

The Chemically Highly Diversified Metabolites from a Red Sea Marine Sponge *Spongia* sp.

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Table S1. Selected ^1H and ^{13}C NMR data of **2** and similar compounds **13** and **14** in CDCl_3 and $\text{C}_5\text{D}_5\text{N}$.

position	2			13			14		
	$\delta_{\text{H}}^{\text{a}}$	$\delta_{\text{H}}^{\text{b}}$	$\delta_{\text{C}}^{\text{b}}$	$\delta_{\text{H}}^{\text{c}}$	$\delta_{\text{H}}^{\text{d}}$	$\delta_{\text{C}}^{\text{d}}$	$\delta_{\text{H}}^{\text{c}}$	$\delta_{\text{H}}^{\text{d}}$	$\delta_{\text{C}}^{\text{d}}$
3	4.07 m	4.68 m	67.2 CH	4.02 m	4.65 m	66.8 CH	4.06 m	4.65 m	66.8 CH
5	—	—	80.2 C	—	—	79.8 C	—	—	79.8 C
6	—	—	199.6 C	—	—	199.2 C	—	—	199.2 C
7	5.66 br s ^e	5.96 d (1.5)	120.7 CH	5.67 d (2.1)	5.95 d (1.8)	120.3 CH	5.67 d (2.0)	5.93 d (1.8)	120.3 CH
8	—	—	164.6 C	—	—	164.2 C	—	—	164.2 C
9	—	—	75.4 C	—	—	75.1 C	—	—	75.0 C
10	—	—	42.7 C	—	—	42.3 C	—	—	42.3 C
14	2.72 m	3.00 m	52.3 CH	2.74 m	3.00 m	52.0 CH	2.74 m	2.97 m	51.9 CH
17	1.39 m	1.37 m	56.7 CH	—	—	57.0 CH	—	—	56.3 CH
21	0.94 d (6.0)	0.99 d (4.5)	19.5 CH ₃	0.96 d (6.6)	1.00 d (6.6)	20.3 CH ₃	0.94 d (5.8)	1.00 d (6.6)	19.2 CH ₃
OH-3	—	—	—	—	6.34 d (4.8)	—	—	6.33 d (4.8)	—
OH-5	3.34 br s	8.66 br s	—	—	8.63 s	—	—	8.62 s	—
OH-9	4.12 br s	6.31 br s	—	—	6.32 br s	—	—	—	—

^a. ^1H spectra recorded at 600 MHz in CDCl_3 ; ^b. ^{13}C and ^1H spectra recorded at 125 and 500 MHz in $\text{C}_5\text{D}_5\text{N}$; ^c. ^1H spectra recorded at 270 MHz in CDCl_3 [31]; ^d. ^{13}C and ^1H spectra recorded at 150 and 600 MHz in $\text{C}_5\text{D}_5\text{N}$ [31]; ^e. Broad signal.

Table S2. Selected ^{13}C NMR data at C20-C29 of **2** and related compounds **15–18**.

position	2	15 (24S)	16 (24R)	2	17 (24S)	18 (24R)
	$\delta_{\text{C}}^{\text{a}}$	$\delta_{\text{C}}^{\text{b}}$	$\delta_{\text{C}}^{\text{b}}$	$\delta_{\text{C}}^{\text{c}}$	$\delta_{\text{C}}^{\text{d}}$	$\delta_{\text{C}}^{\text{d}}$
20-CH	36.44	36.29	36.17	37.23	37.05	36.91
21-CH ₃	18.86	18.82	18.82	19.53	19.16	19.16
22-CH ₂	33.68	33.95	33.95	34.42	34.11	34.16
23-CH ₂	26.35	26.43	26.13	27.16	26.78	26.50
24-CH	46.00	46.07	45.85	46.74	46.32	46.09
25-CH	28.90	28.98	29.18	29.72	29.28	29.51
26-CH ₃	18.96	19.07	19.84	19.61	19.16	20.00
27-CH ₃	19.55	19.62	19.07	20.23	19.78	19.27
28-CH ₂	23.03	23.09	23.09	23.83	23.34	23.39
29-CH ₃	12.29	12.32	12.32	12.96	12.52	12.17

^a. ^{13}C spectra recorded at 150 MHz in CDCl_3 ; ^b. ^{13}C spectra recorded at 25.16 MHz in CDCl_3 [32].

^c. ^{13}C spectra recorded at 150 MHz in $\text{C}_5\text{D}_5\text{N}$; ^d. ^{13}C spectra recorded at 149.9 MHz in $\text{C}_5\text{D}_5\text{N}$ [35].

Table S3. ^{13}C and ^1H NMR data of **3** and related compound **19**.

(22 <i>E</i> ,24 <i>S</i>)-Ergosta-7,22-dien-3 β ,5 α -diol-6,5-olide (3) ^a			(22 <i>E</i> ,24 <i>R</i>)-Ergosta-7,22-dien-3 β ,5 α -diol-6,5-olide (19) ^b	
position	δ_{H}	δ_{C}	δ_{H}	δ_{C}
1	1.95, m 1.40, m	35.9, CH ₂	1.93, m 1.40, m	35.8, CH ₂
2	1.86, m 1.40, m	30.4, CH ₂	1.85, m 1.40, m	30.4, CH ₂
3	3.79, m	67.6, CH	3.79, m	67.6, CH
4	2.25, m 2.10, m	46.2, CH ₂	2.28, m 2.11, m	46.1, CH ₂
5	–	104.3, C	–	104.3, C
6	–	166.4, C	–	166.6, C
7	5.70, br s ^c	115.3, CH	5.69, s	115.2, CH
8	–	159.7, C	–	159.4, C
9	2.26, m	51.7, CH	2.25, m	51.7, CH
10	–	43.1, C	–	42.8, C
11	1.82, m 1.64, m	25.3, CH ₂	1.82, m 1.64, m	25.3, CH ₂
12	2.05, m 1.42, m	39.9, CH ₂	2.05, m 1.41, m	39.9, CH ₂
13	–	46.6, C	–	46.6, C
14	2.14, m	58.1, CH	2.13, m	58.0, CH
15	1.58, m 1.50, m	23.1, CH ₂	1.53, m 1.49, m	23.0, CH ₂
16	1.77, m 1.33, m	28.0, CH ₂	1.76, m 1.36, m	27.7, CH ₂
17	1.37, m	56.3, CH	1.35, m	56.4, CH
18	0.62, s	12.4, CH ₃	0.62, s	12.5, CH ₃
19	1.06, s	17.8, CH ₃	1.05, s	17.7, CH ₃
20	2.05, m	40.4, CH	2.03, m	40.3, CH
21	1.01, d (6.0) ^d	21.0, CH ₃	1.01, d (6.6)	21.0, CH ₃
22	5.13, dd (15.0, 7.8)	135.2, CH	5.14, dd (15.2, 8.5)	135.0, CH
23	5.21, dd (15.0, 8.4)	132.7, CH	5.23, dd (15.3, 7.8)	132.6, CH
24	1.85, m	43.1, CH	1.85, m	42.8, CH
25	1.47, m	33.2, CH	1.47, m	33.0, CH
26	0.82, d (6.6)	19.6, CH ₃	0.84, d (6.9)	19.9, CH ₃
27	0.84, d (7.2)	20.1, CH ₃	0.82, d (6.8)	19.6, CH ₃
28	0.92, d (7.2)	18.0, CH ₃	0.91, d (6.8)	17.6, CH ₃
OH-5			3.99, brs	

^a. ^{13}C and ^1H spectra recorded at 150 and 600 MHz in CDCl_3 ; ^b. ^{13}C and ^1H spectra recorded at 150 and 600 MHz in CDCl_3 [36]; ^c. Broad signal; ^d. J values (Hz) in parentheses.

Table S4. Cytotoxicity of compounds **1–9**.

Compound	Huh7 cell viability		
	12.5 μ M (%, mean \pm SD)	50 μ M (%, mean \pm SD)	200 μ M (%, mean \pm SD)
1	— ^a	—	—
2	—	—	—
3	—	—	—
4	—	—	—
5	—	83.0 \pm 3.8	56.8 \pm 2.3
6	—	—	—
7	—	—	—
8	—	68.1 \pm 1.1	46.6 \pm 0.7
9	—	100.2 \pm 2.7	97.7 \pm 2.3
Sorafenib	47.7 \pm 0.3		

^a viability %>100. Results are presented as mean \pm SD. (n = 3).

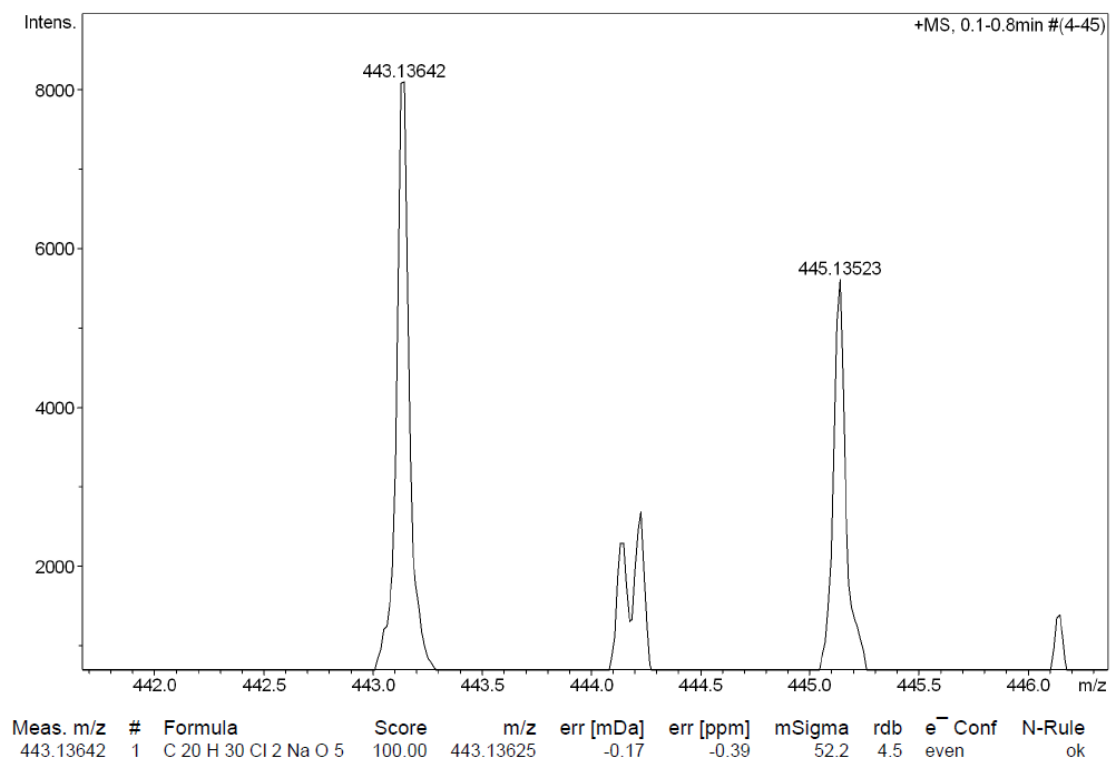


Figure S1. HRESIMS spectrum of **1**.

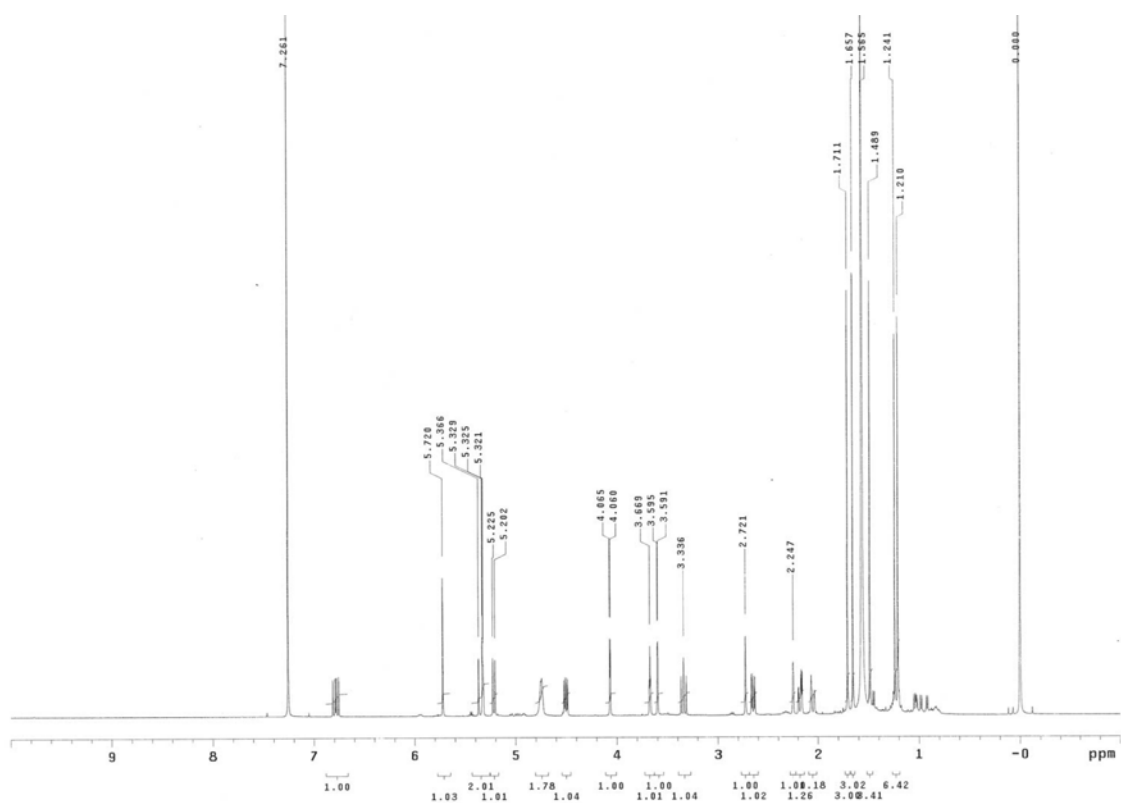


Figure S2. ¹H NMR spectrum of **1**.

