

Supplementary Material

Newly Discovered Irbesartan Disinfection Byproducts by Chlorination: Investigating Potential Environmental Toxicity

Antonietta Siciliano, Antonio Medici, Marco Guida, Giovanni Libralato, Lorenzo Saviano,
Lucio Previtera, Giovanni Di Fabio and Armando Zarrelli*

* Correspondence: zarrelli@unina.it; Tel.: +39-081-674-472

Spectral Data

DBP7: *Tetrazolo[1,5-f]phenanthridine-6-carbaldehyde*; white powder; ^1H and ^{13}C NMR in CDCl_3 (see Table S1); MS-TOF (positive ions): m/z calculated for $\text{C}_{14}\text{H}_8\text{N}_4\text{O}$ 248.07 $[\text{M}]^+$; found 249.11 $[\text{M} + \text{H}]^+$ (72%); 220.23 (15%); UV (MeOH) $\lambda_{\text{max}}/\text{nm}$ 236 ($\epsilon/\text{dm}^3 \text{ mol}^{-1} \text{ cm}^{-1}$, 41,000), 253 (28,000).

DBP8: *1-Amino-N-(2-hydroxypentanoyl)-N-(tetrazolo[1,5-f]phenanthridin-6-ylmethyl)cyclopentanecarboxamide*; white powder; ^1H and ^{13}C NMR in $\text{CDCl}_3/\text{CD}_3\text{OD}$ 9:1 (see Table S2); MS-TOF (positive ions): m/z calculated for $\text{C}_{25}\text{H}_{28}\text{N}_6\text{O}_3$ 460.22 $[\text{M}]^+$; found 461.23 $[\text{M} + \text{H}]^+$ (58%); 443.26 (18%). UV (MeOH) $\lambda_{\text{max}}/\text{nm}$ 239 ($\epsilon/\text{dm}^3 \text{ mol}^{-1} \text{ cm}^{-1}$, 41,000), 254 (36,000).

DBP9: *9-((2'-(2H-tetrazol-5-yl)-[1,1'-biphenyl]-4-yl)methyl)-7-hydroxy-7-propyl-6,9-diazaspiro[4.5]decane-8,10-dione*; white powder; ^1H and ^{13}C NMR in CD_3OD (see Table S3); MS-TOF (positive ions): m/z calculated for $\text{C}_{25}\text{H}_{28}\text{N}_6\text{O}_3$ 460.22 $[\text{M}]^+$; found 461.24 $[\text{M} + \text{H}]^+$ (78%). UV (MeOH) $\lambda_{\text{max}}/\text{nm}$ 240 ($\epsilon/\text{dm}^3 \text{ mol}^{-1} \text{ cm}^{-1}$, 37,000), 253 (35,000).

DBP10: *N-((2'-(2H-Tetrazol-5-yl)-[1,1'-biphenyl]-4-yl)methyl)-1-amino-N-(2-hydroxypentanoyl)cyclopentanecarboxamide*; white powder; ^1H and ^{13}C NMR in CDCl_3 (see Table S4); MS-TOF (positive ions): m/z calculated for $\text{C}_{25}\text{H}_{30}\text{N}_6\text{O}_3$ 462.24 $[\text{M}]^+$; found 463.26 $[\text{M} + \text{H}]^+$ (61%); 645.27 (33%); UV (MeOH) $\lambda_{\text{max}}/\text{nm}$ 240 ($\epsilon/\text{dm}^3 \text{ mol}^{-1} \text{ cm}^{-1}$, 42,000), 255 (36,000).

DBP11: *1-Amino-N-pentanoyl-N-((2'-(trichloromethyl)-[1,1'-biphenyl]-4-yl)methyl)-cyclopentanecarboxamide*; white powder; ^1H and ^{13}C NMR in $\text{CDCl}_3/\text{CD}_3\text{OD}$ 9:1 (see Table S5); MS-TOF (positive ions): m/z calculated for $\text{C}_{25}\text{H}_{29}\text{Cl}_3\text{N}_2\text{O}_2$ 494.13 $[\text{M}]^+$; found 500.17 $[\text{M} + 6]^+$ (2%); 499.13 $[\text{M} + 5]^+$ (7%); 498.13 $[\text{M} + 4]^+$ (27%); 497.14 $[\text{M} + 3]^+$ (28%); 496.15 $[\text{M} + 2]^+$ (92%); 495.88 $[\text{M} + \text{H}]^+$ (27%); 494.13 $[\text{M}]^+$ (87%).

