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

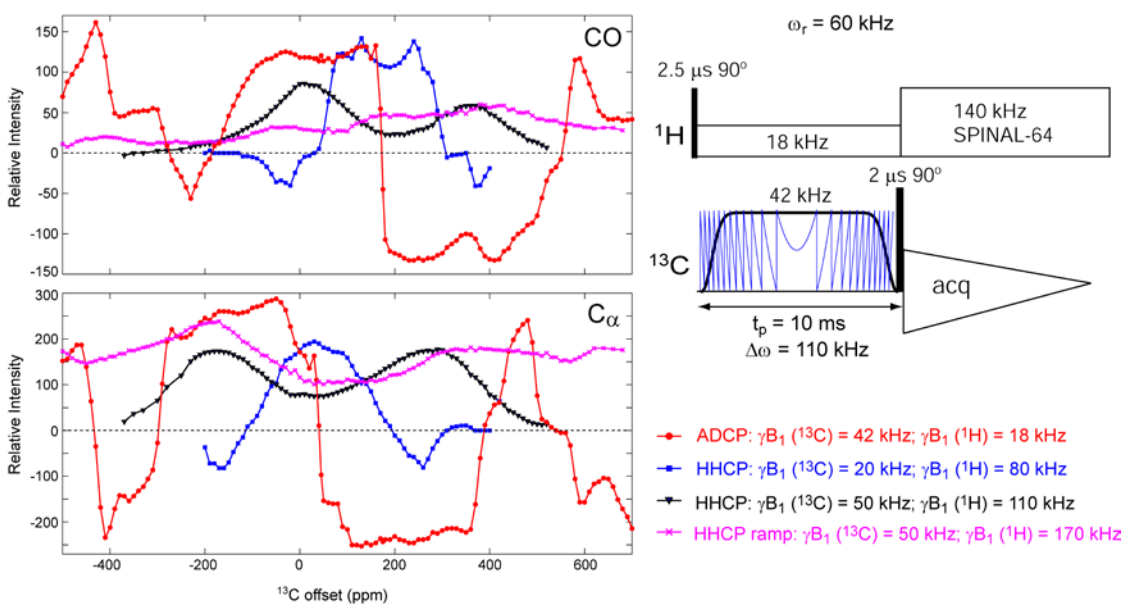
One of our proposed research activities was to develop and implement solid-state NMR pulse techniques that can be utilized at ultrahigh magnetic fields and spinning speeds. Uniform, efficient pulse excitation, inversion, and cross-polarization involving a X-nucleus spanning over wide spectral frequency ranges are extremely difficult to achieve at an ultrahigh magnetic field because the spectral dispersion of these nuclei becomes extremely wide. We explored an efficient ^{1}H-X 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 a spinning speed, ω_r = 60 kHz, utilizing an adiabatic inversion pulse block, Wurst-40, along the ^{13}C channel replacing the conventional spin-lock pulse block. The pulse power and duration of each block in the CP sequence is specified in the figure. We tested the performance of this novel CP method on powdered 1-^{13}C Ala (red). Also shown for comparison are results obtained by the conventional CP methods based on the Hartman-Hahn CP (HHCP) method.

Results and Discussion

Blue and black symbols and lines are results obtained by HHCP at 20 and 50 kHz of ^{13}C pulse power ($\omega_1[^1\text{H}] = \omega_1[^{13}\text{C}] + \omega_r$), respectively, and black are HHCP results obtained at 50 kHz ^{13}C power, with ramped ^{1}H spin-lock [ω_1 = 170 kHz, 90-110%]). All HHCP results were obtained without the 90° read pulse along the ^{13}C channel that is required when an adiabatic inversion scheme is employed. As can be seen in the figure the CP profile obtained by the adiabatic pulse scheme excels the HHCP methods not only in terms of the efficiency but by the width, which is obtained by changing the offset frequency of ^{13}C. One noticeable feature is the presence of null points at the on-resonance and ± ω_r positions. We are currently developing a theoretical scheme explaining this feature numerically and analytically.



Conclusions

An adiabatic inversion scheme along the X-channel under a ultrahigh spinning condition is a viable option for an efficient ^{1}H-X CP that covers a wide spectral frequency range with improved sensitivity.

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