High-Power Solid-State Millimeter-Wave Sources to Enable Advanced EPR and DNP-NMR Measurements

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DNP uses a high-power millimeter-wave source to irradiate electron spins and transfer high electron polarization to the nuclear spins, thereby enhancing the NMR signal. This fundamentally reduces the time required to acquire the desired data; making a much broader range of measurements feasible. The critical barrier to the greater use of DNP-NMR and advanced EPR systems is the lack of sufficiently powerful and frequency agile solid-state sources. Currently, DNP is performed with either relatively low-power (~50mW) solid-state sources on low temperature samples (20-30K), with higher power TWT amplifiers at higher cryogenic temperatures, or with single-frequency high-power Gyrotrons. In this work, we have developed and demonstrated an electronically tunable multiplier-based solid-state source with 256 mW output power at 262 GHz. This source was used to measure DNP enhancements of 50 using a 3.2 mm sample diameter probe and 120 using a 1.3 mm diameter sample probe, both at 100 K sample temperature. This is one-fifth and one-third respectively the maximum achievable DNP enhancement using a gyrotron oscillator with several Watts output power. Frequency tuning also allows fast acquisition of DNP field dependence profiles, eliminating the need to sweep NMR magnet for optimal DNP efficiency.

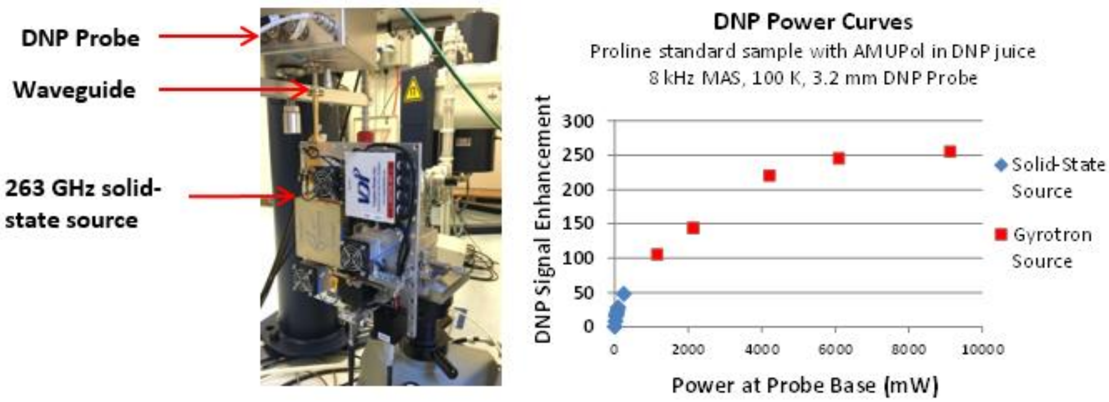

Figure 1. Left: Photograph of 262 GHz solid-state source mounted beneath 400 MHz NMR magnet and DNP probe. Right: Measured DNP enhancement versus mm-wave power for the solid-state source and gyrotron source using a 3.2 mm DNP probe.

For the 260 GHz frequency doublers, GaAs Schottky diode arrays were fabricated and flip-chip mounted on 0.0012" thick diamond embedding circuits. The choice of diamond provides a high thermal conductance path from the diodes to the aluminum split-block waveguide housing, since overheating of the diodes is a primary limitation to increased power handling. Waveguide housing were designed and fabricated in conjunction with the diode arrays and embedding circuits to combine the output of two diode multiplier chips. Approximately 20% conversion efficiency was measured for the power-combined 260 GHz doublers over a ~10% tuning bandwidth. Similarly, power-combined doublers were developed for 130 GHz. These yielded over 800 mW output power with about 30% conversion efficiency, also over a ~10% tuning bandwidth. In the final x18 active multiplier chain, these two doublers are driven by a power amplifier from Quinstar, Inc. with 2.5-3.0 W output power over a similar fractional bandwidth. The entire active multiplier chain is shown on the left side of Fig. 1 mounted underneath a DNP probe at Bruker BioSpin. The right side of Fig. 1 shows the measured DNP enhancement versus power level using a 3.2 mm probe for both the solid-state active multiplier source and a gyrotron source. Using a 1.3mm probe with optimized sample irradiation, DNP enhancements of 115-120 were measured. Fig. 2. shows a ^{1}H-^{13}C CP-MAS spectra with and without DNP using the solid-state 262 GHz source and 1.3mm probe

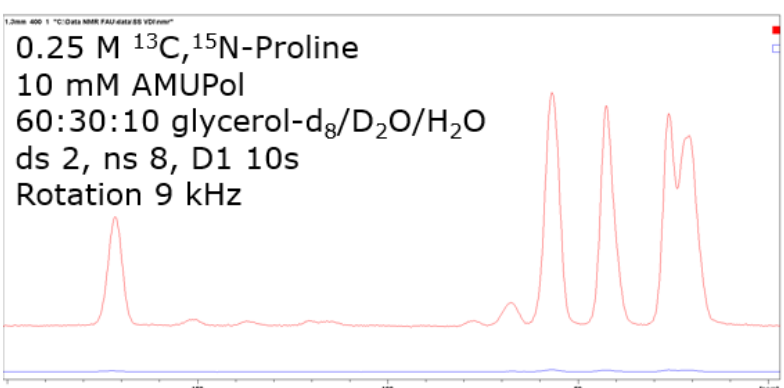

Figure 2. Measured ^{1}H-^{13}C CP-MAS spectra with and without DNP using the VDI solid-state 262 GHz source and 1.3mm probe.