SCHEDULE

Section	Day - Time	Room	Activity
<i>All</i>	Thursday - 1:10-2:00 p.m.	7 th Floor Lounge	Mentoring Session
<i>Friday</i>	Friday - 1:00– 6:00 p.m.	302-222 Hav	Laboratory Session
<i>Monday</i>	Monday - 1:00 – 6:00 p.m.	302 –222 Hav	Laboratory Session

NUTS AND BOLTS

(1) Learning Blocks

To facilitate your learning in this course, we have grouped the experiments in three sequential learning blocks; the first block will initiate you into collaborative learning and will last two weeks. During this time you will work on structured (laboratory manual-based) experiments that will cover basic laboratory techniques. You will report your findings in the form of in-class and take-home group reports.

The second block will last seven weeks and will be used to cover the theoretical background typical of a first-year laboratory course including kinetics, reactivity, spectroscopy, and quantum mechanics. You will perform structured-contextual experiments on a rotating manner, submitting mostly individual laboratory reports.

The last block of experiments will be dedicated to assess your learning by assigning you a two-week problem-based learning (PBL) project or case study in contexts including Environmental Chemistry, Forensic Chemistry, Food Science, and Materials Science. The supporting material for these case studies will be partially or completely unstructured. You will present your findings to the entire research community in the department of chemistry at the Undergraduate Poster Session.

LEARNING BLOCK	DESCRIPTION
I	Workplace skills
	Wet Techniques (Calibrating Glassware, Acid-Base Titration)
II	Chlorophyll Extraction (Partition Equilibrium, TLC, Column)
	Chlorophyll Photophysics (Visible, Fluorescence, and Laser spectroscopy)
	Fourier Transform Infrared Spectroscopy, FTIR
	Chromatography (HPLC/GC)
	Laser properties
	Flame Atomic Absorption (Coral Tank chemistry)
	Electrochemistry (Coral Tank chemistry)
III	Case Study, PBL project, three weeks
	Undergraduate Poster Session

(2) **Group Formation:**

You will be assigned to your group according to the bench number you will receive during the first laboratory period (1/28-1/31). Each group will be named after an element in the periodic chart (Li through F) and will be assigned to a mentor (TA). You have to remain in this group throughout the semester, unless you request a transfer due to extenuating circumstances.

Transfer will be honored only during the first two weeks (first learning block) under the following conditions: you find another student from a different group who is willing to switch with you, or you make a written request to your mentor for transfer. In the later case, your mentor, in consultation with Professor Avila, will arrange for your transfer only if your reasons deem substantially sufficient. Due to constraints of the course structure, transfer requests cannot be accepted after the beginning of the second learning block, third session (February 11-14).

(3) **Collective Responsibilities:**

The success of your group depends on the contribution of each member to the group goals. In this sense you must show¹:

- a) **Positive interdependence:** as team members, you have to rely on one another to achieve the group goal. If any team member fails to do their part, everyone suffers the consequences.
- b) **Individual accountability:** you are held accountable for doing your share of the work and for mastery of all of the materials to be learned.
- c) **Face-to-face promotive interaction:** you must provide one another with feedback, challenge one another's reasoning, teach each other the intellectual or psychomotor skills acquired, and encourage one another to excel in their own work.
- d) **Appropriate use of collaborative skills:** you must develop and practice trust building, leadership, decision-making, communication, and conflict management skills.
- e) **Group processing.** You must set group goals, periodically assess the progress of the team, and identify and promote group changes.

¹Felder, R. and R. Brent. ERIC Document Reproduction Service Report ED 377038 (1994).

Group responsibilities will be assessed by monitoring your:

- Attendance to the mentoring sessions
- Participation on the preparation of the Plan of Action
- Participation on the preparation of the Case Study Plan of Action
- Participation on the presentation of the Case Study Group

(3-1) Attendance to the Mentoring Session

Mentoring sessions are organizational meetings for the groups held on Thursdays before the experiments start according to the schedule on page 15. The first mentoring session will be held on January 20. Except for the first meeting, depending on the type of report (individual or collective), during this hour each member of the group will present their share of the work for the laboratory report, i.e., review the results from the last experiment, and brainstorm about the Discussion of Results and Conclusions sections of the laboratory report. During this hour you must also prepare the Plan of Action for the experiment you will be doing next session. It is therefore imperative that each team member makes ample preparation before attending the mentoring session such as analyzing the data from the previous experiment, reading the manual for the next experiment and review the pre-lab lecture on the course website. You need to agree on a way to communicate before these meetings so that you can maximize the help provided by your mentor by asking pertinent questions. We recommend that you allocate the tasks/portions of the report before leaving the laboratory.

