Demonstration of 2D NMR Spectroscopy in a Powered Magnet at 25 T

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Introduction

Two-dimensional NMR (2D NMR) experiments require the temporal stability and spatial homogeneity afforded by persistent mode magnets. The majority of 2D NMR experiments have previously been infeasible in powered magnets because of large temporal magnetic field fluctuations and poor spatial homogeneity. Leveraging recent improvements in field regulation and passive shims, we demonstrate the well-known TOCSY (total correlation spectroscopy) experiment using the Keck powered magnet operating at 25 T.

Experimental

The field regulation system combines inductive and NMR measurements of field fluctuations to attenuate high- and low-frequency field fluctuations, respectively. Field regulation reduces the 60 Hz component by 64 dB, from 160 mG rms (0.640 ppm rms) to 0.1mG rms (0.0004 ppm) and the low-frequency fluctuations under 20 Hz by 43 dB from 1.43 G rms (5.72 ppm rms) to 9.84 mG rms (0.0394 ppm rms). Passive shims correct the field homogeneity from about 90 ppm to within 10 ppm over a 1 cm volume. Spinning the sample at a rate of about 4000 revolutions per second further reduces the linewidth to 50 ppm. The TOCSY sequence is MLEV-17 with B_1 about 5 kHz and a mixing time of 4 ms.

Results and Discussion

The 2D TOCSY sequence correlates spins with scalar J-coupling. The acquisition of the 2D experiment requires a constant magnetic field. Any field fluctuation and drift would cause misalignment of the peaks in the 1D spectra and ridges of t1-noise along the F1-dimension after the 2D Fourier transform. Thus, performing an 2D experiment is the ultimate test of field regulation. Figure 1 shows the first 2D TOCSY spectrum acquired on the 25T Keck powered magnet. The result clearly shows both the diagonal and the cross peaks arising from the $^1H/^1H$ J-coupling between the CH_3 and CH_2 peaks in ethanol. Ridges of t1-noise arise from the residual field fluctuations remaining after field regulation.

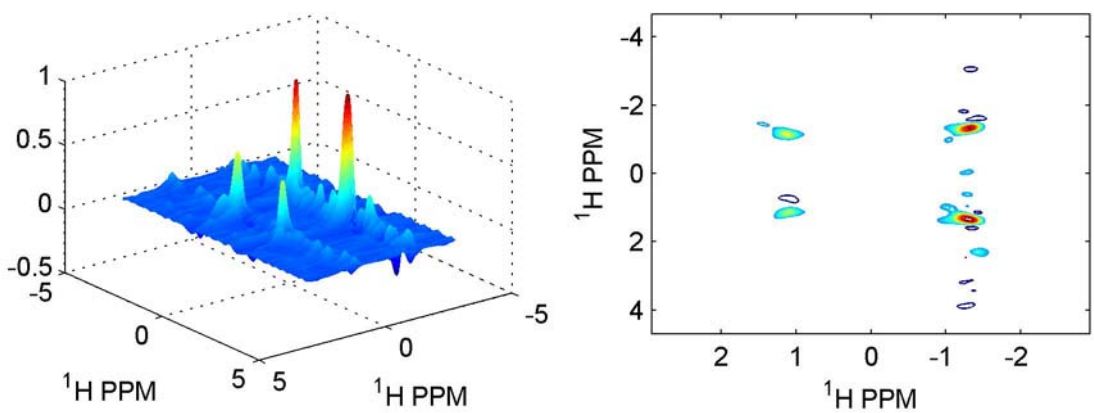

Figure 1. TOCSY 2D spectrum of ethanol acquired at 25 T in the Keck powered magnet with field regulation

Conclusions

To our knowledge, this is the first time that off-diagonal peaks have been resolved during a 2D NMR experiment on a powered magnet. This work supports the development of the 36 T series-connected hybrid magnet scheduled for completion in 2015.

Acknowledgements

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