Organization and Style of Lab Reports

See the schedule for report drafts and revised due dates. The reports are to be organized in a neat and professional manner. ***Write like your job depends on it!*** One of the goals of CHEM 146A is to develop your proficiency in reporting the results of experimental work. The results of scientific research are of little value unless they are known to others who can make use of them. Written reports are more important than oral communications. These take the form of papers in the chemical literature. This body of literature serves as an important repository for past experimental, theoretical, and conjectural results on a variety of topical areas.

A written report should be both concise and clear. Clarity in writing rests on a thorough understanding of the problem undertaken and of the significance of the results. One must be able to convey this understanding to the reader in a coherent, organized, and concise fashion. ***Lab reports written with poor grammar and spelling will be returned and graded only after revisions are made.***

Students should be accustomed to the style of technical writing outlined in CHEM 8L/M. Revisit the writing guidelines from your previous courses and/or access this online. The following points are of particular importance in 146A reports.

* Reports should be typed, double-spaced, justified text with adequate margins. Pages should be numbered, including attached spectra (hand-written page numbers are OK for spectra). The report should be printed on paper and securely stapled or bound in a folder.

* Use the lab report cover pages provided online.

* Literature references should be sequentially numbered with in-text citations, and placed at the end of the report. See **Appendix II** for the appropriate format, as used in *J. Am. Chem. Soc*. **If a reference is listed at the end of the report, it needs to actually be used and cited in the text.**

* Common abbreviations may be used: mm, cm, g, mL, Hz, NMR, sec, mp, GC, HPLC. Periods should not be placed after these except in. for inch and no. for number which do require periods. You can (and should) define abbreviations parenthetically for anything else the first time they are used. Thereafter use that abbreviation. Do not switch back and forth!

Ex. “The 2,4-Dinitrophenylhydrazine (DNPH) test…”

* To ensure clarity, it is common to sequentially number compounds and structures with boldface integers: **1**, **2**, **3,** etc. These numbers are used to clearly refer to compounds in the body of the text.

* All reports should use ***grammatically correct, complete English sentences***. While your lab book can be written using phrases, the report should be written professionally, with proper grammar and spelling. The ACS style guide sets the following protocol for scientific writing: use past tense for your experimental findings, and use the present tense for "statements of fact" from the literature. Reference all information retrieved from the literature with footnotes (see 3 above!).

* In *scientific writing*, **avoid the use of the first person ("I" or "we")**. For example, instead of writing "I recrystallized my neutral unknown from ethanol to give 0.66 g of a solid as white needles;" instead write: "The neutral unknown was recrystallized from ethanol to give a white solid (0.66 g)."

* Include a document header including your name and unknown number so that this information appears on every page.

* EVERY figure, table, and scheme is given a number and descriptive title. EVERY one of these should be referenced in the text, preferably before the figure, table, or scheme.

- Scheme = reaction; Figure = anything not a reaction or table, ex. mechanisms
- Ex. **Table 1.** ^{1}H NMR Shifts of Acetanilide
- "The chemical shifts, integration, and splitting patterns for acetanilide can be found in **Table 1** below."

  …or…
- "The structure of acetanilide was confirmed by ^{1}H NMR spectral analysis (**Table 1**)."

* Elements and chemical names are NOT proper nouns.

* Do not start sentences with numbers or abbreviations. Write out the name for numbers below ten (one, two, three…10, 11, 12…).

**** The teaching team (Caitlin, Grant, and Patrick) are available to help with your reports. Help us help you by reading the provided guidelines carefully before asking for help. It makes a lasting impression when students clearly have taken the effort to work out issues on their own, then discuss the outcome with the instructor. That being said, don't wait too long to ask for help!***

FORMAT FOR LITERATURE REFERENCES

There is a standard A.C.S. (American Chemical Society) format for listing references in the chemical literature that you are required to follow. 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 or endnote. Footnotes and endnotes can be referred to more than once. Use superscripts[1] with corresponding numbered references at the bottom of the page or at the end of the report.

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 University Press: New York, 2010; pp. 67-70.

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

JOURNALS

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

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

These are not recommended for this class. **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. Use full websites addresses so a reader could locate your referenced material on the web.

Example

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

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 depending on the field. Students will 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 is provided online and contains much more information than CHEM 146A students are expected to include.*** *Use passive voice and past tense.*

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 146A students*. Next, define the abbreviations and list the specifications for NMR (MHz of instrument) and IR (medium for analysis, such as salt plates or Teflon) only if used in the experiment.

Experimental Methods

Following general methods, each organic compound or reaction gets its own paragraph (one paragraph per reaction/compound). Depending on the forms of analysis available to students (based on experimental techniques as well as spectra provided), some or all of the following should be included in the experimental methods and compound characterization section.

- Reaction scheme - including reactants, reagents, products, solvent(s), and % yield
  (structures and reaction schemes can be 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)."

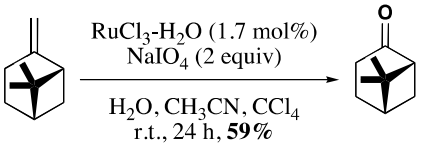
Characterization follows in the same paragraph (after reporting the yield) and includes some or all of the following.

- ^{1}H NMR data – peaks listed downfield to upfield with chemical shift, integration, splitting, and coupling (*J*) values
- Melting point or boiling point
- Optical rotation
- Distinctive IR stretch(es) – one or two distinguishing peaks, such as carbonyl or O-H stretches

Sample Experimental Methods: The following is an excerpt of the supporting information for Caitlin's Ph.D. dissertation and contains significantly more details than are applicable to CHEM 146A students. Follow the general format below and apply to reports where experimental methods sections are required.

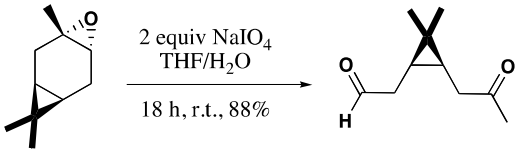
General Methods.

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}.

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).