SPRING 2019 SYLLABUS

CHEM 344 ORGANIC II LABORATORY: SYNTHESIS & CHARACTERIZATION

Senior Instructor: Dr. Andrea Gorce

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Office Hours: T 10 am-11 am, Th 1 pm-2 pm, or by appointment

Sections:	344-01	M 1:00 pm-4:55 pm CB 214	
	344-02	Th 1:00 pm-4:55 pm CB 214	
	344-03	W 1:00 pm-4:55 pm CB 214	
	344-08	T 1:00 pm-4:55 pm CB 216	
	344-09	T 1:00 pm-4:55 pm CB 214	
	344-10	F 8:00 am-11:55 am CB 214	
	344-11	TBA	
	344-12	T 8:00 am-11:55 am CB 214	
	344-75	T 7:00 pm-10:55 pm CB 214	

Text: The Organic Chem Lab Survival Manual, any edition Author: James W. Zubrick

Teaching assistants will provide you with their office hours and other necessary information in the first lab session. Please contact them for help/questions about the experiments. TAs are responsible for the grading of all assignments, quizzes, and the final exam, therefore, grading questions should be directed to your TA.

The tentative schedule of experiments is shown below. Ordering of the experiments may be changed if doing so better aligns the session with the lecture progress.

WEEK OF	EXPERIMENT
1/7/19:	
ALL SECTIONS	Check-in; Safety Instruction; Lab protocol
1/14/19:	
ALL SECTIONS	An Organic Redox Sequence: Part 1: (1S)-[endo]-borneol to (1S)-camphor (Packet procedure adapted by Dr. K Grant Taylor from Pavia, Lampman, Kriz & Engel)
	QUIZ 1

1/21/19:

Monday, 1/21/19 NO LAB-MLK DAY

Tuesday, 1/22/19-Friday, 1/25/19 An Organic Redox Sequence: Part 2: (1S)-camphor to Isoborneol (Packet procedure adapted by Dr. K Grant Taylor from Pavia, Lampman, Kriz &

Engel); **Diels-Alder Reaction** (Adapted from France, M.B., Alty, L.T., Earl, T.

Markley. J. Chem. Ed. 1999, 76, 659-660)

QUIZ 2

1/28/19:

Monday, 1/28/19 An Organic Redox Sequence: Part 2: (1S)-camphor to Isoborneol

(Packet procedure adapted by Dr. K Grant Taylor from Pavia, Lampman, Kriz & Engel); **Diels-Alder Reaction** (Adapted from France, M.B., Alty, L.T., Earl, T.

Markley. J. Chem. Ed. 1999, 76, 659-660)

QUIZ 2

Tuesday, 1/29/19-

Diels-Alder Product Isolation/Characterization

Friday, 2/1/19 **QUIZ 3**

2/4/19:

Monday, 2/4/19 **Diels-Alder Product Isolation/Characterization**

QUIZ 3

Tuesday, 2/5/19-

Friday, 2/8/19

Electrophilic Aromatic Substitution I: Microscale Bromination of Acetanilide. Which isomer predominates? Characterization by tlc, mp, and 1H NMR spectroscopy. Using splitting patterns to determine structural features. (Packet procedure adapted from Palleros and Schoffstall, Gaddis, and Druelinger).

QUIZ 4

2/11/19:

Monday, 2/11/19 **Electrophilic Aromatic Substitution I:** Microscale Bromination of Acetanilide.

Which isomer predominates? Characterization by tlc, mp, and 1H NMR spectroscopy. Using splitting patterns to determine structural features. (Packet

procedure adapted from Palleros and Schoffstall, Gaddis, and Druelinger).

