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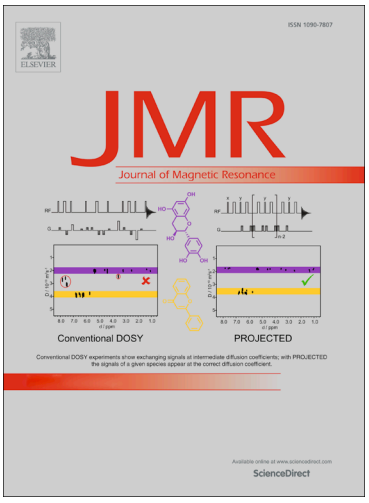

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Indirect detection of broad spectra in solid-state NMR using interleaved DANTE trains

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Abstract.

We analyze the performances and the optimization of ^{1}H-{*I*} HMQC experiments using basic and interleaved DANTE schemes for the indirect detection of nuclei I = 1/2 or 1 exhibiting wide lines dominated by chemical shift anisotropy (CSA) or quadrupole interaction, respectively. These sequences are first described using average Hamiltonian theory. Then, we analyze using numerical simulations (i) the optimal lengths of the DANTE pulse and DANTE train, (ii) the robustness of these experiments to offset, and (iii) the optimal choice of the defocusing and refocusing times for both ^{1}H-{*I*} *J*- and *D*-HMQC sequences for ^{195}Pt (I = 1/2) and ^{14}N (I = 1) nuclei subject to large CSA and quadrupole interaction, respectively. These simulations are compared to ^{1}H-{^{14}N} *D*-HMQC experiments on γ-glycine and L-histidine.HCl at B_0 = 18.8 T and MAS frequency of 62.5 kHz. The present study shows that (i) the optimal defocusing and refocusing times do not depend on the chosen DANTE scheme, (ii) the DANTE trains must be applied with the highest rf-field compatible with the probe specifications and the stability of the sample, (iii) the excitation bandwidth along the indirect dimension of HMQC sequence using DANTE trains is inversely proportional to their length, (iv) interleaved DANTE trains increase the excitation bandwidth of these sequences, and (v) dephasing under residual ^{1}H-^{1}H and ^{1}H-*I* dipolar couplings, as well as ^{14}N second-order quadrupole interaction, during the length of the DANTE scheme attenuate the transfer efficiency.

Key words. Solid-state NMR, DANTE, Broadband excitation, fast MAS, ^{14}N, very large CSA.

I. Introduction

In the last decades, the advancement of Magic-Angle Spinning (MAS) [1–4] with presently rotation frequencies up to 150 kHz, magnetic fields up to 35.2 T [5–7], and Dynamic Nuclear Polarization (DNP) at fields up to 21.1 T [8–11], has enabled a considerable improvement in the resolution and/or sensitivity of solid-state NMR spectroscopy. Furthermore, the sensitivity of this technique can be further enhanced by the transfer of polarization from abundant nuclei with high gyromagnetic ratio, such as ^{1}H, to dilute isotopes with lower ratio, such as ^{13}C or ^{15}N, using pulse sequences such as Cross-Polarization under MAS (CPMAS) [12]. The resolution of NMR spectra can be improved by the suppression of dipolar couplings with protons using homo- [13] or hetero-nuclear [14] dipolar decoupling schemes. In the case of half-integer spin quadrupolar nuclei, the sensitivity can be increased by irradiating the satellite transitions [15], whereas the resolution can be improved by removing the second-order quadrupole broadening using MQMAS or STMAS (Multiple-Quantum or Satellite Transition MAS) [16,17].