Supporting Information

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A New Azaphilone Derivative from the Co-culture of *Aspergillus versicolor* and *Aspergillus chevalieri*

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S1: Characterization of compounds 2–8

*Lunatinin (**2**):* UV (MeOH) λ_{max} 245, 330 nm. ^{1}H NMR (DMSO-d_6, 600 MHz) δ 2.49 (overlap, H-1′), 3.97 (m, H-2′), 1.13 (d, J = 6.6 Hz, H-3′), 6.43 (s, H-4), 6.42 (s, H-5), 2.00 (s, H-9); ^{13}C NMR (DMSO-d_6, 150 MHz) δ 166.2 (C-1), 154.2 (C-3), 105.3 (C-4), 136.3 (C-4a), 101.8 (C-5), 164.5 (C-6), 109.7, (C-7), 159.8 (C-8), 97.2 (C-8a), 8.0 (C-9), 42.5 (C-1'), 63.9 (C-2'), 23.3 (C-3'). MS *m/z* 251 $[M+H]^+$.

*(2S,3S)-5,6-dihydroxy-2,6-dimethyl-3-(2-oxopentyl)-2-cyclohexen-1-one (**3**)*: [α] +6.3 (c 0.95 MeOH); UV (MeOH) λ_{max} 249 nm. ^{1}H NMR (DMSO-d_6, 600 MHz) δ 0.92 (t, J = 7.2 Hz, H-11), 1.30 (s, H-12), 1.60 (t, J = 7.2 Hz, H-10), 1.74 (s, H-13), 2.46 (br d, J = 18.0 Hz, H-4b), 2.52 (t, J = 7.2 Hz, H-9), 2.79 (br d, J = 18.0Hz, H-4a), 3.39 (d, J = 16.8 Hz, H-7b), 3.60 (d, J = 16.8Hz, H-7a), 3.94 (t, J = 3.6 Hz, H-3); ^{13}C NMR (DMSO-d_6, 150 MHz) δ 202.7 (C-1), 77.2 (C-2), 74.8 (C-3), 38.6 (C-4), 147.7 (C-5), 131.3 (C-6), 49.0 (C-7, overlapped), 208.5 (C-8), 45.6 (C-9), 18.1 (C-10), 13.9 (C-11), 22.9 (C-12), 11.6 (C-13); MS *m/z* 241 $[M+H]^+$.

*Sterigmatocystin (**4**):* UV (MeOH) λ_{max} 244, 325 nm. ^{1}H NMR (DMSO-d_6, 600 MHz) δ 6.75 (m, H-4), 7.62 (t, J = 8.4 Hz, H-5), 6.98 (d, J = 8.4 Hz, H-6), 6.71 (s, H-11), 6.97 (d, J = 7.2 Hz, H-14), 4.86 (dd, J = 7.2, 2.4 Hz, H-15), 5.53, (t, J = 2.4 Hz, H-16), 6.75, (m, H-17), 3.89 (s, H-18), 13.29 (s, OH-3); ^{13}C NMR (DMSO-d_6, 150 MHz) δ 180.4 (C-1), 108.2 (C-2), 161.3 (C-3), 110.7 (C-4), 136.1 (C-5), 106.4 (C-6), 154.4 (C-7), 153.3 (C-8), 106.5 (C-9), 164.3 (C-10), 91.0 (C-11), 162.8 (C-12), 104.9 (C-13), 113.3 (C-14), 47.3 (C-15), 102.6 (C-16), 145.6 (C-17), 56.8 (C-18). MS *m/z* 325 $[M+H]^+$.

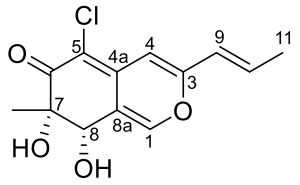
*Glyantrypine (**5**) :* UV (MeOH) λ_{max} 223 nm. ^{1}H NMR (DMSO-d_6, 600 MHz) δ 7.56 (d, J = 7.8 Hz, H-3), 7.83 (t, J = 7.8 Hz, H-4), 7.55 (t, J = 7.8 Hz, H-5), 8.21 (d, J = 7.2 Hz H-6), 3.81 (dd, J = 16.8, 4.8 Hz, H-11a), 3.08 (d, J = 16.8 Hz, H-11b), 8.33 (d, J = 4.8 Hz, H-12), 5.27 (t, J = 4.8 Hz, H-14), 3.43 (m, H_2-15), 6.87 (d, J = 2.4 Hz, H-17), 10.97 (brs, H-18), 7.32 (d, J = 7.8 Hz, H-21), 7.00 (t, J = 7.8 Hz, H-22), 6.78 (t, J = 7.8 Hz, H-23), 7.26 (d, J = 7.8 Hz, H-24); ^{13}C NMR (DMSO-d_6, 150 MHz) δ 147.0 (C-2), 126.3 (C-3), 136.0 (C-4), 126.6 (C-5), 126.7 (C-6), 119.9 (C-7), 167.6 (C-8), 149.3 (C-10), 43.8 (C-11), 159.9 (C-13), 56.5 (C-14), 26.5 (C-15), 124.4 (C-16), 107.8 (C-17), 135.6 (C-19), 127.2 (C-20),118.6 (C-21), 121.2 (C-22), 117.7 (C-23), 111.4 (C-24). MS *m/z* 345 $[M+H]^+$.

*Cottoquinazoline A (**6**):* UV (MeOH) λ_{max} 205, 228 nm. ^{1}H-NMR (DMSO-d_6, 600 MHz) δ 9.07 (d, J = 5.4 Hz, H-2), 5.24 (d, J = 4.8 Hz, H-3), 7.74 (d, J = 7.8 Hz, H-7), 7.85 (t J = 7.8 Hz, H-8), 7.56 (t, J = 7.8 Hz, H-9), 8.13 (d, J = 8.4 Hz, H-10), 5.26 (m, H-14), 2.40 (dd, J =15.0, 2.4 Hz, H-15a) 3.07 (dd, J = 15.0, 5.4 Hz, H-15b), 4.88 (d, J = 1.8 Hz, H-18), 4.08 (brq, J = 6.0 Hz,, H-20), 7.29 (m, H-24), 7.29 (m, H-25), 7.10 (m, H-26),7.42 (d, J = 7.8 Hz, H-27), 1.47 (d, J = 6.0 Hz, H_3-29), 5.56 (brs, OH-17); ^{13}C NMR (DMSO-d_6, 150 MHz) δ 167.8 (C-1), 65.5 (C-3), 147.5 (C-4), 146.7 (C-6), 127.1 (C-7), 134.5 (C-8), 127.2 (C-9), 126.2 (C-10), 120.7 (C-11), 159.4 (C-12), 53.9 (C-14), 36.0 (C-15), 74.1 (C-17), 79.8 (C-18), 63.4 (C-20), 165.6 (C-21), 136.0 (C-23), 113.8 (C-24), 129.3 (C-25), 124.4 (C-26), 124.4 (C-27), 139.8 (C-28), 14.8 (C-29). MS *m/z* 412 $[M+H]^+$.

*Preechinulin (**7**):* UV (MeOH) λ_{max} 224, 280nm. ^{1}H-NMR (DMSO-d_6, 600 MHz) δ 10. 55 (s, H-1), 7.33 (d, J = 7.8 Hz, H-4), 6.94 (t, J = 7.8 Hz, H-5), 7.03 (t, J = 7.8 Hz, H-6), 7.43 (d, J = 7.8 Hz, H-7), 3.35 (overlapped, H-8a), 3.06 (dd, J = 8.0 Hz, H-8b), 3.96 (m, H-9), 8.17 (d, J = 3.0 Hz, H-11), 3.79 (m, H-12), 7.50 (d, J = 3.0 Hz, H-14), 6.18 (dd, J = 17.4, 10.8 Hz, H-16), 5.08 (d, J = 17.4 Hz, H-17a), 5.04 (d, J = 17.4 Hz, H-17b), 1.49 (s, H-18), 1.48 (s, H-19), 1.23 (d, J = 7.1 Hz, H-20); ^{13}C NMR (DMSO-d_6, 150 MHz) δ 141.4 (C-2), 104.6 (C-3), 128.9 (C-3a), 117.9 (C-6), 118.3 (C-5), 120.5 (C-4), 110.8 (C-7), 134.8 (C-7a), 31.0 (C-8), 55.7 (C-9), 167.3 (C-10), 50.3 (C-12), 167.9 (C-13), 39.2 (overlapped, C-15), 146.5 (C-16), 111.0 (C-17), 28.0 (C-18), 28.0 (C-19), 20.7 (C-20). MS *m/z* 326 $[M+H]^+$.

