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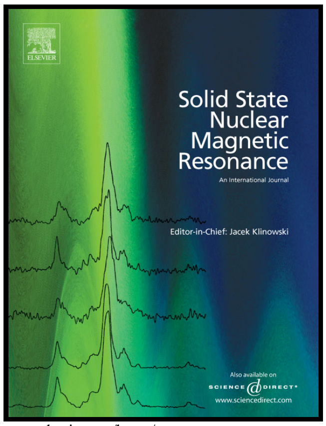

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An optimal double-magic flip angle for performing the distance measurement REDOR experiment on a spin S=1

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Abstract

Distance measurements between a half-spin and a quadrupolar S=1 spin having a small quadrupolar coupling constant can be performed using the rotational echo double resonance (REDOR) experiment. We derived an analytical expression for the probability of transitions between energy levels resulting from the application of an arbitrary pulse flip angle to the quadrupolar spin and consequently minimized the probability that populations of individual levels do not undergo a spin transition during the pulse. As a result we discovered that if the flip angle of the quadrupolar spin pulse is 109.47°, the maximal recoupling values are the largest possible and the signal reaches a maximum value of 8/9, larger than in the use of either a 90° pulse or a 180° pulse. In addition, the slope of the initial decay is higher than the 90° pulse. The recoupling signal can be modeled by an exact analytical formula in the ideal case and simulations show that the advantage of the 109.47° pulse is preserved when the quadrupolar coupling constant C_Q has a finite value typical of ^{2}H and ^{6}Li spins (up to C_Q~200 kHz). Experimental results on two spin pairs, ^{2}H-^{13}C and ^{6}Li-^{13}C, demonstrate the validity and accuracy of this method.

1. Introduction

The rotational echo double resonance (REDOR) sequence[1] is one of the primary methods for measuring the heteronuclear dipolar interaction between a pair of atoms. REDOR was initially developed to measure distances between isolated spin-1/2 pairs; however, it can easily be utilized to measure distances to nuclei with spin quantum values larger than one half when the nuclear quadrupolar coupling constant C_Q is not large[2]. As this value increases, or when other interactions such as homonuclear dipolar couplings and chemical shift anisotropies are large, REDOR becomes less efficient or more sensitive to spinning frequency fluctuations and a distance cannot be easily extracted. Therefore, new sequences based on a similar approach were developed that utilize adiabatic transfers, relaxation induced transitions and symmetry-based dipolar recoupling to obtain recoupling signals that report on the inter-nuclear distance[2–9].

During REDOR, two pulses are applied every rotor period thereby selectively recoupling the dipolar interaction. In the REDOR sequence shown in Fig. 1 the implementation is such that only a single pulse is applied to the coupled spin while other pulses are applied to the detected spin. Theoretically, this scheme is identical to the original implementation where pulses are alternating between the two channels. Practically, this approach is preferable for coupled spins that have a large anisotropy or have a spin > 1/2 since then only a single pulse is applied