Writing Guidelines: Experimentals & References

Please review **Parts A-B** of the writing guidelines from CHEM 8L (available on Canvas) for general format and writing style (passive voice, no personal pronouns).

Part D: Experimental Methods and Compound Characterization

Experimental methods and compound characterization are found at the end of scientific journal articles, dissertations, and other technical documents to give the reader instructions on how to recreate the experiment and confirm the structure of the newly synthesized compounds. The format and general content differs by field (chemistry vs. biology). Include this section at the end of the final lab report using the generally accepted guidelines followed by synthetic organic chemists: one General Methods paragraph followed by one additional paragraph per compound synthesized. ***A sample Experimental Methods section follows and contains much more information than CHEM 110L students are expected to include.*** *Use passive voice and past tense; feel free to adopt the language used in Caitlin's sample provided.*

General Methods

Reagents and by-products do not get full descriptions but are mentioned in the "General Methods" section with the following statement: "**All reagents were commercially available**, unless otherwise stated." Typically researchers would then describe how reagents and solvents were purified, but *this does not apply to 110L students*. Next, define the abbreviations and list the specifications for NMR (MHz of instrument). List the **specifications for IR** (medium for analysis, such as salt plates and neat or nujol) only if used in the experiment.

Experimental Methods & Characterization

Following general methods, each organic compound or reaction gets its own paragraph (one paragraph per reaction/compound). Some or all of the following should be included in the experimental methods and compound characterization section. This is based on experimental techniques you performed in the lab, omitting any data that was provided by instructors unless otherwise stated in the lab report guidelines.

- **Reaction scheme** - including reactants, reagents, products, solvent(s), and % yield (hand-written)
- **Full chemical name of product in bold** (common and/or IUPAC)
- Brief description of **reaction set up** and **workup** including…
  - Names and amounts of each reactant and reagent (mmol and mL or mg)
  - Name and amount of solvent (mL)
  - Order of addition, if pertinent, and reaction conditions (time, temperature)
  - Description, name, and amount of product obtained and % yield:
    - Ex. "Benzhydrol was obtained as a clear liquid (1.00 g, 87% yield)."

Characterization follows in the same paragraph (after reporting the yield) and includes the following.

- **Melting point or boiling point**, if determined in the lab
- Distinctive **IR stretch(es)** – one or two distinguishing peaks, such as carbonyl or O-H stretches
- **^{1}H and ^{13}C NMR data *see sample*** - report the solvent and MHz of the instrument.
  - Check out the online predictor tool nmrdb.org for examples including *J* values! DO NOT COPY directly from nmrdb.org without checking with instructors and/or understanding what you're looking at (just ask!).
  - **Proton:** All signals listed from downfield to upfield (high to low) chemical shifts, integration, splitting
    - ^{1}H NMR ($CDCl_3$, 600 MHz) δ (ppm): 9.78 (t, *J*=1.8 Hz, 1H), 2.34 (m, 4H), 2.17 (s, 3H)…
      - The "J value" is a coupling constant; NOT required, but just ask if you'd like to learn more!
  - **Carbon:** All signals listed from downfield to upfield (high to low) chemical shifts
    - ^{13}C NMR ($CDCl_3$, 500 MHz) δ (ppm): 208.5, 202.1….

Experimental Methods Worksheet
Guidance on components to incorporate into your reports

1. Draw the **reaction scheme** by hand and list the **name of the product**.
- *The reaction scheme includes reactant, reagents over arrow, solvent under arrow, and product.*

2. What **glassware** and **equipment** was used for the reaction setup?

3. How much **of reach reagent** was used in the reaction setup?
- Solids: Convert **mass** to **mmol (xx g, xx mmol)**.
  - *Calculate or look up the molecular weight of the reagent (g/mol) = (mg/mmol).*
- Liquids: Convert volume to mmol **(xx mL, xx mmol)**.
  - *Use density (g/mL) and molecular weight (g/mol) for this calculation.*
- Solutions: How much solution (**chemical name**) was used and in what **concentration (xx M, xx mL)**?
  - Fill in the "xx" and calculate the quantity of **reagent in mmoles.**
  - *Recall Molarity = (moles / Liter) … M = (mol / L) = (mmol / mL).*
- Solvents: Report only the chemical name and volume for the reaction solvents that are not otherwise involved in the chemical reaction; no mole calculation required.

4. Determine the **limiting reagent** then calculate the **theoretical yield** (mmol and mg). Show your work, including units with every value. The theoretical yield isn't explicitly listed in the experimentals, but will be used to calculate percent (%) yield later.
- *Determine the mole ratio in the reaction (x mol reactant / x mol product).*
- *Calculate or look up the molecular weight of product (g/mol) = (mg/mmol)*

5. What was the **reaction temperature** and **time**? Was the reaction stirred, refluxed, or standing?

6. What **technique** was used to monitor **reaction progress**, if any, ex. TLC? What **solvent(s)** were used in this analysis?