*(–)-Neoechinulin (**8**):* UV (MeOH) λ_{max} 223, 335 nm. ^{1}H-NMR (DMSO-d_6, 600 MHz) δ 7.18 (d, J = 7.8 Hz, H-4), 7.01 (dd, J = 7.8, 7.8 Hz, H-6), 7.08 (dd, J = 7.8, 7.8 Hz, H-5), 7.41 (d, J = 7.8 Hz, H-7), 6.88 (s, H-14), 8.32 (s, H-8), 4.15 (q, J = 7.2 Hz, H-12), 6.09 (dd, J = 18.0, 10.2 Hz, H-16), 5.05 (d, J = 10.2 Hz, H-17a), 5.02 (d, J = 18.0 Hz, H-17b), 1.46 (s, H-18), 1.46 (s, H-19), 1.37 (d, J = 7.2 Hz, H-20); ^{13}C NMR (DMSO-d_6, 150 MHz) δ 143.9 (C-2), 103.4 (C-3), 125.9 (C-3a), 118.9 (C-4), 119.3 (C-5), 120.7 (C-6), 111.6 (C-7), 135.1(C-7a), 111.6 (C-8), 124.9 (C-9), 159.9 (C-10), 50.5 (C-12), 166.4 (C-13), 38.9 (overlapped, C-15), 145.1 (C-16), 110.1 (C-17), 27.5 (C-18), 27.5 (C-19), 19.6 (C-20). MS *m/z* 324 $[M+H]^+$.

Table S1: The ^{1}H and ^{13}C NMR data for compound **1** in DMSO-d_6.



1

	Compound **1**	
No.	δ_H, mult. (*J* in Hz)	δ_C, mult.
1	7.52, s	144.3, CH
3		156.8, C
4	6.51, s	103.4, CH
4a		140.0, C
5		108.3, C
6		190.9, C
7		76.1, C
7-CH_3	1.09, s	19.2, CH_3
8	4.32, s	70.8, CH
8a		120.0, C
9	6.39, d (16.2)	123.5, CH
10	6.57, dq (16.2, 7.2)	134.8, CH
11	1.89, d (7.2)	18.3, CH_3

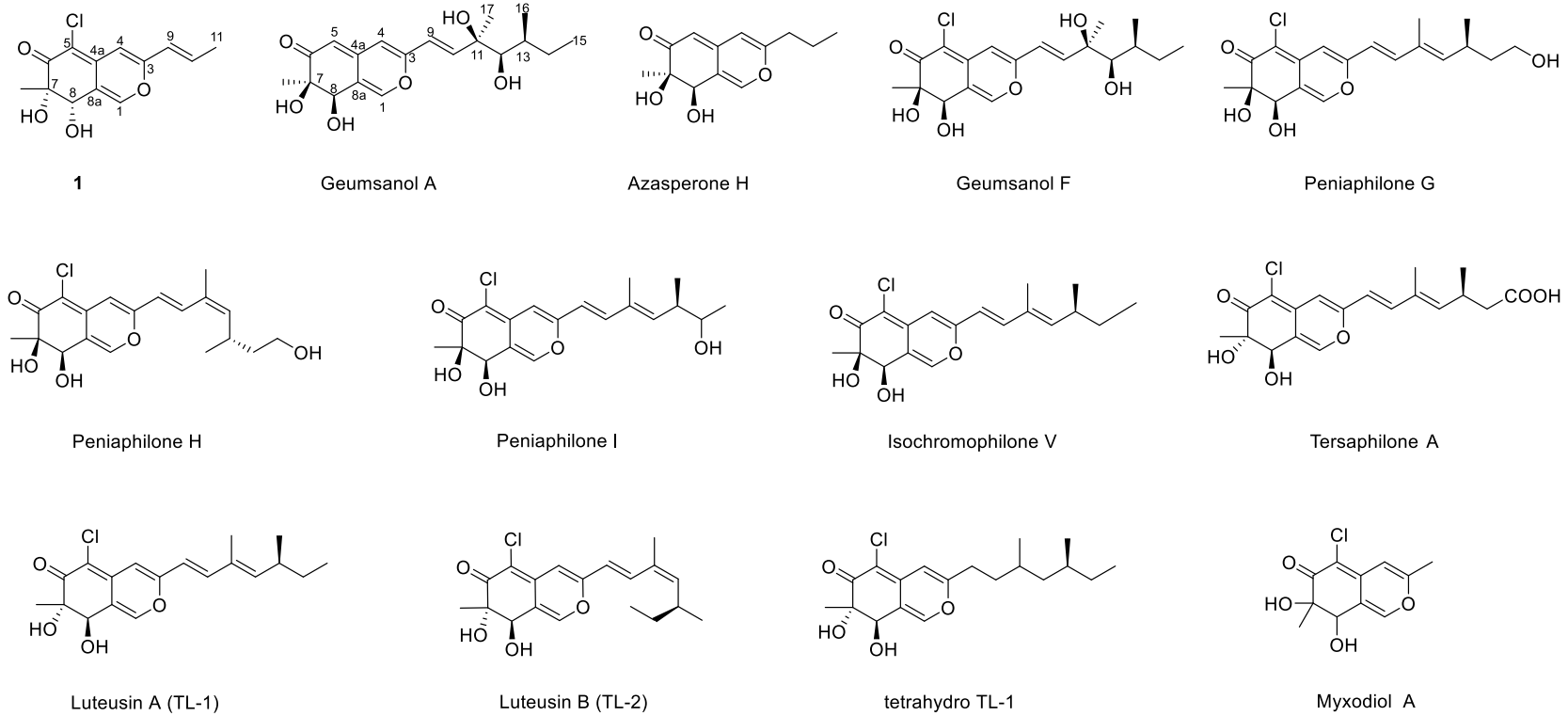


Figure S1: Natural products containing the 7,8-dihydro-6H-isochromen-6-one skeleton and the related compounds.

Table S2: ^{1}H NMR Data for natural products containing the 7,8-dihydro-6H-isochromen-6-one skeleton and the related compounds.

no.	7,8-*cis*								
	1	GeumsanolA[1]	Azasperone H[2]	Geumsanol F[3]	Geumsanol F[4]	Peniaphilone G[5]	Peniaphilone H[5]	Peniaphilone I [5]	Isochromophilone V[6]
	DMSO-d_6	DMSO-d_6	DMSO-d_6	DMSO-d_6	CD_3OD	$CDCl_3$	$CDCl_3$	$CDCl_3$	unknown[a]
1	7.52	7.54	7.54	7.62	7.59	7.36	7.37,s	7.36,s	7.38
4	6.51	6.34	6.19	6.55	6.61	6.52	6.55,s	6.53,s	6.53
5		5.24	5.18						
7-CH_3	1.09	1.10	1.10	1.17	1.28	1.28	1.28	1.29	1.30
8	4.32	4.15	4.14	4.21	4.33	4.34	4.34	4.34	4.36
9	6.39	6.24	2.36	6.42	6.42	6.05	6.14	6.08	6.06
10	6.57	6.67	1.57	6.80	6.87	6.99	7.44	7.04	7.03
11	1.89		0.87						
12		3.27		3.29	3.45	5.65	5.49	5.77	5.66
13		1.67		1.67	1.75	2.76	2.95	2.57	2.46
14		1.31 1.18		1.31 1.18	1.29 1.44	1.67 1.54	1.68 1.49	3.67	1.20-1.40
15		0.82		0.83	0.91	3.63 3.57	3.63 3.56	1.19	0.86
16		1.23		1.25	1.36	1.03	1.03	1.02	1.01
17		0.70		0.71	0.85	1.89	1.89	1.86	1.83

[a] The solvent for recording NMR data was not reported in the literature.

