Supporting Information for:

Restraints on backbone conformations in solid state NMR studies of uniformly labeled proteins from quantitative amide ^{15}N-^{15}N and carbonyl ^{13}C-^{13}C dipolar recoupling data

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Figures S1 and S2; Tables S1 and S2

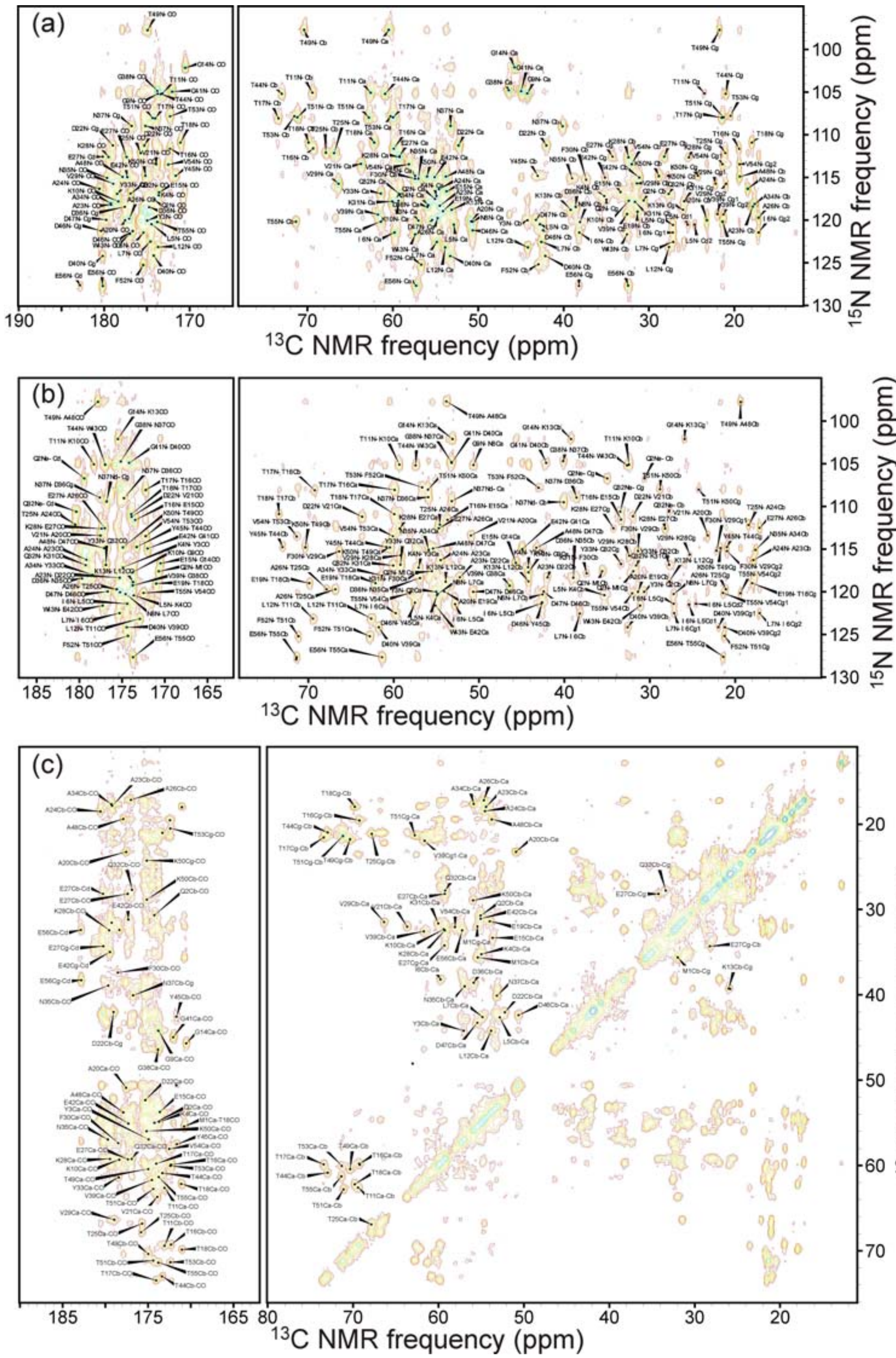

Figure S1: 2D NCACX (a), NCOCX (b), and CC (c) correlation spectra of microcrystalline, uniformly ^{15}N,^{13}C-labeled GB1, recorded at 17.6 T with MAS at 17.00 kHz. Assignments were obtained from these spectra using the MCASSIGN2 program [Hu *et al.*, J Biomolec. NMR **50**, 267-276 (2011)]. Assignments are listed in Table S1.

