

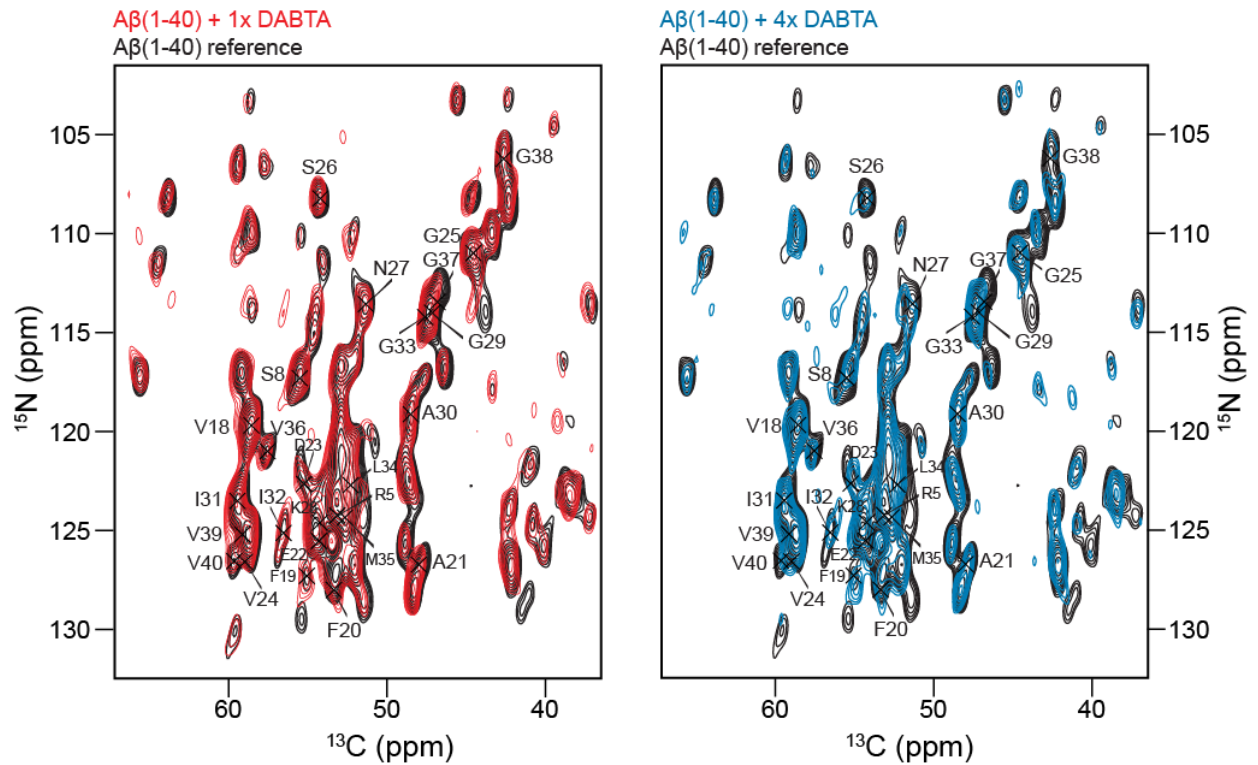
# ChemBioChem

## Supporting Information

### **Mapping the Binding Interface of PET Tracer Molecules and Alzheimer Disease A $\beta$ Fibrils by Using MAS Solid-State NMR Spectroscopy**

