

Supplementary material

Similarities and differences within members of the Ff family of filamentous bacteriophage viruses

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Table S1: Experimental parameters.

Experimental parameters are shown for the 2D and 3D experiments recorded on the M13 bacteriophage sample. The data for the DARR experiments is noted for both M13 and fd (fd parameters, if different, are in brackets). For the RFDR, zfr-INADEQUATE and 3D NCACX/NCOCX, parameters are given only for M13.

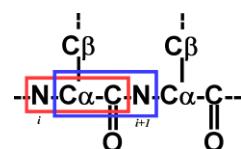
2D experiments			
sequence	DARR	RFDR	zfr-INADEQUATE
¹ H frequency [MHz]	600.2	600.2	600.2
spinning frequency (v_r) [kHz]	13.5	12	12
set temperature [°C]	3	3	3
acquisition points (t1/t2)	1024/4990 (1024/4096)	1024/4990	712/3988
acquisition times (t1/t2) [ms]	12.8/25 (12.8/20.5)	12.8/25	5/20
¹³ C carrier frequency [ppm]	103.6	110	97.1
H_{90}/C_{90} [μ s]	2.5/5	2.5/5	2.5/5
CP power (v_H/v_C) [kHz]	~65/~50	~65/~50	~65/~50
CP contact time [ms]	2.4 (1.2)	2.4	3
mixing time [ms]	15, 50, 100, 250, 500	3	τ (scalar coupling)=3
¹ H Decoupling [kHz]	~80	~80	~80
swf-tppm decoupling. Tangent pulse 78-122% / phase [μ s/phase].	6/20°	6/20°	6/20°
relaxation delay/scans[sec]	2.7/16	3/32	3/128
SW (F1/F2) [kHz]	40/100	40/100	67.9/100
processing parameters F1/F2			
processing software	NMRPipe	TOPSPIN	NMRpipe
total # of points	4096/8192	2048/8192	4096/8192
apodization functions	Lorentz to Gauss transformation in (F1;F2): (G50/L10; G65/L40)	exponential: (50Hz; 20Hz)	1. exponential: (50Hz; 100Hz) 2. squared cosine-bell

3D experiments		
Experiment	NCACX	NCO CX
¹ H frequency [MHz]	600.2	600.2
spinning frequency (v_r) [kHz]	12	12
set temperature [°C]	3	3
acquisition points (t1/t2/t3)	68/160/4990	92/68/4990
acquisition time (t1/t2/t3) [ms]	10.0/11.8/25	13.6/10/25
carrier frequency [ppm]	116.8/54.0/102.0	117.5/178.6/99.7
H_{90}/C_{90} [μs]	2.5/5	2.5/5
CP power (v_H/v_N) [kHz]	~65(ramp 90-100%)/~50	~65(ramp 90-100%)/~50
CP contact time [ms]	3	3
DCP shape (on ¹³ C)	tangent 80-120%	tangent 80-120%
DCP power (v_C/v_N) [kHz]	~1.5v _r /3.3v _r	~3.5v _r /1.5v _r
DCP contact time [ms]	5.5	5
DARR mixing time [ms]	15	15
¹ H Decoupling [kHz]	~80	~80
swf-tppm [μs/phase]	7/20°	5.5/20°
relaxation delay/scans	2.7s/16	2.7s/16
SW (F1/F2/F3) [kHz]	3.3/6.7/100	3.3/3.3/100
processing parameters F1/F2/F3 (NMRPipe)		
total number of points	512/512/8192	
apodization	¹⁵ N dim. (F1) exponential (20 Hz); ¹³ C dim. (F2,F3) squared-cosine	

Samples:

U-[¹³C, ¹⁵N] PEG precipitated wild-type M13 virus (fully hydrated) in a 50μL NMR (12mg) zirconium rotor (of 4mm diameter).

