Writing Guidelines for Second-year Reports, 2019

Due Wednesday, noon, 27 November 2019

The form of the examination is a paper in the style of the *Journal of Organic Chemistry* or similar American Chemical Society journal. Your examination will be evaluated on the basis of content (research progress) and form (organization, clarity, writing quality, etc.). The main body of the paper will consist of an Introduction, a Results section, a Discussion section, and a Conclusion and Future Work section. The main body is not to exceed 10 total pages in length (double-spaced). In addition, an Experimental Section, References and Notes, and Supporting Information Section should be included. There are no page limits on these sections.

Brief descriptions of some of these sections follow:

- Introduction. This section allows you to explain the significance of your research. What is the problem that you are studying? Why are you studying it? What has been done by others to address this problem? You should provide sufficient background information that would enable a student in a different research group to understand the rationale for your research.
- Results. Summarize your data. Use figures and tables to help present the data in a clear fashion. Organize this section carefully. It should not be a chronological narrative of each experiment that you have conducted, but a logical presentation of the experimental highlights. In some cases, unsuccessful or inconclusive experiments can provide insight into your research problem.
- Discussion. This section should provide interpretation of the data and what its implications are for the project as a whole. Relate your findings to material presented in the introduction. If you are studying a particular chemical reaction, postulate possible mechanisms and explain how your data is consistent with your proposal. When employing other authors' arguments, cite relevant references.
- Conclusions and Future Work. Conclude with the key findings of your experiments. You should discuss future directions of your research.
- Experimental Section. This important section will contain the experimental details of how your research was conducted. New compounds should be characterized completely, and procedures should be documented in detail. Do not report experimental or characterization data for previously reported compounds unless you have changed the procedure by which the experiment was conducted or have additional characterization data. This section should document your research. Citations to the literature should be included where appropriate; if a compound you prepare is known, a reference should be included. Detailed instructions for composing experimental sections are provided in the *Guidelines for Authors* for ACS journals (see http://pubs.acs.org/about.html, select the appropriate journal, and go into "info for authors," and the *Guidelines* can be found there).

- Supporting Information. Material that is not appropriate for the body of your paper but will be included in your dissertation should be included as Supporting Information (for example, graphs, GC or HPLC traces, etc.). Include ^{1}H and ^{13}C NMR spectra of new compounds that are described in the Experimental Section. Such material is often very useful to reviewers (for example, to determine whether ^{1}H NMR spectroscopic data are consistent with pure compounds).

Stylistic aspects of the paper can be found in the *Guidelines for Authors*. Please note that you do not need to use the templates and other forms, as these are designed for electronic manuscript handling.

Writing (style, abbreviations, journal citations, etc.) must conform to the *ACS Style Guide*, which is available for free online https://pubs.acs.org/isbn/9780841239999#. For attractive chemical drawings, follow the directions provided in the *Guidelines for Authors*. Structures drawn following these guidelines and reduced to 75% will look the same as they look in the journals. If your paper does not fit within the 10- page space constraint, we will request that you conduct the appropriate editing before it will be evaluated.

Characterization of Organic Compounds

To report new compounds in the chemical literature, both identity and purity must be carefully documented. Determination of a compound's identity requires a detailed survey of the properties of a compound using various techniques: mass spectrometry, ^{1}H and ^{13}C NMR spectroscopy, infrared spectroscopy, DNA sequencing, and/or biological activity. Establishing the purity of newly reported compounds is necessary to judge the significance of the data from identity characterization. For example, does the synthetic scheme employed provide clean materials? Is any new biological activity due to the newly reported compound or it is an artifact of an impurity?