Table S2: ^{1}H NMR Data for natural products containing the 7,8-dihydro-6H-isochromen-6-one skeleton and the related compounds. (Continue)

no.	7,8-*cis*	7,8-*trans*					C-7,8 configuration unassigned
	1	Tersaphilone A[7]	Luteusin A[7]	Luteusin A[8]	Luteusin B[8]	tetrahydro TL-1[9]	Myxodiol A[10]
	DMSO-d_6	CD_3OD	CD_3OD	$CDCl_3$	$CDCl_3$	unknown[a]	unknown[a]
1	7.52	7.53	7.54	7.47	7.50	7.47	7.48
4	6.51	6.66	6.66	6.54	6.57	6.47	6.51
7-CH_3	1.09	1.18	1.19	1.22	1.22	1.20	1.22
8	4.32	4.54	4.55	4.67	4.67	4.65	4.67
9	6.39	6.32	6.30	6.08	6.17	2.52	2.31
10	6.57	7.09	7.12	7.08	7.46		
11	1.89						
12		5.72	5.68	5.68	5.51	0.95-1.76	
13		3.11	2.52	2.48	2.64		
14		2.30 2.37	1.46 1.32	1.43 1.32	1.32 1.43		
15			0.88	0.86	0.86	0.86	
16		1.90	1.87	1.84	1.91	0.85 or 0.93	
17		1.09	1.02	1.01	1.02	0.85 or 0.93	

[a] The solvent for recording NMR data was not reported in the literature.

Table S3: ^{13}C NMR Data for natural products containing the 7,8-dihydro-6H-isochromen-6-one skeleton and the related compounds.

no.	7,8-*cis*								
	1	GeumsanolA[1]	Azasperone H[2]	Geumsanol F[3]	Geumsanol F[4]	Peniaphilone G[5]	Peniaphilone H[5]	Peniaphilone I[5]	Isochromophilone V[6]
	DMSO-d_6	DMSO-d_6	DMSO-d_6	DMSO-d_6	CD_3OD	$CDCl_3$	$CDCl_3$	$CDCl_3$	unknown[a]
1	144.3	145.6	146.7	145.0	147.4	146.7	146.7	146.7	146.5
3	156.8	154.7	161.5	156.9	159.0	158.2	158.2	158.0	158.0
4	103.4	107.5	106.7	104.1	106.1	106.1	106.1	106.3	105.6
4a	140.0	143.2	143.9	139.2	141.7	140.8	140.8	140.8	140.5
5	108.3	105.4	104.5	108.2	109.5	107.4	107.4	107.0	106.0
6	190.9	197.8	198.3	190.6	193.5	191.5	191.5	191.5	191.1
7	76.1	75.0	75.4	75.9	77.7	76.4	76.5	76.5	76.2
7-CH_3	19.2	22.6	23.1	21.9	23.1	24.1	24.1	24.1	23.8
8	70.8	72.2	72.6	71.5	73.5	71.9	71.9	71.9	71.7
8a	120.0	119.8	120.6	119.7	120.4	117.0	117.0	116.9	116.5
9	123.5	118.1	34.8	118.2	120.3	117.1	117.1	117.4	116.3
10	134.8	143.2	20.1	144.9	146.0	141.9	141.9	141.7	142.2
11	18.3	74.8	13.8	74.9	76.7	132.6	132.6	134.6	131.9
12		79.3		79.3	81.0	146.8	146.9	142.9	147.9
13		34.3		34.3	36.3	30.2	30.3	41.3	35.0
14		28.5		28.5	30.0	40.2	40.2	71.9	30.0
15		11.9		11.9	12.2	61.3	61.3_2	21.0	11.9
16		27.5		27.3	26.6	20.8	20.	17.0	20.2
17		14.0		14.0	14.3	12.5	12.5	12.8	12.3

[a] The solvent for recording NMR data was not reported in the literature.

Table S3: ^{13}C NMR Data for natural products containing the 7,8-dihydro-6H-isochromen-6-one skeleton and the related compounds. (*Continued*)

no.	7,8-*cis*	7,8-*trans*					C-7,8 configuration unassigned
	1	Tersaphilone A[7]	Luteusin A[7]	Luteusin A[8]	Luteusin B[8]	Tetrahydro TL-1[9]	Myxodiol A[10]
	DMSO-d_6	CD_3OD	CD_3OD	$CDCl_3$	$CDCl_3$	Unknown[a]	Unknown[a]
1	144.3	145.9	145.9	144.3	144.4	144.8	144.8
3	156.8	160.4	160.6	159.2	159.0	165.9	161.8
4	103.4	105.9	105.6	104.7	105.4	103.7	104.5
4a	140.0	143.6	143.7	142.7	142.6	142.4	142.1
5	108.3	109.3	109.2	107.2	107.5	106.8	106.7
6	190.9	193.8	193.7	191.5	191.6	191.7	191.8
7	76.1	78.5	78.5	77.4	77.4	72.4	77.0
7-CH_3	19.2	19.1	19.1	19.0	19.0	18.9	18.9
8	70.8	72.7	72.7	72.2	72.1	77.1	72.3
8a	120.0	121.4	121.4	118.9	118.9	119.0	118.9
9	123.5	118.9	118.2	116.1	119.0	44.2	20.0
10	134.8	142.4	142.8	142.3	133.8	29.2	
11	18.3	134.2	133.7	131.9	129.9	29.8	
12		145.3	148.1	147.8	145.3	31.5	
13		31.6	36.2	35.0	34.1	31.6	
14		42.3	31.2	30.1	30.3	33.8	
15		176.0	12.4	11.9	12.0	11.2	
16		12.4	12.5	12.3	20.1	19.7	
17		20.6	20.7	20.2	21.	20.0	

[a]The solvent for recording NMR data was not reported in the literatures.

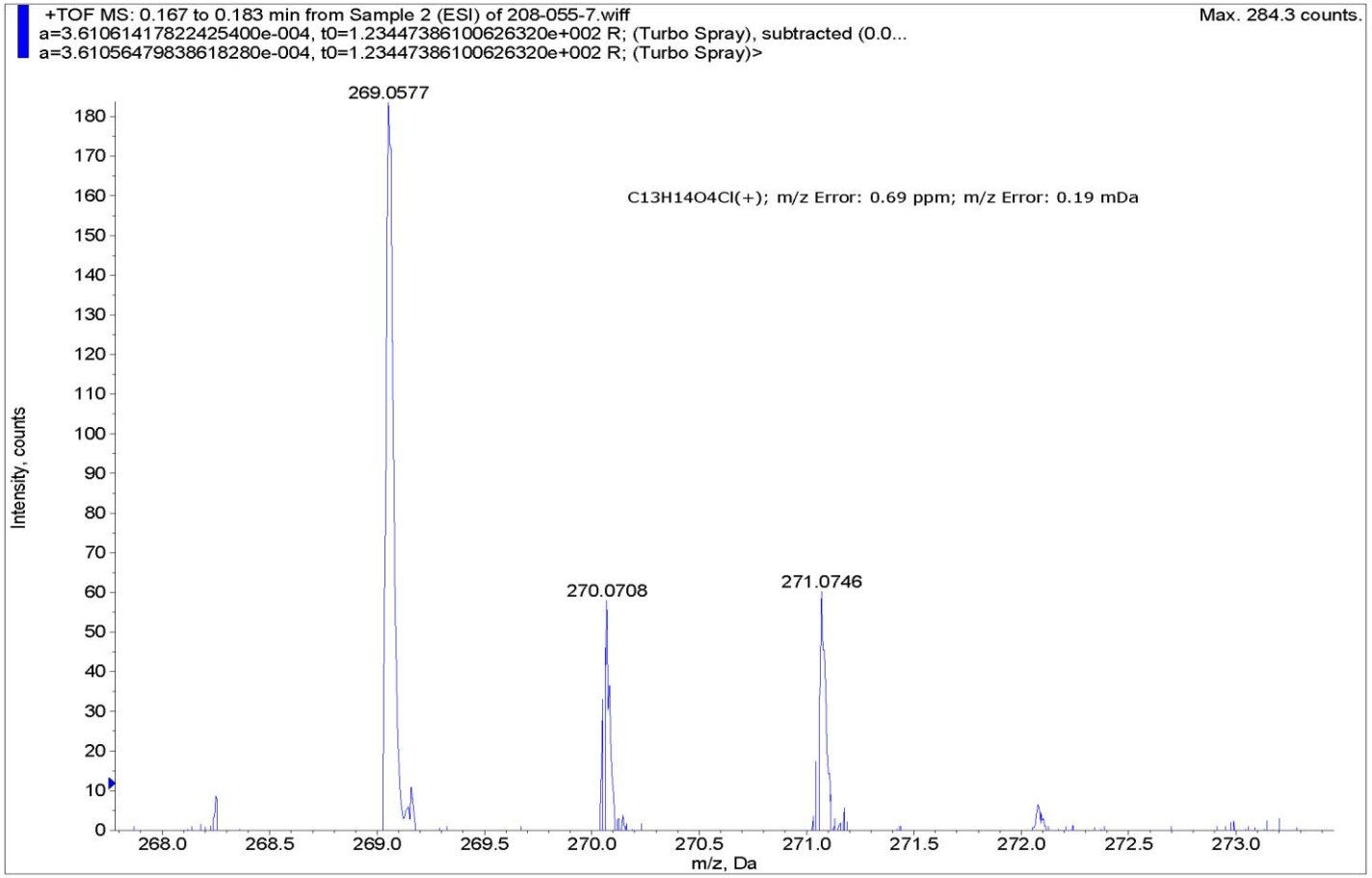


Figure S2: The HR-ESI-MS spectrum of **1**.

