

Supplemental material for
CACA-TOCSY with alternate ^{13}C - ^{12}C labeling: a $^{13}\text{C}^{\alpha}$ direct
detection experiment for mainchain resonance assignment, dihedral
angle information, and amino acid type identification

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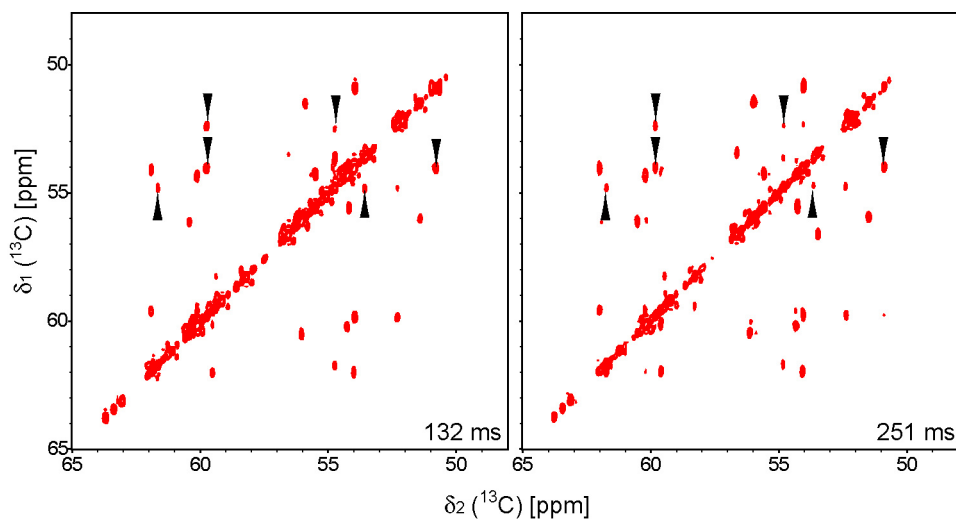


Figure S1. 2D CACA-TOCSY spectra with $[U\text{-}^2\text{H}, ^{15}\text{N} + 1,3\text{-}^{13}\text{C}]$ -GB1 (2.5 mM) recorded at 285K in D_2O . The 2D CACA-TOCSY spectra at mixing times of 132 ms (left) and 256 ms (right) are shown. Black arrowheads indicate sequential correlations from Leu C^α (indirect dimension) to neighboring residues (direct dimension). The measuring time for each spectrum was 5.5 h.

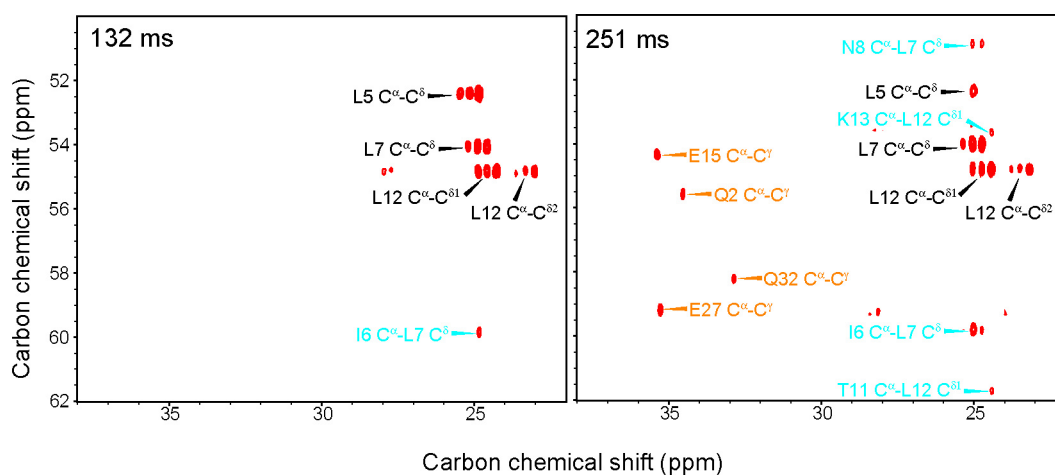


Figure S2. C^α to sidechain carbon correlations in the $[U\text{-}^2\text{H}, ^{15}\text{N} + 1,3\text{-}^{13}\text{C}]$ -GB1 sample. The spectra were recorded at the same conditions as Figure S1 with mixing times of 132 ms (left) and 251 ms (right). The measuring time for each experiment was 5.5 h. Black and

cyan arrowheads indicate intra-residual and sequential C–C correlations, respectively. Orange arrowheads indicate intra-residual C–C correlations. Leu C resonances feature splittings in the direct dimensions due to insufficient proton decoupling during acquisition.

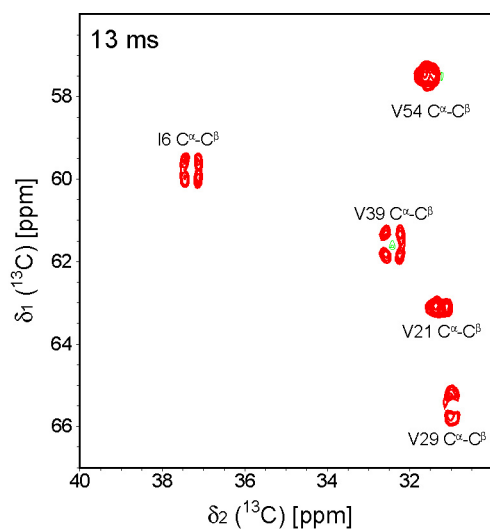


Figure S3. C^α to C^β 1J correlations for the valines and isoleucines in a $[U\text{-}^2\text{H}, ^{15}\text{N} + 2\text{-}^{13}\text{C}]$ -GB1 sample. The spectrum was recorded at the same condition as in Fig. 2 with a mixing time of 13 ms. The measuring time was 5.5 h. The sequence specific resonance assignments are indicated. Note that line shapes are distorted by the presence of C^α - C^β couplings as well as the interference of TOCSY and ROESY contributions.