Demonstration of purity is critical to all forms of chemical research, although some compounds are more amenable to such analysis than others. Elemental analysis is a time- honored technique that provides evidence for both purity and molecular formula. Elemental analysis is not readily applicable to all compounds, however (for example, DNA plasmids, salts, polymers, and hydrated materials). In such cases, establishing the homogeneity of the compound is an acceptable demonstration of composition. Techniques used to establish homogeneity include chromatographic methods (such as GC or HPLC), electrophoresis gels, GPC, ^{1}H NMR and ^{13}C NMR spectroscopy, and DNA sequencing. The specific requirements for your compound may require consultation with your research advisor. See also the PowerPoint slides for the 2nd-Year Report Advising Meeting posted at: https://www.chem.uci.edu/Graduate-Advising

For reports that rely heavily on organic synthesis of small molecules (not proteins or nucleic acids), assignments of NMR resonances must be made when possible, in order to demonstrate complete understanding of structural assignments.

The following text from the *Journal of Organic Chemistry* "Information for Authors" indicates some issues you will need to consider when conducting your experiments and writing the appropriate experimental data.

"The Journal upholds a high standard for compound characterization to ensure that compounds being added to the chemical literature have been correctly identified and can be synthesized in known yield and purity by the reported preparation, isolation, and purification methods. For all **new** compounds, evidence adequate to establish both *identity* and *degree of purity* (homogeneity) must be provided. Purity documentation must be provided for **known** compounds whose preparation by a new or improved method is reported. For combinatorial libraries containing more than 20 compounds, complete characterization data must be provided for at least 20 diverse members."

You should consult the "Information for Authors" for an exhaustive discussion of characterization.

Some Tips for Writing Manuscripts

A worthwhile guide to writing would take about 100 pages. Mercifully, Strunk and White have written just such a guide: *Elements of Style* (an outstanding investment if you happen to be at a bookstore). Believe it or not, computer grammar checkers are quite good, so I would try that first. They are imperfect, however, so I have compiled a few reminders for you. These topics tend to be the same ones I notice with every paper or grant I read:

1. **Paragraphs and topic sentences.** Paragraphs are units that include related sentences. Paragraphs have topic sentences that indicate how the paragraph is related. These sentences are the first sentence of every paragraph. For example, you might have a series of sentences that describe important NMR data such as hydrogen bonding or a variable-temperature NMR experiment. These sentences should flow logically from one to the next and not jump around. One might use the following topic sentence as the first sentence: "Variable temperature NMR experiments provided important information about the hydrogen bonding pattern of compound **11**."
2. **Verb tense.** You should not switch verb tense in the middle of a paper. Usually past tense is preferred, although sometimes present is more appropriate.
3. **Subject/verb agreement**. Check to make sure subjects and verbs are either both singular ("Amide **6** was crystallized") or both plural ("Amides **6** and **8** were crystallized"). Be aware that sometimes the subject defines a collection but is itself singular ("A mixture of amides **6** and **8** was crystallized" with "mixture" as the subject).
4. **Extraneous words.** Words such as "surprisingly," "interestingly," "much to our delight," and "in the event" add little to text. "Very" also adds little to a qualifier... just think, would you trump "very" with "oh so very" or, more simply, "very very?"
5. **Colloquial expressions**. The above words are colloquial as well, but some other colloquial expressions are better rendered more formal. For example, "The ester was reacted with the amine" sounds fine in the laboratory, but this level of informality is not

appropriate for a report. The word “treated” can often be substituted for “reacted” and therefore papers can be made more formal rather easily. Be aware, though, that sometimes you can overuse the same sentence structure, so change it up a bit. We also do not perform operations on a “reaction” in formal writing. We filter/concentrate/etc. the “reaction mixture.”

6. **Contractions.** Contractions aren’t (I mean, “are not”) appropriate for formal writing.
7. **Imprecise subjects of sentences.** “This” cannot serve as the subject of a sentence. Although it can serve as a noun, it is not a precise one. Specifying what “this” is can be much better: the expressions “this phenomenon,” “this compound,” or “this experiment” can serve as subjects of sentences.
8. **Weak sentence constructions.** A few common sentence constructions are believed to be weak because the infinitive “to be” must be employed as the verb. For example, sentences starting with “There is...” or “There are...” need no other verbs. Consider the following:

   “There are no examples of silacyclopropanes used as synthetic intermediates.”

