Guide to Writing – Holland Group

updated 2/14

Filenames:
- It is very useful when transferring files to know when they're from, and who the last person to modify it is. So, for your papers, it's advisable to use filenames like TMP0406-ph to indicate a paper on TMP complexes, modified April 6 by PLH.

Notes and common mistakes with papers:
- The "highlight changes" function in Microsoft Word is great for keeping track.
- Figures in a journal will typically need to be 3.5 inches wide or less. Make sure that they will fit in this space with sufficiently large letters/numbers to allow visibility. Don't use color unless necessary: stick with black/white.
- Graphs look good created in Kaleidagraph, with the following typical parameters: use ticks out from the axis; no grid, and no axes on top and right; black & white only; export as tiff at 200% with "minimize white space" box checked, then convert to GIF using GraphicConverter before pasting into document.
- Many students have a tendency to paste in TIFF files or other HUGE files. Turn them to GIF format first, which will make them much smaller. If you're unsure, look at the size of the picture file before putting it in. There's rarely a reason to use a picture that's more than about 200 KB.
- For ACS journals, make sure you format references with FirstAuthor, J. C.; SecondAuthor, C. J. *Journal* **Year**, *Volume*, Page Range. There is no punctuation before or after the name of the journal! The "Insert endnote" function works best. To get the formatted reference, I recommend using EndNote: select the reference, use {apple} K (i.e. "copy formatted"), then paste this in. Use "insert cross-reference" if you use that reference a second time. Before showing it to someone else, you should update the cross-references (select all text, then F9 on a Mac).
- For *Angew. Chem.*, put the references in a numbered list at the end, and then use cross-references for all of the endnote markers in the text. Again, update cross-references.
- The reference marker comes AFTER punctuation, except in *Science*. So, it's like this.[10] Not like this[10].
- I usually use ## in the text to remind myself of something that I need to revisit. (This is convenient because the # symbol is rarely used otherwise, and then it's possible to find the ## symbols using the "search" function.)
- Calculate error bars on your numbers whenever possible. This is very important.

Example of a good synthetic writeup for the Experimental Section follows. Notice that the flow is strictly chronological, that amounts are generally in parentheses, and that it avoids vague terms like "crystallized," "refluxed," and "in vacuo."

L^{tBu}NiCl. A Schlenk flask was loaded with $NiCl_2(THF)_{0.7}$ (1.75 g, 10.2 mmol), L^{tBu}Li(THF) (3.72 g, 6.41 mmol), and THF (30 mL) and heated to 70 °C overnight. Volatile materials were removed from the dark green mixture under vacuum, and the residue was extracted with CH_2Cl_2 (70 mL), filtered, and concentrated to 15 mL. Addition of diethyl ether (10 mL) and cooling to -35 °C gave green crystals of LNiCl. A second crop of crystals was collected to give a total yield of 2.5 g (66%). ^{1}H NMR (400 MHz, CD_2Cl_2) δ 29.3 (4H, *m*-aryl), 18.5 (4H, iPr methine), 5.6 (12H, iPr methyl), 4.1 (12H, iPr methyl), 2.5 (18H, tBu), -14.7 (2H, *p*-aryl), -123.5 (1H, backbone). μ_{eff} (CD_2Cl_2, 295 K)= 3.1 μ_B. IR (CH_2Cl_2): 2967(vs), 2869(m), 1587(w), 1521(m), 1464(m), 1385(m), 1366(s), 1315(s), 1268(vs), 1221(w), 1099(w), 1057(w) cm^{-1}. UV-vis (CH_2Cl_2, ε in mM^{-1}cm^{-1}): 372 (8.4), 467 (sh, 1.7), 838 (1.4) nm. Anal. Calcd for $C_{35}H_{53}N_2NiCl$: C, 70.54, H, 8.96, N, 4.70. Found: C, 69.25, H, 9.02, N, 4.63.

template:

Compound Name. Description. ^{1}H NMR (400 MHz, T, solvent) δ # (H, assignment), # (H, assignment) ppm. μ_{eff} (solvent, T) = # BM. IR (method): #(#), #(#) cm^{-1}. UV-vis (solvent, ε in mM^{-1}cm^{-1}): # (#), # (#) nm. Anal. Calcd for empirical formula: C, #, H, #, N, #. Found: C, #, H, #, N, #.

