Solid-State MAS NMR Characterization of Two Tripeptides

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Introduction

Weak hydrogen bonds, such as those in the form C-H…O=C, have been of great interest in the solid-state chemistry community. Several years ago, we identified two tripeptides with unusually short hydrogen bond contacts of this type. We have been engaged in an investigation of these two peptides, Ala-Gly-Gly and Ala-Ala-Gly, to further understand their structure. We have employed solid-state NMR and diffraction as the main experimental tools. The NMR and crystallographic signatures of the central hydrogen is the focus of our attention. We have obtained high-resolution neutron diffraction structures for these two compounds. With our collaboration at the NHMFL, we attempted to measure a solid-state MAS ^{1}H NMR spectra.

Experimental

Polycrystalline samples of the tripeptides Ala-Gly-Gly and Ala-Ala-Gly were prepared by slow solvent evaporation, and their polymorphs were authenticated by powder X-ray diffraction. One-dimensional ^{1}H and ^{13}C and two-dimensional ^{1}H-^{13}C HETCOR and ^{1}H-^{1}H SQ-DQ NMR spectra were acquired at the NHMFL using a 14.1 T NMR system equipped with a 600 Bruker Avance spectrometer and Bruker 1.3 mm MAS probe spinning at 60 kHz MAS.

Results and Discussion

Figure 1 below shows the ^{1}H-^{1}H SQ-DQ spectrum for the tripeptide Ala-Ala-Gly. In the coming months, we will bring together the NMR data with diffraction data, along with high-level ab initio calculations to understand fully the nature of the packing in these two special tripeptides.

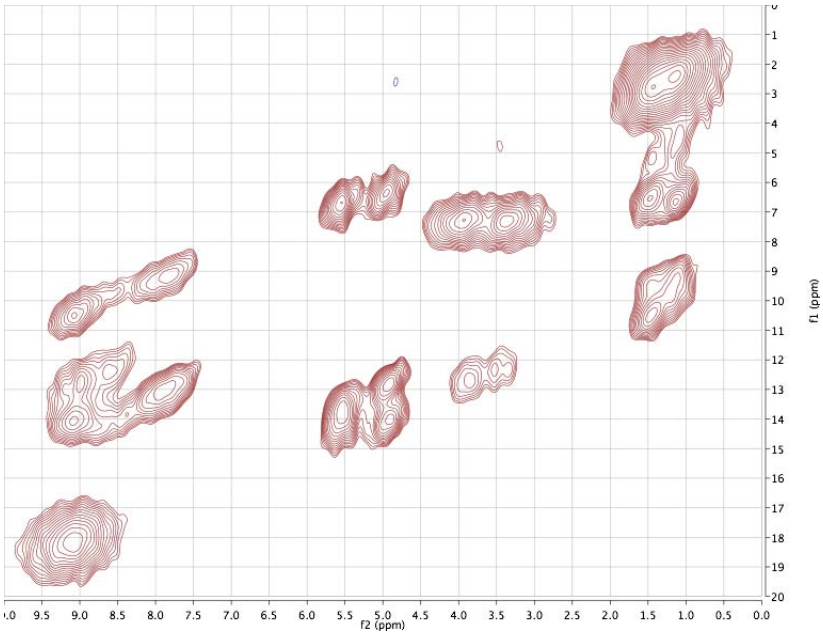

Figure 1. Solid-state ^{1}H SQ-DQ NMR spectrum of the tripeptide Ala-Ala-Gly at 60 kHz MAS. The spectrum was collected at 600 MHz (^{1}H) field strength. The horizontal and vertical axes show the single-quantum and double-quantum (DQ) dimensions, respectively. The DQ correlations among the proximal protons are clearly evident.

Acknowledgements

This project is funded by NSF-RUI grant (CHE-1012813).