

Chemistry 205L. Laboratory in Organic Chemistry II Fall 2007

“A good observer is one who sees what he is not looking for if it is there and who does not see what he is looking for if it is not there:” Frank Galbraith

Course Description: Laboratory work related to CHEM-205; a continuation of 106L. This course must be taken concurrently with CHEM-205.* Prerequisite: CHEM-106L. Three hours per week. *One semester hour.*

Class meetings: Monday or Tuesday or Wednesday or Thursday;
1:30 – 4:20 PM in the J.R. Lovett Organic Chemistry Laboratory (Pfahler 315).
Pre-lab discussion/lecture in Pfahler 210 at 1:30 PM.

Textbook: John W. Lehman, *Multiscale Operational Organic Chemistry*;
Prentice Hall: Upper Saddle River, NJ, 2002 [ISBN: 0-13-015495-4].

Notebook: Student Lab Notebook with Permanent Binding (100 pages),
Hayden-McNeil Publishing Company [ISBN: 1-930882-00-9].

Instructors: Mon: Dr. Ronald Hess Pfahler 313C Ext. 2345 e-mail: rhess
 Tues: Dr. Victor Tortorelli Pfahler 313B Ext. 2239 e-mail: vtortorelli
 Wed: Dr. Ronald Hess Pfahler 313C Ext. 2345 e-mail: rhess
 Thu: Dr. Amy Brown Pfahler 205B Ext. 2383 e-mail: mknechel

Safety: Suitable safety glasses must be worn at all times in the laboratories by all students. Appropriate clothing must be worn to ensure your personal safety. Read carefully the material contained in your textbook on pages 9 – 18; these topics are entitled “Laboratory Safety” and “Reacting to Accidents.”

Grading: Your grade in this course will be based on the following components, the quoted number of points being approximate and subject to change:

	<u>approximate % of final grade</u>
Laboratory reports, safety reports, quizzes	50
Laboratory notebook	10
Examinations	40

Examinations: Two examinations will be given—one focusing on work of the first six lab periods; the other on the last seven lab periods:

Thursday, 18 October 2007; 7:00 – 8:30 PM. in Pfahler Auditorium and Pfahler 207

Thursday, 06 December 2007; 7:00 – 8:30 PM. in Olin Auditorium

Quizzes: Quizzes will be given to different sections during different weeks, at the discretion of the individual instructor. To prepare for a quiz, simply have your notebook fully completed as per the instructions contained in this syllabus. The quizzes begin promptly at 1:30 PM. **You may use only your laboratory notebook**, which may not

contain any photocopied or other electronically-reproduced sections. The notebook may contain only that which is hand-written in pen by you. You may not use the text or any other source. There will be no make-up quizzes given or extensions of time for lateness granted. The message? Arrive on time.

Safety Concerns: On the first page(s) of your notebook entry for each new experiment, you need to construct a ***hand-written*** table of ***reagents, reactants, and products***—anything you might come in contact with, ***structural formulas*** for each, important ***physical properties***, and ***safety concerns/hazards*** potentially associated with their usage (you must provide the ***source(s)*** for the safety concerns you cite.). For example, it is vital for you to know that diethyl ether and ethanol are highly flammable, that bromine liquid causes really nasty burns, and that sodium hydroxide is caustic. **The carbon copy of this notebook page must be submitted to the laboratory instructor no later than 5:00 PM of the day before your lab meets (for Monday lab students, this means the preceding Friday).** Late submissions will earn no credit, but this report of safety issues must be submitted prior to your going into lab.

For your safety as well as the safety of everyone else in the laboratory, you will not be allowed to enter the laboratory if this form is not satisfactorily completed and submitted in a timely fashion. ***Your signature and date at the end of your document testifies that the work is yours and only yours. Please note that you must sign and date the safety document; no credit will be given otherwise.***

We are determined to continue our tradition of safe labs at Ursinus. (Also see page 11 of this syllabus.)

Laboratory reports: For each lab period, a “Report of Results” form must be completed and submitted to your lab instructor by 5:00 PM of the day following your laboratory session. (Also see page 12 of this syllabus.)

Laboratory Notebooks: Please see the bottom of page 12 of this syllabus.

Classroom Etiquette: Please think not only of yourself but of others in the laboratory. A clean lab is a safe lab, so work to keep your own work space free from clutter as well as those common areas. Clean up your spills and other messes. Keep the balances and the surrounding space free of chemicals. When using an instrument, return it to its proper condition—clean and ready for the next person. Do not place disposable or broken glass in the glass disposal containers in such a fashion that sharp, pointed, or ragged pieces of glass protrude from the box. If you use the last of any chemical or lab supply, please tell the instructor or a lab assistant.

