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Magn. Reson. Discuss., referee comment RC1
https://doi.org/10.5194/mr-2021-39-RC1, 2021


# Comment on mr-2021-39

Anonymous Referee #1

Referee comment on "Efficient polynomial analysis of magic-angle spinning sidebands and application to order parameter determination in anisotropic samples" by Günter Hempel et al., Magn. Reson. Discuss., https://doi.org/10.5194/mr-2021-39-RC1, 2021

In the presented work, the authors introduced the polynomial representation of spinning sideband (SSB) intensities due to chemical shift anisotropy (CSA) of spin one-half under magic-angle spinning. At 1D level, the exact polynomial expansion of a powder-averaged spectrum was given, and a convenient CSA estimation based on the sum and difference of the 1st-order SSB intensity was introduced. Later, the same detailed deduction was further applied to the analysis for 2D "syncMAS" spectra from the partially oriented samples with uniaxial distribution, from which orientational information can be extracted from the 2D SSB intensities.
Glycine powder sample was used to test the 1st-order polynomial approximation, and the extracted CSA was in reasonable agreement with literature values. Furthermore, the author used a pre-drawn polycarbonate (PC) to demonstrate the capability of their approach, where orientational moments up to 4th-order for multiple carbon atoms were extracted and were qualitatively in agreement with previous results based on static experiments.

Although I somewhat disagree with the authors' argument about the relation between CSA tensor and SSB intensities given by Herzfeld and Berger being too complicated to be applied, the dependence of the sum and difference of the 1st order SSB intensities upon δ and η is still very insightful. The following analysis of 2D syncMAS data truly demonstrated the benefit of the polynomial approach, where the fitting procedure is fully under control.
Overall, the manuscript is nicely written. I only have a few questions and comments:
1. Even though the samples and experimental conditions are not the major concern of the study, and the information is given somewhere in the manuscript, it is still easier to read if summarized in a dedicated section.
2. In line 487 reads "The second to last column in Table 3 is the noise-related sum squared uncertainty of 10 SSB intensities considered" and in Table 3 the 2nd last column is 10ΔI2. Does this supposed to mean 10-time the integration uncertainty of some sort of "average SSB", assuming each SSB has similar line width?
3. Following #2 in line 488 reads "Only for the C1+C2+C5 combined signal does χ2 from the best fit exceed this value significantly". From Table 3, χ2 of C3 and C4 is even bigger compared to the uncertainty, as discussed in the later section. Did I misunderstand something?

Besides, there are a few typos to be corrected:
1. Line 36, "different order are separated"
2. In Fig. 2 caption, should be "for η=1" instead of "for q=1"
3. Line 389, "the intensity of this valuable signal"
4. In eqn. 40, a missing second half parentheses after ω2.

This work should be helpful for the determination of orientational information in the anisotropic sample under MAS.