(continued on next page)

Notes and common mistakes with experimental descriptions:

- If you use compound numbers, the whole compound name should be given as well in the Experimental Section.
- Our standard notation now is L^{tBu} and L^{Me} for the two most common ligands.
- Every sentence should sound normal without the parenthetical sections, which list the numbers. The amount is given in g or mg *and* in mmol or μmol. There is *always* a space between the number and the units (even in 70 °C). Thus, "Pentane (5 mL) was added to KC_8 (50 mg), and the mixture was stirred…" because without the parentheses it reads normally as "Pentane was added to KC_8, and the mixture…"
- The number of significant figures in the weight should match that in the number of moles.
- A common mistake is to say that xxx (# g, # mol) was heated. It was the *flask* or the *solution* that was heated/filtered/cooled, etc.
- "Volatiles" is not a word. We typically use "volatile materials."
- Avoid Latin abbreviations like *in vacuo*, *vide infra*, *e.g.* –use English "under vacuum," "see below"
- Abbreviations for units of time: y, d, min, s. "Molar equivalents" is abbreviated equiv. These standard ACS abbreviations *do not* have periods after them.
- Quadrupole splittings from a zero-field Mössbauer measurements could be positive or negative, and therefore it is best to give them as an absolute value, $|\Delta E_Q|$.
- "Ether" is a class of compound. When you have used diethyl ether, you must specify the "diethyl."
- Don't speculate about what you filtered off/evaporated/removed. Only describe what you did… if you want to say what really happened with the chemicals, you need to have *evidence*.
- Leave out unimportant information like what it was filtered through, the size of the flask, how long it was cooled, unless it's important for reproducibility.
- A *slurry* is solid + solution. A *solution* implies that it is all dissolved.
- You cannot *re*crystallize something unless you crystallized it once already. Usually, it's best to avoid the issue by saying what you did: you cooled it or slowly diffused in a solvent, and then collected crystals.
- List the amounts of solvents used: this is often important.
- You don't need to list the amount in each crop: a composite yield is fine.
- You don't need to describe the method for growing analytically pure crystals or for single crystals unless it differs substantially from the crystallization method you described.
- Carefully look at the spacing used in the spectroscopic descriptions in the template above!
- You should list the frequency of the NMR spectrometer, unless it was listed in the General part of the Experimental. Note that the same magnet has *different* frequencies for different nuclei, based on differences in the gyromagnetic ratio. Also, in the general part, you should describe how you calibrated the frequencies in your spectra (ex. 7.15 ppm for C_6D_6).
- Don't give chemical shifts for paramagnetic compounds to the hundredth place, because this is rarely reproducible. Use the same number of decimal places for all peaks. For paramagnetic compounds give the temperature at which the NMR spectrum was taken, because the chemical shifts are sensitive to *T*.
- The first number after the chemical shift is the integration. You don't need the "H" for integrations.
- We used to give T_2 values or some other estimate of peak broadness for paramagnetic compounds, but now I think the best way is to put the actual NMR spectrum in Supporting Information.
- Your labeling for the peak assignments should be clear to someone who's not familiar with our chemistry.
- Magnetic moments need to have the solvent and temperature. If it is a multinuclear compound, specify "per iron" or "per dimer" or whatever.
- Yes, you need to list all major peaks in IR spectra. Yes, I know it's a hassle. Sorry!
- Don't give molar extinctions (or any other quantity) to more significant figures than you trust to be reproducible.
- Note the formatting of the analysis data. Another acceptable format is: Elem. Anal. Found(calcd): C, 69.25(70.54); H, 9.02(8.96); N, 4.63(4.70). Journal standards are that all need to be within 0.4%.
- The empirical formula in your crystal structure report might be wrong if you have solvent in the crystal.

You should consult these guidelines carefully several times while writing your paper to make sure that you are conforming to the guidelines. If you learn these things early and well, it will save everyone time (my time editing, and your time revising), and you will be able to devote more of your energy to becoming an effective writer!