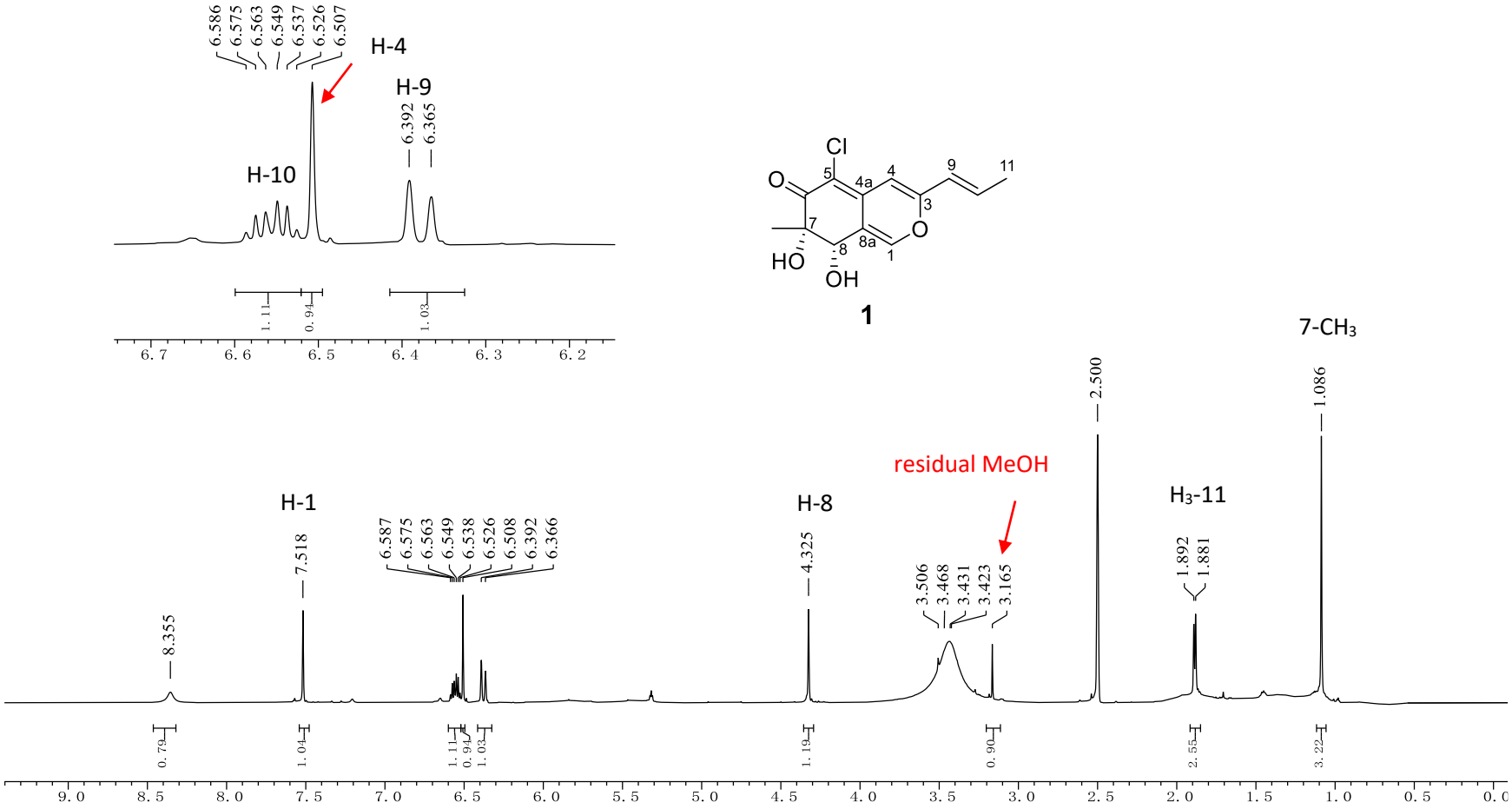


Figure S3: The ^{1}H-NMR spectrum of **1** (600 MHz, DMSO-d_6).

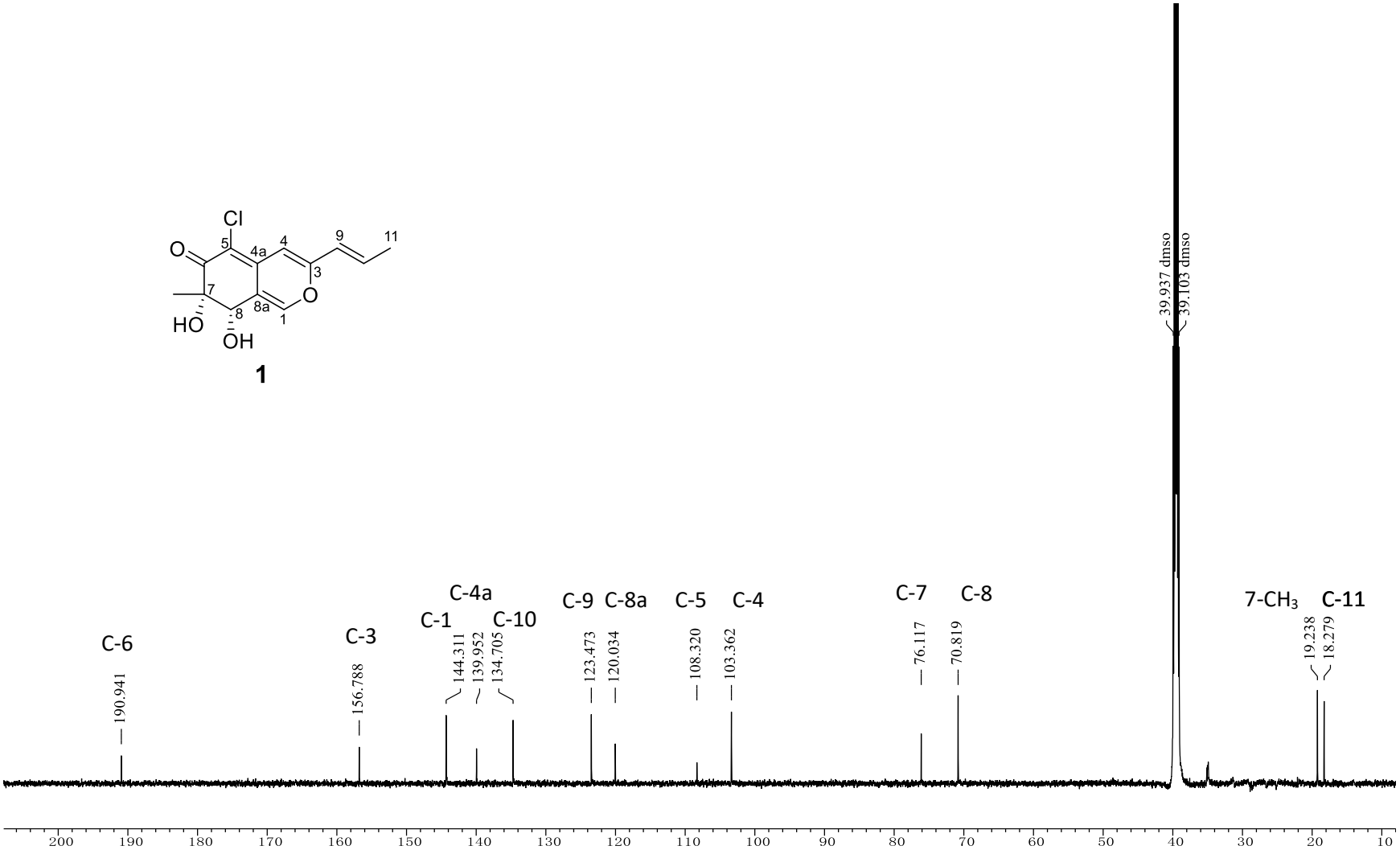


Figure S4: The ^{13}C-NMR spectrum of **1** (150 MHz, DMSO-d_6).

