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Magn. Reson. Discuss., author comment AC1
https://doi.org/10.5194/mr-2021-12-AC1, 2021

Reply on RC1

Thomas Kress et al.

Author comment on "A novel sample handling system for dissolution dynamic nuclear polarization experiments" by Thomas Kress et al., Magn. Reson. Discuss., https://doi.org/10.5194/mr-2021-12-AC1, 2021

Dear editor,

please find our point-to-point discussion below. We have responded to all points raised by the referee (responses in italics). We hope that you will consider our manuscript for a revised submission.

Sincerely,

Dennis Kurzbach

Referee 1

The manuscript by Kress et al. entitled "A novel sample handling system for dissolution dynamic nuclear polarization experiments" entails a discussion of a new method of sample retrieving and handling technique in dissolution dynamic nuclear polarization (DNP) in which a three-fold advantages were noted over currently used dissolution method: (1) cryostat operation is uninterrupted, (2) dissolution does not require overpressurization of the sample space, (3) the use of a confined airlock minimizes freezing and blockages in case of dissolution mishaps.

Based from my experience in homebuilt dissolution DNP instrumentation assembly, dissolution mishaps (e.g. hot solvent leak into the cryostat) can result to freezing of dissolution stick, high boiloff of liquid Helium bath, and in some cases, calling the day off for experiments since the dissoltuion stick is stucked in the cryostat. This can also be problematic even for commercial systems such as hypersense in which accidental spray of superheated water or solvent into the cryostat, often requiring a visit by technical engineer for repair. The confined airlock technique presented here by the authors is a novel way to bypass these potential issues and it sounds like the low vacuum pressure in the cryostat sample space is maintained during dissolution which implies that the next DNP sample can be brought to low temperature rather relatively quickly. In light of this new development that solves current challenges in dissolution DNP, I highly recommend publication of this manuscript by Kress et al., with minor revision and suggestions:

Page 1 abstract, line 10: change "For DDNP," to "In DDNP,"

Page 1 abstract, line 15: change "Here," to "Herein,"

Page 1, line 28: change "here" to "herein"

Page 1, line 46: change "widely used" to "widely-used"

Page 2, line 1: put a comma after "To minimize the heat load"

Page 2, line 27: Change the first 3 words to "Herein, we demonstrate"

Page 5, Figure 5, lines 2: indicate the unit "t = 0 s".

We will correct this.

In addition, I have a question for the authors: is there any particular reason why the hyperpolarization was done the 1H rather than the staple 13C tracers in DDNP? This manuscript is self-sufficient and great in its current form with the 1H studies--thereby recommended for publication, but I was just wondering why 1H was measured instead of 13C spins in which majority of the DDNP metabolic imaging groups are working on.

We presented ^{1}H data, since the low-field spectrometer used for detection cannot detect ^{13}C nuclei, and hyperpolarised water can provide a valuable help in protein NMR to study folded sites (Szekely et al. J. Am. Chem. Soc. 2020, 142, 9267−9284). However, we have also recorded ^{13}C data on another spectrometer (^{13}C-labeled acetate and natural-abundance glycerol data detected at 11.8 T), which will be added to the manuscript.