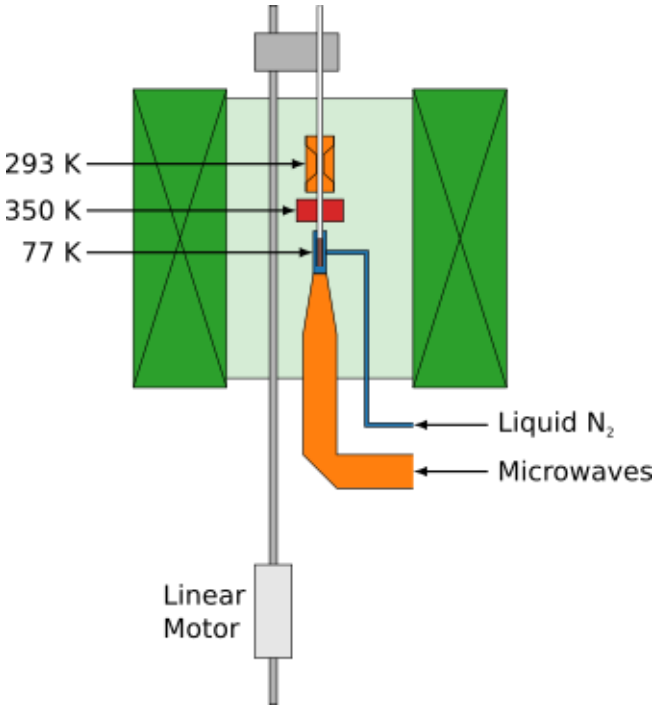
Rapid-Melt DNP-NMR: A novel approach for high-sensitivity NMR.

S. G. J. van Meerten, A. P. M. Kentgens
Magnetic Resonance Research Center, IMM, Radboud University, Nijmegen

The greatest bottleneck of NMR is the inherently low sensitivity. Over the years a number hyperpolarization techniques have been developed to address this issue. A particularly promising method is Dynamic Nuclear Polarization (DNP), where the large polarization of electron spins is transferred to the nuclear spins by microwave irradiation. This polarization transfer is quite efficient in solids. However, for chemical analysis with NMR a liquid sample is often preferred for high resolution spectra.
To obtain large enhancements in the liquid state using solid state DNP, a novel Rapid-Melt NMR probe has been developed. In this probe a small volume sample is located inside a fused silica capillary, which can be moved between three positions using a linear motor. At the bottom position the sample is frozen using liquid nitrogen and irradiated with microwaves. The DNP at low temperature results in a large polarization of the nuclear spins. The sample is then moved to a heated melting block. Due to the low heat capacity, the sample melts in 100 ms. Since this melting time is short with respect to the relaxation time, a greatly enhanced polarization will be available in the liquid state. The NMR signal is recorded using a stripline detector located at the top position in the probe.
We report new results repeating the cycle of DNP, melt, and NMR to record high sensitivity multidimensional experiments. The time of a single cycle is on the order of a few seconds. The capillary in the probe is constructed in such a way that is can also be used in stopped-flow experiments. These features make Rapid-Melt DNP a versatile method for recording NMR spectra with high sensitivity.



1. Figure: Schematic of the Rapid-Melt NMR probe