

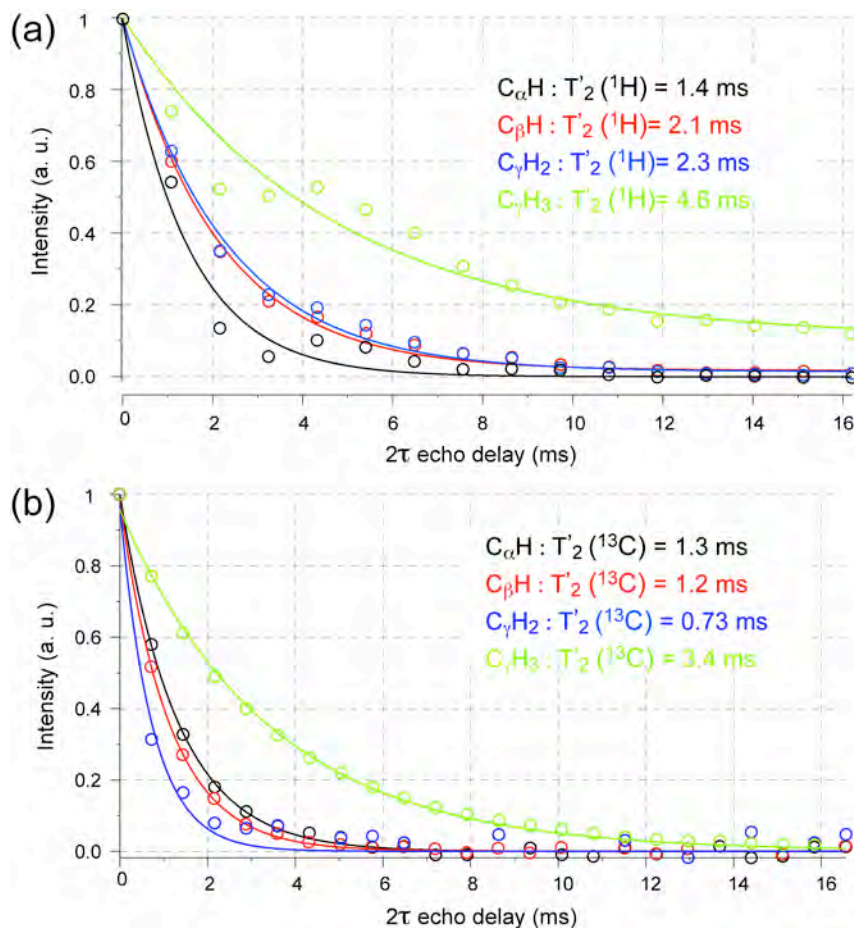
## Supporting Information

# Methyl Proton Contacts Obtained Using Heteronuclear Through-Bond Transfers in Solid- State NMR Spectroscopy

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**Figure S1:** Measurement of proton (a) and carbon (b) refocused transverse dephasing time  $T_2'$  under homonuclear decoupling on fully  $^{13}\text{C}$ -labeled L-isoleucine. Proton  $T_2'$  were measured using the following pulse sequence:  $90^\circ$  proton pulse,  $\tau$ - $\pi$ - $\tau$  echo period under eDUMBO-1 homonuclear decoupling, short CP step of  $50 \mu\text{s}$  to transfer the magnetization to the adjacent carbon nuclei, and detection of the  $^{13}\text{C}$  magnetization. Carbon  $T_2'$  were measured using the following pulse sequence:  $90^\circ$  proton pulse, CP to the carbon spins,  $\tau$ - $\pi$ - $\tau$  echo period under eDUMBO-1 homonuclear decoupling, Z-filter (1 ms) and detection of the  $^{13}\text{C}$  magnetization. The  $\pi$  pulse employed was a selective Q3 refocusing pulse of 2.3 ms. In both measurements the  $\tau$  delays were incremented by multiples of the rotor period and 16 transients were used for each value of  $\tau$ . The spinning frequency was 22 kHz and eDUMBO-1 decoupling was applied at a RF field strength of 150 kHz. The carbon peak intensities were then fitted to a single exponential decay function.

Seg	Res	aa	Atom	Seg	Res	aa	Atom
a	16	ALA	HB	a	51	MET	HE
a	19	ALA	HB	a	47	ILE	HD1
a	44	ALA	HB	a	26	ALA	HB
a	33	VAL	HG	a	44	ALA	HB
a	57	THR	HG	a	12	THR	HG
a	77	LEU	HD	a	26	ALA	HB
a	63	LEU	HD	a	77	LEU	HD
a	81	VAL	HG	a	35	LEU	HD
a	55	VAL	HG	a	14	LEU	HD
a	23	VAL	HG	a	47	ILE	HG2
a	26	ALA	HB	a	33	VAL	HG
a	33	VAL	HG	a	63	LEU	HD
a	42	VAL	HG	a	35	LEU	HD
a	61	VAL	HG	a	35	LEU	HD
a	44	ALA	HB	a	35	LEU	HD
a	61	VAL	HG	a	81	VAL	HG
a	44	ALA	HB	a	63	LEU	HD
a	44	ALA	HB	a	77	LEU	HD
a	47	ILE	HD1	a	20	ALA	HB
a	47	ILE	HD1	a	23	VAL	HG
a	51	MET	HE	a	19	ALA	HB
a	73	ALA	HB	a	33	VAL	HG
a	64	ILE	HD1	a	62	THR	HG
a	54	ALA	HB	a	55	VAL	HG

a	19	ALA	HB	a	14	LEU	HD
a	78	ALA	HB	a	74	LEU	HD
a	20	ALA	HB	a	16	ALA	HB
a	21	LEU	HD	a	20	ALA	HB
a	12	THR	HG	b*	55	VAL	HG
a	74	LEU	HD	b*	2	VAL	HG
a	35	LEU	HD	b*	6	VAL	HG
a	2	VAL	HG	b*	74	LEU	HG
a	78	ALA	HB	b*	6	VAL	HG
a	14	LEU	HD	b*	12	THR	HG

**Figure S2:** Distance restraints identified from the 2D J-CHHC spectrum of the [U-<sup>13</sup>C/<sup>15</sup>N] labeled Crh protein. \* These restraints involve proton contacts between the two monomers (segment a and b) of the dimeric Crh.