DBP12: *N-((2'-(2H-Tetrazol-5-yl)-[1,1'-biphenyl]-4-yl)methyl)-1-amino-N-pentanoyl cyclopentanecarboxamide*; white powder; ^1H and ^{13}C NMR in $\text{CDCl}_3/\text{CD}_3\text{OD}$ 9:1 (see Table S6); MS-TOF (positive ions): m/z calculated for $\text{C}_{25}\text{H}_{30}\text{N}_6\text{O}_2$ 446.24 $[\text{M}]^+$; found 447.25 $[\text{M} + \text{H}]^+$ (57%); 361.26 (12%); UV (MeOH) $\lambda_{\text{max}}/\text{nm}$ 239 ($\epsilon/\text{dm}^3 \text{ mol}^{-1} \text{ cm}^{-1}$, 47,000), 250 (32,000).

DBP13: *N-(1-Aminocyclopentanecarbonyl)-2'-(2H-tetrazol-5-yl)-[1,1'-biphenyl]-4-carboxamide*; white powder; ^1H and ^{13}C NMR in CD_3OD (see Table S7); MS-TOF (positive ions): m/z calculated for $\text{C}_{20}\text{H}_{20}\text{N}_6\text{O}_2$ 376.16 $[\text{M}]^+$; found 377.18 $[\text{M} + \text{H}]^+$ (38%); UV (MeOH) $\lambda_{\text{max}}/\text{nm}$ 237 ($\epsilon/\text{dm}^3 \text{ mol}^{-1} \text{ cm}^{-1}$, 44,000), 256 (31,000).

DBP14: *2'-(2H-Tetrazol-5-yl)-[1,1'-biphenyl]-4-carbaldehyde*; White powder; ^1H and ^{13}C NMR in CDCl_3 (see Table S8); MS-TOF (positive ions): m/z calculated for $\text{C}_{14}\text{H}_{10}\text{N}_4\text{O}$ 250.09 $[\text{M}]^+$; found 251.11 $[\text{M} + \text{H}]^+$ (68%); 222.12 (25%); UV (MeOH) $\lambda_{\text{max}}/\text{nm}$ 236 ($\epsilon/\text{dm}^3 \text{ mol}^{-1} \text{ cm}^{-1}$, 38,000), 253 (23,000).

DBP15: *N-((2'-(2H-Tetrazol-5-yl)-[1,1'-biphenyl]-4-yl)(hydroxy)methyl)-1-pentanamidocyclopentanecarboxamide*; white powder; ^1H and ^{13}C NMR in CDCl_3 (see Table S9); MS-TOF (positive ions): m/z calculated for $\text{C}_{25}\text{H}_{30}\text{N}_6\text{O}_3$ 462.24 $[\text{M}]^+$; found 463.26 $[\text{M} + \text{H}]^+$ (37%); UV (MeOH) $\lambda_{\text{max}}/\text{nm}$ 240 ($\epsilon/\text{dm}^3 \text{ mol}^{-1} \text{ cm}^{-1}$, 41,000), 254 (36,000).

DBP16: *N-(1-Pentanamidocyclopentanecarbonyl)-2'-(2H-tetrazol-5-yl)-[1,1'-biphenyl]-4-carboxamide*; white powder; ^1H and ^{13}C NMR in CDCl_3 (see Table S10); MS-TOF (positive ions): m/z calculated for $\text{C}_{25}\text{H}_{28}\text{N}_6\text{O}_3$ 460.22 $[\text{M}]^+$; found 461.23 $[\text{M} + \text{H}]^+$ (31%); UV (MeOH) $\lambda_{\text{max}}/\text{nm}$ 237 ($\epsilon/\text{dm}^3 \text{ mol}^{-1} \text{ cm}^{-1}$, 40,000), 252 (27,000).