Figure S1 - Strip plots from 3D heteronuclear correlation experiments showing residues 12-17 of the M13 capsid protein. Red and blue spectra correspond to intra-residue NCACX and sequential NCO CX experiments, respectively. Horizontal bars link the strips from ¹⁵N planes of residues j and $j-1$ that share the C'-Cα peaks of residue $j-1$. The bottom sub-spectra correspond to 2D planes at the same ¹⁵N shift and display N-C'-Cβ or N-Cα-Cβ cross-peaks. Two adjacent amino-acids and the magnetization transfer in the 3D experiment are shown above.



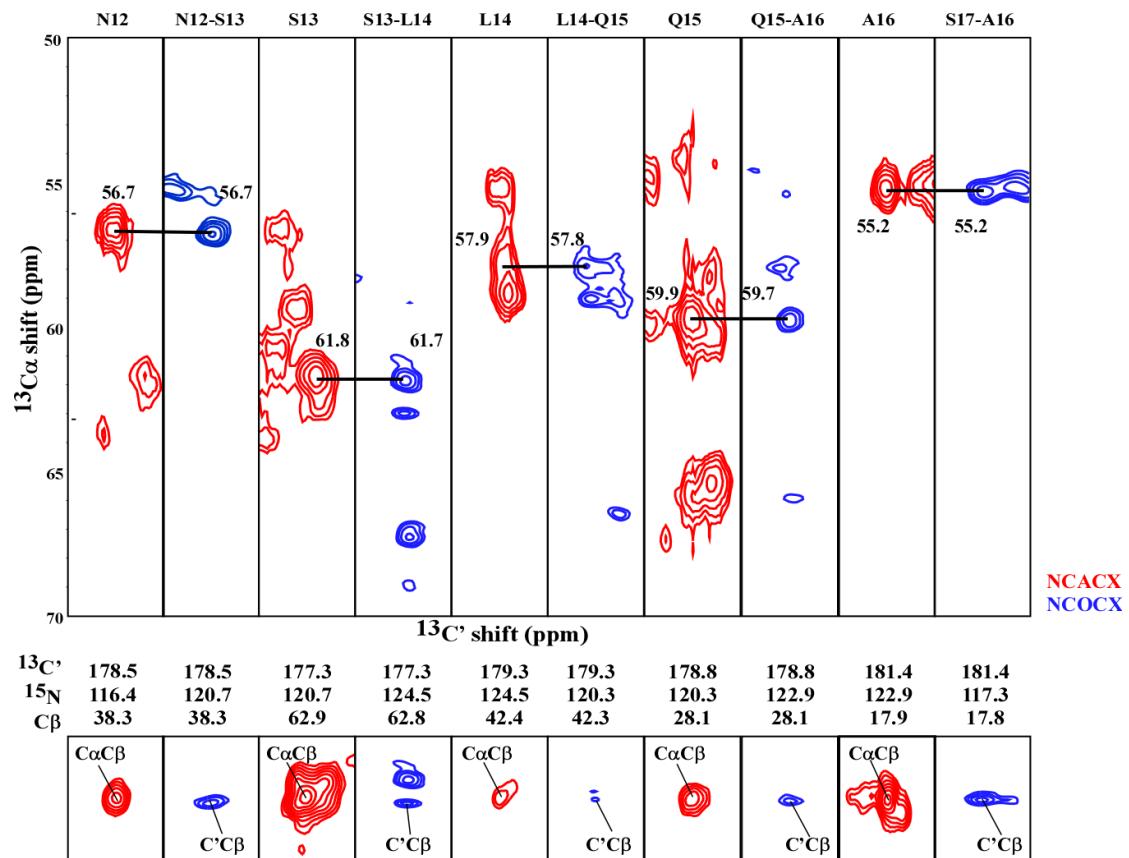


Table S2. Chemical shift assignments of the magic-angle spinning solid-state NMR resonances of the M13 phage coat protein. Deviation from fd Chemical shifts are indicated in the brackets. Chemical shift values were generated by Sparky and are given in ppm. Unassigned atoms are indicated by empty cells and the dashes in the shaded cells (-) indicate that the side-chain of the specific residue does not have the atom in the column. Absence of brackets suggests that the atom chemical shift was either ambiguous or unassigned for M13 and/or for fd. Averaged standard deviations (calculated by Sparky) are 0.08 ± 0.04 and 0.12 ± 0.06 ppm for ^{13}C and ^{15}N , respectively.

