Written Research Reports
The Second-Year Report

Due: Wednesday, noon, Nov. 21, 2018
(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.
Main Objective: Learn to Think and Write

1. Encourage **deep, scholarly thought** about your research problems, including background, significance, and goals.

2. Develop **good research habits**, including knowledge of the literature and careful documentation of experimental findings.

3. Teach **effective communication** of scientific material, including writing of an experimental procedure and accurate citation of the literature.

4. Provide additional **feedback** to students from outside their own research group and advisor.

5. Determine that students are developing as **independent** research scientists.
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
   • Why is the project important to society?
   • What is the long term objective of the science?
   • What is the short term objective of your research?
   • What obstacles did you overcome?
   • If you worked with someone else, which accomplishments are yours?
• 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.
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?
Characterization of New Chemical Substances
Give Evidence for Identity and Purity

• **Identity** = the chemical structure
  - the connectivity of atoms and bonds
  - the stereochemistry ($E/Z$, cis/trans, $R/S$)

• **Purity** = which molecules are present?
  - unless you say otherwise, it is assumed that you have prepared compounds with 100% purity

How to say otherwise: *Compound was 97% pure by $^1$H NMR with a small amount of ethyl acetate.*
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) δ: 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).
Algebra: Derive Wt% from NMR Mole Ratios

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\[
\begin{align*}
\text{EtOAc} & : n = \text{mole EtOAc}; \quad m = \text{mole product} \\
\frac{n}{m} & = \frac{5}{95} \quad \text{from NMR} \\
m \times 219.23 \, \text{g/mol} + n \times 88.1 \, \text{g/mol} & = 3.61 \, \text{g} \\
\end{align*}
\]

\[n = 0.00085 \text{ moles EtOAc}\]

75 mg EtOAc + 3.54 g product

\[
\text{corrected yield} = 87.8\% \quad \text{vs. uncorrected yield} = 89.5\%
\]
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).
Small Molecule Characterization Data Checklist:

1. **Yield.** Significant figures should match measured reagents.
2. **$^1$H NMR Spectroscopy ($^1$H NMR):** Critical to establishing both structure and purity of compounds.
3. **$^{13}$C NMR Spectroscopy ($^{13}$C NMR):** Critical to establishing both structure and purity of compounds.
4. **Infrared Spectroscopy (IR).** Some information about functional groups (structure)
5. **Melting Point.** (solids only) Informs about stability and purity.
6. **High-Resolution Mass Spectrometry (HRMS).** Provides evidence for the molecular weight and elemental composition of a compound, but provides no indication of purity. Low-resolution data is less preferable, but still useful.
7. **Optical Rotation.** (enantiomerically enriched compounds only) Provides evidence of optical and/or chemical purity
8. **Gas or Liquid Chromatography Data.** For purity and product ratios (isomeric purity)
9. **Elemental Analysis.** Absolute criterion of purity; i.e., if it’s “off,” the compound is not pure.
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.

![Chemical structures](image)

• **Unexpected chemical structures:**
  Require more detailed characterization.
Evaluation Criteria for the Second Year Research Report

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


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
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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.
How is the Second-Year Report Reviewed?

Two faculty members will be assigned the report as primary readers.
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