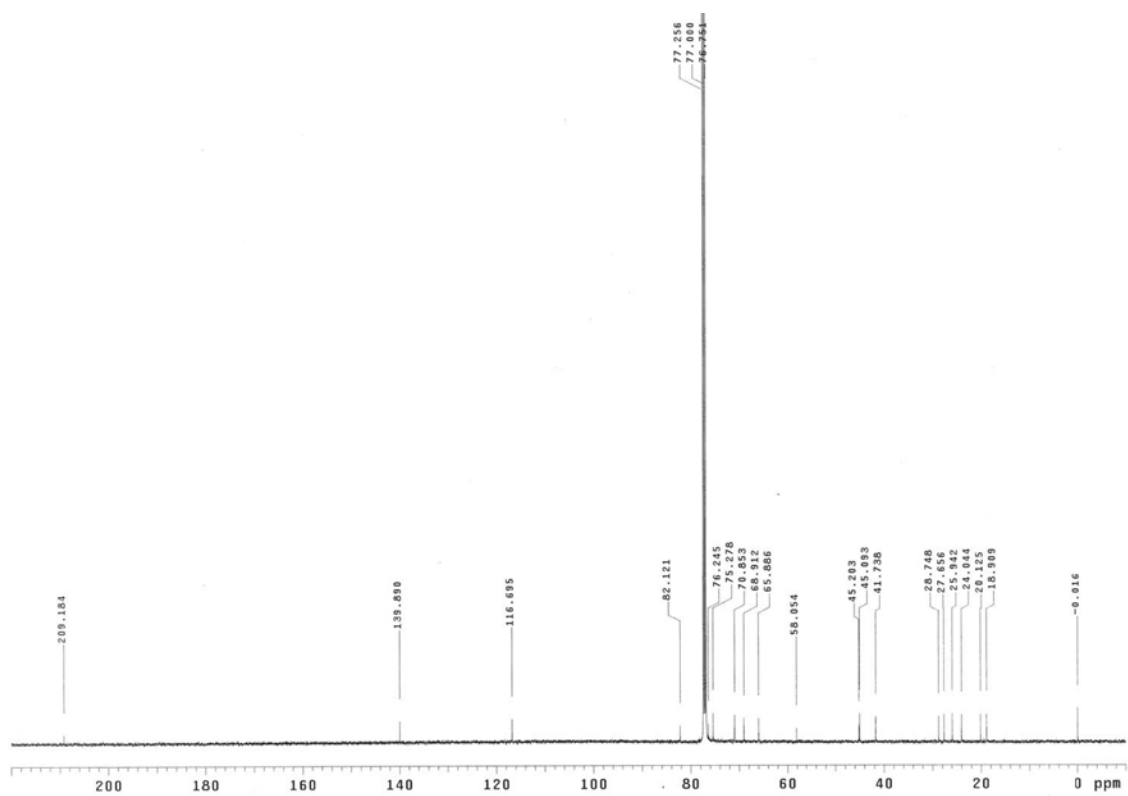


Figure S3. ¹³C NMR spectrum of **1**.

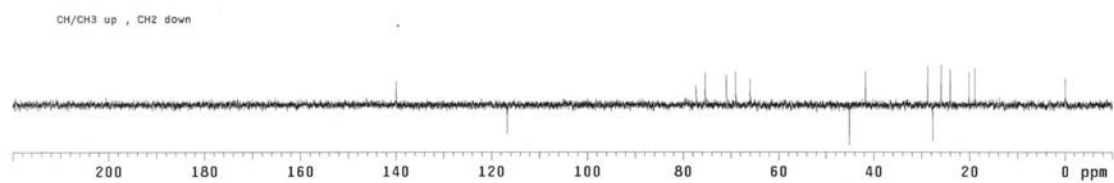


Figure S4. DEPT spectrum of **1**.

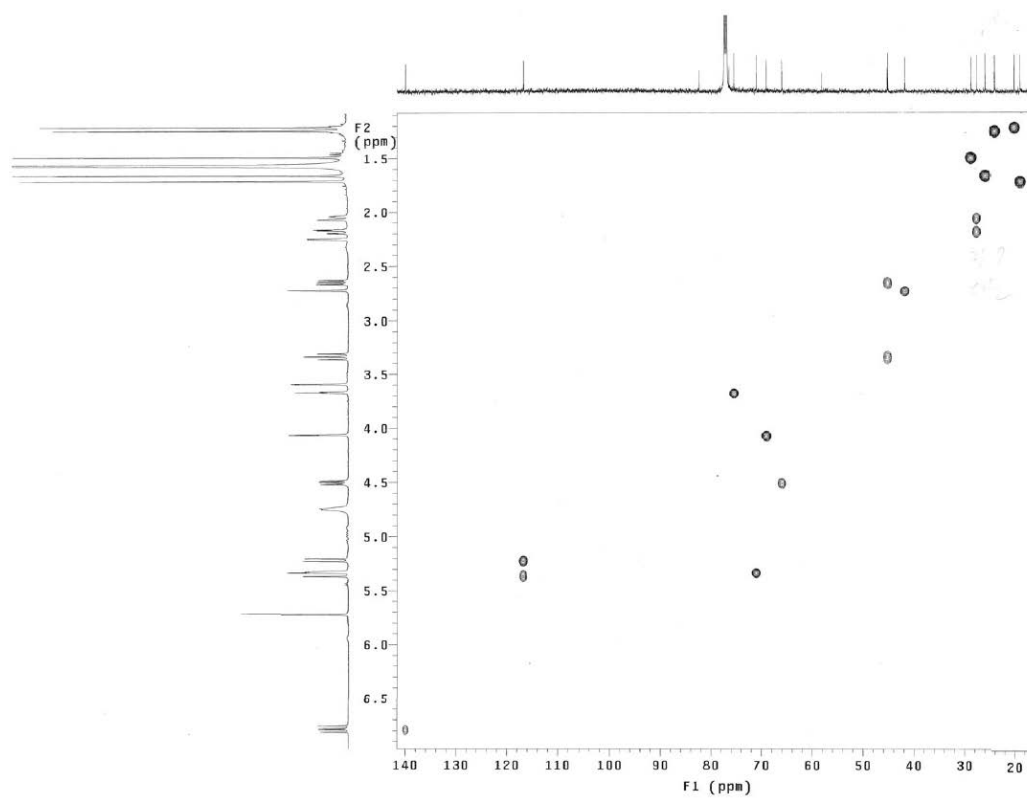


Figure S5. HSQC spectrum of **1**

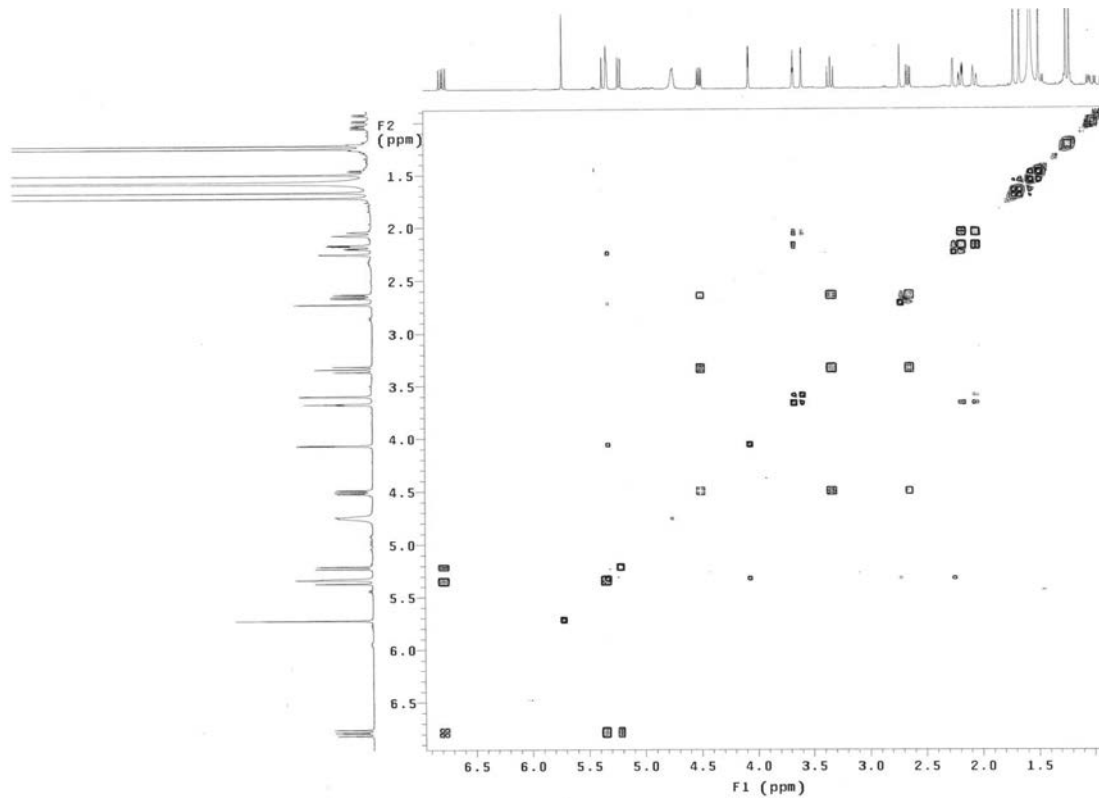


Figure S6. ^1H - ^1H COSY spectrum of **1**.

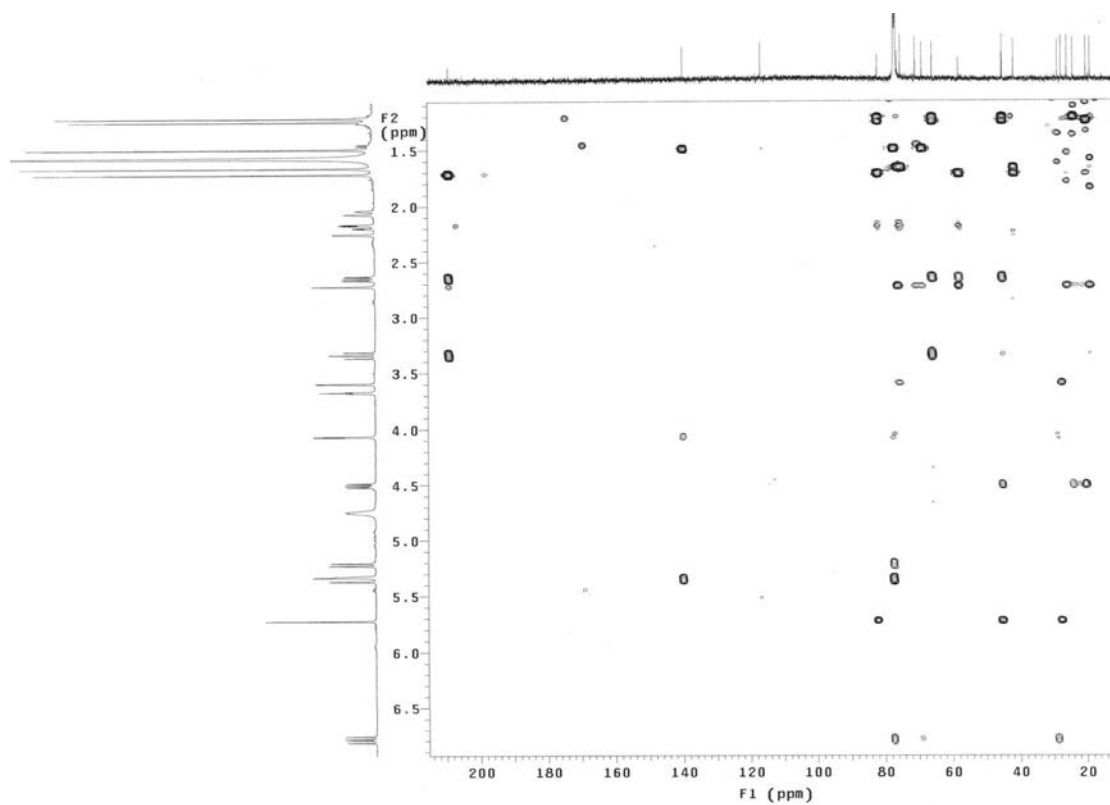


Figure S7. HMBC spectrum of **1**.

