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Magn. Reson. Discuss., referee comment RC1
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# Comment on mr-2022-12

Daniella Goldfarb (Referee)

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Referee comment on "Reverse dynamic nuclear polarisation for indirect detection of nuclear spins close to unpaired electrons" by Nino Wili et al., Magn. Reson. Discuss., https://doi.org/10.5194/mr-2022-12-RC1, 2022

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This manuscript presents a new pulse sequence which offers a way to probe the nuclear polarization obtained via DNP by transferring it back to electron polarization after a step of saturation of the electron spins. The authors call this experiment reversed DNP. This is an elegant and original idea and the technique, based on the NOVEL DNP sequence, will be useful in the future to study nuclear spin diffusion and may find applications in ENDOR. The presentation is generally clear (though many experimental details are missing, see below) .The authors present a convincing series of experiments, which clearly demonstrate that their idea works and do the right control. I liked the work and I think it fits very well to MR. I recommend publication after addressing the following:

- The new method is demonstrated at Q-band frequencies (for the electrons) , ~ 50 MHz for protons, which is way below the standard NMR frequencies at which DNP is applied. This is a general problem of NOVEL. It will be nice to have some outlook regarding possibilities of using this at more relevant frequencies.
- The technique is demonstrated on trityl, which at Q-band has a rather narrow EPR signal and with the help of chirp pulses the authors managed to excite the whole spectrum. At a high field the spectrum of trityl broadens and full excitation may not be possible. What will be the consequences of partial excitation (saturation) ? please add to the discussion
- Related to point 2 – will this work for nitroxides, which are often used as polarizers for DNP?
- Many experiential details are not given, like length of pulses, delays, properties of the chirp pulses when used, details on the train of saturating pulses, their number and interval , how long it takes to acquire the data. Please give these details in the fig. captions or in the Experimental part for each figure shown in the manuscript and the SI.
- Figure 5b –it is not clear to me what was the sequence , where are inversion pulses used ? , the sequences show only saturation train.
- In the introduction – around line 20,  maybe mention that schemes different than just CW irradiation exist.
- S9 – please remove the application ruler.