(3-2) Plan of Action

The Plan of Action for each experiment will be conceived during the mentoring session before the experiment (Thursday, January 27, 2005). This document will be clearly handwritten and should describe the distribution of the experimental tasks among the group members. It must include a well thought strategy to complete those tasks in the allotted time. Essentially you should cover: what you want to do and how you will do it; indicating division of labor and time frames for the tasks. You need to keep track of task allocation by member and have a signed contract for it. Material such as the MSDS (Material Safety data Sheets) of the main chemicals to be used, instrumentation guidelines, and the waste disposal protocol for the experiment must be written prior to the

mentoring session as described in the Section on laboratory notebook.

(4) Individual Responsibilities

(4-1) Preparation for the experiment

You must prepare for the experiment before the mentoring session. Read and comprehend the experiment in the manual and the recommended links on the website, which includes the pre-lab lecture. You need to check the preparation of your team members and make sure they also understand and come prepared to the mentoring session. Your mentor will assess your preparation during the laboratory period.

(4-2) Laboratory Notebook

During the mentoring session each team member must write the Plan of Action on his/her own laboratory notebook. Although the manuals for specific instrumentation will be available, the laboratory manual will not be permitted in the laboratory. At the end of the period, turn in all duplicate pages of your records for assessment purposes.

Leave the first page of the notebook for a Table of Contents. For each experiment, write the title, date, and names of the team members on the top of the first page of the record. Divide the notebook into two columns as depicted below, use the left column for procedural observations and reserve the right column for experimental observations.

<p><u>Name</u> <u>Date</u> <u>Experiment title</u> John 1/28/02 Density of peanut butter</p> <p><u>Group members:</u> Kathy, Max, Marie</p> <p><u>Purpose:</u> The purpose of this experiment is to determine the density of peanut butter.</p> <p><u>Safety notes:</u> Do not eat the peanut butter used for the experiment; it might be contaminated with chemicals.</p> <p><u>Procedural observations:</u></p> <p style="text-align: center;">(...)</p> <p>Transfer the peanut butter into the flask containing water.</p> <p style="text-align: center;">(...)</p>	<p><u>Pre-Lab assignment:</u> Density = Mass/Volume If Mass =10g and Volume =1.00 cm³ Then Density=10 g/cm³</p> <p><u>Data & Observations:</u></p> <p style="text-align: center;">(...)</p> <p>During the transfer process a few drops of water were spilled out.</p> <p style="text-align: center;">(...)</p>
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Be organized in writing observations; when appropriate draw data tables to organize data. Before coming to the laboratory, you must have completed the left column

for that day's experiment including date, names, title, purpose, procedure, MSDS of the main chemicals to be used, instrumentation guidelines, and the waste disposal protocol. Perform calculations as required and write a flowchart containing each step of the experiment as if you were doing the experiment on your own. Include a list of any changes from the manual's procedure. During the mentoring session you will agree on the distribution of the tasks, and hence which parts of the flow chart you will be using.

At the end of the laboratory period, sign at the end and tear down the duplicate pages of your record. Staple all pages together and submit them to your mentor.

(4-3) Laboratory Reports

Experiments 3, 4, 6 and 7 will be assessed through individual laboratory reports as shown on table 1 in italics. The reports are due as shown on table 1. Late lab reports will not be accepted.

Table 1

Exp. #	Experiment Name	Assessment Activity
E1	Workplace skills	In-Class Poster Session (group assessment)
E2	Wet Techniques I	Group Report Sheet (Calculations and Discussion of Results)
<i>E3/E4</i>	<i>Chlorophyll Extraction and Photophysics</i>	<i>Individual report including extraction and photochemistry. Due the week after both parts of the experiment have been completed.</i>
E5	FT-IR	Answers to IR-Tutor handout. Individually handed in at end of period.
<i>E6</i>	<i>Chromatography</i>	<i>Individual report. Due next week after experiment</i>
<i>E7</i>	<i>Laser properties</i>	<i>Individual report. Due next week after experiment</i>
E8	Coral Tank chemistry I	Group report on Coral Tank Chemistry including AAS and electrochemistry results. Due the week after both parts of the experiment have been completed.
E9	Coral Tank chemistry II	
CS	Case Study	Poster Session (group assessment)

The goal of writing a lab report is to communicate your findings in a concise, thorough, and thought-out, manner, which clearly demonstrates the meaning of what you did in the laboratory. If in doubt, ask someone in your group or someone from the teaching team to clarify concepts to you.

