

Characterization

For each of the compounds that you synthesize in lab, you will need to choose which of the following data you should compile:

^1H -NMR spectrum (samples prepared in lab; run outside of lab)

IR spectrum (run in lab)

UV-vis spectrum (run in lab)

(No acetone or organic solvents in the plastic cuvetts! Organic solvents can dissolve plastic.)

Melting point (if solid)

TLC

Training for NMR, IR, and UV-Vis will take place the first three weeks of lab.

What do I do if I don't have enough time or material to do all of these?

If you run out of time or material, then you get the chance to do some decision making. You have to convince your GSI that you made your target product, so which technique will give you the best data to make the best argument and write the best discussion section for your lab report.

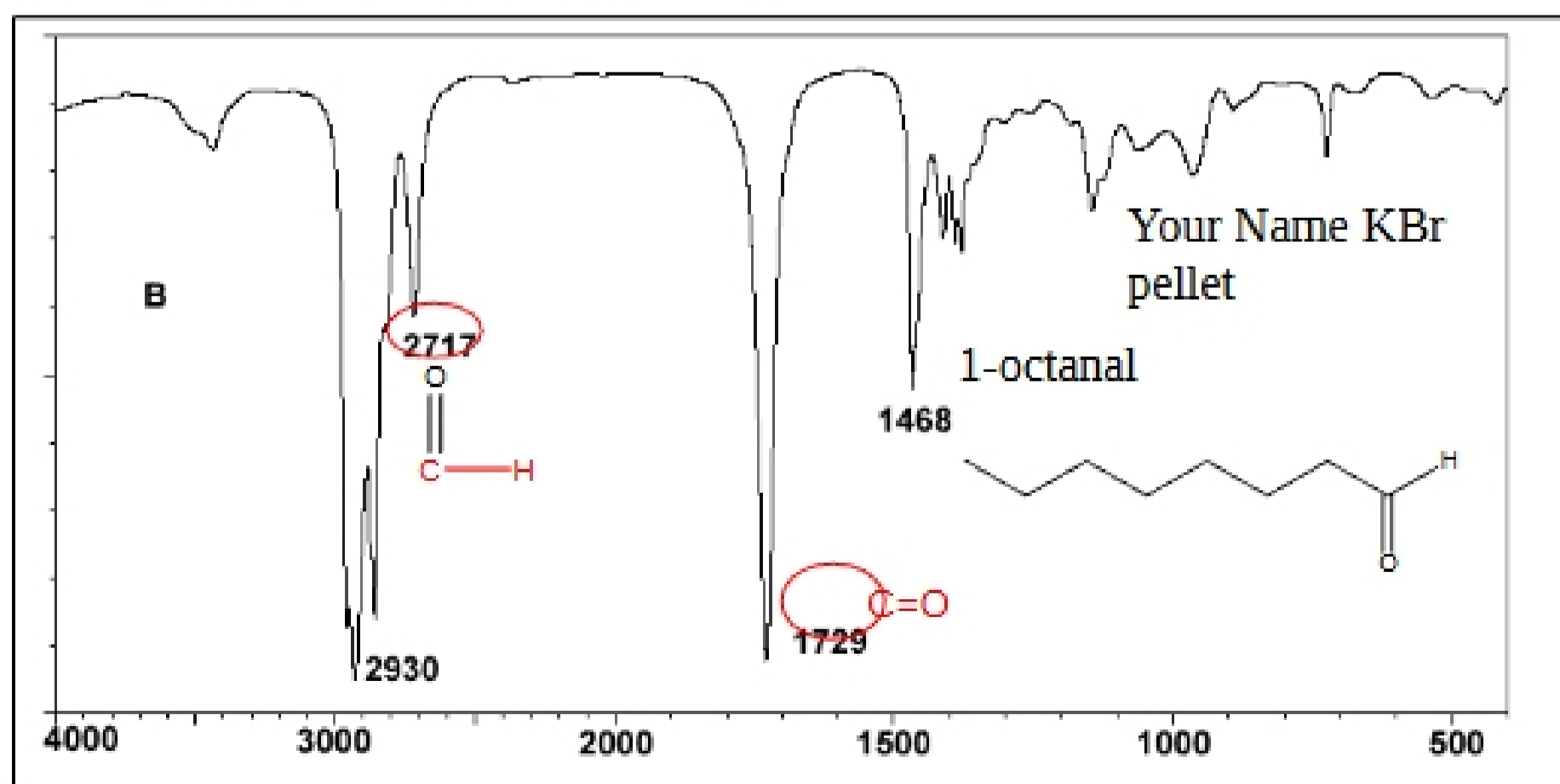
What do I do with all of those spectra?

Each completed spectrum should be labeled with the name of the person who recorded the data, the molecular structure and name of the compound, and the collection conditions (solvent, KBr pellet, etc.)

Make judgements about the spectra you collect—is the spectrum clear? Can a logical conclusion be based on/supported by the data?

