Development of a Dissolution DNP Instrument: For Applications and Technique Development

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Introduction

The inherent insensitivity of the NMR/MRI technique is well known. Many avenues have been followed in an attempt to attenuate this problem, for example building higher field magnets, designing more sensitive RF circuits and obtaining higher quality samples. Recently, dynamic nuclear polarization (DNP) has emerged as a promising technique for NMR signal enhancement. DNP enhances the NMR signal by transferring the higher polarization of an unpaired electron to a nucleus of interest. Dissolution DNP has been demonstrated as a powerful orthogonal technique for enhancing small molecule polarization by up to three orders of magnitude. We are implementing dissolution DNP technology in concert with our existing programs for developing RF coils and mid to high magnetic fields to enable *in-vivo* metabolic studies. In a typical dissolution DNP experiment a metabolite of interest is hyperpolarized in one instrument (a DNP polarizer) and then transferred to a second instrument (either a conventional NMR spectrometer or an MRI scanner) where the hyperpolarized metabolite is utilized in any number of experiment types. This work has focused on the development of the first instrument, a DNP polarizer.

Results and Discussion

Various components are required for a dissolution DNP system, the most important being: a magnet, cryostat, and microwave source. Our system utilizes a 300 MHz 89 mm magnet, which is currently charged to 5 T (212 MHz). To ensure the unpaired electron has maximal polarization to transfer to the nucleus of interest the sample is cooled to sub 1.2 K temperatures, to achieve these temperatures a cryostat is necessary. We have chosen a system that was developed by Comment A. *et al*[1] at the EPFL which also includes a means for the rapid dissolution of the sample and its transfer to the second magnet system. The cryostat reaches low temperature by evaporative cooling of liquid helium, thus the cryostat is connected to a vacuum pump system, composed of a rotary vane and roots blower pumps, capable of a displacement of 500 m^3h^{-1}. To induce the polarization transfer irradiation of the sample with microwaves (MW) is required. A 140 GHz MW source capable of 135 mW of output power was selected for this system. Figure 1 shows the system assembled in the AMRIS facility of the NHMFL. Many groups in the dissolution DNP arena have focused on metabolic studies of acetate; therefore, we chose sodium acetate as the metabolite to demonstrate hyperpolarization in our instrument. Polarization was transferred from the unpaired electron on 4-oxo-TEMPO to sodium acetate labeled with ^{13}C at the carbonyl position. Figure 2 shows the solid-state enhanced signal generated in the DNP polarizer. We have demonstrated an enhancement of approximately 430 compared to non-polarized signal at 1.2 K.

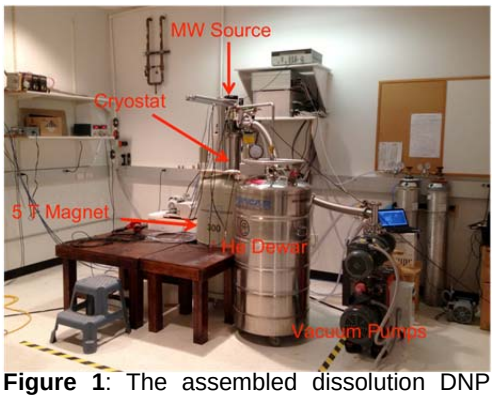

Figure 1: The assembled dissolution DNP polarizer in the AMRIS facility of the NHMFL1.

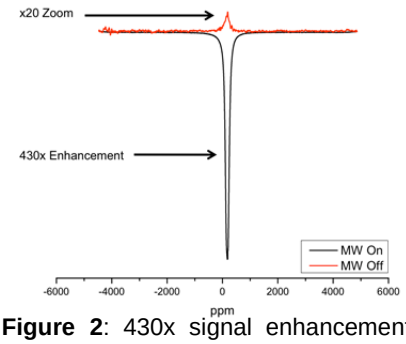

Figure 2: 430x signal enhancement of Na Acetate at 1.2 K.

References

1. Comment A *et al Conept. Magn. Reson. B* 2007, *31B, 255-269*