Supplementary Information

Isolation and structure elucidation of natural products of three soft corals and a sponge from the coast of Madagascar

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Content

Table S1. Comparison of ^1H and ^{13}C NMR data and optical rotation values of (+)-(7*S*,8*S*)-epoxy-7,8-dihydrocembrene C [(+)-**1**] from the soft corals *Sarcophyton stellatum* and *Sarcophyton ehrenbergi* [1]

Position	**^1H NMR**		**^{13}C NMR**	
	from *Sarcophyton stellatum* δ_H (*J* in Hz) (600 MHz, $CDCl_3$)	from *Sarcophyton ehrenbergi* [1] δ_H m (*J* in Hz) (500 MHz, $CDCl_3$)	from *Sarcophyton stellatum* δ_C (150 MHz, $CDCl_3$)	from *Sarcophyton ehrenbergi* [1] δ_C (125 MHz, $CDCl_3$)
1			148.16	148.2
2	6.03 d (10.9)	6.01 d (10.9)	118.40	118.4
3	5.96 dq (11.3, 1.1)	5.95 d (10.9)	121.40	121.4
4			134.21	134.2
5a+5b	2.17–2.31 m	2.23–2.24 m	35.75	35.8
6a 6b	1.77–1.83 m 1.66–1.72 m	2.17–2.18 m 1.97–1.99 m	25.72	37.4
7	2.84 t (5.6)	2.82–2.84 m	61.61	61.6
8			60.09	60.1
9a 9b	1.92 ddd (14.3, 7.5, 2.3) 1.51 ddd (13.9, 10.5, 3.0)	1.78–1.80 m 1.67–1.68 m	37.48	25.7
10a+10b	1.98–2.06 m	1.99–2.01 m	22.49	22.5

11	5.06 t (7.1)	5.05 t (6.3)	125.54	125.5
12			135.63	135.6
13a 13b	2.17–2.31 m 1.98–2.06 m	2.18–2.19 m 2.00–2.01 m	39.44	39.5
14a+14b	2.17–2.31 m	2.24–2.25 m	28.23	28.2
15	2.33 sp (6.7)	2.31 –2.32 m	34.89	34.9
16+17	1.04 d (7.1) 1.06 d (6.8)	1.04 d (7.2) 1.04 d (7.2)	22.14 22.32	22.1 22.3
18	1.75 s	1.74 s	17.10	17.1
19	1.26 s	1.24 s	17.84	17.8
20	1.58 d (0.8)	1.57 s	16.95	17.0
$[\alpha]_D$ (T [° C])	+44.6 (20, c 0.5, MeOH)	+19 (25, c 0.5, $CHCl_3$)		

Table S2. Comparison of ^{1}H and ^{13}C NMR data and optical rotation values of (1*E*,3*E*,11*E*)-7,8-epoxycembra-1,3,11,15-tetraene (**2**) from *Sarcophyton stellatum* and *Sarcophyton crassocaule*[2]

Position	**^{1}H NMR**		**^{13}C NMR**	
	(+)-**2** from *Sarcophyton stellatum* δ_H (J in Hz) (600 MHz, $CDCl_3$)	(–)-**2** from *Sarcophyton crassocaule* [2] δ_H m (J in Hz) (100 MHz, $CDCl_3$)	(+)-**2** from *Sarcophyton stellatum* δ_C (150 MHz, $CDCl_3$)	(–)-**2** from *Sarcophyton crassocaule* [2] δ_C m [a] (15 MHz, $CDCl_3$)
1			139.81	139.6 s
2	6.39 d (10.9)	6.30 (12)	122.58	122.8 d
3	6.10 dq (11.1, 1.3)	6.05 (12)	122.24	122.8 d
4			137.70	137.0 s
5a+5b	2.20–2.35 m		36.10	36.4 t
6a	1.79–1.86 m		25.62	27.1 t
6b	1.68–1.74 m			
7	2.83 t (5.6)	2.65 t (6)	61.03	59.5 d
8			60.16	58.9 s
9a	1.89 ddd (14.4, 7.7, 2.6)		37.04	37.0 t
9b	1.60 ddd (14.3, 9.4, 3.0)			
10a+10b	1.95–1.99 m		22.28	22.4 t

11	5.02–5.06 m	5.00 m	126.00	126.5 s
12			135.84	135.4 s
13a 13b	2.20–2.35 m 2.05 dt (13.6, 6.5)		39.12	39.1 t
14a+14b	2.54 t (6.4)		22.81	25.9 t
15			143.28	142.9 s
16	1.96 s	1.92 s	21.22	24.0 q
17a 17b	5.08 s 4.98 s	5.00 s 4.90 s	112.15	112.4 t
18	1.81 s	1.80 s	17.23	17.5 q
19	1.27 s	1.20 s	18.21	21.4 q
20	1.55 d (1.1)	1.52 s	17.31	18.7 q
$[\alpha]_D$ (T [° C])	+12.0 (20, c 0.5, MeOH)	–14.4 (c 0.1, $CHCl_3$)		

[a] allocated according to our assignment

Table S3. Comparison of ^{1}H and ^{13}C NMR data and optical rotation values of (+)-(7*R*,8*R*,14*S*,1*Z*,3*E*,11*E*)-14-acetoxy-7,8-epoxycembra-1,3,11-triene [(+)-**3**] from the soft corals *Sarcophyton stellatum* and *Sarcophyton trocheliophorum*[3].

Position	**^{1}H NMR**		**^{13}C NMR**	
	from *S. stellatum* δ_H (*J* in Hz) (600 MHz, $CDCl_3$)	from *S. trocheliophorum* [3] δ_H m (*J* in Hz) (300 MHz, $CDCl_3$)	from *S. stellatum* δ_C (150 MHz, $CDCl_3$)	from *S. trocheliophorum* [3] δ_C [a] (75.5 MHz, $CDCl_3$)
1			143.83	143.3
2	6.23 d (11.3)	6.23 d (11.4)	121.02	119.8
3	6.07 d (11.7)	6.07 d (11.4)	120.19	120.6
4			136.22	135.5
5a 5b	2.19–2.27 m	2.26 m 2.21 m	35.70	35.2
6a 6b	1.72–1.77 m	1.83 m 1.74 m	25.12	24.8
7	2.78 t (5.8)	2.79 t (5.9)	59.61	58.9
8			59.61	58.9
9a 9b	1.80–1.85 m	1.83 m	35.92	35.5
10a+10b	1.91 q (5.5)	1.90 m	21.44	21.0

11	5.18 t (6.4)	5.18 br t (6.0)	129.75	129.4
12			131.28	130.7
13a	2.43 dd (13.6, 3.8)	2.43 dd (12.0, 3.8)	43.63	43.2
13b	2.19–2.27 m	2.24 dd (12.0, 9.5)		
14	5.90 dd (9.4, 3.8)	5.91 dd (9.5, 3.8)	73.93	73.4
15	2.53 sp (6.8)	2.53 sp (6.9)	28.22	27.8
16	1.05 d (6.8)	1.05 d (6.8)	24.72	24.3
17	1.15 d (7.2)	1.16 d (7.0)	23.92	23.5
18	1.78 s	1.79 s	16.90	16.4
19	1.29 s	1.29 s	18.62	18.2
20	1.50 s	1.51 s	17.96	17.5
C=O			170.19	169.3
$COCH_3$	2.04 s	2.06 s	21.37	20.8
$[\alpha]_D$ (T [° C])	+171.8 (20, c 0.1, MeOH)	+136 (c 1.1, $CHCl_3$) +150 (c 1.02, $CHCl_3$)[4]		

Table S4. Comparison of ^{1}H and ^{13}C NMR data, melting points and optical rotation values of (–)-sarcophytoxide [(–)-**4**] from the soft corals *Sarcophyton stellatum*, *Sarcophyton birklandi*,[5] and *Sarcophyton ehrenbergi*[1]

Position	**^{1}H NMR**		**^{13}C NMR**		
	from *S. stellatum* δ_H (J in Hz) (600 MHz, $CDCl_3$)	from *S. birklandi* [5] δ_H m (J in Hz) (300 MHz, $CDCl_3$)	from *S. stellatum* δ_C (150 MHz, $CDCl_3$)	from *S. ehrenbergi* [1] δ_C (125 MHz, $CDCl_3$)	from *S. birklandi* [5] δ_C (75 MHz, $CDCl_3$)
1			133.19	133.1	133.5
2	5.50–5.56 m	5.53 m	83.63	83.6	83.8
3	5.22 d (9.8)	5.22 d (10.2)	126.27	126.3	126.4
4			139.30	139.3	139.2
5	2.30–2.38 m	2.3	37.65	37.7	37.6
6a 6b	1.86–1.94 m 1.60–1.65 m	1.9 1.3	25.28	25.2	25.4
7	2.71 t (4.1)	2.71 t (4.1)	61.90	61.8	61.9
8			59.84	59.8	59.8
9a 9b	2.10 ddd (13.1, 5.2, 2.8) 1.00 td (13.2, 3.0)	2.0 1.0 dt (13.0, 2.9)	39.83	39.8	39.7
10a 10b	2.25 dddd (14.3, 10.2, 4.9, 3.4) 1.86–1.94 m	2.2 1.9	23.52	23.5	23.5

11	5.09 dd (9.8, 5.3)	5.09 dd (10.8, 5.1)	123.59	123.6	123.7
12			136.84	136.8	136.7
13	1.86–1.94 m	1.9	36.66	36.7	36.7
14a	2.51–2.59 m	2.6	26.12	26.1	26.0
14b	1.60–1.65 m	1.6			
15			127.86	127.8	127.5
16	4.45–4.53 m	4.49 s	78.40	78.3	78.4
17	1.64 s	1.64 s	10.20	10.1	10.1
18	1.81 s	1.81 s	15.58	15.5	15.6
19	1.26 s	1.26 s	16.91	16.9	17.0
20	1.59 s	1.58 s	15.06	15.1	15.2
mp [°C]	60–61	79–81[6], 78–79[2]			
$[\alpha]_D$ (T [° C])	–129.4 (20, c 0.1, MeOH)	–191 (c 0.4, $CHCl_3$)[6] –183 (c 0.1)[2]		–128 (25, c 1.0, $CHCl_3$)	

Table S5. NMR data ($CDCl_3$) of ethyl 5-[(1*E*,5*Z*)-2,6-dimethylocta-1,5,7-trienyl]furan-3-carboxylate (**6**) recorded at 600 MHz (^1H) and 150 MHz (^{13}C)

Position	**δ_H (*J* in Hz)**	**δ_C** [a]	**HMBC** [b,c]	**NOESY** [b]
1	7.88 s	145.42, CH	3	5, 14, 16, 17
2		120.63, C	1, 3	
3	6.49 s	106.74, CH	1, 5	5, 14
4		154.61, C	1, 3, 14	
5	6.05 br s	113.65, CH	3, 7, 14	1, 3, 7, 14
6		140.50, C	5, 7, 8, 14	
7	2.22 t (7.9)	40.66, CH_2	5, 8, 9, 14	5, 8, 9, 14
8	2.36 q (7.5)	25.80, CH_2	7, 9	7, 9, 11, 14
9	5.37 t (7.3)	129.75, CH	7, 8, 11, 15	7, 8, 15
10		132.84, C	8, 11, 12a, 12b, 15	
11	6.76 ddd (17.3, 10.8, 0.9)	133.46, CH	9, 12a, 12b, 15	8, 12a, 12b
12a	5.29 br d (17.3)	113.77, CH_2		11, 12b, 15
12b	5.09 dt (10.5, 1.5)			11, 12a
13		163.38, C	3	
14	1.96 d (1.1)	18.71, CH_3	5, 7	1, 3, 5, 7, 8
15	1.80 q (1.1)	19.74, CH_3	9, 11	9, 12a
16	4.29 q (7.1)	60.38, CH_2	17	1
17	1.34 t (7.2)	14.11, CH_3	16	1

[a] Number of attached protons determined by the DEPT experiment. [b] Only selected signals are shown. [c] HMBC correlations are from carbon atoms (position) to protons.

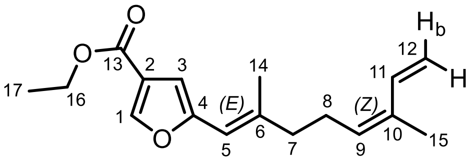


Table S6. NMR data ($CDCl_3$) of ethyl 5-[(1*E*,5*E*)-2,6-dimethylocta-1,5,7-trienyl]furan-3-carboxylate (**7**)

Position	***from Capnella fungiformis***				**Synthetic product**[7]	
	δ_H (*J* in Hz) 600 MHz	**δ_C**[a] 150 MHz	**HMBC**[b,c]	**NOESY**[b]	**δ_H** (*J* in Hz) 200 MHz	**δ_C**[d] 50 MHz
1′	7.88 s	145.42, CH	3′	5′, 14′, 16′, 17′	7.86 s	145.4
2′		120.63, C	1′, 3′			120.6
3′	6.49 s	106.74, CH	1′, 5′	5′, 14′	6.50 s	106.7
4′		154.61, C	1′, 3′, 14′			154.6
5′	6.05 br s	113.65, CH	3′, 7′, 14′	3′, 1′, 7′, 14′	6.05 br s	113.6
6′		140.50 C	5′, 7′, 8′, 14′			140.4
7′	2.24 t (7.9)	40.27, CH_2	5′, 8′, 9′, 14′	5′, 9′, 14′	2.26 m	40.2
8′	2.33 q (7.9)	26.70, CH_2	7′, 9′	9′, 14′, 15′		26.7
9′	5.47 br t (7.2)	131.75, CH	11′, 8′, 7′, 15′	7′, 8′, 11′	5.46 br t (7)	131.7
10′		132.84, C	11′, 12a′, 12b′, 8′, 15′			134.5
11′	6.35 dd (17.1, 10.7)	141.34, CH	9′, 12a′, 15′	9′, 12a′, 12b′	6.35 dd (17, 11)	141.3
12a′	5.09 d (17.3)	110.84, CH_2		11′, 12b′, 15′	5.08 br d (17)	110.8
12b′	4.93 d (10.9)			11′, 12a′	4.93 br s (11)	
13′		163.38, C	3′, 16′			163.3
14′	1.97 d (1.1)	18.71, CH_3	5′, 7′	1′, 3′, 5′, 7′, 8′	2.00 br s	18.6
15′	1.74 s	11.69, CH_3	11′, 9′	8′, 12a′	1.74 br s	11.6
16′	4.29 q (7.1)	60.38, CH_2	17′	1′	4.29 q (7)	60.3
17′	1.34 t (7.2)	14.32, CH_3	16′	1′	1.34 t (7)	14.3

[a] Number of attached protons determined by the DEPT experiment. [b] Only selected signals are shown. [c] HMBC correlations are from carbon atoms (position) to protons. [d] Assignment according to the compound from *Capnella fungiformis*

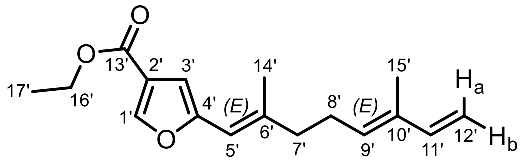


Table S7. Dihedral angles (°) determined from the minimum energy conformations of the eight possible diastereoisomers **9a**–**9h** by quantum chemical calculation

