
Chemistry 391: Advanced Laboratory 1

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<https://www.smkenedy.org/>**Office Hours:**

Please see D2L for office hours schedule. Meetings outside of weekly office hours are available by email appointment only.

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Lab Section 01A (CRN 4347): Mondays 12:30 – 3:20 pm; 327 Caputo Hall
Chemistry Department Seminar: Mondays 4:00 – 5:00 pm; 149 Roddy Hall

COURSE DESCRIPTION

Chemistry 391 (Advanced Laboratory 1) builds on some of the prior knowledge obtained during your Chemistry 231 and 232 lecture and in-person laboratory courses. Selected skills and techniques relevant to the modern organic and organometallic synthesis research laboratory will be introduced and developed during the semester; these include: searching the chemical literature, reaction planning, exploring reaction methods scope and limitations, reaction set-up, reaction monitoring by thin-layer-chromatography, chemical isolation, chemical purification, NMR spectroscopic analysis of reaction products and mixtures, employing mechanistic proposals utilizing electron-pushing formalism (EPF, a.k.a. curved-arrow notation) to describe elementary reaction steps, teamwork to compile meaningful data sets, and formal communication of experimental results via semi-formal report writing (utilizing an undergraduate research journal style).

COURSE LEARNING OBJECTIVES

Upon completion of Chemistry 391 you should be able to...

1. Perform SciFinder chemical literature searches of a target organic molecule.
2. Plan reactions based on chemical literature experimental procedures.
3. Adapt experimental procedures to different substrates, reaction size (scale), and available supplies.
4. Set up chemical reactions safely and efficiently.
5. Monitor reactions using thin-layer-chromatography (TLC) analytical techniques.
6. Isolate, purify, and characterize reaction products using chemical, physical, and spectroscopic techniques.
7. Obtain, formally analyze, and utilize NMR data to determine the composition of reaction mixtures.
8. Work as a team to compile, share, analyze, and interpret raw experimental data.
9. Begin to explore a reaction method's scope and limitations, as part of a team.
10. Communicate experimental results via detailed laboratory reports using the *Journal of Undergraduate Chemical Research (JUCR)*; <https://bit.ly/3iWl8Wj> & <https://bit.ly/3BORbA9> style format with additional guidelines & criteria discussed during laboratory meetings.
11. Propose plausible reaction mechanisms using curved-arrow notation electron-pushing formalism (EPF) to demonstrate and reinforce your understanding of plausible elementary reaction steps from CHEM 231 & 232.

REQUIRED & RECOMMENDED MATERIALS

1. Required Materials & Supplies:
 - Bound Composition Notebook: any notebook will work fine.
 - One (1) set of Safety Goggles or Glasses.
2. Highly Recommended: a modern (published after 2010) sophomore-level organic chemistry textbook. This will help you to propose reasonable reaction mechanisms using curved-arrow notation.

COURSE ORGANIZATION

This is a blended laboratory course in which new topics and assignments are first presented online via the D2L course website, or in laboratory, or via handouts. Required readings, preparation for laboratory, literature searches, data analysis, and laboratory report writing will all require some time spent outside of laboratory to successfully complete this advanced laboratory course. The scheduled class laboratory periods will be used primarily for wet chemistry and chemical analysis. This course is also organized around the following ideas related to synthesis...

1. Green chemistry techniques and reaction modifications
2. Small scale test reactions as a green research and reaction development technique
3. Reactions that form C–C, C–N, and C–O bonds using modern synthetic methods including catalysis
4. Reactions used in the pharmaceutical and materials industries
5. Understanding mechanisms of organic and organometallic reactions
6. Exploring the scope, limitations, and applications of reaction methods by using chemical literature precedent
7. Creating small libraries of organic molecules utilizing known reactions

