

Magn. Reson. Discuss., referee comment RC2  
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## Comment on mr-2021-25

Anonymous Referee #2

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Referee comment on "Solid-state  $^1\text{H}$  spin polarimetry by  $^{13}\text{CH}_3$  nuclear magnetic resonance" by Stuart J. Elliott et al., Magn. Reson. Discuss.,  
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Elliott and coworkers are using a  $^{13}\text{C}$ -enriched sodium acetate species as a probe of  $^1\text{H}$  polarization under DNP. I am generally supportive of publication of this work as a communication of early data (after responding to a number of points/clarifications) because it will spur further studies, where the hyperpolarization interactions can be considered. The effects that the group reports are a potentially interesting finding that merits follow-up. I hope the group is pursuing sodium formate actively, since that may be an easier system to describe.

After reading the manuscript, I was left with a number of questions that I couldn't understand from my reading of the material:

- The appearance of the spectra in Figures 3a) and 3d) are not "single peaks" but rather spectra with multiple shoulders (spanning several 100 ppm). What is the origin of those? I would be curious to know if the appearance of the spectra is identical at shorter  $^1\text{H}$  DNP times in the sequence – does this line(shape) build homogeneously?
- What is the role of CSA or possible dipole-dipole interactions, and how are those manifest under both positive and negative microwave irradiation? What is the preferred energy state for coupling to  $P(^1\text{H}) = +$  versus  $P(^1\text{H}) = -$  ?
- Presumably the glycerol carbon and the quaternary carbon of the formate both contribute to the spectrum. Where are those, and how are those influenced by both cross-polarization and microwave irradiation?
- At extended  $^1\text{H}$  DNP times, there are additional intriguing details – the claim that these are now two separate resonances doesn't quite fit with the initial picture (of a "single [peak]")
- Defining Eq 1 based on the fractional intensities of these two "peaks" feels somewhat arbitrary. Without knowing what these  $I_{\text{h}}$  and  $I_{\text{l}}$  features represent, it's somewhat difficult to tell this is arising from the  $^1\text{H}$  polarization or from some other effect.
- The different slopes for Figure 5 are explained empirically (lines 36-37) but is there a physical reason why the  $^1\text{H}$  polarization (or the asymmetry of the carbon resonances) would be more sensitive to negative microwave irradiation?
- Minor point: the term "crusher" is unfamiliar to me. Do you mean "saturation" sequence or "saturating comb"?