QUIZ 4

Tuesday, 2/12/18-

Electrophilic Aromatic Substitution II: A Variation of the Friedel-Crafts Reaction

Friday, 2/15/18 **QUIZ 5**

2/18/19		
Monday, 2/18/19	Electrophilic Aromatic Substitution II: A Variation of the Friedel-Crafts Reaction QUIZ 5	
Tuesday, 2/19/19- Friday, 2/22/19	Grignard Synthesis of Triphenylmethanol Part 1. Synthesis and purification QUIZ 6	
2/25/19:		
Monday, 2/25/19	Grignard Synthesis of Triphenylmethanol Part 1. Synthesis and purification QUIZ 6	
Tuesday, 2/26/19- Friday, 3/1/19	Grignard Synthesis of Triphenylmethanol Part 2. Characterization: TLC, NMR, IR, and melting point. QUIZ 7	
3/4/19:		
Monday, 3/4/19	Grignard Synthesis of Triphenylmethanol Part 2. Characterization: TLC, NMR, IR and melting point. QUIZ 7	
Tuesday 3/5/19- Friday, 3/8/19	A Stereoselective Wittig reaction: Microscale synthesis and purification of 9-(2-phenylethenyl)-anthracene. Is it cis or trans? Using ¹ H NMR coupling constants to determine stereochemistry. (Packet procedure adapted from Schoffstall, Gaddis, Druelinger). QUIZ 8	
3/11/19:	NO LABS - SPRING BREAK	
3/18/19:		
Monday, 3/18/19	A Stereoselective Wittig reaction: Microscale synthesis and purification of 9-(2-phenylethenyl)-anthracene. Is it cis or trans? Using ¹ H NMR coupling constants to determine stereochemistry. (Packet procedure adapted from Schoffstall, Gaddis, Druelinger). QUIZ 8	
Tuesday 3/19/19-	The Suzuki Coupling Reaction: 21st Century Chemistry, Microscale	

synthesis, purification and NMR characterization of the Pd- catalyzed Suzuki coupling of p-bromoacetanilide with phenylboronic acid. (Procedure developed

by Devin Pantess and Christine Rich)

QUIZ 9

Friday, 3/22/19

3/25/19:

Monday, 3/25/19 The Suzuki Coupling Reaction: 21st Century Chemistry, Microscale

synthesis, purification and NMR characterization of the Pd- catalyzed Suzuki coupling of p-bromoacetanilide with phenylboronic acid. (Procedure developed

by Devin Pantess and Christine Rich)

QUIZ 9

Tuesday, 3/26/19-Friday, 3/29/19 Microscale Fisher Esterification: Making Fruity Fragrances. Synthesis,

purification, and characterization of fragrant esters.

QUIZ 10

4/1/19:

Monday, 4/1/19- Microscale Fisher Esterification: Making Fruity Fragrances. Synthesis,

purification, and characterization of fragrant esters.

QUIZ 10

Tuesday, 4/2/19-

Friday, 4/5/19

What was in that aldol?: Crossed aldol reaction from two unknowns;

determination of starting materials and product structure from spectroscopic

data and mp.

QUIZ 11

4/9/18:

Monday, 4/8/19 What was in that aldol?: Crossed aldol reaction from two unknowns;

determination of starting materials and product structure from spectroscopic

data and mp.

QUIZ 11

Tuesday, 4/9/19

Friday, 4/12/19

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Final Exam During Regular Lab Time/Check-out

4/16/18:

Monday, 4/16/19

Final Exam During Regular Lab Time/Check-out

Other information:

<u>Safety</u> is always the #1 priority. Lab safety glasses and appropriate dress are mandatory. No shorts, tanktops, or sandals are allowed. Other appropriate dress is discussed in Zubrick's textbook. You may store sweat pants and tennis shoes in your lab tote. Long hair must be tied back. Gloves are available and sometimes will be mandatory. Please pay attention when the TA or Senior Instructor points out the safety features in the lab. Pay particular attention when we discuss any safety tips for a given experiment. Precautions are noted at the end of each experiment's write up.

Proper waste disposal is an integral part of practicing responsible chemistry. Syringe needles <u>must</u> be disposed of in the sharps container. Waste solvents (from extractions, NMR samples, etc) must be placed in the appropriate liquid waste containers; solid waste should be placed in the solid waste container. There are also broken glassware containers and glass pipette disposal containers in each lab. The handout often gives guidelines for proper disposal of consumables and chemical waste. When in doubt, please ask the TA or senior instructor for information. If you break a mercury thermometer, *immediately* notify your teaching assistant. There are special precautions we must take to clean up mercury spills.

ALL FOOD AND DRINK ARE PROHIBITED IN THE LAB this includes water bottles.