The information in the lab report will be distributed by sections, which include Title, Abstract, Introduction, Experimental Method, Data and Results, Discussion and Conclusions, and References.

Abstract and Title page (10 points): When all sections of the report are completed to your satisfaction, write an abstract of no more than 120 words summarizing the report. Ideally, the abstract should have one sentence per section to highlight the main points of the report. Address the research questions and summarize the results/findings of the experiment. The Title Page should contain the title of the experiment, the abstract, the name of the members in the group, the date the experiment was performed, and the date the report was submitted.

Introduction (10 points): explain the theory behind the experiment as if you were describing it to someone in our class who hasn't yet done the experiment, e.g., if you were writing an individual report for experiment E2, you would define accuracy, precision, and standard deviation, but you can assume that the reader knows what an Erlenmeyer flask is. Include references for all sources consulted.

Experimental Method (10 points): Indicate specific details of the apparatus used [e.g., "A Matson Galaxy FT-IR spectrometer equipped with a DTGS (deuterated triglycine sulfate) detector with optimal optical path difference (OPD) resolution of 1.0 cm^{-1} was used to collect the infrared spectra of the reaction products and starting materials]; the physical properties of chemicals used, the number of runs, the experimental conditions (concentration range(s), temperature, pressure, and other experimental variables of relevance). Mention any modification from the procedure described in the lab manual.

Data and Results (30 points): Tabulate all data used in the calculation of results, number the tables and title them properly. Perform error analysis of your data. Provide one sample calculation for the results and the associated uncertainty and then tabulate the rest. Use a different numbering pattern for tables, figures, graphs and equations. Describe how you calculated the results using the proper units and significant figures.

Discussion and Conclusions (30 points): The purpose of the discussion is to expand on your observations or to comment on possible causes of poor results. Make a quantitative comparison of your results with literature values; based on this, evaluate the experiment's accuracy and precision. Answer any questions given at the end of the experiment. Summarize the main conclusions and results.

References (10 points):

Bibliographic styles depend on the journal that has defined the bibliographic format; the following citation norms pertain to the Journal of Physical Chemistry. Notice that in this format you are to number the citations along the text and present a list of the references at the end of the report as exemplified below.

a. Citing an article:

- (1) Selco, J. I.; Roberts, J., Jr.; Wacks, D. B. *Journal of Chemical Education* **2003**, *80*, 54.

b. Citing a book:

- (2) Szafran, Z.; Pike, R.; Singh, M. *Microscale Inorganic Chemistry*, John Wiley & Sons, Inc Publishers: New York, 1991.

c. Citing a chapter in a book of several authors:

- (3) Avila, L.; Fine, L. Infrared spectroscopy and education. In *Handbook of vibrational spectroscopy*; Chalmers, J. M., Griffiths, P. R., Eds.; John Wiley & Sons, Ltd.: Chichester, UK, 2002; Vol. 4; pp 3207.

To facilitate handling bibliographic files you should download the EndNote bibliographic software, which is available for downloading, free of charge, to all current students, faculty and staff of Columbia University from the AcIS software server:

<http://www.columbia.edu/cu/lweb/help/howto/endnote/index.html>

In order to better organize your report, you can use the templates provided by the American Chemical Society. The site below corresponds to the Journal of Physical Chemistry template:

https://paragon.acs.org/paragon/application?pageid=content&parentid=authorchecklist&mid=mt_jp.html&headername=Manuscript%20Templates%20-%20The+Journal+of+Physical+Chemistry

Individual responsibilities will be assessed by monitoring your:

- Completion of individual laboratory reports.
- Answers to the IR-Tutor navigation questionnaire (E5).
- Preparation for lab. Your mentor will monitor your active participation during the mentoring session.