For UV-Vis spectra, you should include them in your lab report (tape them to a 8½ x 11 sheet of paper); include the λ -max in the experimental section.

For IR spectra, you may circle or draw arrows to the relevant peaks and label the functional group stretch that the absorption represents.

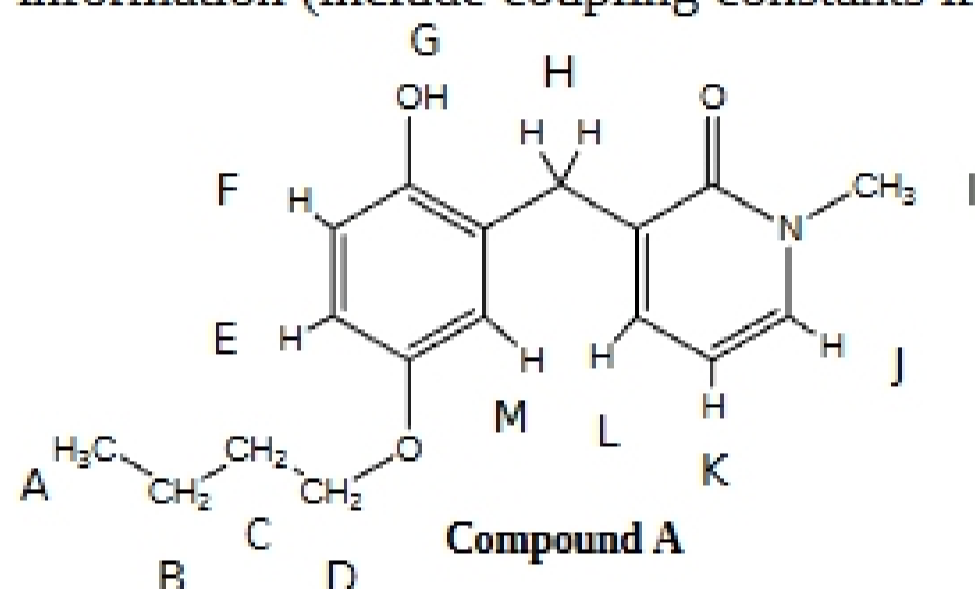


Working up NMR spectra

For NMR spectra, remember to phase your spectrum. Then set a reference peak to TMS, CDCl_3 , or another known compound so that you can read the correct chemical shifts. Integrate all of the peaks. Pick one peak from your compound that you are confident you have identified. Set the integral of that peak to the number of protons that it represents. (Methyl groups work well as they are often unique peaks that have an integral of 3; or a hydrogen on an aromatic ring that you know is unique has an integral of 1). Finally, pick peaks to display chemical shifts.

Including NMR spectra in your report

On the spectrum, draw the molecule (by hand) and label each group of protons on the molecule and the corresponding peak on the spectra. Then use a table to organize all of the spectral information (include coupling constants if you can):



Assign-ment	Chemical shift	Integration	Splitting pattern	Coupling
A	0.95	3 H	triplet	$J = _ \text{ Hz}$
B	1.47	2 H	sextet	
Etc.....				

Don't forget to look up reference compounds. There may be spectral data available for your compound. A solid claim to making your target compound would come from matching your data to that which is published. And, what are those other peaks in your NMR? Are they solvent or starting material? Do you know the spectral characteristics of your starting material so you can describe how they changed as the molecule was synthetically transformed?

Some common solvent peaks:

- CHCl_3 7.25 ppm (this is almost always found in CDCl_3)
- H_2O 1.5 ppm (often broad)
- Hexanes 0.88 ppm (t), 1.26 ppm (m)
- CH_2Cl_2 5.3 ppm
- Acetone 2.17 ppm
- Ethyl Acetate 2.05 ppm (s), 4.12 ppm (q), 1.26 ppm (t)
- Diethyl ether 1.2 ppm (t), 3.5 ppm (q)
- TMS 0 ppm (added to CDCl_3 as a reference)

There are two on-line databases that contain spectra you may be interested in:

SDBS <http://www.aist.go.jp/RIODB/SDBS/menu-e.html>.

NIST <http://webbook.nist.gov>

Keep in mind these databases do not include all compounds nor are they always taken in the same solvent or under the same conditions as the spectra you are comparing them to. All spectra obtained from this site or any other source must always be properly referenced.

If the internet does not hold what you are looking for, try the library:

The Atlas of near infrared spectra by Sadtler Shapiro Science - Reference Collection | QC457 .A771 1981

The Aldrich library of FT-IR spectra Shapiro Science Reference Collection Tables

Nuclear magnetic resonance spectra by Sadtler [Shapiro Science - Reference Collection](#) | QD 96 .N8 S13

The Aldrich library of NMR spectra Shapiro Science Reference Collection Tables

And, if you can't find a spectrum, try looking up the chemical shift data using Beilstein.