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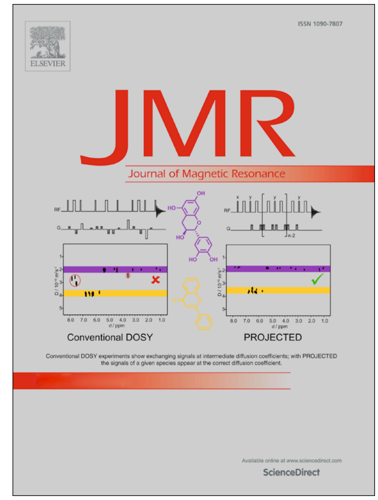

Magic Angle Spinning NMR Below 6 K with a Computational Fluid Dynamics Analysis of Fluid Flow and Temperature Gradients

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Magic Angle Spinning NMR Below 6 K with a Computational Fluid Dynamics Analysis of Fluid Flow and Temperature Gradients

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ABSTRACT

We report magic angle spinning (MAS) up to 8.5 kHz with a sample temperature below 6 K using liquid helium as a variable temperature fluid. Cross polarization ^{13}C NMR spectra exhibit exquisite sensitivity with a single transient. Remarkably, ^{1}H saturation recovery experiments show a ^{1}H T_1 of 21 s with MAS below 6 K in the presence of trityl radicals in a glassy matrix. Leveraging the thermal spin polarization available at 4.2 K versus 298 K should result in 71 times higher signal intensity. Taking the ^{1}H longitudinal relaxation into account, signal averaging times are therefore predicted to be expedited by a factor of >500. Computer assisted design (CAD) and finite element analysis were employed in both the design and diagnostic stages of this cryogenic MAS technology development. Computational fluid dynamics (CFD) models describing temperature gradients and fluid flow are presented. The CFD models bearing and drive gas maintained at 100 K, while a colder helium variable temperature fluid stream cools the center of a zirconia rotor. Results from the CFD were used to optimize the helium exhaust path and determine the sample temperature. This novel cryogenic experimental platform will be integrated with pulsed dynamic nuclear polarization and electron decoupling to interrogate biomolecular structure within intact human cells.

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