Dr. Steven Merwin Kennedy  
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Lab Section 01 (CRN 4347): Mondays 12:30 – 3:20 pm; 327 Caputo Hall  
Chemistry Department Seminar: Mondays 4:00 – 5:00 pm; 149 Roddy Hall  
Lab Section 02 (CRN 11049): Thursdays 1:10 – 4:00 pm; 327 Caputo Hall

COURSE DESCRIPTION
Chemistry 391 (Advanced Laboratory 1) builds on the prior knowledge obtained during your Chemistry 231 and 232 lecture and laboratory courses. Passing both Chemistry 232 and Chemistry 265, with at least C letter grades, is pre-requisite for this course. Skills and techniques relevant to the modern organic and organometallic synthesis research laboratory will be 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 formal report writing (utilizing a standard undergraduate research journal style).

COURSE LEARNING OBJECTIVES
Upon completion of Chemistry 391 you should be able to...  
1. Use SciFinder to search the chemical literature  
2. Preform literature searches of a target organic molecule  
3. Plan reactions based on literature reported experimental procedures  
4. Explore reaction methods scope and limitations, as part of a team  
5. Adapt experimental procedures to different substrates, reaction size (scale), and available supplies  
6. Set up chemical reactions safely and efficiently  
7. Efficiently monitor reactions using thin-layer-chromatography (TLC) analytical techniques  
8. Isolate, purify, and characterize reaction products using chemical, physical, and spectroscopic techniques  
9. Obtain, analyze, and utilize NMR data to correctly determine the composition of reaction mixtures  
10. Write quality abstracts for each laboratory experiment  
11. Work as a team to share, analyze, and interpret raw experimental data  
12. Communicate experimental results using the Journal of Undergraduate Chemical Research (JUCR) style format, information in the required textbook for this course, and information discussed during laboratory meetings  
14. Formally analyze your NMR spectral data to include with your laboratory report supporting information (SI)  
15. Propose reasonable reaction mechanisms, in your SI, using curved-arrow notation (EPF) to demonstrate your understanding of plausible elementary reaction steps from Chemistry 231 and Chemistry 232

POSSIBILITIES FOR PUBLICATION
If your experimental procedure and data are of high enough quality, and have never been previously published in CSSP, then they may be considered for publication in the Royal Society of Chemistry’s ChemSpider SyntheticPages (CSSP) database (http://bit.ly/2t0RMPr), and...

If your laboratory report is of high enough quality, it may be considered for inclusion in a manuscript for submission to the JUCR (http://bit.ly/2rzNUQP)
**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 (laboratory procedures and problem sets). So, check your MU email regularly for updates. Required readings, preparation for laboratory, literature searches, data analysis, and laboratory report writing will all require time spent outside of laboratory to successfully complete this advanced laboratory course with at least a C letter grade. The scheduled class laboratory periods will be used primarily for wet chemistry and chemical analysis. This course is also organized around the following “Current Ideas in Organic 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 and possible side 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

**REQUIRED & Highly Recommended Laboratory Books, Materials, & Resources**


2. **Required Materials & Supplies**:
   - Bound Composition Notebook: 80 sheets, dimensions 9.75 x 7.5 inches, 5 x 5 graph ruled.
   - One (1) set of Safety Goggles or Glasses.

3. **Required daily online access to our Chemistry 391 D2L course website and your Millersville email account.**

4. **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.

5. **Highly Recommended (If you do not purchase the 7th Ed., then please consider the 6th Ed., or online access)**: Spectrometric Identification of Organic Compounds, 7th Edition, Robert M. Silverstein, Francis X. Webster, David Kiemle, ©2005. ISBN 9780471393627. This is a valuable resource for interpreting IR spectra, NMR spectra, and more. Most graduate programs in organic chemistry require a version of this text.

6. **Highly Recommended**: access to ChemDraw or ChemDoodle software.

**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 and every laboratory experiment. 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. All things are toxic, and many of the chemicals that we will work with in CHEM 391 are very toxic. While much of synthetic chemistry research is still empirical, in a world with instant electronic access to the chemical literature (e.g. Millersville Library and SciFinder Scholar) and chemical safety information (e.g. Safety Data Sheets – SDS) **there is absolutely no excuse for carrying out unplanned or poorly literature researched chemical reactions**. General safety guidelines will be presented throughout this laboratory course and should be followed at all times. Specific safety precautions for each experiment will be covered before each lab. Failure to follow safety guidelines is grounds for dismissal from a laboratory session and a grade of zero on the experiment. Additional laboratory guidelines including notebook guidelines and report instructions are covered in the weekly handouts.
LABORATORY ATTENDANCE AND LATE WORK