DBP17: *N-((2'-(2H-Tetrazol-5-yl)-[1,1'-biphenyl]-4-yl)(hydroxy)methyl)pentanamide*; white powder; NMR spectra conform to those recorded for the available standard (Luongo et al., 2020); UV (MeOH) $\lambda_{\text{max}}/\text{nm}$ 236 ($\epsilon/\text{dm}^3 \text{ mol}^{-1} \text{ cm}^{-1}$, 32,000), 253 (21,000).

DBP18: 4'-((2-Butyl-4-oxo-1,3-diazaspiro[4.4]non-1-en-3-yl)methyl)-[1,1'-biphenyl]-2-carboxamide; white powder; ^1H and ^{13}C NMR in CDCl_3 (see Table S11); MS-TOF (positive ions): m/z calculated for $\text{C}_{25}\text{H}_{29}\text{N}_3\text{O}_2$ 403.52 $[\text{M}]^+$; found 404.54 $[\text{M} + \text{H}]^+$ (42%).

Table S1. ^1H , ^{13}C and 2D NMR data of DBP7

Position	residue	$^{13}\text{C}^a$	$^1\text{H}^a$ (J in Hz)	^1H - ^1H COSY	^1H - ^{13}C HMBC
11	CH	190.01	10.30 (s, 1H)		129.68, 127.19, 125.41
12	C	129.68			
13	CH	127.19	9.19 (d, J = 1.7, 1H)	8.28	190.01, 125.41
14	C	148.81			
15	C	127.19			
16	CH	126.57	8.73 (d, J = 8.3, 1H)	8.28	125.41
17	CH	125.41	8.28 (dd, J = 8.3; 2.0, 1H)	8.73, 9.19	169.01, 127.19, 126.57
18	C	136.92			
19	CH	120.18	8.61 (d, J = 8.5, 1H)	7.98, 7.92	127.19, 136.92, 130.95, 119.70
20	CH	125.09	7.98 (dt, J = 7.5; 1.5, 1H)	8.61, 7.92	136.92, 124.03
21	CH	130.95	7.92 (dt, J = 7.4; 1.3, 1H)	7.98, 8.86	120.18, 119.70
22	CH	124.03	8.86 (dd, J = 8.3; 1.3, 1H)	7.98, 7.92	136.92, 125.09, 119.70, 150.62
23	C	119.70			
24	C	150.62			

^aChemical shifts in ppm.

Table S2. ^1H , ^{13}C and 2D NMR data of DBP8

Position	residue	$^{13}\text{C}^a$	$^1\text{H}^a$ (J in Hz)	^1H - ^1H COSY	^1H - ^{13}C HMBC
1,2	CH_2	25.82	1.89 (m, 2H) 1.91 (m, 2H)	1.91	25.82, 37.00, 77.20
3,5	CH_2	37.00	1.91 (m, 4H)	1.89	25.82, 37.00, 186.43
4	C	77.20			
7	C	159.43			
9	C	186.43			
11	CH_2	43.30	4.94 (d, J = 16.6, 1H) 5.16 (d, J = 16.6, 1H)	5.16, 4.94	159.43, 186.43, 114.89, 125.77
12	C	138.78			
13	CH	114.89	8.36 (d, J = 1.5, 1H)	8.45	43.30, 125.77
14	C	129.20			
15	C	121.86			
16	CH	124.93	8.49 (d, J = 8.2, 1H)	8.45	138.78, 129.20, 121.86, 129.40
17	CH	125.77	8.45 (dd, J = 8.0; 1.7, 1H)	8.36, 8.49	43.30, 114.89, 121.86
18	C	129.40			
19	CH	123.06	7.50 (d, J = 7.8, 1H)	7.74	121.86, 129.58, 118.05
20	CH	132.23	7.74, t, J = 7.9, 1H)	7.50, 7.85	129.40, 126.38
21	CH	129.58	7.85, t, J = 7.7, 1H)	7.74, 8.64	123.06, 118.05
22	CH	126.38	8.64 (d, J = 7.4, 1H)	7.85	129.40, 132.23, 147.16
23	C	118.05			
24	C	147.16			
29	CH	52.50	4.34 (t, J = 7.7, 2H)	1.27	159.43, 35.52, 19.37
30	CH_2	35.52	1.27 (m, 2H)	4.34, 1.15	159.43, 52.50, 19.37, 12.71
31	CH_2	19.37	1.15 (m, 2H)	1.27, 0.69	52.50, 35.52, 12.71
32	CH_3	12.71	0.69 (t, J = 7.5, 3H)	1.15	19.37, 12.71

^aChemical shifts in ppm.