	9a	**9b**	**9c**	**9d**	**9e**	**9f**	**9g**	**9h**
H2α–C2–C3–H3α	-34.3	-34.1	35.9	36.6	-33.8	-33.5	35.4	35.2
H2α–C2–C3–H3β	85.8	85.4	155.9	156.6	86.0	86.0	155.3	155.2
H2β–C2–C3–H3α	-155.2	-155.2	-85.5	-84.5	-154.7	-154.5	-85.8	-85.6
H2β–C2–C3–H3β	-35.1	-35.7	34.4	35.5	-34.9	-34.9	34.1	34.4
H3α–C3–C4–H4	32.5	27.9	-39.1	-39.4	157.4	157.4	89.5	87.7
H3β–C3–C4–H4	-87.3	-91.5	-158.0	-158.3	38.7	38.6	-30.1	-32.0

🟧 Dihedral angles ~ 90 °

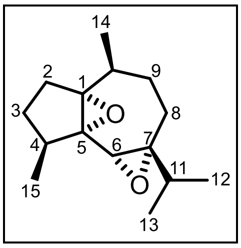
Table S8. Comparison of estimated and experimental $^3J_{HH}$ coupling constants (Hz) in the five-membered ring of the eight possible diastereoisomers **9a**–**9h**

	9a	**9b**	**9c**	**9d**	**9e**	**9f**	**9g**	**9h**	**Exp**
^3J(H-2α,H-3*cis*) [a]	2.0	2.0	11.2	11.3	8.1	8.1	7.8	7.9	< 1 Hz
^3J(H-2α,H-3*trans*) [a]	8.0	8.0	7.8	7.6	2.0	2.0	11.2	11.1	8.4 Hz
^3J(H-2β,H-3*cis*) [a]	7.9	7.8	8.0	7.8	11.1	11.0	2.0	2.0	8.3 Hz
^3J(H-2β,H-3*trans*) [a]	11.1	11.1	2.0	2.0	7.9	7.9	8.0	8.0	10.4 Hz
^3J(H-3*cis*,H-4) [a]	2.0	2.0	11.5	11.6	11.4	11.4	2.0	2.0	< 1 Hz
^3J(H-3*trans*,H-4) [a]	8.3	8.9	7.2	7.2	7.3	7.3	8.6	8.3	7.6 Hz

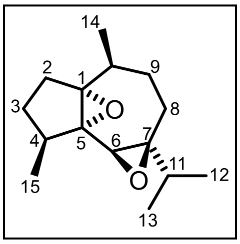
[a] *cis* and *trans* with respect to the 4-Me group

🟧 $J \leq 2$ Hz

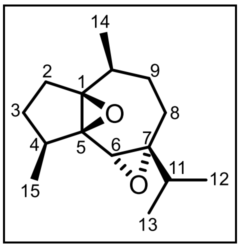
Estimated from the torsion angles by the Bothner-By equation ($^3J_{HH} = 7 - \cos\theta + 5\cos2\theta$)[1, 2]



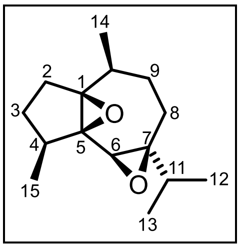
9a



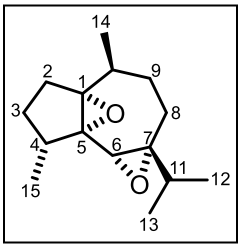
9b



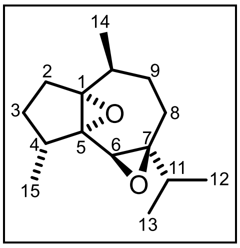
9c



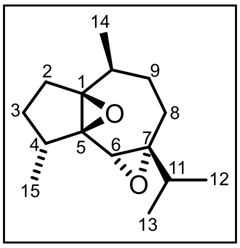
9d



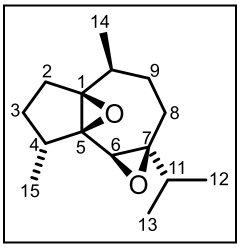
9e



9f



9g



9h

Table S9. Comparison of calculated (GIAO) and experimental ^{1}H NMR shifts (δ, $CDCl_3$) for the structures **9a** and **9b**

		9a		**9b**	
	δ_H exp. (ppm)	δ_H calcd. (ppm)	Δ	δ_H calcd. (ppm)	Δ
2α	1.693	1.353	0.340	1.541	0.152
2β	1.794	1.971	-0.177	1.652	0.142
3α	1.642	1.552	0.090	1.618	0.024
3β	1.122	0.974	0.148	0.984	0.138
4	2.373	2.160	0.213	2.512	-0.139
6	3.052	2.923	0.129	3.135	-0.083
8α	1.870	1.663	0.207	2.260	-0.390
8β	1.910	1.991	-0.081	1.510	0.400
9α	1.774	2.052	-0.278	1.423	0.351
9β	1.229	0.861	0.368	1.371	-0.142
10	2.357	2.061	0.296	1.894	0.463
14	1.019	1.106	-0.087	0.975	0.044
15	0.974	1.007	-0.033	1.130	-0.156

average absolute deviation	0.188		0.202
maximum absolute deviation	0.368		0.463

Table S10. Comparison of selected calculated (GIAO) and experimental ^{13}C NMR shifts (δ, $CDCl_3$) for the structures **9a** and **9b**

		9a		**9b**	
	δ_C exp. (ppm)	δ_C calcd. (ppm)	Δ	δ_C calcd. (ppm)	Δ
1	73.690	73.630	0.060	71.689	2.001
4	37.600	39.811	-2.211	38.052	-0.452
5	69.340	69.934	-0.594	65.016	4.324
6	58.080	61.403	-3.323	60.845	-2.765
7	68.590	68.312	0.278	61.045	7.545
10	31.450	33.692	-2.242	34.547	-3.097
11	36.500	39.177	-2.677	36.829	-0.329
14	17.300	13.647	3.653	17.593	-0.293
15	16.010	15.034	0.976	17.742	-1.732

average absolute deviation	1.779		2.504
maximum absolute deviation	3.653		7.545

Table S11. Comparison of ^{1}H NMR data of 24-methylenecholesterol (**10**), (24*S*)-24-methylcholesterol (**11**), gorgosterol (**12**) and aplysterol (**13**)

Position	**10**		**11**		**12**		**13**	
	from *Capnella fungiformis* δ_H (*J* in Hz) (600 MHz, $CDCl_3$)	synthetic sample [8] δ_H (*J* in Hz) (500 MHz, $CDCl_3$)	from *Capnella fungiformis* δ_H (*J* in Hz) (600 MHz, $CDCl_3$)	from *Posidonia oceanica* and *Cymodocea nodosa* [9] δ_H (*J* in Hz) (500 MHz, $CDCl_3$)	from *Capnella fungiformis* δ_H (*J* in Hz) (600 MHz, $CDCl_3$)	from *Peridinium foliaceum* [10] δ_H (*J* in Hz) (360 MHz, C_6D_6)	from *Pseudoceratina arabica* δ_H (*J* in Hz) (600 MHz, $CDCl_3$)	from *Aplysina fistularis* [11] δ_H (*J* in Hz) (360 MHz, $CDCl_3$)
1a	1.80–1.90 m						1.80–1.86 m	
1b	1.03–1.10 m						1.03–1.11 m	
2a	1.80–1.90 m						1.80–1.86 m	
2b	1.47–1.60 m						1.47–1.57 m	
3	3.52 tt (11.2, 4.6)	3.54 tt (11.5, 5.0)			3.52 tt (11.2, 4.6)		3.51 tt (11.2, 4.7)	
4a	2.29 ddd (12.8, 4.9, 1.9)						2.29 ddd (13.2, 5.3, 2.3)	
4b	2.19–2.26 m						2.19–2.26 m	
6	5.34 dt (5.0, 2.0)	5.35 dt (3.0, 2.5)	5.36 dt (5.3, 2.0)		5.35 dt (5.0, 2.2)		5.34 dt (5.0, 2.6)	
7a	1.94–2.03 m						1.96 dtd (17.3, 4.9, 2.6)	
7b	1.47–1.60 m						1.47–1.57 m	
8	1.40–1.47 m						1.40–1.47 m	
9	0.90–0.94 m						0.90–0.92 m	
11a	1.47–1.60 m						1.47–1.57 m	
11b	1.40–1.47 m						1.47–1.57 m	
12a	1.94–2.03 m						2.00 dt (12.4, 3.4)	
12b	1.10–1.19 m						1.15 td (12.8, 4.9)	
14	0.99–1.01 m				1.00 br s		0.96–0.99 m	
15a	1.47–1.60 m						1.55–1.60 m	
15b	1.03–1.10 m						1.03–1.11 m	
16a	1.80–1.90 m						1.80–1.86 m	

Position	**10**		**11**		**12**		**13**	
	from *Capnella fungiformis* δ_H (*J* in Hz) (600 MHz, CDCl$_3$)	synthetic sample [8] δ_H (*J* in Hz) (500 MHz, CDCl$_3$)	from *Capnella fungiformis* δ_H (*J* in Hz) (600 MHz, CDCl$_3$)	from *Posidonia oceanica* and *Cymodocea nodosa* [9] δ_H (*J* in Hz) (500 MHz, CDCl$_3$)	from *Capnella fungiformis* δ_H (*J* in Hz) (600 MHz, CDCl$_3$)	from *Peridinium foliaceum* [10] δ_H (*J* in Hz) (360 MHz, C$_6$D$_6$)	from *Pseudoceratina arabica* δ_H (*J* in Hz) (600 MHz, CDCl$_3$)	from *Aplysina fistularis* [11] δ_H (*J* in Hz) (360 MHz, CDCl$_3$)
16b	1.25–1.30 m						1.23–1.27 m	
17	1.10–1.19 m						1.03–1.11 m	
18	0.67 s	0.68 s	0.66 s	0.678 s	0.65 s	0.685 s	0.67 s	0.681 s
19	1.00 s	1.01 s	1.00 s	1.008 s	1.00 s	0.953 s	1.00 s	1.008 s
20	1.40–1.47 m				0.98–1.01 m		1.32–1.37 m	
21	0.94 d (6.4)	0.95 d (6.5)	0.91 d (6.8)	0.919 d (6.7)	0.98–1.01 m	1.138 d (6.4) [c]	0.90 d (6.8)	0.909 d (6.5)
22a	1.47–1.60 m				0.13–0.19 m	0.20 m [c]	1.27–1.32 m	
22b	1.10–1.19 m						1.03–1.11 m	
23a	2.05–2.11 m						1.17–1.20[a] m	
23b	1.80–1.90 m						1.03–1.11 m	
24					0.24 dqd (8.8, 7.0, 1.8)	0.20 m [c]	1.27–1.32 m	
25	2.19–2.26 m						1.23–1.27 m	
26	1.011 d (6.8)	1.02 d (7.0) 1.03 d (7.0)	0.77 d (6.8)	0.858 d (7.0)	0.85 d (6.4)	0.902 d (8.1) [c]	0.80 d (7.2)	0.798 d (6.6) [b]
27a	1.014 d (6.8)		0.84 d (6.8)	0.782 d (6.9)	0.93 d (7.5)	1.018 d (6.7) [c]	1.32–1.37 m	
27b							1.03–1.11 m	
27-Me							0.85 t (7.3)	0.861 t (7.3)
28a	4.64 br d (1.5)	4.66 d (1.5)	0.76 d (6.8)	0.775 d (6.8)	0.94 d (6.9)	1.026 d (6.9) [c]	0.79 d (6.8)	0.812 d (6.7) [b]
28b	4.70 br s	4.71 d (1.5						
29					0.89 s	0.923 s		
30a					0.45 ddd (9.1, 4.3, 2.6)	0.50 dd (9.2, 4.5) [c]		
30b					–0.14 ddd (5.8, 4.4, 1.3)	–0.11 dd (6.0, 4.5) [c]		

[a] Chemical shift derived from the HSQC spectrum. [b] Assignments may be interchanged. [c] Signals not assigned to specific protons.

Table S12. Comparison of ^{13}C NMR data of 24-methylenecholesterol (**10**), (24*S*)-24-methylcholesterol (**11**), gorgosterol (**12**) and aplysterol (**13**)

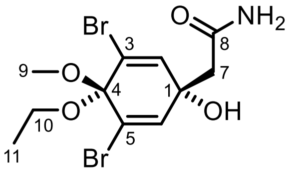
Position	**10**		**11**		**12**		**13**	
	from *Capnella fungiformis* δ_C (150 MHz, $CDCl_3$)	from *Litophyton viridis*[12] δ_C (68 MHz, $CDCl_3$)	from *Lobophytum crassum* δ_C (150 MHz, $CDCl_3$)	unspecified origin [13] δ_C (90 MHz, $CDCl_3$)	from *Capnella fungiformis* δ_C (150 MHz, $CDCl_3$)	from *Alcyonium molle*[14] δ_C (75 MHz, $CDCl_3$)	from *Capnella fungiformis* δ_C (150 MHz, $CDCl_3$)	from *Verongia* sp.[14] δ_C (25 MHz, $CDCl_3$)
1	37.24	37.2	37.25	37.3	37.23	37.3	37.27	37.3
2	31.65	31.6	31.67	31.7	31.61	31.7	31.70	31.7
3	71.82	71.7	71.81	71.8	71.92	71.8	71.83	71.7
4	42.28	42.3	42.31	42.4	42.23	42.3	42.34	42.3
5	140.73	140.7	140.76	140.7	140.68	140.8	140.72	140.6
6	121.71	121.5	121.73	121.7	121.80	121.7	121.72	121.6
7	31.89	31.9	31.90	31.9	31.86	31.9	31.93	31.9
8	31.89	31.9	31.90	31.9	31.96 or 32.03 or 32.14	32.0 (2 C), 32.1	31.93	31.9
9	50.11	50.1	50.12	50.1	50.14	50.2	50.17	50.1
10	36.49	36.5	36.50	36.5	36.51	36.5	36.52	36.5
11	21.07	21.1	21.08	21.1	21.07	21.2	21.10	21.1
12	39.76	39.8	39.76	39.8	39.85	39.9	39.80	39.8
13	42.35	42.3	42.31	42.4	42.76	42.8	42.34	42.3
14	56.75	56.7	56.75	56.8	56.49	56.6	56.79	56.8
15	24.28	24.3	24.29	24.3	24.51	24.5	24.30	24.3
16	28.21	28.2	28.19	28.2	28.21	28.2	28.23	28.3
17	55.97	56.0	55.98	56.0	57.90	57.9	56.17	56.1
18	11.85	11.0	11.85	11.9	11.90	11.9	11.87	11.9
19	19.39	19.4	19.40	19.4	19.40	19.4	19.40	19.4
20	35.74	35.7	36.18	36.1	35.28	35.3	35.88	35.9
21	18.70	18.7	18.88	18.9	21.17	21.1	18.71	18.7
22	34.67	34.7	33.71	33.8	31.96 or 32.03 or 32.14	32.0 (2 C), 32.1	33.90	33.9

Position	**10**		**11**		**12**		**13**	
	from *Capnella fungiformis* δ_C (150 MHz, $CDCl_3$)	from *Litophyton viridis*[12] δ_C (68 MHz, $CDCl_3$)	from *Lobophytum crassum* δ_C (150 MHz, $CDCl_3$)	unspecified origin [13] δ_C (90 MHz, $CDCl_3$)	from *Capnella fungiformis* δ_C (150 MHz, $CDCl_3$)	from *Alcyonium molle*[14] δ_C (75 MHz, $CDCl_3$)	from *Capnella fungiformis* δ_C (150 MHz, $CDCl_3$)	from *Verongia* sp.[14] δ_C (25 MHz, $CDCl_3$)
23	30.96	31.0	30.56	30.6	25.80	25.8	29.04	29.0
24	156.89	156.7	39.06	39.1	50.80	50.8	37.53	36.2
25	33.79	33.8	31.45	31.5	31.96 or 32.03 or 32.14	32.0 (2 C), 32.1	39.86	37.5
26	21.86	21.9	17.58	17.6	21.53	21.5	15.90	15.9
27	21.99	22.0	20.52	20.5	22.18	22.2	25.78	25.8
27-Me							12.22	12.3
28	105.91	105.9	15.44	15.5	15.45	15.4	16.54	16.6
29					14.27	14.3		
30					21.29	21.3		