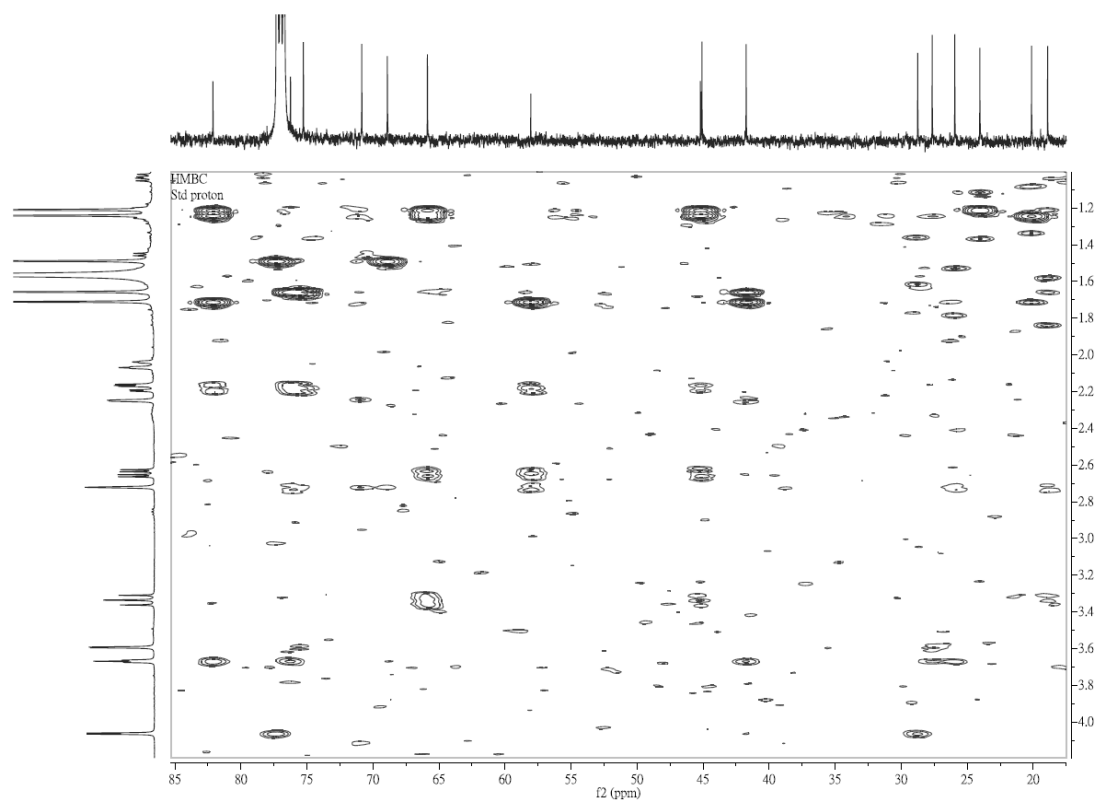


Figure S8. The partial HMBC spectrum of **1**.

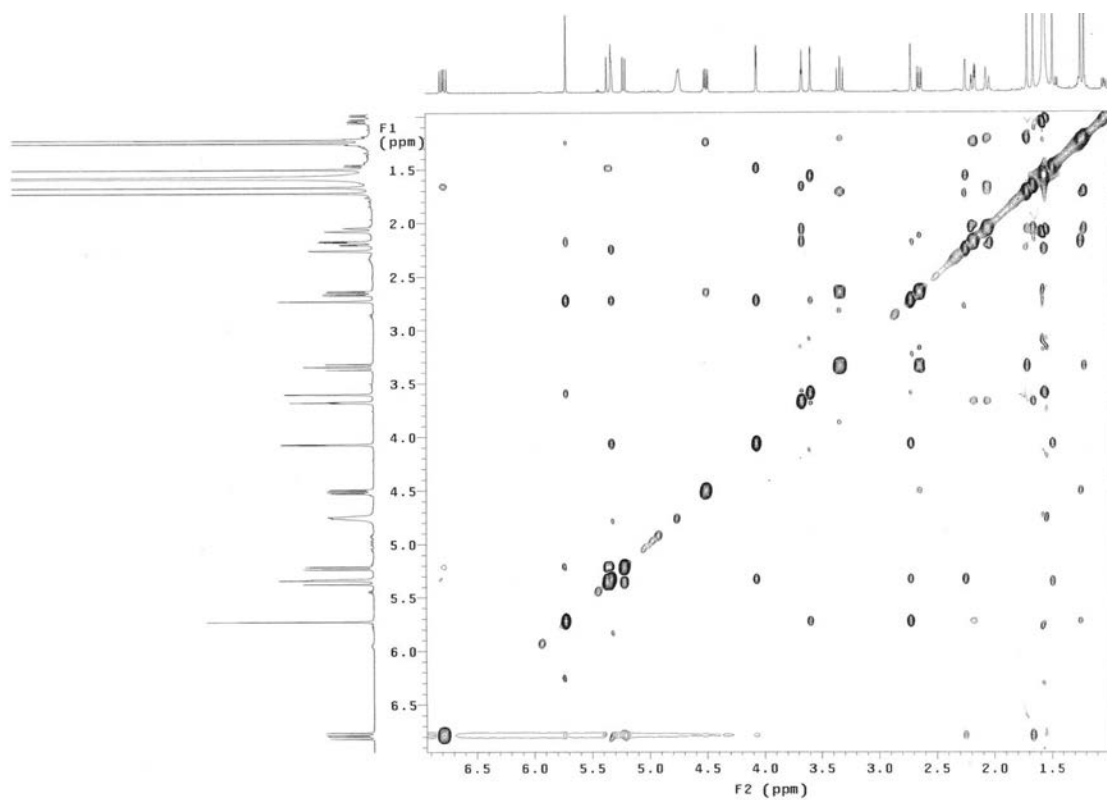


Figure S9. NOESY spectrum of **1**.

Table S5. The cartesian coordinates of conformer of compound **1** at the B3LYP/6-311+G(d,p) level of theory.

C	-2.9986360	1.8877370	-0.0588340	H	-1.2717860	-2.2681090	1.3382890
C	-3.7943200	0.6610420	-0.5062620	H	-1.7591470	-2.9274280	-0.2096550
C	-3.3435710	-0.6788630	0.1338760	H	0.6487820	-3.2629890	0.2542500
C	-1.7765200	-0.7617600	-0.1585970	H	0.5068930	0.1495050	-1.1191040
C	-0.8948330	0.4397040	0.4432860	H	2.5349820	1.0369040	-1.7287260
C	-1.4938990	1.6834840	-0.2455520	H	1.1921810	2.2557270	-0.2214640
C	-1.1849450	-2.1240730	0.2590610	H	-3.5397210	0.1184680	2.2012730
C	0.2780340	-2.3128360	-0.1543390	H	-4.8162240	-0.9733200	1.6892420
C	1.1922890	-1.1769320	0.3706160	H	-3.2346040	-1.6193630	2.1099760
C	0.5856710	0.2001460	-0.0288130	H	-5.1329310	-1.6542570	-0.6238330
C	3.4894010	-0.4005040	-0.4287980	H	-3.6990360	-1.9308070	-1.6346750
C	2.8443320	0.9877060	-0.6832990	H	-3.8998910	-2.7904210	-0.0913640
C	1.6165450	1.3357250	0.1818480	H	-0.9610090	-1.2045570	-1.8999630
O	-0.8595970	2.4765970	-0.9186200	H	-1.0266780	-0.3072980	2.5200490
C	-3.7416620	-0.7821250	1.6215800	H	-0.0961750	1.1794210	2.3240370
C	-4.0547590	-1.8375840	-0.6064030	H	-1.8527440	1.2217580	2.2843840
O	-1.6889970	-0.6228420	-1.5902820	H	1.2521800	-2.1763810	-1.8186740
C	-0.9807530	0.6416750	1.9829120	H	1.8956500	-0.5863440	2.3698540
O	0.3252970	-2.3854420	-1.5893640	H	0.5679170	-1.7439430	2.3913890
C	1.4756330	-1.4494030	1.8593440	H	2.1800200	-2.2838030	1.9230240
O	1.9441120	1.5255760	1.5582090	H	2.4776600	2.3363060	1.6134200
Cl	4.0927740	2.3301760	-0.5249340	H	3.8594310	0.1052020	1.6969080
C	4.2863140	-0.4509240	0.8693510	H	5.9148390	-1.1134010	2.0277610
C	5.4269760	-1.1162060	1.0564060	H	5.9237080	-1.6830200	0.2744800
C	4.3164920	-0.8257280	-1.6459740	H	5.1872700	-0.1779720	-1.7731310
O	2.3998580	-1.3713070	-0.4146020	H	4.6584920	-1.8573050	-1.5300960
Cl	-5.5885870	1.0222630	-0.2108500	H	3.7013190	-0.7728530	-2.5492760
H	-3.2870290	2.7628160	-0.6449270				
H	-3.2114450	2.1065750	0.9924680				
H	-3.7362000	0.5670940	-1.5887490				

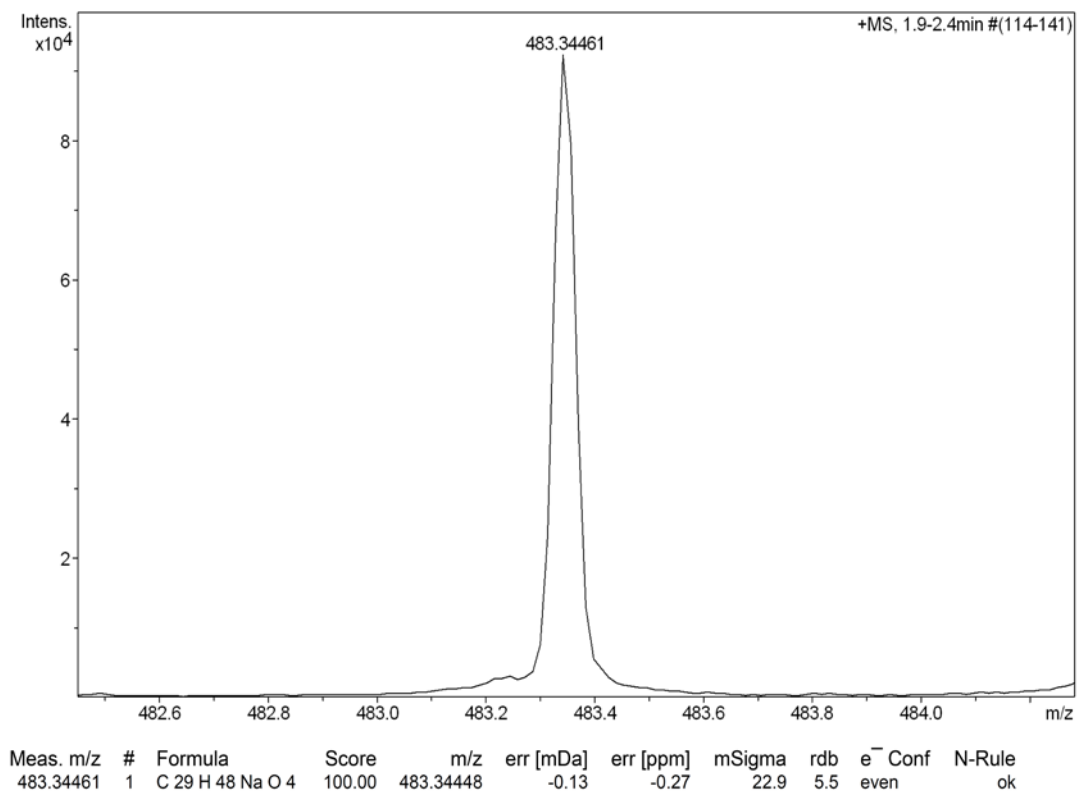


Figure S10. HRESIMS spectrum of **2**.