	C α	C β	C'	C γ	C γ 1/2	C δ	C δ 1/2	C ε	C ε 1/2	C ζ	C ζ 2	N
A1	51.6 (0.2)	19.5 (0.2)	174.2	-	-	-	-	-	-	-	-	
E2	57.5 (0.1)	29.8 (0.1)	177.1	36.0 (0.1)	-	183.9	-	-	-	-	-	120.3
G3	45.1 (0)	-	174.1 (0.1)	-	-	-	-	-	-	-	-	111.3
D4	54.3 (0)	41.3 (0.1)	176 (0)	179.9	-	-	-	-	-	-	-	120.3
D5	51.8 (0.1)	41.6 (0.1)	175.1 (0.1)	180.0 (0.1)	-	-	-	-	-	-	-	122 (0.1)
P6	63.9 (0)	31.7 (0.1)	178 (0.1)	26.6 (0.2)	-	50.8 (0.1)	-	-	-	-	-	
A7	54.6 (0)	19.3 (0.1)	179.1 (0.3)	-	-	-	-	-	-	-	-	119.6 (0.2)
K8	60.1 (0.1)	32.8 (0.3)	178.7 (0.6)	24.7 (0.3)	-	30.0 (0.9)	-	42.3 (0.3)	-	-	-	118.8 (0.1)
A9	54.7 (0.1)	17.9 (0)	180.4 (0.1)	-	-	-	-	-	-	-	-	119.4 (0.3)
A10	54.9 (0)	17.8 (0.2)	180.3 (2.2)	-	-	-	-	-	-	-	-	117.7 (2)
F11	63.7 (0.2)	38.1 (0.1)	178.8 (0.1)		-	-		-	-	-	-	116.9 (2.2)