   compared with:

   “Silacyclopropanes have not been employed as synthetic intermediates.” The second sentence has a more active verb and is thus a “stronger” sentence.
9. **Qualification.** Do not have gradations of yield like high/good/satisfactory/moderate/modest/poor. Calling something “high yield” or “poor yield” is descriptive, but one person’s “modest” is another’s “poor.” The same idea applies to selectivities, etc.
10. **Numbers.** Numbers less than or equal to twelve are normally written out (whereas 13 and higher are not).
11. **Experimental data.** In figures, put only the most important experimental details. Too much information will only serve to hide key data. You will find that a description of reagents is sufficient, and the details of solvent and temperature are not critical. Do not include too much experimental detail in the text of a paper, either. Save the details like volume of solvent, etc. for the experimental section.

Writing Experimentals:

Symbols, Punctuation, and Formatting for Experimental Data

Note: The keystrokes were checked using Microsoft Word for Macintosh OSX, so if your symbols do not look ideal, you will need to figure out which are the best keystrokes for your word processing program and computer.

Temperature: 65 °C	Degree sign: SHIFT+OPTION+8
Temperature: –78 °C	Minus = en dash: OPTION+hyphen (without space)
Selectivity: 98:2	No space before and after ':'; normalized to 100 (use spaces in ChemDraw figures, though, because the 75% reduction makes the numbers microscopic)
Yield: 95%	No space between number and '%'; no decimals
Joining names: Horner–Emmons reaction	En dash: OPTION+hyphen
A melting point: mp = 65–67 °C	Use en dash and degree sign. Add spaces around '=' and after the number but not around the en dash.
A weight: 73.0 mg	Space after number; select grams or milligrams to make the number easy to read. Try to avoid switching between milligrams and grams in a single procedure.
A volume: 35 mL of water	Use small m, capital L, then "of" before the solvent.
Hours: 7 h	Space before "h", same symbol for singular and plural
Micromoles: 13.2 μmol	μ: OPTION+m; μ: m in Symbol font.
Compound Formulas: $BF_3 \cdot OEt_2$	'·' character: SHIFT+OPTION+9
NMR Format: δ 7.25 (s, 1H)	δ: d in Symbol font
Coupling: (dd, *J* = 10.0, 6.7, 1H)	Italicize J. Spaces around '=' , no space in the integration. "Hz" does not need to be written, as the general experimental has this unit by default.
	1.7–1.4 (m, 3H) Use en dash for a range of values.

^{1}H NMR Data: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.20–7.35 (m, 5H), 5.05 (d, *J* = 2.9, 1H), 4.24 (dq, *J* = 6.4, 2.0, 1H),

If overlapping data, use the following:
1.08 (s, 9H and m, 1H)

^{13}C NMR Data: $^{13}C\{^{1}H\}$ NMR (125 MHz, $CDCl_3$) δ 143.3, 128.0, 126.9,

The $\{^{1}H\}$ indicate proton decoupled. Only use the hundredths place when you have two peaks that are so close together that they would round to the same number. Example: $^{13}C\{^{1}H\}$ NMR (125 MHz, $CDCl_3$) δ 143.3, 128.0, 126.91, 126.85, 124.0

Rotation: $[\alpha]_{24}^{D}$ = 2.1 (*c* 0.91, $CHCl_3$)

α: a in Symbol font, c italicized, space after it. Specific optical rotations do not have units, so do not put in a degree sign.

MS: HRMS (ESI/Q-TOF) m/z: $[M + H]^+$ Calcd for $C_{19}H_{33}O_2Si$ 321.2250; Found 321.2249.

Space around '+' (or '–'). No spaces in m/z. The '+' is superscript. If the ion is an M^+, then use $[M]^+$ with the "+" as superscript. Also, if you have lost a *t*-Bu group for example, use $[M - C_4H_9]^{+\cdot}$ List the molecular formula of the ion (not the molecule as a whole) and any added elements in the ion (e.g. H^+, Na^+). Electrons have a mass of ca. 0.0005. A cationic ion has this mass subtracted. Many calculators give you neutral masses, so remember to check and subtract an electron mass if necessary. ChemDraw Analysis window subtracts the electron mass when the structure has an explicit positive charge.

