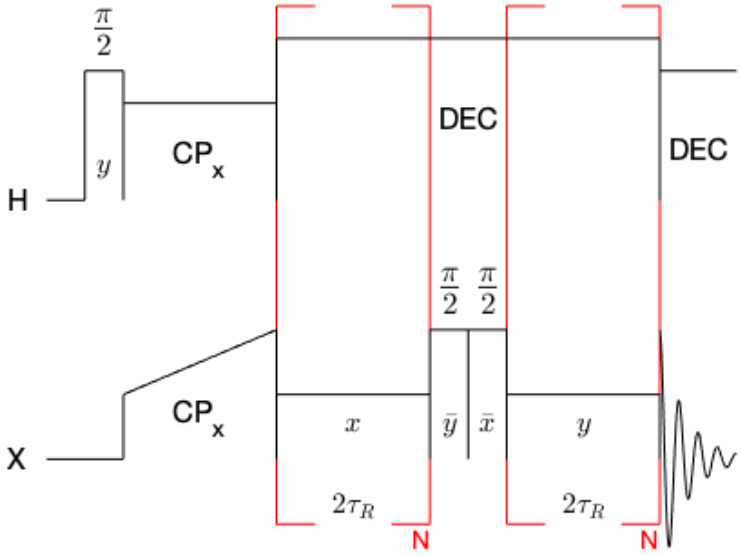
October 2022 NMR Topic of the Month: Oh the HORROR!



For what does the acronym HORROR stand in NMR?

HORROR = HOmonucleaR ROtary Resonance

What is the role of the HORROR sequence, and how does it work?

HORROR is a solid-state NMR recoupling technique, like the REDOR experiment (see the topic from six months ago) except that HORROR utilizes homonuclear direct dipole couplings. To reintroduce the X_1-X_2 (note: X_1 may be the same as X_2) spin coupling a rf-field on the X-spins of strength $\omega_R/2$ (where ω_R is the spinning frequency) is applied between them following cross polarization from the protons. The mechanical spinning of the rotor is thereby partially undone by the applied spin-lock, which in principle reintroduces other terms of the Hamiltonian. However, under the correct conditions, the dipolar coupling is the principal term recoupled by HORROR.

What does a pulse strength in frequency mean?

When we refer to a pulse strength in frequency, we mean the frequency required to make a $\pi/2$ pulse. Specifically, for a pulse of duration t and therefore frequency υ:

$$\omega t = \frac{\pi}{2} \Rightarrow 2\pi\upsilon t = \frac{\pi}{2} \rightarrow \upsilon = \frac{1}{4t}$$

So if a $\pi/2$ pulse is determined at 50μs, the frequency of the pulse is 5kHz. If that power level were the spin-lock for the HORROR experiment, the correct spinning frequency would be 10kHz.

What is the role of the two X-pulses between CP and acquisition?

Those pulses make a double-quantum filter. In some versions of the HORROR these pulses are absent, but most often this sequence is run on spin labeled material for rare spins. The double-quantum filter eliminates the natural abundance signals from the rest of the molecule.

References

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