7-CH_3

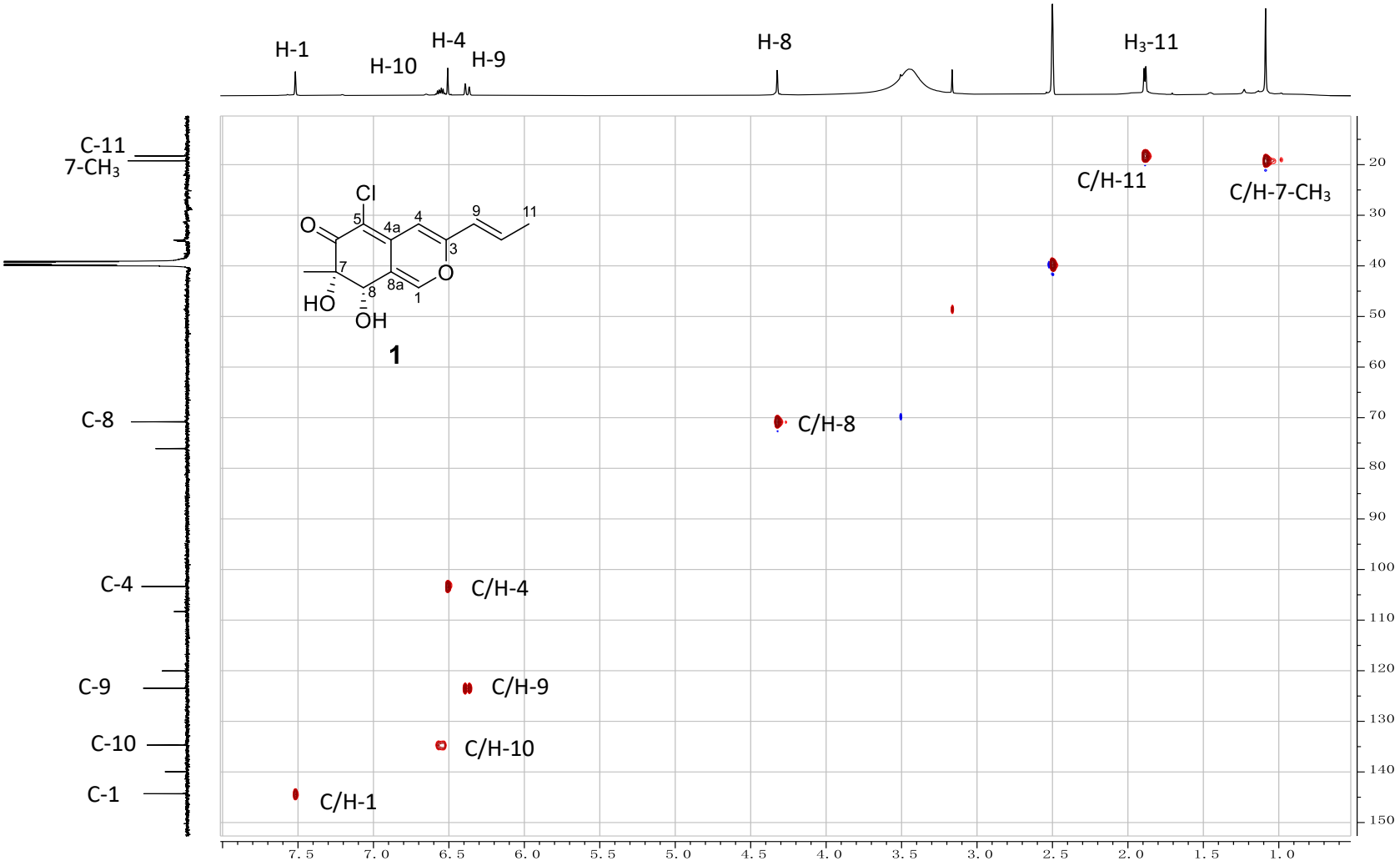


Figure S5: The HSQC spectrum of **1** (600 MHz, DMSO-d_6).

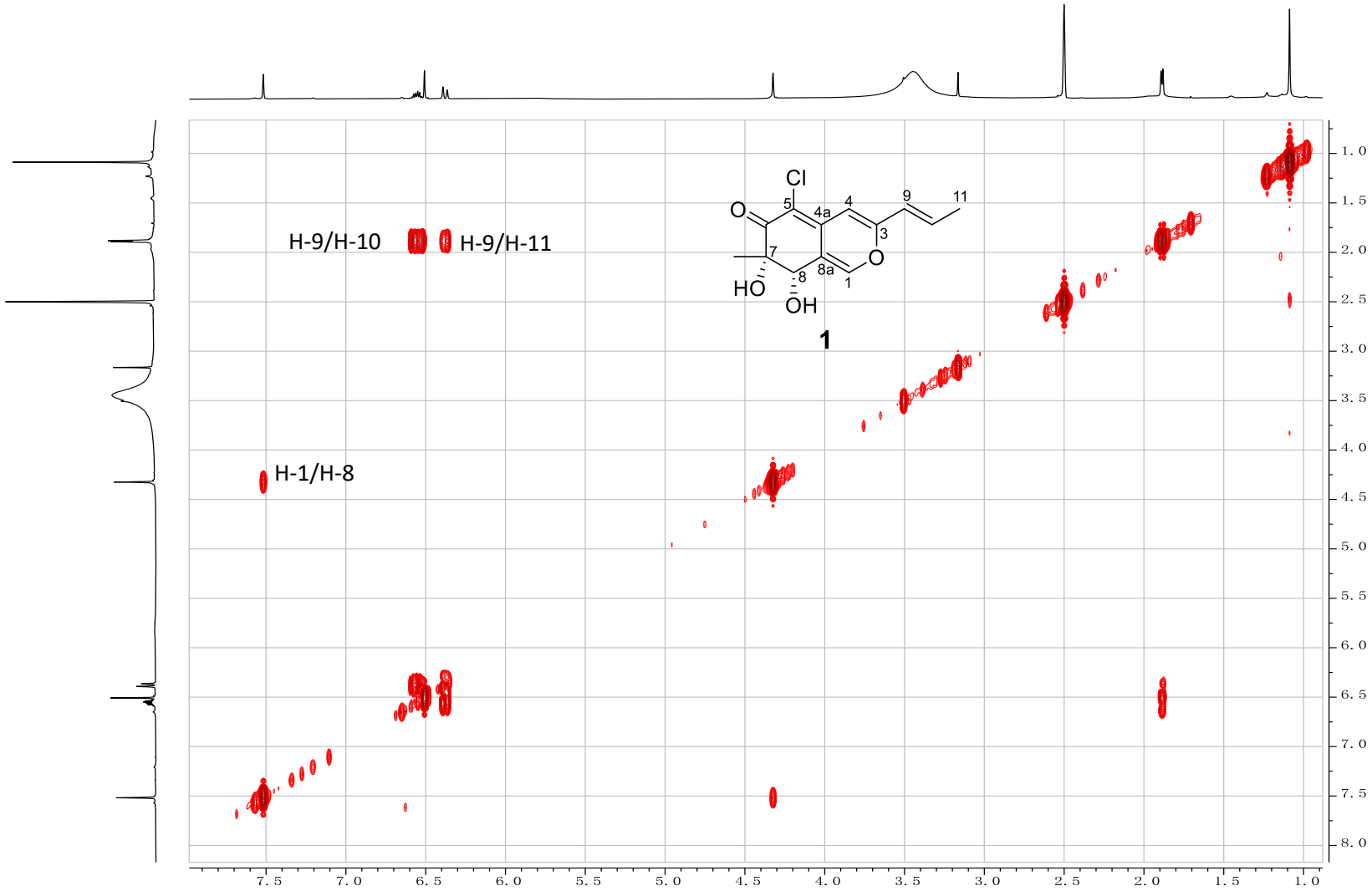


Figure S6: The ^{1}H-^{1}H COSY spectrum of **1** (150 MHz, DMSO-d_6).