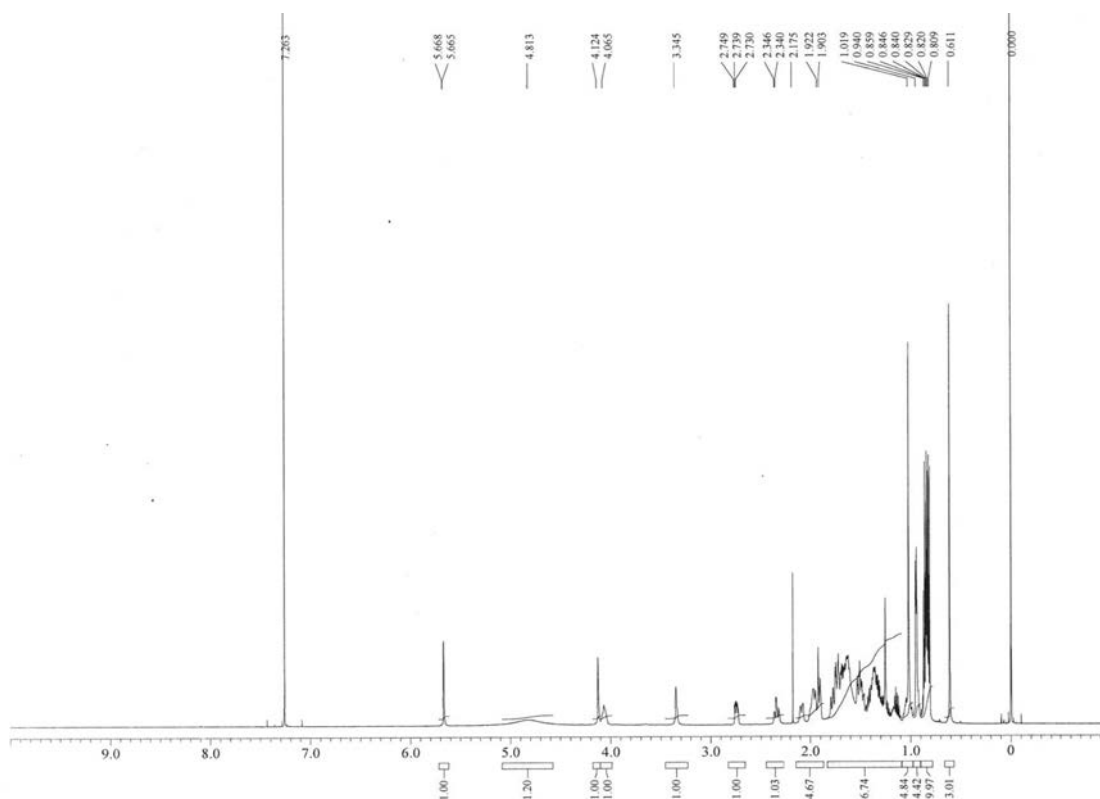


Figure S11. ^1H NMR spectrum of **2**.

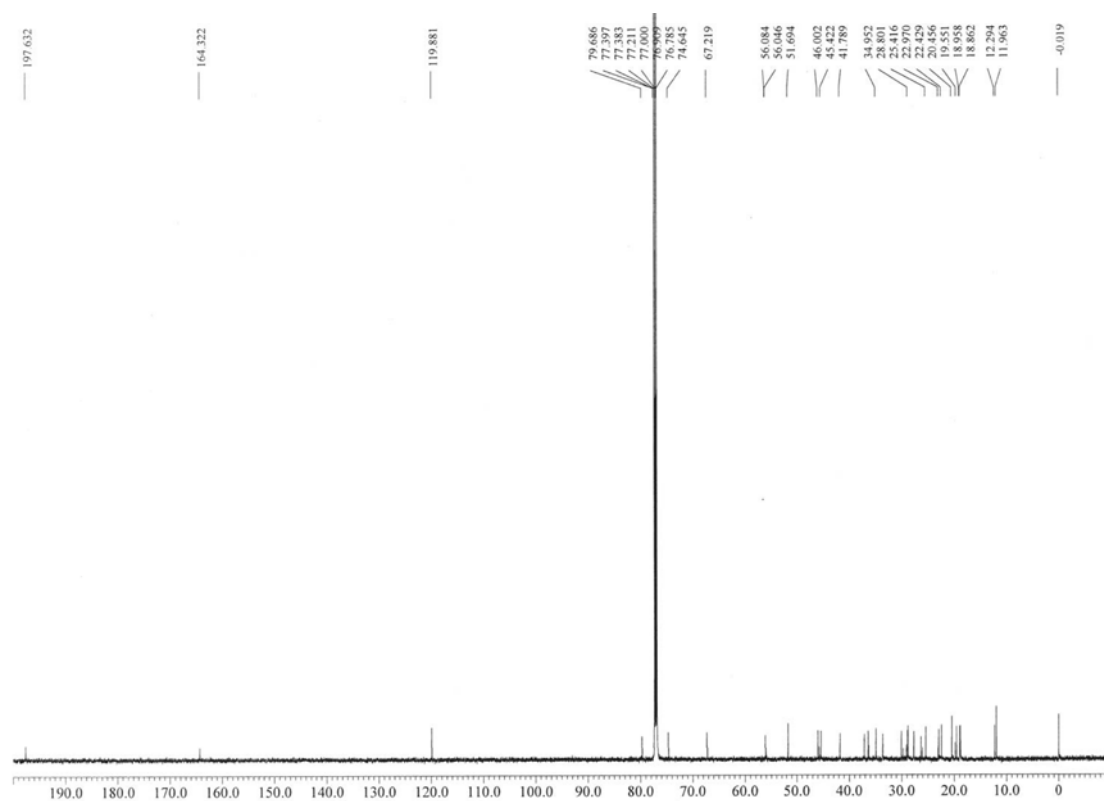


Figure S12. ^{13}C NMR spectrum of **2**.

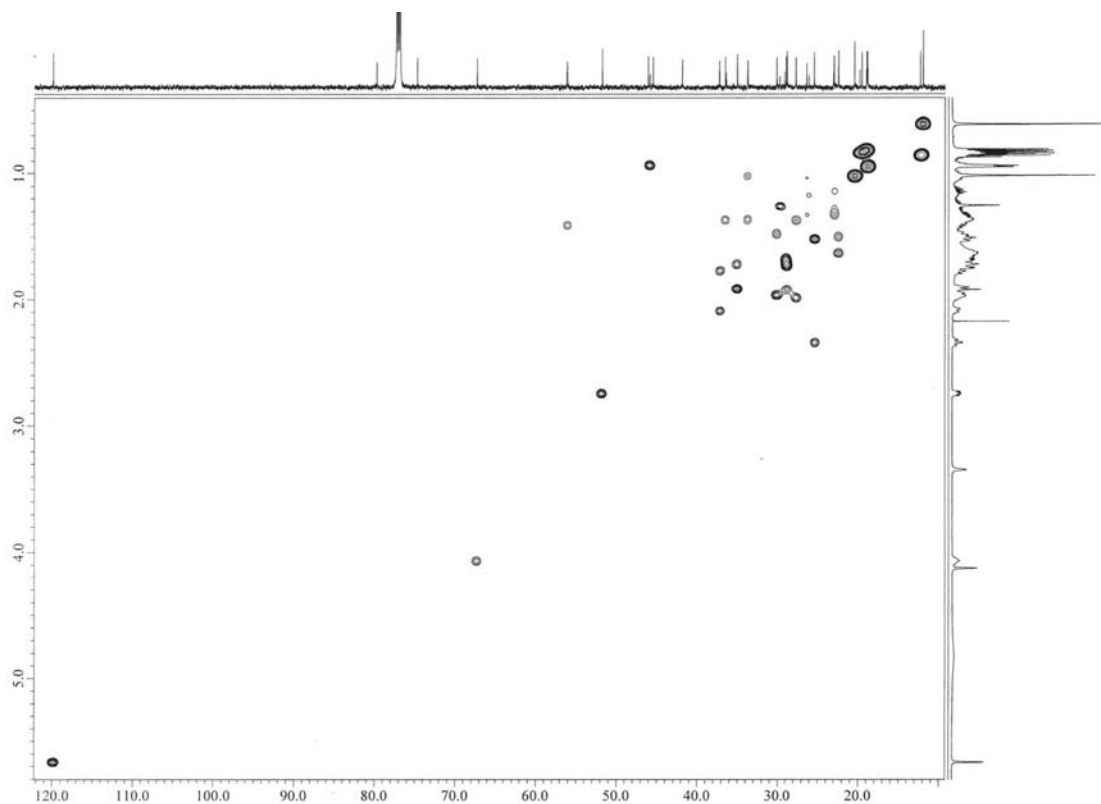


Figure S13. HSQC spectrum of **2**.

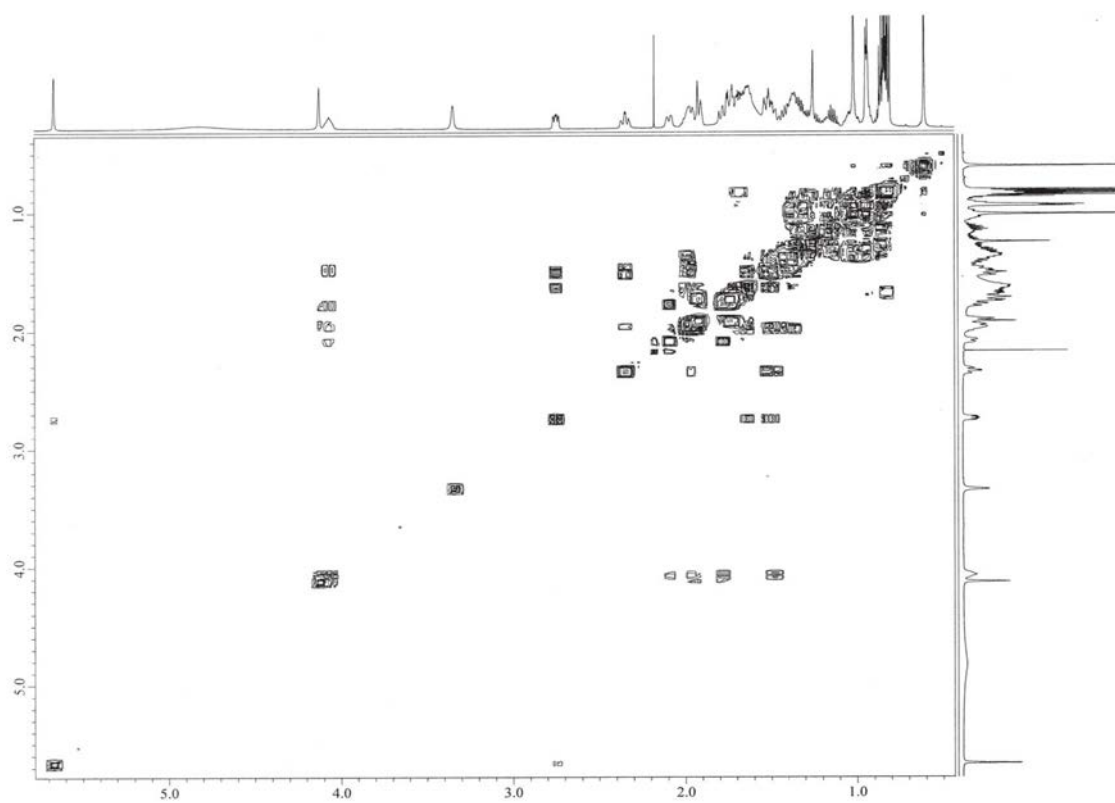


Figure S14. ^1H – ^1H COSY spectrum of **2**.

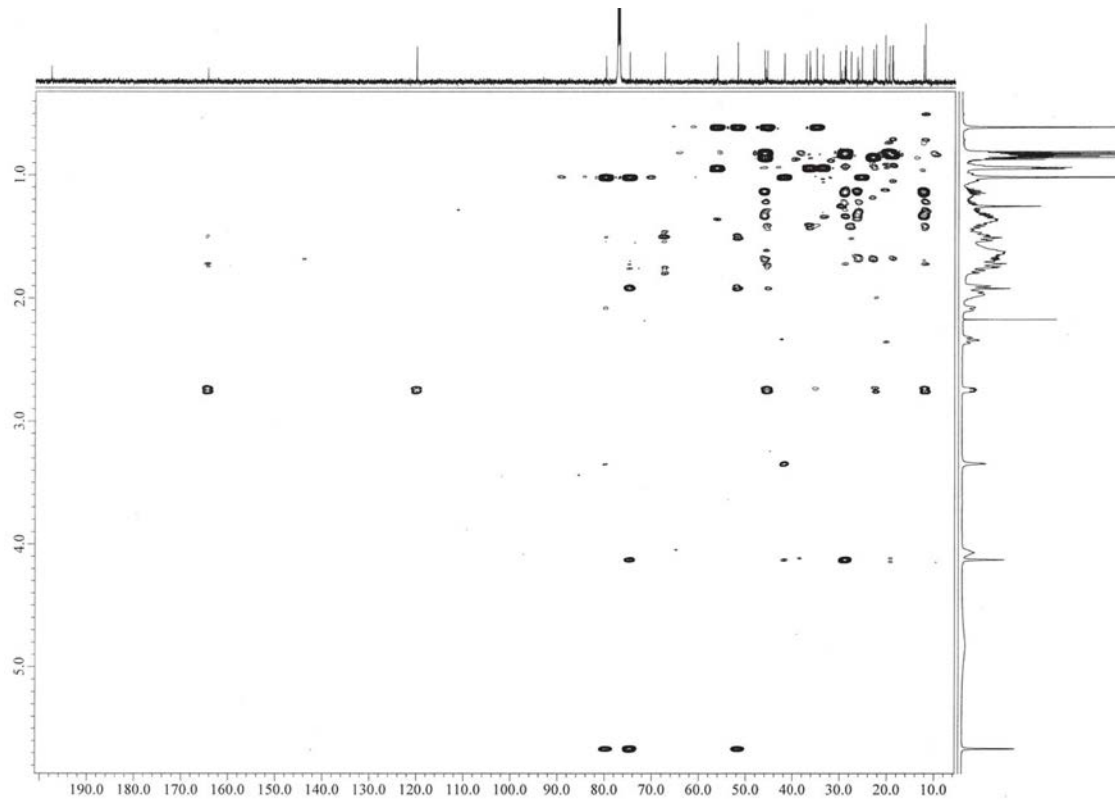


Figure S15. HMBC spectrum of **2**.