<u>Course Objectives:</u> The primary objective of this laboratory course is to give hands-on experience with the basic techniques and synthesis employed by organic chemists. Important characterization/product analysis techniques such as NMR and IR spectroscopy will be used more extensively in this lab course than in previous courses. The course goal is to make the connection between concepts and theories discussed in lecture and their application and execution in the laboratory. This means that there will be an emphasis not only the "how to" but also the "why and when to" inherent in organic experimentation.

Concerning i2A, this lab course will provide many opportunities to practice and develop **critical thinking skills**. In part, this will be (explicitly) modeled during class discussions and/or group problemsolving sessions. Key elements of critical thinking include: identifying the question or problem, analyzing evidence and developing arguments, integrating knowledge and demonstrating an awareness of multiple points of view, and drawing conclusions based upon reasons, arguments, and evidence. Your progress in this area will be assessed informally during prelab discussion and formally in written lab reports.

<u>Lab Preparation</u>: Please come prepared. All of the basic organic lab techniques and many other helpful hints for success in the lab are discussed in Zubrick's text. The syllabus lists the basic techniques you'll employ to carry out the experiment. Reading the appropriate section describing these techniques in the lab manual is required so that you'll be prepared when you come to lab. **Pre-lab questions are turned in at the start of the experiment for a portion of that session's grade.**

Each week before entering the lab, the lab report template should be filled out with the title, objective, introduction, reaction/mechanism, and procedure sections already typed. During the lab, observations should be hand-written in the lab report and the **unchanged** (ie. Do not rewrite them) **handwritten observations must be attached to the submitted final report.** Please note that every lab rubric says "Observations may be handwritten or typed". **Both handwritten and typed observations are required.** To submit your lab for SafeAssign, observations must be typed. When the report is given to your TA, the original handwritten observations must be included as well.

You must have the procedure written out before you enter lab. Your TA will check for the procedure each week. If you come to lab without a written procedure, you will be asked to leave the lab until you complete the write-up of the procedure and you will lose the points for that week's procedure.

<u>Grading</u>: Grades are on a 10 point scale (100-90 = A, 89-80 = B, etc). Grades will be computed based on a possible 1650 points. If you earn the following number of points, you are <u>guaranteed</u> the letter grade shown:

1485-1650	90% - 100%	Α
1320-1484	80%-89%	В
1155-1319	70%-79%	С
990-1154	60%-69%	D
below 1200 pts	below 60%	F

Lab Reports/Questions: One week after the completion of each experiment you will be required to turn in a type-written lab report which includes the handwritten observations. The template for this lab report can be found in the lab packet. As noted above, typed and handwritten observations a required, Your TA will then grade the report and return it the following week. You will then place the graded lab reports in a 3-ring binder for the remainder of the semester. This binder will become your "lab notebook." You may also keep a traditional lab notebook, but these will not be collected or graded.

Be sure each report is complete. Post-lab questions are given at the end of the experimental procedure and those questions should be answered directly at the end of the lab report; pre-lab questions are turned in at the start of the experiment for a portion of that session's grade. The TA or Senior Instructor will discuss the format of the lab report with you in more detail during class. Lab reports are due during your scheduled lab time one week following completion of the experiment unless your TA tells you otherwise. Late reports are docked 10% per day.

Notice on the syllabus that there are 9 single session experiments, each worth 100 points, and 1 double session experiment, worth 200 points, for a total of 1100 points. You will be allowed to drop one lab grade worth 100 points (or one-half of a double session grade). Therefore, lab reports/lab questions constitute 1000 points (or ~61%) of your overall course grade.

<u>Quizzes</u>: Your TA will give eleven 30-point quizzes during the course of the semester. Quizzes will be given *during each lab session* starting the day of your first experiment. Your lowest quiz score will be dropped. Therefore, quizzes will constitute 300 points (or ~18%) of your overall course grade. Please note that the online Safety quiz must be done before the first lab and is NOT counted towards your quiz grade.

<u>Final Lab Exam</u>: A comprehensive final exam for the course will be given during the week of April 8th and on April 15th during the regularly scheduled lab times. The Final will constitute 350 points (or ~21%) of your overall course grade.