Table S3. ^1H , ^{13}C and 2D NMR data of DBP9

Position	residue	$^{13}\text{C}^a$	$^1\text{H}^a$ (J in Hz)	^1H - ^1H COSY	^1H - ^{13}C HMBC
1,2	CH_2	26.11	2.02 (m, 4H)	1.89	26.11, 37.72, 77.0
3,5	CH_2	37.72	1.89 (m, 2H) 2.02 (m, 2H)	2.02 1.89	26.11, 37.72, 187.48
4	C	77.0			
7	C	158.02			
9	C	187.48			
11	CH_2	46.84	5.25 (s, 2H)		158.02, 187.48, 127.48, 127.48
12	C	137.21			
13,17	CH	127.48	7.08 (d, J = 8.3, 2H)	7.07	46.84, 138.26, 127.48
14,16	CH	129.07	7.07 (d, J = 8.3, 2H)	7.08	137.21, 129.07, 140.47
15	C	138.26			
18	C	140.47			
19	CH	130.80	7.46 (d, J = 7.3, 1H)	7.62	138.26, 128.53, 122.03
20	CH	131.49	7.62 (t, J = 7.7, 1H)	7.46, 7.57	140.47, 130.62
21	CH	128.53	7.57 (t, J = 7.4, 1H)	7.62, 8.23	130.80, 122.03
22	CH	130.62	8.23 (d, J = 7.4, 1H)	7.57	140.47, 131.49, 154.38
23	C	122.03			
24	C	154.38			
29	C	83.09			
30	CH_2	46.84	2.67 (m, 2H)	1.88	158.02, 18.23, 13.54
31	CH_2	18.23	1.88 (m, 2H)	2.67, 1.09	83.09, 46.84, 13.54
32	CH_3	13.54	1.09 (t, J = 8.1, 3H)	1.88	46.84, 18.23

^aChemical shifts in ppm.

Table S4. ^1H , ^{13}C and 2D NMR data of DBP10

Position	residue	$^{13}\text{C}^a$	$^1\text{H}^a$ (J in Hz)	^1H - ^1H COSY	^1H - ^{13}C HMBC
1,2	CH_2	26.14	1.81 (m, 2H) 1.93 (m, 2H)	1.93 1.81	26.14, 37.46, 77.0
3,5	CH_2	37.46	1.93 (m, 4H)	1.81	26.14, 37.46, 186.67
4	C	77.0			
7	C	159.67			
9	C	186.67			
11	CH_2	43.48	4.66 (d, J = 16.5, 1H) 4.98 (d, J = 16.5, 1H)	4.98, 4.66	159.67, 186.67, 126.74
12	C	135.90			
13,17	CH	126.74	7.06 (d, J = 8.3, 2H)	7.09	43.48, 126.74, 139.12
14,16	CH	129.76	7.09 (d, J = 8.3, 2H)	7.06	135.90, 129.76, 140.90
15	C	139.12			
18	C	140.90			
19	CH	131.19	7.41 (dd, J = 7.7; 1.2, 1H)	7.57, 7.48	139.12, 128.13, 123.04
20	CH	131.70	7.57 (dt, J = 7.6; 1.2, 1H)	7.41, 7.48	140.90, 131.19
21	CH	128.13	7.48 (dt, J = 7.3; 1.3, 1H)	7.57, 7.79	131.19, 123.04
22	CH	131.19	7.79 (d, J = 7.5, 1H)	7.48	140.90, 131.70, 155.60
23	C	123.04			
24	C	155.60			
29	CH	52.81	4.31 (dd, 8.3; 6.6, 1H)	2.03, 1.39	159.67, 19.54
30	CH_2	35.71	2.03 (m, 2H)	4.31, 1.28, 1.39	159.67, 13.10
31	CH_2	19.54	1.28 (sest, J = 6.7, 1H) 1.39 (sest, J = 6.7, 1H)	2.03, 0.78	52.81, 13.10
32	CH_3	13.10	0.78 (t, J = 7.3, 3H)	1.28, 1.39	19.54, 35.71

^aChemical shifts in ppm.

