

Anticholinesterase and Antityrosinase Secondary Metabolites from the Fungus *Xylobolus subpileatus*

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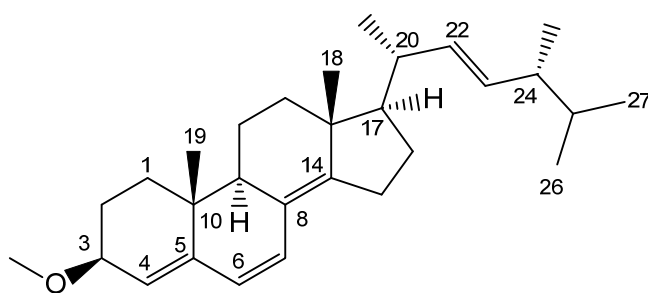
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Spectra and spectral data on compound **1**



3 β -methoxy-ergosta-6,8¹⁴,22-trien (**1**): amorphous solid; $\alpha_D^{25}+87.8$ (c 0.42, CHCl₃, 25.4 °C); HRESIMS m/z 377,31924 [$M + H - CH_3OH$]⁺ (Δ 2.2 ppm; C₂₈H₄₁); HRESI-MSMS (CID = 15%, 30%, 45%; rel int %) m/z 293 (16), 251 (24).

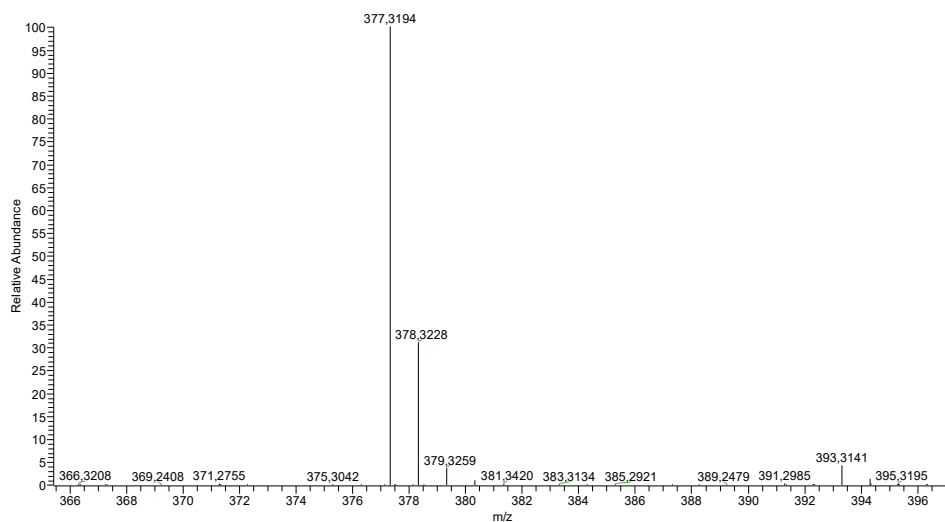


Figure S1. HRESI-MS spectrum of compound **1**

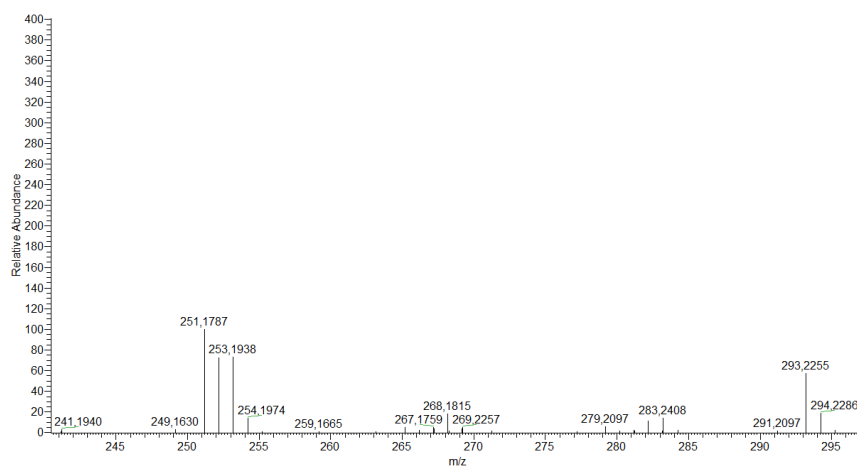


Figure S2. MS-MS spectrum of compound **1**

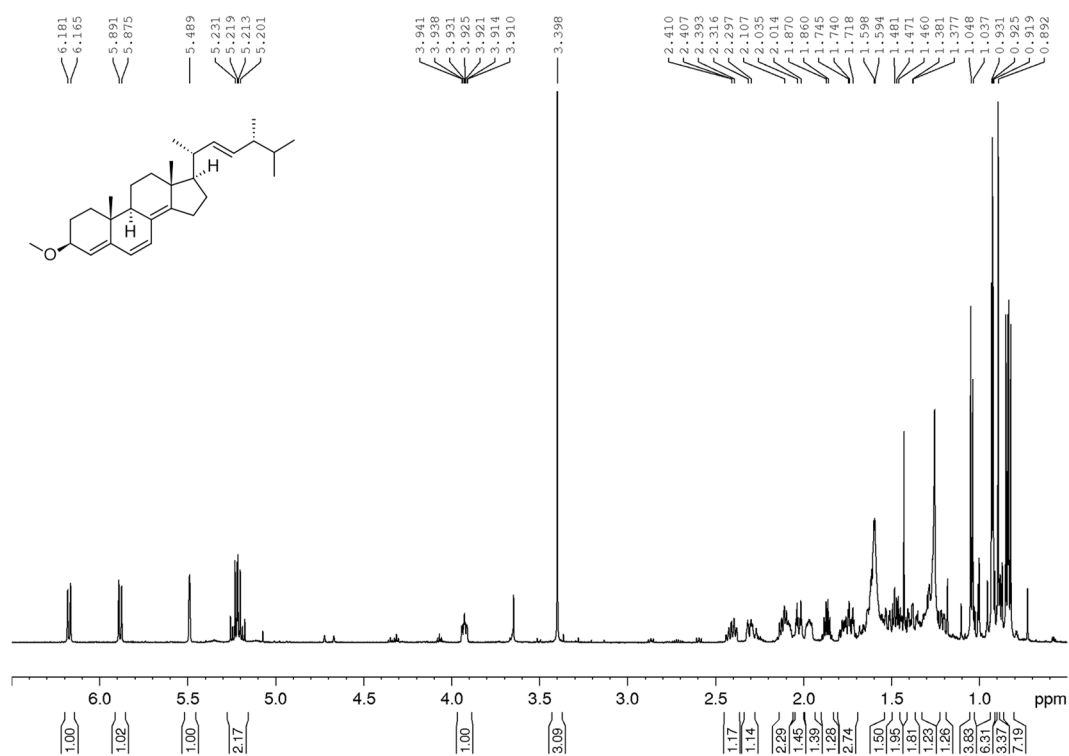


Figure S1. ¹H spectrum of compound **1** (600 MHz, CDCl₃, 295 K)

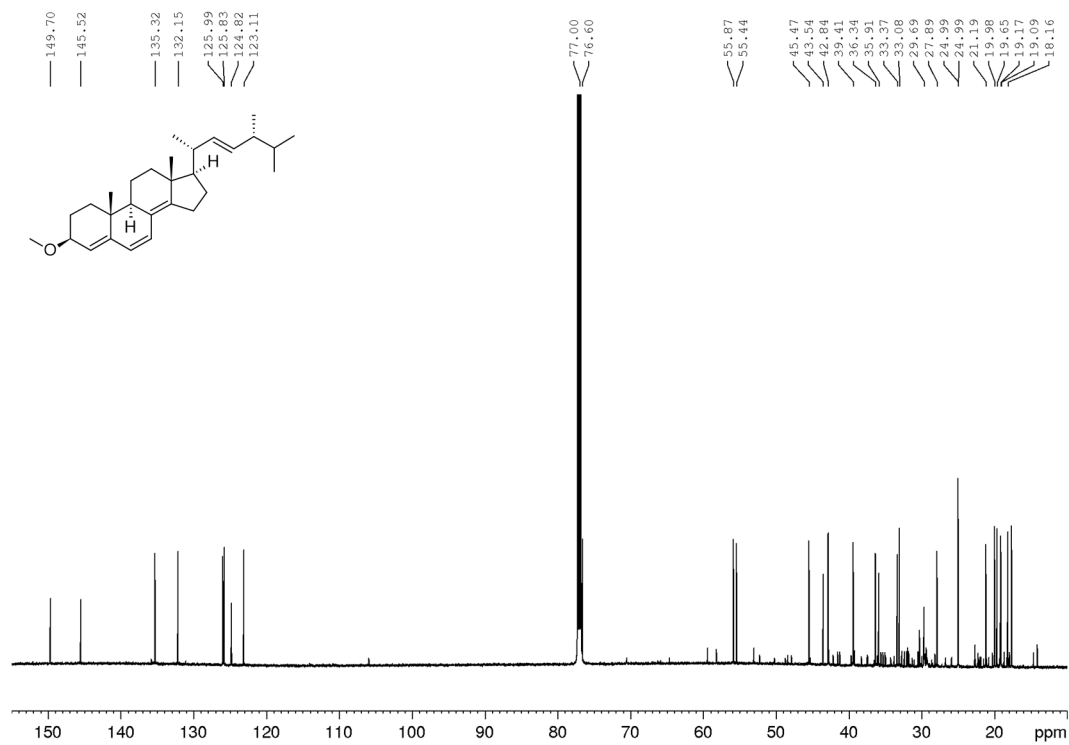


Figure S2. ¹³C spectrum of compound **1** (150 MHz, CDCl₃, 295 K)

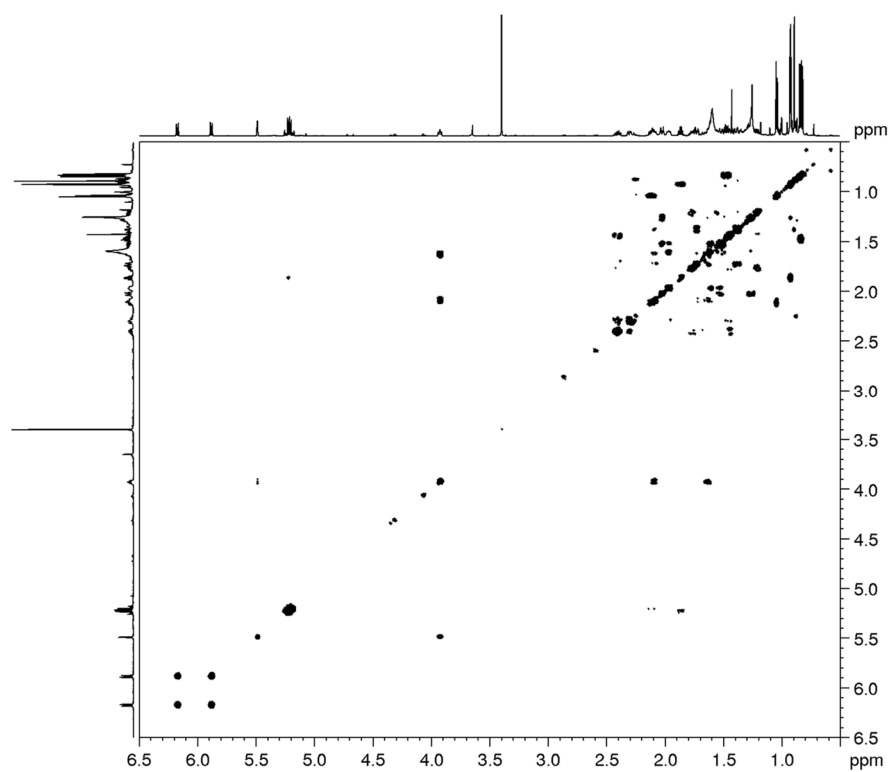


Figure S3. COSY spectrum of compound **1** (600 MHz, CDCl₃, 295 K)

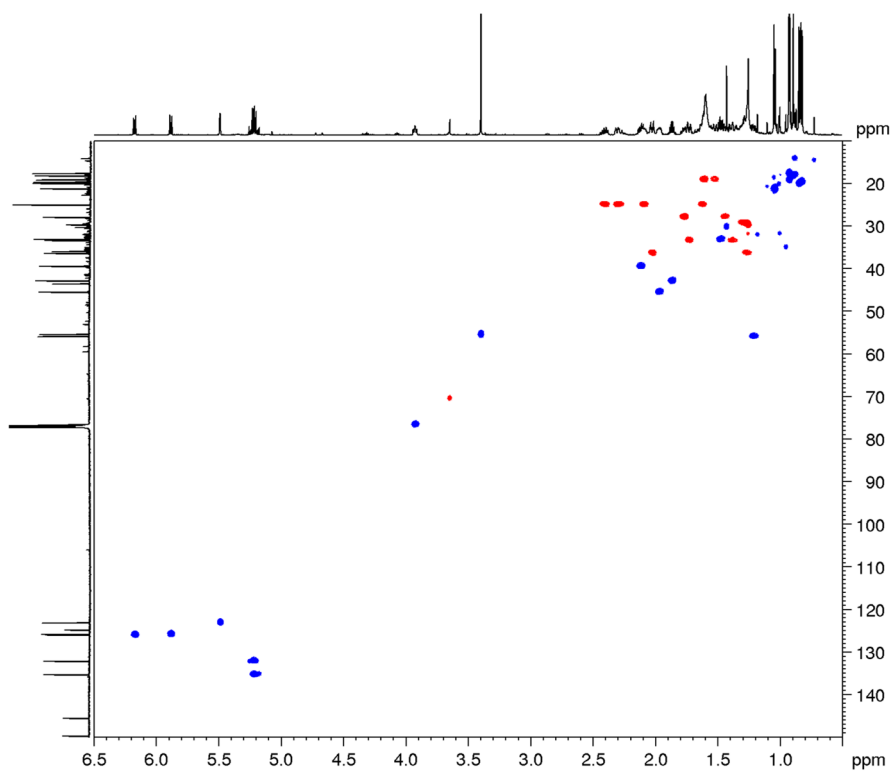


Figure S4. DEPT-edited HSQC spectrum of compound **1** (600 MHz, CDCl₃, 295 K)

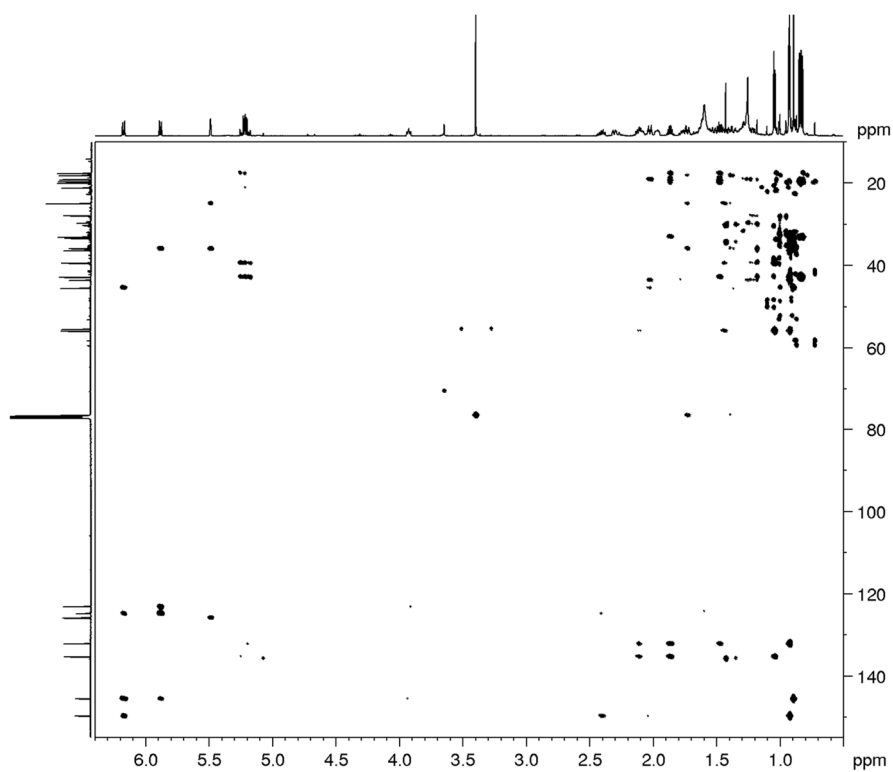


Figure S5. HMBC spectrum of compound **1** (600 MHz, CDCl₃, 295 K)