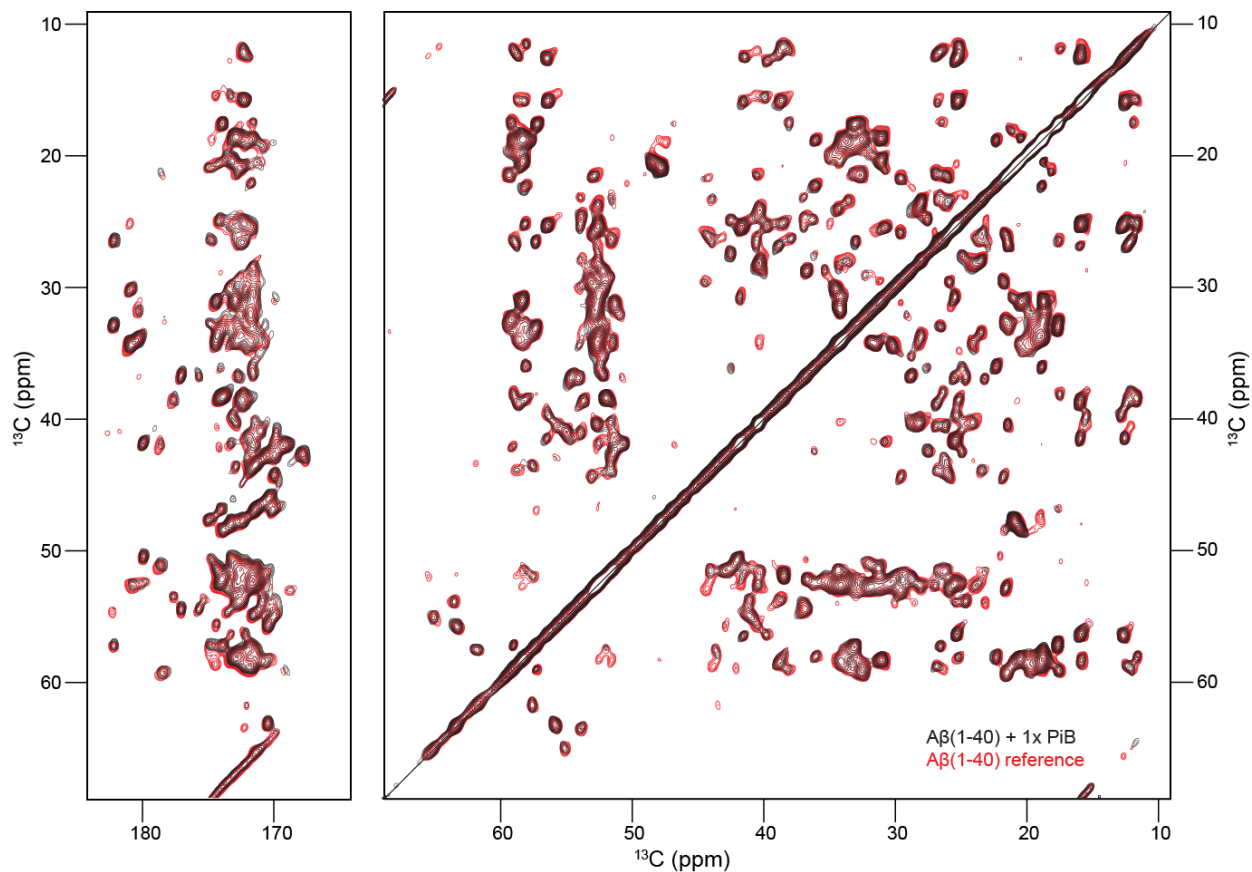
Zheng Niu, Riddhiman Sarkar, Michaela Aichler, Hans-Jürgen Wester, Behrooz Hooshyar Yousefi<sup>+,\*</sup> and Bernd Reif<sup>+,\*</sup> © 2020 The Authors. Published by Wiley-VCH Verlag GmbH & Co. KGaA. This is an open access article under the terms of the Creative Commons Attribution License, which permits use, distribution and reproduction in any medium, provided the original work is properly cited. This article is part of a Special Collection on the occasion of Horst Kessler's 80th birthday. To view the complete collection, visit our