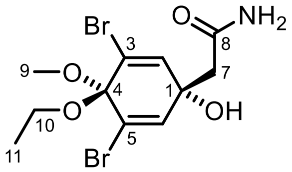
Table S13. Comparison of the ^{13}C NMR data of the secondary metabolites **14a** and **14b** isolated from the sponges *Pseudoceratina arabica* and *Aplysina* sp.[15]

Position	^{1}H NMR				^{13}C NMR		
	14a	**14a**	**14b**	**14b**	**14a**	**14b**	**14b**
	from *P. arabica* δ_H (*J* in Hz) (600 MHz, $CDCl_3$)	from *Aplysina* sp.[15] δ_H (*J* in Hz) (700 MHz, $CDCl_3$)	from *P. arabica* δ_H (*J* in Hz) (600 MHz, $CDCl_3$)	from *Aplysina* sp.[15] δ_H (*J* in Hz) (700 MHz, $CDCl_3$)	from *P. arabica* δ_C (150 MHz, $CDCl_3$)	from *P. arabica* δ_C (150 MHz, $CDCl_3$)	from *Aplysina* sp.[15] δ_C (75 MHz, $CDCl_3$)
1					71.92	70.90	70.9
2	6.72 s	6.73 s	6.72 s	6.73 s	139.44	139.42	139.5
3					124.41	124.42	124.4
4					96.12	96.13	96.2
5					124.41	124.42	124.4
6	6.72 s	6.73 s	6.72 s	6.73 s	139.44	139.42	139.5
7	2.50 s	2.51 s	2.50 s	2.51 s	43.67	43.71	43.8
8					172.31	172.31	172.4
9	3.14 s	3.16 s	3.20 s	3.21 s	50.88	51.16	51.2
10	3.34 q (7.2)	3.36 q (6.8)	3.24 q (7.2)	3.27 q (6.7)	59.74	59.34	
11	1.26 t (7.0)	1.275 t (6.8)	1.27 t (6.8)	1.28 t (6.7)	14.94	15.07	15.1

OH	5.20 s	5.17 br s	5.16 s	5.10 br s			
N-Ha	5.73 br s	5.825 br s	5.73 br s	5.76 br s			
N-Hb	5.59 br s	5.67 br s	5.59 br s	5.59 br s			



14a



14b

Fig. S1. ^{1}H NMR (600 MHz, $CDCl_3$) spectrum of **6** and **7**.

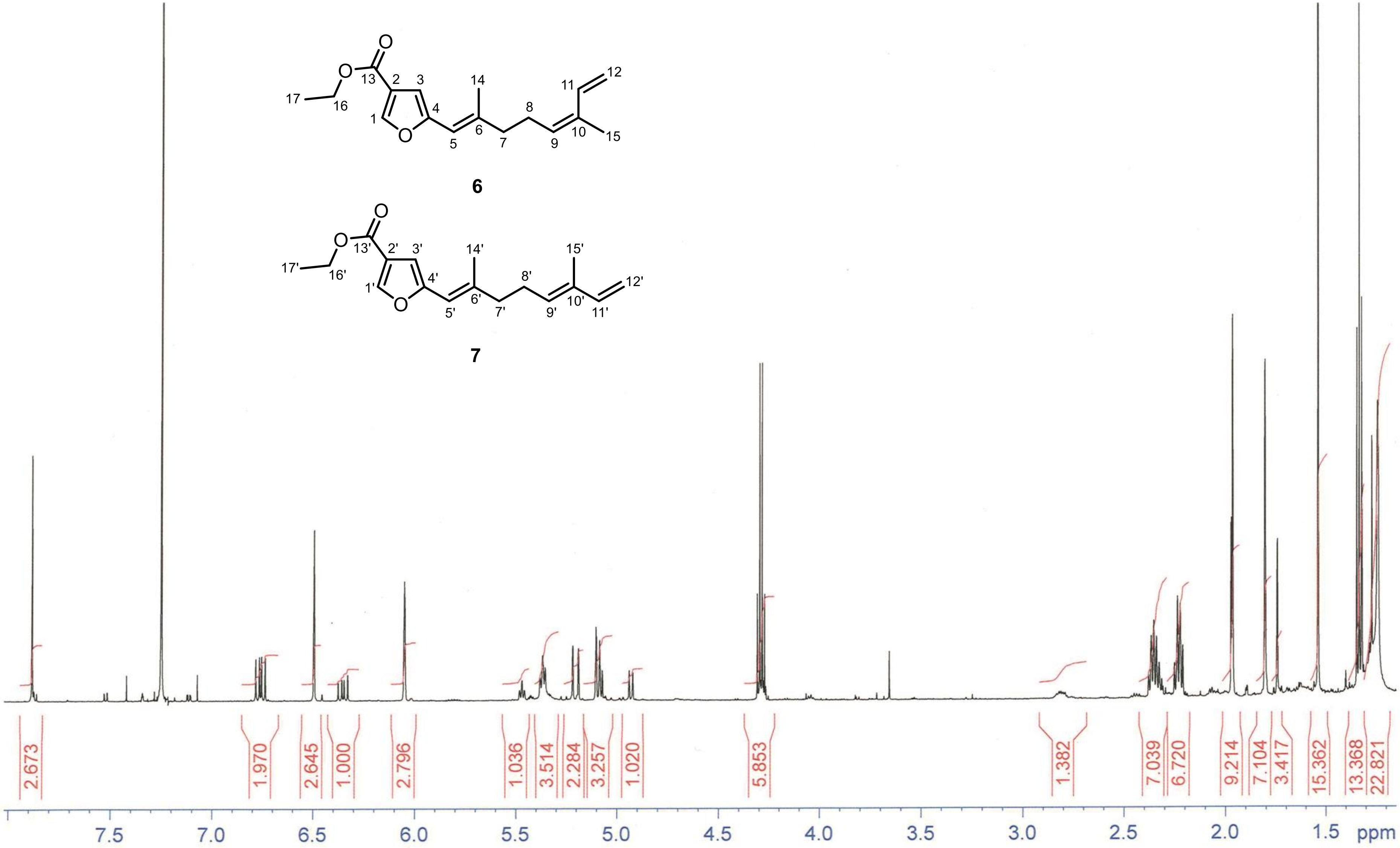


Fig. S2. ^{13}C NMR (150 MHz, $CDCl_3$) spectrum of of **6** and **7**.

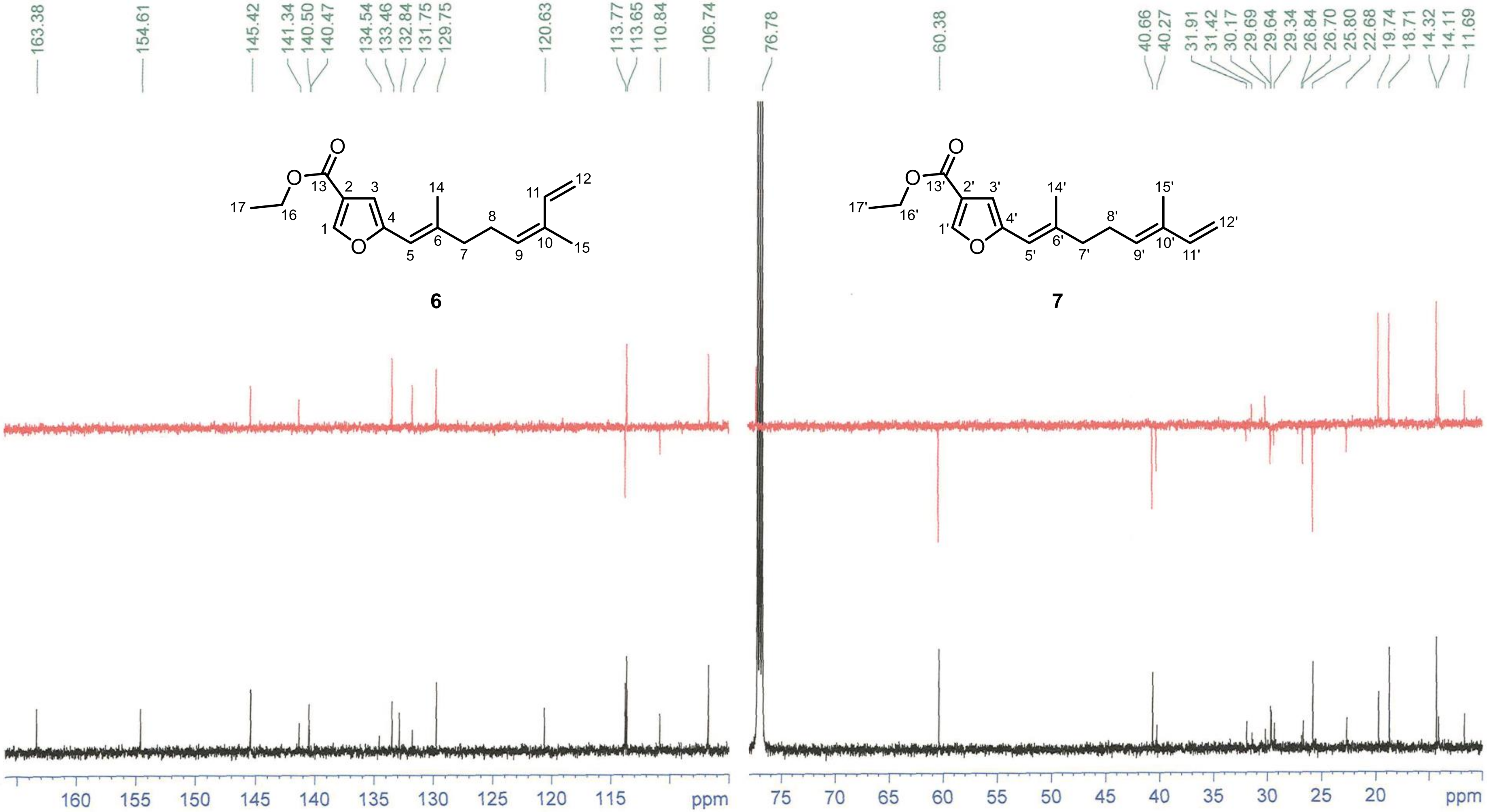
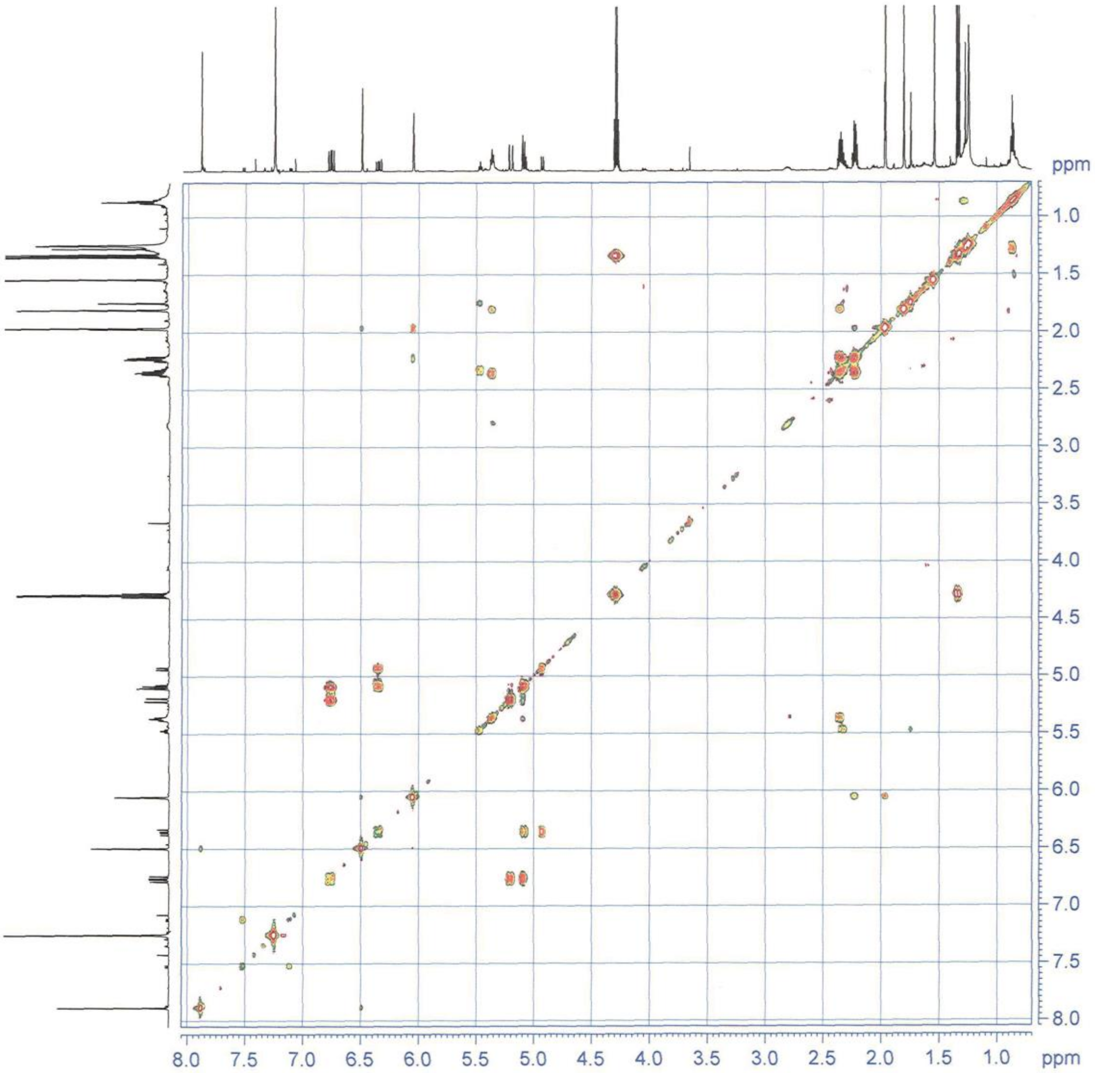
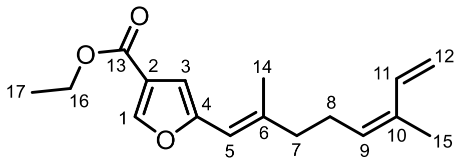


Fig. S3. COSY spectrum of **6** and **7**.





