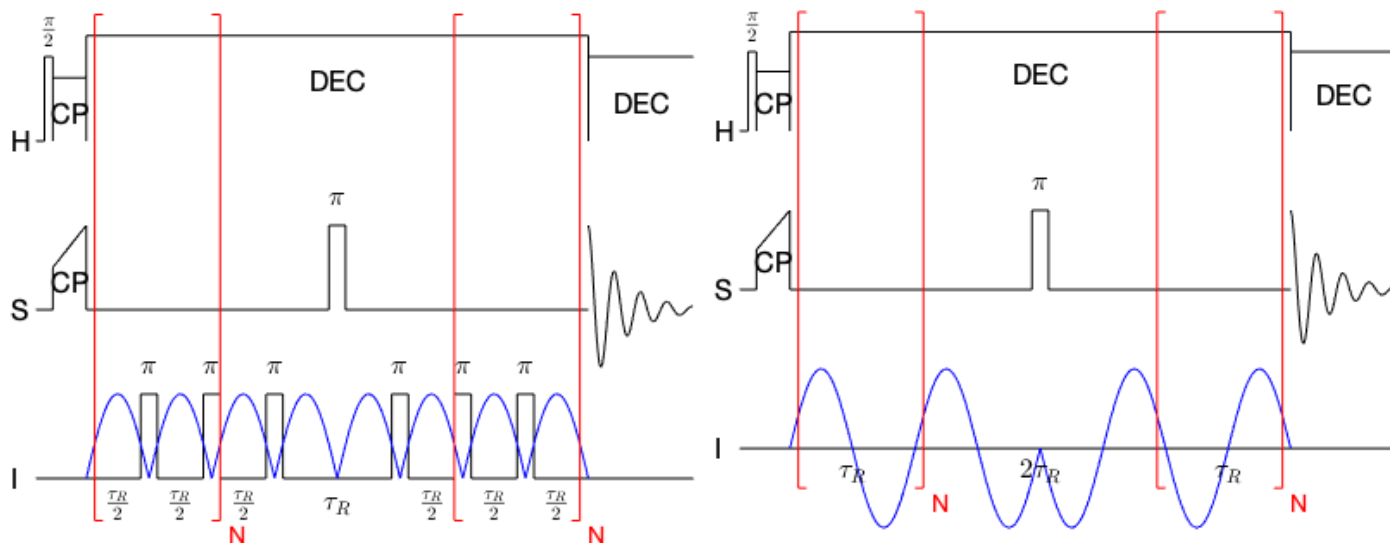


April 2022 NMR Topic of the Month: The REDOR Pulse Sequence



For what does the acronym REDOR stand?

REDOR = Rotational Echo DObble Resonance

What is the role of the REDOR sequence?

REDOR is a solid-state NMR sequence for determining the distance between spins under fast magic angle spinning.

How does the REDOR work?

Fast magic angle spinning normally leads to the direct dipolar coupling averaging to zero, but the rotor-synchronized π pulses interfere with this averaging in a controlled way. In the pulse sequences above the blue wave represents the sine component of the direct dipole coupling, notice how the sum is non-zero with the I channel π pulses but zero without. The REDOR experiment is collected using both of the sequences shown above over many choices of N , and the result is plotted as $1-S/S_0$ where S is the signal from the experiment with the I channel π pulses and S_0 is the signal from the simple spin echo. By fitting this resulting universal curve, the distance between the I and S spins can be determined. Therefore, the experiment allows for distance determination using isotropic (narrow) lines gained from fast magic angle spinning.

What is the role of the S_0 curve?

The recoupling reduces the overall signal, but so does relaxation. The simple spin echo compensates for relaxation.

How easy is REDOR to do?

It is trickier than it looks. You should use well-calibrated pulses that are on resonance; and, even then, need to account for them being non-ideal. You need a stable MAS rate ($<0.05\%$ off) and a probe that can take the power. You should try to get the universal $(1-S/S_0)$ curve out past the first maximum, and ideally past the immediately following minimum. If your sample is dense and labeled you often need to cut it with natural abundance material and account for those signal contributions. There are several variants to the sequence which may facilitate the analysis, but the REDOR concept and setup requirements do not change. In general, it is not a difficult experiment to do, but it can take some work to do well.

References

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