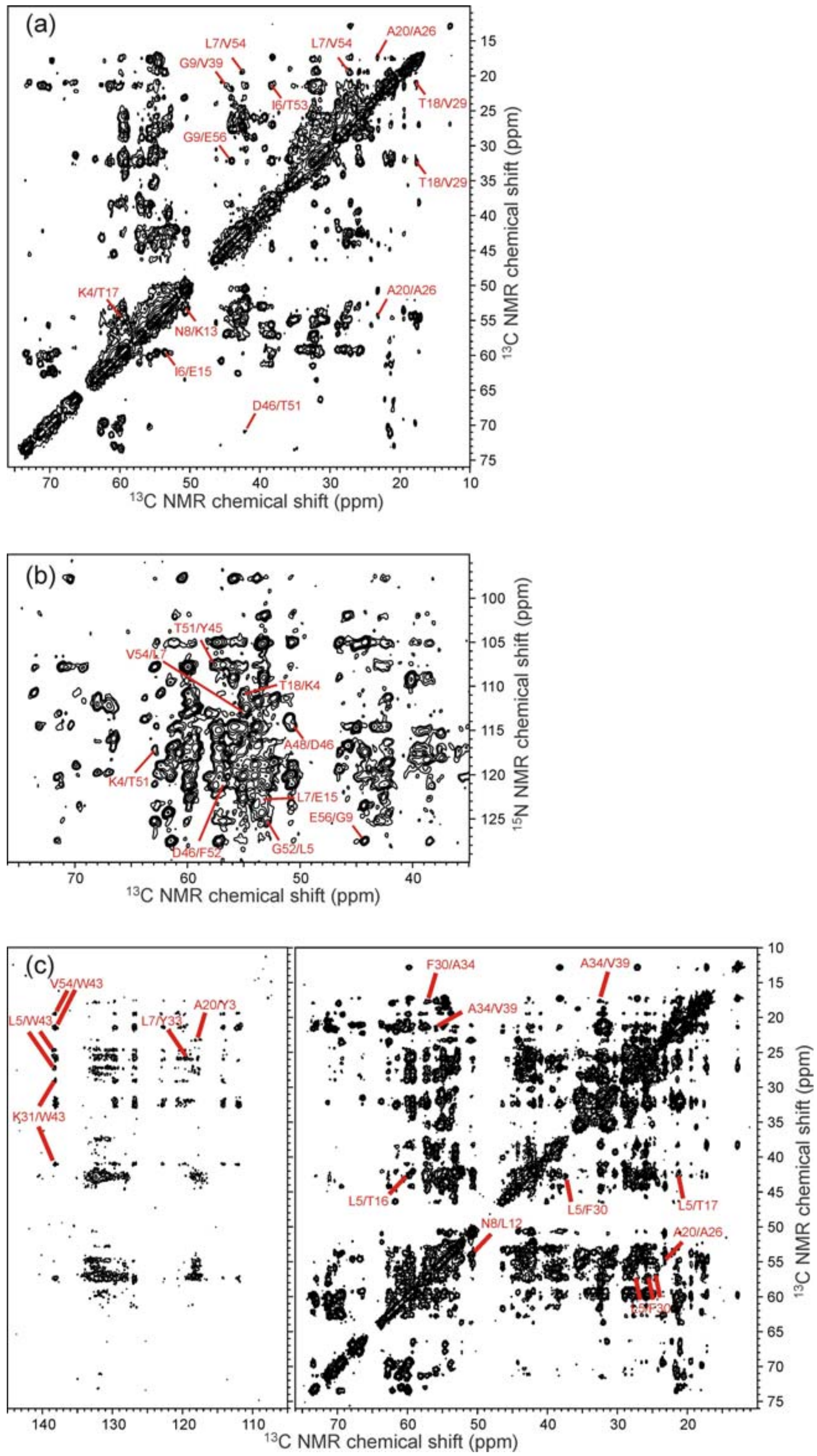

Figure S2: 2D CHHC (a), NHHC (b), and RAD (c) spectra of GB1, from which inter-residue distance restraints in Table S2 were obtained. These spectra were recorded at 17.6 T with MAS at 17.00 kHz.

Table S1: Site-specific chemical shift assignments and predicted backbone torsion angles for microcrystalline GB1. Assignments were generated in a semi-automated manner from spectral data in Fig. S1, using the program MCASSIGN2. ^{13}C shifts are in ppm relative to DSS, ^{15}N shifts are in ppm relative to liquid NH_3. Torsion angle predictions (in degrees) were generated by TALOS+. Only "good" predictions are shown. Predictions that were included in Xplor-NIH calculations are listed in bold font. Uncertainties were doubled in Xplor-NIH calculations. Torsion angles determined from atomic coordinates in PDB file 2GI9 are shown for comparison.

residue type	^{13}CO shift	^{13}Cα shift	^{13}Cβ shift	^{13}Cγ shift	^{13}Cδ shift	backbone ^{15}N shift	sidechain ^{15}N shift	TALOS+ ϕ prediction	TALOS+ ψ prediction	ϕ,ψ in PDB 2GI9
M1	170.7	55.5	35.6	31.9		33.5				---,152
Q2	174.2	55.0	30.7	35.0	179.4	117.6	106.7	-135 ± 22	**147 ± 14**	-94,127
Y3	174.9	57.0	44.2			120.0		**-130 ± 16**	**157 ± 13**	-115,152
K4	173.6	54.9	35.3			117.1		- 152 ±10	**150 ± 9**	-114,146
L5	174.4	52.8	42.7	27.2	25.5, 24.6	120.5		**-117 ± 16**	**133 ± 17**	-122,125
I6	175.3	59.8	38.2	17.2, 27.2		121.4				-105,123
L7	174.6	54.9	42.6	27.2		122.5				-104,126
N8		50.7	38.1			120.2		**-117 ± 28**	164 ± 26	-120,65
G9	173.7	44.2				105.2				-86,169
K10	178.3	59.3	32.4			117.7		-61 ± 7	-34 ± 10	-62,-40
T11	173.0	62.6	69.4	23.5		105.1		-105 ± 19	-2 ± 16	-107,-43
