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Discussions

Interactive comment on "Surprising absence of strong homonuclear coupling at low magnetic field explored by two-field NMR spectroscopy" *by* Ivan V. Zhukov et al.

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Referee Report for the manuscript: "Surprising absence of strong homonuclear coupling at low magnetic field explored by two-field NMR spectroscopy" from I.V. Zhukov, A. S. Kiryutin, Z. Wang, M. Zachrdly, A. V. Yurkovskaya, K.L. Ivanov, F. Ferrage

1. General Comments:

This paper describes theoretically and experimentally the phenomen that the 13C polarization transfer efficiency between various labeled 13C nuclei in an amino acid after a field cycling experiment (from 14.1 T to 0.3 T and back) is reduced significantly in

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Discussion paper

the absence of 1H decoupling. This is due to hetero-nuclear 1H-13C J-coupling, which alters the strong coupling condition between different 13C sites. If proton decoupling is performed at low field (0.33 T), the 1H-13C J coupling is switched off and efficient 13C polarization transfer takes place between homo-nuclear strongly J-coupled 13C sites, where the chemical shift difference is smaller than the homo-nuclear J-coupling. This work is a nice contribution to the field of biomolecular NMR methodologies. There are, however, a number of issues that need to be resolved before I can recommend this article for publication in JMR.

2. Individual scientific comments and questions:

(a) A small confusion arises due to Figure 1, where the 13C spectrum from L-leucine measured at 100.62 MHz is shown. Later in Figure 5 the 13C spectra is presented at 150.9 MHz. The reader will ask why the13C NMR spectrum has not been measured at 150,9 MHz ?

(b) In section B in line 99 an unprecise statement is made “First, a non-equilibrium state is generated at B_HF by applying a selective pulse”. Please specify here that a selective 180° pulse is applied to Cˆ(delta2), leading to a non-equilibrium state, i.e into a population inversion (refer here to Figure 2).

(c) There is an error in the signs in Equation (1), either all signs in the Zeeman terms (- om1 I1z- om2 I2z- om3 Sz) are negative or all are positive, but mixed signs cannot be true. Furthermore Equation (1) and (10) are inconsistent with Equation (2), where all signs before I1z and Sz are negative.

(d) The exact form of om1, om2 and om3 should be stated in line 139, i.e. om1 = gam_C(1+delt2) B_HF, om2 = gam_C (1+deltgam) B_HF and om3 = gam_H B_HF.

(e) The experimental results in Figures 5B and 6B are in good agreement with the theoretical expectation only in the first 10 ms. Especially in Figure 6B the experimental data does not show the oscillating features as suggested by the theory. An explanation for this deviation would be very helpful.

3. Technical corrections, typing errors and references:

(a) In Figure 1 each panel should be labelled with (A), (B)…. (E), where (A) should be the molecule, (B) the overall 13C spectrum, (C) C′- spectrum, and so on.

(b) Caption in Figure 3 (line 156) should be (A) and (B) instead of (a) and (b). Similar in Figure 3,4,5,6 with labels (A) and (B) instead of A and B. Please be consistent with (A), (B), .. between captions and figures.

(c) I plotted the paper in black and white. Some indices, for example the “alpha, beta” symbols in Figs. 4 (A,B) can be hardly identified. The same problem in Figs.5,6 with Cˆ(gam), Cˆ(delt1), and Cˆ(delt2).

(d) A reference studying the polarization transfer form para-hydrogen onto 13C in a HA-HB-13C three spin system is: P. Türschmann, J. Colell, T. Theis, B. Blümich and S. Appelt, PCCP (2014), (DOI: 10.1039/c4cp01807a). Some aspects described there are related to the present manuscript and might be suitable as a reference.

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