Ultrafast high-resolution *J*-resolved spectroscopy in inhomogeneous fields

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Introduction

Recently, Pelupessy and co-workers proposed a spatial encoding method to obtain high-resolution NMR spectra from inhomogeneous magneitc fields [1]. The pity is that uncoupled spins (singlets) cannot be detected and chemical shift information cannot be directly obtained from the resulting spectra. In this abstract, we proposed a new excitation scheme for high-resolution 2D *J*-resolved spectra in the presence of B_0 heterogeneities, which combines spatial encoding with intermolecular zero-quantum coherence (iZQCs) and does not suffer from the forementioned problems.

Methods

The pulse sequence is shown in Fig. 1. The spatially encoded block (composed of a chirped π pulse and a gradient), which plays an important role of monitoring spin evolution in an indirect fashion, is inserted into the IDEAL sequence [2]. The second π/2 RF pulse is solvent selective. The gradients in the detection period are used to decode iZQC signals. The *J*-modulated technique is used in the detection period [3].

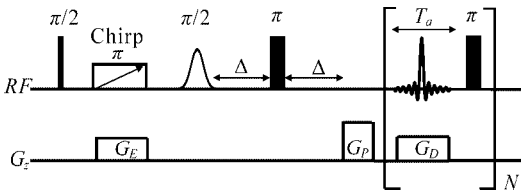

Fig. 1 Pulse sequence for high-resolution 2D *J*-resolved spectra based on iZQCs.

Experiments were performed at 298 K using a Varian NMR System 500 MHz spectrometer, equipped with a 5 mm indirect detection probe with three-dimensional (3D) gradient coils. A sample of a solution of propanol in dimethyl sulfoxide (DMSO) was used to verify the proposed sequence. To test the feasibility of the proposed sequence in inhomogeneous fields, different shimming coils were intentionally deshimmed to produce a linewidth of 2000 Hz. N = 64 ($t_2^{\max} = 462$ ms), and a two-step phase cycling is used. All the acquired data were processed in the same way: the time-domain data points were first separated into two independent bidimensional data sets related to odd and even G_D periods respectively. These two data sets were then experienced inverse Fourier transform (IFT). Finally, they were subjected to 2D FT, and the two resulting spectra were co-added into a single spectrum.

Results and Discussion

The experimental results are presented in Fig. 2. Hardly any spectral information can be obtained from the conventional single-quantum coherence (SQC) spectrum in the inhomogeneous field (Fig. 2b). In the two-dimensional (2D) spectrum obtained from the proposed sequence (Fig. 2c), the chemical shift and *J*-coupling information are separated along the F1 and F2 dimensions respectively, *i.e.* the 2D spectrum is a *J*-resolved spectrum, as we expect. Since all the peaks have a frequency shift of ω_I (frequency offset of solvent spin) in the F1 dimension, we can set the spectrometer reference frequency to ω_I, *i.e.* $\omega_I = 0$, then the absolute chemical shifts can be directly displayed in the F1 dimension. In inhomogeneous field, the resulting spectrum still has good resultion (Fig. 2d). The accumulated projection onto the F1 dimension gives a linewidth of 33 Hz (data not shown), much smaller than the linewidth (2000 Hz) of conventional one-dimensional (1D) SQC spectrum obtained from the same magnetic field. This example highlights the efficiency of present sequence for high-resolution 2D *J*-resolved spectra in inhomogeneous fields.

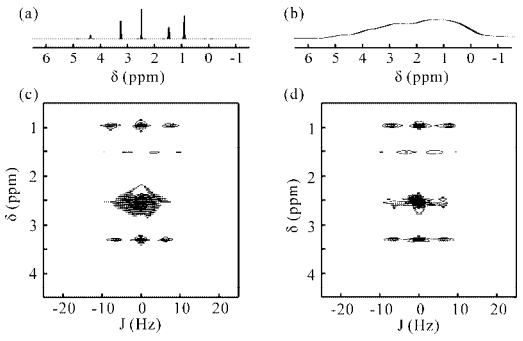

Fig. 2 NMR spectra of propanol in DMSO. (a,b) Conventional 1D SQC spectrum in homogeneous and inhomogeneous fields respectively; (c,d) 2D iZQC *J*-resolved spectra in homogeneous and inhomogeneous fields respectively.

Intermolecular multiple-quantum coherence is attractive for high-resolution NMR spectroscopy in inhomogeneous fields. However, 2D or 3D acquisition is often required for 1D or 2D high-resolution spectra, which results in much longer experimental time than 1D experiments since the indirect dimension evolution is monitored *via* a stepwise increment of time parameter and a collection of hundreds of independent transients in each indirect dimension is needed to obtain a multi-dimensional (mD) spectrum with well resolution. The present sequence pave a way to speed up mD iMQC experiments.

Acknowledgment

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References

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