

Magn. Reson. Discuss., referee comment RC2
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Comment on mr-2022-14

Benno Meier (Referee)

Referee comment on "Fine optimization of a dissolution dynamic nuclear polarization experimental setting for ^{13}C NMR of metabolic samples" by Arnab Dey et al., Magn. Reson. Discuss., <https://doi.org/10.5194/mr-2022-14-RC2>, 2022

This manuscript on "Fine optimization of a dissolution-DNP experimental setting for ^{13}C NMR of metabolic samples" represents a systematic exploration of experimental parameters used in D-DNP experiments, with the objective of achieving optimal sensitivity. The results of course depend on the instrument used to conduct the experiment.

The improvements in resolution and sensitivity upon changing solvent from D₂O to methanol, and upon accelerating the dissolution process are perhaps not too surprising, but the study has clearly been conducted in a very thorough way, is valuable for the field, and should be published.

The study is empirical and one cannot argue with the findings. I do however have a few comments regarding the presentation:

- The authors have chosen to first discuss all the parameters they optimize (section 3 Experiments and parameters), and then give the results of each optimization in another section (section 4 Results and Discussion).

This structure leads to a rather long manuscript, and so the results of the optimization should be summarised in a table, stating resolution before and after the optimisation, as well as the polarisation levels that were obtained in the liquid state, before and after optimisation. The final values of course should be stated for each of the two solvents D₂O and Methanol-d₄.

- It is well known that high-resolution spectra can be recorded using aqueous solvents if a back-pressure technique is used (see, e.g., the works by Bowen / Hilty, and Katsikis / Günther). These works should be discussed. (BTW our group has implemented a similar back-pressure technique for the bullet system.)

- Figure 1 suggests that the field during the sample transfer never drops below 0.56 T. This is probably not correct.

- In section C1 the authors write that the heat transfer coefficient is different for different solvents. Can they give numbers? Perhaps naively I would have thought that the heat capacity of the solvents plays a more important role.

- Figure 3 plots the thermal signal, and the ¹H DNP signal, as well as the ¹³C DNP signal in arbitrary units. I assume that the "thermal signal" is the ¹H thermal signal (if so, this should be stated explicitly).

In that case, the ¹H DNP signal is only 15 times larger than the thermal signal. What is the estimate for the proton background?

- Negative DNP shows larger polarization (Fig. A1 a), but the authors opted for positive DNP. Why? Was the dependence of DNP enhancement on sweep width studied previously? If so, this study should be cited explicitly.

- The results in table 2 should be rounded appropriately.

My other comments are minor language corrections.

- p1 line 13: relies on 1D instead of rely (analysis is singular).

- line 29: unparalleled instead of unparallel?

- p2 line 42: of ¹³C signal /detection/

- line 51: the references apply to dissolution-DNP, so the line should read ... such as Dissolution Dynamic ...

- line 56: "dissolution state DNP" should read dissolution-DNP

- p3 line 83: provides instead of provided

- line 97f: this sentence should be split into two

- line 101: during /the/ d-DNP experiment

- p4: Magnetic instead of Magentic

- p5. 112: identify instead of identified

- p6. l 153: /The/ PA plays /a/ central part.. A broad variety of PAs is available

- p7. l 164: the efficiency ... depend/s/

- p8. l. 211: B1a 1.2 K should probably read B1a at 1.2 K.

- p9. l. 236: in detail instead of in details.

- l. 240: pressurized instead of pressured

- p10. l263: the ... delays... contain (no s)

- l. 267: we focused on instead of in.

- l. 271: all parameters listed instead of each parameter enlisted

- l. 286: "smaller" is better than "less efficient"

- l. 295: mL with captial L, missing period at end of line

- p12 l. 332: How much is 11 % in Hertz?

- p16 line 435: the words "¹³C signal" appear twice.

- I don't understand the sentence in line 436ff. Is this a sensitivity vs resolution tradeoff?

- l. 439: what about the irregularity in TSP?

- p17 l. 449: this sentence should read "This often results in failure of signal acquisition.

