Written Research Reports
The Second-Year Report

Due: Wednesday, noon, Nov. 27, 2019
(Just before Thanksgiving break)
Six Basic Requirements for a Ph.D. in Chemistry

http://www.chem.uci.edu/

1. Completion of a minimum of seven approved courses with maintenance of an average grade of B or better.
2. **Completion of a second-year Written Examination.**
3. Completion of an Oral Examination for **Advancement to Candidacy.**
4. Completion of the teaching requirement (four quarters).
5. Completion of six quarters in residence at UCI.
GOAL OF THE 2\textsuperscript{ND}-YEAR WRITTEN EXAMINATION

https://www.chem.uci.edu/graduate/current-students

PREPARE PUBLICATION-READY MANUSCRIPTS
Organization of the Research Report
Second-Year Report and Orals

Generally, you will compile:

1. Descriptive Title + Abstract
2. Introduction
3. a. Results and b. Discussion
4. Conclusions and Future Work
5. Scholarly References
6. Experimental Section
7. Supporting Information

≤10 pages

combined or separate
What are we looking for in a Second-Year Report?

1. Satisfactory research progress
   • Fully characterized products
   • Yields for many, many reactions
   • Purified proteins or peptides
   • Developed and validated new assays

2. A written document that communicates effectively

Scholarly Background
   • Why is the project important to society?
   • What was done in the past?
   • What is the gap in technology/knowledge that needs to be filled?

Research
   • What obstacles did you overcome?
   • If you worked with someone else, which accomplishments are yours?
   • Future goals
I can not overemphasize the importance of making significant research progress.

A well written report identifies challenges you faced - unstable compounds, poor protein expression, low transformation efficiencies, difficulties in purification, assay (ir-reproducibility).

Faculty understand the difference between lack of effort and challenging projects.

### About Research Progress on the Second-Year Report

**Conditions and Yields**

<table>
<thead>
<tr>
<th>Conditions</th>
<th>Yield 7</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 M HCl, 100 °C</td>
<td>0%</td>
</tr>
<tr>
<td>6 M HCl, 100 °C</td>
<td>0%</td>
</tr>
<tr>
<td>0.1 M H$_2$SO$_4$, 100 °C</td>
<td>0%</td>
</tr>
<tr>
<td>0.05 eq. p-TsOH, 100 °C</td>
<td>0%</td>
</tr>
<tr>
<td>1 M KOH/EtOH, 25 °C</td>
<td>0%</td>
</tr>
<tr>
<td><em>C. ligrea</em> lipase, phosphate buffer, 25 °C</td>
<td>0% (S.M.)</td>
</tr>
<tr>
<td>0.1 M acetic acid, H$_2$O/dioxane, 25 °C</td>
<td>0% (S.M.)</td>
</tr>
<tr>
<td>0.1 M acetic acid, H$_2$O/dioxane, 100 °C</td>
<td>0%</td>
</tr>
</tbody>
</table>

**Explain WHY it didn't work:**

- Identify the side-products.
- Look up reactivity of similar functional groups.
Experimental and Characterization Data

1. Follow the standards of the community
   See: "Author Guidelines."
   • The Journal of Organic Chemistry
   • ACS Chemical Biology

2. Follow the standards of your research group
   • See perfected research reports from senior students.
   • See published papers from your group
   • See dissertations from your group
Experimental and Characterization Data

I. WHAT YOU GOT (e.g., ... 36.2 mg of diene 43 (3:1 E/Z) as a yellowish solid.)
   A. Yield. Significant figures should match measured reagents.
      1. Isolated mass
      2. Spectroscopic/chromatographic peak ratios versus an internal standard
   B. Stereoisomeric ratios
      (e.g., (±)-2-hydroxybut-3-ene, 4:1 E/Z, 95:5 syn/anti, 74% ee)
   C. Physical state (for solids, include a m.p.)
   D. $R_f$ and eluant system