The quality and thoroughness of the lab reports will contribute strongly to the overall grade; **Please do not attempt to falsify data or copy lab reports from other sources.** Cheating will not be tolerated. We check all labs through SafeAssign. We are aware that the procedure part of the lab report is going to show duplications. However, if your discussion shows that over 60% of it is copied from 1 source, that is considered as plagiarism. The following actions will be taken in case of plagiarism:

- 1. First offense- a warning will be given
- 2. Second offense- a zero will be given for the lab
- 3. Third offense- a grade of F will be given for the course and student will be reported to the Dean.

Please see the University Policy on Plagiarism for more details. https://louisville.edu/artsandsciences/academics/undergraduate-education/files/plagarism.pdf/view

We understand that an experiment may go wrong and that low yields may be obtained, or the product may not be pure. In that case, please offer reasonable explanations for the observed results.

<u>There are no make-up labs.</u> If you must miss a lab session <u>for any reason</u>, <u>consider that to be the lab grade that you will drop</u>. It is highly recommended that you do not skip any lab unless it is absolutely unavoidable.

What if I miss a lab? Students who miss <u>one</u> lab/quiz will be required to use that miss as their dropped lab/quiz grade for the semester (see exclusion below for UEAP absences).

If you must miss a <u>second</u> lab/quiz: What sorts of absences are excused?

First, be aware that it is **always** up to the instructor to decide whether an absence is excused or not. There isn't a specific reason(s) that automatically excuses absence from class; all cases must be brought to the instructor. The burden of proof is on the student and hard copies of documentation will be required (email is usually not accepted). In any case, discuss any potential absences with the instructor as far in advance as possible.

Generally excused:

- Absences due to medical emergency: provide a note from your doctor or the student health services stating that you were unable to attend lab. The note must be dated; we reserve the right to call your health care provider to confirm that the excuse is genuine. We don't want to know the nature of your medical emergency. We will not ask for, nor disclose, any personal medical information.
- Absences due to the death of an immediate family member: provide a copy of the obituary or other document stating the time and place of memorial services.
- Absences relating to University-sponsored travel (e.g. for athletes or members of other University organizations) require official documentation from the appropriate departments.

May be excused, at the discretion of the instructor:

- Court dates: provide a document from the court stating that you were required to be present at the time and date in question. We do not want to know the purpose of your court date.
- Emergencies, such as being involved in a car accident on the way to class: at a minimum, appropriate documentation showing that you were unable to be in class at the time in question (e.g., police or insurance report, tow truck receipt.) Injuries are covered above, under medical emergencies.
- Absences stemming from family emergencies or work-related conflicts: appropriate documentation will be required and excuses are not granted automatically.

Never or rarely excused: any absence that could be seen as avoidable, including but not limited to

- Absences due to inclement weather, if the University is operating.
- Oversleeping alarm, power outage, dead battery/car not starting, missed/late bus, ride not showing up, etc.
- Being stranded out of town, ride for break leaving early or returning late.
- Accident or illness at a time other than during the class or exam.
- Lack of child care (with rare exceptions that may constitute a family emergency-see above).
- Studying for another class, exam in another class, paper required in another class, etc.
- Social engagements, family reunions, family in town, picking people up at the airport, personal travel.
- Club sports, club events, fraternity or sorority events, etc. (exception for "University-sponsored travel").

How are excused absences generally handled?

- For students with an excused absence due to an accident, illness or emergency, or any other excused absence NOT due to a University-sponsored activity: if a student has exhausted his/her dropped grade for the course and must miss a <u>second</u> lab due to an excused absence, the missed lab/quiz is dropped from the semester grade calculation for that student: the value of that lab/quiz is not included in the total points for the semester when that student's semester grade is calculated. It is a good idea, if you have an excused absence on a lab/quiz, to remind your TA during the week before finals, just to make sure your grade is calculated and recorded correctly. Excused absences on the lab final exam are rare and will be handled individually.
- Students who miss a lab because of a University-sponsored activity, and who invoke the University Excused Absence Policy (UEAP): students who invoke UEAP for a missed lab/quiz will not be required to use the missed lab/quiz as their dropped grade for the course. However, these students can choose to drop any or all UEAP-covered missed labs/quizzes from their grade calculation as above, or they can

attend lab with another section. To attend a different lab section, the student must submit their intention, in writing, to Dr. Gorce, *no less than 7 days in advance of the scheduled lab*. This submission is necessary for scheduling purposes. The student must provide their weekly course schedule, with times and course numbers indicated. The lab may be scheduled by the professor to take place before or after the regularly scheduled lab. Unless all student obligations under this policy are met, a student will not be able to attend another section of lab.