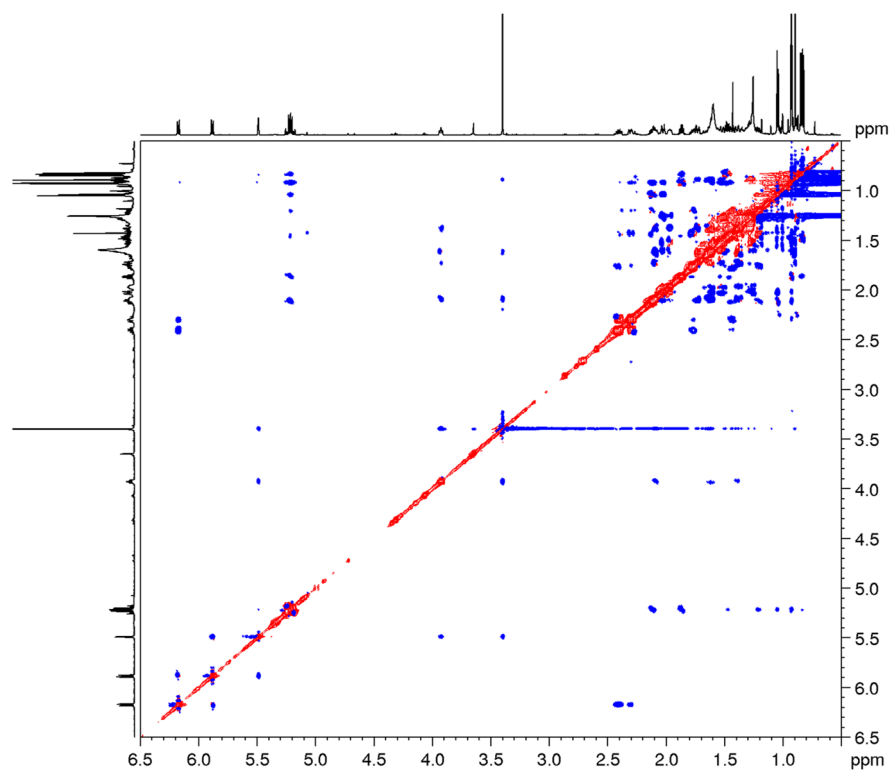
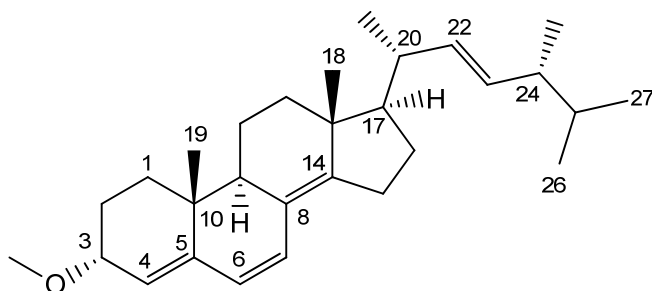


Figure S6. ROESY spectrum of compound **1** (600 MHz, CDCl₃, 295 K)

Spectra and spectral data on compound **2**



3 α -methoxy-ergosta-6,8¹⁴,22-trien (**2**): amorphous solid; α_D^{25} -10.0 (*c* 0.09, CHCl₃, 25.5 °C); HRESIMS *m/z* 377,3194 [M + H - CH₃OH]⁺ (Δ 2.2 ppm; C₂₈H₄₁); HRESI-MSMS (CID = 15%, 30%, 45%; rel int %) *m/z* .293, 251.

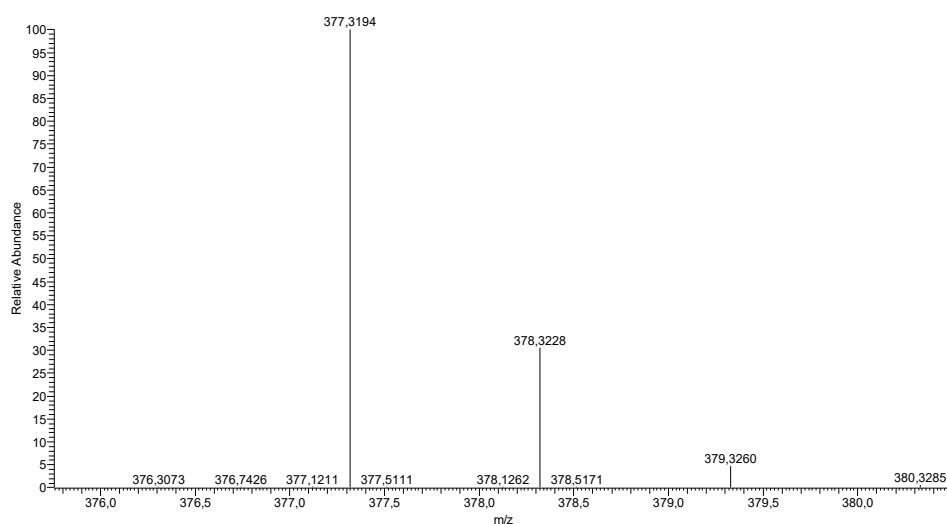


Figure S9. HRESI-MS spectrum of compound **2**

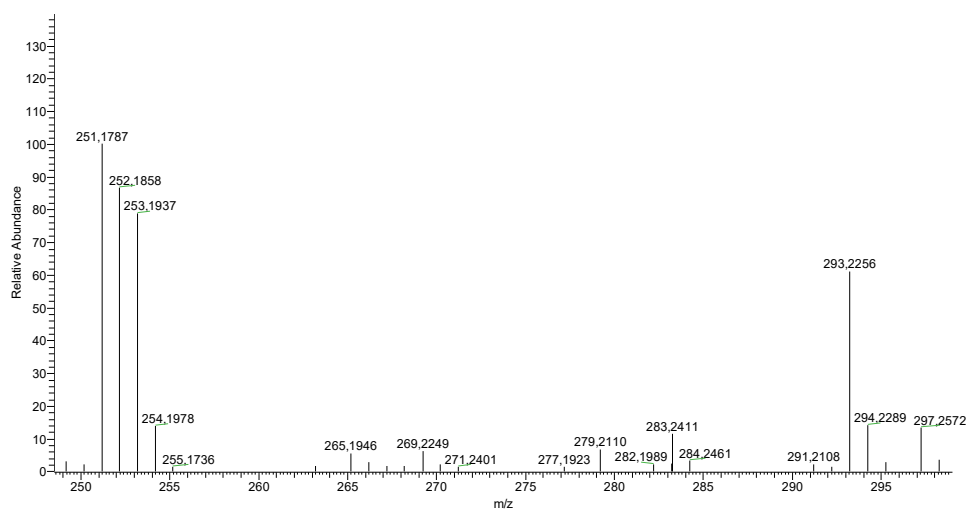


Figure S10. MS-MS spectrum of compound **2**

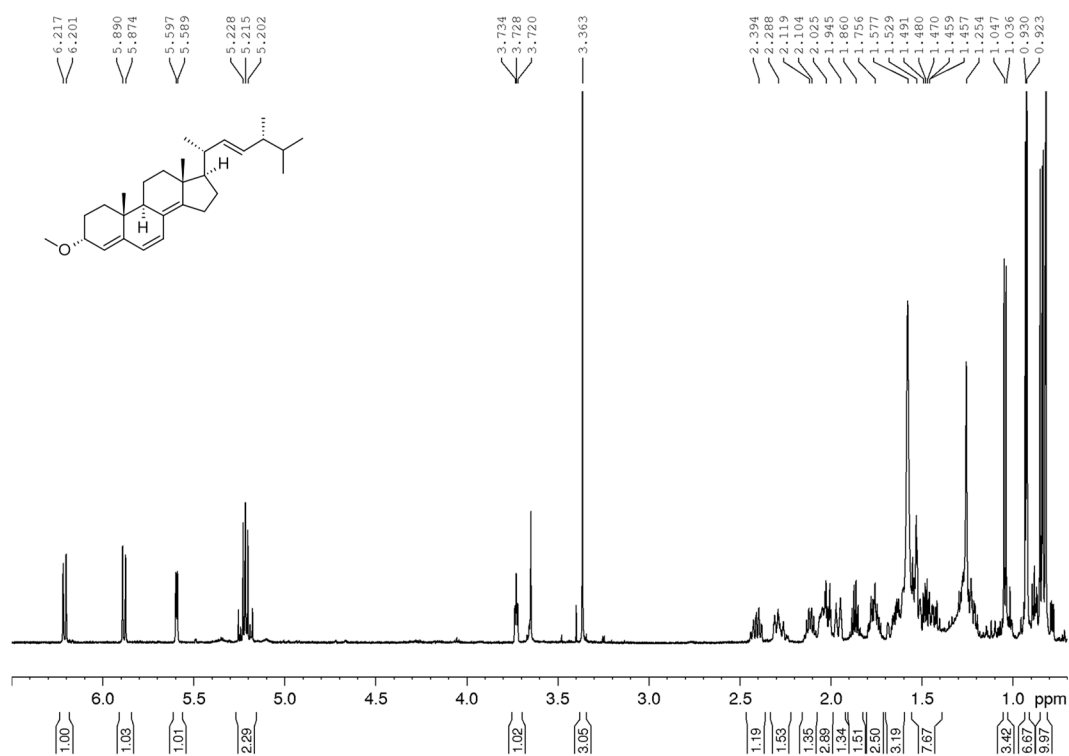


Figure S7. ¹H spectrum of compound **2** (600 MHz, CDCl₃, 295 K)

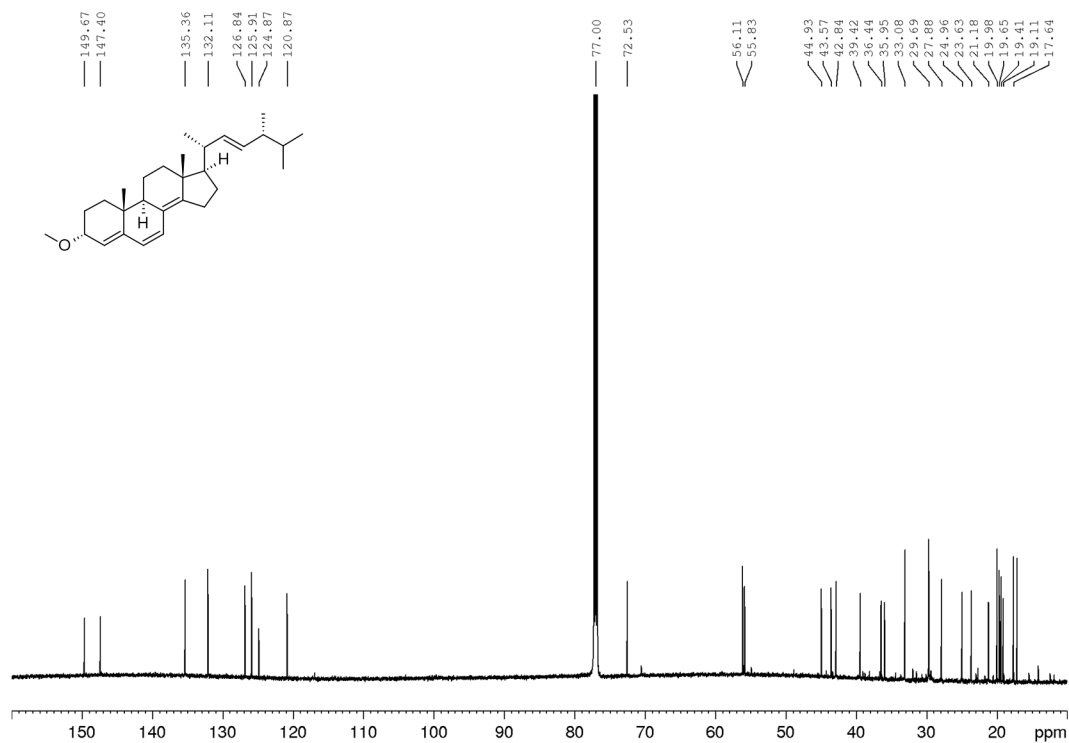


Figure S8. ¹³C spectrum of compound **2** (150 MHz, CDCl₃, 295 K)

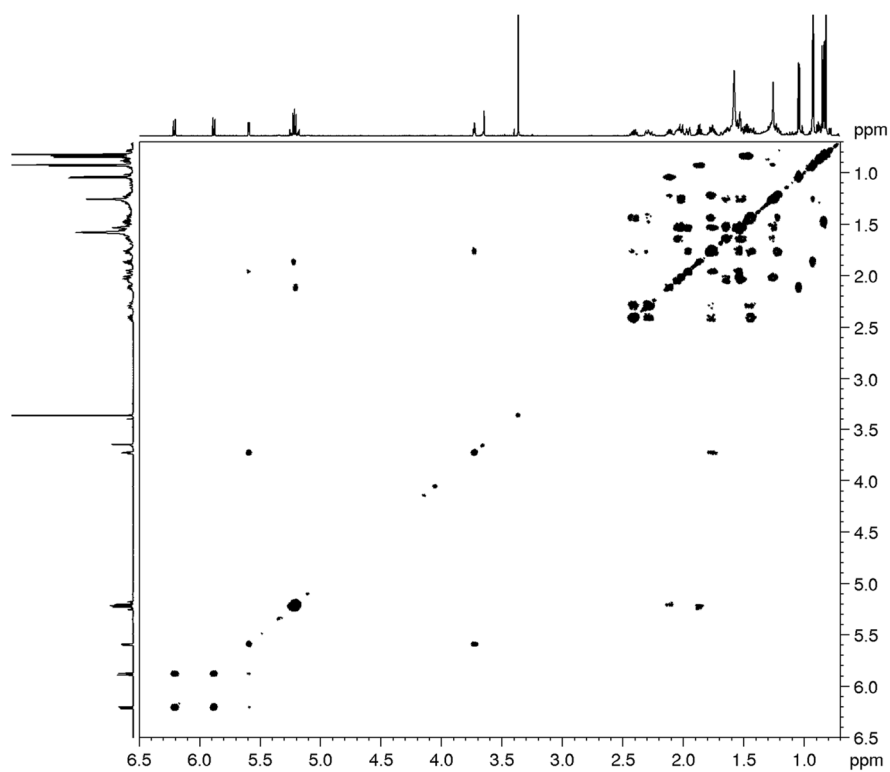


Figure S9. COSY spectrum of compound **2** (600 MHz, CDCl₃, 295 K)

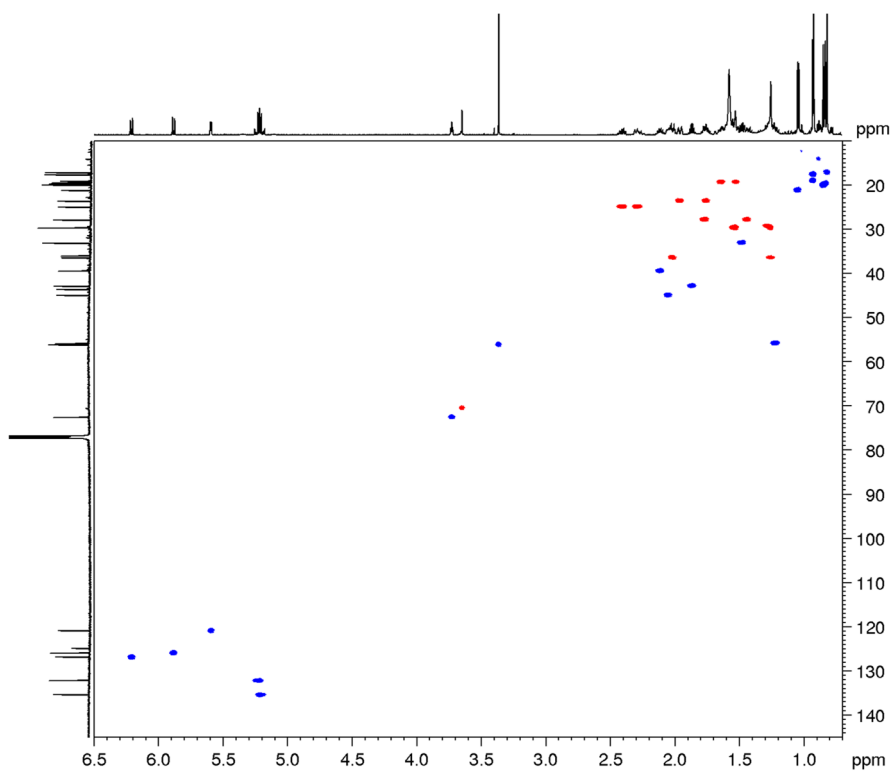


Figure S10. DEPT-edited HSQC spectrum of compound **2** (600 MHz, CDCl₃, 295 K)