## Supporting Information



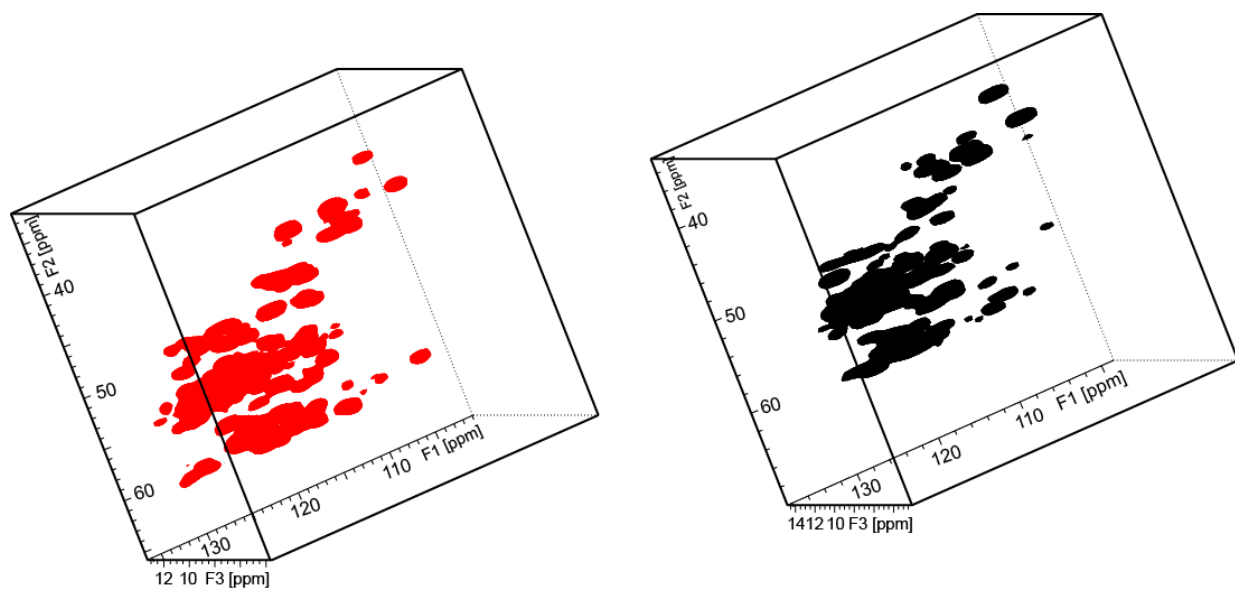
**Supporting figure 1.** Superposition of 2D NCACX spectra recorded for Aβ(1-40) reference fibrils (black) and Aβ(1-40) fibrils titrated with an equimolar amount of DABTA (red) and with a 4-fold molar excess of DABTA (right, blue). The experiments were carried out using a Bruker Avance III 750 MHz spectrometer. The MAS rotation frequency has been adjusted to 10 kHz. The PSD mixing time was set to 50 ms. The effective sample temperature was 10 °C.





**Supporting Figure 3.** Superposition of 2D  $^{13}\text{C}$ ,  $^{13}\text{C}$  PDSD spectra of  $^{13}\text{C}$ ,  $^{15}\text{N}$ -labeled  $\text{A}\beta(1-40)$  reference fibrils (black) and  $\text{A}\beta(1-40)$  fibrils in the presence of an equimolar amount of PiB (red).  $\text{A}\beta(1-40)$  fibrils were produced without seeding. As a consequence, two sets of resonances are observed in the spectra. For none of the two polymorphs, chemical shift changes can be observed upon addition of PiB. The experiments were acquired using a Bruker Avance III operating at a proton Larmor frequency of 750 MHz spectrometer. The MAS rotation frequency was adjusted to 10 kHz. The PDSD mixing time was set to 50 ms.





**Supporting figure 5.** 3D cube of hCANH experiments of A $\beta$ (1-40) reference fibrils (left) and A $\beta$ (1-40) fibrils in the presence of equimolar PiB (right).

