Supporting Information

Solid state NMR study of amyloid nanocrystals and fibrils formed by the peptide GNNQQNY from yeast prion protein Sup35p

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Supporting information:

- A selection of 2D spectra used for assigning the orthorhombic GNNQQNY crystal resonances.
- Assignment data illustrating the assignment of each of the three forms in a GNNQQNY fibril sample.

2D assignment data for orthorhombic GNNQQNY nanocrystals

Figure 1 shows a subset of the 2D data used to assign the GNNQQNY orthorhombic nanocrystal resonances. The measurements were performed at 700 MHz ^{1}H frequency using isotopically dilute samples consisting of [U-^{13}C,^{15}N-GNNQ]QNY (left) and GNN[U-^{13}C,^{15}N-QQNY] (right). ^{13}C-^{13}C correlations on [U-^{13}C,^{15}N-GNNQ]QNY were obtained via 1.6 ms RFDR mixing (using 50 kHz ^{13}C power) at 15 kHz MAS. Broadband ^{13}C-^{13}C correlations on GNN[U-^{13}C,^{15}N-QQNY] were determined using 8ms of homonuclear ^{13}C-(^{1}H)-^{13}C TSAR-recoupling with the center frequency at 120.548 ppm and ^{13}C and ^{1}H powers of 42 kHz and 45 kHz respectively (Lewandowski, J. R., De Paëpe, G., Griffin, R. G. *J. Am. Chem. Soc.* **2007,** 129(4): 7-8). ^{13}C-^{15}N correlations were determined using NCO, NCOCX, NCA, and NCACX experiments analogous to those performed on the fibril samples, as described in the manuscript.

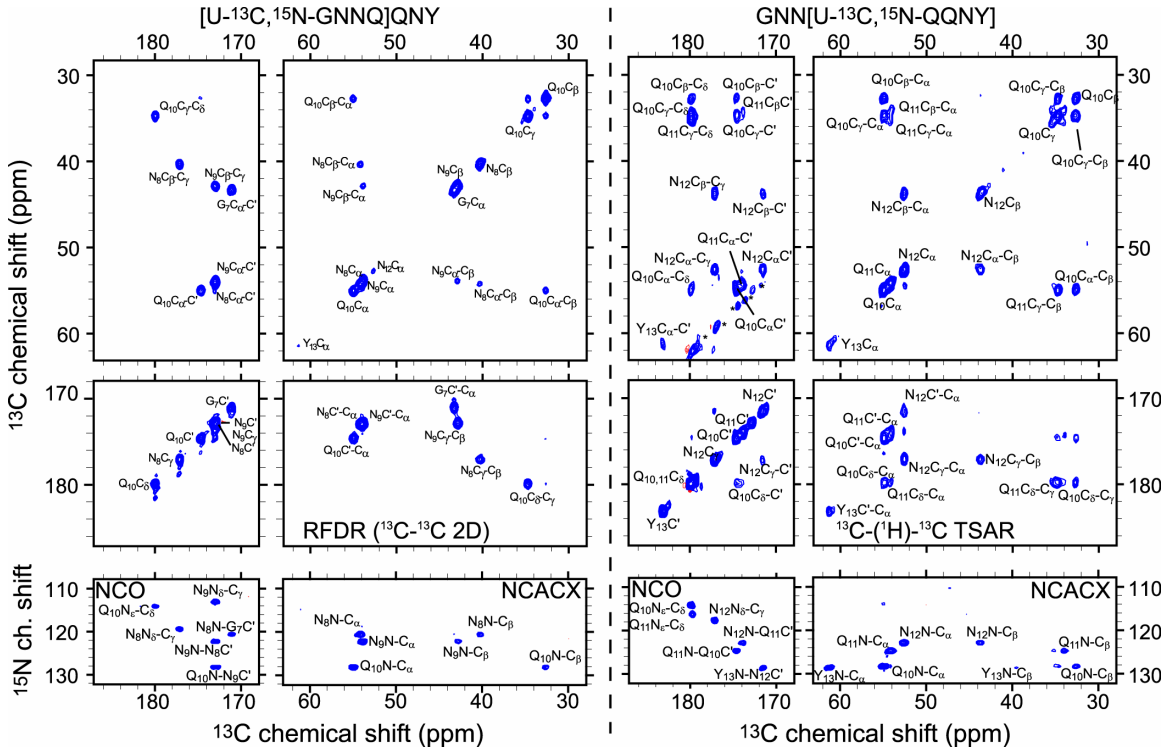

Figure 1: 2D assignment data for orthorhombic nanocrystals of GNNQQNNY. Spinning side bands are indicated with (*).