Attendance: We expect you to attend your regularly scheduled lab section each week. All labs are full. Consequently, the Department sometimes simply cannot accommodate every request for a change in section, although we try hard to honor every legitimate request. If at all possible, ***please make your request by no later than Thursday, 5:00 PM of the preceding week by means of an e-mail to Dr. Hess (ALL SECTIONS).***

Clearly state your reasons for requesting the change, and indicate those sections that you could attend. (You may be asked to provide appropriate documentation.) The Department cannot guarantee that we can allow you to make the change, but we will try our best to do so. If the change can be made, you will be notified by means of an e-mail from Dr. Hess, and a copy of this e-mail will serve as your “boarding pass” to the other section. Requests based upon attempted deception or dishonesty will be considered cases of academic dishonesty.

Our expectations: Each student is expected to do every experiment, using the proper amounts of materials specified in the Lehman text, the syllabus, or the *Blackboard*[®] site, whichever pertains. Each student is also expected to submit the results that he or she actually obtained. It is a serious breach of laboratory ethics (i.e., cheating) to do otherwise. Your laboratory notebook should reflect your work, should be accurate, well-organized, and kept up-to-date at all times. Your notebook may be collected for grading by your lab assistant or your instructor at any time.

ACADEMIC HONESTY. Your work must be written in your own words and comprise your own ideas. We hold you responsible for knowing **ALL** the rules on academic honesty, including plagiarism, spelled out in the Student Handbook, some of which follow:

“... ***You are cheating if you:***

1. Copy answers or use information from a fellow student’s paper during a quiz, test, or examination.
2. Divulge answers or information, or otherwise give improper aid to another student during a quiz, test, examination or accept such aid.
3. Relay or receive any improperly obtained or confidential information concerning a quiz, test, or examination. (Example: if one sees the test before it is given and transmits information concerning its contents or whereabouts to other students.)
4. Use or refer to any unauthorized notes, books, calculators, problem solving aids such as “cheat sheets” during a quiz, test, or examination.
5. Collaborate improperly with another student on an open-book or take-home quiz, test, or examination; or obtain information from an unsuspecting fellow student during such an exercise.
7. Commit an act of plagiarism in any form.
8. Borrow under false pretenses, steal or otherwise improperly obtain lecture or research notes, laboratory data, or any information gathered by another student and present it as your own work (examples ... laboratory reports or experimental yields ...), or knowingly collaborate with another student by making such material available to him/her.
9. Falsify laboratory data, notes, results, or research data of any type in any course and present it as your own work.
10. Steal or intentionally damage or destroy notes, research data, laboratory projects, library projects, library materials, computer software (including the intentional passing of a computer virus), or any other work of another student (or faculty member), out of malice, or for the purpose of sabotaging that person’s work and thereby gaining an unfair advantage to yourself.
11. Knowingly and willingly violate any special rules concerning research procedures, group assignments, or inter-student collaboration, which may be established by any instructor in any course.
13. Misrepresent yourself to an instructor or an administrator for the purpose of gaining special favors or extensions for academic work missed. Examples include but are not limited to lying about your health or the health of a relative, forging doctor’s notes.
14. Forge signatures on forms, documents, or letters pertinent to College business. This may include but is not limited to course of study sheets, drop/add forms, or doctors’ notes.

You are an accessory to cheating, and penalties may be applied to you, if you:

1. Witness or have direct knowledge of any of the aforementioned forms of cheating and fail to inform an authorized person (faculty member, administrator, proctor, or student assistant).
 2. You bring unauthorized materials into a testing area and fail or refuse to remove them when instructed to do so.
 3. You fail or refuse to comply with admonitions from a faculty member or authorized proctor to cease any activity, which might aid other students in cheating.”
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Department of Chemistry
Policy on attire in chemistry teaching laboratories and
chemistry research laboratories

1. Approved safety goggles or glasses must be worn at all times.
 2. No open-toed shoes, sandals, or clogs. Feet must be completely covered, including heels. Socks are recommended.
 3. Arms should be covered up to the elbow. No tank tops or muscle shirts.
 4. Torso area should be completely covered.
 5. Long hair should be tied back.
 6. Long pants that come down to the ankles must be worn. No shorts, skirts, or kilts.
- Approved Fall 2005*

The Nature of Organic Chemistry Laboratory 2007 - 2008

The undergraduate organic chemistry laboratory experience generally has two goals: to teach the basic manipulative skills (such as recrystallization, distillation, reaction set-ups) and to make the organic chemistry lecture material come alive. In CHEM-106L and CHEM-205L, both of these goals are addressed.

