Characterizing intermolecular multiple-quantum coherence signals between spin-1/2 and spin-3/2 nuclei

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Introduction

Quadrupolar spin probes are becoming increasingly important in a wide range of applications. Among these probes, the spin-3/2 nuclei constitute an important class [1,2]. Intermolecular multiple-quantum coherence (iMQC) techniques involving spin-3/2 nuclei may find new applications. In this abstract, the heteronuclear ^{23}Na-^{1}H spin system in liquid state was taken as an example to study the properties of iMQC signals between spin-3/2 (^{23}Na) and spin-1/2 (^{1}H) nuclei in heteronuclear CRAZED experiments.

Theory and Experiments

The pulse sequence is shown in Fig. 1. The dependences of iMQC signal intensity on the RF pulse flip angles are deduced as follows:

$$M_{obs} \propto \begin{cases} \cos\alpha\sin\beta\sin\theta\sin\varphi & (+\text{iSQCs with } G_1 : G_2 = \gamma_I : \gamma_S) \\ \cos\alpha\sin\beta\sin\theta\sin\varphi & (-\text{iSQCs with } G_1 : G_2 = -\gamma_I : \gamma_S) \\ \sin\alpha\sin\beta\sin^2(\theta/2)\sin\varphi & (+\text{iDQCs with } G_1 : G_2 = \gamma_I : (\gamma_S + \gamma_I)) \\ \sin\alpha\sin\beta\cos^2(\theta/2)\sin\varphi & (-\text{iDQCs with } G_1 : G_2 = -\gamma_I : (\gamma_S + \gamma_I)) \\ \sin\alpha\sin\beta\cos^2(\theta/2)\sin\varphi & (-\text{iZQCs with } G_1 : G_2 = \gamma_I : (\gamma_S - \gamma_I)) \\ \sin\alpha\sin\beta\sin^2(\theta/2)\sin\varphi & (+\text{iZQCs with } G_1 : G_2 = -\gamma_I : (\gamma_S + \gamma_I)) \end{cases}$$

where γ_I and γ_S are the gyromagnetic ratios of I and S spins, respectively. All experiments were performed on a Varian NMR system 500 MHz spectrometer using a 5 mm indirect detection probe equipped with 3D gradient coils. A sample of saturated NaCl water solution in D_2O was used. To remove the residual unwanted signals due to longitudinal relaxation, two-step phase cycling schemes were used. To verify the purity of resulting iMQC signals, 2D iMQC spectra were acquired. The frequency offsets of ^{1}H and ^{23}Na spins were set to 100 Hz and 250 Hz, respectively.

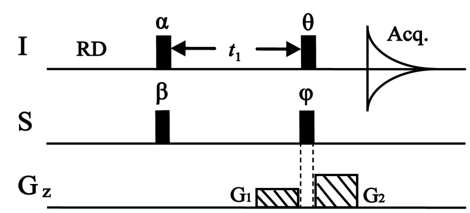

Fig.1 Heteronuclear CRAZED pulse sequence for investigating different coherence order iMQC signals between spin-1/2 and spin-3/2 nuclei.

Results and Discussion

The variations of ^{23}Na iMQC signal intensities with the RF pulse flip angle θ are presented in Fig. 2, together with normalized theoretical curves. When θ was varied, the RF pulse flip angles α, β and φ were kept at their theoretically optimal values, and vice versa. It can be seen that the optimal θ angles for positive intermolecular double- and zero-quantum coherence (+iDQC and +iZQC) signals are both π, while the optimal θ angles are both 0 for -iDQCs and -iZQCs. For intermolecular single-quantum coherence (±iSQC) signals, the optimal θ angles are both π/2. The experimental results of the dependences of ^{23}Na iMQC intensities on α, β, and φ angles are also in accordance with the theoretical predictions (data not shown). The 2D iMQC spectra (Fig. 3) confirm that the signals are indeed from the iMQCs. The variations of ^{1}H iMQC signal intensities with the RF pulse flip angles show the same regularities.

In spin systems with spin-1/2 and spin-3/2 nuclei, all the experimental results indicate that the heteronuclear CRAZED experiment allows coherence transfer from spin-3/2 nuclei to spin-1/2 nuclei, and vice verse. No matter which spin is detected, the dependences of the iMQC signal intensities on the RF pulse flip angles follow the same rules. These results are identical to those for other heteronuclear systems [3,4], implying that heteronuclear iMQCs have same properties in liquid NMR.

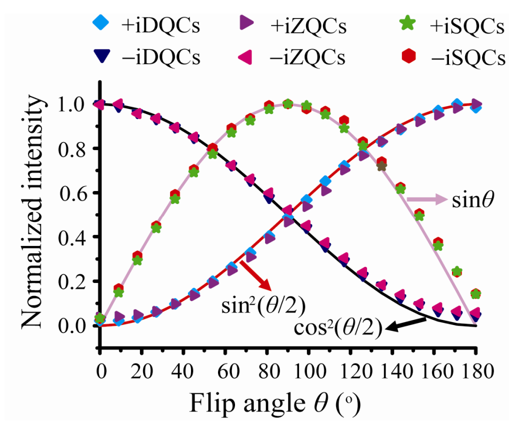

Fig. 2 Variations of ^{23}Na heteronuclear iMQC signal intensities with RF pulse flip angle θ.

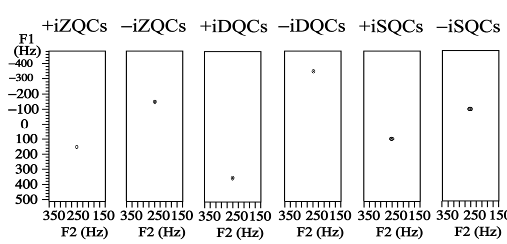

Fig. 3 2D ^{23}Na heteronuclear iMQC spectra with optimal RF pulse flip angles.

Acknowledgment

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References

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