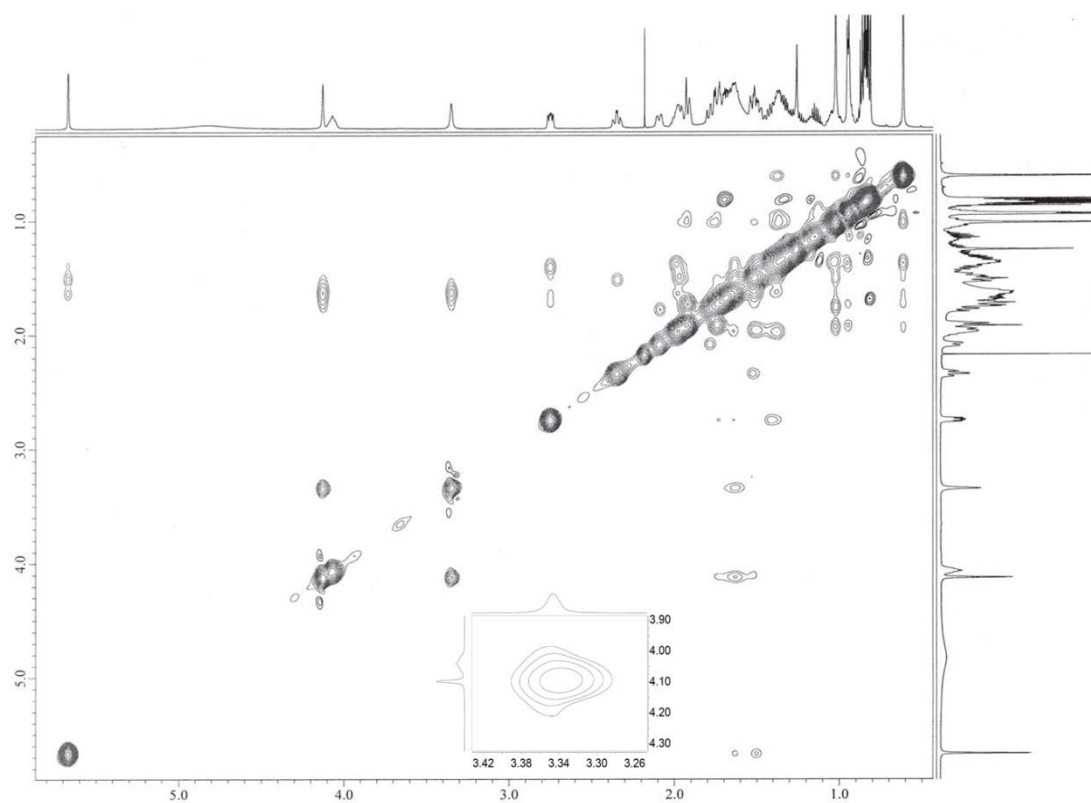


Figure S16. NOESY spectrum of **2**.

Table S6. The cartesian coordinates of conformer of compound **2** at the CAM-B3LYP/6-311+G(d,p) level of theory.

C	6.2305810	-1.6396570	0.6163380	H	7.0443720	-0.4354460	-0.9734960
C	6.9197530	-0.3659450	0.1172350	H	6.0775560	1.0369490	1.5178150
C	6.0833720	0.8735400	0.4354540	H	6.5476330	1.7652380	-0.0008870
C	4.6527430	0.7467790	-0.1018590	H	4.8416870	-1.9113270	-1.0211990
C	3.9133330	-0.5410780	0.3848590	H	4.3361900	-2.6725280	0.4762660
C	4.8022170	-1.7714030	0.0624760	H	1.8160400	2.8104100	-0.0927900
C	3.7993310	1.9943870	0.2080710	H	0.4470720	0.2183380	-1.8739620
C	2.3680720	1.8742740	-0.0745270	H	-0.3558200	-2.5972080	0.2326450
C	1.7637800	0.7018150	-0.3616830	H	0.1822250	-2.0516460	-1.3514400
C	2.5123280	-0.6476710	-0.3537570	H	1.5843870	-1.6550070	1.3361150
C	0.3468450	0.6156240	-0.8511720	H	2.1040440	-2.7180240	0.0398270
C	-0.5350890	-0.4351170	-0.1071540	H	-0.4593810	2.4618680	-0.0190630
C	0.1741860	-1.7917200	-0.2855740	H	-0.2226800	2.5279840	-1.7622000
C	1.6191700	-1.7585730	0.2498520	H	-2.3406930	1.5186580	-2.1288010
C	-0.5250940	1.8712670	-0.9398680	H	-2.6516210	1.7665400	-0.4198670
C	-1.9565030	1.3001400	-1.1279640	H	-1.7378920	-0.7330280	-1.8637430
C	-1.8821670	-0.2466420	-0.8857930	H	2.9097440	0.2620790	2.1839030
C	3.7031370	-0.4429460	1.9188000	H	3.4379350	-1.4192340	2.3330460
C	-0.7047540	-0.0792590	1.3853950	H	4.6095310	-0.1195030	2.4347230
C	-3.1950030	-0.8180830	-0.2898440	H	-1.2374510	-0.8751020	1.9173470
C	-3.1204970	-2.3251150	0.0058390	H	0.2581290	0.0626860	1.8865630
C	-4.3740290	-0.5063500	-1.2471110	H	-1.2772860	0.8438870	1.5220340
C	-5.7842360	-0.9275460	-0.7866140	H	-3.3828190	-0.2968940	0.6602140
C	-6.2862030	-0.3293570	0.5473580	H	-4.0809920	-2.7062630	0.3675820
C	-6.2972590	1.2301070	0.5650850	H	-2.8577470	-2.8921460	-0.8968100
C	-6.7589520	1.7894870	1.9210240	H	-2.3782420	-2.5567320	0.7741110
O	8.1957620	-0.1684150	0.7364680	H	-4.3826550	0.5667780	-1.4664940
O	4.3144650	3.0518070	0.5631810	H	-4.1719520	-1.0040350	-2.2079890
O	4.7190940	0.6686540	-1.5549500	H	-5.8309200	-2.0215250	-0.7047040
C	-7.5861180	-1.0383980	1.0127870	H	-6.4822810	-0.6699480	-1.5932830
C	-8.8284550	-0.9530020	0.1132270	H	-5.5457880	-0.6045360	1.3149290
C	-7.0600960	1.9179080	-0.5813810	H	-5.2443550	1.5282780	0.4531350
O	2.6782600	-1.0242650	-1.7337970	H	-7.8326900	1.6335680	2.0808980
H	6.8191310	-2.5171190	0.3163080	H	-6.5740910	2.8691580	1.9761120
H	6.2270070	-1.6245040	1.7136290	H	-6.2245750	1.3160290	2.7541620

H	8.7503770	-0.9293080	0.4995580
H	5.1537180	1.4722370	-1.8872640
H	-7.8518300	-0.6802450	2.0152950
H	-7.3339510	-2.1008830	1.1403500
H	-9.6138730	-1.6163450	0.4951960
H	-8.6128970	-1.2607870	-0.9160140
H	-9.2478760	0.0572560	0.0774890
H	-6.8922180	3.0013980	-0.5418690
H	-8.1405780	1.7547490	-0.5157230
H	-6.7297230	1.5722150	-1.5667160
H	3.3211070	-0.3840290	-2.0991460

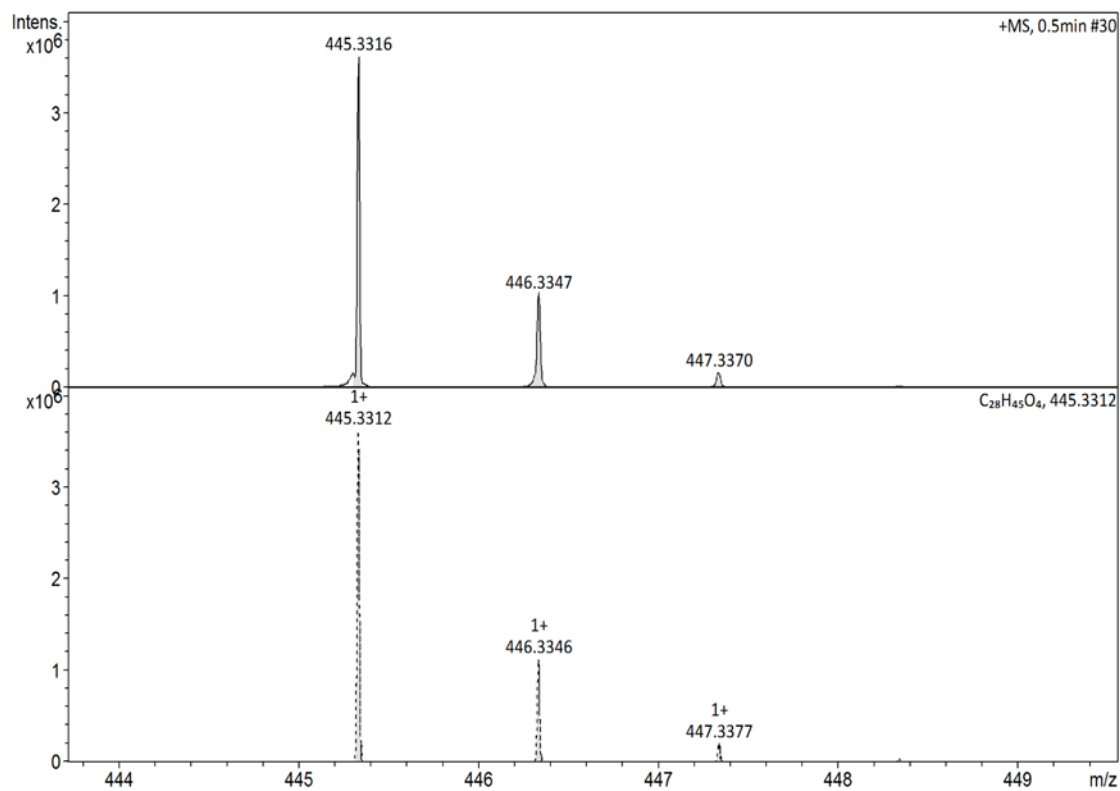


Figure S17. HRESIMS spectrum of **3**.