Elemental Analysis:

Anal. Calcd for $C_{19}H_{32}O_2Si$: C, 71.18; H, 10.08. Found: C, 70.97; H, 9.98.
Found values should be within 0.4% of calculated.

Naming: (2*R*,3*S*)-Dimethyl…

'*R*' and '*S*' are italicized and always put in parentheses.
Use no spaces in a name. Only numbers and italicized words have dashes around them.

Volumes: 3 x 10 mL

"x" is "x" in Helvetica or Arial font

(–)-roxaticin

Use en dash for minus sign, separate name fragments with a hyphen (no line break); natural products are not proper nouns, so they are not capitalized

N-methylmorpholine *N*-oxide

(*E*)-2-butene.

Italicize atom symbols in a chemical name.
Italicize '*E*' and '*Z*' when they are part of the name of a compound, but not otherwise.

tert-butyl alcohol	Italicize *tert, sec*
15:85 – 20:80 EtOAc:hexanes	Spaces around '– (en dash)' but not ":"
>94% er	No space between '>' or '%' and number. Space between '%' and 'ee'. Use er in preference to ee.

References

A space but no punctuation before '*J.*' and '**1969**'. References should now include titles. For example:

Dale, J. A.; Dull, D. L.; Mosher, H. S. α-Methoxy-α-trifluoromethylphenylacetic Acid, a Versatile Reagent for the Determination of Enantiomeric Composition of Alcohols and Amines. *J. Org. Chem.* **1969**, *34*, 2543–2549.

Note that one word journal titles are not abbreviated:

Clark, T. B.; Woerpel, K. A. Silver-Catalyzed Silacyclopropenation of 1-Heteroatom-Substituted Alkynes and Subsequent Rearrangement Reactions. *Organometallics* **2005**, *24*, 6212–6219.

A complete list of correct reference abbreviations can be found in the *ACS Style Guide*.
The formatting of references may be handled by EndNote or many other programs for managing bibliographic information.

Miscellaneous Symbols:

hyphen '-'	'-': hyphen
en dash '–'	'–': OPTION+hyphen
em dash '—'	'—': SHIFT+OPTION+hyphen
Line return (not a new paragraph)	(insert page break from Word menu)
heat: 'Δ'	'Δ': OPTION-j.

Words that should not be abbreviated: saturated, quantitative, aqueous, powdered.
Nouns that take singular verbs: grams, milligrams, equivalents, number, mixture,

Useful abbreviations: h (hour(s)), min (minutes), equiv (equivalents), eq (equation), mmol (millimoles), calcd (calculated)

Numbering:

Scheme 1, 2, 3…
Figure 1, 2, 3 …
eq 1, 2, 3 …
Table 1, 2, 3 …

Abbreviations:
Use standard abbreviations that are acceptable for the journal (consider defining any that might be less common). For example, the *Guidelines for Authors* for *J. Org. Chem.* gives a detailed list that might be helpful.

Miscellaneous Chemistry Words:
to describe "rotovapping": "concentrated *in vacuo*"
Celite (capitalized)
one-pot, overalkylate, transmetalation
"reflux" is not a verb: "heated at reflux" is the more accurate description

The following text is a sample experimental in JOC format (we thank Dr. J. H. Smitrovich from the Woerpel lab for assembling this experimental). Please note the grammar used to describe the procedure and the format for reporting characterization data.