SCHEDULE OF EXPERIMENTS

Week	Date	Experiments // Activities // Assignments // Due Dates
1	August 22 nd	Check-in, Syllabus, Safety, Introduce Acetal Formation Reaction , & SciFinder
2	August 29 th	Run AFR // JUCR report guidelines introduced
3	September 5 th	Holiday = Labor Day = No Lab
4	September 12 th	Isolate, TLC, & run NMR of AFR product
5	September 19 th	IR, MP, & NMR analysis of AFR product
6	September 26 th	Introduce Biginelli Reaction & Discuss AFR report (200 points; due 5-OCT)
7	October 3 rd	Run BR , isolate crude product, TLC, & run NMR experiments
8	October 10 th	Holiday = Fall Break = No Lab
9	October 17 th	IR, MP, & NMR analysis of BR product
10	October 24 th	Introduce Suzuki-Miyaura Cross-Couplings & Discuss BR report (200 points; due 2-NOV)
11	October 31 st	Run two SMCCs (control and experimental), TLC monitor, isolate crude products
12	November 7 th	Pipet column to purify SMCC crude products, TLC, NMR w/ DMSO ₂ internal standard
13	November 14 th	IR, MP, & NMR analysis of SMCC products
14	November 21 st	Discuss SMCC lab report
15	November 28 th	Extra week to finish work & Clean-up 327 Caputo Hall
16	December 5 th	Notebooks (200 points) & SMCC report (400 points) both due to D2L on 12/5 @ 3:20 pm

COURSE EVALUATION OVERVIEW

Notebook (200 points)	20 %
3 Typed Laboratory Reports (800 points)	<u>80 %</u>
	$\Sigma = 100 %$

GRADE DISTRIBUTION (in percentage points)

A	92.0 – 100	B	82.0 – 87.9	C	70.0 – 77.9	D	55.0 – 59.0
A-	90.0 – 91.9	B-	80.0 – 81.9	C-	65.0 – 69.9	D-	50.0 – 54.9
B+	88.0 – 89.9	C+	78.0 – 79.9	D+	60.0 – 64.9	F	<50.0

LABORATORY SAFETY

You are expected to have read all assigned reading thoroughly, to have prepared a detailed notebook entry, and to have considered safety concerns prior to each laboratory experiment. Please be prepared to complete focused and efficient work during laboratory time. Being prepared for laboratory is the best way to stay safe. Most of the compounds that we (as chemists) work with have some level of toxicity; and many of the chemicals that we will work with in CHEM 391 should always be handled in the fume hood. General safety guidelines will be presented throughout this laboratory course and should be always followed. Specific safety precautions for each experiment will be covered before each lab. Additional laboratory guidelines including notebook guidelines and report instructions are covered in the weekly handouts.

Academic Honesty & Dishonesty (<https://bit.ly/3iXLVBF>): plagiarism is the deliberate or even accidental representation of another's work as your own without proper reference. Although you may work together on some material and experiments, this does not mean that lab reports and assignments should be identical. Each participant uses the collective data and discussion to prepare their own individual report. You should be familiar with the University policy on academic honesty and dishonesty as outlined in the Student Handbook and Academic Honesty and Dishonesty brochure; the content applies to this course.

Millersville University Approved Policy (<http://bit.ly/2ChS479>):

1. Students are expected to attend all classes. It is the student's responsibility to complete all course requirements even if a class is missed. If a student missed class for an officially excused reason, then he/she is entitled to make up the missed work but only at the convenience of the faculty member. Responsibility for materials presented in, assignments made for, and tests/quizzes given in regularly scheduled classes lies solely with the student.
2. The University policy is that faculty will excuse absences for the following reasons: personal illness, death or critical illness in the family, participation in a university-sponsored activity, jury duty, military duties, religious holidays
3. Faculty judge the validity of student absences from class within the University's approved guidelines and may require documentation for excused absences. Faculty will evaluate any reason, other than those listed above, for a student missing class and determine whether the absence is justified. In these circumstances, a student may make up missed work at the discretion of the instructor.
4. In the case of foreseeable absences, students are encouraged to notify the faculty member in advance. A student who will miss class due to participation in an official University activity must notify the instructor well in advance of the activity to assure that the absence is excused.

Title Nine (IX) Statement:

Millersville University and its faculty are committed to assuring a safe and productive educational environment for all students. In order to meet this commitment and to comply with Title IX of the Education Amendments of 1972 and guidance from the Office for Civil Rights, the University requires faculty members to report incidents of sexual violence shared by students to the University's Title IX Coordinator. The only exceptions to the faculty member's reporting obligation are when incidents of sexual violence are communicated by a student during a classroom discussion, in a writing assignment for a class, or as part of a University-approved research project. For more information on Title IX: <https://bit.ly/2V19BgD>