N12	56.7 (1.1)	38.3 (1.9)	178.5 (1.3)	175.8 (2.9)	-	-	-	-	-	-	-	-	116.3 (2.7)
S13	61.7 (0)	62.7 (0.2)	177.3 (0.1)	-	-	-	-	-	-	-	-	-	117.3 (0.5)
L14	57.8 (0)	42.5 (0.1)	179.3 (0.1)	26.4 (0)	-	-	23.5 (0)	-	-	-	-	-	124.4 (0.3)
Q15	59.6 (0.1)	28.0 (0.3)	178.8 (0.1)	33.3 (0.3)	-	-	-	-	-	-	-	-	120.5 (0.3)
A16	55.2 (0.1)	17.7 (0.1)	181.3 (0.2)	-	-	-	-	-	-	-	-	-	122.9 (0.1)
S17	62.4 (0.1)	62.4 (0.1)	174.8 (0.1)	-	-	-	-	-	-	-	-	-	117.2 (0)
A18	55.9 (0)	17.2 (0.1)	179 (0)	-	-	-	-	-	-	-	-	-	122.2 (0.1)
T19	68.7 (0.1)	67.1 (0)	177.1 (0)	-	-21.6 (0)	-	-	-	-	-	-	-	111.5 (0.1)
E20	58.8 (0)	28.7 (0)	179.1 (0.1)	35.6 (0.1)	-	183.2 (0.1)	-	-	-	-	-	-	124.3 (0.1)
Y21	58.2 (0.2)	36.1 (0.2)	178.2 (0.1)	129.7 (0)	-	-	130.8 (0)	-	118.7 (0)	158.7 (0.2)	-	-	120 (0.2)
I22	65.5 (0)	38.4 (0)	178.3 (0.3)	-	31(0.2)/18.4 (0)	-	15.6 (0.1) / -	-	-	-	-	-	120.4 (0)
G23	47.3 (0.1)	-	177.9 (0.1)	-	-	-	-	-	-	-	-	-	106.5 (0.2)
Y24	57.9 (0)	35.3 (0.2)	179.1 (0)	129 (0.4)	-	-	132.5 (0.1)	-	118.2 (0.2)	157.7 (0.1)	-	-	122.0 (0.4)
A25	55.1 (0.1)	18.5 (0.1)	179.7 (0.1)	-	-	-	-	-	-	-	-	-	123.7 (0.2)
W26	60.7 (0.2)	29.0 (0.2)	178.7 (0.1)	113.9 (0.2)	-	-	127.7(0.2) /129.1(0.1)	-	-139.9 (0)	-	113.0 (0.3) /	-	117.7
A27	54.8 (0.1)	20.0 (0)	180.6 (0.1)	-	-	-	-	-	-	-	-	-	121.4 (0.4)
M28	57.8 (0.1)	33.1 (0.3)	177.8 (0)	32.7 (0)	-	-	-	17.9 (0)	-	-	-	-	119.2
V29	67.2 (0.1)	31.8 (0)	179.3 (0)	-	21.7/	-	-	-	-	-	-	-	120.4
V30	65.0	31.5 (0)	178.5 (0)	-	24.1/21.9	-	-	-	-	-	-	-	120.4
V31	65.9 (0.1)	32.2 (0)	178.8 (0.1)	-	22.9/21.1	-	-	-	-	-	-	-	121.3
I32	66.2	37.6 (0.4)	177.9 (1)	-	29.3/16.7	-	13.7 (0.2) / -	-	-	-	-	-	122
V33	65.3	31.5 (0)	177.9 (0)	-	25.0/22.3	-	-	-	-	-	-	-	120.5
G34	47.7 (0.1)	-	177.3 (0)	-	-	-	-	-	-	-	-	-	106.4 (0.3)
A35	55.1 (0)	21.4 (0.1)	179.1 (0.2)	-	-	-	-	-	-	-	-	-	123.0 (0.3)
T36	67.1 (0.1)	65.7 (0)	178.5 (0.2)	-	22.8	-	-	-	-	-	-	-	112.3 (0.6)
I37	61.1 (0.2)	34.2 (0.2)	178.2 (0.1)	-	28.3(0) /17.6(0.1)	-	8.2(0.2) / -	-	-	-	-	-	123.6 (0.2)
G38	48.4 (0)	-	175.8 (0)	-	-	-	-	-	-	-	-	-	110.2 (0.3)
I39	66.4 (0)	38.0 (0)	177.9 (0)	-	28.9(0) /17.1(0.1)	-	12.3(0.1) / -	-	-	-	-	-	122.6 (0.3)
K40	60.5 (0.1)				-		-	42.5 (0)	-	-	-	-	
L41	57.7 (0.1)	41.5 (0.1)	179	29.2	-	-	25.0	-	-	-	-	-	124.6
F42	61.7 (0.2)	39.7 (0.2)	178.9 (0.3)	138.6 (0.2)	-	-		-	-	-	-	-	123.1
K43	60.1 (0.1)	33.2 (0)			-		-		-	-	-	-	120.8
K44	59.4 (0)	33.4 (0.1)	177.7		-		-		-	-	-	-	117.7 (0)
F45	61.8 (0)	37.8		141.1 (0.2)	-	-	130.5(0.1)	-	-	-	-	-	120.3
T46	65.7 (0)	68.8 (0)	177.4 (0.1)	-	-24 (0.1)	-	-	-	-	-	-	-	108.8
S47	62.9(0)			-	-	-	-	-	-	-	-	-	115.9 (0.2)
^a K48				-			-		-	-	-	-	
A49	52.2(0.2)	21.1(0.1)	174(0)	-	-	-	-	-	-	-	-	-	119.3 (0.1)
S50	57.8(0.1)	66.7(0)	179.7(0.2)	-	-	-	-	-	-	-	-	-	110.9 (0.2)

^aK48 could not been assigned due to high peak overlap.