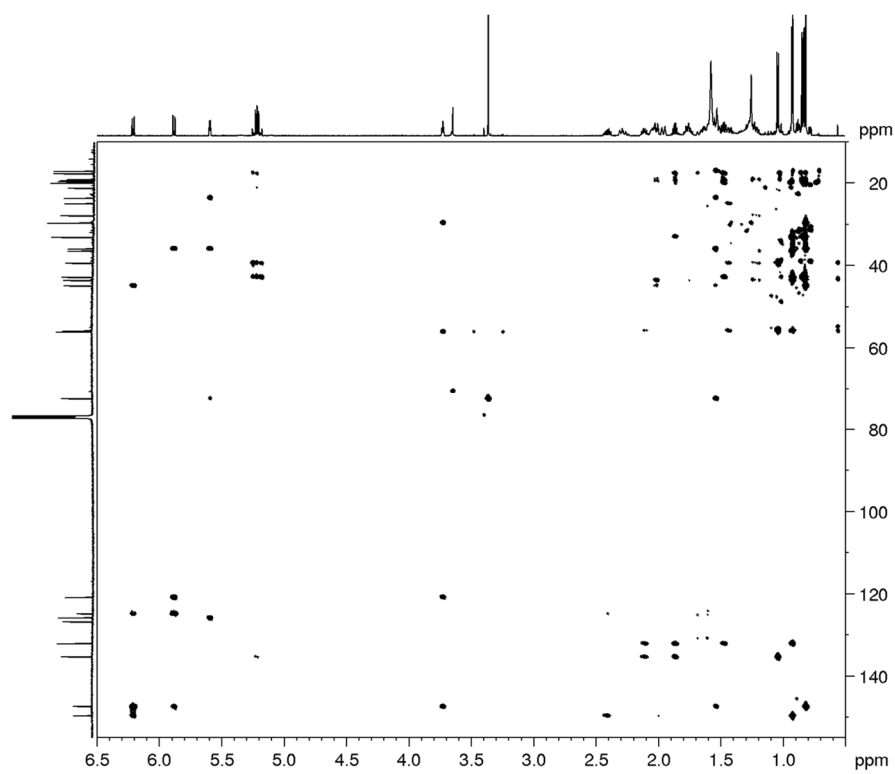


Figure S11. HMBC spectrum of compound **2** (600 MHz, CDCl₃, 295 K)

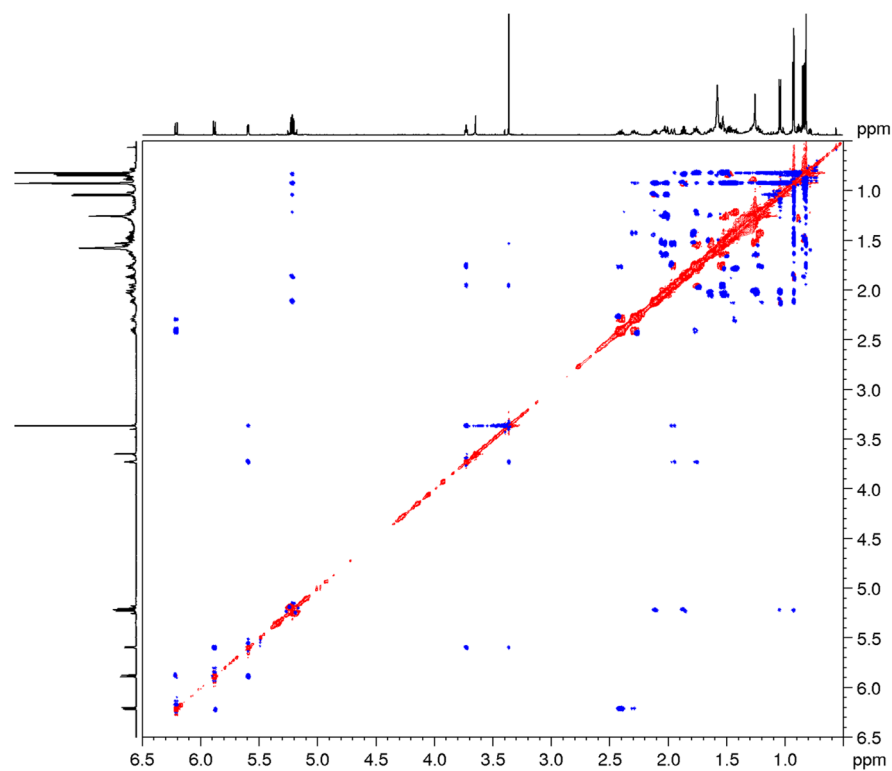
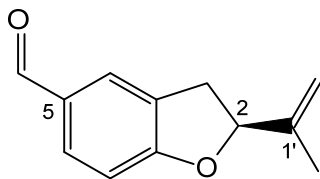


Figure S12. ROESY spectrum of compound **2** (600 MHz, CDCl₃, 295 K)

Spectra and spectral data on compound 3



Fomannoxin (3): amorphous solid; HRESIMS m/z 189,09061 $[M + H]^+$ (Δ 2.1 ppm; $C_{12}H_{13}O_2$); HRESI-MSMS (CID = 15%, 30%, 45%; rel int %) m/z 161 (97), 143 (23), 133 (33).

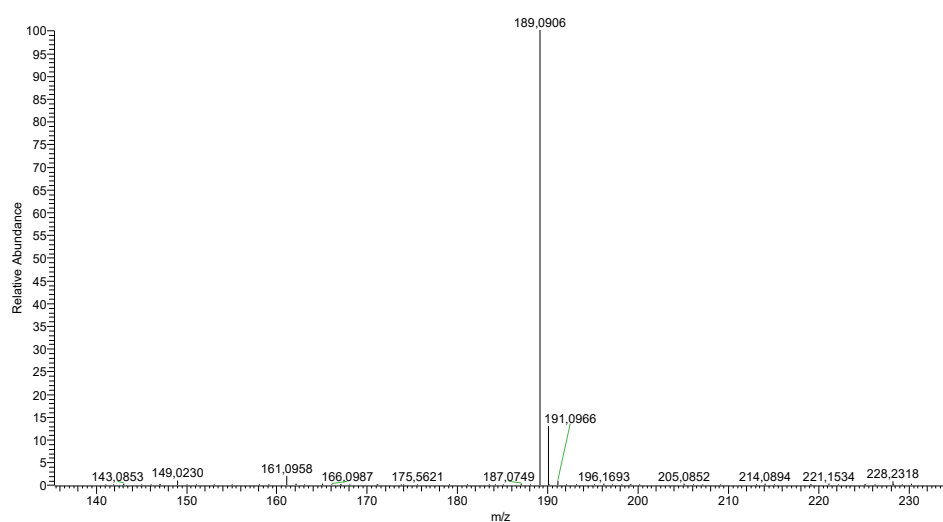


Figure S13. HRESI-MS spectrum of compound 3

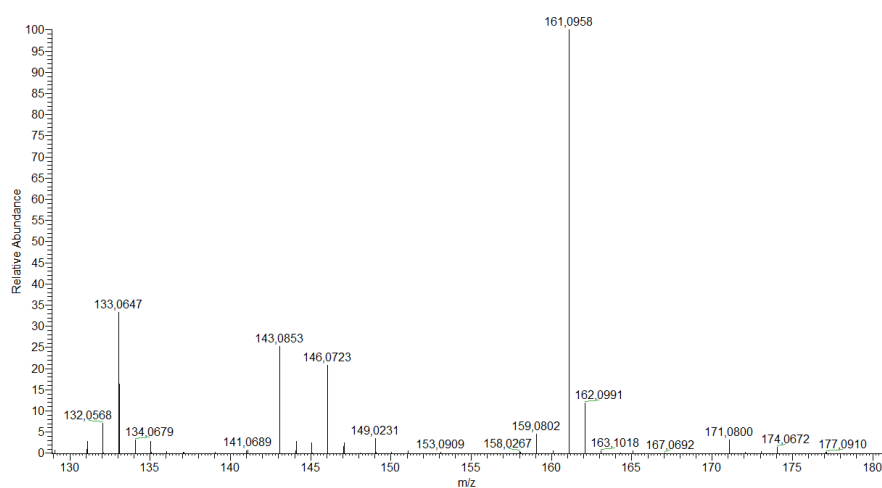


Figure S14. MS-MS spectrum of compound 3

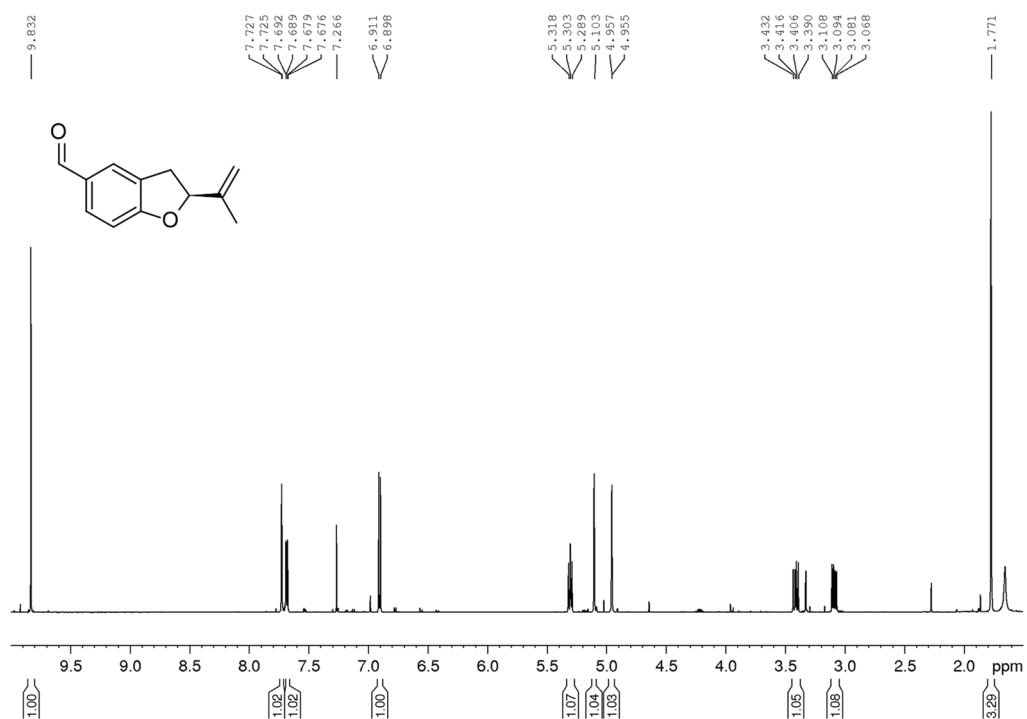


Figure S15. ¹H spectrum of compound 3 (600 MHz, CDCl₃, 295 K)

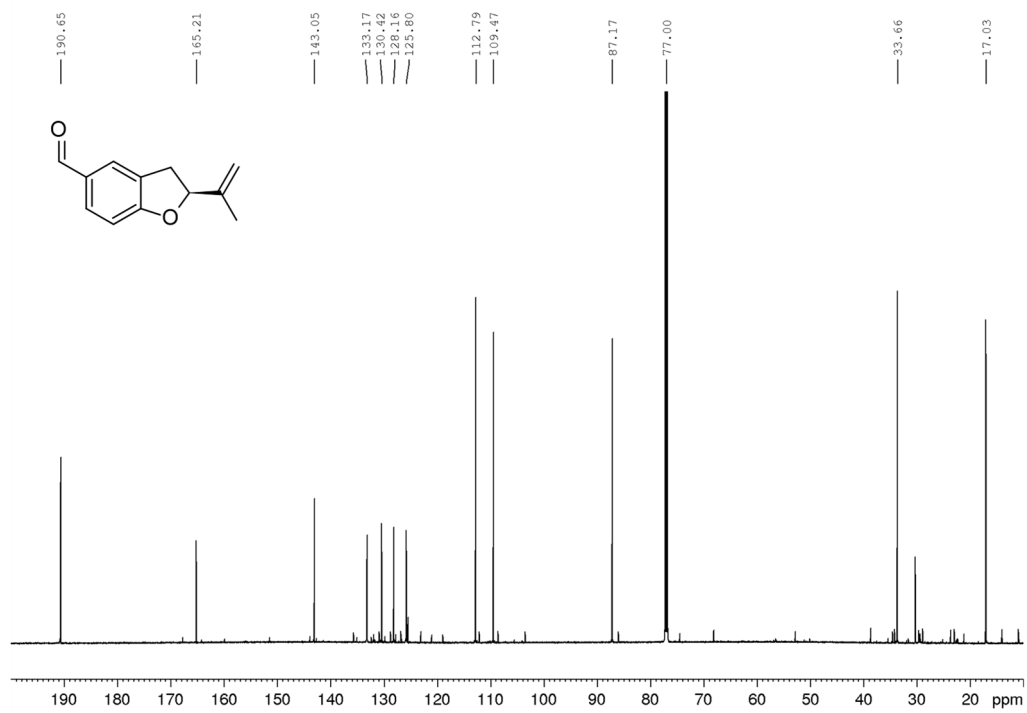


Figure S16. ¹³C spectrum of compound 3 (150 MHz, CDCl₃, 295 K)

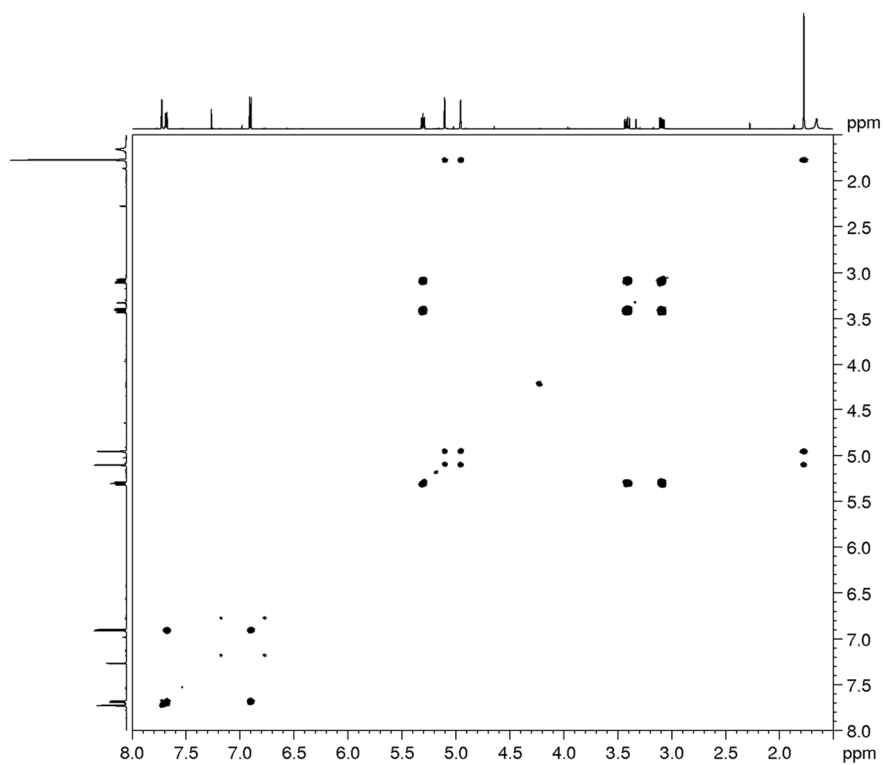


Figure S17. COSY spectrum of compound **3** (600 MHz, CDCl₃, 295 K)

