Development and Implementation of the State-of-the-Art Solid-State NMR Pulse Techniques 1) at Ultrahigh Magnetic Fields and 2) for Utilizing ^{14}N Overtone Transition

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Introduction

We have developed and implemented solid-state NMR pulse techniques for obtaining ^{2}H spectra that can be utilized at ultrahigh magnetic fields and spinning speeds. We explored an efficient ^{1}H-^{2}H cross-polarization (CP) scheme under an ultrafast magic-angle-spinning (MAS) condition, employing an adiabatic pulse scheme along the X-channel.

Experimental

The figure shown below demonstrates the outcome of this method under spinning rates of ν_r = 40 and 60 kHz, utilizing an adiabatic inversion pulse[1,2] block along the ^{2}H channel while employing a conventional rectangular pulse block along the ^{1}H channel. We tested the performance of this CP method, BRoadband Adiabatic INversion CP (BRAIN-CP),[3,4] on powdered 2,2-d_2-Gly. Also shown for comparison are results obtained by the conventional Hartman-Hahn CP (HH-CP) method and directly excited ^{2}H solid-echo spectrum at each MAS spinning rate. A directly polarized static ^{2}H spectrum is also obtained and compared in the figure. Each measurement mode was obtained based on an iterative rf power optimization.

Results and Discussion

As can be seen in the figure the BRAIN-CP profile obtained by the adiabatic pulse scheme exceeds the HH-CP method in terms of the efficiency at higher spinning rate (ν_r = 60 kHz). At ν_r = 40 kHz both CP modes are comparable in terms of CP efficiency and they are still better than the directly excited ^{2}H spectrum. One noticeable feature is that the efficiency of the BRAIN-CP method exceeds the other cases as the spinning rare increases as can be seen in the figure.

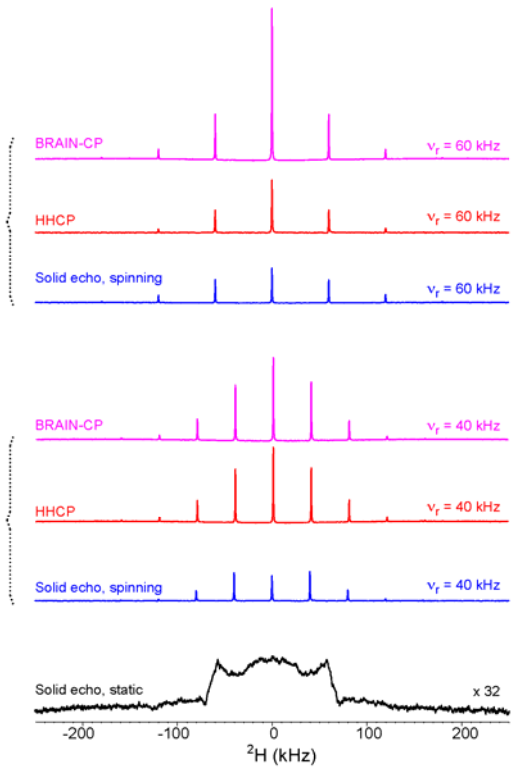

Fig.1 ^{1}H-^{2}H CP spectra measured by HH- and BRAIN-CP methods measured at ν_r = 40 and 60 kHzs. Each CP spectrum measured at each spinning rate is compared to the directly polarized solid echo spectrum. Solid echo spectrum measured at the static case is also included at the bottom for an overall comparison.

Conclusions

An adiabatic inversion scheme along the ^{2}H-channel in a standard CP scheme is a favorable option for obtaining a ^{2}H spectral efficiently under an ultrahigh spinning rate compared to either a direct polarization or a conventional HH-CP method.

Acknowledgements

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References

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