Attendance at each laboratory session is mandatory. Missing one laboratory session will result in a zero grade for the experiment. Missing two laboratory sessions equals failure in this course. Students are responsible for all notes, discussions, assignments and handouts presented at each laboratory meeting (whether you are absent or present). Absences that you know about in advance may be excused. In the event of an anticipated excused absence, arrangements should be made to make up work prior to the absence. Excused absences include attendance at university events (athletic and educational), military duty, bereavement, critical illness in the family, or personal illness in which a physician indicates that you were too ill to attend class. A request for an excused absence should include a written justification of the reason for the absence and be signed by the appropriate individual (coach, faculty member, physician, etc.). Only valid excuses will be accepted to make up any missed assignments. Work not made up will receive a grade of zero. Departure times for travel before university breaks are not a valid excuse to miss class, so please make your travel arrangements accordingly. Please see the University Attendance Policy for more information. https://www.millersville.edu/registrar/faculty/attendance-policy.php

SCHEDULE OF EXPERIMENTS*

<table>
<thead>
<tr>
<th>Week</th>
<th>Date</th>
<th>Experiments // Activities // Assignments // Due Dates</th>
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</thead>
<tbody>
<tr>
<td>2</td>
<td>3-or-6-SEPT</td>
<td>Labor Day = No Lab (Due by 7-SEPT @ 3:00 pm: BH Notebook entry picture, BH abstract outline &amp; draft, &amp; screen grabs of SciFinder, Request-it, &amp; ChemDraw) Open lab from 1:10 pm to 4:00 pm on 6-SEPT</td>
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<tr>
<td>3</td>
<td>10-or-13-SEPT</td>
<td>Run BH // JUCR manuscript style lab report guidelines introduced // NMR, IR, MP procedures explained</td>
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<tr>
<td>4</td>
<td>17-or-20-SEPT</td>
<td>NMR (1H, 13C12 scans, DEPT, gCOSY, gHMQC), TLC, IR, MP of BH reaction product(s)</td>
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<tr>
<td>5</td>
<td>24-or-27-SEPT</td>
<td>Introduce Suzuki-Miyaura Cross-Coupling (BH Report due by 5-OCT @ 3:00 pm)**</td>
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<tr>
<td>6</td>
<td>1-or 4-OCT</td>
<td>Run SMCC and monitor reaction via Thin-Layer-Chromatography (TLC), isolate crude product (Notebooks due by 5-OCT @ 3:00 pm)</td>
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<tr>
<td>7</td>
<td>8-or-11-OCT</td>
<td>Holiday = Fall Break = No Lab (pick up BH report and notebooks by 12-OCT @ 3:00 pm) Open lab from 1:10 pm to 4:00 pm on 11-OCT</td>
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<td>8</td>
<td>15-or-18-OCT</td>
<td>Pipet column purify SMCC crude product, TLC, NMR (1H &amp; 13C) w/ DMSO2 internal standard Introduce Imine-Formation-Reaction (IFR)</td>
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<td>9</td>
<td>22-or-25-OCT</td>
<td>Run IFR, then recrystallize imine product(s) (SMCC Report due by 11/2 @ 3:00 pm)</td>
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<tr>
<td>10</td>
<td>29-OCT-or-1-NOV</td>
<td>TLC, Night Queue NMR (1H, 13C1024 scans, DEPT, gCOSY, gHMQC), IR, &amp; MP of IFR product(s)</td>
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<tr>
<td>11</td>
<td>5-or-8-NOV</td>
<td>Introduce Inverse-Electron-Demand Diels-Alder reaction</td>
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<tr>
<td>12</td>
<td>12-or-15-NOV</td>
<td>Run IEDDA &amp; TLC monitor (IFR Report due by 11/20 @ 3:00 pm)</td>
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<tr>
<td>13</td>
<td>19-or-22-NOV</td>
<td>Holiday = Thanksgiving</td>
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<tr>
<td>14</td>
<td>27-or-29-NOV</td>
<td>TLC, Night Queue NMR (1H, 13C1024 scans, DEPT, gCOSY, gHMQC), IR, MP of IEDDA product(s)</td>
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<tr>
<td>15</td>
<td>4-or-6-DEC</td>
<td>Finish IEDDA data collection work, Post-Survey, &amp; Clean-up 327 Caputo Hall</td>
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<tr>
<td>16</td>
<td>10-DEC</td>
<td>Notebooks &amp; IEDDA Report both due by 12/14 @ 3:00 pm</td>
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**Data due on D2L one week prior to report due date.  *Note: experiments subject to change.
COURSE EVALUATION OVERVIEW
Chemistry Seminar Attendance (100 points) 10 %
Notebook (300 points) 30 %
4 Typed Laboratory Reports (600 points) 60 %
\[ \sum = 100 \% \]