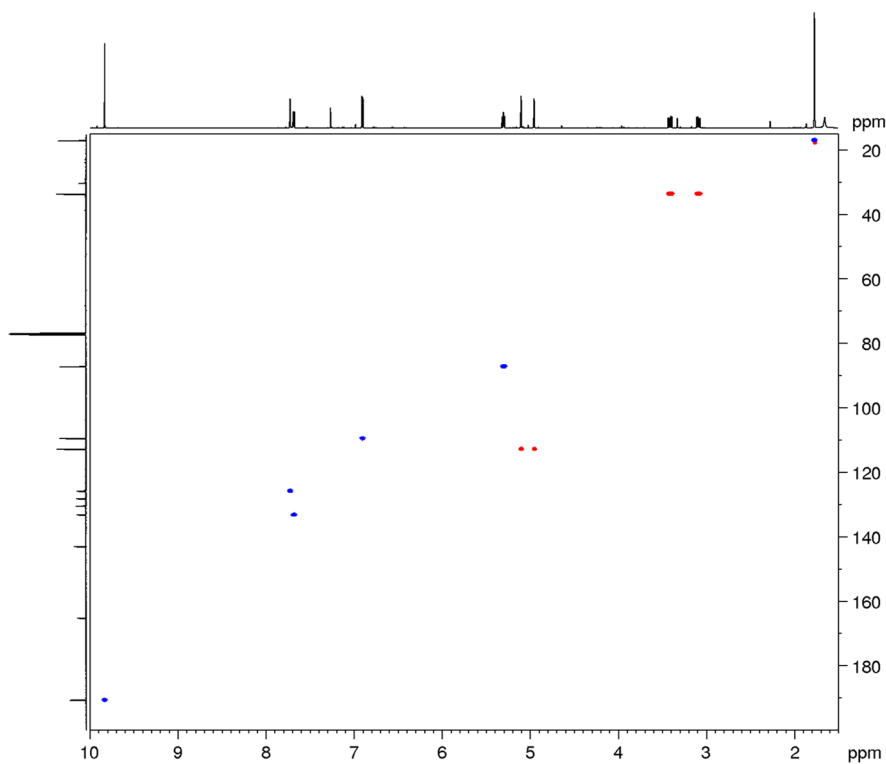


Figure S18. DEPT-edited HSQC spectrum of compound **3** (600 MHz, CDCl₃, 295 K)

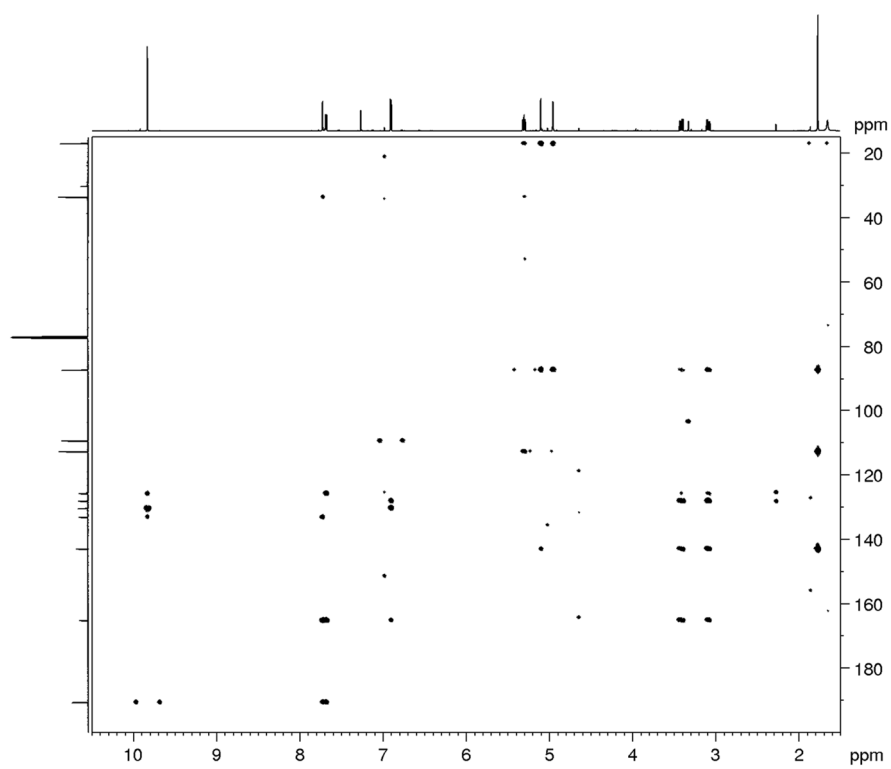
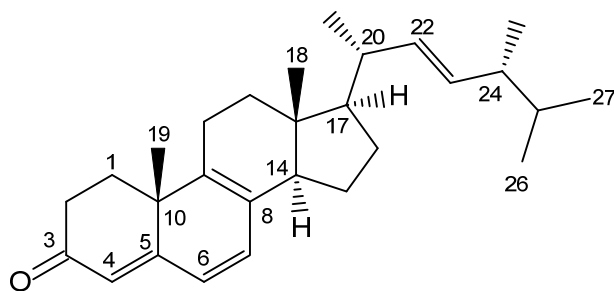


Figure S19. HMBC spectrum of compound **3** (600 MHz, CDCl_3 , 295 K)

Spectra and spectral data on compound 4



Ergosta-4,6,8[9]22-tetraene-3-one (4): amorphous solid; HRESIMS m/z 393.31458 $[M + H]^+$ (Δ 1.6 ppm; $C_{28}H_{44}O$); HRESI-MSMS (CID = 15%, 30%, 45%; rel int %) m/z 268 (54).

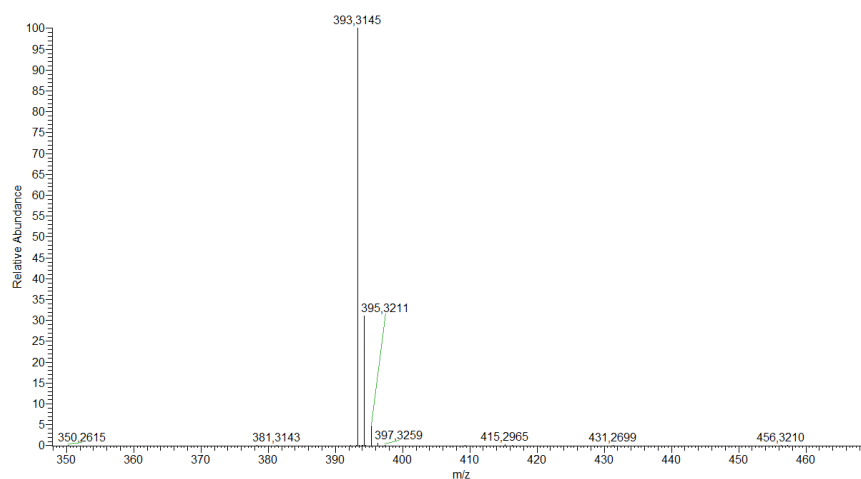


Figure S20. HRESI-MS spectrum of compound 4

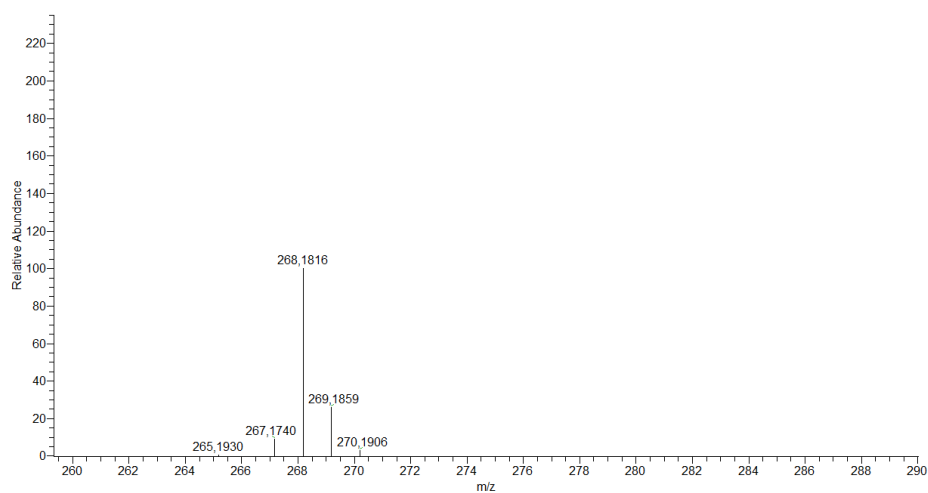


Figure S21. MS-MS spectrum of compound 4

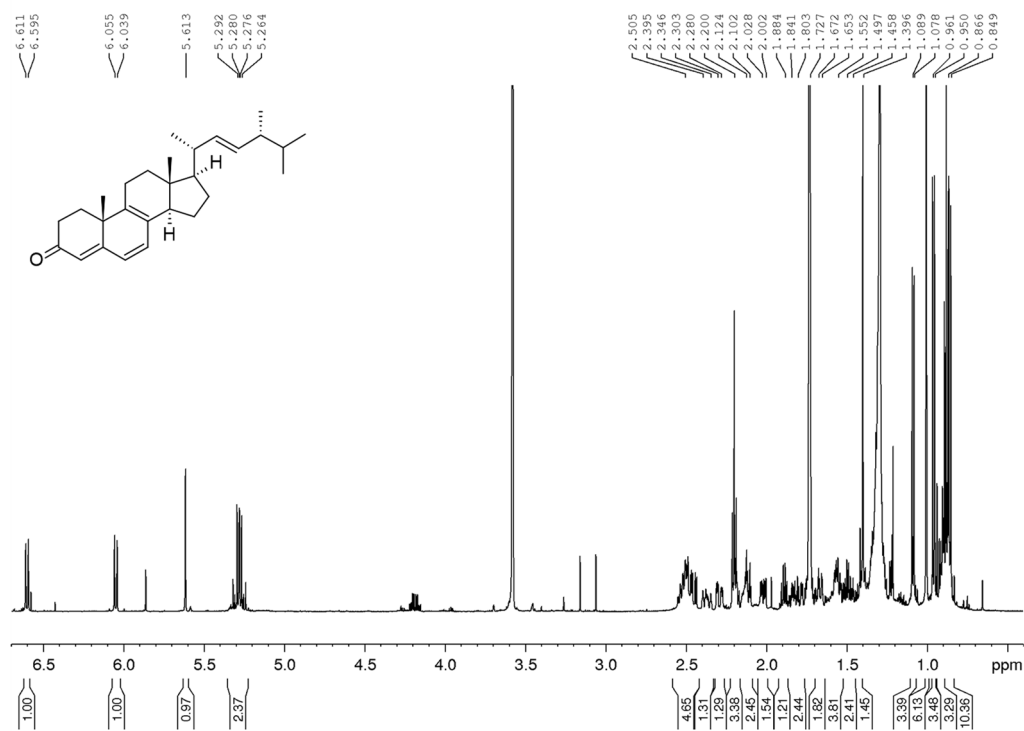


Figure S22. ¹H spectrum of compound 4 (600 MHz, THF-*d*₈, 295 K)

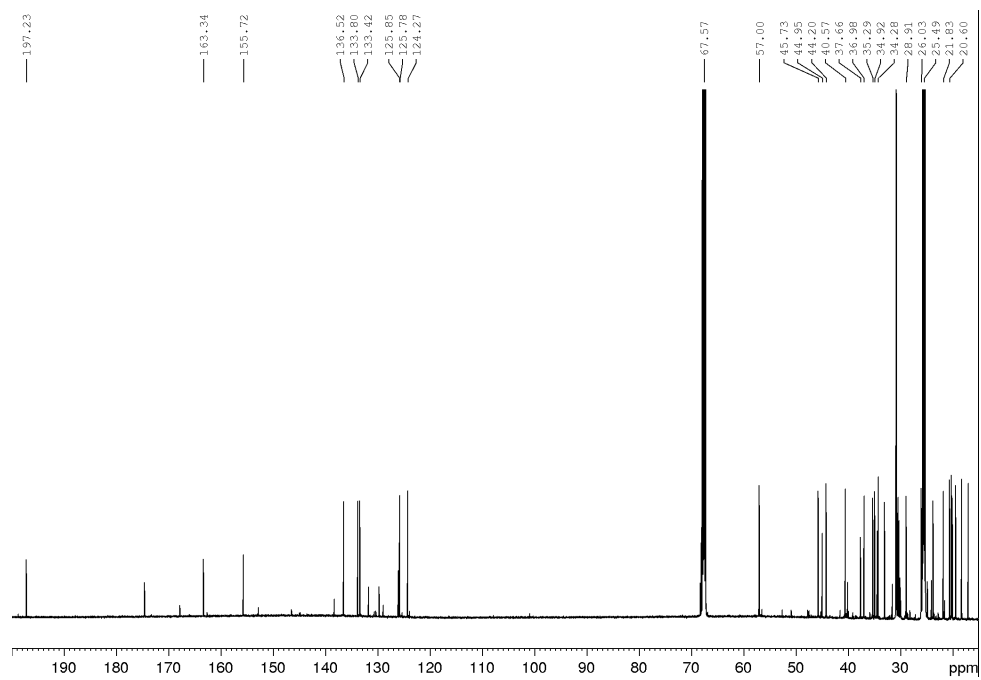


Figure S23. ¹³C spectrum of compound 4 (150 MHz, THF-*d*₈, 295 K)

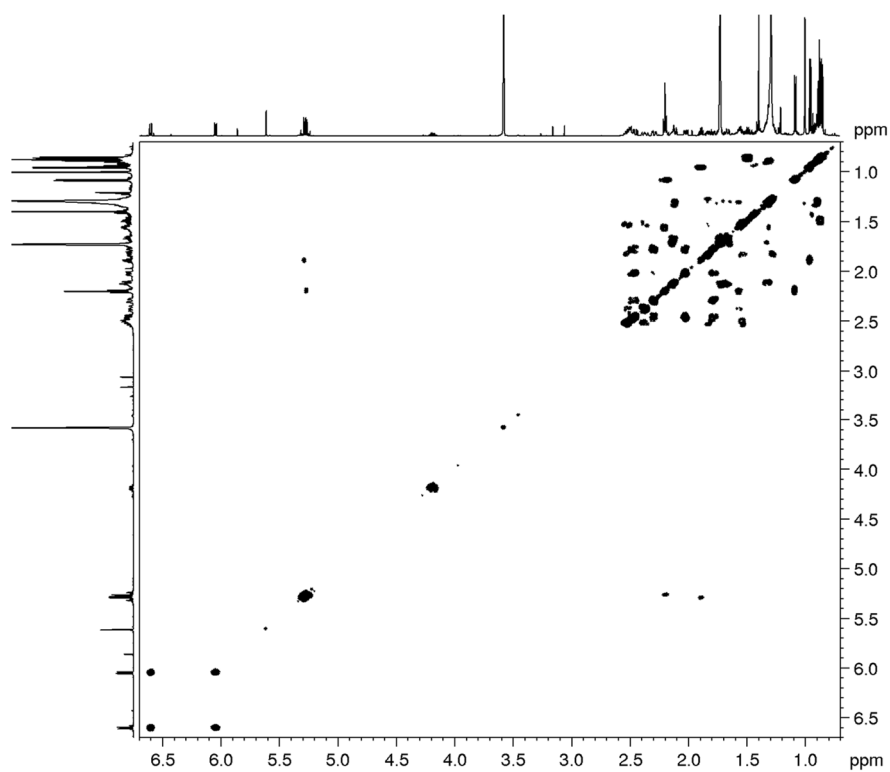


Figure S24. COSY spectrum of compound **4** (600 MHz, THF-*d*₈, 295 K)

