

Supporting Information

3D $^{15}\text{N}/^{15}\text{N}/^1\text{H}$ Chemical Shift Correlation Experiment Utilizing an RFDR-based $^1\text{H}/^1\text{H}$ Mixing Period at 100 kHz MAS

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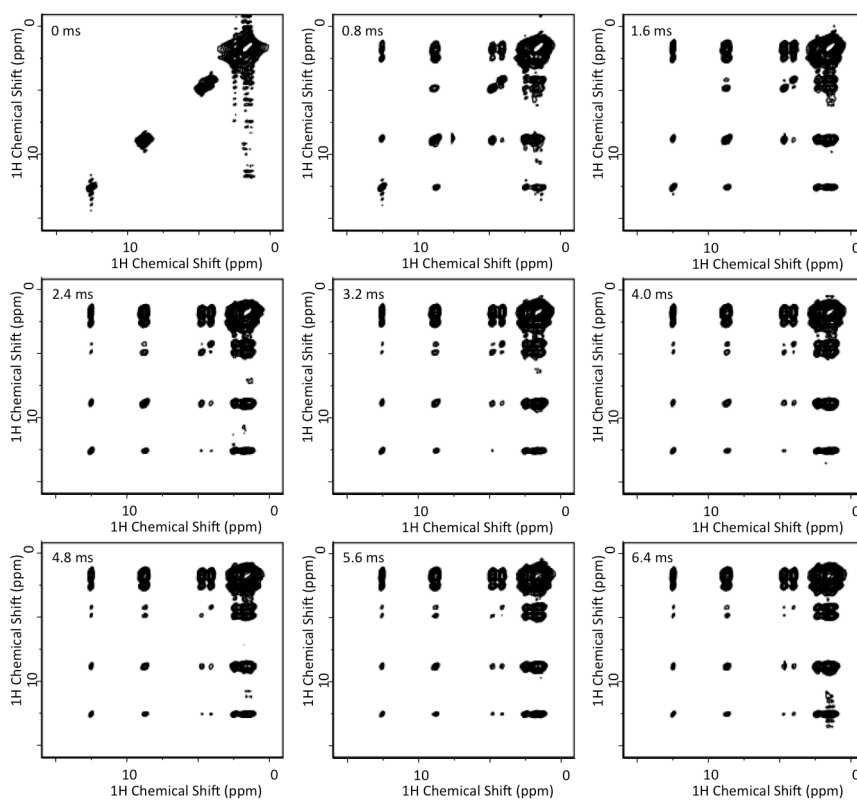


Figure S1. 2D $^1\text{H}/^1\text{H}$ chemical shift correlation spectra of NAVL powder sample obtained using the pulse sequence given in Figure 1(A) at the indicated RFDR mixing times; all other conditions are as explained in the main text.