7. List the **identity** and **quantities** of the **chemicals (xx mL)** used in the **reaction work-up**.
- *Note: quantities of drying agents need not be included.*

8. What **additional processes** were involved in the final isolation of product?
- Ex. rota-vap = "concentrated *in vacuo*"

9. What is the yield of **product (_____ mg, _____ % yield)**? Fill in the blanks, including units on every value.
- Calculate **percent (%) yield** with the actual yield obtained at the end of lab and the theoretical yield from above.

$$\% \text{ yield} = \frac{\text{actual yield (mg)}}{\text{Theoretical yield (mg)}} \times 100\%$$

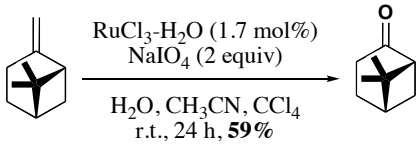
Put it all together:
- Review the Sample General & Experimental Methods section on the next page..
  - Use a similar format and writing style to incorporate necessary content from the worksheet above.
- Organize the **key information (see page 1)** into **complete, concise sentences** to allow an experienced synthetic chemist to carry out this experiment.
- Note: the experimental methods section is an *abbreviated* version of the procedure and will omit many procedural details.

Sample from Binder, C. M. **"**Novel (-)-β-Pinene-Derived Amino Alcohols as Asymmetric Directors for the Addition of Organozinc Reagents to Aldehydes" *UC Santa Cruz*, **2010**.

Sample 1: Nopinone synthesis

General Methods.

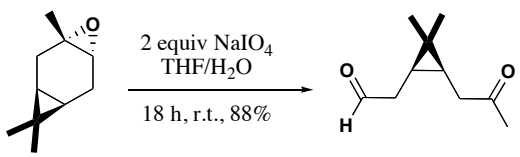
All reagents were commercially available, unless otherwise stated. All air and moisture sensitive reactions were carried out under argon atmosphere using flame- or oven-dried glassware and standard syringe technique. Tetrahydrofuran (THF), dichloromethane (DCM), cyclohexane, triethylamine (Et_3N), morpholine, *tert*-butanol (*t*-BuOH), and dimethyl sulfoxide (DMSO) were distilled over CaH_2. Oxalyl chloride was distilled without drying agent prior to use. Column chromatography was carried out with Silica Gel 60. Proton (^{1}H NMR) and carbon (^{13}C NMR) nuclear magnetic resonance spectra were carried out at 300, 500, or 600 MHz. Chemical shifts are reported relative to TMS (δ=0 ppm), $CHCl_3$ (δ=7.27 ppm) or DMSO (δ=2.54 ppm) for ^{1}H NMR and $CHCl_3$ (δ=77 ppm) for ^{13}C NMR. The following abbreviations were used to describe peak patterns where appropriate: br=broad, s=singlet, d=doublet, t=triplet, q=quartet, app=apparent, sep=septet, and m=multiplet. IR spectra were carried out on NaCl plates with v_{max} in inverse centimeters. Optical rotations were obtained on a digital polarimeter at 20 °C. High resolution mass measurements were obtained on a benchtop ESITOF mass spectrometer.



(+)-Nopinone. $NaIO_4$ (44.96 g, 210 mmol) was added to a 2-L round-bottom flask equipped with a magnetic stir bar and dissolved in water (300 mL), CCl_4 (200 mL), and CH_3CN (200 mL). (–)-β-Pinene (13.88 g, 102.0 mmol) was added followed by $RuCl_3$-$3H_2O$ (457 mg, 1.7 mmol). The reaction was stirred overnight while open to the atmosphere (24 h). The crude reaction mixture was filtered through a pad of celite and rinsed with DCM, creating two distinct layers. The aqueous layer was extracted with DCM (3 x 100 mL). The combined organic extracts were washed with water (2 x 30 mL), dried ($MgSO_4$), filtered, and concentrated *in vacuo* to a black liquid. This was purified by column chromatography (500 mL SiO_2, 100% hexane to elute β-pinene, 4:1 Hexane/EtOAc to elute nopinone) and the nopinone fractions were concentrated to a clear oil (8.3 g, 59% yield). ^{1}H NMR ($CDCl_3$, 600 MHz) δ (ppm): 2.60 (m, 1H), 2.57 (m, 1H), 2.53 (m, 1H), 2.35 (ddd, *J*=19.2 Hz, *J*=9.6 Hz, *J*=1.8 Hz, 1H), 2.24 (tt, *J*=6.6 Hz, *J*=1.8 Hz, 1H), 2.05 (dddd, *J*=13.2 Hz, *J*=9.0 Hz, *J*=3.6 Hz, *J*=1.8 Hz, 1H), 1.95 (m, 1H), 1.58 (d, *J*=10.2 Hz, 1H), 1.33 (s, 3H), 0.86 (s, 3H). ^{13}C NMR ($CDCl_3$, 500 MHz) δ (ppm): 215.3, 58.0, 41.3, 40.4, 32.8, 25.9, 25.3, 22.2, 21.4. bp 74-76 °C (2 mm Hg), $[\alpha]_D^{22}$ +34.43° (*c* 4, MeOH), IR (neat) 1714 cm^{-1}.

