# Comment on mr-2021-12

Anonymous Referee #2

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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-RC2, 2021

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The authors present a potentially very interesting work where they study a specific configuration for dissolution DNP. Hyperpolarization and DDNP has attracted much attention and is an important subject in magnetic resonance. However, the authors present only spare data and an insufficient description of their system. The technical description should be further detailed. Data should be presented that not only support the claim of robustness, but thoroughly characterizes the performance of the system (polarization for more samples and conditions, sample recovery, volume, temperature, …).

P.1 L.29: The spins are not parallel or antiparallel to the magnetic field. All spins are in a superposition of the two eigenstates. Hyperpolarization is a strong increase of the population difference between the populations of the two eigenstates (or a strong net alignment of the spins with the magnetic field).

P.1 L.41: Please elaborate on the link between the heat shock and the prolonged polarization time and dissolution loss?

P.2 L.22: The sentence is written as a fact rather than speculation. Is there any evidence in literature or this work that supports better robustness of configuration 2? The authors should state the argument in principle and present data in support of their own work. What is the robustness of their implementation (1 out of 100 failures, 2, 5, 10, …)? What are the failure modes?

P.3 L.10: Can the authors provide any data on the leak rate (static and dynamic) of the described seal (mbar L/s)? Or say something about the ingression of air and its potential effect? How fast can the sample tube move?

P.3 L.19: 1.5 MPa (15 bar) and 513 K (240 C) seems very excessive compared to other polarizer systems? Please comment on the choice of dissolution conditions and what the effect would be of operation at lower temperature and pressure.

P.4 L.2: 37 mT is much less than the field generated by magnetic tunnels based on permanent magnets and the field is too low to effectively avoid T1 shortening by the nitroxide radical. Later it is stated that 25% of the polarization is lost during the approx. 1 s transfer time. Please comment on the choice of magnetic field and the effectiveness.

P.4 L.14: Fig. 4b would be easier to read if the x-axis was expanded, e.g. 0 to 300 s. The temperature increase seems quite substantial (approx. rising to 6 K). This does not seem to be a small heat load? Is this due to the slow retraction of the sample tube? How fast is it removed?

P.4 L.16: How big is the sample (mg or uL) and what is the largest size that the sample tube can contain? What is the recovery of the sample in the 600 uL in the NMR tube, i.e. how much is lost or undissolved?

P.4 L.20: Why 1.8 K? It has been stated that the polarizer operates at 1.3 K (P.2 L.28) earlier in the paper.

P.4 L.26: The relaxation rate constant is stated to be 0.14 s-1. Fig. 5 states 0.21 s-1. What is correct?

P.4 L.31: It has not been demonstrated that the polarizer is capable of polarizing UV generated radicals. These require fast cold loading not to quench. This claim should be removed unless supporting data can be provided.

Fig 1b does not seem to zoom in on the right part of the main photo?

Conclusion: It is unclear what the operating temperature of the system is (1.3 K or 1.8 K)? The heat load during dissolution does not seem insignificant. What is the heat load during sample loading and how fast can this be done? Maintaining the sample space a low temperature and avoiding to break the vacuum during sample loading and dissolution has already been demonstrated by several other systems. No data has been presented on the reliability of the system. How many samples have been dissolved without failure?