II. EVIDENCE FOR IDENTITY AND PURITY (J. Org. Chem. Dec, 2018)
   http://pubsapp.acs.org/paragonplus/submission/joceah/joceah_authguide.pdf?
   A. Evidence for IDENTITY (chemical structure and stereochemistry)
      1. $^1$H NMR and $^{13}$C NMR
         http://pubsapp.acs.org/paragonplus/submission/acs_nmr_guidelines.pdf?
      2. HRMS or Elemental Analysis to support molecular formula
      3. IR absorptions of distinctive functional groups (C=O), O-H, sp C-H, et
Experimental and Characterization Data

II. EVIDENCE FOR IDENTITY AND PURITY (J. Org. Chem. Dec, 2018)

B. Degree of and Evidence of PURITY

The Journal of Organic Chemistry requires "that the purity level that has been attained be faithfully documented. When new or known synthesized compounds are the study materials for physical measurements or bioassays, a purity level of at least 95% needs to be documented."

1. $^1$H NMR spectrum with all peaks integrated (0-10 ppm)
2. Combustion Analysis (%C, %H, %N)
3. Narrow melting point range when matching known solids
Selective Analysis of $^1$H NMR Spectra?

20.24 mmol in DCM (60 mL) was stand overnight. After the solvent removal, the residue was purified with gel column chromatograph (EtOAc/petrol ether, 1:5), and the compound 25 (3.61 g, 90%) was obtained as pale yellow oil. $^1$H NMR (CDCl$_3$, 300 MHz) $\delta$: 1.45 (s, 9H), 2.31 (br, 1H), 3.79 (s, 3H), 3.85-4.00 (m, 2H), 4.39 (br, 1H), 5.45 (br, 1H).
20.24 mmol in DCM (60 mL) was stand overnight. After the solvent removal, the residue was purified with gel column chromatograph (EtOAc/petrol ether, 1:5), and the compound 25 (3.61 g, 90%) was obtained as pale yellow oil. $^1$H NMR (CDCl$_3$, 300 MHz) $\delta$: 1.45 (s, 9H), 2.31 (br, 1H), 3.79 (s, 3H), 3.85-4.00 (m, 2H), 4.39 (br, 1H), 5.45 (br, 1H).

### Derived Wt% from NMR Mole Ratios

![Diagram showing NMR spectrum and mole ratios]
Alternative writeup: …After the solvent removal, the residue was purified with gel column chromatography (EtOAc/petrol ether, 1:5). After evaporation of solvent, compound 25 was obtained as a pale yellow oil (3.61 g) containing a small amount (5 mol%, 2.1 wt% by $^1$H NMR) of ethyl acetate (88% yield of 25).
Characterization of New Biological Substances

**Identity and Homogeneity**

- **Identity** = the sequence of an oligonucleotide, gene, or protein
- **Homogeneity** = usually means only one type of biooligomer. Biologists rarely account for water content, buffer molecules, or ions. An enzyme might be considered “pure” even if contaminated with DNA, as long as they don’t affect the biological activity.

- **ACS Chemical Biology**: “Provide evidence to firmly establish both the identity and the purity of new substances. The criteria vary according to substance categories but may include electrophoretic, chromatographic, spectrometric, spectroscopic, crystallographic, or other analytical methods. Supply sequencing or functional data for all biological constructs, such as fusion proteins, plasmids, etc.”
Exceptions to Standard Characterization

Consult with your advisor in these situations.

- **Sensitive (unstable) compounds**
  Explain their reactivity (thermal ring-opening, oxygen autoxidation, acid-sensitivity). Estimate the final level of purity and offer convincing evidence of the chemical structure. You can convert it to a stable, characterizable derivative.

- **Unexpected chemical structures:**
  Require more detailed characterization.

\[
\begin{align*}
\text{H}_2\text{N} & \quad \text{CO} \\
\text{H}_2\text{N} & \quad \text{CH}_3\text{CH} & \text{C} & \text{H}_2 \\
\text{H}_2\text{N} & \quad \text{CH}_3\text{CH} & \text{C} & \text{H}_2 \\
\text{H}_2\text{N} & \quad \text{CH}_3\text{CH} & \text{C} & \text{H}_2 \\
\text{H}_2\text{N} & \quad \text{CH}_3\text{CH} & \text{C} & \text{H}_2 \\
\end{align*}
\]

Your report should be perfect.

