

Comment on mr-2021-12

Anonymous Referee #3

Referee 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-RC3>, 2021

In this manuscript the authors present a new design for an apparatus that could make dissolution DNP more fluent and less prone to malfunction. The operation apparatus is demonstrated in a cryogen-free magnet. The problem of the associated helium consumption during the dissolution process is clearly less crucial in a cryogen-free magnet but this does not negate the benefits of this new apparatus. The polarization of HDO water with an apparent T1 of 7 s is demonstrated. This is a challenging sample and goes to show that the authors put their apparatus into a challenging test. My comments relate to the organization of the manuscript and the overall presentation of the work. Regarding Figure 1 and the text relating to it: 1. Figure 1, The terminology blue are green magnets is doing disservice for understanding. Can't figure out which is which, only after reading the entire manuscript it becomes clear. The picture is good, I suggest to keep it and add labels and arrows to describe the various parts. Also not very clear, I would presume that the sample cup should enter the centre of the magnet but the airlock apparatus appears on the side, not sure how this is working. 2. The device is not a result and also does not belong in an introduction. This should be described as methods. The manuscript should be re-structured. 3. It is hard to understand the sequence of events that the sample undergoes from the current wording. I suggest to add more schematics of the various steps in sample handling and dissolution with the new device. Introduction: 4. Page 2, "cryogen consumption-free DNP system" should be spelled out more. Do you mean a system that does not use both liquid helium and liquid nitrogen? In the discussion, page 5 line 11, the authors talk about a helium bath. Is there or isn't there liquid helium in the system? Results and discussion 5. Figure 2, 3, and 4, and the relevant text – move to methods. The results should be started at Figure 5, page 4 line 16. 6. Results and discussion should be separated for clarity. 7. Page 4 line 21, 3 out of 15 is 20%, not 25%. 8 out of 15 is 53% not 57%. Not sure how the authors calculate percent polarization loss. 8. This sentence: "Furthermore, the hybrid system is fully compatible with recent polarization approaches capable of providing polarization levels of up to 70%, obtained using samples containing UV-induced radicals. (Pinon, Capozzi et al. 2020)" is not supported and in its present form, a bit misleading. 9. The results are very thin, it seems like the only result is a single dissolution of a single HDO sample. At least 3 HDO samples should be measured

and averaged. In addition, I would recommend that the authors provide a bit more proof. For example, other concentrations of D₂O in H₂O could be tested (less H₂O for longer T₁), and it would also be useful to see the results of hyperpolarizing other nuclei. Conclusions 10. Page 5 line 14: Could the authors be specific if their suggested apparatus could be installed on a HyperSense system? I would imagine this would be of the most interest to readers in the field. Could the authors speculate on the level of reduced liquid helium consumption in a cryogen-based DDNP device such as the HyperSense per dissolution? Experimental 11. Zero-filling and apodization parameters should be given