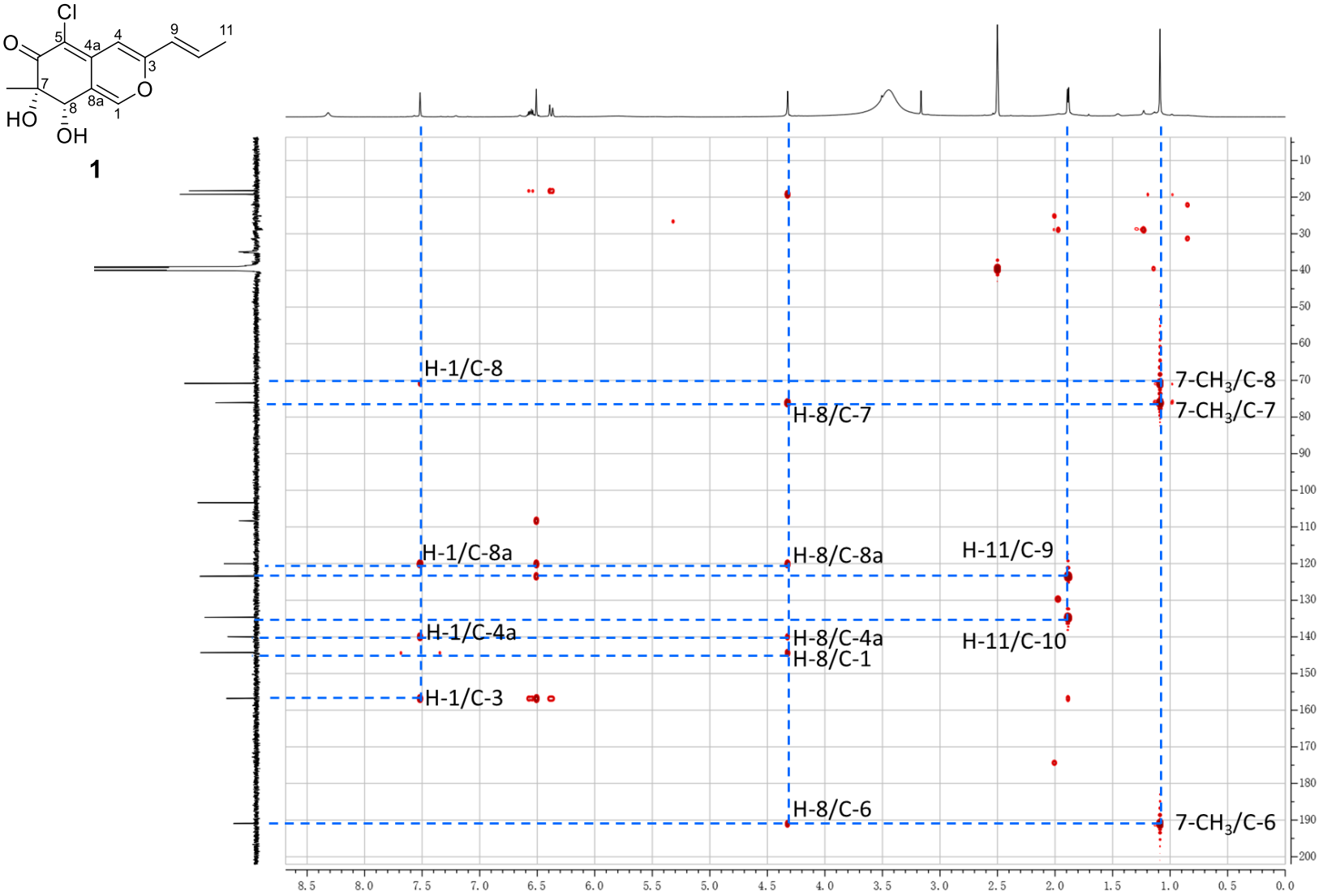


Figure S7: The HMBC spectrum of **1** (600 MHz, DMSO-d_6).

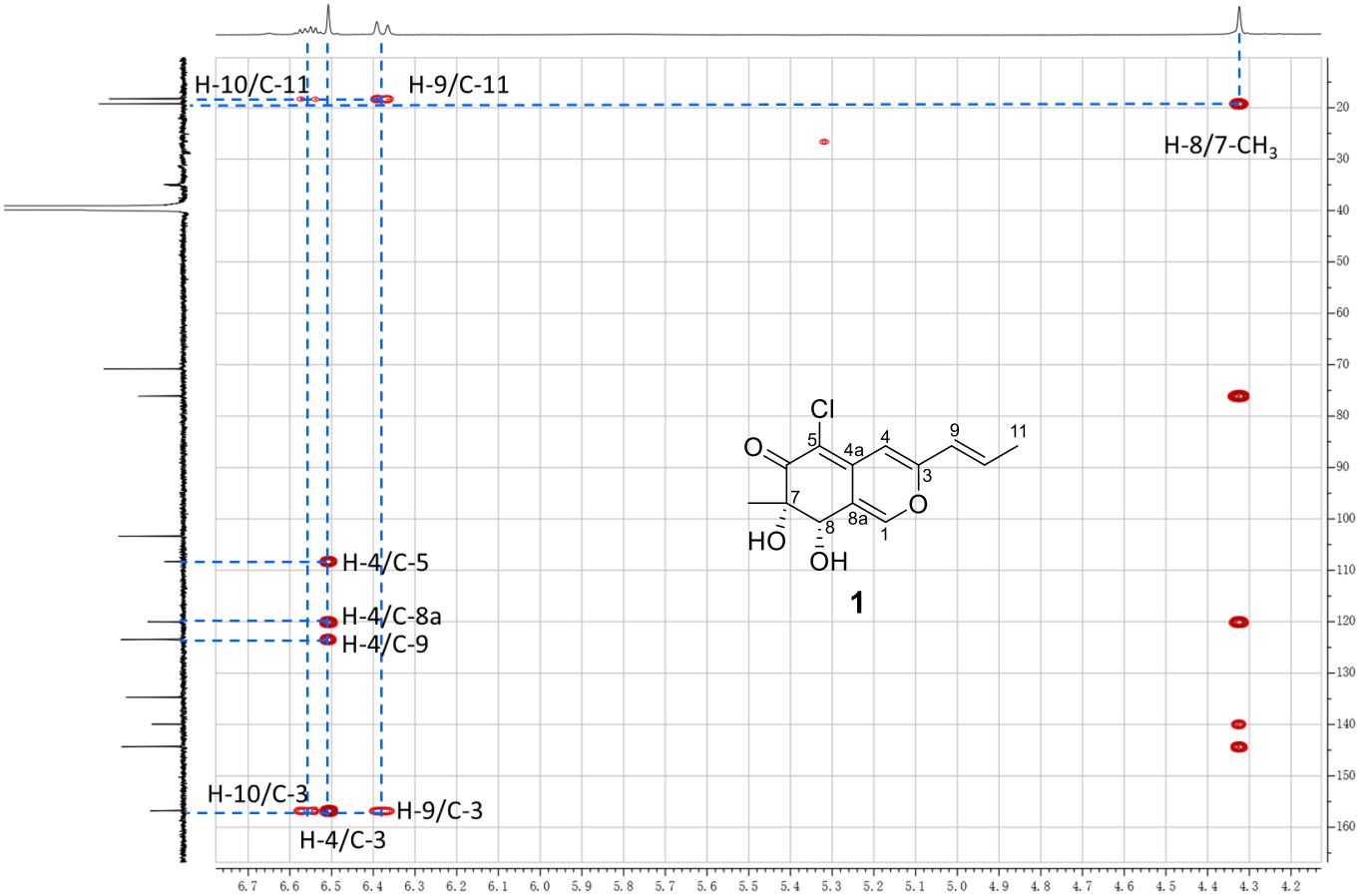


Figure S8: The enlarged HMBC spectrum of **1** (600 MHz, DMSO-d_6)

