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Multi-Sample Dissolution DNP with a Cryogen-Free Polariser

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Dissolution Dynamic Nuclear Polarisation (DNP) has shown great potential in providing large signal enhancement to metabolites of interest in low gamma metabolic magnetic resonance imaging. Originally DNP polarisers were based on pumped-helium cryostats, which provide a high cooling power to contain the extra heat load introduced during the dissolution. However, these systems are not efficient at running at low temperatures for extended periods of time; neither are they cost effective due to the rising price of helium.

We present a closed-cycle cryogen-free 7T polariser which requires no input of liquid helium or any other cryogens. The polariser is based on a modified commercial dilution refrigerator. The closed system can run continuously at 1.4K for many weeks without interruption, useful for solid state measurements of samples with prolonged longitudinal relaxation times as well as making it a highly interesting system for dissolution DNP. Liquid-state 13C polarization larger than 40% were obtained on different samples.

Traditional methods of sample dissolution are not suitable for a cryogen-free system due to the need to introduce warm helium to pressurise the sample space prior to introduce the dissolution apparatus into the cryostat. Instead, in order to minimise the heat load introduced during the dissolution process, we used a fluid path.

The sample space in the polariser is sufficiently large to house a maximum of 4 fluid paths meaning up to 4 mL of sample can be polarised at once. We implemented an insert that allows polarising two samples in parallel and dissolving them consecutively within an interval of 20min. These consecutive dissolutions can be carried out without significant deleterious heat-loads at the cooling stages of the cryostat. Limiting temperature increases on any of the cooling stages in this way allows a rapid recovery of the base temperature and prevents a potential quench of the magnet.

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