High-Resolution Magic-Angle Spinning NMR Using 36 T SCH for Bio-Solids

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Introduction

The 36 T Series-Connected-Hybrid (SCH) magnet offers a 50% increase in field strength over the highest superconducting NMR magnet available today. This report presents initial results of high-resolution magic-angle spinning (MAS) NMR at this high field using nano-crystalline GB1 and membrane ChiZ protein samples. Their comparisons with spectra acquired at an 800 MHz superconducting magnet show the promises as well as challenges of high-field and high-resolution NMR for bio-solids with sub-ppm resolution using the SCH.

Experimental

Experiments at 1500 MHz were performed on the 36 T SCH magnet operating at 35.2 T with a Bruker Avance NEO console and modified lock board. A triple-resonance Low-E MAS probe designed and built at the NHMFL using a 2.0 mm Revolution NMR stator was used, which also has an external ^{7}Li lock sample and circuit. Experiments at 800 MHz (18.8 T) were performed using a Bruker Avance III HD console and a 3.2 mm Low-E MAS probe designed and built at the NHMFL.

Results and Discussion

Field regulation using a customized field-frequency lock enables the acquisition of 2D spectra for the first time with a powered magnet at 35.2 T and sub-ppm resolution [1]. The ^{13}C-^{13}C DARR correlation spectrum in Fig. 1a show comparable line width at 35.2T for GB1 and for some peaks better resolution than at 18.8 T. The contour levels just above the thermal noise show t_1-noise for the sharp and intense methyl and solvent peaks due to the residual field fluctuations. The ^{15}N dimension in the NCA spectrum in Fig. 1b also shows some additional line broadening. When the intrinsic line width gets broader than 1 ppm, such as for the membrane protein ChiZ in Fig. 1c, the effects from the residual field fluctuations become negligible. The improvement in resolution is evident mostly because of the reduction of the homogeneous linewidth in ppm at the higher field. The higher magnetic field of 35.2 T requires faster MAS spinning frequencies and consequently restricts usage to small rotors. It is noteworthy that the sample volume of the 2 mm MAS probe is less than 1/3 of that the 3.2 mm thin-wall MAS probe used at 18.8 T. The F_2 projections show similar S/N between the two spectra acquired under approximately the same total acquisition time.

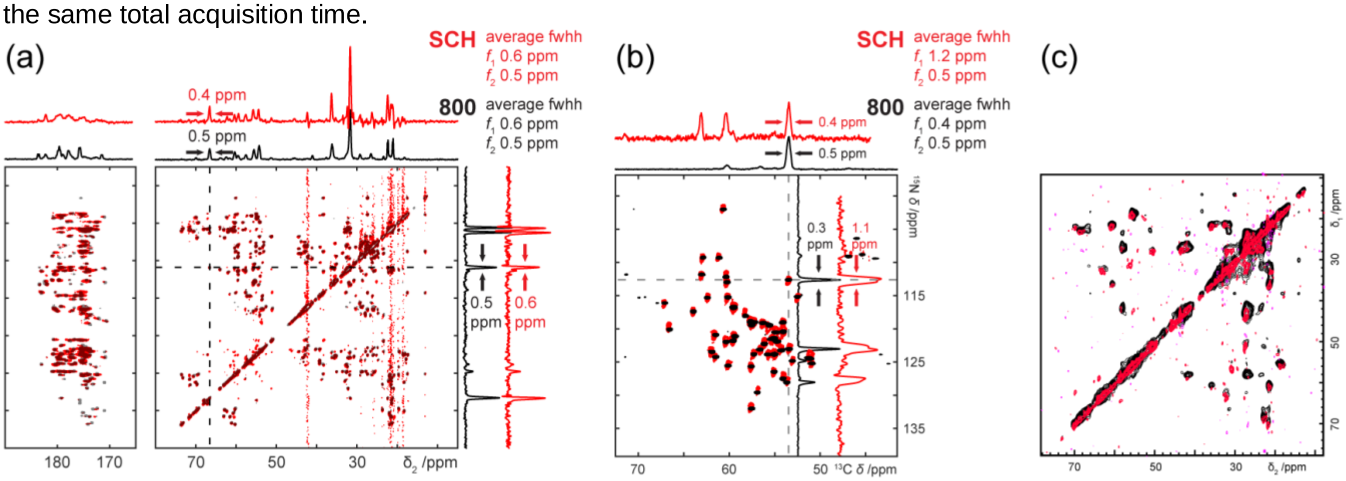

Fig. 1 a) ^{13}C/^{13}C DARR, b) ^{15}N/^{13}C NCA correlation spectra of GB1 and c) ^{13}C/^{13}C DARR of ChiZ acquired at 35.2T (red) and 18.8T (black).

Acknowledgements

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References

[1] Gan, Z., *et al.,* J. Magn. Reson., **284**, 125-136 (2017).