Fibril assignment data

The figures below show representative segments from our 2D assignment data on 100% labeled [U-^{13}C,^{15}N-GNNQ]QNY fibrils, obtained at 700 MHz. The assignments for each fibril form are indicated and traced out (solid lines indicate backbone correlations, dashed lines are side-chain correlations), with color-coding according to residue number. The top four panels are part of a ^{13}C-^{13}C correlation using 12 ms DARR/RAD mixing at 15 kHz MAS. The NCOCX and NCACX experiments were performed using 10ms ^{13}C-^{13}C DARR/RAD mixing, following the DCP N-C transfer, at 15 kHz MAS. Solid lines indicate backbone-backbone correlations, whereas dashed lines involve side chain-side chain, or backbone-side chain correlations. Very similar data were obtained for a number of other samples, which were prepared using isotopically dilute peptide material. Each of the fibril forms displays a self-consistent correlation pattern, while no cross-peaks are seen connecting the different forms to each other (even with longer DARR/RAD mixing times).

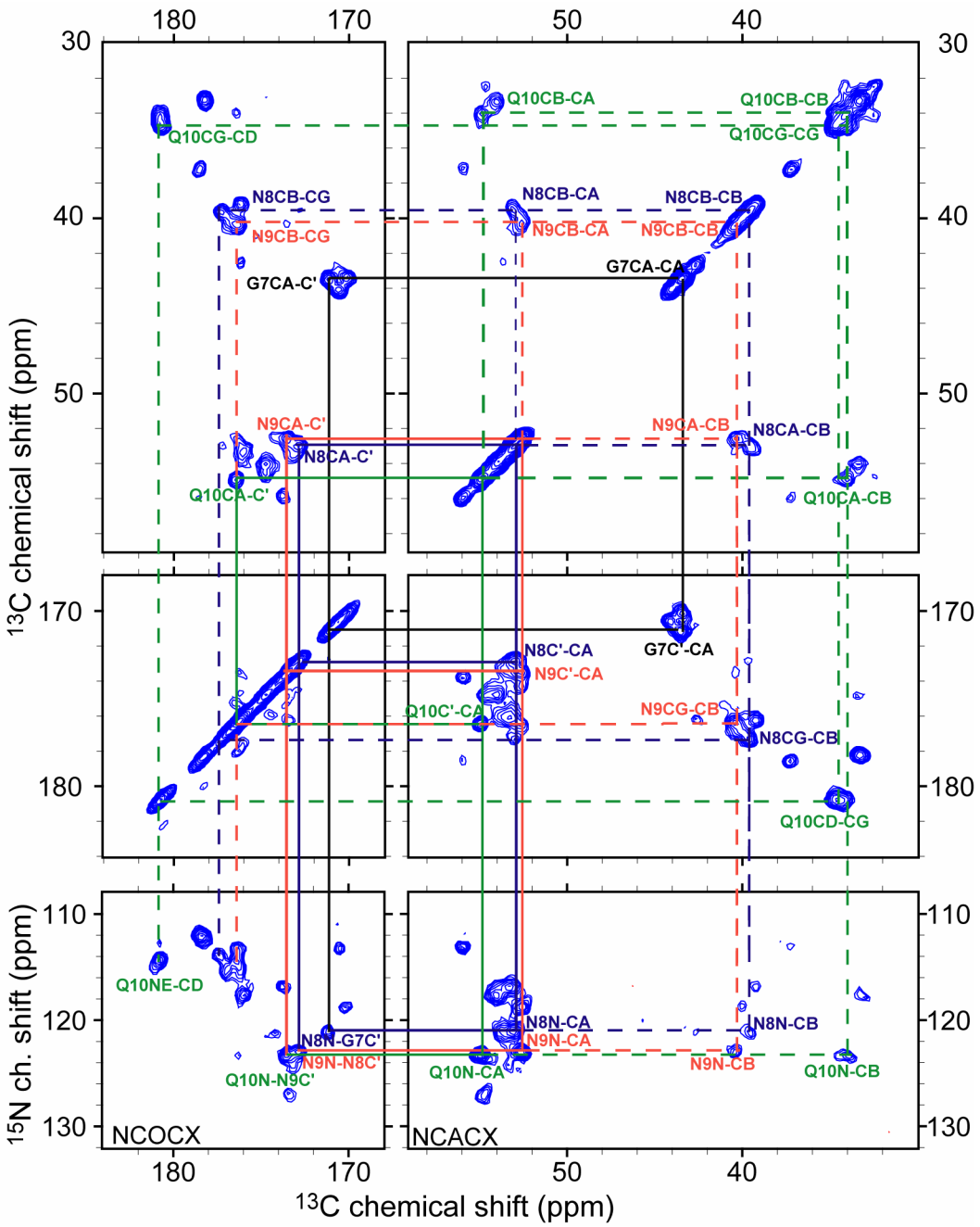

Figure 2: Correlation traces of fibril form 1 in 100%-labeled [U-^{13}C,^{15}N-GNNQ]QNY fibrils.

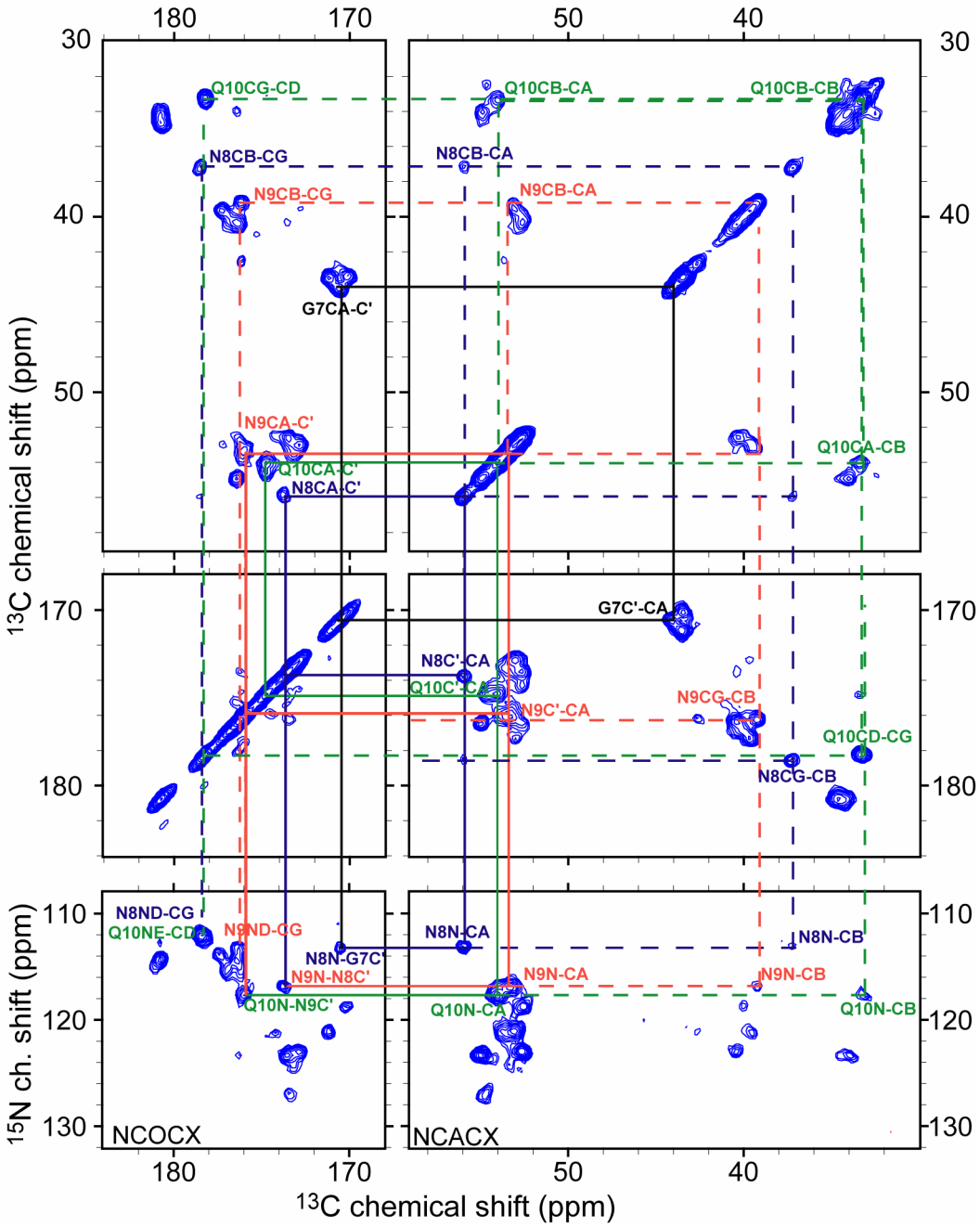

Figure 3: Correlation traces of fibril form 2 in 100%-labeled [U-^{13}C,^{15}N-GNNQ]QNY fibrils.