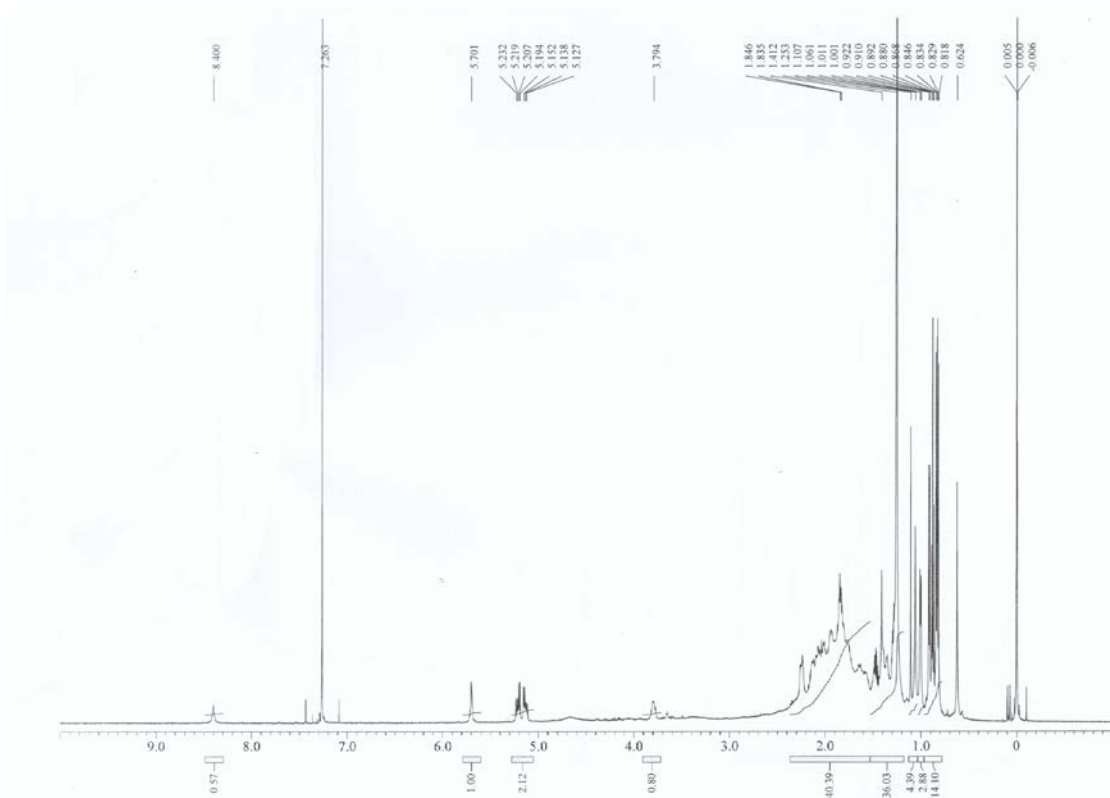


Figure S18. 1H NMR spectrum of **3**.

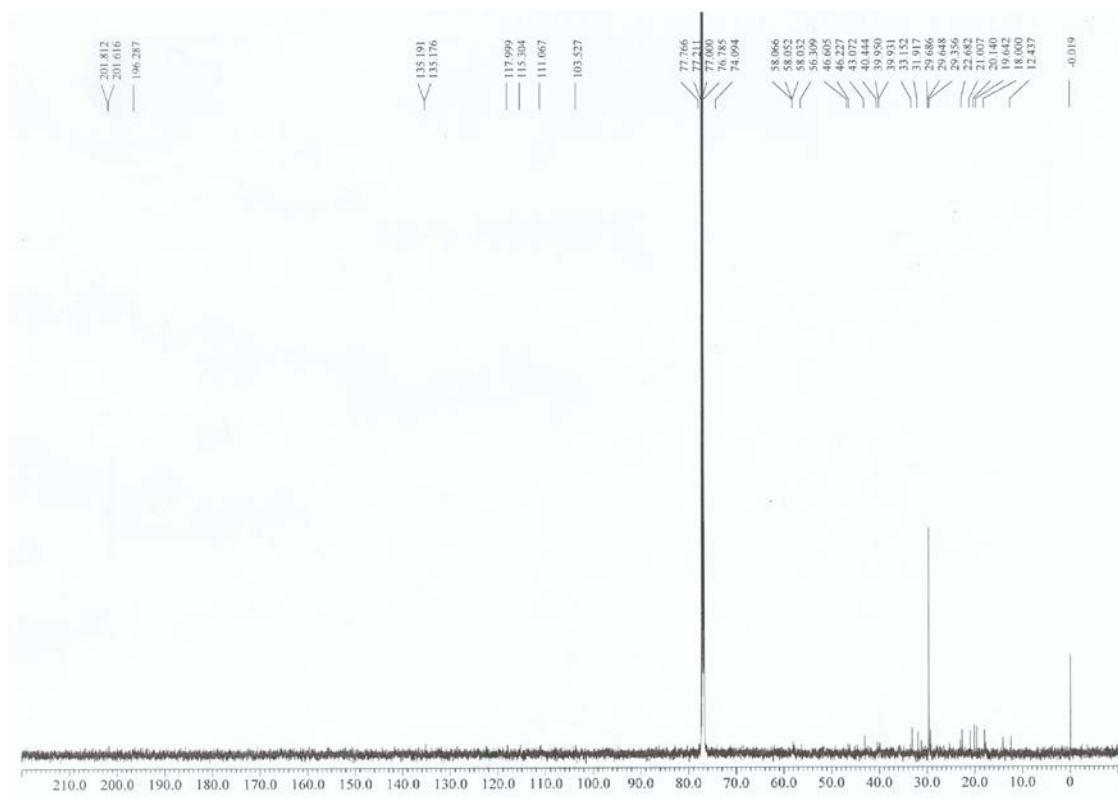


Figure S19. ¹³C NMR spectrum of **3**.

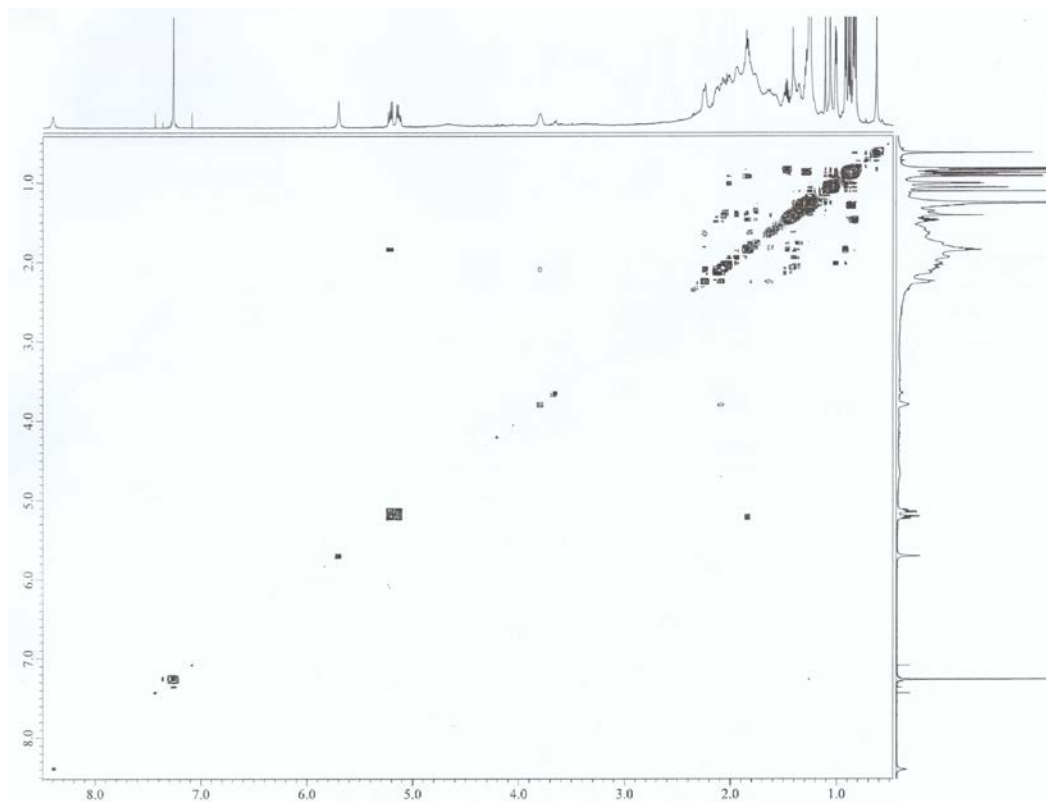


Figure S20. ¹H-¹H COSY spectrum of **3**.

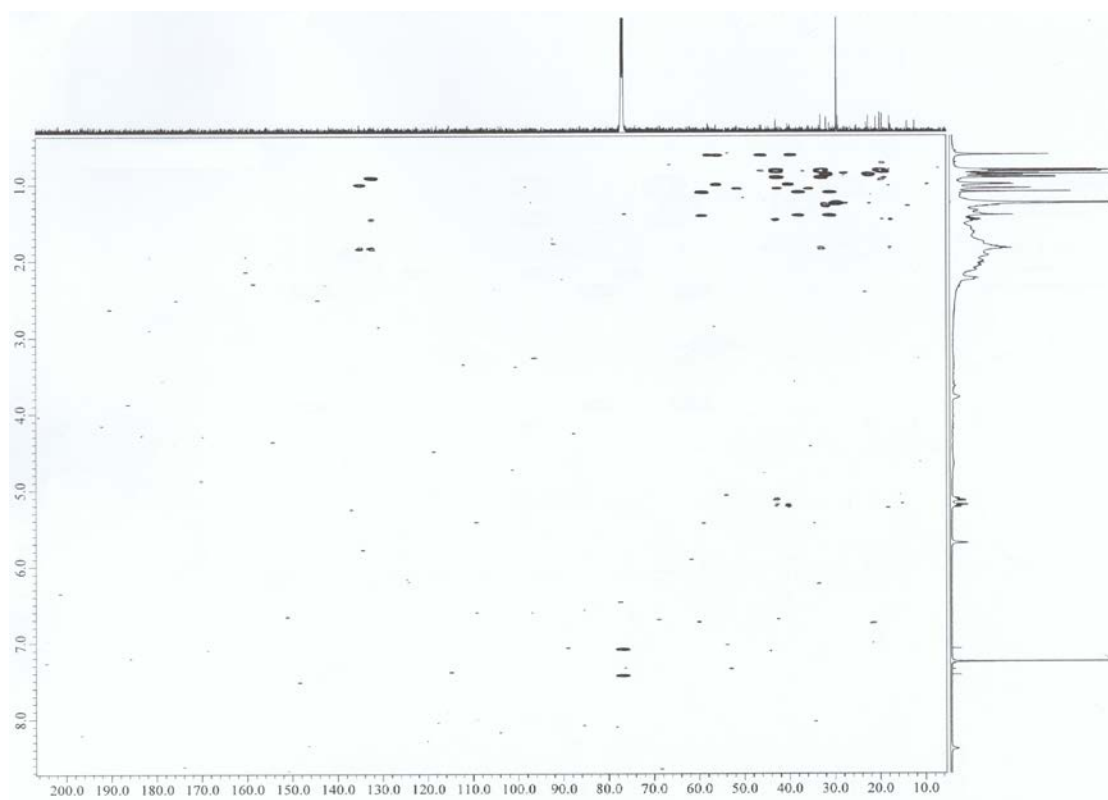


Figure S21. HMBC spectrum of **3**.

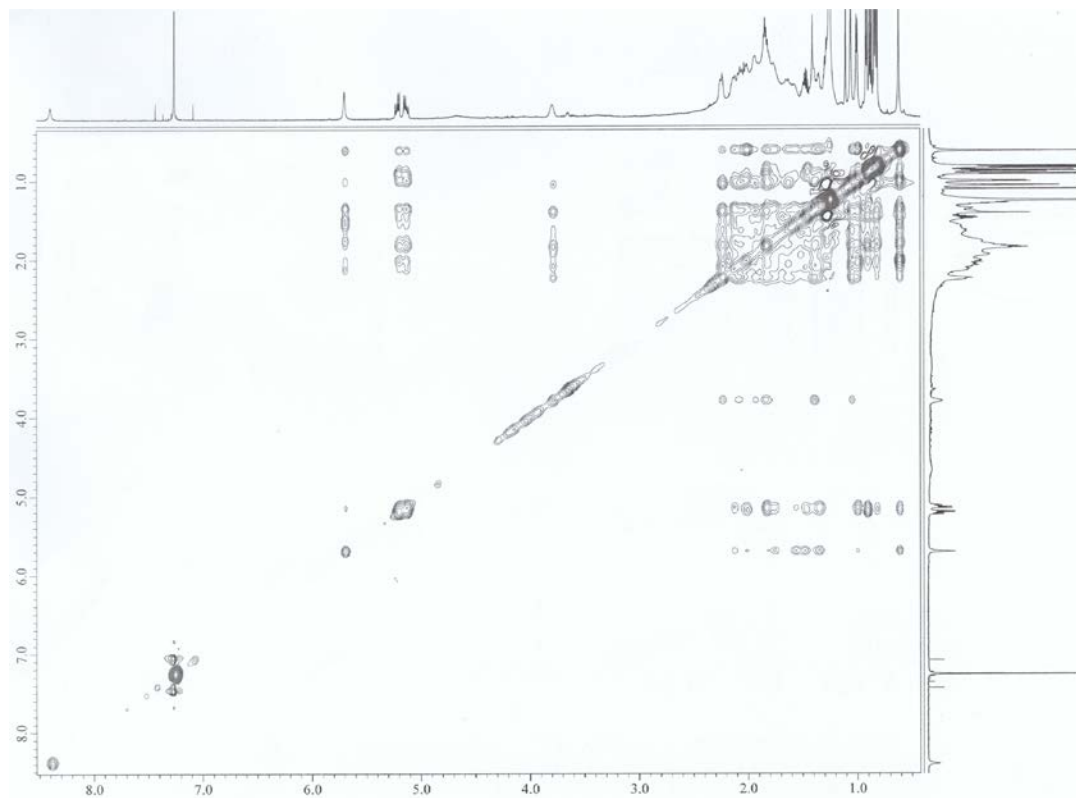


Figure S22. NOESY spectrum of **3**.