Sample 2 - Methodology

Representative procedure for $NaIO_4$-mediated cleavage of epoxides. To a round-bottom flask equipped with magnetic stir-bar, finely powdered sodium periodate (99% pure, 2 - 5 equiv) was stirred with the appropriate solvent mixture (2:1 THF/H_2O or CH_3CN/H_2O, 40 mL) for five minutes. The epoxide (10 mmol) was then added and the reaction mixture was stirred at room temperature. Upon reaction completion, as monitored by TLC, the reaction mixture was filtered and solids were washed with 30 mL Et_2O, creating two distinct layers. The aqueous layer was extracted with Et_2O (2 x 30 mL), washed with water and brine, dried ($MgSO_4$), filtered, and concentrated *in vacuo*. Spectra of compounds **2.3**-**2.11** were consistent with literature data.



3.4-(Dimethylcyclopropyl)-heptan-1-al-6-one, 2.7. (3*R*)-3-Carene oxide (11.3 mmol, 1.6 mL) was reacted with sodium periodate (20 mmol, 4.28 g) for 24 h in 2:1 THF/H_2O (30 mL). **2.7** was isolated as an orange oil (1.678 g, 88% yield). ^{1}H NMR (600 MHz, $CDCl_3$) δ (ppm): 9.78 (t, *J*=1.8 Hz, 1H), 2.34 (m, 4H), 2.17 (s, 3H), 1.15 (s, 3H), 0.98 (m, 2H), 0.91 (s, 3H). ^{13}C NMR ($CDCl_3$, 500 MHz) δ (ppm): 208.5, 202.1, 39.7, 39.4, 29.7, 28.5, 21.1, 19.4, 17.2, 15.2. $[\alpha]^{20}_D$ -4.8° (*c* 4.0, MeOH), IR (neat) 1719 cm^{-1}. ESITOFMS *m/z* $[M+H]^+$ 169.1266 (calcd for $C_{10}H_{17}O_2$ 169.1223).

Part E. Format for Literature References (for future reference in lab reports)

There is a standard A.C.S. (American Chemical Society) format for listing references in the chemical literature that you are required to follow (http://pubs.acs.org/books/references.shtml). This format, illustrated below, must be used in the reference section of your report, if appropriate. Be sure to document all assertions and past work described in your reports with a footnote. Footnotes can be referred to more than once. Use superscripts with corresponding numbered references at the bottom of the page or at the end of the report.

Details matter!

- Pay special attention to punctuation (period / comma / semicolon).
- **Years in bold**,
- *Volumes and titles in italics.*

BOOKS

Author's last name, first initial, *Title of Book,* Publisher: City of publication, **Year of pub**.; pages used.

Examples

Crews, P.; Rodríguez, J.; Jaspars, M. *Organic Structure Analysis, 2nd Ed.;* Oxford: New York, **2010**; pp. 67-70.

Palleros, D.R., *Experimental Organic Chemistry;* Wiley: New York, **2000**; pp. 61-70.

JOURNALS

Author's last name, initials.; 2nd author's last name, initials.; (continue for each author). *Journal abbrev.* **Year**, *Vol.,* first to last page of article.

Proper journal abbreviation used in italics*, **year in bold, *volume in italics*, no issue number

Examples

Tansakul, C.; Lilie, E.; Walter, E. D.; Rivera III, F.; Wolcott, A.; Zhang, J. Z.; Millhauser, G. L.; R. Braslau, R. *J. Phys. Chem. C,* **2010**, *114,* 7793-7805.

Sanchez, L. M.; Lopez, D.; Vesely, B. A.; Della Togna, G.; Gerwick, W. H.; Kyle, D. E.; Linington, R. G. *J. Med. Chem.*, **2010**, *53*, 4187-97.

Woehrmann, M. H., Gassner, N. C., Bray, W. M.; Stuart, J. M.; Lokey, S. *J. Biomol. Screen.* **2010**, *15,* 196-205.

WEB SITES

Use full website addresses to allow the reader to locate referenced material on the web. **Be wary of the content. The info on the web is usually not peer reviewed, and can be erroneous!** If you do cite a website, include the date the website was accessed.

Example

http://organicchemistry.wordpress.com/2007/08/18/tips-for-writing-organic-chemistry-lab-reports/ accessed 7-23-09.