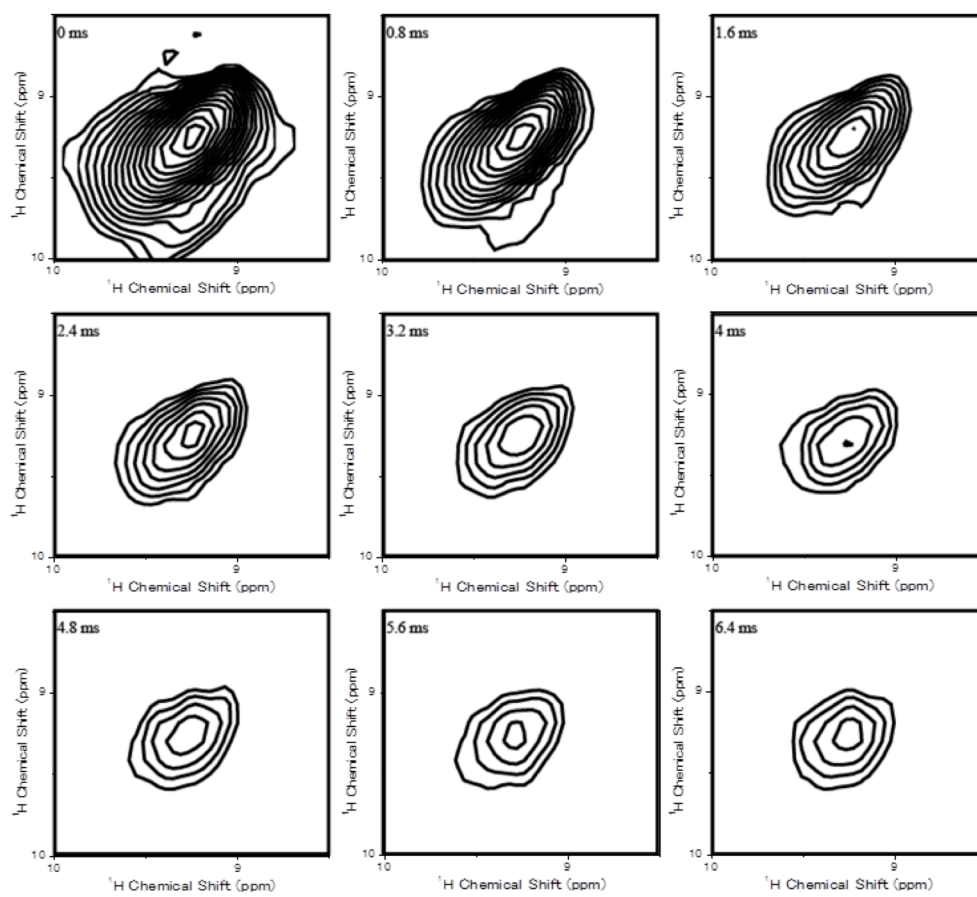


Figure S2. Expanded amide-¹H chemical shift region of 2D spectra given in Figure S1.

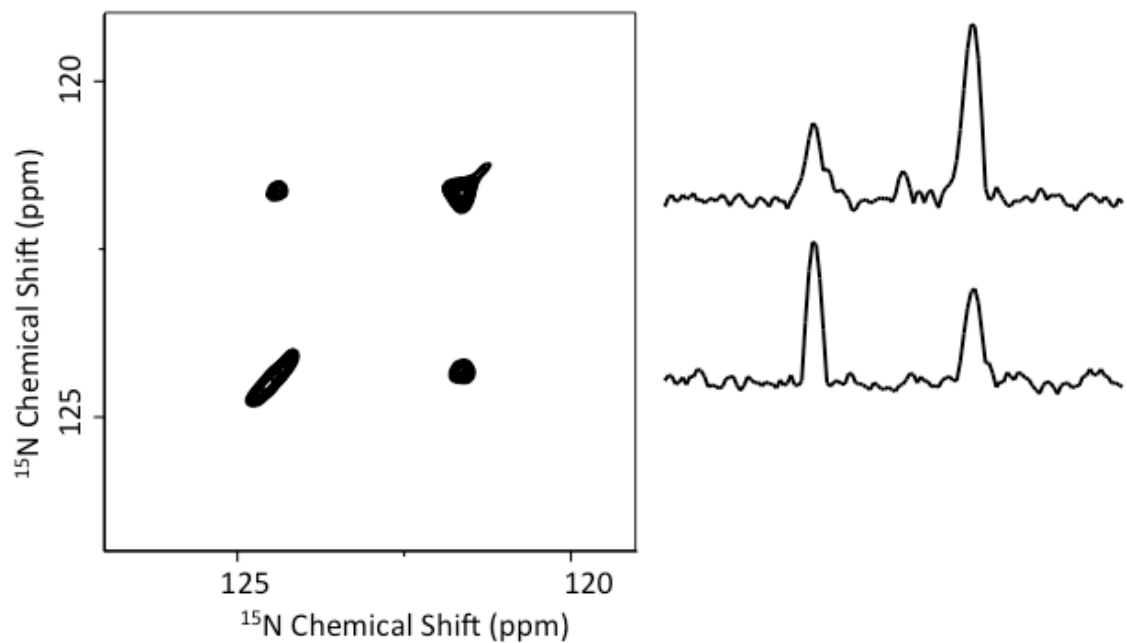


Figure S3. Proton-detected 2D $^{15}\text{N}/^{15}\text{N}$ chemical shift correlation spectrum of NAVL powder sample obtained with a 3.2 ms RFDR mixing in the ^1H channel using the 3D $^{15}\text{N}/^{15}\text{N}/^1\text{H}$ pulse sequence given in Figure 1(C) and XY4^{1_4} phase cycling at 70 kHz MAS. 16 t_1 and 16 t_2 points were observed with a recycle delay of 2 s. The measurement time was 4.6 hour. The 2D spectrum is a project of the 3D $^{15}\text{N}/^{15}\text{N}/^1\text{H}$ spectrum on to the $^{15}\text{N}/^{15}\text{N}$ plane. 1D spectral slices extracted from the 2D $^{15}\text{N}/^{15}\text{N}$ spectrum are shown (right). A contact time of 1 ms was used for all the CP transfers.