NOTEBOOK EVALUATIONS (200 points)
Detailed guidelines are in the APOC textbook. Laboratory notebooks will be collected twice, at the end of week 6 (100 points, 20 of these 100 points are for picking up your notebook and the BH report before 10/12 @ 3:00 pm), then at the end of week 16 (200 points). Requirements and advice for maintaining adequate notebooks will be provided during the first week of the course.

LABORATORY REPORT EVALUATIONS (600 points)
Four laboratory reports will be due during the course of the semester. All laboratory reports must be typed in a word processor correctly following the JUCR style guide & any additional guidelines discussed during Chemistry 391. For full points, all chemical structures must be drawn in the ACS 1996 style (JOC style) using ChemDraw (available on the PCs in 331 Caputo Hall or at http://bit.ly/2ueqIfT for $100, the student discount price) or using ChemDoodle (less user friendly than ChemDraw, available for as little as $19 per six months http://bit.ly/2t0QpAt). Or, see ChemDraw Direct for a free web version (http://bit.ly/2u57lFD).

Point breakdown: the Baylis-Hillman Report is worth 100 points (due week 6). The Suzuki-Miyaura Cross-Coupling Report is worth 135 points (due week 10). The Imine Formation Report is worth 170 points (due week 13). The Inverse-Electron-Demand Diels-Alder Report is worth 195 points (due week 16). More instructions for completing laboratory reports will be provided during week 3. My expectation is that both your adherence to the JUCR style guide and your writing will improve with each report. More details concerning laboratory report structure and grading will be discussed in lab.

GRADE DISTRIBUTION (in percentage points)

<table>
<thead>
<tr>
<th>Grade</th>
<th>Percentage</th>
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<tbody>
<tr>
<td>A</td>
<td>92.0 – 100</td>
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<tr>
<td>A-</td>
<td>90.0 – 91.9</td>
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<tr>
<td>A</td>
<td>92.0 – 89.9</td>
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<tr>
<td>B-</td>
<td>80.0 – 81.9</td>
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<tr>
<td>B</td>
<td>82.0 – 79.9</td>
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<tr>
<td>C</td>
<td>70.0 – 77.9</td>
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<tr>
<td>C-</td>
<td>65.0 – 69.9</td>
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<tr>
<td>D</td>
<td>55.0 – 59.0</td>
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<tr>
<td>D-</td>
<td>50.0 – 54.9</td>
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<tr>
<td>D</td>
<td>&lt;50.0</td>
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Tips for Success: this is an advanced laboratory course focused on the skills necessary to plan, successfully implement, analyze, and discuss (written, symbolic, and verbal) synthetic reactions. This course also focuses on organic and organometallic reactions. Additional advice on how to be successful during this advanced laboratory course... Be curious. Ask questions! Make safety a priority. Be well prepared for each laboratory. Get organized for this course and don’t miss due dates. Do all relevant and assigned readings before the laboratory. Stop by during office hours or schedule a meeting by email. Make NMR data analysis a game and learn to enjoy it.

Out of Class Assistance: please feel free to stop by my office during office hours for assistance outside of class. Ask me questions during laboratory. Set up an appointment via email if office hours don’t fit your schedule. Email me your question. Start a discussion on D2L. Form a laboratory report review and editing group.

Learning Accommodations: please see the Office of Learning Services in Lyle Hall (http://bit.ly/2txPSmb) as soon as possible if you have special learning needs for this class. If you have a condition that may affect your ability to perform laboratory exercises, to exit lab safely from the premises in an emergency, or which may cause an emergency during class, or lab, please discuss this in confidence with your instructor and someone at the Office of Student Support Services. Appropriate accommodations may then be provided.
Academic Honesty & Dishonesty: 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 his or her 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. If you are caught, you will be removed from the course, assigned an F letter grade for the course, and a formal report—a formal report to become part of your permanent academic record—will be filed with the associate Provost for Academic Programs and Services. http://www.millersville.edu/english/for-faculty/academic-integrity/violations.php http://www.millersville.edu/provost/files/academic-honesty-violation-form.pdf


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 misses a 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: http://www.millersville.edu/titleix/title-ix-policies-and-procedures.php