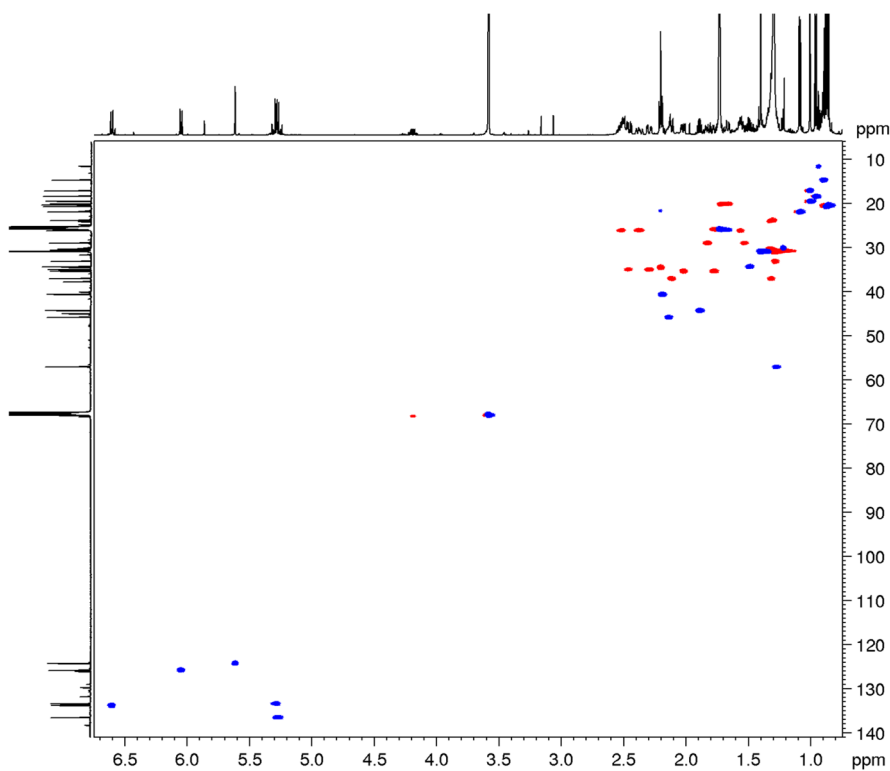


Figure S25. DEPT-edited HSQC spectrum of compound **4** (600 MHz, THF-*d*₈, 295 K)

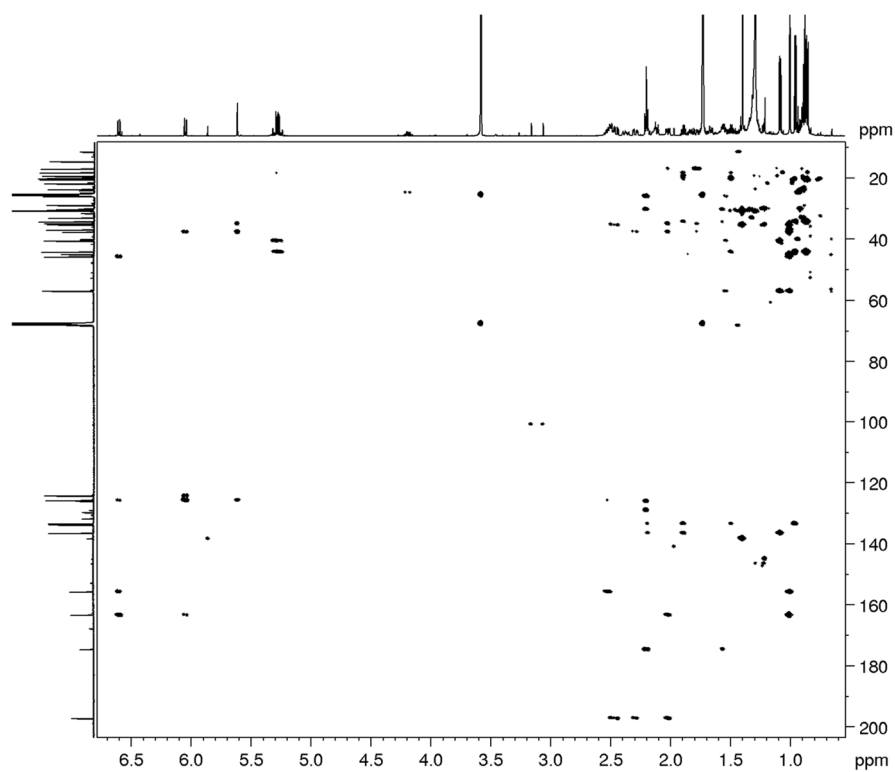


Figure S26. HMBC spectrum of compound **4** (600 MHz, $\text{THF-}d_8$, 295 K)

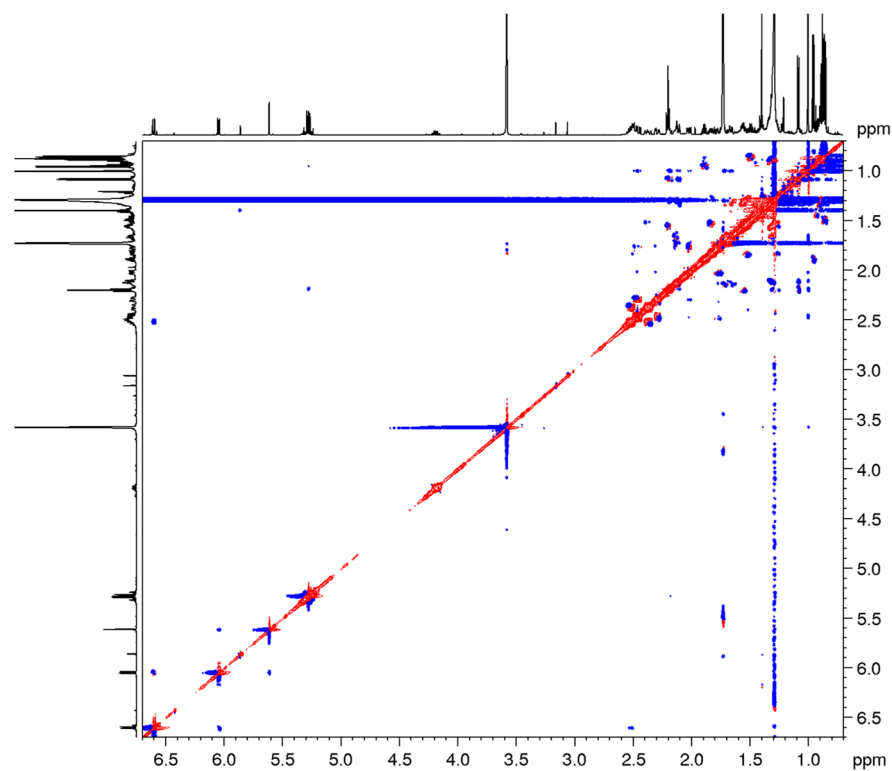
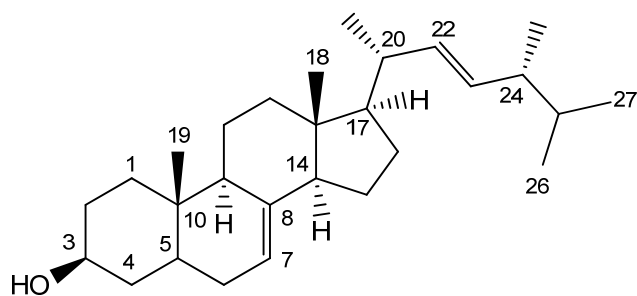


Figure S27. ROESY spectrum of compound **4** (600 MHz, $\text{THF-}d_8$, 295 K)

Spectra and spectral data on compound 5



Ergosta-7,22-diene-3-ol (5): amorphous solid; HRESIMS m/z no evaluable data.

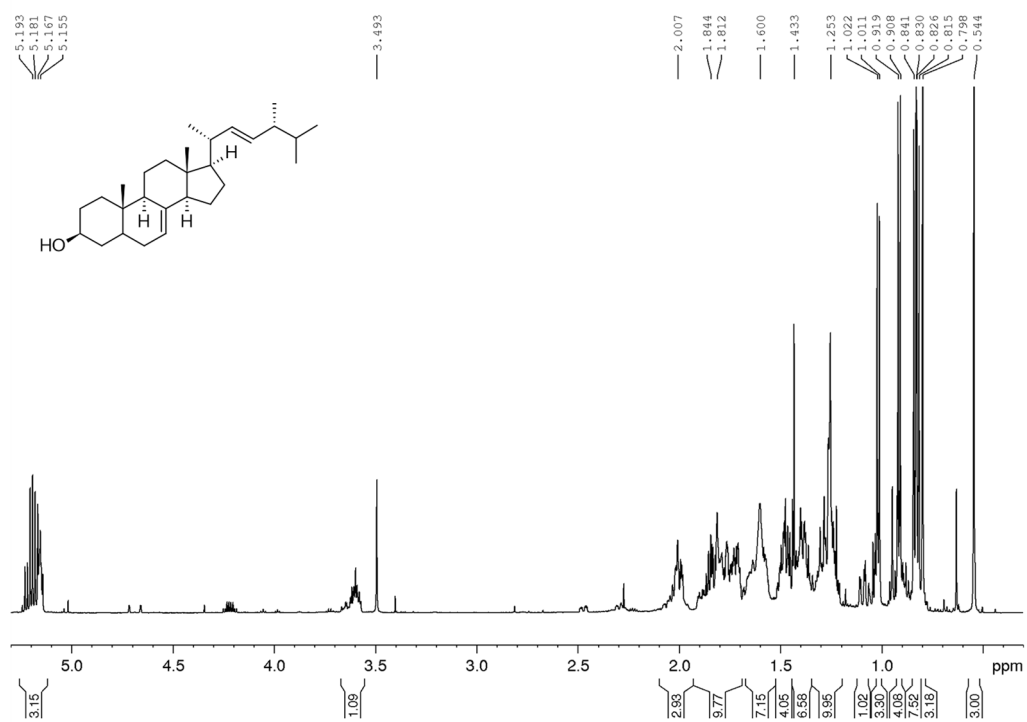


Figure S28. ^1H spectrum of compound 5 (600 MHz, CDCl_3 , 295 K)

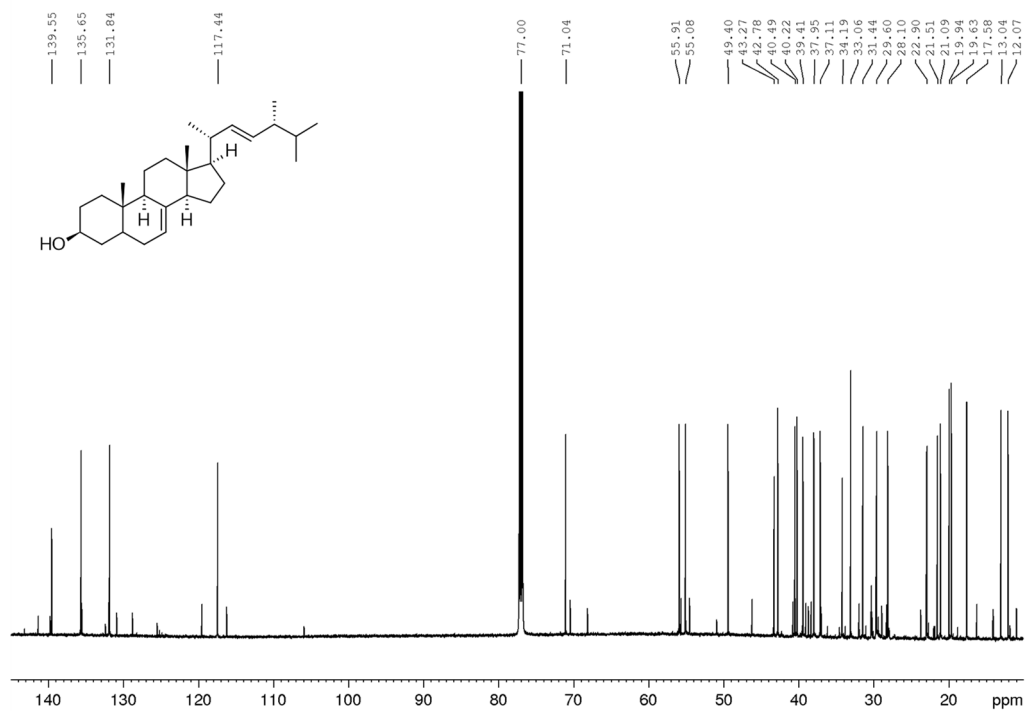


Figure S29. ¹³C spectrum of compound 5 (150 MHz, CDCl₃, 295 K)

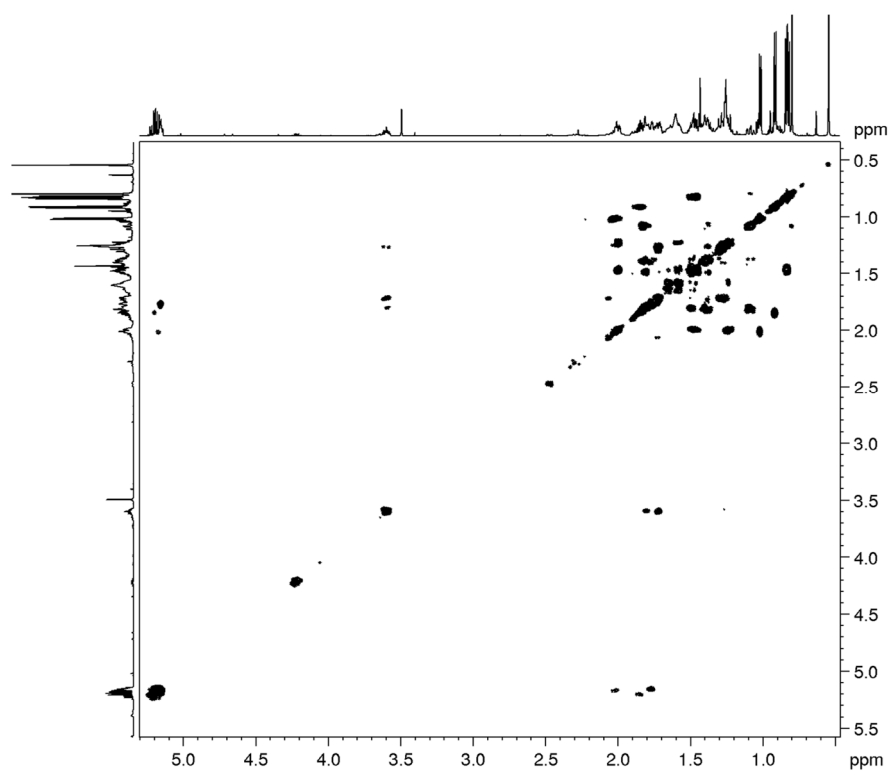


Figure S30. COSY spectrum of compound 5 (600 MHz, CDCl₃, 295 K)

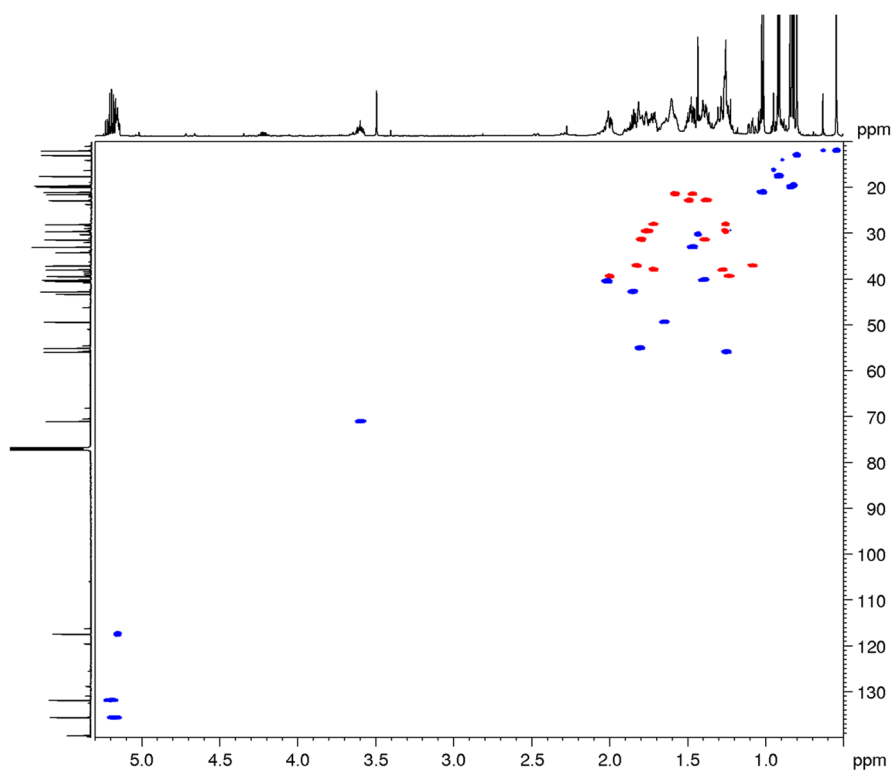


Figure S31. DEPT-edited HSQC spectrum of compound **5** (600 MHz, CDCl₃, 295 K)