L12	173.7	53.9	44.2	27.7		123.2		-128 ± 35	153 ± 12	-111,125
K13	175.5	53.1	39.2	26.0		118.8		**-134 ± 13**	**158 ± 16**	-133,148
G14	170.5	45.7				102.1		-169 ± 28	162 ± 11	144,-153
E15	173.5	53.7	33.3			116.7		**-143 ± 17**	**160 ± 0**	-139,140
T16	172.3	59.9	69.3	19.5		111.6		**-125 ± 31**	**153 ± 14**	-139,171
T17	174.0	59.9	73.4	21.5		108.0		**-126 ± 19**	**162 ± 6**	-135,162
T18	171.0	62.2	69.8	18.0		110.9				-153,162
E19		54.1	31.4			119.0		**-135 ± 23**	156 ± 14	-111,128
A20	177.5	50.9	23.2			120.0		-132 ± 35	167 ± 15	-150,156
V21	173.9	63.7	32.0			113.4		- 84 ± 28	-20 ± 23	-67,-31
D22	175.3	52.3	42.0	179.0		111.2				-156,177
A23	179.2	54.5	17.9			118.3				-70,-39
A24	180.5	54.6	18.5			116.3		**-64 ± 6**	**-38 ± 7**	-61,-37
T25	175.7	66.8	67.8	21.1		112.1		**-67 ± 9**	**-38 ± 13**	-67,-47
A26	177.0	54.7	17.2			119.7		**-64 ± 6**	**-40 ± 14**	-60,-40
E27	177.4	59.3	28.2	34.3	180.2	112.5		**-65 ± 10**	**-40 ± 13**	-58,-43
K28	179.5	59.2	32.4	25.3		112.6		**-67 ± 9**	**-45 ± 9**	-62,-42
V29	178.9	66.4	31.4	21.0, 22.2		115.7		**-69 ± 10**	**-40 ± 9**	-63,-48
F30	178.5	57.3	37.4			115.2		**-69 ± 9**	**-38 ± 12**	-68,-36
K31	179.3	60.0	31.6	25.9		117.8		**-69 ± 9**	**-38 ± 11**	-65,-38
Q32	176.9	59.2	27.7	33.4	180.4	116.9	110.6	**-63 ± 5**	**-45 ± 4**	-64,-41
Y33	178.4	61.4				116.8		**-62 ± 6**	**-44 ± 7**	-59,-46
A34	179.3	55.9	17.6			118.1		**-61 ± 6**	**-39 ± 6**	-63,-46
N35	179.7	57.0	39.0			115.1		**-62 ± 4**	**-41 ± 6**	-60,-45
D36	174.8	55.9	38.6	177.6		118.4		**-66 ± 9**	**-29 ± 13**	-63,-46
N37	175.2	53.2	40.1	176.8		109.0	109.4	-91 ± 11	1 ± 14	-103,12
G38	173.8	46.4				104.8		85 ± 23	15 ± 11	86,10
V39	174.5	61.7	32.6	21.2, 22.0		119.3		-104 ± 14	126 ± 24	-97,134