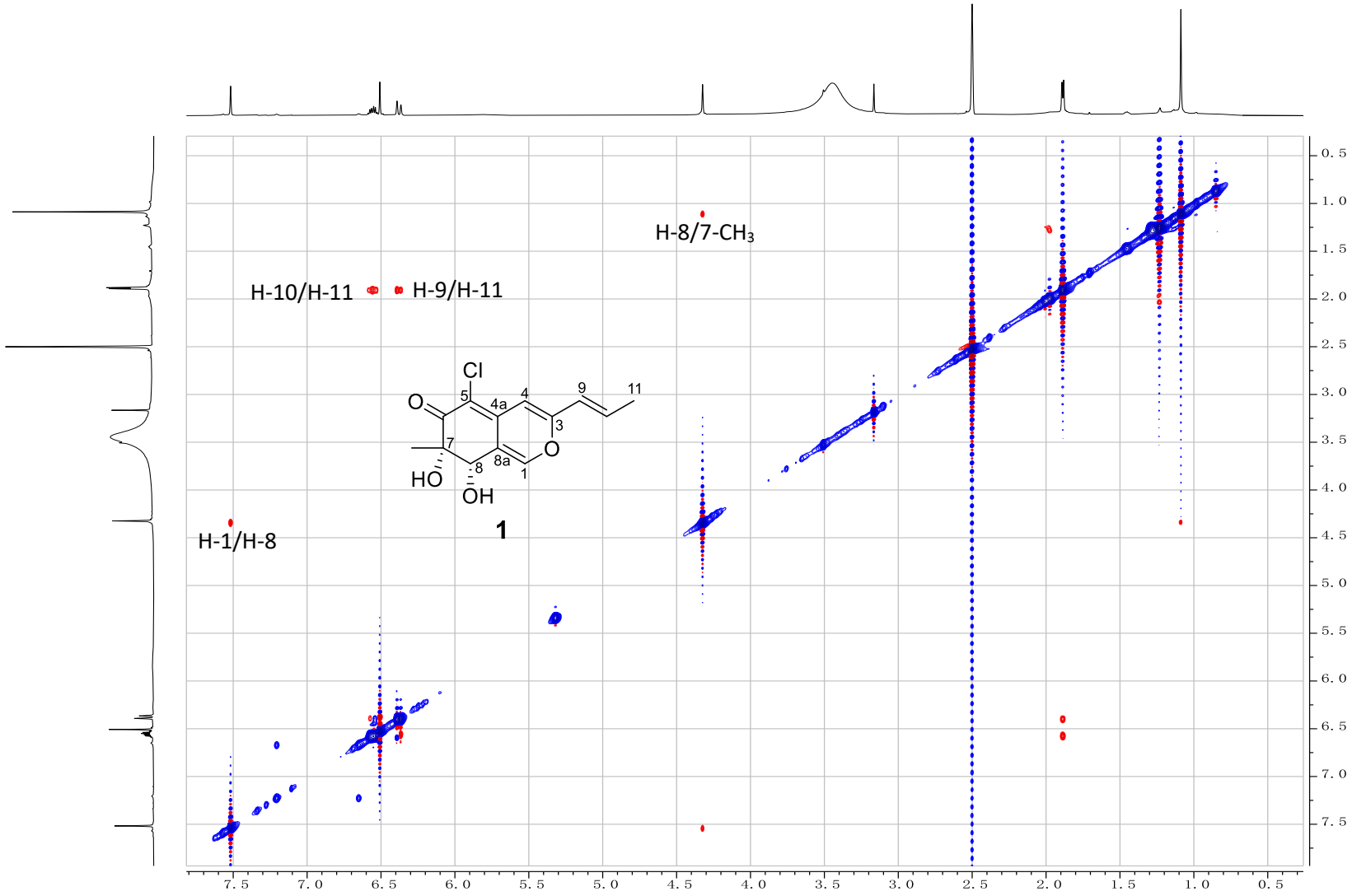


Figure S9: The ROESY spectrum of **1** (600 MHz, DMSO-d_6).

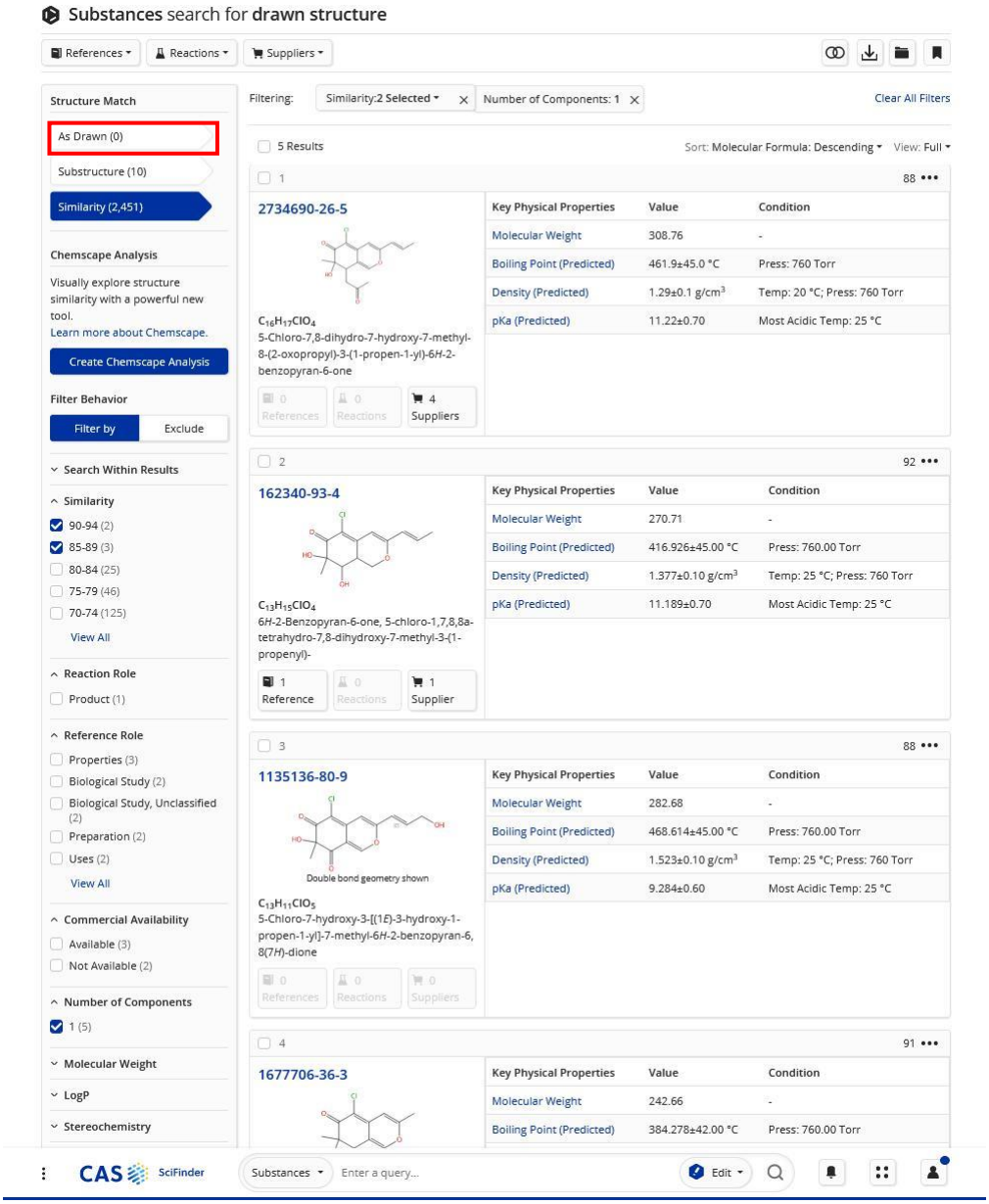


Figure S10: Search report from SciFinder for **1**.