Table S5. ¹H, ¹³C and 2D NMR data of DBP11

Position	residue	¹³ C ^a	¹ H ^a (J in Hz)	¹ H- ¹ H COSY	¹ H- ¹³ C HMBC
1,2	CH ₂	26.93	1.99 (m, 4H)	1.96, 2.12	38.41, 73.05
3,5	CH ₂	38.41	1.96 (m, 2H) 2.12 (m, 2H)	1.99, 2.12 1.96, 1.99	26.93, 73.05, 180.05
4	C	73.05			
7	C	176.01			
9	C	180.05			
11	CH ₂	44.40	4.93 (s, 2H)		126.98, 134.75, 176.01, 180.05
12	C	134.75			
13,17	CH	126.98	7.23 (d, J = 8.2, 2H)	7.41	44.40, 126.98, 141.54
14,16	CH	132.14	7.41 (d, J = 8.2, 2H)	7.23	132.14, 134.75, 141.89
15	C	141.51			
18	C	141.89			
19	CH	131.47	7.50 (m, 1H)	7.19, 7.51	127.05, 128.82, 141.51
20	CH	134.75	7.51 (m, 1H)	7.19, 7.50	127.72, 141.89
21	CH	128.82	7.19 (m, 1H)	7.51, 8.24	127.05, 131.47
22	CH	127.72	8.24 (d, J = 7.8, 1H)	7.19	97.40, 134.75, 141.89
23	C	127.05			
24	C	97.40			
29	CH ₂	37.85	2.49 (t, J = 7.0, 2H)	1.35, 1.54	23.19, 28.32, 176.01
30	CH ₂	28.32	1.54 (quint, J = 6.8, 2H)	1.35, 2.49	13.93, 23.19, 176.01
31	CH ₂	23.19	1.35 (sest, J = 6.8, 2H)	0.89, 1.54	13.93, 28.32, 37.85
32	CH ₃	13.93	0.89 (t, J = 7.4, 3H)	1.35, 1.54	23.19, 28.32

^aChemical shifts in ppm.

Table S6. ¹H, ¹³C and 2D NMR data of DBP12

Position	residue	¹³ C ^a	¹ H ^a (J in Hz)	¹ H- ¹ H COSY	¹ H- ¹³ C HMBC
1,2	CH ₂	28.34	1.65 (m, 4H)	2.05, 2.20	28.34, 39.32, 79.3
3,5	CH ₂	39.32	2.05 (m, 2H) 2.20 (m, 2H)	1.65, 2.20 1.65, 2.05	28.34, 39.32, 178.59
4	C	79.3			
7	C	180.55			
9	C	178.59			
11	CH ₂	47.06	4.31 (s, 2H)		180.55, 178.59, 131.42
12	C	142.16			
13,17	CH	131.42	6.96 (d, J = 8.1, 2H)	7.15	47.06, 145.44, 131.42
14,16	CH	132.99	7.15 (d, J = 8.3, 2H)	6.96	142.16, 132.99, 141.88
15	C	145.44			
18	C	141.88			
19	CH	131.76	7.43 (d, J = 8.0, 1H)	7.58	145.44, 134.62, 126.65
20	CH	135.19	7.58 (t, J = 7.6, 1H)	7.43, 7.50	141.88, 134.52
21	CH	134.62	7.50 (t, J = 7.4, 1H)	7.58, 7.85	131.76, 126.65
22	CH	134.52	7.85 (d, J = 7.3, 1H)	7.50	141.88, 135.19, 159.06
23	C	126.65			
24	C	159.06			
29	CH ₂	37.38	2.41 (t, J = 8.1, 2H)	1.50	180.55, 30.40, 26.31
30	CH ₂	30.40	1.50 (quint, J = 8.0, 2H)	2.41, 1.29	37.38, 30.40, 17.56
31	CH ₂	26.31	1.29 (sest, J = 7.5, 2H)	1.50, 0.84	37.38, 30.40, 17.56
32	CH ₃	17.56	0.84 (t, J = 7.5, 3H)	1.29	30.40, 26.31

^aChemical shifts in ppm.