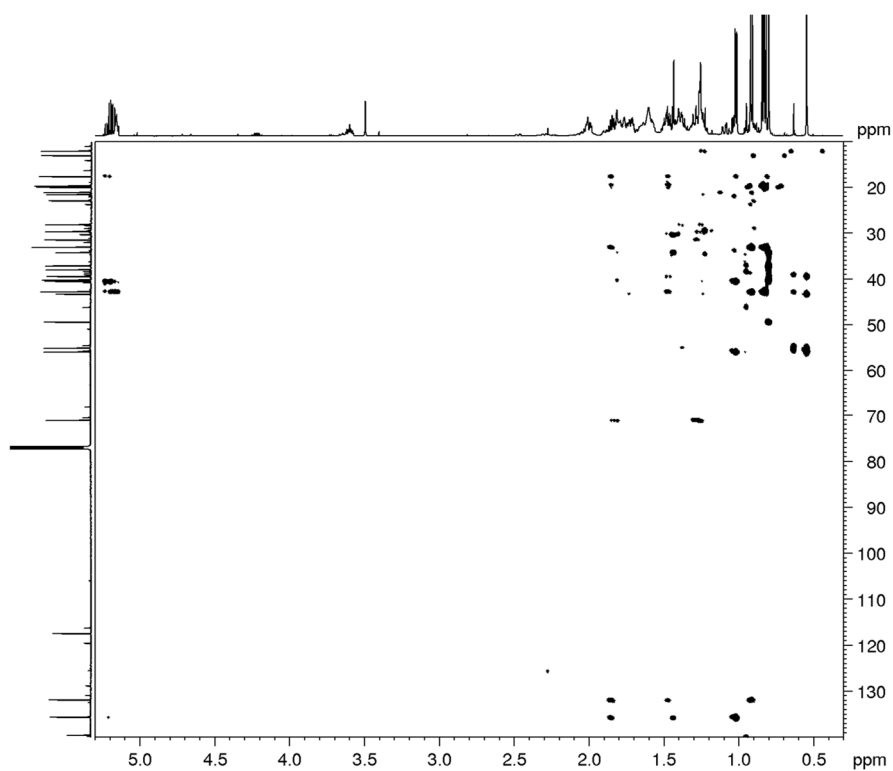


Figure S32. HMBC spectrum of compound **5** (600 MHz, CDCl₃, 295 K)

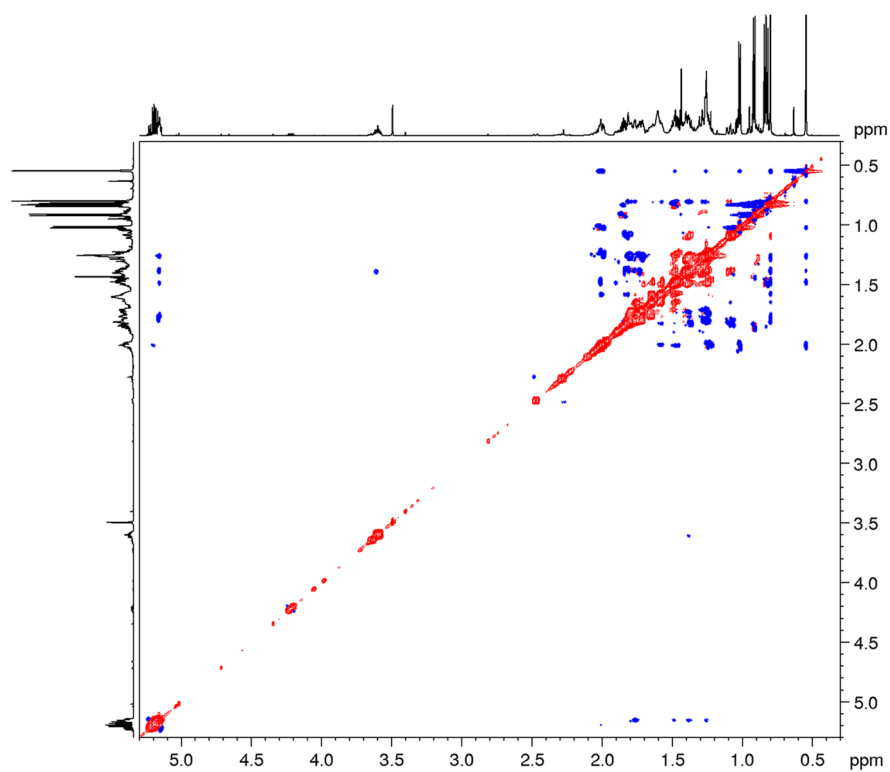
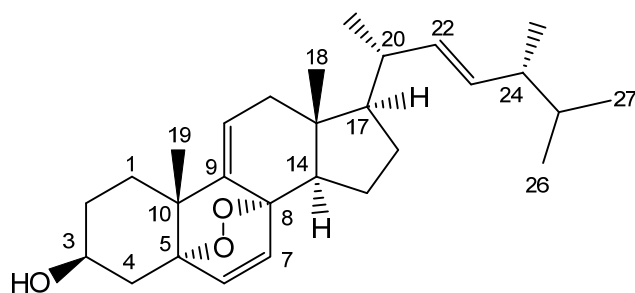


Figure S33. ROESY spectrum of compound **5** (600 MHz, CDCl₃, 295 K)

Spectra and spectral data on compound 6



9,11-Dehydroergosterol peroxide (6): amorphous solid; HRESIMS m/z 427.32028 $[M + H]^+$ (Δ 1.1 ppm; $C_{28}H_{43}O_3$); HRESI-MSMS (CID = 15%, 30%, 45%; rel int %) m/z 409(16), 391 (7), 375 (100).

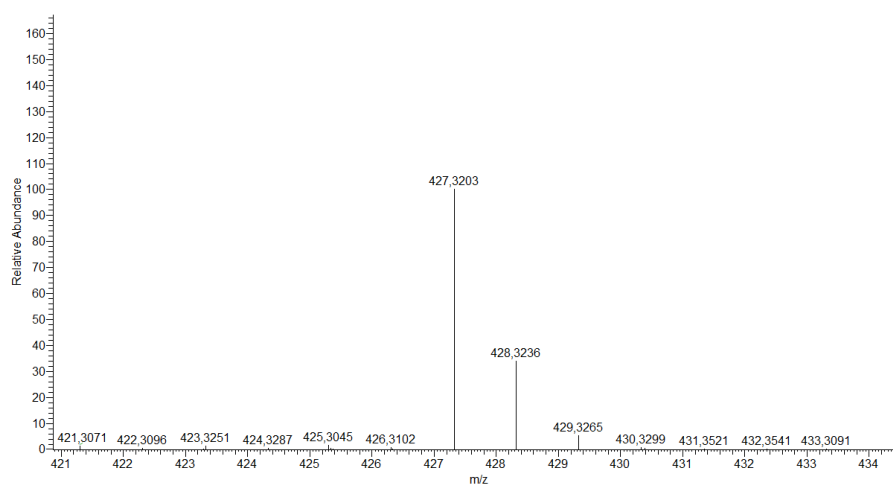


Figure S34. HRESI-MS spectrum of compound 6

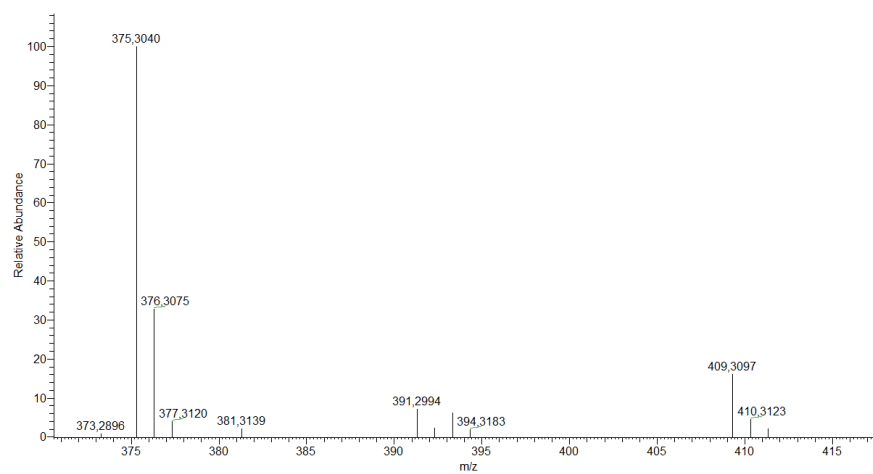


Figure S35. MS-MS spectrum of compound 6

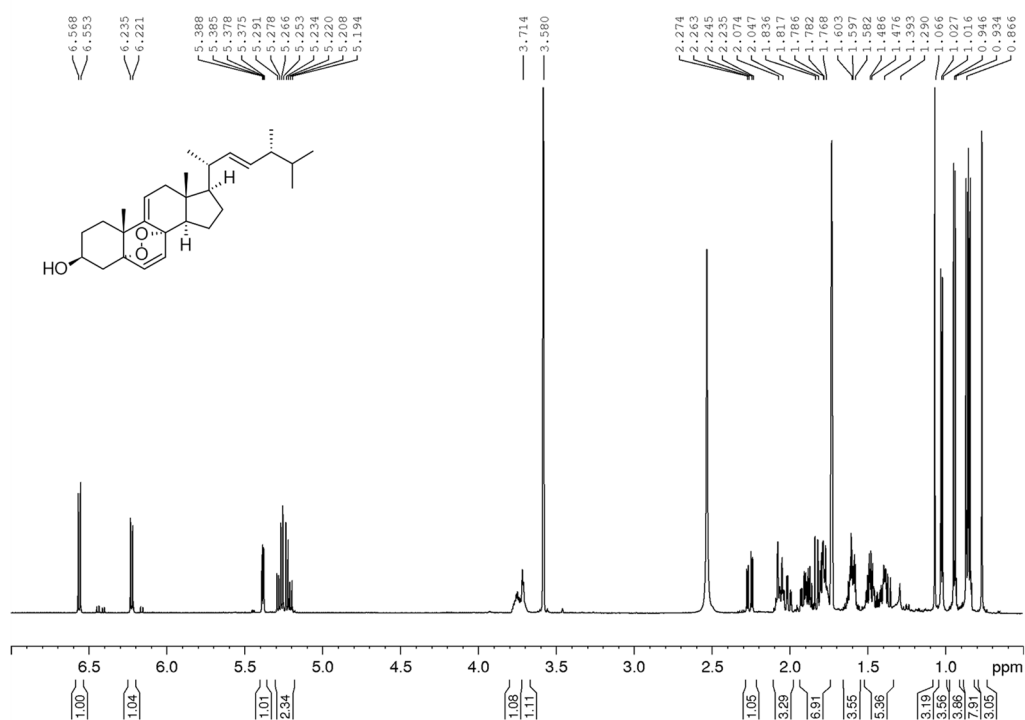


Figure S36. ¹H spectrum of compound 6 (600 MHz, THF-*d*₈, 295 K)

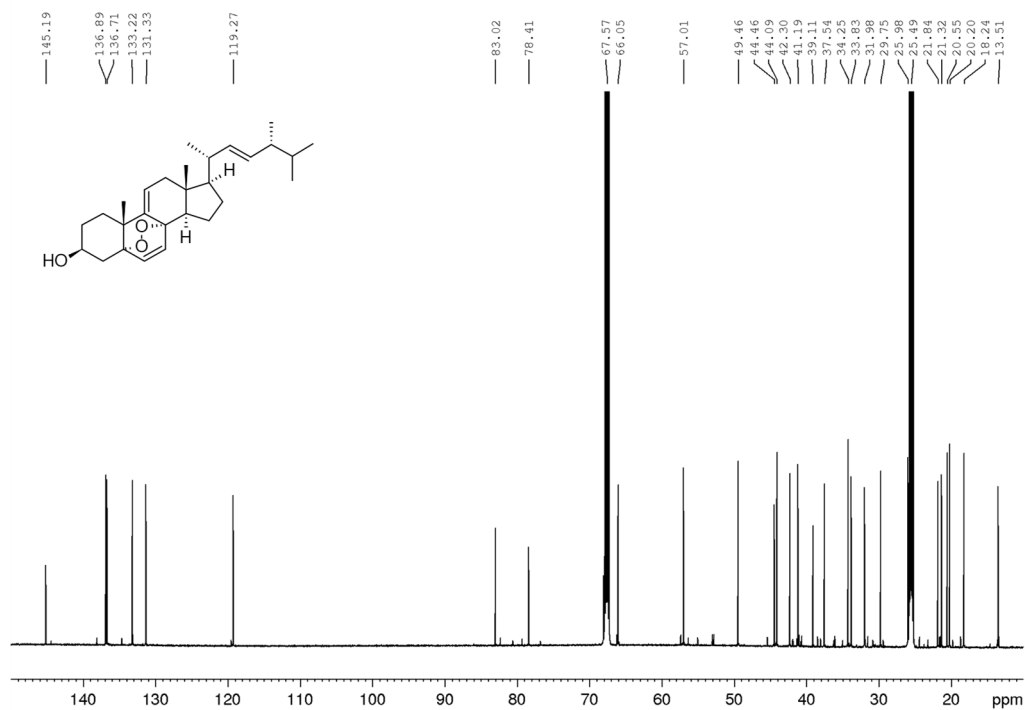


Figure S37. ¹³C spectrum of compound 6 (150 MHz, THF-*d*₈, 295 K)

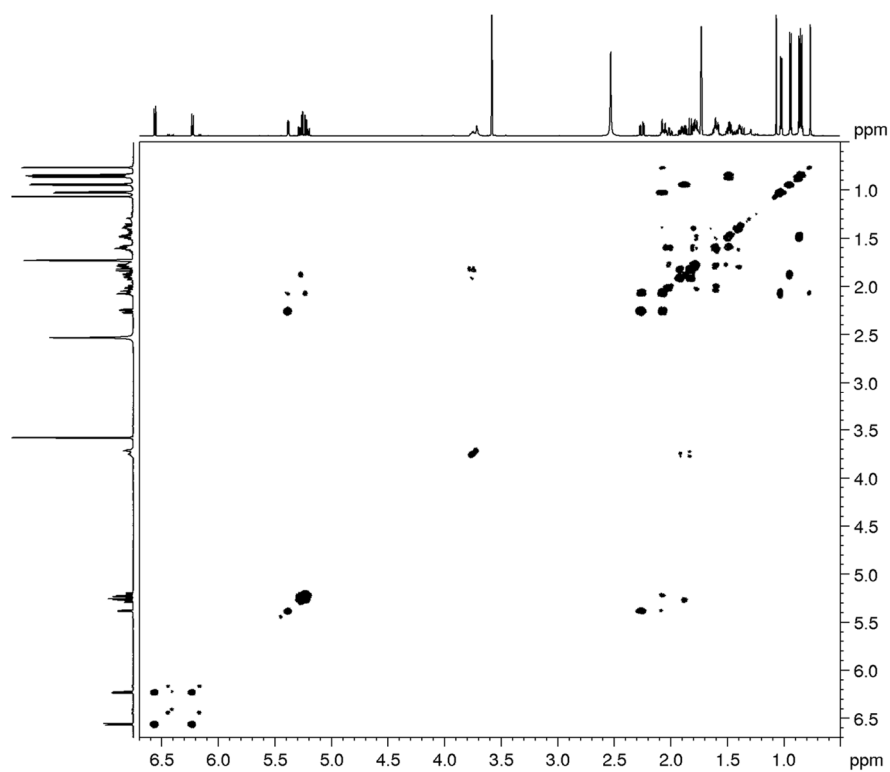


Figure S38. COSY spectrum of compound **6** (600 MHz, THF-*d*₈, 295 K)