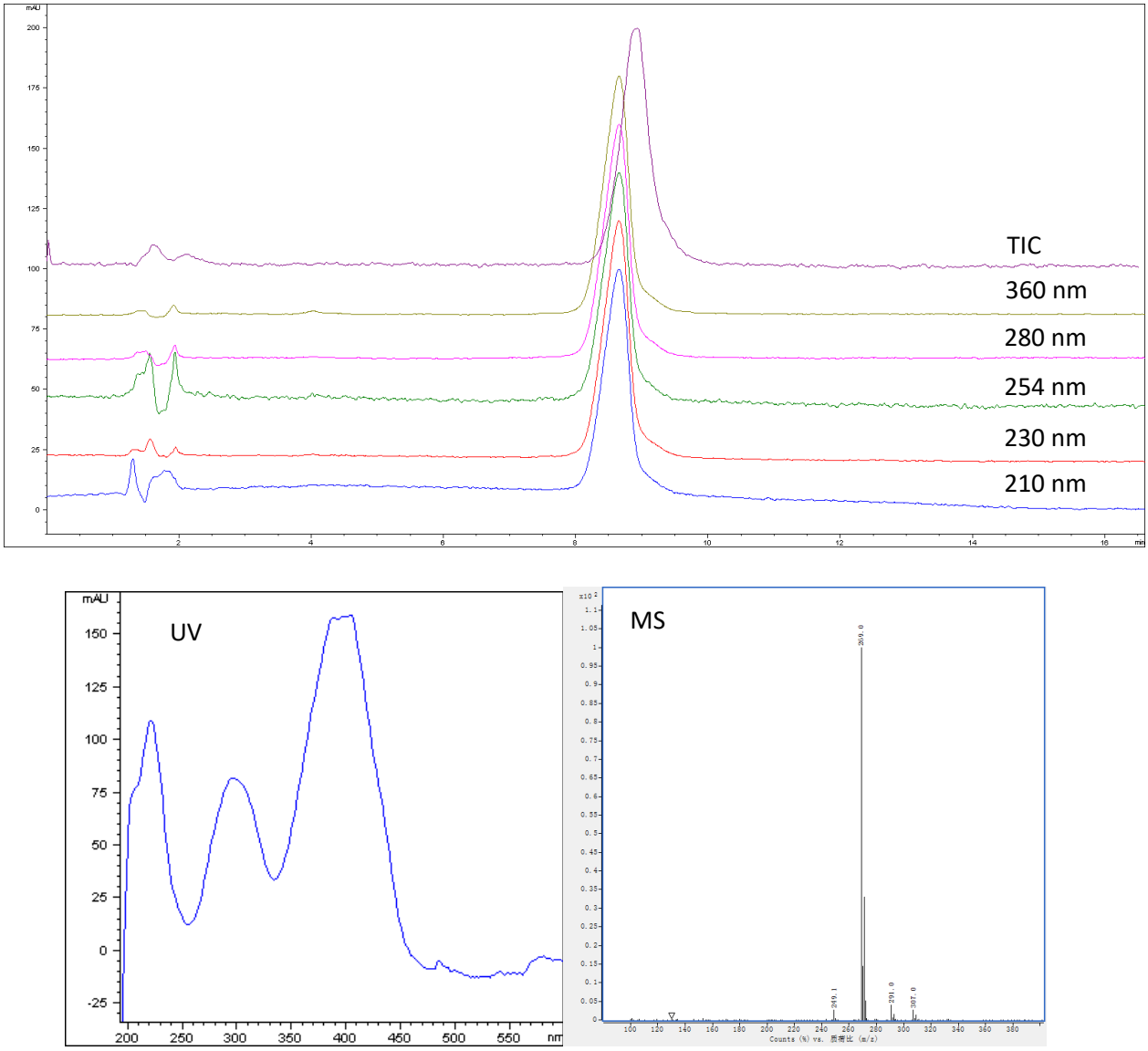


Figure S11: HPLC-MS analysis profile for **1**.

Table S4: Experimental and calculated ^{13}C NMR data of **1**.

No.	δexp	δcal		δscaled		Corrected error (Δδ)		*t* distribution		Probability	
		1A	1B	1A	1B	1A	1B	1A	1B	1A	1B
7-CH_3	19.2	22.0	20.3	17.2	16.8	-2.0	-2.4	0.80	0.84	0.20	0.16
11	18.3	22.1	22.9	17.3	19.4	-1.0	1.1	0.67	0.68	0.33	0.32
8	70.8	73.5	76.6	71.0	74.7	0.2	3.9	0.54	0.94	0.46	0.06
7	76.1	77.7	78.4	75.4	76.6	-0.7	0.5	0.62	0.58	0.38	0.42
4	103.4	104.1	99.3	103.0	98.2	-0.4	-5.2	0.57	0.98	0.43	0.02
5	108.3	112.6	108.7	111.9	107.8	3.6	-0.5	0.93	0.59	0.07	0.41
9	123.5	120.8	119.8	120.4	119.3	-3.1	-4.2	0.90	0.95	0.10	0.05
8a	120.2	121.1	120.6	120.8	120.0	0.6	-0.2	0.59	0.53	0.41	0.47
10	134.8	139.7	140.5	140.2	140.5	5.4	5.7	0.98	0.98	0.02	0.02
4a	140.0	140.8	142.1	141.3	142.2	1.3	2.2	0.71	0.82	0.29	0.18
1	144.3	145.0	143.5	145.7	143.7	1.4	-0.6	0.72	0.61	0.28	0.39
3	156.8	156.7	159.1	158.0	159.7	1.2	2.9	0.69	0.88	0.31	0.12
6	190.9	181.9	186.4	184.3	187.9	-6.6	-3.0	0.99	0.89	0.01	0.11
Product of probabilties										6.09E-11	3.63E-12
DP4+ probability (%)										94.4	5.6

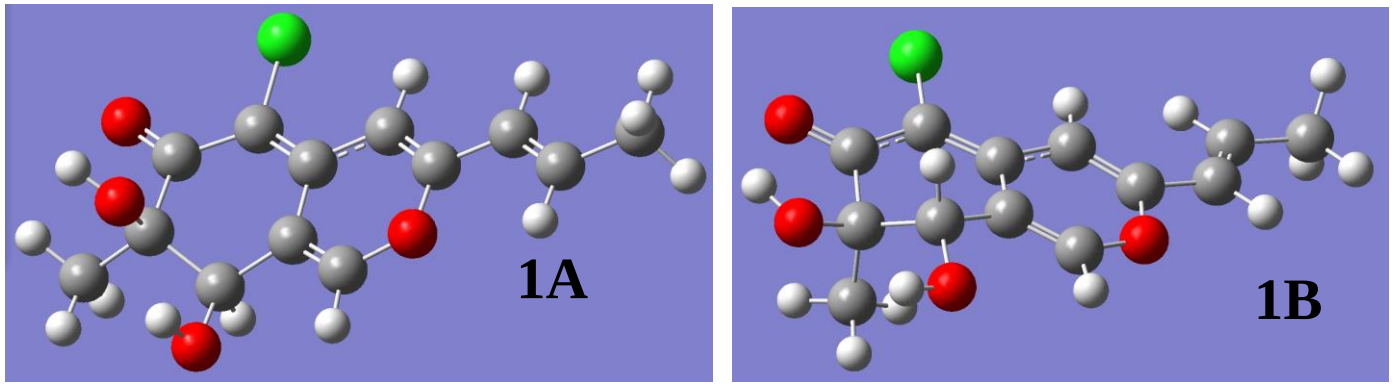


Figure S12: Relative structures of diastereomers for **1** (7,8-*cis*-**1A** and 7,8-*trans*-**1B**).

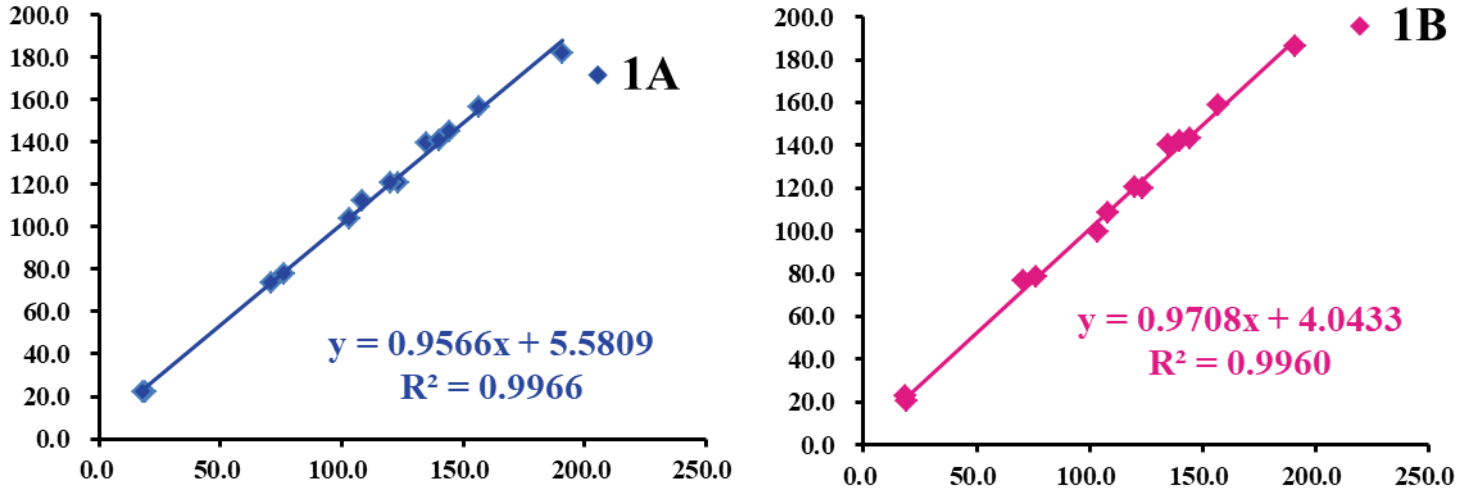


Figure S13: Regression analysis of experimental versus calculated ^{13}C NMR chemical shifts of 7,8-*cis*-**1A** and 7,8-*trans*-**1B**.

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