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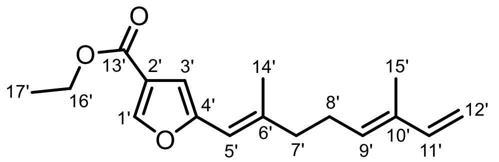

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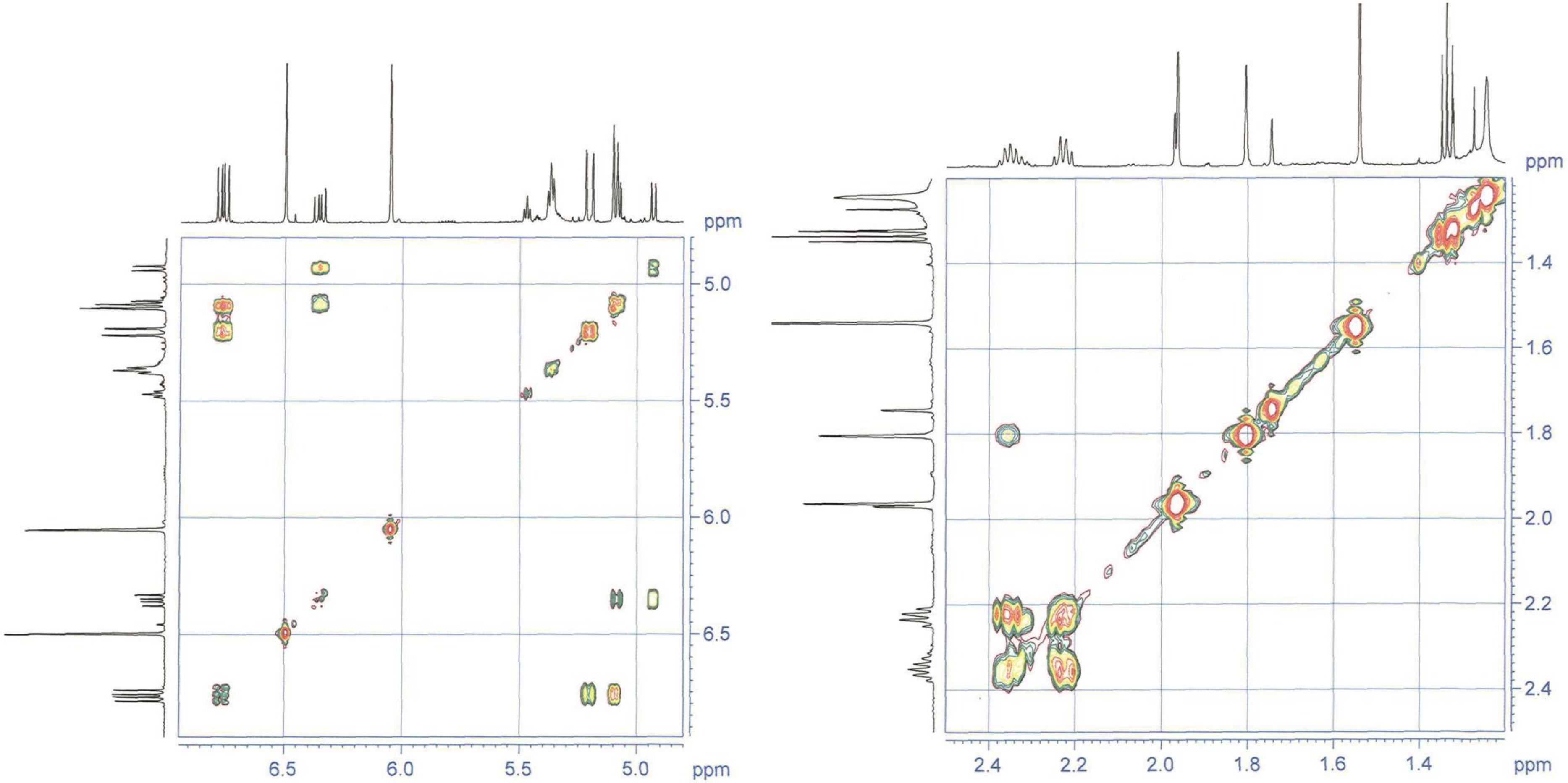
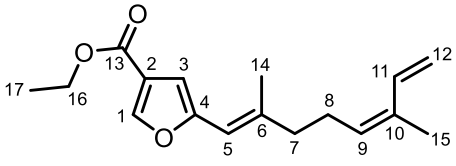
Fig. S4. Details of the COSY spectrum of **6** and **7**.





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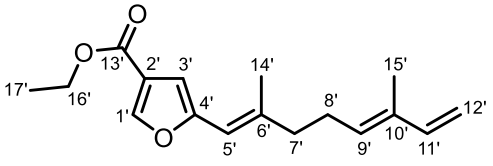

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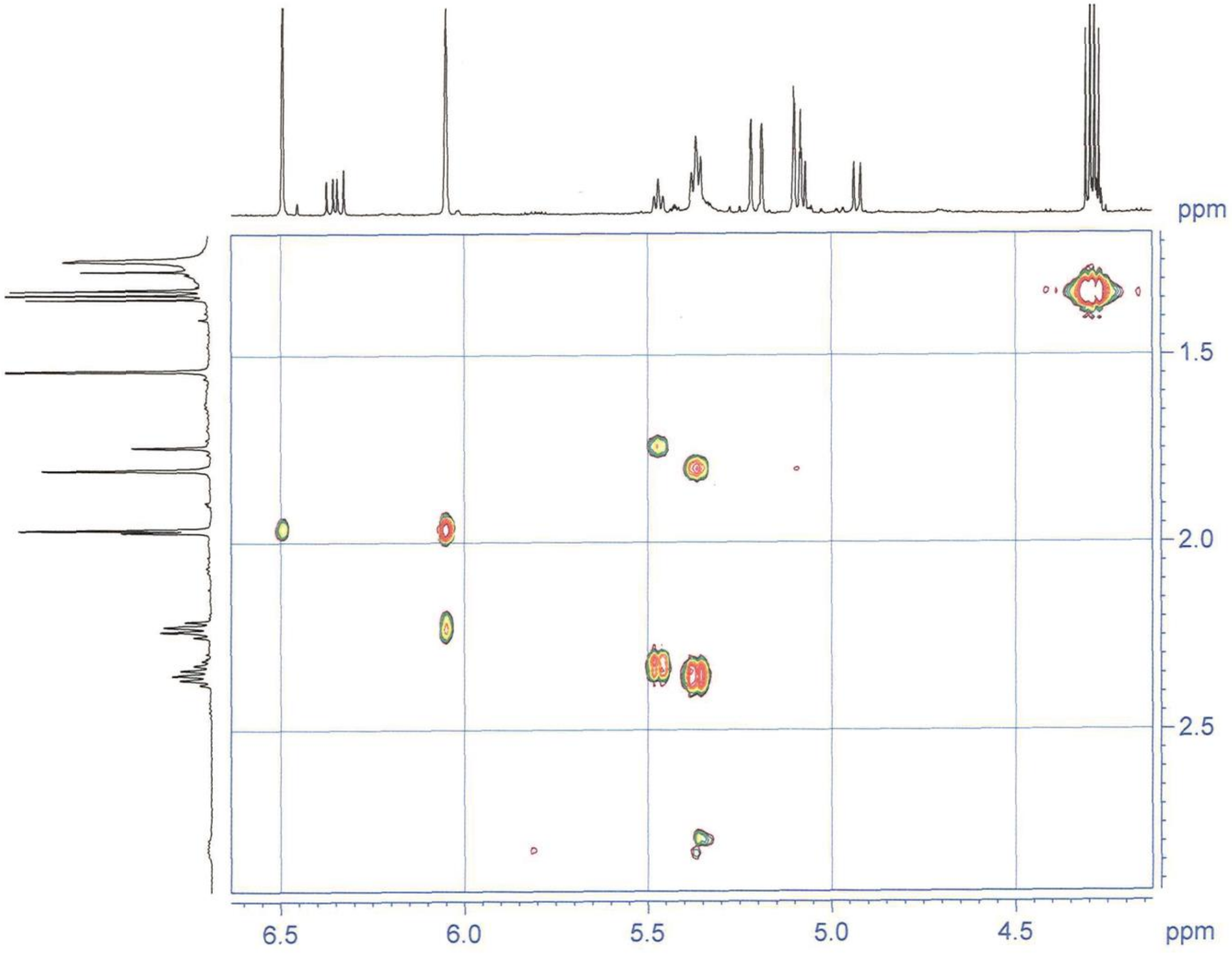
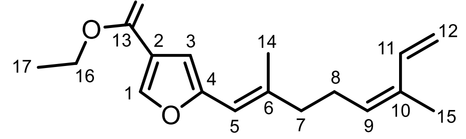
Fig. S5. Details of the COSY spectrum of **6** and **7**.





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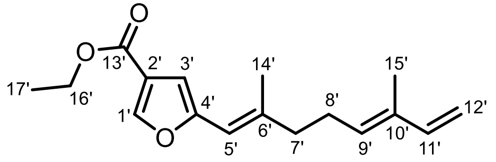

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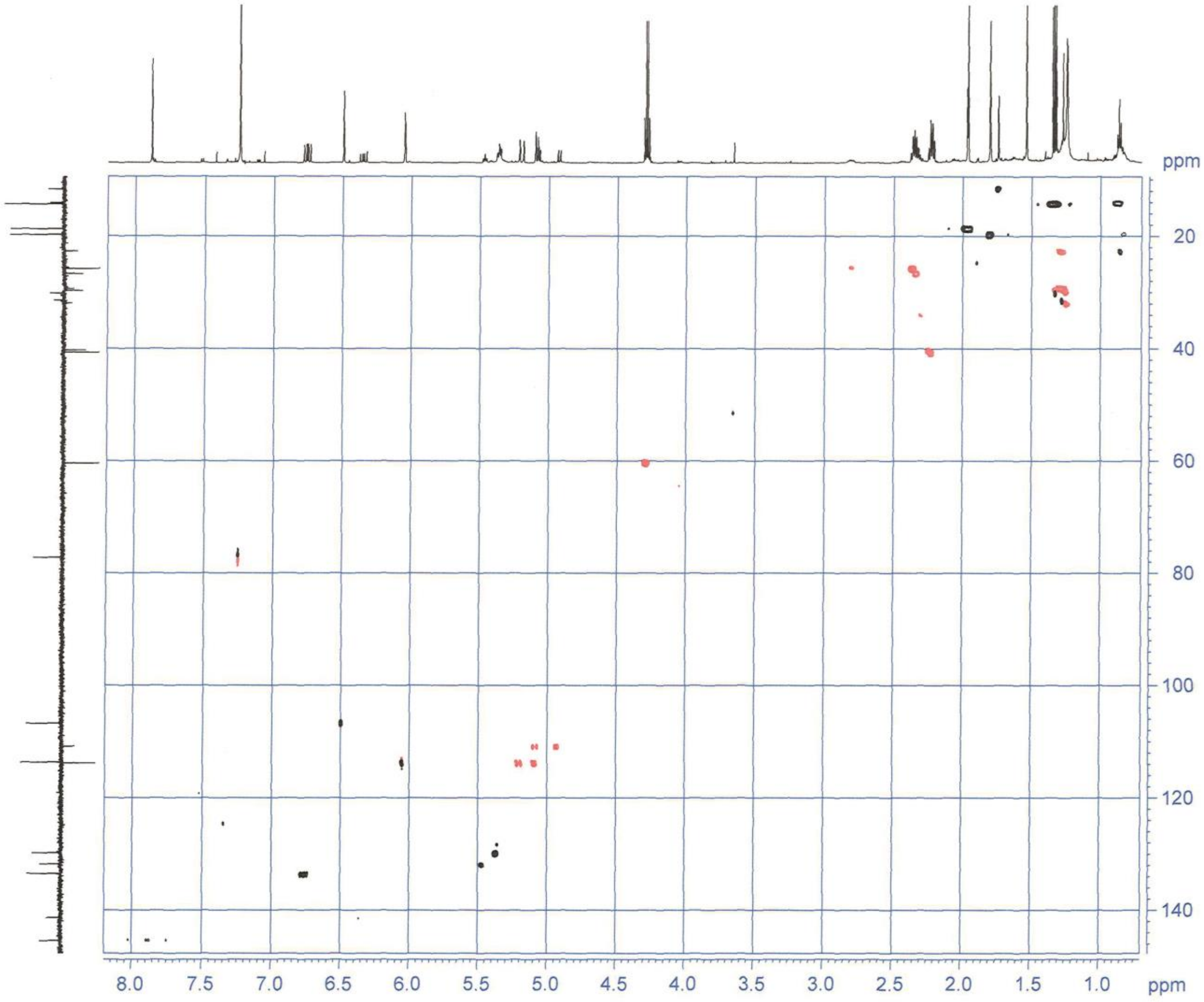
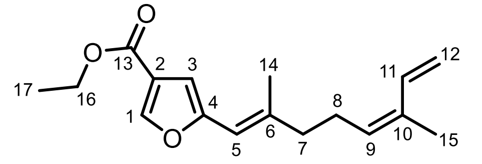
Fig. S6. HSQC spectrum of **6** and **7**.





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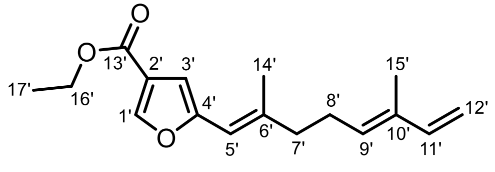

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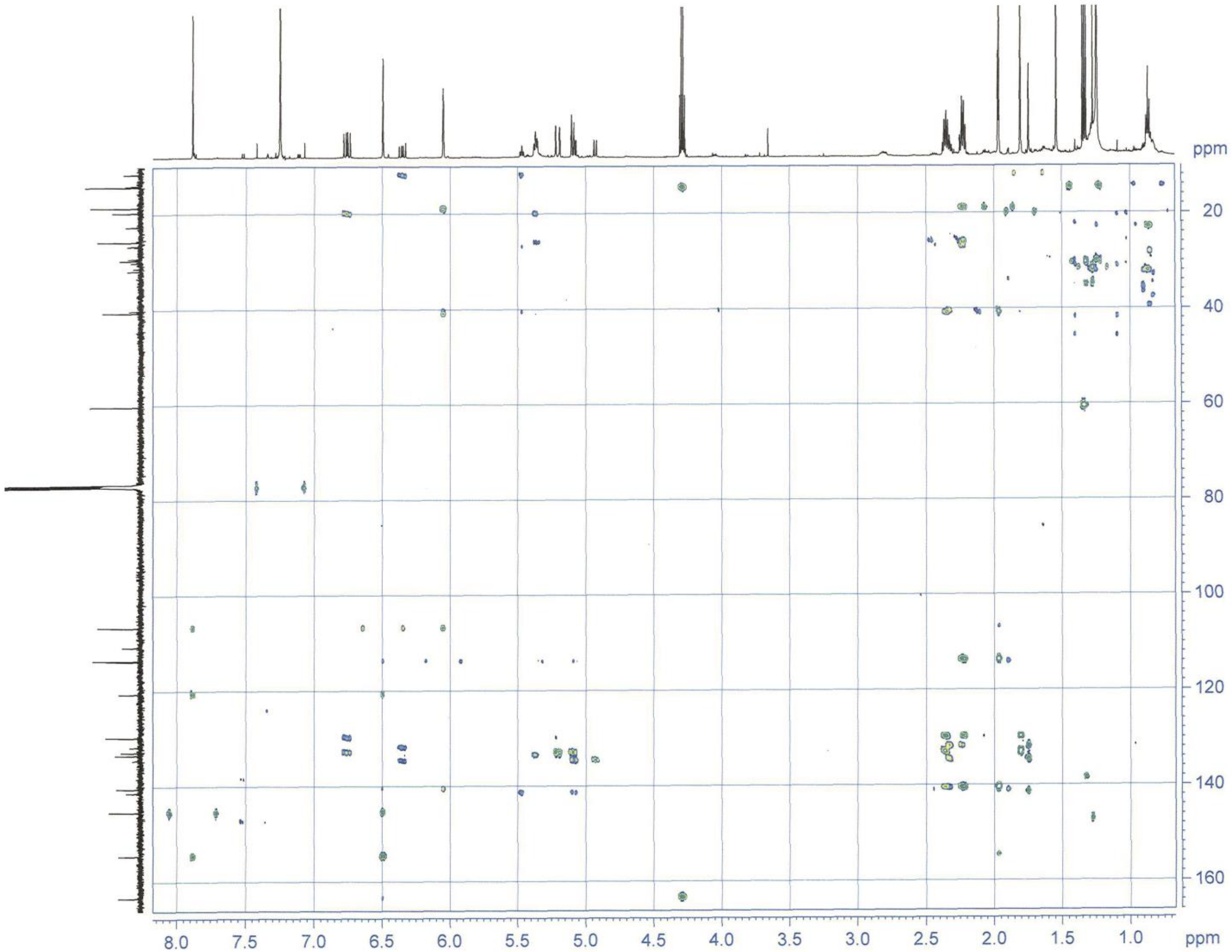
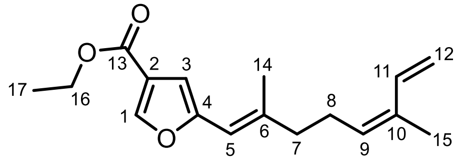
Fig. S7. HMBC spectrum of **6** and **7**.