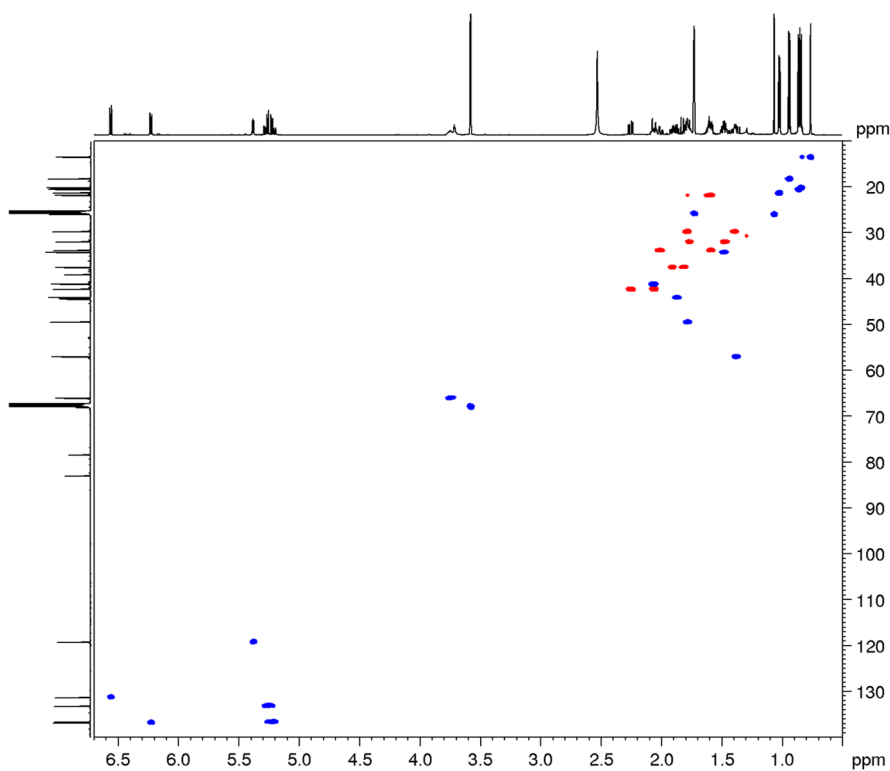


Figure S39. DEPT-edited HSQC spectrum of compound **6** (600 MHz, THF-*d*₈, 295 K)

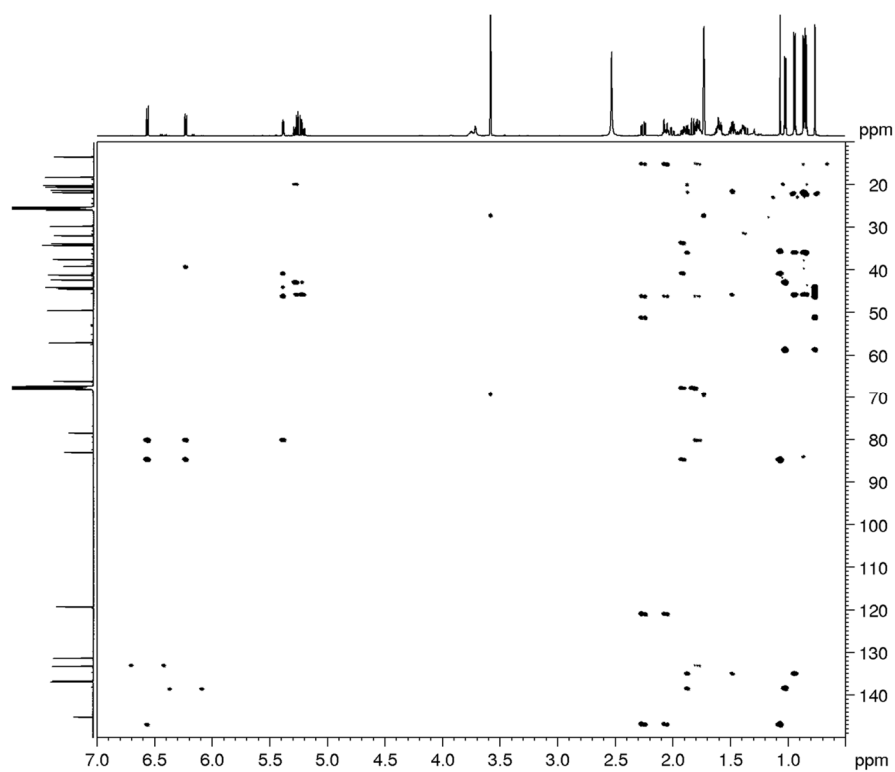


Figure S40. HMBC spectrum of compound **6** (600 MHz, $\text{THF-}d_8$, 295 K)

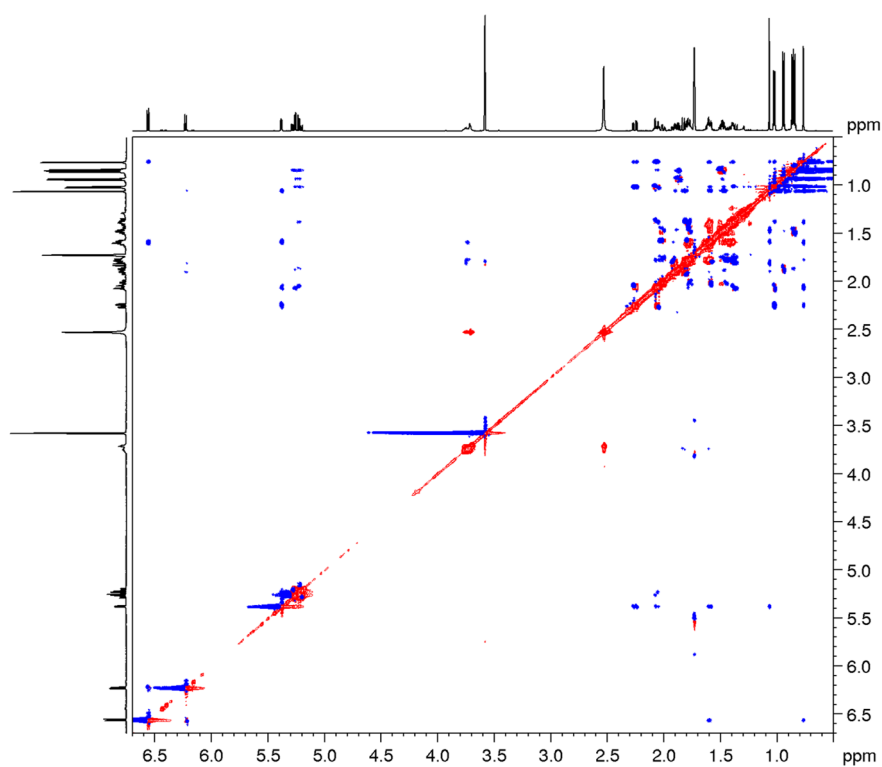
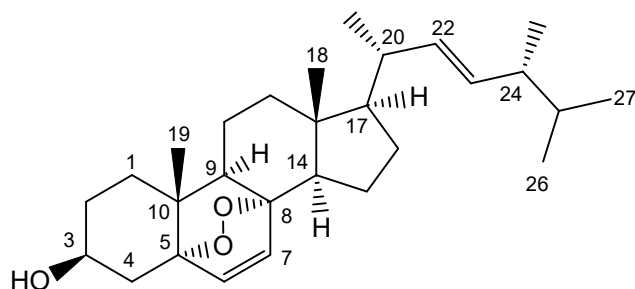


Figure S41. ROESY spectrum of compound **6** (600 MHz, $\text{THF-}d_8$, 295 K)

Spectra and spectral data on compound 7



Ergosta-5,7,22-triene-3-ol (7): amorphous solid; HRESIMS m/z 429.33597 $[M + H]^+$ (Δ 0.8 ppm; $C_{28}H_{45}O_3$); HRESI-MSMS (CID = 15%, 30%, 45%; rel int %) m/z 411 (82), 393 (28), 377 (100).

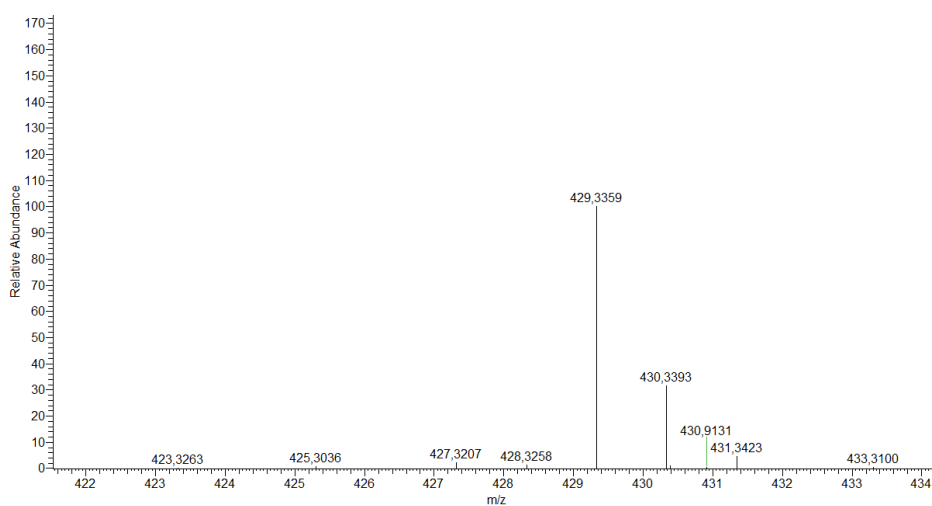


Figure S42. HRESI-MS spectrum of compound 7

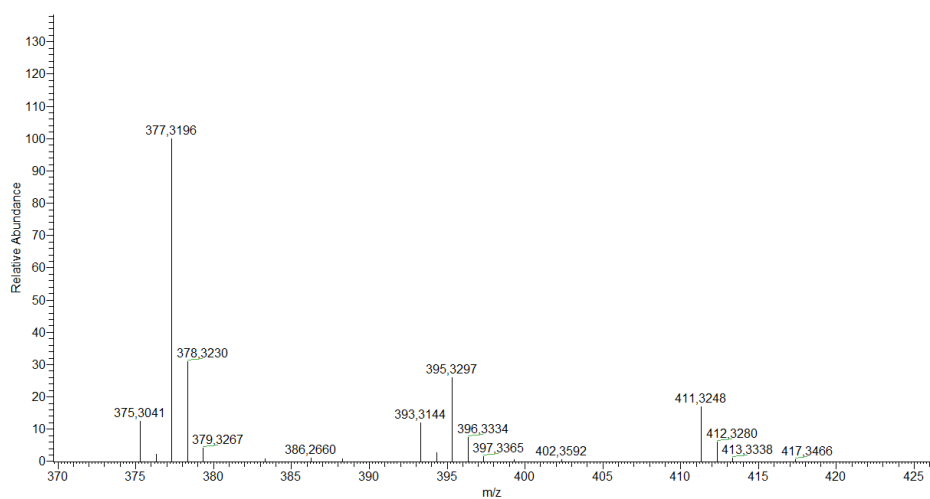


Figure S43. MS-MS spectrum of compound 7

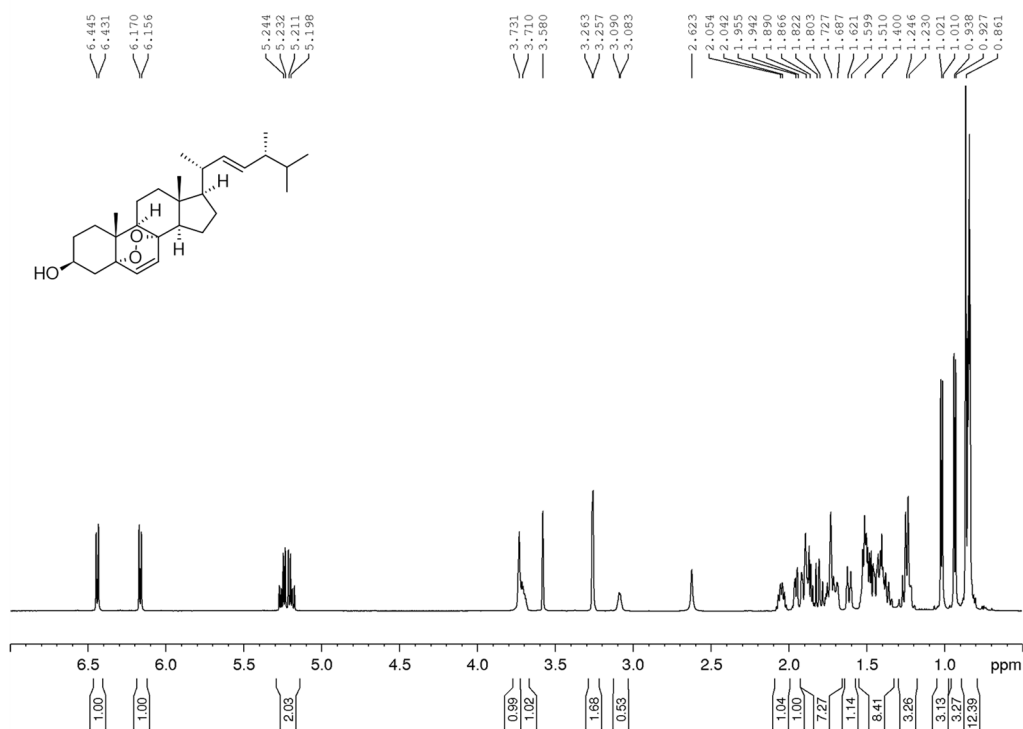


Figure S44. ¹H spectrum of compound 7 (600 MHz, THF-*d*₈, 295 K)

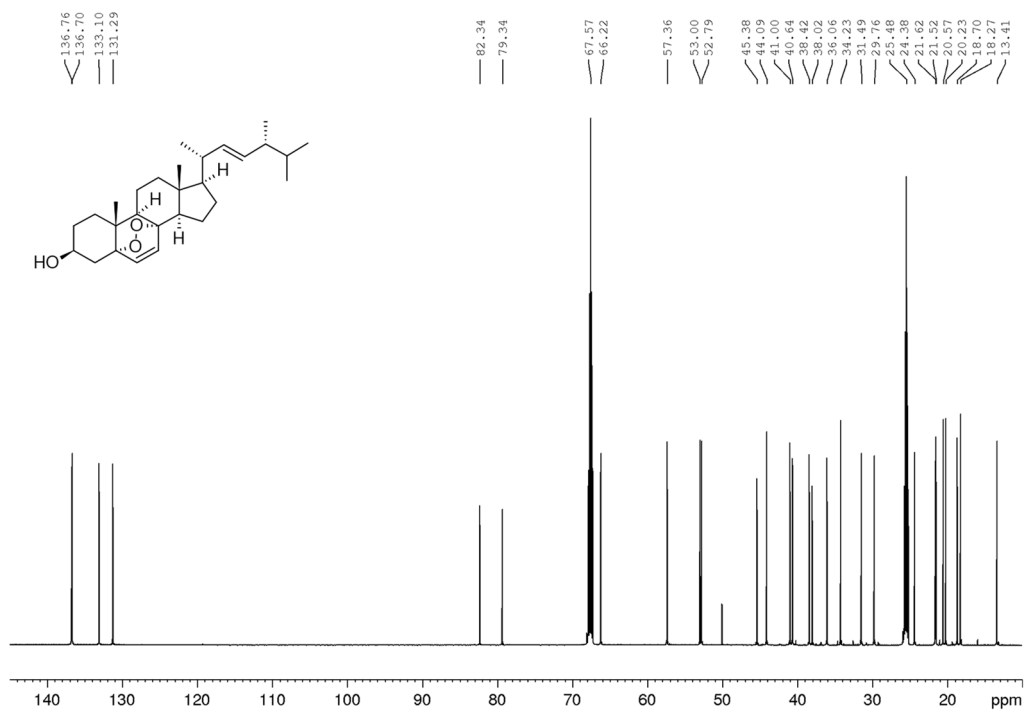


Figure S45. ¹³C spectrum of compound 7 (150 MHz, THF-*d*₈, 295 K)