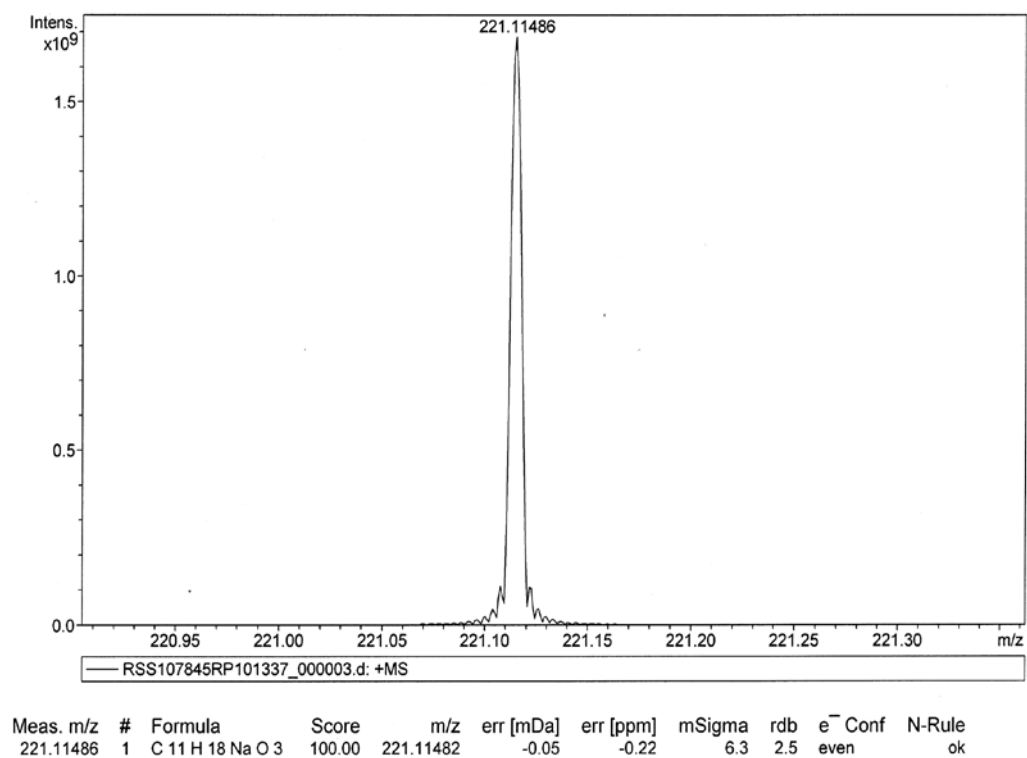


Figure S23. HRESIMS spectrum of **4**.

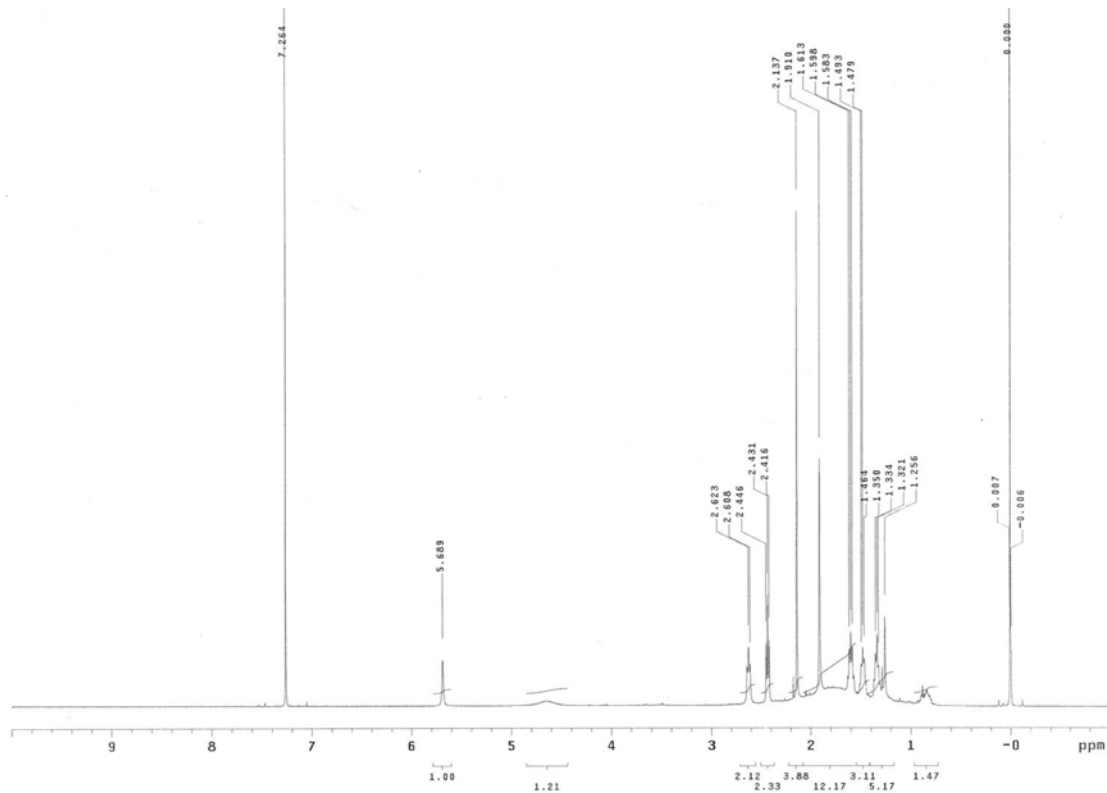


Figure S24. ¹H NMR spectrum of **4**.

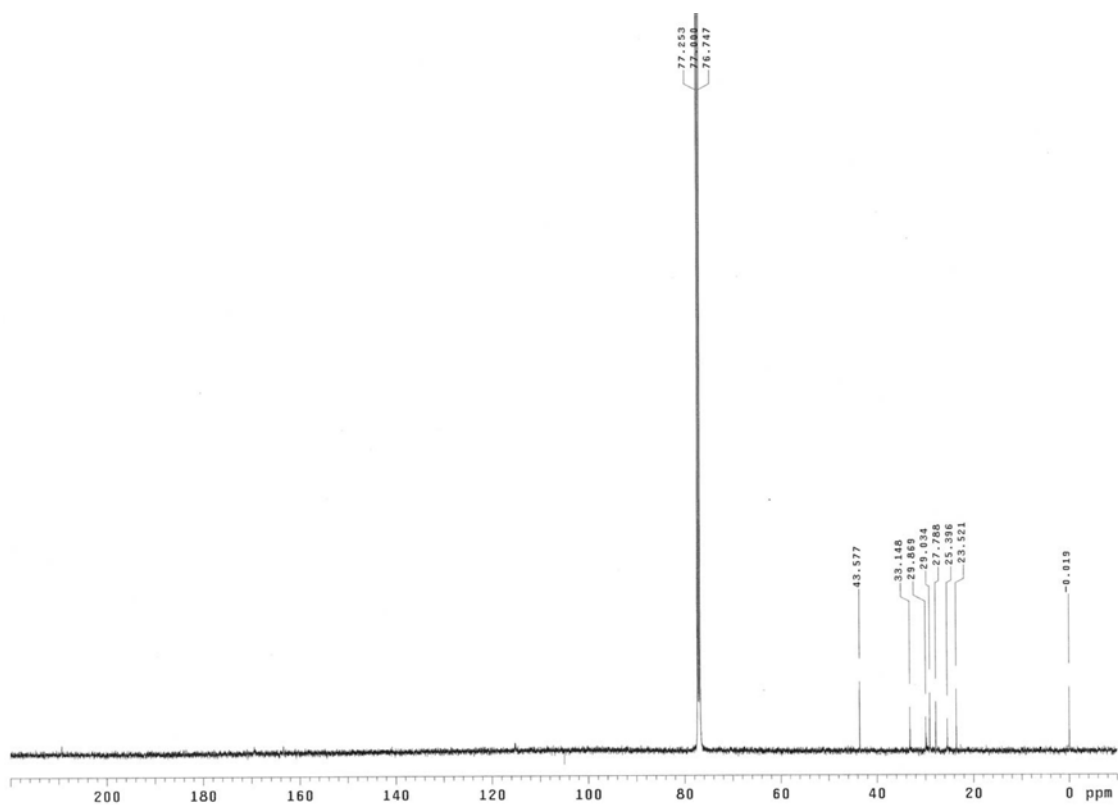


Figure S25. ¹³C NMR spectrum of **4**.

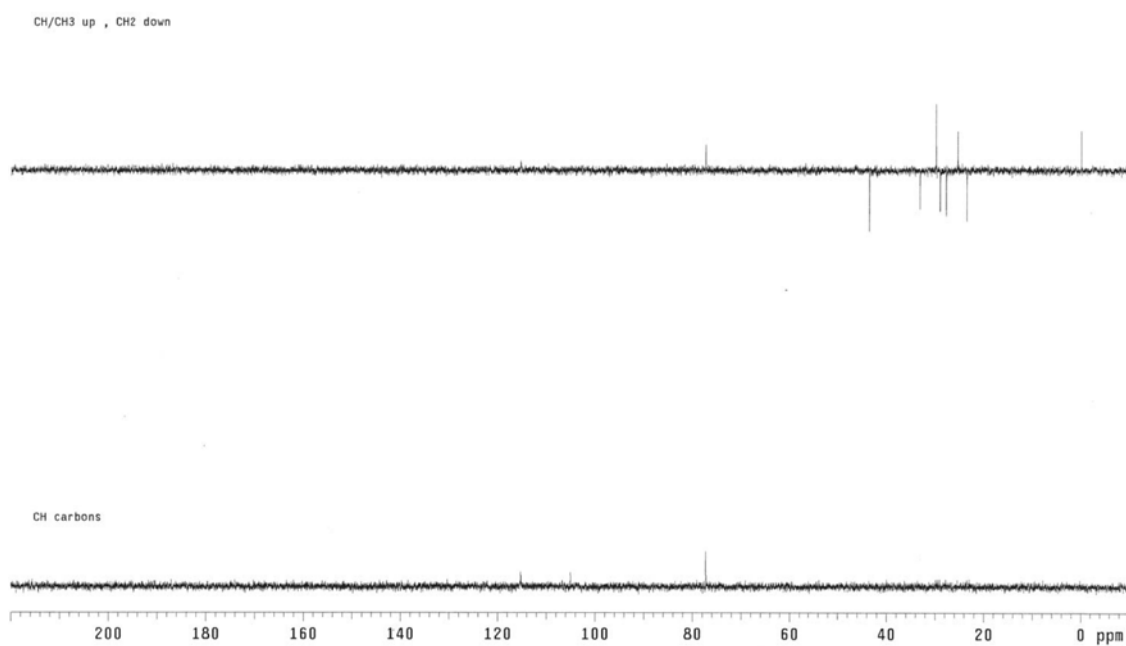


Figure S26. DEPT spectrum of **4**.

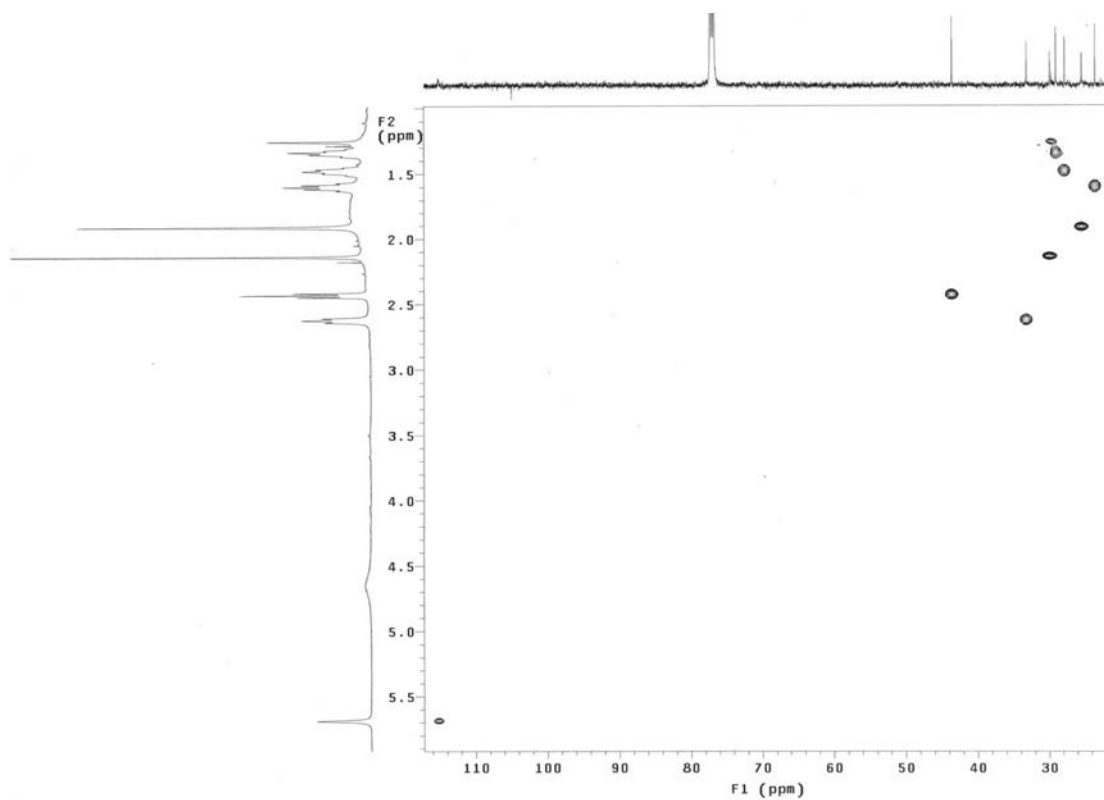


Figure S27. HSQC spectrum of **4**.