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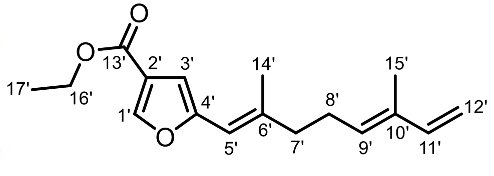
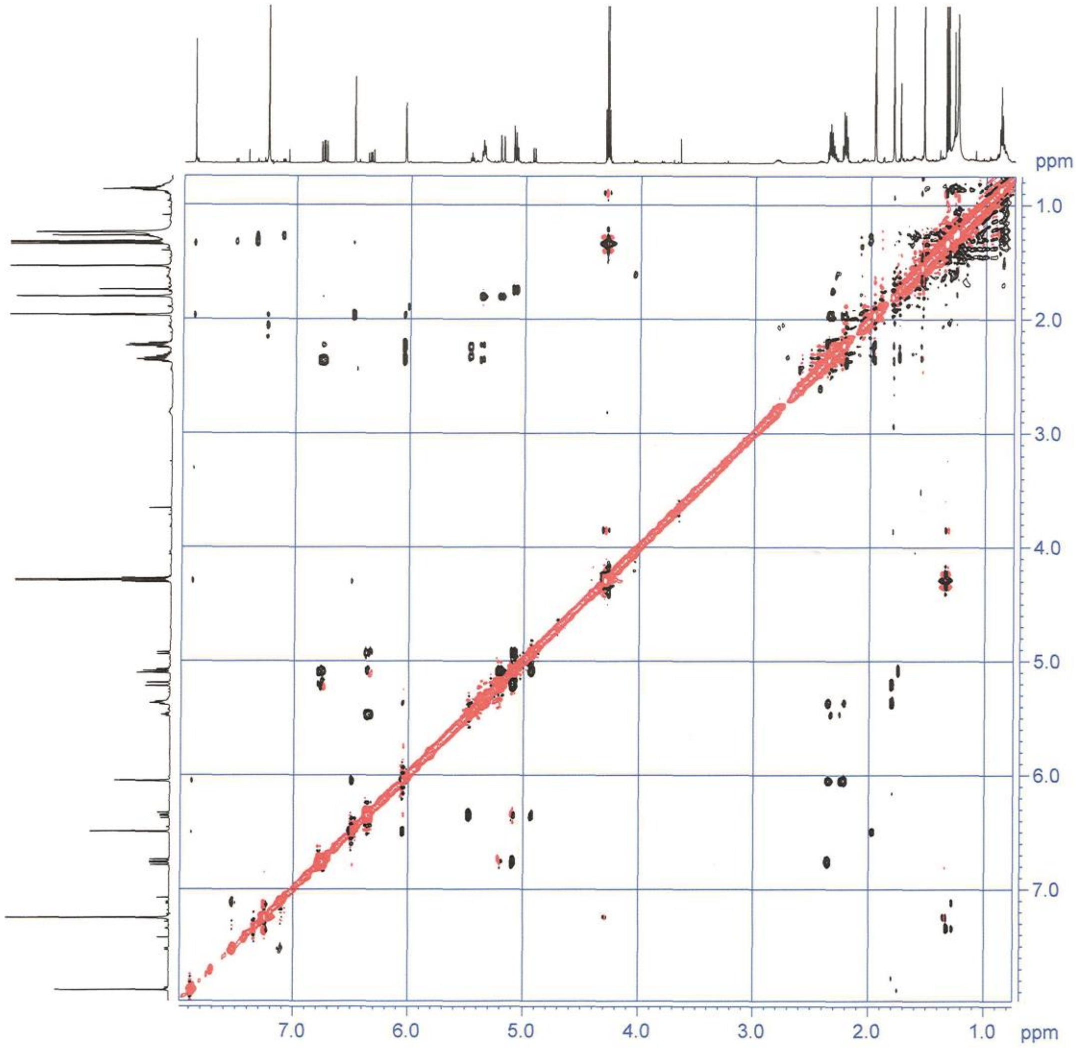
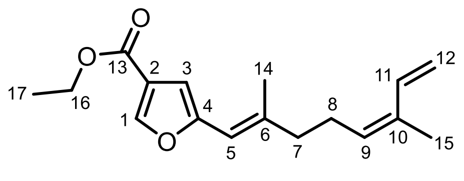


7

Fig. S8. NOESY spectrum of **6** and **7**.





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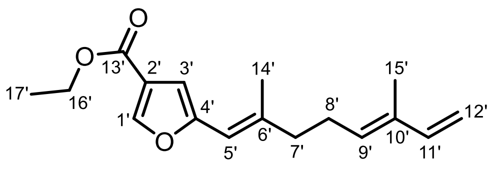

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Fig. S9. ^{1}H NMR (600 MHz, CDCl$_3$) spectrum of **9a**.

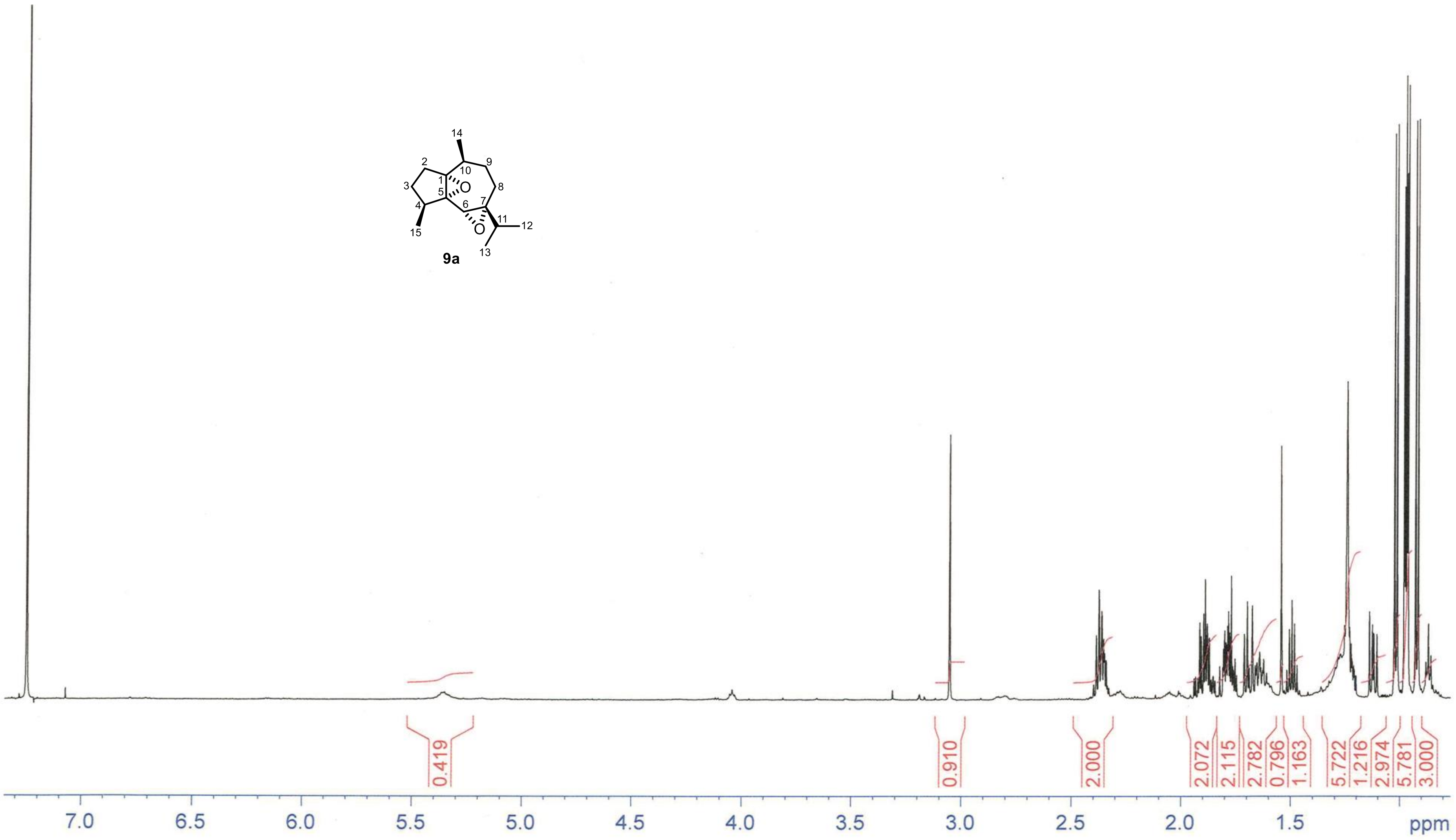


Fig. S10. Details of the ^{1}H NMR (600 MHz, CDCl$_3$) spectrum of **9a**.

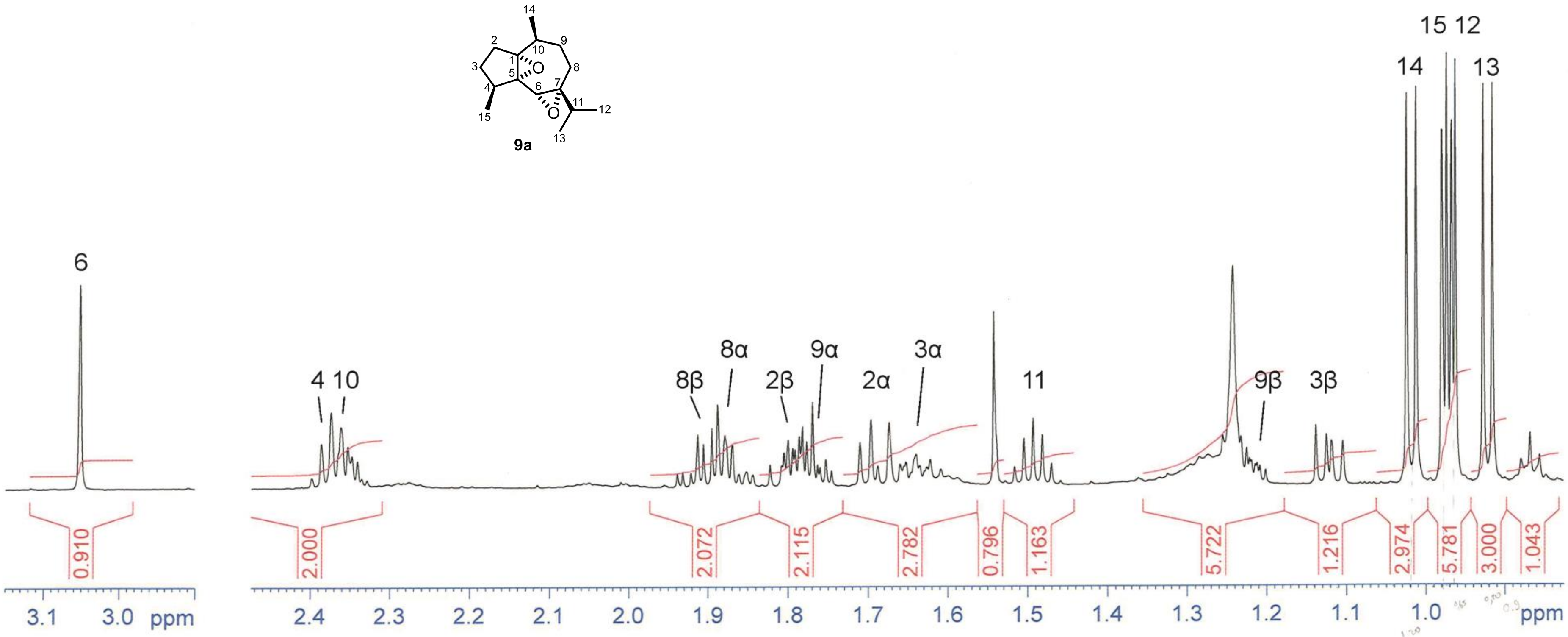


Fig. S11. Expansion of the measured (top) (600 MHz, $CDCl_3$) and simulated (bottom) ^1H NMR spectrum of **9a**.

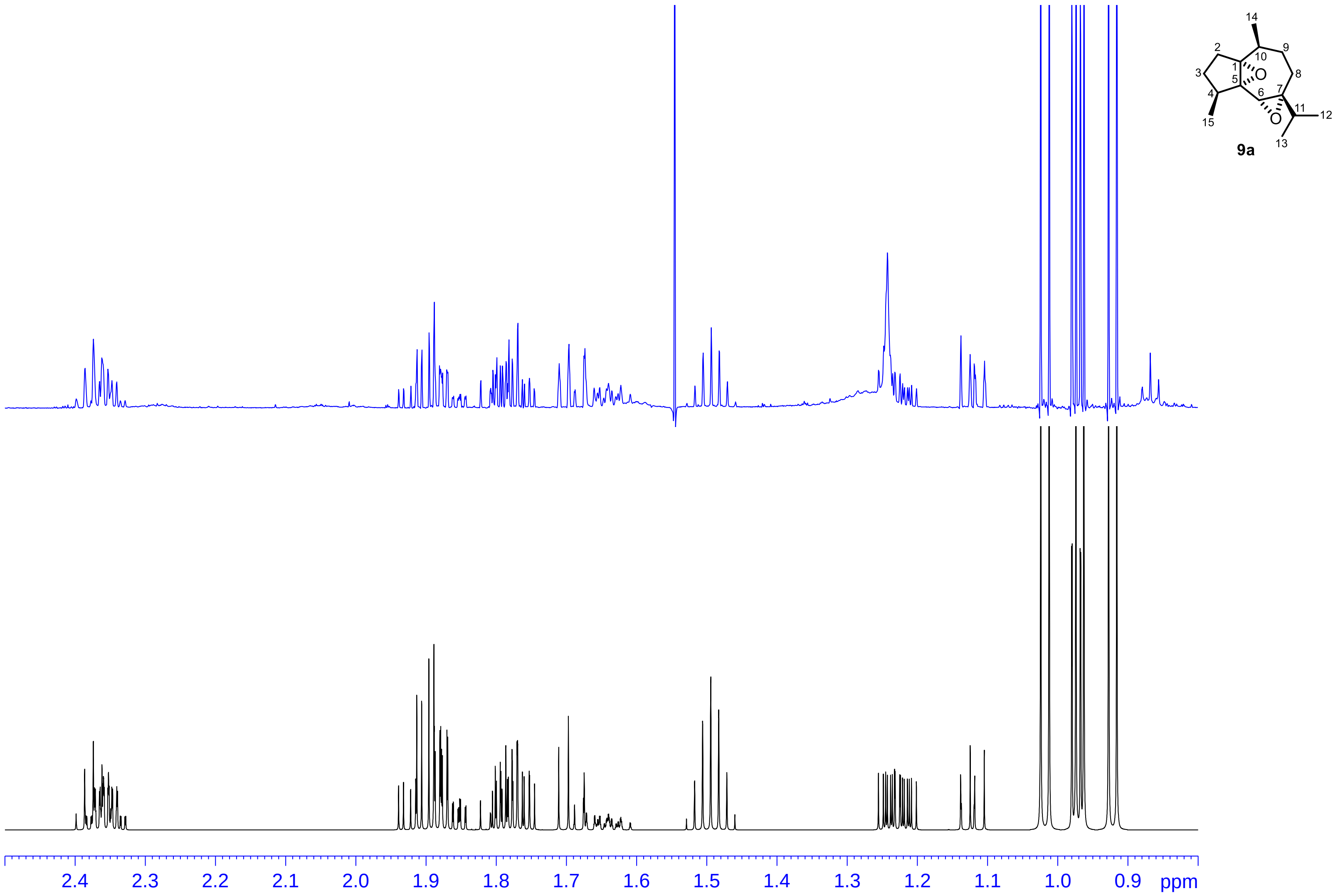


Fig. S12. Signals for H-4 and H-10 in the measured (top) (600 MHz, $CDCl_3$) and simulated (bottom) ^{1}H NMR spectrum of **9a**.