Figure 2S – Y21\Y24 C β -C γ region extracted from 2D ^{13}C - ^{13}C DARR spectra (100 ms) of intact M13 (blue) and fd (red) bacteriophages. For Y24 a chemical shift deviation of 0.4 ppm is observed. Acquisition and processing parameters are the same as in Figure 2,3 and 4 in the text.

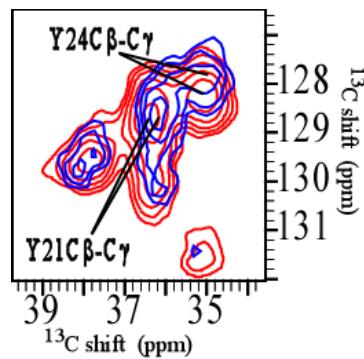
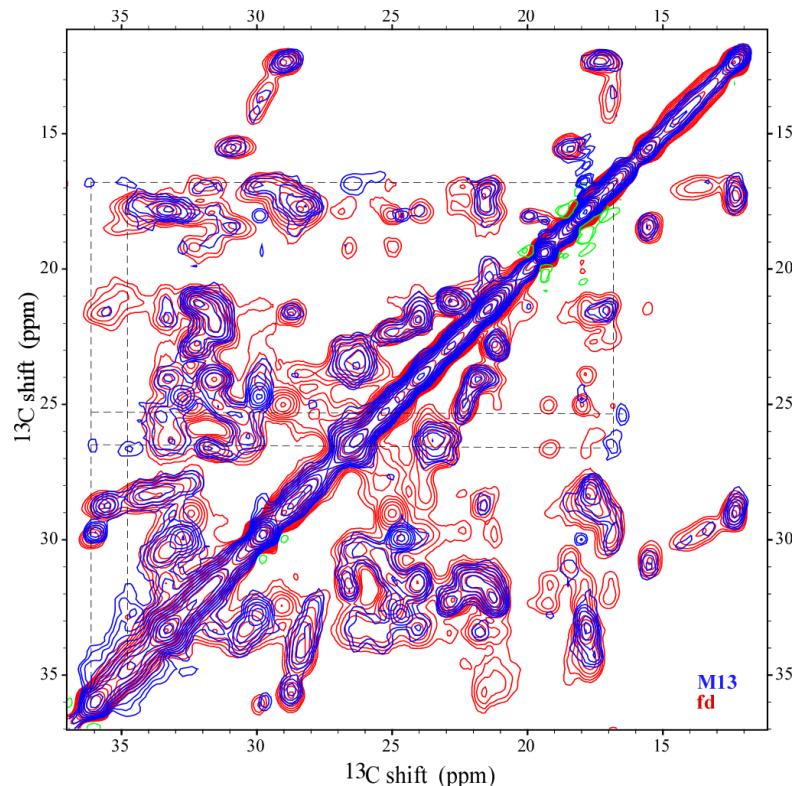


Figure S3 – Overlay of the aliphatic region in the DARR spectra (100 ms) of M13 (blue) and fd (red) showing the appearance of an additional pattern of crosspeaks (marked with dashed line) that exists only in M13. The chemical shifts of these resonances could possibly match Ile side chain carbons. As was noted in the text our assumption is that these correlations belong to Ile32. Acquisition and processing parameters are the same as in Figure 2, 3 and 4 in the text. Contour levels are shown from $\sim 9\sigma$ for fd and $\sim 8\sigma$ for M13 (σ being the noise root-mean-square determined by SPARKY), with each additional level multiplied by 1.4.



Full author list for references 36 and 78:

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