• The spelling should be perfect.
• The grammar should be perfect.
• The references should be perfect with perfect formatting, perfect spelling, and perfect scholarship.
• The layout should be perfect.
  • Consistent fonts
  • Consistent structure drawing settings
  • Consistent orientation of chemical structures
• Drawings should appear professional
  • Key features should be clearly labeled
  • Easily readable font sizes should be chosen
• Writing or figures from other sources should be clearly referenced.
Java-Based Experimental Data Checker

OnLine Version at:
http://www-jmg.ch.cam.ac.uk/tools/magnus/checker.html

Advice For Preparation

1. Read a perfected report and a from a senior student and look at some completed Ph.D. theses from your group.

2. Talk about the report with a senior lab member and your advisor.

3. Get help. Have senior students and your advisor read over it.

4. Consult the UCI Department of Chemistry web page on Graduate Advising Information.

https://www.chem.uci.edu/Graduate-Advising
https://www.chem.uci.edu/Graduate-Advising

Graduate Advising Information

Expectations for your first year:
Your first year in graduate school will start out with heavy course work and teaching responsibilities, and transition into focusing on your Ph.D. research. You are required to complete at least seven courses, including both specialization-specific core courses and electives. Chem 200, 280, 281, 290, 399 do not count as one of the seven required courses. If a course is offered on an annual basis you are required to enroll. If the course is offered biannually, it may be taken in the second year of study. Students who are well prepared should take three four-unit courses each quarter. Students who feel a little overwhelmed by the coursework and teaching responsibilities may elect to take two.

Advising Handouts by Research Area

Chemical Biology (Prof. David Van Vranken) and Organic Synthesis (Prof. Chris Vanderwal)
- UCI Organic/Inorganic/Chem Bio Pre-advising Handout
- 2nd-Year Research Report
- Advancement to Candidacy Examination (Orals)

Inorganic Chemistry (Prof. Bill Evans)
- UCI Organic/Inorganic/Chem Bio Pre-advising Handout
- 2nd-Year Research Report

posted down here
Who Reviews the Second-Year Report?

- The second-year report is assigned for review by **two faculty referees**, much like a manuscript or grant application.
- Your Ph.D. advisor does not review your report.
- Each faculty member reviews 2-4 reports, depending on the number of reports to be reviewed.
- Faculty meet to discuss their reviews, identify weak and strong students, and determine the outcome.
Evaluation Criteria for the Second Year Report

1. Research Progress
   Is student making satisfactory progress in their research?

2. Clarity of presentation
   a. Introduction: Have the problem and the current state of knowledge been clearly defined?
   b. Results: Are results clearly presented? Did you distinguish your accomplishments from those of coworkers?
   c. Discussion: Are the conclusions supported by the data?
   d. Future Goals: Is there a plan to circumvent problems or capitalize on key findings?
   e. Experimental Procedures: Are compounds characterized with respect to identity and purity? Details are important. Format is important.
   f. Literature Cited: Is literature cited generously? Are correct formats employed?
   g. Supporting Information: Are the data accurately and clearly portrayed?
How is the Second-Year Report Reviewed?
Two faculty members will be assigned the report as primary readers.

Second-Year Written Examination in Organic Chemistry
2013

Student: ____________________________________________________________

The outcome of this exam is based on an assessment of research progress as well as the quality and accuracy of the written report. These factors are considered independently, and satisfactory completion of both components is required to receive a “Pass.” A “Rewrite required” rating will require that a revised version of the document be deemed satisfactory before the exam has been formally passed.

<table>
<thead>
<tr>
<th>Research Progress</th>
<th>Satisfactory □</th>
<th>Marginal Progress □</th>
<th>Unsatisfactory □</th>
</tr>
</thead>
</table>

Reviewer’s Comments:

<table>
<thead>
<tr>
<th>Written Report</th>
<th>Accept without revision □</th>
<th>Accept with minor revision □</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Accept after major revision □</td>
<td>Unsatisfactory □</td>
</tr>
</tbody>
</table>

Reviewer’s Comments:
Outcomes of the Second-Year Written Report / Exam

1. Pass
2. Deferred for written, pass upon rewrite/approval
3. Research progress needs to accelerate, but acceptable
4. Fail