

MASSACHUSETTS INSTITUTE OF TECHNOLOGY

ORGANIC CHEMISTRY 5.46

The following is an approximate algorithm for determining the structure of a molecule.

Determine the unsaturation number (UN), a.k.a. index of hydrogen deficiency or IHD, to see how many rings or double/triple bonds should be in the final structure.

Make a table of shifts, integrals, and multiplicities for the proton 1D spectrum.

Inspect the proton 1D spectrum and pick out any obvious functionalities such as para-substituted benzene rings (a non-first-order doublet of doublets around 7.2p), exchanging protons (broad, featureless peaks), or basic aliphatic multiplets (e.g., ethyl, isopropyl).

Inspect the carbon 1D spectrum and make general observations and possibly some initial hypotheses as to what sort of carbon skeleton you might have. Make a table of shifts for the carbon resonances.

Look at the HMQC / HSQC spectrum and establish proton-carbon pairs. Remember that a methylene group near an asymmetric center will have two proton shifts for one carbon, and also remember that methyl groups have very strong cross peaks.

Examine the TOCSY spectrum to identify isolated proton spin systems. Use the TOCSY data for confirm or refute some of your initial assumptions about some of the functionalities present. Be aware that the TOCSY may contain additional, spurious cross peaks. Use the proton 1D spectrum to identify and discard these cross peaks.

Put the pieces together in the various possible combinations. Rule out those structures that are not consistent with the chemical shifts you observe in the proton and carbon 1D spectra. Proton couplings will also help you piece together the correct structure.

View the HMBC spectrum (if available) and look for any long-range couplings that will allow you to establish the overall connectivity of your molecular fragments. Beware of HMQC / HSQC peaks that will appear with one carbon but two proton shifts (consult the HMQC / HSQC to look for the center point between the two peaks) caused by having the decoupler off during the acquisition of the FID. Also be cognizant that the HMBC spectrum may have many symmetry-related artifacts that should be ignored.