- Organization of laboratory notebook. Your mentor will check that you have a complete flowchart for each experiment and that you keep organized notes of your work in the laboratory.
- Workplace skills. At the end of the term you will fill up an assessment rubric about your peers' interpersonal, conflict management, time management, and other relevant workplace skills. You will fill up the same rubric evaluating your own group performance for self-assessment.
- Safety and housekeeping habits, laboratory techniques, and your ability to coordinate tasks. Occasionally your mentor will ask questions about safety and procedures or will request you to demonstrate a technique during the lab period.

(5) Attendance Policy and Make-up

As described earlier in the section on Collective Responsibilities, the success of the group depends on the contribution of each individual to the group goal. Therefore, absence from a laboratory period is unacceptable, except for a reason such as medical, religious observance, or any other emergency. Also, due to the course structure, it is impossible to make-up experiments. The following attendance policy will be in effect:

- You may miss only one laboratory period provided that you notify your group members and mentor and that you have a valid excuse.
- If you missed an experiment, your team members have to assure that you understand and master the principles covered in the experiment, and you will have to write an individual lab report. In addition, your mentor will interview you to complete the assessment.
- Absence beyond the limit of one would seriously affect your grade in the course.

(6) Grading

Your letter grade for the course will be computed according to the following scale:

Group grade 50%

<u>Assessment form</u>	<u>Total points</u>
Plan of action (E2 through E9)	160
Group reports (E1, E2, E8/E9)	60
Case Study plan of action	30
Case Study poster and presentation	30
Total Group Grade	280 pts.

Individual grade 50%

<u>Assessment form</u>	<u>Total points</u>
Three Individual laboratory reports (E3/E4, E6, E7)	120
In-class FT-IR assessment (E5)	30
Preparation for lab	30
Laboratory notebook	40
Peer Assessment	30
Safety, techniques and procedure grade	30
Total Individual Grade	280 pts.

(7) Safety Guidelines

http://www.hr.columbia.edu/ehrs/html/laboratory_safety.html

All laboratory safety protocols in CHEM C2507 will be strictly enforced. Violation of any safety protocol may result in point deduction on the Procedures, Techniques and Safety section of your individual Evaluation Sheet, and/or dismissal from the laboratory.

Safety has two aspects: prevention of accidents and response to emergency. The golden rule in emergency situations is to use your common sense. Treat your classmates and chemicals in the laboratory with respect. Do not work in the laboratory alone or perform unauthorized experiments. Ask your instructor whenever you do not know how to perform a procedure. Notify your instructor immediately when there is an accident (including broken glassware, chemical spill, and bodily injury).

(7-1) Material Safety Data Sheet (MSDS)

http://www.hr.columbia.edu/ehrs/html/accessing_msd.html

Federal law requires that manufacturers and distributors of chemicals provide users with Material Safety Data Sheets (MSDS). MSDS is a fairly concise technical document that gives information on any particular chemical among the over 10,000 frequently encountered chemicals in research laboratories and industries. The information includes contact address and phone number of the chemical supplier, chemical names, physical and chemical properties, physical hazards (such as flammability, reactivity), toxicity data and health hazards, storage and handling procedures, emergency and first-aid procedures, disposal, and transportation information. The MSDS for a chemical can be conveniently located by submitting the name of the chemical to a MSDS searchable database on the Internet. In the Case Study, you will come into contact with many chemicals that you may be unfamiliar with. It is your responsibility to read the MSDS for any chemical that you plan to use for your particular experiment or analysis.

(7-2) Chemical Waste Disposal

http://www.hr.columbia.edu/ehrs/html/hazardous_regulated_waste.html

No chemicals can go down the sink. Aqueous, organic, and solid wastes should be disposed properly in clearly labeled containers. Ask your instructor when in doubt.

(7-3) Chemicals Storage

Toward the end of a lab period, you will be asked to clean up your working area in the laboratory. At that time, decide what samples of chemicals you will save for the next lab period. Glass vials of various sizes should be used for aqueous and organic solutions and solid samples. If you are not certain how to store properly your particular sample, ask your instructor.

(7-4) Off-hour Use of the Laboratory

Unauthorized use of the laboratory facilities is a serious violation. If you need to repeat a measurement during any morning (Monday through Friday) when there is no class, you must ask permission from your mentor.