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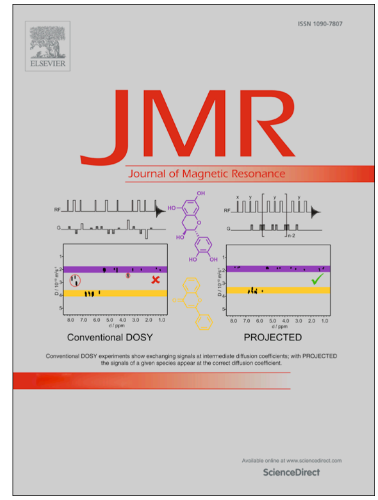

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A quasi-optical and corrugated waveguide microwave transmission system for simultaneous dynamic nuclear polarization NMR on two separate 14.1 T spectrometers

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Abstract

Nuclear magnetic resonance (NMR) is an intrinsically insensitive technique, with Boltzmann distributions of nuclear spin states on the order of parts per million in conventional magnetic fields. To overcome this limitation, dynamic nuclear polarization (DNP) can be used to gain up to three orders of magnitude in signal enhancement, which can decrease experimental time by up to six orders of magnitude. In DNP experiments, nuclear spin polarization is enhanced by transferring the relatively larger electron polarization to NMR active nuclei via microwave irradiation. Here, we describe the design and performance of a quasi-optical system enabling the use of a single 395 GHz gyrotron microwave source to simultaneously perform DNP experiments on two different 14.1 T (^{1}H 600 MHz) NMR spectrometers: one configured for magic angle spinning (MAS) solid state NMR; the other configured for solution state NMR experiments. In particular, we describe how the high power microwave beam is split, transmitted, and manipulated between the two spectrometers. A ^{13}C enhancement of 128 is achieved via the cross effect for alanine, using the nitroxide biradical AMUPol, under MAS-DNP conditions at 110K, while a ^{31}P enhancement of 160 is achieved via the Overhauser effect for triphenylphosphine using the monoradical BDPA under solution NMR conditions at room temperature. The latter result is the first demonstration of Overhauser DNP in the solution state at a field of 14.1 T (^{1}H 600 MHz). Moreover these results have been produced with large sample volumes (~100 µL, i.e. 3 mm diameter NMR tubes).

Graphical abstract