

Supporting Information for

**Fast Magic Angle Spinning NMR with Heteronucleus Detection for Resonance
Assignments and Structural Characterization of Fully Protonated Proteins**

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Table S1. Acquisition and processing parameters for the 3D heteronuclear-detected and 2D HETCOR MAS NMR spectra.

Experiment	Acquisition parameters			Processing parameters		
	ω_3	ω_2	ω_1	ω_3	ω_2	ω_1
<i>5mM Cu(II)-EDTA doped U-¹³C, ¹⁵N-LC8, 19.9 T, $\omega_r = 62$ kHz</i>						
HNCA	2064 complex; SW = 64.1 kHz; 256 scans	24 complex; SW = 7.5 kHz;	48 complex; SW = 5.0 kHz;	30-degree/60- degree sinebell;	forward linear prediction of 40 points; 30-degree/60- degree sinebell;	forward linear prediction of 80 points; 30-degree/60- degree sinebell;
HNCO	2064 complex; SW = 64.1 kHz; 128 scans	24 complex; SW = 7.5 kHz;	48 complex; SW = 5.0 kHz;	30-degree/60- degree sinebell;	forward linear prediction of 40 points; 30-degree/60- degree sinebell;	forward linear prediction of 80 points; 30-degree/60- degree sinebell;
HNCOCX	2064 complex; SW = 64.1 kHz; 512 scans	24 complex; SW = 7.5 kHz;	48 complex; SW = 5.0 kHz;	30-degree/60- degree sinebell;	forward linear prediction of 40 points; 30-degree/60- degree sinebell;	forward linear prediction of 80 points; 30-degree/60- degree sinebell;
<i>5mM Cu(II)-EDTA doped U-¹³C, ¹⁵N-LC8, 19.9 T, $\omega_r = 60$ kHz</i>						
¹³ C-detected HETCOR		2048 complex; SW = 64.1 kHz; 128 scans	128 complex; SW = 20.0 kHz;		30-degree sinebell;	forward linear prediction of 128 points; 30-degree sinebell;
¹ H-detected HETCOR		1024 complex; SW = 34.5 kHz; 32 scans	512 complex; SW = 45.0 kHz;		30-degree sinebell;	forward linear prediction of 512 points; 30-degree sinebell;

Table S2. Additional chemical shifts of 5mM Cu(II)-EDTA doped U-¹³C, ¹⁵N-LC8 assigned based on 3D ¹³C-detected MAS NMR HNCA, HNC0 and HNCOCX spectra.

Residue	C γ (ppm)	C γ 1 (ppm)	C γ 2 (ppm)	C δ (ppm)	C ϵ (ppm)	C ζ (ppm)
I38			17.9			
I42		28.8				
N51	180.5					
T70			21.5			
F86	138.4			131.6	131.0	130.2

Table S3. ^1H and ^{13}C linewidths of cross peaks in $^1\text{H}_\text{N}$ - $^{13}\text{C}\alpha$ regions of 2D ^{13}C -detected HETCOR and ^1H -detected HETCOR spectra.

Chemical Shifts (ppm)		Linewidths (Hz)			
^1H	^{13}C	^1H	^{13}C	^1H	^{13}C
		^{13}C -detected HETCOR		^1H -detected HETCOR	
9.4	56.8	354	208	411	205
9.4	58.5	427	185	418	189
9.1	55.9	455	152	525	196
8.9	61.5	269	122	322	143
8.8	55.1	401	129	499	149
8.7	62.5	475	121	463	169
8.6	59.4	747	177	803	233
8.4	55.3	384	177	350	174
8.3	54.0	504	186	415	207
8.3	56.9	812	179	768	182
8.3	60.2	642	201	581	210
8.2	62.6	390	172	671	178
8.1	59.2	540	204	606	192
8.0	61.8	448	199	526	225
8.0	70.6	396	130	298	186
7.9	57.9	364	198	333	213
7.8	60.1	1100	237	1192	259
7.8	65.2	280	197	226	189
7.7	59.0	376	712	375	285
7.7	52.4	506	143	674	153
7.6	63.6	700	194	805	186
7.6	66.1	923	397	839	498
7.5	57.0	368	155	328	157
7.4	61.7	391	235	415	240
7.4	60.7	373	209	328	234
7.3	57.9	308	132	296	145
7.2	69.9	240	139	296	239
7.2	71.8	357	161	347	167
7.2	65.8	366	169	410	177
7.2	66.9	379	149	327	174
7.0	63.0	513	282	547	215
7.0	67.4	443	143	471	192
6.9	68.7	237	124	227	134
6.8	57.1	358	130	344	139

Note: The linewidths are extracted from spectra processed with 60-degree sinebell apodization.