Nuclear Magnetic Resonance

Homonuclear Decoupling

Homonuclear decoupling in proton spectra can be extremely helpful in determining atom connectivity, especially in complex spectra and spectra with AMX coupling.

Procedure

Fully Coupled Spectrum

Insert a sample and lock the magnet. Shim the magnetic field as carefully as possible. The use of autoshim (`tune`) is not discouraged.

Read the standard proton parameter file (`rpar`). Establish a new dataset (`edc`). Adjust the receiver gain (`rga`) and acquire a proton spectrum. Adjust the phase of the spectrum, provide a title, adjust the peak-picking parameters, and integrate (if desired).

If desired, adjust the spectral window and spectrometer frequency offset to show only the proton spectrum, minimizing any excess baseline. Phasing and integration may need to be readjusted.

Record the values for `o1p` and `sw`. Print the spectrum.

Decoupled Spectrum

Establish a new `expno` (typically it is simply incremented by one) using `edc` for the current dataset. Read the standard parameter file for acquiring homonuclear decoupled proton spectra. Set `o1p` and `sw` to the settings for the proton spectrum taken previously.

Set `o2p` to the first peak to decouple. The setting of `pl24` (power level 24) sets the decoupler power. A setting of 45-50 dB is usually sufficient to decouple the protons. The lower the setting, the higher the decouple transmitter power. Setting the decoupler transmitter power too high will cause energy "bleed" to adjacent protons.

Acquire the spectrum. Observe what peaks in the spectrum change due to decoupling. There is an artifact in the spectrum of the decoupler transmitter at the transmitter frequency. This can be ignored.

Move the decoupler transmitter frequency to every peak for decoupling, observing the effects. Print each spectrum.

Analysis

Based on the information obtained by decoupling, identify which protons are coupled and assign the spectrum based upon the molecular structure.