Table S7. ^1H , ^{13}C and 2D NMR data of DBP13

Position	residue	$^{13}\text{C}^a$	$^1\text{H}^a$ (J in Hz)	^1H - ^1H COSY	^1H - ^{13}C HMBC
1,2	CH_2	27.06	1.94 (m, 4H)	1.92, 1.99	27.06, 38.67, 77.27
3,5	CH_2	38.67	1.92 (m, 2H) 1.99 (m, 2H)	1.94, 1.99 1.92, 1.94	27.06, 38.67, 192.25
4	C	77.27			
9	C	192.25			
11	C	166.70			
12	C	127.40			
13,17	CH	128.61	7.84 (d, J = 8.4, 2H)	7.28	166.70, 146.21, 128.61
14,16	CH	130.87	7.28 (d, J = 8.4, 2H)	7.84	127.40, 130.87, 142.10
15	C	146.21			
18	C	142.10			
19	CH	131.49	7.56 (d, J = 7.4, 1H)	7.63	146.21, 129.37, 128.05
20	CH	131.58	7.63 (t, J = 7.5, 1H)	7.56, 7.55	142.10, 131.84
21	CH	129.37	7.55 (t, J = 7.8, 1H)	7.63, 7.65	131.49, 128.05
22	CH	131.84	7.65 (d, J = 7.8, 1H)	7.55	142.10, 131.58, 159.35
23	C	128.05			
24	C	159.35			

^aChemical shifts in ppm.

Table S8. ¹H, ¹³C and 2D NMR data of DBP14

Position	residue	¹³ C ^a	¹ H ^a (J in Hz)	¹ H- ¹ H COSY	¹ H- ¹³ C HMBC
11	CH	191.54	10.06 (s, 1H)		135.88, 130.67
12	C	135.88			
13,17	CH	130.67	7.94 (d, J = 8.4, 2H)	7.44	191.54, 145.57, 130.67
14,16	CH	129.83	7.44 (d, J = 8.4, 2H)	7.94	135.88, 129.83, 139.93
15	C	145.57			
18	C	139.93			
19	CH	130.67	7.51 (d, J = 7.4, 1H)	7.69	145.57, 129.14, 122.37
20	CH	131.61	7.69 (t, J = 7.4, 1H)	7.51, 7.64	139.93, 130.88
21	CH	129.14	7.64 (t, J = 7.4, 1H)	7.69, 8.13	130.67, 122.37
22	CH	130.88	8.13 (d, J = 7.4, 1H)	7.64	139.93, 131.61, 155.09
23	C	122.37			
24	C	155.09			

^aChemical shifts in ppm.

Table S9. ¹H, ¹³C and 2D NMR data of DBP15

Position	residue	¹³ C ^a	¹ H ^a (J in Hz)	¹ H- ¹ H COSY	¹ H- ¹³ C HMBC
1,2	CH ₂	27.52	1.62 (m, 4H)	1.94	27.52, 31.88, 77.0
3,5	CH ₂	31.88	1.94 (m, 4H)	1.62	27.52, 31.88, 169.00
4	C	77.0			
7	C	172.37			
9	C	169.00			
11	CH	83.04	6.66 (s, 1H)		172.37, 169.00, 135.15, 127.53
12	C	135.15			
13,17	CH	127.53	7.26 (d, J = 8.3, 2H)	7.28	83.04, 133.02, 127.53
14,16	CH	129.63	7.28 (d, J = 8.3, 2H)	7.26	135.15, 129.63, 140.15
15	C	133.02			
18	C	140.15			
19	CH	130.83	7.43 (dd, J = 7.4; 1.4, 1H)	7.61, 7.56	133.02, 128.73, 122.01
20	CH	131.54	7.61 (dd, J = 7.6; 7.4, 1H)	7.43, 7.56	140.15, 130.83
21	CH	128.73	7.56 (dd, J = 7.6; 7.4, 1H)	7.61, 8.19	130.83, 122.01
22	CH	130.83	8.19 (dd, J = 7.6; 1.8, 1H)	7.56, 7.61	140.15, 131.54, 153.90
23	C	122.01			
24	C	153.90			
29	CH ₂	36.13	2.95 (t, J = 7.5, 2H)	1.62	172.37, 25.80, 22.16
30	CH ₂	25.80	1.62 (m, 4H, 2H)	2.95, 1.36	172.37, 36.13, 22.16, 13.74
31	CH ₂	22.16	1.36 (quint, J = 7.6, 2H)	1.62, 0.92	36.13, 25.80, 13.74
32	CH ₃	13.74	0.92 (t, J = 7.6, 3H)	1.36	25.80, 22.16

^aChemical shifts in ppm.