How will an unexcused absence affect my grade? (Can I still pass?)

A <u>second</u> missed lab due to an unexcused absence will be recorded as a score of 0 and cannot be dropped from the student's course grade. BUT: Each weekly experiment and associated quiz counts as ~8% of the total grade for the semester. In principle, a student who missed two labs/quizzes, but earned high grades on the other work in the course could still earn an A or a B without any accommodations at all.

NOTE: ANY STUDENT WHO MISSES 3 OR MORE LABS (EXCUSED OR UNEXCUSED) MUST CONTACT THE SENIOR INSTRUCTOR AND MAY HAVE TO TAKE AN INCOMPLETE FOR THE SEMESTER

What appeals processes are in place?

If you believe you are not being treated fairly under the terms of the syllabus and University policy, you should meet with Dr. Gorce. If after doing so, you feel that you are still being treated unfairly, please make an appointment with the Chair of the Department of Chemistry. (I will be happy to help you set this up). You may request that I be present or not. The Department Chair can inform you of the other appeal options available to you under University policy.

TECHNOLOGY EXPECTATIONS

Because Blackboard is used extensively for communication in this course you must have access to a computer and a reliable Internet service. If you do not have access at home, you can use the University computer lab in the library or one of the IT student computer labs on campus.

You must be able to access, use, and navigate Blackboard, use email, and know how to download documents.

For your privacy and security, only your official U of L email account will be used for communication. No information will be sent to personal email accounts. Please check your U of L email account daily.

STUDENT SUPPORT

Tech Support

Blackboard Student and After-Hours Support: HelpDesk Call Service: (502-852-7997)

Email Service: helpdesk@louisville.edu

The HelpDesk provides support to the entire University of Louisville community: faculty, staff and students. If you need assistance with accessing your university accounts, unlocking your password, accessing wireless or more, please let the support staff know.

Academic Support

Supplemental Instruction (SI) sessions are done by the **REACH Program.** Tutoring by appointment may also be scheduled through REACH. Go to the <u>REACH website</u>, (www.reach.louisville.edu), click "Appointment Tutoring" to fill out a tutoring-request form or go in person to Strickler Hall, Room 107 and complete a request form.

DISABILITY SERVICES

Students with disabilities who require accommodations (academic adjustments and/or auxiliary aids or services) for this course must contact the Disability Resource Center (DRC). Please do not request accommodations directly from the professor. The DRC can be reached at (502)852-6938.

SEXUAL HARASSMENT

Sexual misconduct (sexual harassment, sexual assault, and sexual/dating/domestic violence) and sex discrimination are violations of University policies. Anyone experiencing sexual misconduct and/or sex discrimination has the right to obtain confidential support from the PEACC Program 852-2663, Counseling Center 852-6585 and Campus Health Services 852-6479. Reporting your incident to any other University employee (including, but not limited to, professors and instructors) is an official, non-confidential report to the University.

To file an official report, please contact the Dean of Student's Office 852-5787 and/or the University of Louisville Police Department 852-6111. For more information regarding your rights as a victim of sexual misconduct, visit the Office for Civil Rights (http://www2.ed.gov/about/offices/list/ocr/docs/know-rights-201404-title-ix.pdf) and for more information about resources and reporting at UofL, visit the Sexual Misconduct Resource Guide (http://louisville.edu/hr/employeerelations/sexual-misconduct-brochure).

OTHER UNIVERSITY POLICIES

Please refer to the university policies on religious holy days/observances and diversity as detailed in the Student Handbook (https://louisville.edu/dos/students/studentpoliciesandprocedures/student-handbook).

MISCELLANEOUS INFO: This course has an underlying focus on microscale and Green Chemistry. Each of you will have your own personal microscale kits. Not only is our microscale effort an environmentally sound approach to bench chemistry (reduced reaction scale = less chemical usage = less chemical waste generated = less student exposure to toxic and explosive chemicals = safer lab conditions), microscale experiments enjoy another benefit...shorter reaction times!

Other Problems Both the TAs and Senior Instructor have office hours.

*The syllabus is subject to change at the discretion of Dr. Gorce. Please check Blackboard daily for updates and announcements.