**Table S1.** Chemical shift assignments for protonated A $\beta$ (1-40) fibrils.

aa	N	N $\zeta$	CO	C $\alpha$	C $\beta$	C $\gamma$	C $\delta$	C $\epsilon$	C $\zeta$
R5	124.24	-	-	53.14	31.76	26.10	42.08	-	158.13
D7	-	-	174.49	51.87	42.00	179.10	-	-	-
S8	117.31	-	172.03	55.50	65.61	-	-	-	-
Q15	-	-	170.13	53.09	31.94	34.84	180.76	-	-
V18	119.74	-	172.38	58.81	34.46	19.20 20.85	-	-	-
F19	127.31	-	170.64	55.10	42.19	-	-	-	-
F20	128.01	-	171.53	53.35	40.90	-	-	-	-
A21	126.72	-	173.78	47.97	19.37	-	-	-	-
E22	125.53	-	173.07	54.29	30.41	34.73	181.33	-	-
D23	122.65	-	173.54	55.24	37.89	182.71	-	-	-
V24	126.58	-	173.15	59.00	31.38	18.71 21.39	-	-	-
G25	111.00	-	170.44	44.72	-	-	-	-	-
S26	108.27	-	172.59	54.16	64.05	-	-	-	-
N27	113.63	-	172.63	51.21	37.18	175.10	-	-	-
K28	124.61	32.85	173.46	54.26	28.60	24.71	-	40.78	-
G29	114.00	-	171.63	47.20	-	-	-	-	-
A30	119.12	-	173.72	48.50	21.08	-	-	-	-
I31	123.52	-	172.79	59.46	38.42	26.71 17.87	12.45	-	-
I32	125.07	-	174.70	56.52	40.98	25.69 16.16	12.45	-	-
G33	114.22	-	170.51	47.57	-	-	-	-	-
L34	122.70	-	171.71	52.30	44.21	27.02	23.89	-	-
M35	124.35	-	172.13	52.67	37.16	29.16	-	-	-
V36	121.07	-	174.28	57.65	33.48	17.98	-	-	-
G37	113.46	-	171.11	46.81	-	-	-	-	-
G38	106.22	-	169.84	42.63	-	-	-	-	-
V39	125.20	-	172.82	59.17	33.20	20.00	-	-	-
V40	126.60	-	180.03	59.76	32.89	19.19 21.91	-	-	-



**Table S2.** Chemical shift assignments for deuterated A $\beta$ (1-40) reference fibrils, and A $\beta$ (1-40) fibrils in presence of  $^{13}\text{C}$ -Me-PiB.

aa	H <sup>N</sup>	N	C $\alpha$	C $\beta$	CO
A21	8.50 8.47	121.96 121.81	47.39 47.38	18.74 -	170.02 -
E22	8.30 -	119.17 -	52.02 -	- -	171.96 -
D23	9.86 -	126.64 -	51.63 -	35.16 -	173.22 -
V24	9.27 -	123.63 -	51.47 54.58	- -	171.85 171.10
G25	8.52 8.11	111.13 112.43	43.15 42.11	- -	173.00 173.45
S26	8.21 9.08	109.70 118.53	56.76 54.59	58.07 -	170.56 171.80
N27	9.01 8.21	118.49 115.37	51.94 55.13	- -	170.49 172.85
K28	8.83 9.10	125.63 128.74	53.12 52.65	30.69 -	172.44 171.61
G29	8.73 8.65	107.06 106.48	38.26 38.17	- -	172.75 171.44
A30	7.38 7.33	123.91 123.25	49.67 49.68	20.48 -	172.48 171.67
I31	9.46 9.39	121.95 121.47	58.44 58.33	36.09 -	171.29 -
I32	9.18 9.11	125.20 125.58	58.21 56.53	36.58 -	171.79 172.76
G33	8.07 8.73	112.71 116.61	42.42 42.44	- -	177.20 166.93
L34	8.58 8.61	128.95 128.57	50.72 50.73	43.41 -	171.91 171.25
M35	9.45 9.30	125.90 125.59	50.73 50.75	32.84 -	172.19 171.86
V36	8.57 8.56	127.19 126.97	58.05 58.09	29.73 -	175.48 173.73
G37	9.02 8.93	111.34 110.17	43.12 43.29	- -	174.63 169.28
G38	7.88 7.81	106.05 105.20	41.22 41.14	- -	175.43 167.79
V39	8.76 8.69	123.19 122.77	58.21 58.07	31.62 -	174.04 -
V40	9.29 -	132.34 -	58.40 -	- -	- -

## References

- [1] E. Barbet-Massin, A. J. Pell, J. S. Retel, L. B. Andreas, K. Jaudzems, W. T. Franks, A. J. Nieuwkoop, M. Hiller, V. Higman, P. Guerry, A. Bertarello, M. J. Knight, M. Felletti, T. Le Marchand, S. Kotelovica, I. Akopjana, K. Tars, M. Stoppini, V. Bellotti, M. Bolognesi, S. Ricagno, J. J. Chou, R. G. Griffin, H. Oschkinat, A. Lesage, L. Emsley, T. Herrmann, G. Pintacuda, *J. Am. Chem. Soc.* 2014, 136, 12489-12497.