EXPERIMENTS AVAILABLE ON VARIAN MERCURY 400

August 9, 2016

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.

d1, the relaxation delay (the time between the end of one acquisition and the next excitation pulse); if this is too short, then spectra may not be quantitative (integrals may not appear correct). Increase it to 5 or even to 10 s for more quantitative spectra.

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 8 scans and takes 29 s (plus sample change, locking, and shimming). The main parameter to change is ns, or number of scans: consider using 16, 32, or even 64 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.

Phosphorus

The basic phosphorus experiment has 64 scans and takes 2 min (plus sample change, locking, and shimming). It is proton decoupled. This spectrometer is much less sensitive than the Bruker spectrometers or the Varian 500 (which requires special training for phosphorus). The main parameter to change is ns, or number of scans; consider using 128 or 256 to increase your signal-to-noise ratio.

Hmbc_P31 or gHMBC

These are the same experiment, a ^{31}P HMBC, which correlates ^{1}H and J-coupled ^{31}P sites (usually two or three bonds away from the ^{1}H). Consider changing the parameter dof (Acquire / Channels / P31 Offset) to a value that is close to the expected ^{31}P chemical shift. If this parameter is very different from the actual phosphorus chemical shift, you will have much

reduced intensity. To make it easier to set dof to the correct value, enter it in ppm.

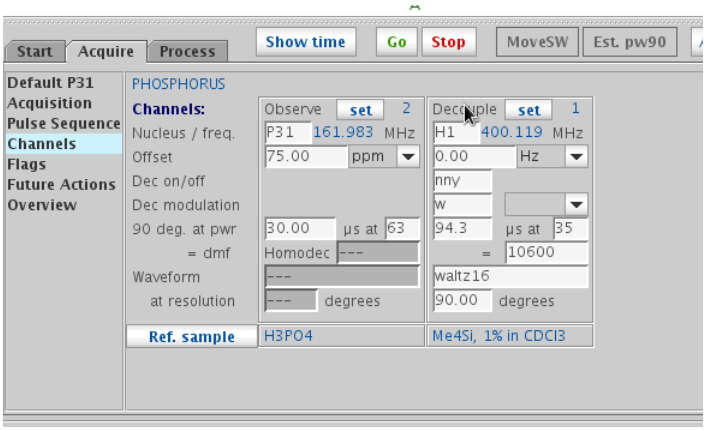

Figure 1: Customizing the centre of the phosphorus dimension of the phosphorus HMBC. Set the phosphorus offset to be close to the expected chemical shift of phosphorus for better results.

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 (16 may be too many), the number of increments (128 may suffice, but will give poorer resolution in the vertical or indirect 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 experiment is a 2D experiment that correlates all protons that are in the same spin system. So, if proton A is J-coupled to proton B, and proton B is J-coupled to proton C, protons A, B, and C will show up in the same row and same column, even if A is not J-coupled to C. The TOCSY mixing time (“Spinlock duration”, under Defaults) can be increased to see longer range J-coupling or decreased to see shorter range J-coupling.