Table S10. ¹H, ¹³C and 2D NMR data of DBP16

Position	residue	¹³ C ^a	¹ H ^a (J in Hz)	¹ H- ¹ H COSY	¹ H- ¹³ C HMBC
1,2	CH ₂	24.20	1.72 (m, 4H)	1.98, 2.33	24.20, 36.16, 68.29
3,5	CH ₂	36.16	1.98 (m, 2H) 2.33 (m, 2H)	1.72, 2.33 1.72, 1.98	24.20, 36.16, 168.48
4	C	68.29			
7	C	176.73			
9	C	168.48			
11	C	168.42			
12	C	132.36			
13,17	CH	127.53	7.60 (d, J = 8.2, 2H)	7.11	168.42, 143.15, 127.53
14,16	CH	129.20	7.11 (d, J = 8.2, 2H)	7.60	132.36, 129.20, 140.52
15	C	143.15			
18	C	140.52			
19	CH	130.53	7.35 (d, J = 7.5, 1H)	7.58	143.15, 127.62, 123.32
20	CH	130.77	7.58 (dd, J = 7.6; 7.5, 1H)	7.35, 7.45	140.52, 131.15
21	CH	127.62	7.45 (dd, J = 7.6; 7.4, 1H)	7.58, 7.77	130.53, 123.32
22	CH	131.15	7.77 (dd, J = 7.6; 1.4, 1H)	7.45, 7.58	140.52, 130.77, 154.13
23	C	123.32			
24	C	154.13			
29	CH ₂	37.13	2.71 (t, J = 8.2, 2H)	1.54	176.73, 26.21, 22.17
30	CH ₂	26.21	1.54 (quint, J = 8.1, 2H)	2.71, 1.30	176.73, 37.13, 22.17, 13.75
31	CH ₂	22.17	1.30 (sest, J = 8.1, 2H)	1.54, 0.86	37.13, 26.21, 13.75
32	CH ₃	13.75	0.86 (t, J = 7.6, 3H)	1.30	26.21, 22.17

^aChemical shifts in ppm.

Table S11. ^1H , ^{13}C and 2D NMR data of DBP18

Position	residue	$^{13}\text{C}^a$	$^1\text{H}^a$ (J in Hz)	^1H - ^1H COSY	^1H - ^{13}C HMBC
1,2	CH_2	27.04	1.98 (m, 4H)	1.81, 2.02	77.55
3,5	CH_2	38.41	1.81 (m, 2H) 2.02 (m, 2H)	1.98, 2.02 1.81, 1.98	77.55, 187.92
4	C	77.55			
7	C	165.32			
9	C	187.92			
11	C	44.22			
12	C	137.01			
13,17	CH	127.62	7.23 (d, J = 8.0, 2H)	7.29	44.22, 127.62, 142.36
14,16	CH	130.08	7.29 (d, J = 8.0, 2H)	7.23	130.08, 137.01, 143.02
15	C	142.36			
18	C	143.02			
19	CH	131.74	7.38 (d, J = 7.7, 1H)	7.45, 7.58	128.52, 137.01, 142.36
20	CH	132.56	7.58 (t, J = 7.8, 1H)	7.38, 7.45	130.73, 143.02
21	CH	128.52	7.45 (t, J = 7.7, 1H)	7.58, 7.78	131.74, 137.01
22	CH	130.73	7.78 (d, J = 8.0, 1H)	7.45, 7.58	132.56, 143.02, 170.51
23	C	137.01			
24	C	170.51			
29	CH_2	29.50	2.50 (t, J = 8.2, 2H)	1.31, 1.52	23.73, 29.45, 165.32
30	CH_2	29.45	1.52 (quint, J = 8.0, 2H)	1.31, 2.50	13.97, 23.73, 29.50
31	CH_2	23.73	1.31 (sest, J = 8.0, 2H)	0.89, 1.52	13.97, 29.45
32	CH_3	13.97	0.89 (t, J = 7.6, 3H)	1.31, 1.52	23.73, 29.45

^aChemical shifts in ppm.