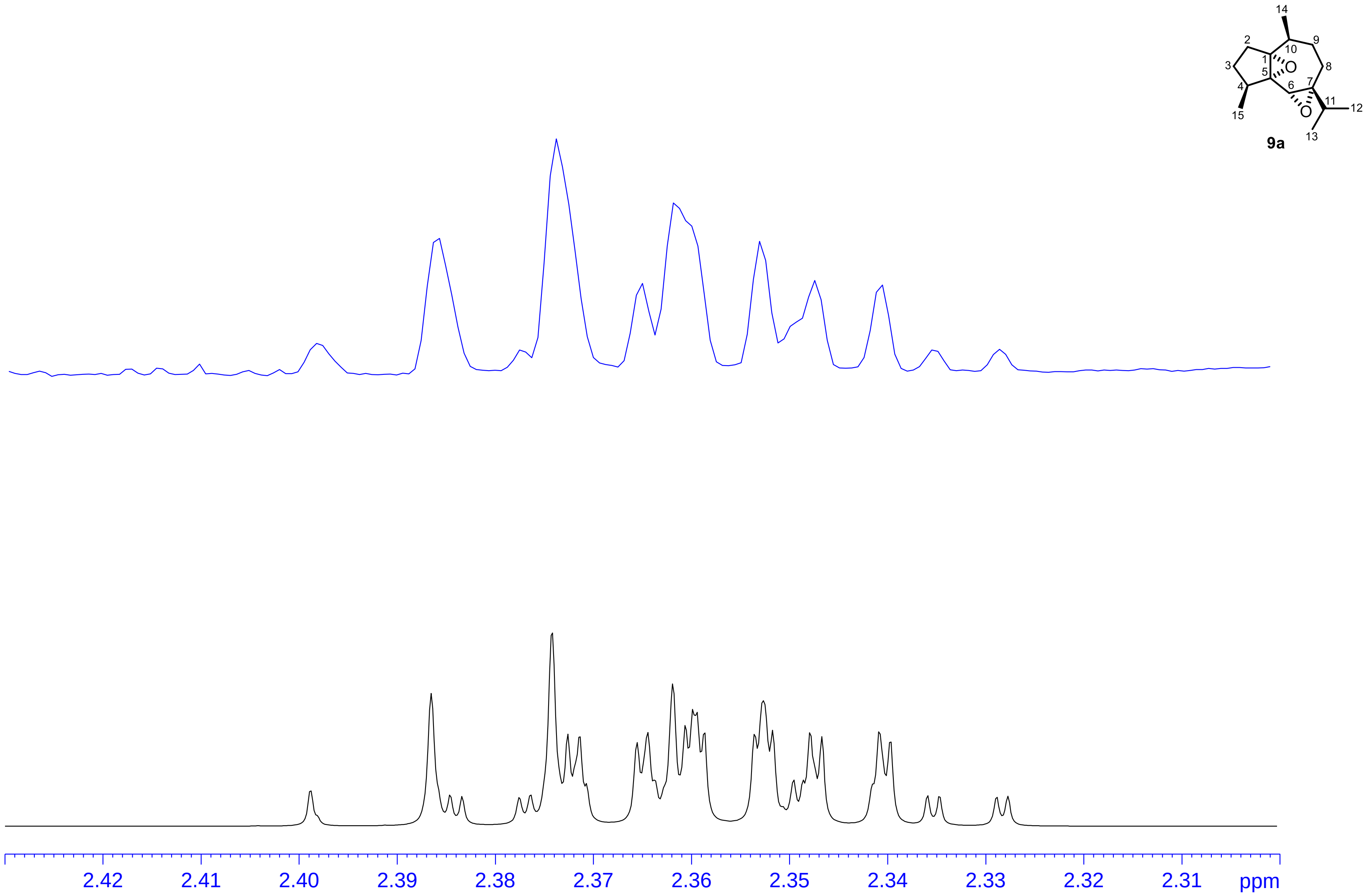
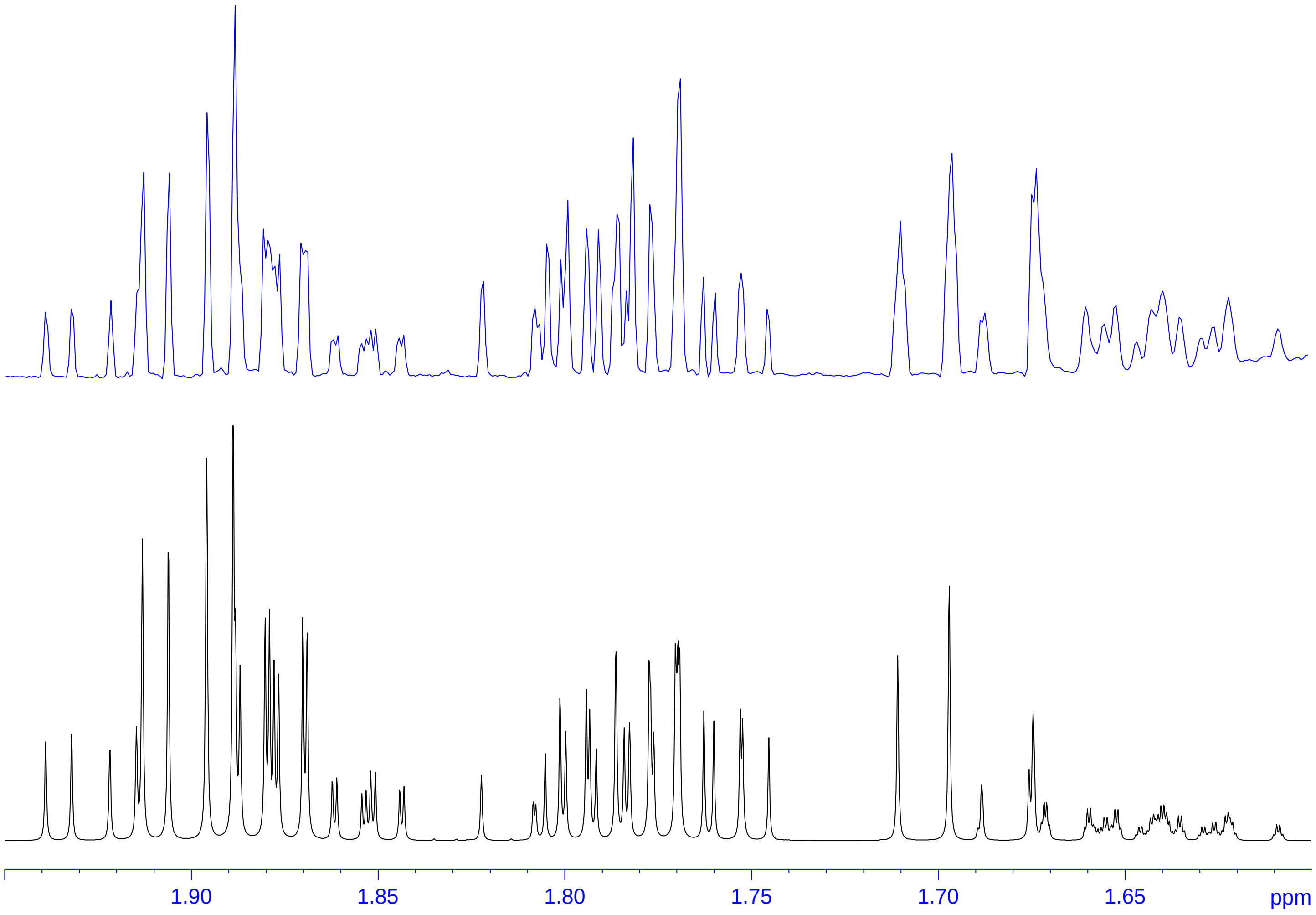
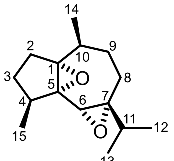


Fig. S13. Signals for H-8β, H-8α, H-2β, H-9α, H-2α and H-3α in the measured (top) (600 MHz, $CDCl_3$) and simulated (bottom) ^{1}H NMR spectrum of **9a**.





9a

Fig. S14. Signals for H-8β and H-8α in the measured (top) (600 MHz, $CDCl_3$) and simulated (bottom) ^{1}H NMR spectrum of **9a**.

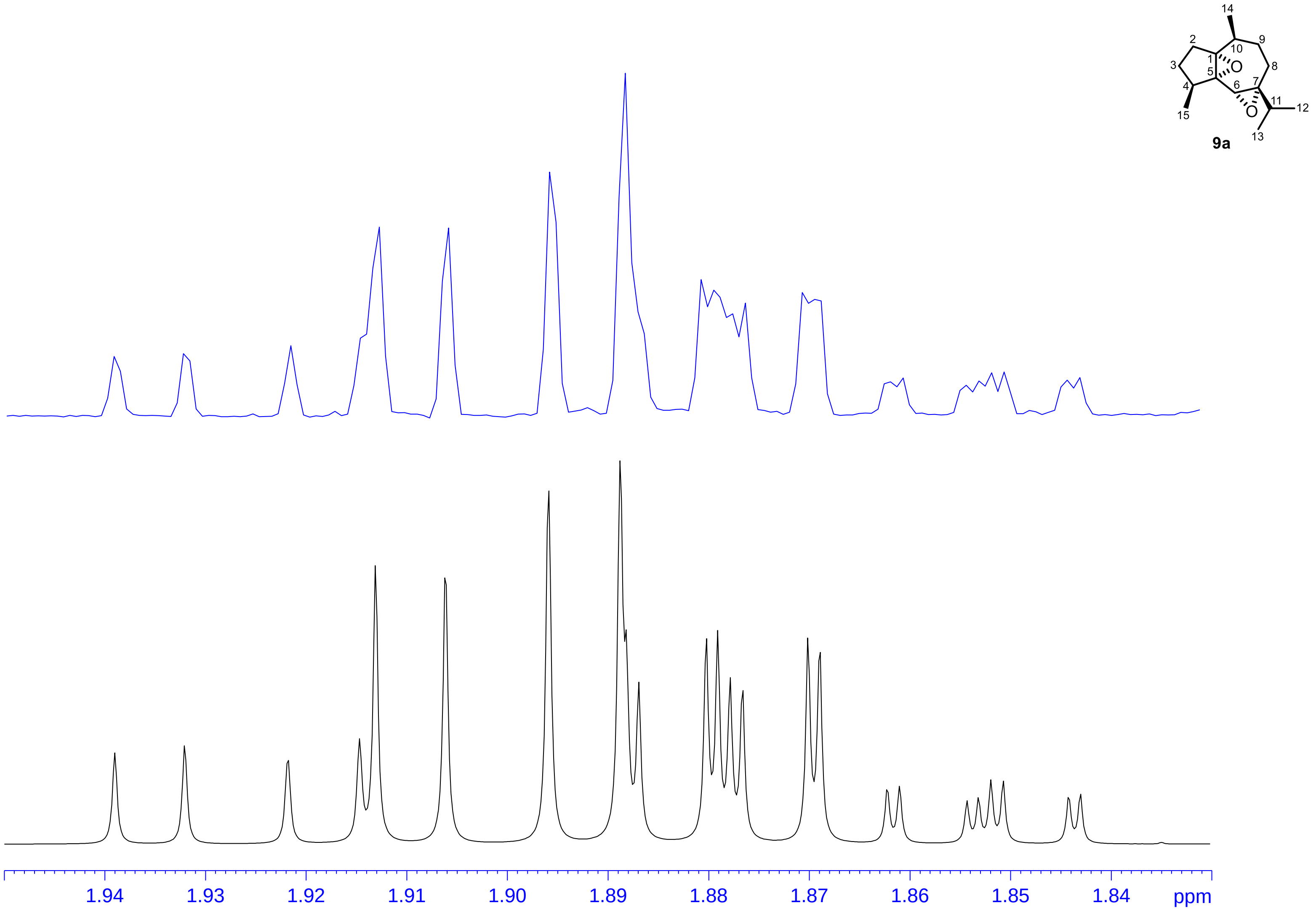


Fig. S15. Signals for H-2β and H-9α in the measured (top) (600 MHz, CDCl$_3$) and simulated (bottom) ^{1}H NMR spectrum of **9a**.

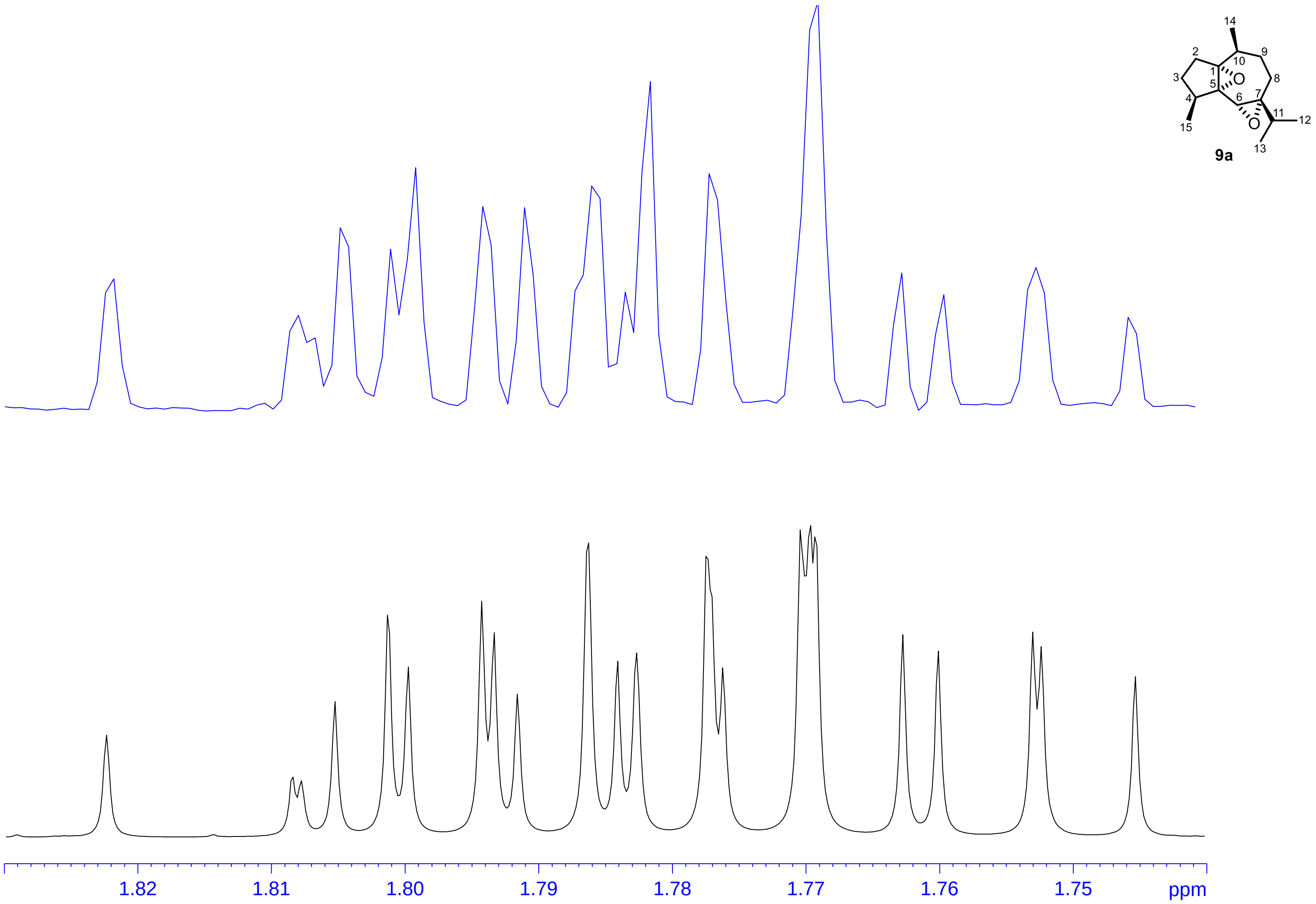


Fig. S16. Signals for H-2α and H-3α in the measured (top) (600 MHz, $CDCl_3$) and simulated (bottom) ^{1}H NMR spectrum of **9a**.

