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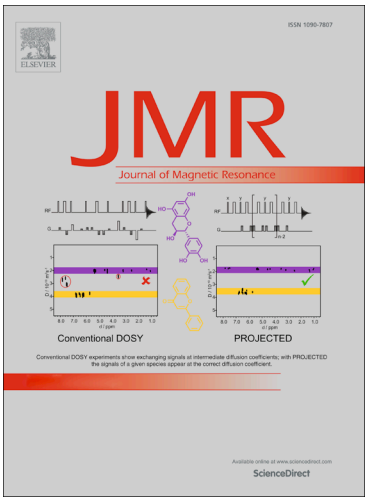

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1. Abstract

Cryogenic sample temperatures can enhance NMR sensitivity by extending spin relaxation times to improve dynamic nuclear polarization (DNP) and by increasing Boltzmann spin polarization. We have developed an efficient heat exchanger with a liquid nitrogen consumption rate of only 90 liters per day to perform magic-angle spinning (MAS) DNP experiments below 85 K. In this heat exchanger implementation, cold exhaust gas from the NMR probe is returned to the outer portion of a counterflow coil within an intermediate cooling stage to improve cooling efficiency of the spinning and variable temperature gases. The heat exchange within the counterflow coil is calculated with computational fluid dynamics to optimize the heat transfer. Experimental results using the novel counterflow heat exchanger demonstrate MAS DNP signal enhancements of 328 ± 3 at 81 ± 2 K, and 276 ± 4 at 105 ± 2 K.

Keywords: Dynamic nuclear polarization, solid-state NMR, magic-angle spinning, cryogenic MAS, heat exchanger, electron decoupling, pulsed DNP

2. Introduction

Magic-angle spinning (MAS) nuclear magnetic resonance (NMR) is a powerful technique to study the structure and dynamics of an array of molecular architectures important to biology and materials science [1–6]. However, the small magnetic moments of nuclear spins result in inherently small NMR signal intensity. Nuclear spin alignment is poor at room temperature thermodynamic equilibrium due to the relatively weak Zeeman interaction. Cooling samples can significantly boost NMR sensitivity by enabling continuous wave dynamic nuclear polarization (DNP), and increasing thermal spin polarization [7–12].

DNP boosts NMR sensitivity by using microwave energy to transfer electron spin polarization to nuclear spins[13,14]. Many contemporary MAS DNP experiments employ exogenous stable organic radicals and continuous wave DNP mechanisms [15–18]. However, continuous wave