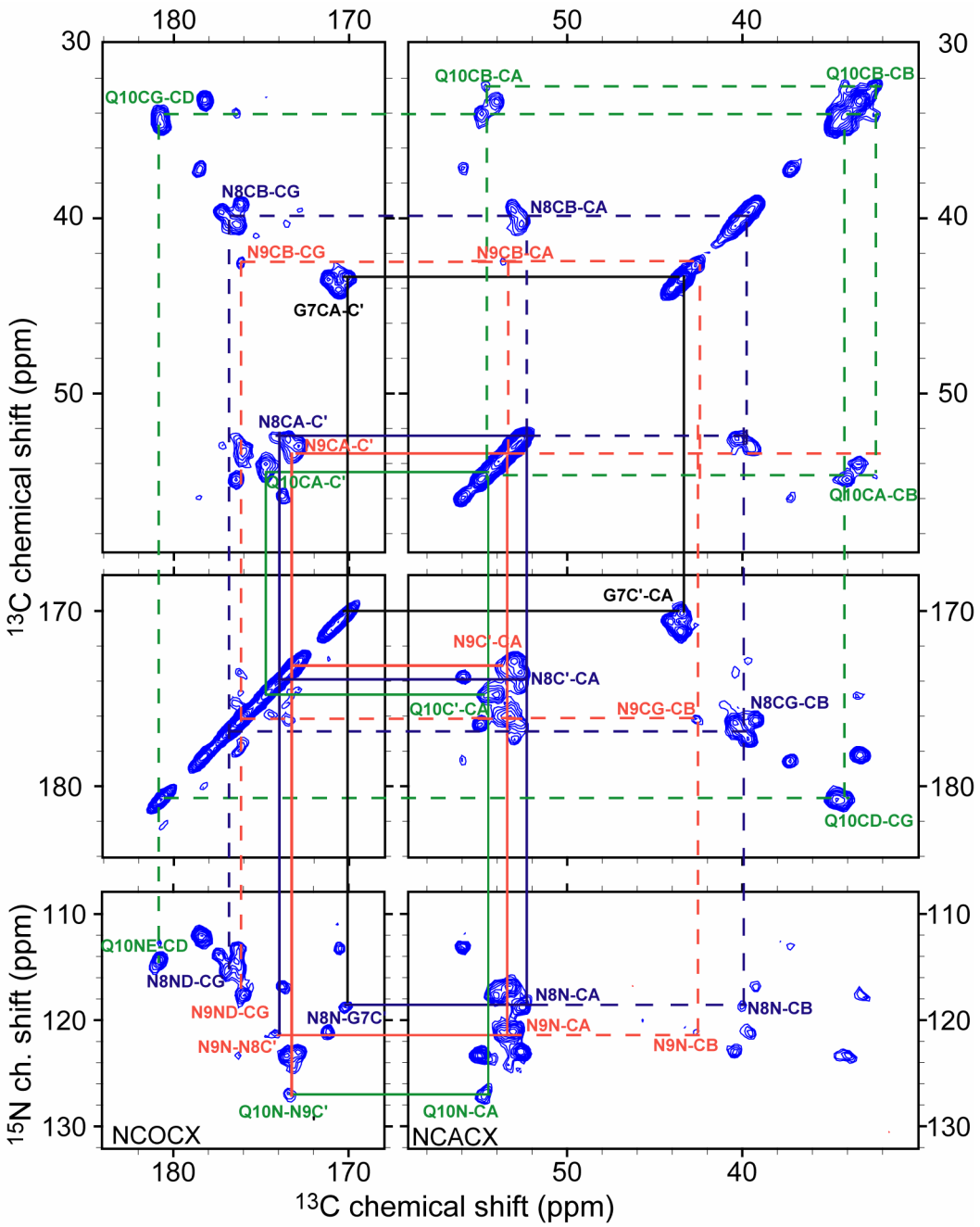

Figure 4: Correlation traces of fibril form 3 in 100%-labeled [U-^{13}C,^{15}N-GNNQ]QNY fibrils.