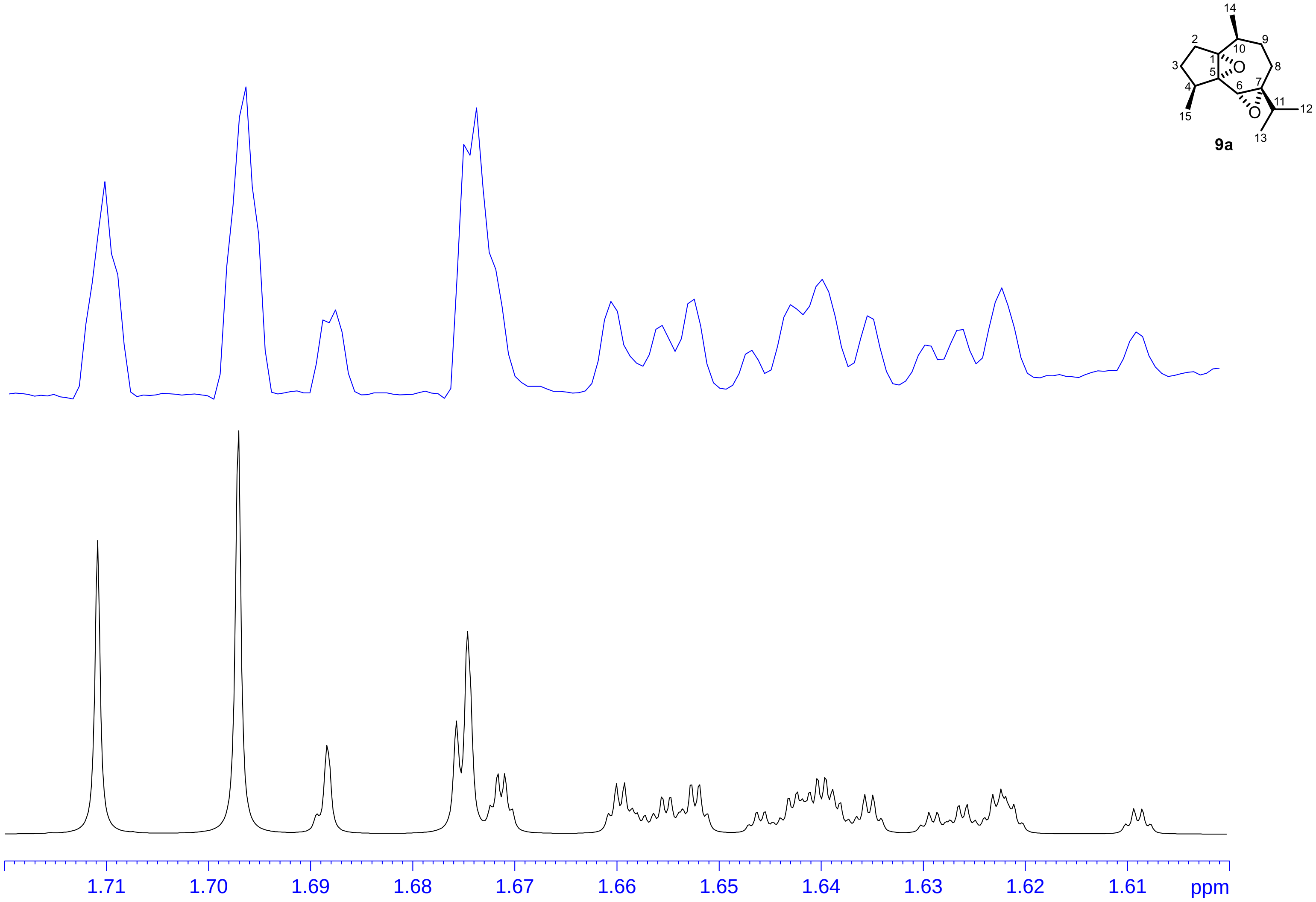


Fig. S17. Signals for H-11, H-9β and H-3β in the measured (top) (600 MHz, $CDCl_3$) and simulated (bottom) ^1H NMR spectrum of **9a**.

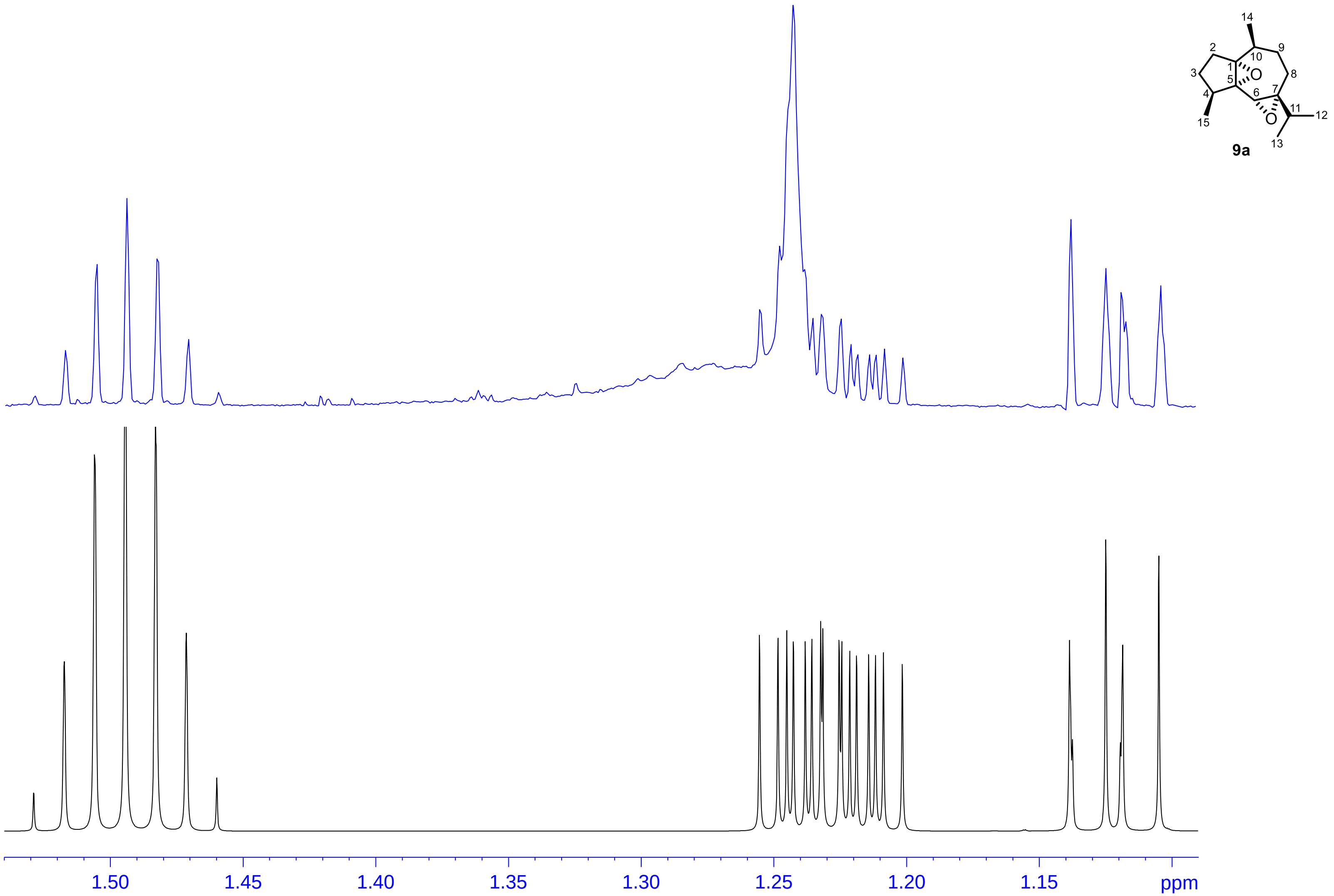


Fig. S18. ^{13}C NMR (150 MHz, CDCl$_3$) spectrum of **9a**.

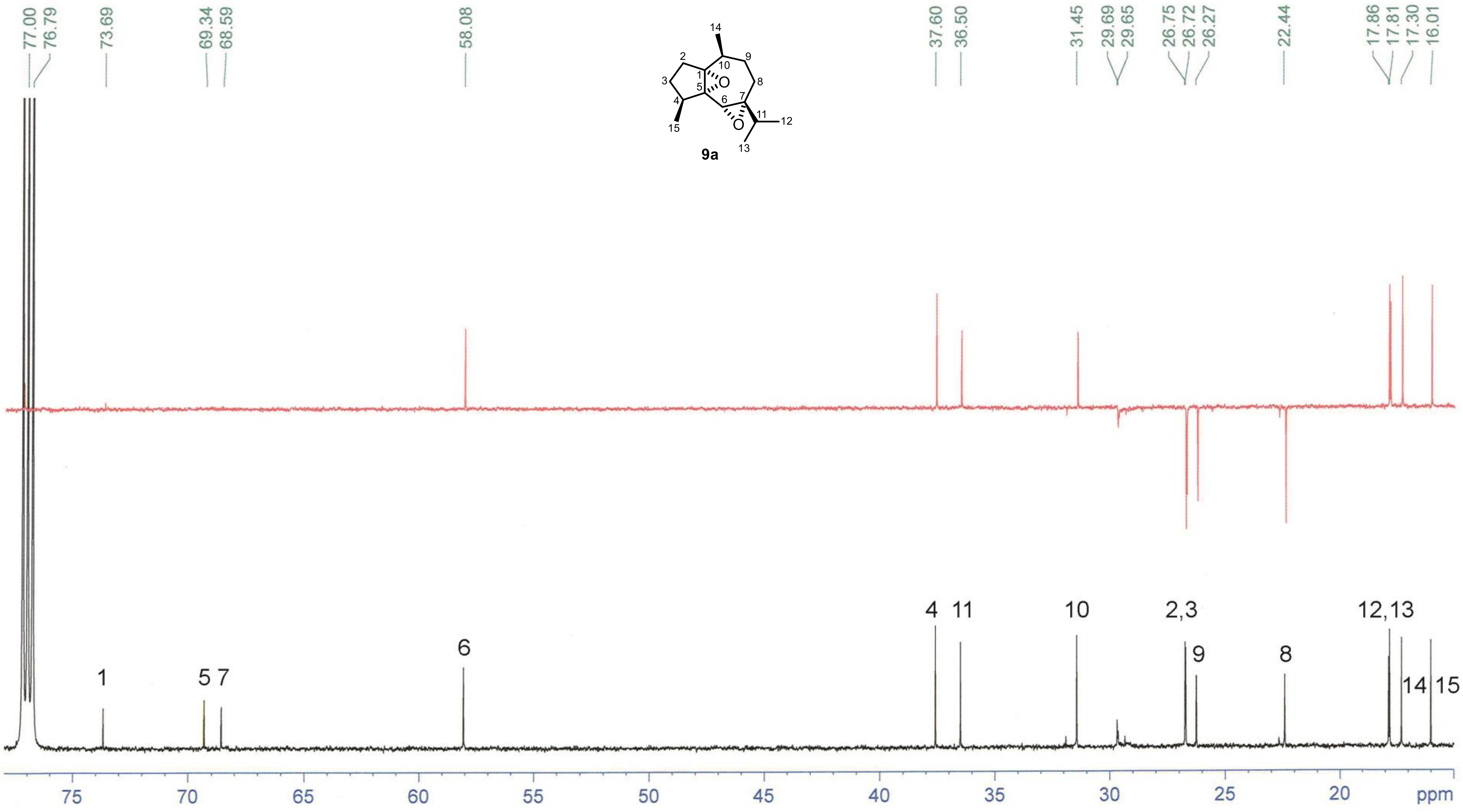
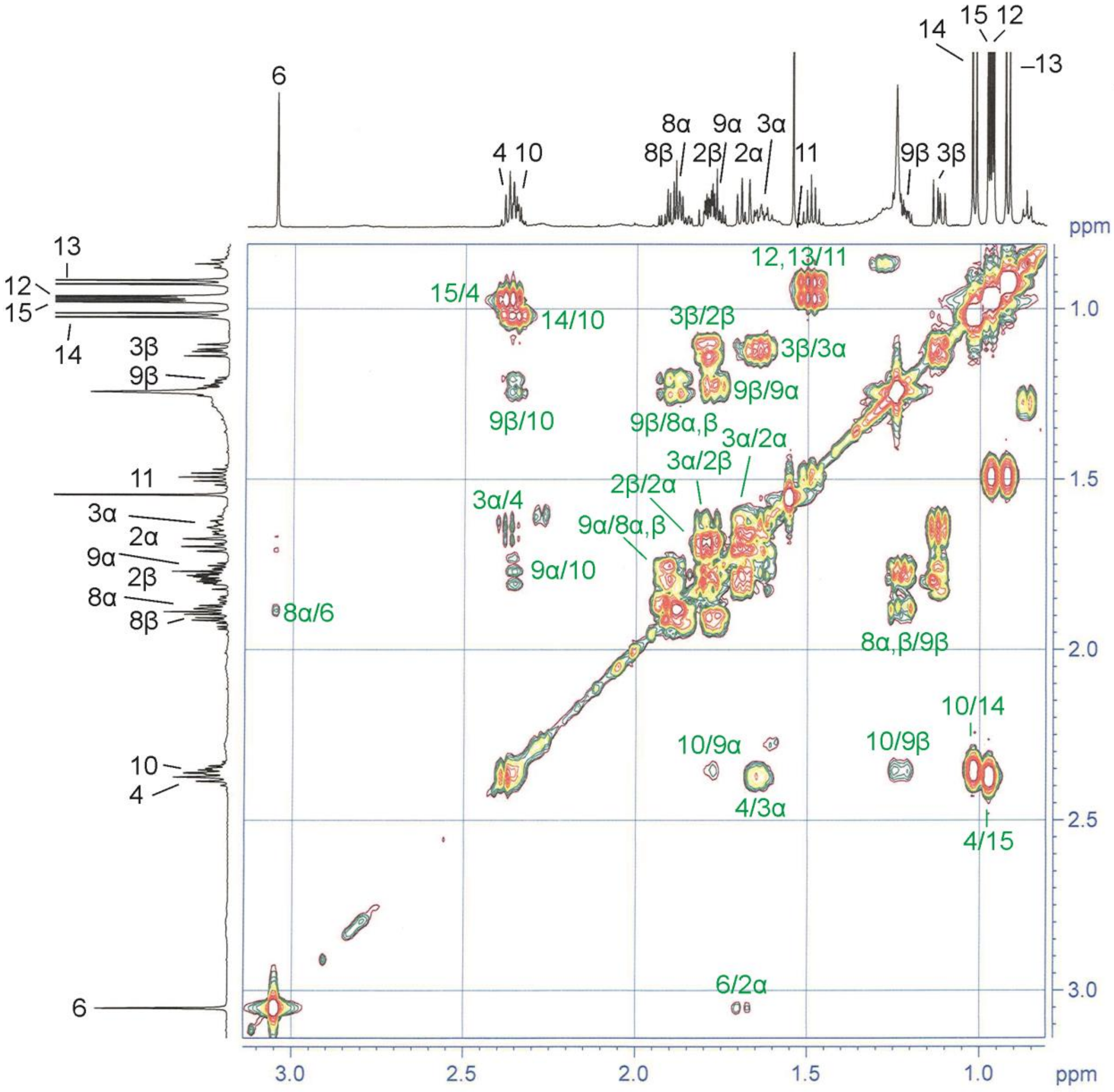
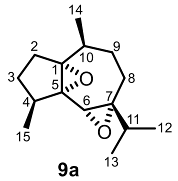