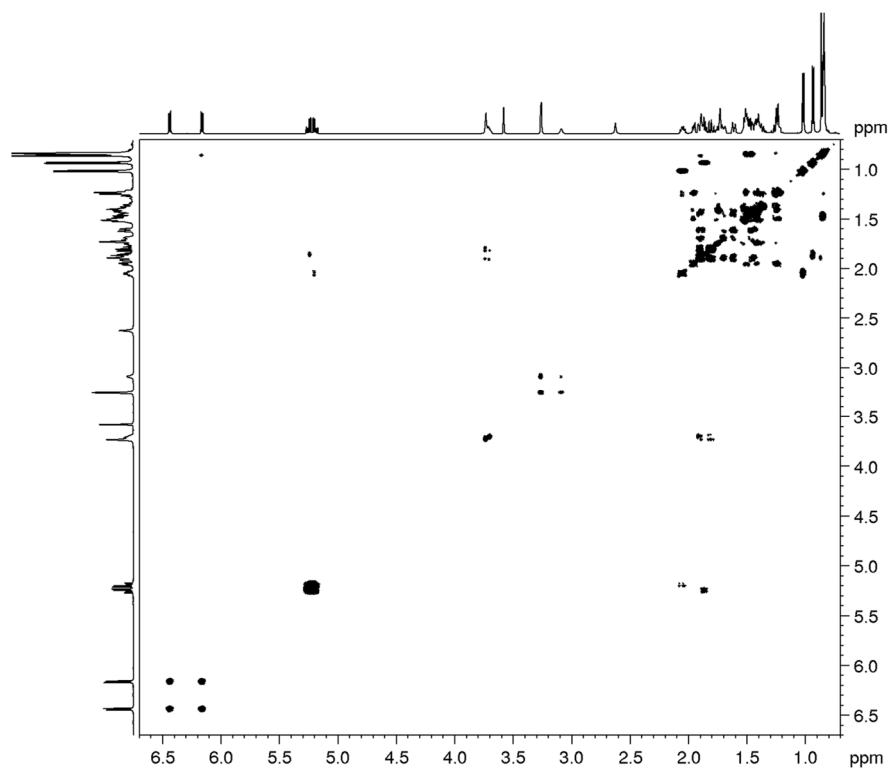


Figure S46. COSY spectrum of compound 7 (600 MHz, THF-*d*₈, 295 K)

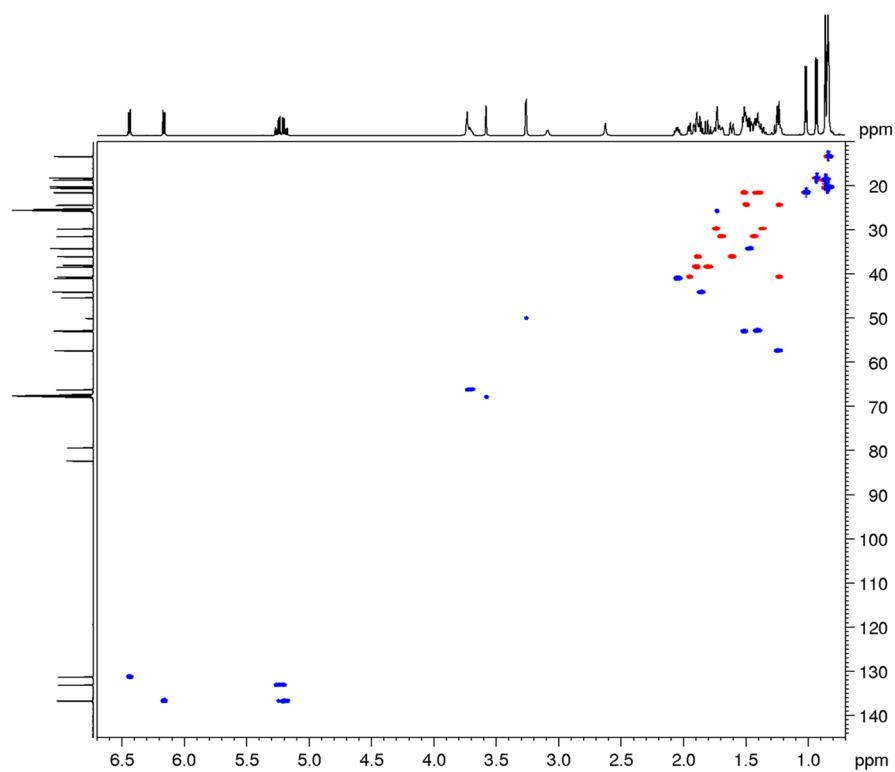


Figure S47. DEPT-edited HSQC spectrum of compound **7** (600 MHz, THF-*d*₈, 295 K)

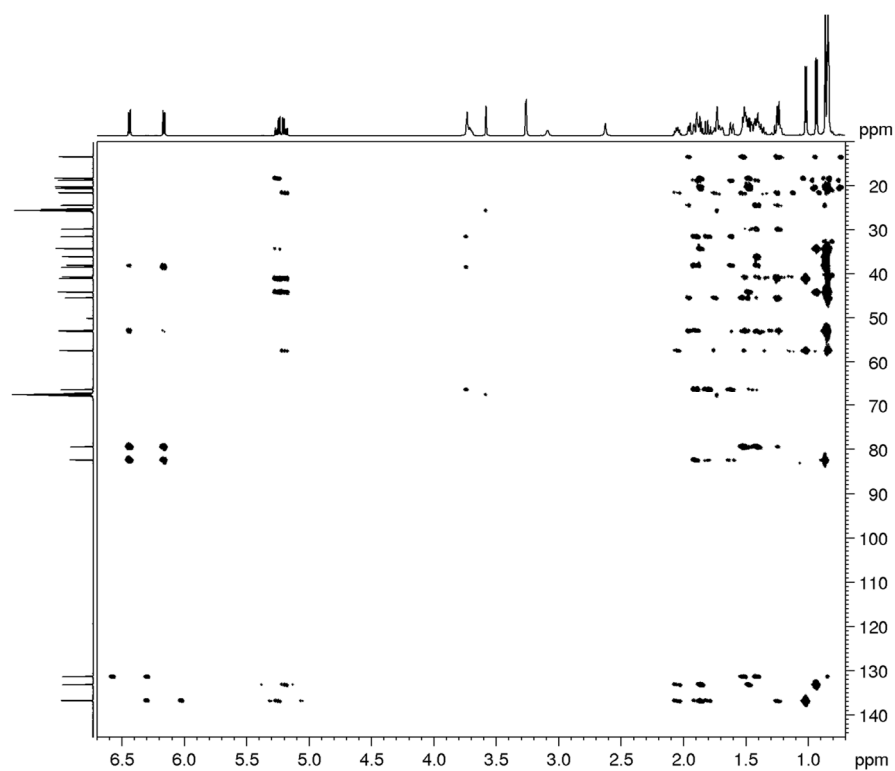


Figure S48. HMBC spectrum of compound **7** (600 MHz, THF-*d*₈, 295 K)

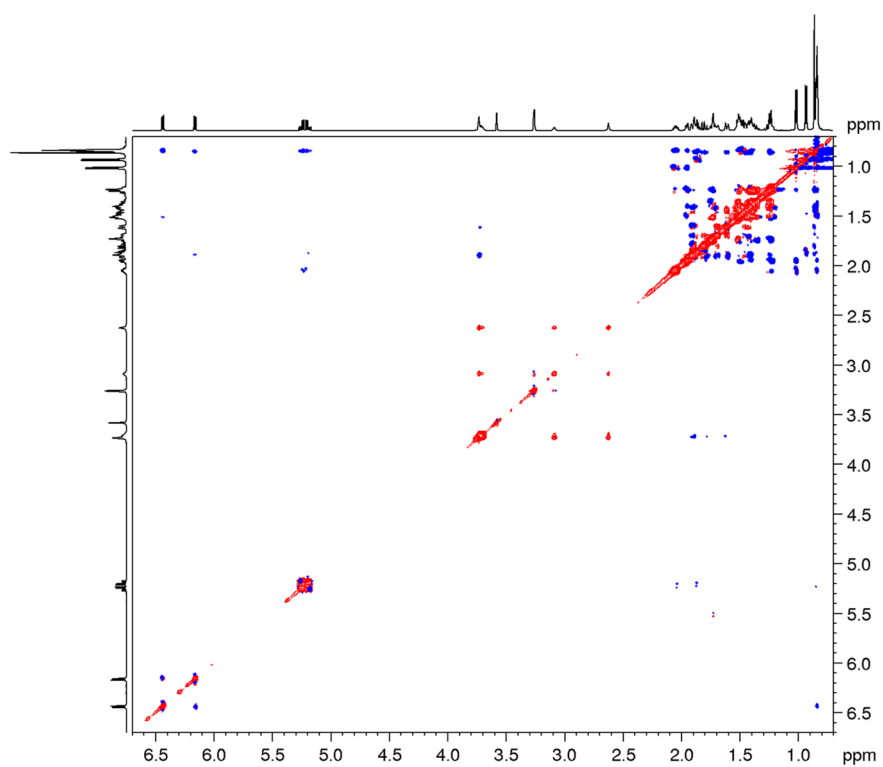


Figure S49. ROESY spectrum of compound 7 (600 MHz, THF-*d*₈, 295 K)

Table S1. Complete ^1H and ^{13}C NMR resonance assignments for compounds **1** and **2**.

No.	1			2		
	$\delta^{13}\text{C}$	$\delta^1\text{H}$	m, J	$\delta^{13}\text{C}$	$\delta^1\text{H}$	m, J
1	33.4	1.73 1.38	m m	29.7	1.53 1.26	m m
2	25.0	2.09 1.62	m m	23.6	1.96 1.76	m m
3	76.6	3.93	ddd, 10.1, 6.5, 2.0 Hz	72.5	3.73	b t, 4.6 Hz
4	123.1	5.49	b s	120.9	5.59	d, 4.6 Hz
5	145.5	-	-	147.4	-	-
6	125.8	5.88	d, 9.6 Hz	125.9	5.88	d, 9.7 Hz
7	126.0	6.17	d, 9.6 Hz	126.8	6.21	d, 9.7 Hz
8	124.8	-	-	124.9	-	-
9	45.5	1.97	m	44.9	2.05	m
10	35.9	-	-	36.0	-	-
11	19.1	1.60 1.52	m m	19.4	1.64 1.52	m m
12	36.3	2.02 1.26	m m	36.4	2.00 1.25	m m
13	43.5	-	-	43.6	-	-
14	149.7	-	-	149.7	-	-
15	25.0	2.40 2.29	m m	25.0	2.40 2.28	m m
16	27.9	1.77 1.44	m m	27.9	1.77 1.44	m m
17	55.9	1.21	m	55.8	1.22	m
18	19.2	0.92	s	19.1	0.92	s
19	18.2	0.89	s	17.1	0.82	s
20	39.4	2.11	m	39.4	2.11	m
21	21.2	1.04	d, 6.7 Hz	21.2	1.04	d, 6.8 Hz
22	135.3	5.21	m	135.4	5.22	m
23	132.2	5.22	m	132.1	5.22	m
24	42.8	1.86	m	42.8	1.87	m
25	33.1	1.48	m	33.1	1.47	m
26	19.7	0.83	d, 6.8 Hz	19.7	0.83	d, 6.8 Hz
27	20.0	0.84	d, 6.8 Hz	20.0	0.84	d, 6.8 Hz
28	17.6	0.92	d, 6.8 Hz	17.6	0.93	d, 6.8 Hz
3-OCH ₃	55.4	3.40	s	56.1	3.36	s

Table S2. Complete ¹H and ¹³C NMR resonance assignments for compounds **4**, **5**, **6** and **7**.

No.	4			6			7			5		
	$\delta^{13}\text{C}$	$\delta^1\text{H}$	<i>m, J</i>	$\delta^{13}\text{C}$	$\delta^1\text{H}$	<i>m, J</i>	$\delta^{13}\text{C}$	$\delta^1\text{H}$	<i>m, J</i>	$\delta^{13}\text{C}$	$\delta^1\text{H}$	<i>m, J</i>
1	35.3	2.02	m	37.1	1.82	m	33.8	2.01	m	36.1	1.88	m
		1.77	m		1.08	m		1.59	m		1.61	m
2	34.9	2.46	m	38.0	1.72	m	32.0	1.76	m	31.5	1.67	m
		2.29	m		1.28	m		1.47	m		1.43	m
3	197.2	-	-	71.0	3.60	m	66.1	3.75	m	66.2	3.71	m
4	124.3	5.61	s	31.4	1.80	m	37.5	1.90	m	38.4	1.89	m
					1.39	m		1.82	m		1.80	m
5	163.3	-	-	40.2	1.40	m	83.0	-	-	82.3	-	-
6	125.8	6.05	d, 9.5 Hz	29.6	1.76	m	136.9	6.23	d, 8.5 Hz	136.7	6.16	d, 8.5 Hz
7	133.8	6.60	d, 9.5 Hz	117.4	5.16	m	131.3	6.56	d, 8.5 Hz	131.3	6.44	d, 8.5 Hz
8	125.9	-	-	139.6	-	-	78.4	-	-	79.3	-	-
9	155.7	-	-	49.4	1.65	m	145.2	-	-	52.8	1.40	m
10	37.7	-	-	34.2	-	-	39.1	-	-	38.0	-	-
11	26.0	2.51	m	21.5	1.58	m	119.3	5.38	dd, 6.0. 1.9 Hz	24.4	1.49	m
		2.37	m		1.47	m					1.23	m
12	37.0	2.11	m	39.4	2.00	m	42.3	2.25	dd, 16.8. 6.0 Hz	40.6	1.94	m
		1.31	m		1.23	m		2.06			1.23	m
13	45.0	-	-	43.3	-	-	44.5	-	-	45.4	-	-
14	45.7	2.13	m	55.1	1.81	m	49.5	1.78	m	53.0	1.51	m
			m	22.9	1.49	m						
15	20.1	1.71	m	28.1	1.38	m	21.8	1.60	m	21.6	1.51	m
		1.66	m		1.72	m					1.40	m
16	28.9	1.82	m	55.9	1.25	m	29.8	1.79	m	29.8	1.73	m
		1.52	m		1.25	m		1.39	m		1.37	m
17	57.0	1.27	m	12.1	0.54	s	57.0	1.38	m	57.4	1.24	m

18	19.4	1.00	s	13.0	0.80	s	13.5	0.76	s	13.4	0.83	m
19	17.1	1.00	s	40.5	2.01	m	26.0	1.07	s	18.7	0.86	m
20	40.6	2.19	m	21.1	1.02	d, 6.8 Hz	41.2	2.07	m	41.0	2.04	m
21	21.8	1.08	d, 6.7 Hz	135.7	5.17	m	21.3	1.02	d, 6.7 Hz	21.5	1.02	d, 6.5 Hz
22	136.5	5.27	m	131.8	5.20	m	136.7	5.23	m	136.8	5.20	m
23	133.4	5.28	m	42.8	1.85	m	133.2	5.26	m	133.1	5.23	m
24	44.2	1.89	m	33.1	1.47	m	44.1	1.87	m	44.1	1.86	m
25	34.3	1.48	m	19.6	0.82	d, 6.8 Hz	34.3	1.48	m	34.2	1.47	m
26	20.2	0.85	d, 6.8 Hz	19.9	0.84	d, 6.8 Hz	20.2	0.84	d, 6.8 Hz	20.2	0.83	m
27	20.6	0.87	d, 6.8 Hz	17.6	0.91	d, 6.8 Hz	20.6	0.86	d, 6.8 Hz	20.6	0.85	m
28	18.3	0.96	d, 6.8 Hz				18.2	0.94	d, 6.8 Hz	18.3	0.93	m

Table S3. Complete ^1H and ^{13}C NMR resonance assignments for compound **3**

No.	3		
	$\delta^{13}\text{C}$	$\delta^1\text{H}$	$\mathbf{m, J}$
2	87.2	5.30	dd, 9.7; 7.9 Hz, 1H
3	33.7	3.41	dd, 15.9; 9.7 Hz, 1H
		3.09	dd, 15.9; 7.9 Hz, 1H
3a	128.2	-	-
4	125.8	7.73	M
5	130.4	-	-
6	133.2	7.68	dd, 8.2; 1.7 Hz
7	109.5	6.90	d, 8.2 Hz
7a	165.2	-	-
1'	143.1	-	-
2'	112.8	5.10	m
		4.95	m
3'	17.0	1.77	s
1''	190.7	9.83	s