EXPERIMENTS AVAILABLE ON MERCURY 300 AUTOSAMPLER

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General notes on parameters

Some parameter to consider changing are:

ns, the number of scans; the more scans you acquire, the better will be your signal-to-noise ratio. But, if you find that your experiments require a long time, you should consider using a higher field spectrometer (400 MHz or 500 MHz).

d1, the relaxation delay (the time between the end of one acquisition and the next excitation pulse); if this is too short, then ^{1}H and ^{19}F spectra may not be quantitative (integrals may not appear correct). Increase it to 5 or even to 10 s for more quantitative spectra. Note that ^{13}C spectra will not usually be quantitative, even with a long d1, because of the NOE effect, which is different for different ^{13}C sites.

sw, the spectral width; if this is too small, then you will not see highly shielded or deshielded protons. Check that the spectral width covered is sufficient for your expected sample.

Proton

The basic proton experiment has 16 scans and takes about one minute (plus sample change, locking, and shimming). The main parameter to change is ns, or number of scans: consider using 32 or 64 scans to increase your signal-to-noise ratio. For better integration, you might increase the relaxation delay by a few seconds. If you expect proton signals to show up with very low chemical shift values (for example, hydrides), then be sure that your spectral width is wide enough.

Carbon

The basic carbon experiment has 1000 scans and takes 50 min (plus sample change, locking, and shimming). The main parameter to change is ns, or number of scans; consider using fewer or more scans, depending on the concentration of your sample. This experiment is proton-decoupled, but fluorine-coupled, so carbons that are one, two, or even three bonds away from ^{19}F will be split by C-F J coupling. So, CF_3 groups will appear as quartets with a large splitting (200-250 Hz); CF groups will appear as doublets.

Fluorine

The basic fluorine experiment has 16 scans and takes about one minute (plus sample change, locking, and shimming). Like with proton experiments, consider changing the number of scans and the recycle delay.

C13_F19DEC

This experiment is a carbon experiment with fluorine decoupling ($^{13}C\{^{19}F\}$). Like the basic carbon experiment, it has 1000 scans and takes about 50 minutes. In this spectrum, carbon signals are split by CH coupling (one bond: 120-160 Hz, two or three bond 2-10 Hz), so CH_3 groups will appear as quartets and CH_2 groups as triplets. But, CF_3 signals will appear as singlets.

Hsqc

This experiment is a 2D experiment. Each peak has two chemical shifts associated with it: the chemical shift of a proton, and the chemical shift of a carbon that is directly bonded to the proton. The experiment is more sensitive than a standard carbon experiment and is a very useful, quick way to obtain carbon chemical shifts. As a bonus, it is DEPT-edited: CH_2 peaks are negative while CH and CH_3 peaks are positive. However, quaternary carbons (carbons not bonded to proton) will not appear. The main parameter to vary is the number of scans (try 8 if 4 is not enough). Increase the t1 increments to 200 or 256 if two carbon chemical shifts are similar, because this number will increase resolution in the carbon dimension. Finally, if you suspect that you have an aldehyde group in your molecule, increase the ^{13}C spectral width to cover the aldehyde region.

Dept

This experiment is a quick way to see protonated carbons: it is more sensitive than a standard carbon, but non-protonated carbons do not appear. The basic experiment acquires the full set of DEPT spectra (DEPT-90: CH only; DEPT-135: CH + CH_3 up / CH_2 down; DEPT-45 (all protonated carbons, up)), but you can choose a particular spectrum under the “XH multiplicity editing” option. You will probably need to increase the number of scans above the default of 128.

Cosy

This experiment is a 2D experiment in which the presence of a cross-peak (a non-diagonal peak) indicates that two protons are J-coupled to each other. You might need to increase the number of scans above 1.

Noesy

This experiment is a 2D experiment in which the presence of a cross-peak (a non-diagonal peak) indicates that two protons are near each other in space. For small and medium sized molecules, the NOESY effect will be greater at lower field than at higher field. Parameters to consider are: the number of scans (4 may not be enough), the number of increments (reduce ni for a faster experiment, albeit at the price of resolution in the indirect or vertical dimension) and the NOE mixing time (the longer the mixing time, the further the correlations that can be seen, though with too long a mixing time, relay transfers become important). Note that chemical exchange between two sites appears in NOESY spectra in the form of peaks that are the same sign as the diagonal peak and the opposite sign as the off-diagonal peak.

Tocsy

This 2D proton experiment shows entire coupled spin systems in the same row or column (if proton A is coupled to B, which in turn is coupled to C, then A, B, and C will appear in the same row or column, even if A is not coupled to C). Parameters to consider are: the number of scans (4 may not be enough), the number of increments (reduce ni for a faster experiment, albeit at the price of resolution in the indirect or vertical dimension) and the TOCSY mixing time (the longer the mixing time, the further the correlations that can be seen).

Hmbc

This experiment complements the HSQC. It correlates protons in the direct dimension with carbons that are two or three bonds away in the indirect dimension. Consider decreasing the Multiple-Bond Jnxh value to 5 Hz to see the further couplings, and consider increasing the number of scans and/or the number of t1 increments to improve signal-to-noise (and resolution in the case of the number of t1 increments).