D40	174.2	53.2	42.1	180.2		124.2		-107 ± 25	151 ± 34	-140,111
G41	172.0	45.0				104.9				-156,-164
E42	177.3	55.1	31.1	35.1	179.5	115.0		**-114 ± 25**	**140 ± 13**	-96,140
W43	177.0	57.4	32.6			121.6				-113,147
T44	173.3	60.9	73.0	21.0		105.2		**-135 ± 24**	**161 ± 14**	-131,159
Y45	171.6	57.5	42.7			114.8		**-149 ± 7**	**152 ± 14**	-136,130
D46	176.7	50.7	42.4	180.4		121.2				-119,104
D47	177.1	55.4	43.3	179.8		120.0		-64 ± 8	-28 ± 13	-62,-25
A48	177.9	53.8	19.4			114.9		-71 ± 16	-28 ± 14	-71,-16
T49	174.9	60.4	70.4	21.7		97.7		-108 ± 11	1 ± 21	-126,8
K50	175.2	56.0	28.9	24.3	27.8	114.8		61 ± 7	34 ± 11	53,40
T51	174.3	62.8	71.1	21.3		108.0				-123,129
F52	175.4	56.6	43.0			125.2		**-102 ± 20**	**152 ± 14**	-99,152
T53	172.3	60.1	71.4	20.5		107.8		**-143 ± 23**	**157 ± 13**	-129,149
V54	172.5	58.1	32.1	19.4, 21.4		113.5		**-122 ± 24**	**145 ± 14**	-131,131
T55	173.7	61.3	71.4	21.4		120.2		**-112 ± 18**	**131 ± 17**	-133,129
E56	180.2	57.2	32.4	38.2	182.8	127.7				-97,---