Laboratory work can be placed in three broad categories:

Structural Analysis. In most organic reactions, the identity of the product must be determined. If the compound is *known*, comparisons with properties reported in the chemical literature allow us to verify its identity. If *new*, the compound is characterized by its physical and chemical properties (especially spectral techniques), and often its structure is proved by independent synthesis or by degradative methods. In the latter case, a molecule is broken down into simpler molecules by known chemical reactions; the smaller pieces are then identified and the original molecule is reassembled employing sound chemical logic, sometimes in the lab but most often in the minds of those who carried out the reaction.

In all cases, the reaction product must be purified; otherwise, the observed properties are those of the combination of the given compound and the impurities present in the sample, not just the compound of interest. Impure compounds are simply mixtures, whose properties are often intermediate to those of the components. Purification techniques that you will encounter in your organic chemistry courses include distillation, crystallization (recrystallization), sublimation, extraction, and chromatography. Compounds will be characterized by morphology (shape and color), melting and boiling points, solubility, refractive index, adsorption behavior, and spectroscopic parameters.

Purification can often be a very time-consuming and frustrating process (be warned!), and this, of course, must be reflected in the cost of a chemical. To illustrate this point, check out the prices of calcium carbonate (CaCO_3 , limestone) in the 2005 – 2006 Aldrich catalog. The price you pay for 100 grams of ACS reagent grade CaCO_3 , $\geq 99.0\%$ purity is \$26.20. However, the price of 100 grams (4 x 25 grams) of CaCO_3 , 99.999+% purity is \$806.00 (of course, plus shipping charges).

Synthesis. As the name implies, a desired compound is prepared from material(s) of known structure, through a sequence of chemical reactions that will optimize yield. In the early 19th century, all organics were natural products. However, the exponential increase in empirical and theoretical knowledge over the past two centuries has allowed us to synthesize myriad compounds not found in nature, some for the good of humankind and some not. We create some molecules simply for the intellectual challenge; we create some molecules to make us well or keep us well, others to make our lives better or more comfortable, still others for less than noble reasons.

Many natural products were synthesized after their structures had been proven. The late Dr. Robert Burns Woodward—Professor of Chemistry at Harvard, 1954 Nobel Laureate in Chemistry, and arguably the greatest synthetic chemist who ever lived—carried out, with numerous co-workers, the total synthesis of such complex molecules as quinine, reserpine, cholesterol, vitamin B12, and chlorophyll, both as proof of the deduced structure and as a challenge to his intellect, imagination, and creativity. Not only a synthetic chemist, Woodward used theoretical predictions to arrive at the correct answers to many questions which had arisen from his synthetic work. (See Woodward-Hoffmann rules of orbital symmetry in Wade, 6th edition, p. 689ff.)

Reaction Dynamics involves the study of the rates of chemical reactions (kinetics) and the energy changes (thermodynamics) that accompany these transformations. Experiments determine the effect of temperature, concentration, solvent, catalysis, molecular structure, light, and other variables on reaction rates and equilibrium constants.

Our physical observations in these experiments often enable us to draw scientifically sound conclusions concerning what is happening at the molecular level, a level we cannot directly observe. In other words, we can learn about reaction mechanisms.

In the year 2006, chemical theory has advanced to a position where we can often make accurate predictions—both qualitative and quantitative—about the course of chemical reactions—the nature of the products and their properties, the rate of the reaction, the effect of catalysis, solvent effects, and the magnitude of the thermodynamic parameters (enthalpy, entropy, and free energy).

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Schedule of Experiments

Preparation for the Laboratory: Success in the laboratory depends upon your understanding of the experimental procedures, your understanding of the underlying theory of the experiment, and your efficient use of time. You should read the recommended pages that appear below, become familiar with the laboratory instructions, and understand what you are supposed to do and why you are doing it. Note carefully any safety instructions/precautions. Be sure to include in your notebook any hazards associated with the materials you will be using. **You will NOT be allowed to have your textbook in the laboratory, only your laboratory notebook.** You must write up the experimental procedure in your notebook prior to entering the laboratory. You should include a section on cleanup after the procedure, stating how you will deal with all waste.

Week #1: 27 – 31 August 2007

Introduction to the Laboratory: Lehman, pp. 2 – 9
The Laboratory Notebook: Lehman, pp. 802 – 803
Laboratory Safety: Introduction, pp. 9 – 18, and the attached syllabus
Planning an Experiment: Appendix V, pp. 810 – 812

Check-in to laboratory lockers [\$5.00 refundable key deposit required]

Blackboard®: The Photochemical Reduction of Benzophenone

You may use a screw cap bottle as the vessel for this reaction.