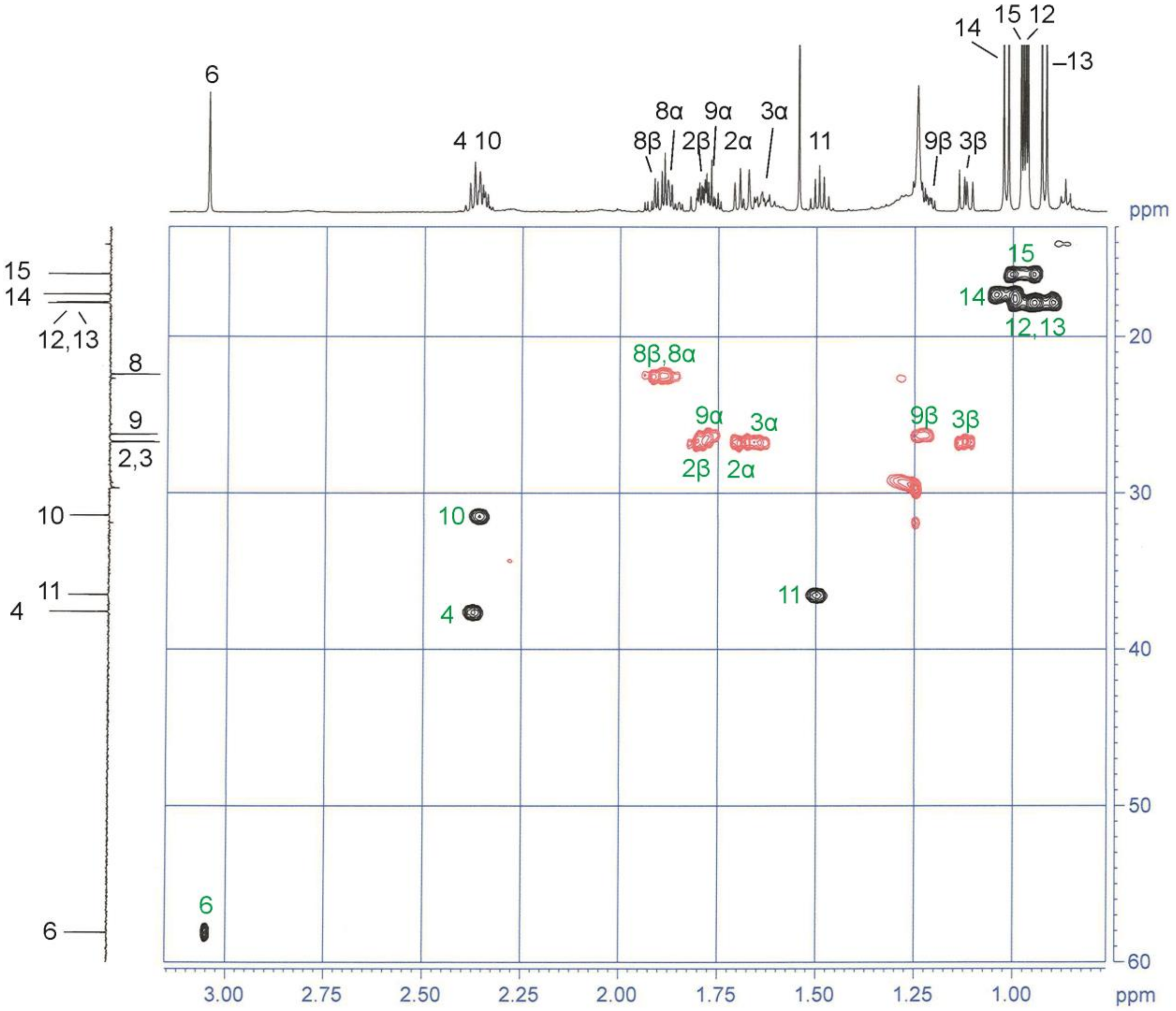
Fig. S19. COSY spectrum of **9a**.

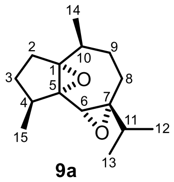




9a

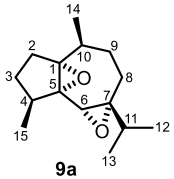
Fig. S20. HSQC spectrum of **9a**.





9a

Fig. S21. HMBC spectrum of **9a**.



9a

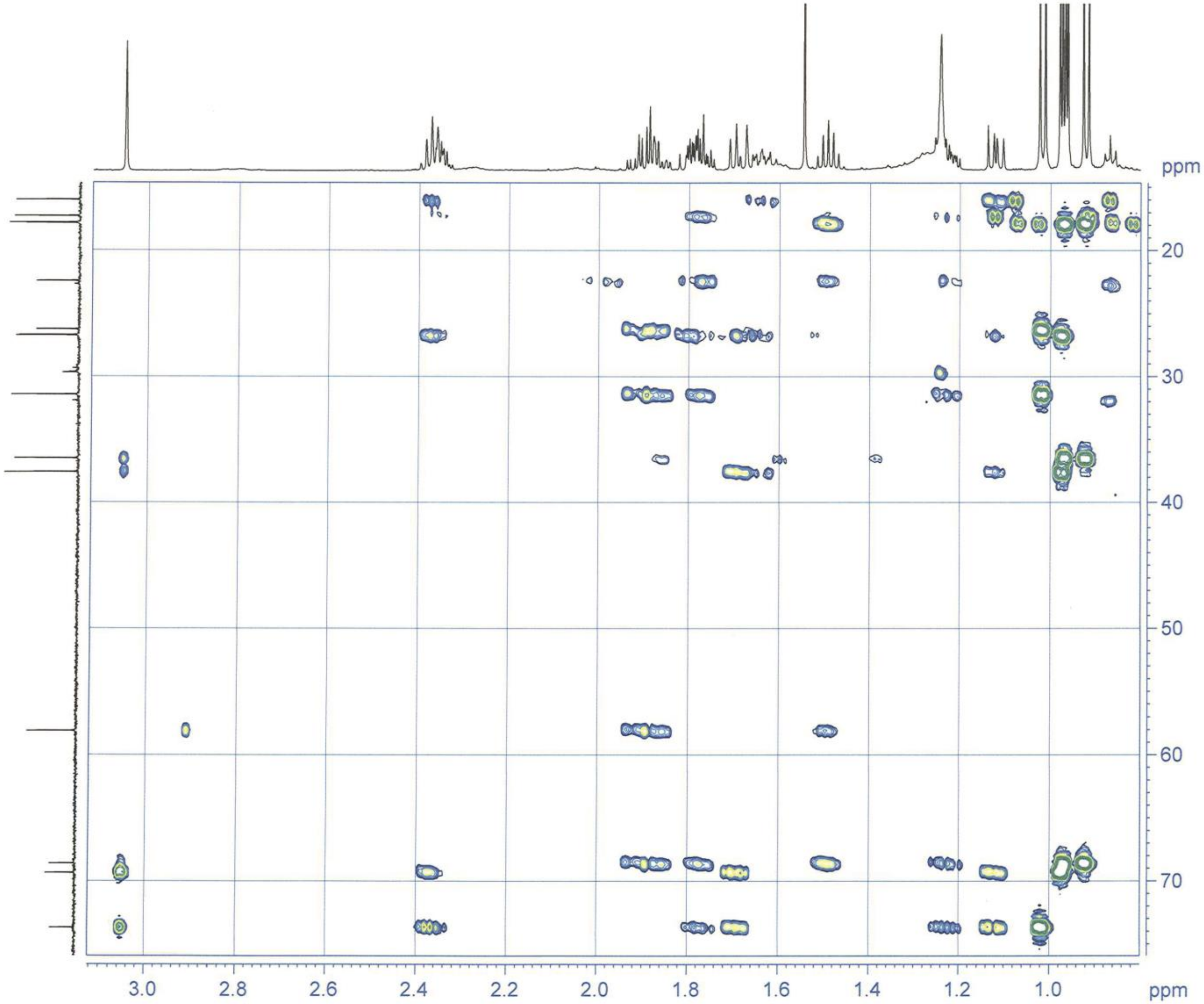
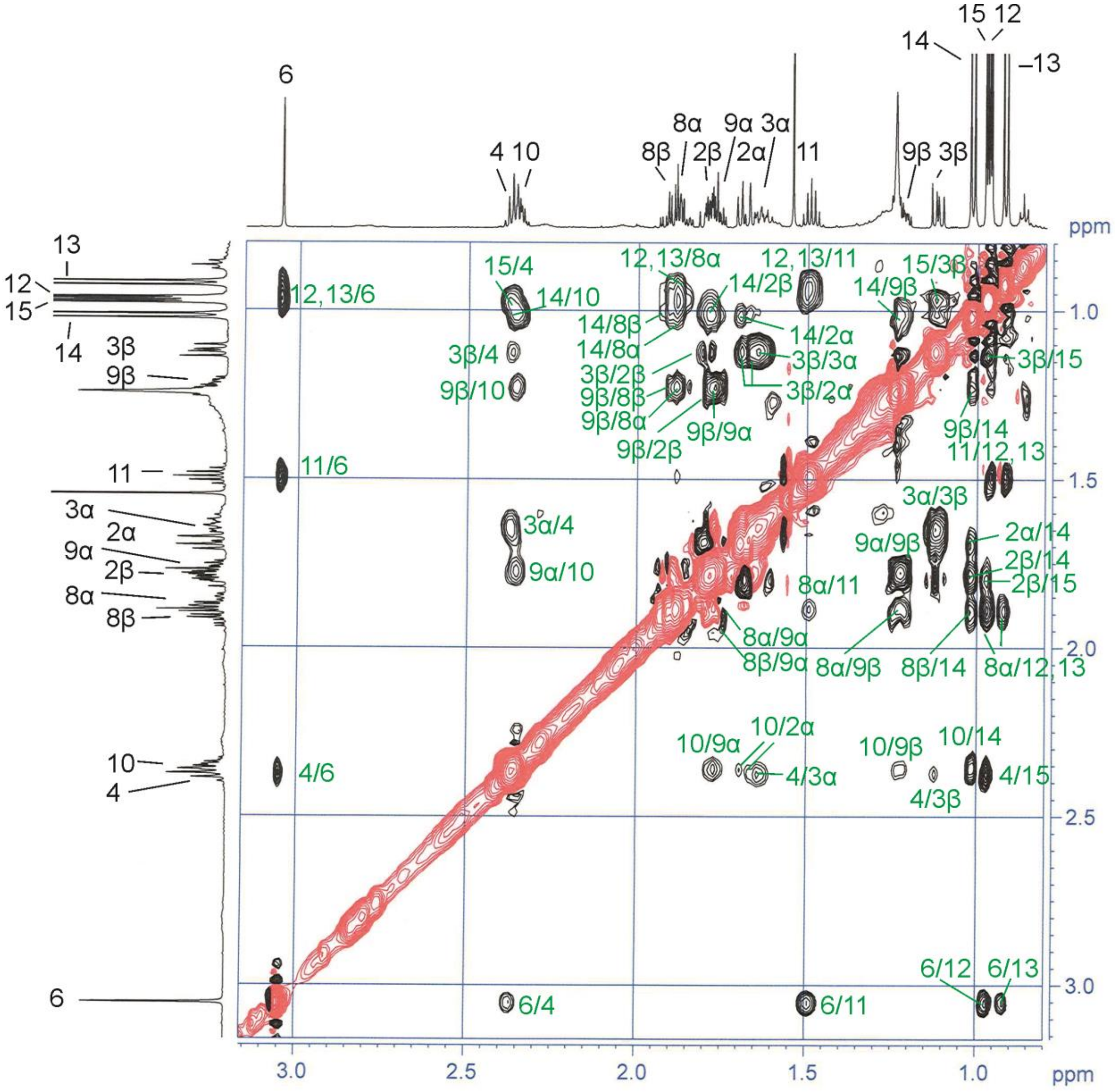
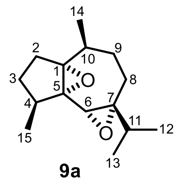


Fig. S22. NOESY spectrum of **9a**.





9a

Antimalarial Assay

Parasite culture. The chloroquine resistant strain (FCM29) of *P. falciparum* was provided by Mr. Michel Ratsimbason, Centre National d'Application de Recherche Pharmaceutique (CNARP), Antananarivo, Madagascar. The strain was maintained *in vitro* by using the Trager and Jensen's method reported earlier.[16, 17] The culture media consisted of standard RPMI 1640 (Sigma, St. Louis, MO) supplemented with 10% heat-inactivated (56 °C, 1 h) human type O+ serum, 25 mM $NaHCO_3$, 2 mM glutamine, and 1 M HEPES (Sigma, St. Louis, MO). The culture was maintained in type AB+ human red blood cell suspensions collected from healthy local donors and prepared in citrate-phosphate-dextrose anticoagulant (Sigma, St. Louis, MO) at a hematocrit of 2%. The parasite density was maintained below 2% parasitemia under an atmosphere of a gas mixture containing CO_2 (5%), O_2 (5%), and N_2 (90%) and at 37 °C. For each experiment the sample of stock sorbitol-synchronized culture was further diluted in culture medium containing sufficient non-infected type AB+ human erythrocytes to yield a final hematocrit of 2% and a parasitemia of 1%.

Fluorimetric susceptibility test. The synchronized rings from cultures (hematocrit 2% and parasitemia 1%) were used to test serial dilutions of extracts in 96-well culture plates. Cultures of *P. falciparum* were placed in a humidified, air-sealed container, flushed with the gas mixture described above, and incubated at 37 °C. Parasites were allowed to grow for a 48-hour incubation period, after which a 150 μL aliquot of culture was transferred to a new 96-well flat bottom plate. Fifty microliters of the fluorochrome mixture, which consists of PicoGreen® (Molecular Probes, Inc., Eugene, OR), 10 mM Tris-HCl, 1 mM EDTA, pH 7.5 (TE buffer), and 2% Triton X-100 diluted with double-distilled water, was then added to liberate and label the parasitic DNA. The plates were then incubated for 5−30 minutes in the dark. The fluorescence signal, measured as relative fluorescence units (RFU) was quantified with a fluorescence microplate reader (FLx 800; Bio-Tek Instruments, Inc., Winooski, VT) at 485/20 nm excitation and 528/20 nm emission. Simultaneously, the RFU from positive (quinine: IC_{50} = 3.5 μg/mL) and negative (solvent, MeOH) control samples were also performed.

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