Table S2: Interatomic distance restraints derived from inter-residue crosspeaks in 2D CHHC, NHHC, and RAD spectra of microcrystalline GB1 in Fig. S2, and from ^{15}N-BARE and ^{13}C-BARE data for N- and C-terminal residues. (Note: If atom 1 and/or atom 2 can represent more than one site, *e.g.*, the three H_β sites of A20, then distance restraints for all possible combinations of atom 1 and atom 2 were included in the Xplor-NIH calculations.)

atom 1	atom 2	distance range (Å)	2D spectrum
I6 H_α	E15 H_α	1.8-2.8	CHHC
N8 H_α	K13 H_α	1.8-2.8	CHHC
K4 H_α	T17 H_α	1.8-2.8	CHHC
L12 H_δ	N37 H_β	1.8-2.8	CHHC
T18 H_γ	V29 H_β	1.8-2.8	CHHC
T18 H_γ	V29 H_γ	1.8-2.8	CHHC
A20 H_β	A26 H_α	1.8-2.8	CHHC
A20 H_β	A26 H_β	1.8-2.8	CHHC
G9 H_α	V39 H_γ	1.8-2.8	CHHC
E42 H_β	T55 H_β	1.8-2.8	CHHC
D46 H_β	T51 H_β	1.8-2.8	CHHC
I6 H_β	T53 H_γ	1.8-2.8	CHHC
L7 H_β	V54 H_γ	1.8-2.8	CHHC
L7 H_γ	V54 H_γ	1.8-2.8	CHHC
G9 H_α	E56 H_β	1.8-2.8	CHHC
K4 H_N	T51 H_α	2.0-4.0	NHHC
L5 H_α	F52 H_N	2.0-4.0	NHHC
L7 H_α	E54 H_N	2.0-4.0	NHHC
G9 H_α	E56 H_N	2.0-4.0	NHHC
L7 H_N	E15 H_α	2.0-4.0	NHHC
K4 H_α	T18 H_N	2.0-4.0	NHHC
D46 H_α	A48 H_N	2.0-4.0	NHHC
D46 H_N	F52 H_α	2.0-4.0	NHHC
Y45 H_α	T51 H_N	2.0-4.0	NHHC
L5 C_δ	F30 C_α	3.0-7.0	RAD
L5 C_δ	W43 $C_{\varepsilon 2}$	3.0-7.0	RAD
L5 C_γ	F30 C_α	3.0-7.0	RAD
L5 C_γ	W43 $C_{\varepsilon 2}$	3.0-7.0	RAD
L7 C_δ	Y33 C_ζ	3.0-7.0	RAD
N8 C_α	L12 C_α	3.0-7.0	RAD
L5 C_β	T16 C_α	3.0-7.0	RAD
L5 C_β	T17 C_γ	3.0-7.0	RAD
Y3 C_ζ	A20 C_β	3.0-7.0	RAD
A20 C_β	A26 C_α	3.0-7.0	RAD
L5 C_β	F30 C_β	3.0-7.0	RAD
K31 C_β	W43 $C_{\varepsilon 2}$	3.0-7.0	RAD
K31 C_δ	W43 $C_{\varepsilon 2}$	3.0-7.0	RAD
K31 C_γ	W43 $C_{\varepsilon 2}$	3.0-7.0	RAD
F30 C_α	A34 C_β	3.0-7.0	RAD
A34 C_β	V39 C_β	3.0-7.0	RAD
Y45 C_ζ	D47 C_β	3.0-7.0	RAD
W43 $C_{\varepsilon 2}$	V54 C_γ	3.0-7.0	RAD
M1 N	Q2 N	3.1-3.5	^{15}N-BARE
T55 N	E56 N	3.1-3.3	^{15}N-BARE
M1 CO	Q2 CO	3.0-3.2	^{13}C-BARE
T55 CO	E56 CO	2.9-3.1	^{13}C-BARE