Week #2: 03 – 07 September 2007

Experiment #29: Synthesis of Triphenylmethanol ...

This is a two-week experiment. Today you will prepare the Grignard reagent, which will decompose if allowed to stand around, and react it with benzophenone:
Reaction of Bromobenzene; Reaction of Phenylmagnesium Bromide
(pp. 238 – 239)

The work-up of the reaction will be done next week.
Standard Scale (SS) procedure: Lehman, pp. 232 – 239.
Operations 7, 10, 11, and 24

Week #3: 10 – 14 September 2007

Experiment #29: Synthesis of Triphenylmethanol ...

This is the week for working up the reaction you carried out last week:
Separation; Purification and Analysis: Lehman, page 239.
You will trace an infrared spectrum of your purified product.
Operations 12, 13, 16, 21, 22, 23, 25, 28, and 36.

You should plan to start next week's experiment; reagents will be available.

Week # 4: 17 – 21 September 2007

Blackboard®: Reactions of Triphenylmethanol; Product Identification by Melting Point

You will synthesize three products from reactions of triphenylmethanol with a variety of reagents, and identify these products by determining their melting points.

Infrared spectra may also help in your identification.

Operations 13, 23, 25, and 30.

Minilab #27: Diels-Alder Reaction of Maleic Anhydride and Furan

Lehman, pp. 489 - 491

Reactants will be mixed and allowed to react until next lab period, when the solid product will be isolated by vacuum filtration.

Week #5: 24 – 28 September 2007

Minilab #27: Diels-Alder Reaction of Maleic Anhydride and Furan

Lehman, pp. 489 - 491

The solid product will be isolated by vacuum filtration.

Blackboard®: The Acetylation of Ferrocene; Column Chromatographic Separation

In this experiment, you will prepare a mixture of ferrocene and acetylferrocene—two organometallic “sandwich” compounds—and then separate the mixture by column chromatography on an alumina adsorbent. **During today’s lab, you will prepare the mixture of ferrocene and acetylferrocene that will be separated next lab period.**

Operation 18: Lehman, pp. 626 – 633.

Week #6: 01 – 05 October 2007

Blackboard®: The Acetylation of Ferrocene; Column Chromatographic Separation

During today’s lab, you will separate the mixture of ferrocene and acetylferrocene that you prepared last lab period..

Operation 18: Lehman, pp. 626 – 633.

Blackboard®: The Pinacol Rearrangement—Conversion of Benzopinacol to Benzopinacolone.

Week #7: 08 - 12 October 2007

Experiment #35. Friedel-Crafts Acylation of Anisole

Standard Scale (SS) procedure: pages 287 – 288

Operations 7, 10, 11, 21, 22, 23, 24, 30, 36

FALL BREAK: 13 – 16 OCTOBER 2007

Laboratory Examination #1: Thursday, 18 October 2007

7:00 – 8:30 PM in Pfahler Auditorium and Pfahler 207.

Week #8: 22 – 26 October 2007

Part IV. Qualitative Organic Analysis.

You will separate and characterize the components of a binary mixture of unknown organic compounds.

Week #9: 29 October – 02 November 2007

Part IV. Qualitative Organic Analysis.

You will continue to separate and characterize the components of your binary mixture of unknown organic compounds

Week #10: 05 – 09 November 2007

Part IV. Qualitative Organic Analysis.

You will continue to separate and characterize the components of your binary mixture of unknown organic compounds

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Week #11: 12 – 16 November 2007

Part IV. Qualitative Organic Analysis.

You will continue to separate and characterize the components of your binary mixture of unknown organic compounds

WEEK of 19 NOVEMBER 2007:

No laboratory periods scheduled this week: Thanksgiving!!!

Week #12: 26 – 30 November 2007

Blackboard®: An Aldol Condensation Experiment Using a Number of Aldehydes and Ketones.

This experiment is found in the *Journal of Chemical Education*, volume 64, 1987, page 367. The original article can be accessed on Blackboard.®

Week #13: 03 – 07 December 2007

You will check-out of your lab locker today.

You must clean all the glassware in your locker, and make sure that all equipment is present and in good condition.

Be sure that you bring your locker key with you today, so that your deposit can be cheerfully refunded.

Pay any outstanding bills for breakage.

Perform some “community service” before leaving the lab.

Laboratory Examination #2: Thursday, 06 December 2007

7:00 – 8:30 PM in Olin Auditorium