(1*R*,2*S*,3*R*)-1-Phenyl-2-methyl-1,3-butanediol (2). To a cooled (0 °C) solution of *tert*-butylhydroperoxide (207 mg, 2.30 mmol, 90%) in 1.3 mL of DMF was added $CsOH·H_2O$ (0.331 g, 1.97 mmol). After the mixture was allowed to warm to 25 °C, a solution of oxasilacyclopentane **1** (50.0 mg, 0.164 mmol) in 0.8 mL of DMF was added dropwise by syringe. After 10 min, *n*-Bu_4NF (214 mg, 0.82 mmol, hydrate, lyophilized from benzene) was added. The reaction mixture was heated at 75 °C for 8 h. After the mixture was cooled to 25 °C, $Na_2S_2O_3$ was added, and the solvent was removed *in vacuo*. The resultant oily solid was partitioned between 5 mL of H_2O and 10 mL of Et_2O. The layers were separated, and the aqueous layer was extracted with 2 × 10 mL of Et_2O. The combined organic layers were washed with 10 × 1 mL of H_2O and 5 mL of brine, dried ($MgSO_4$), and concentrated *in vacuo* to afford 57 mg of a yellow oil. The oil was purified by flash chromatography (25:75 – 35:65 EtOAc:hexanes) to yield the product as a colorless oil (19 mg, 64%): ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.20-7.35 (m, 5H), 5.05 (d, *J* = 2.9, 1H), 4.24 (qd, *J* = 6.4, 2.0, 1H), 3.85 (br s, 1H), 3.4 (br s, 1H), 1.71 (m, 1H), 1.23 (d, *J* = 6.5, 3H), 0.83 (d, *J* = 7.2, 3H); ^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$) δ 143.3, 128.0, 126.9, 125.6, 78.4, 72.0, 44.9, 21.4, 4.0; IR (thin film) 3355, 2975, 1380, 1199, 974, 742 cm^{-1}; HRMS (CI/isobutane) m/z: $[M + H]^+$ Calcd for $C_{19}H_{33}O_2Si$ 321.2250, found 321.2249. Anal. Calcd for $C_{19}H_{32}O_2Si$: C, 71.18; H, 10.08. Found: C, 70.97; H, 9.98.

Tips for Proofreading Experimentals

1. All experimentals should be consistent. Keep in mind, though, that they should all be consistent *and* correct.
2. Significant figures must be considered. If a weight or volume is only precise to two significant figures, then no calculation using that number can be more precise than to two significant figures.
3. Several abbreviations, punctuations, etc. can be inadvertently incorrect. Check things like “mL” instead of its smaller relative “ml”... stuff like that.
4. Solids must have melting points. Melting points are reported as a range.
5. Optically active materials should have optical rotations.
6. Check the IR for key functional groups that the compound should have. Only a half dozen or so important frequencies should be listed.
7. Does the phase (solid or liquid) of the IR match the reported phase?
8. Make sure that the spectral data is in the correct order and is consistent experimental to experimental.
9. Check ^{1}H NMR spectra for the proper number of hydrogens. Make sure integrations are listed for every peak, and that all data is presented consistently.
10. Consider the ^{1}H NMR spectral data. Is it consistent with the structure?
11. Check agreement of coupling data with the proposed structure. Coupling constants must appear twice, naturally.
12. Check ^{13}C NMR for the proper number of carbons. Is the spectrum consistent with the structure?
13. Make sure the HRMS passes. Double-check the format. Is the ionization method indicated?
14. Check the elemental analysis to make sure it passed (±0.4%). Make sure the format is correct and that the molecular formula is consistent with the HRMS.
15. If there are compounds with the same molecular formula (such as diastereomers), most of the data between the two ought to be similar (but not identical). Check the relation, because then you can catch mistakes in both at the same time.
16. Make sure compound numbers agree with the text.

One way to proceed with proofreading repetitious data (compound characterization): Examine one particular type of data for all of the experimentals (the assembly-line method). Make sure the format is right for every compound and that all the data make sense. For example, start with mp, R_f, optical rotation, HRMS. Then proceed to the IR, ^{1}H, and ^{13}C NMR spectra together for each compound, since at that point you will be thinking more intensely about structure and functional groups.

Written by Prof. Keith Woerpel, with some updates by Profs. Chris Vandewal, Scott Rychnovsky, and David Van Vranken