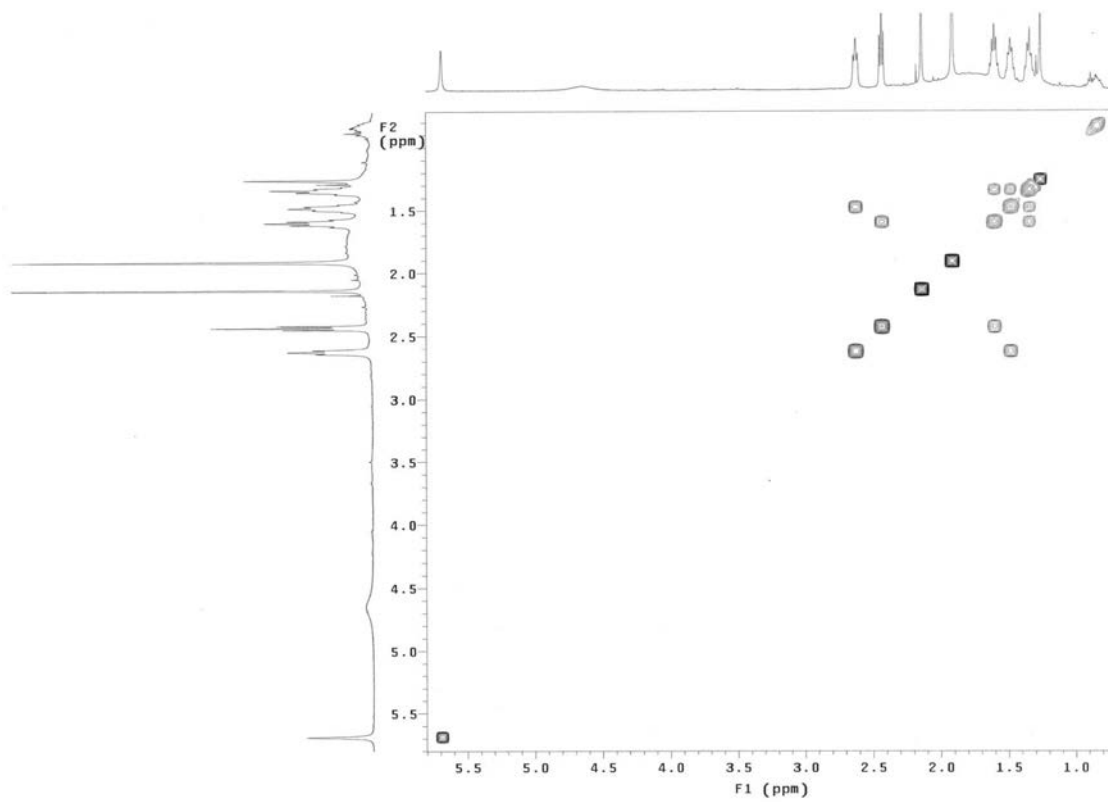


Figure S28. ^1H - ^1H COSY spectrum of **4**.

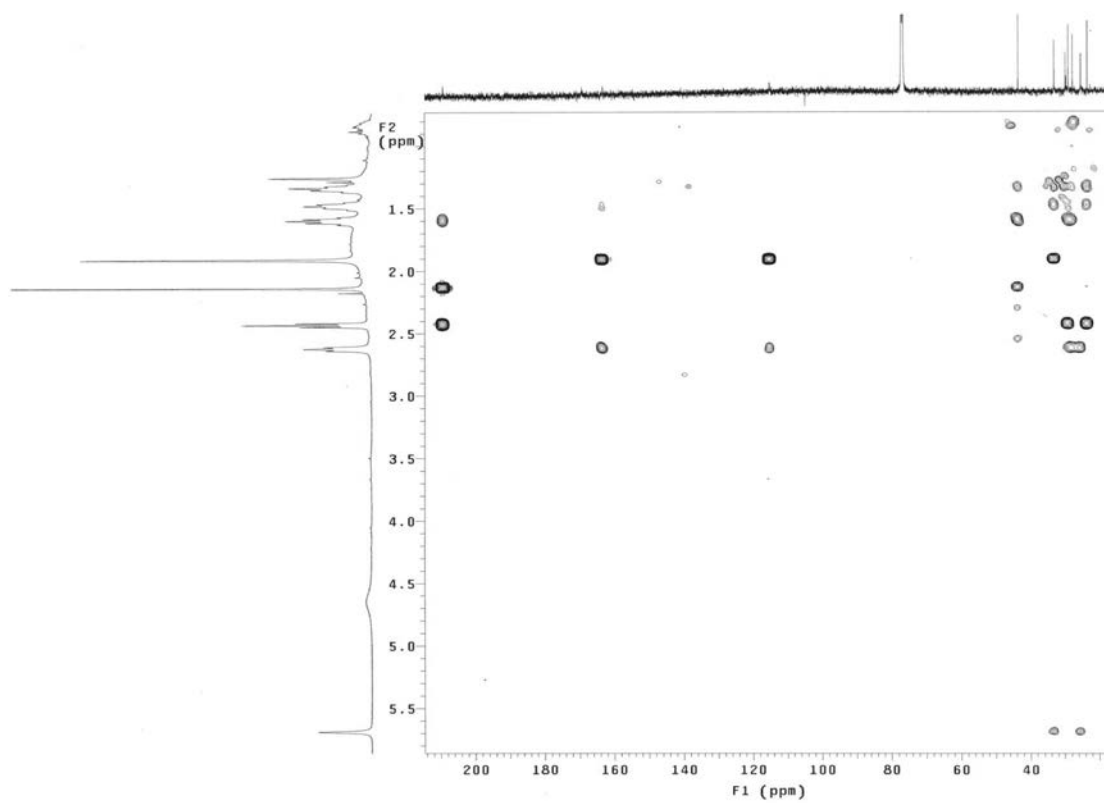


Figure S29. HMBC spectrum of **4**.

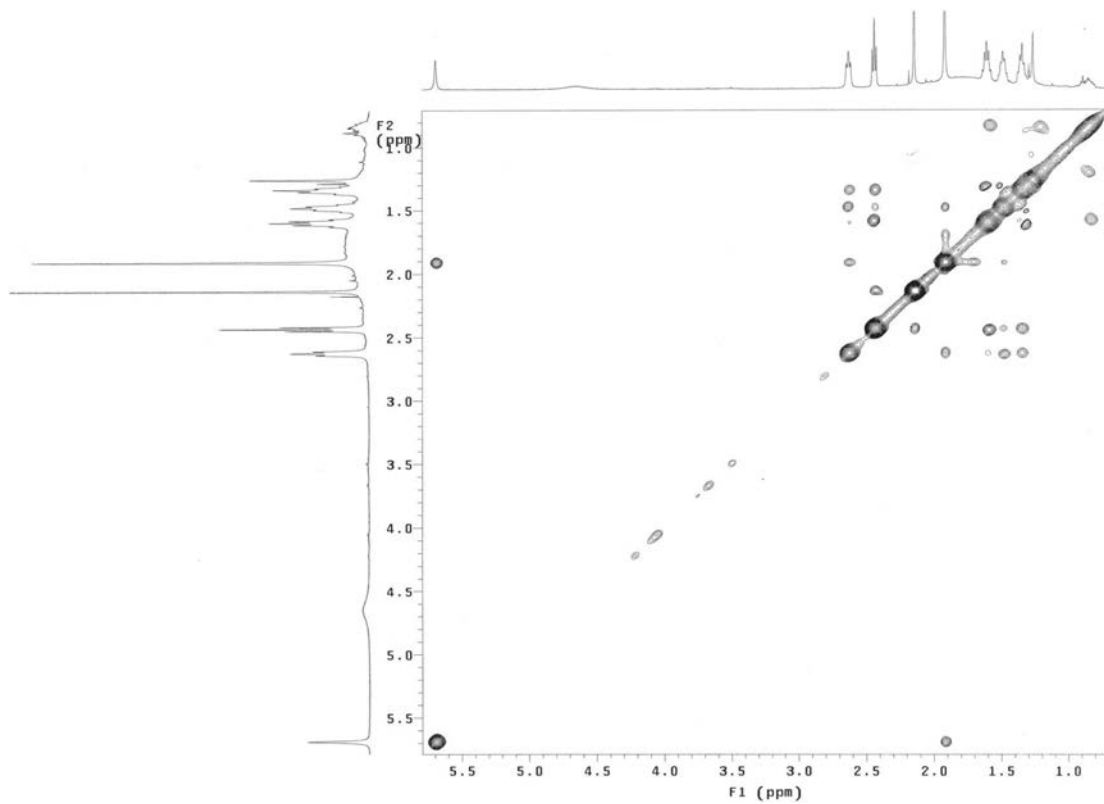


Figure S30. NOESY spectrum of **4**.

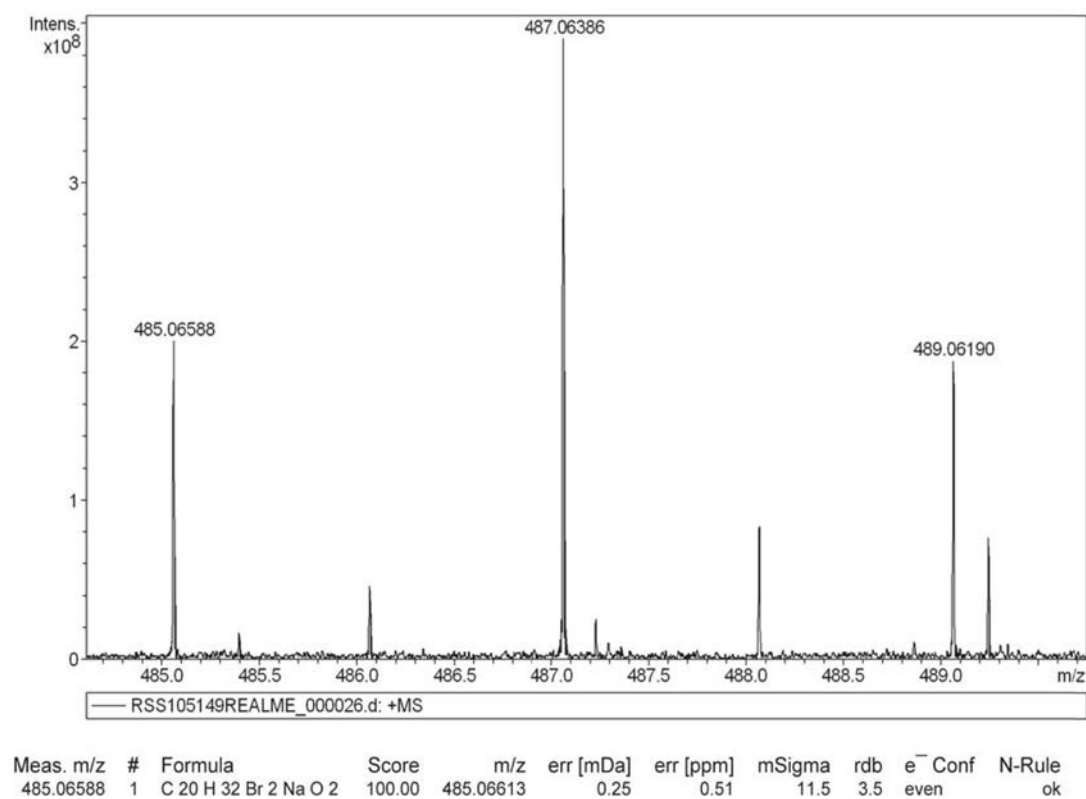


Figure S31. HRESIMS spectrum of **5**.

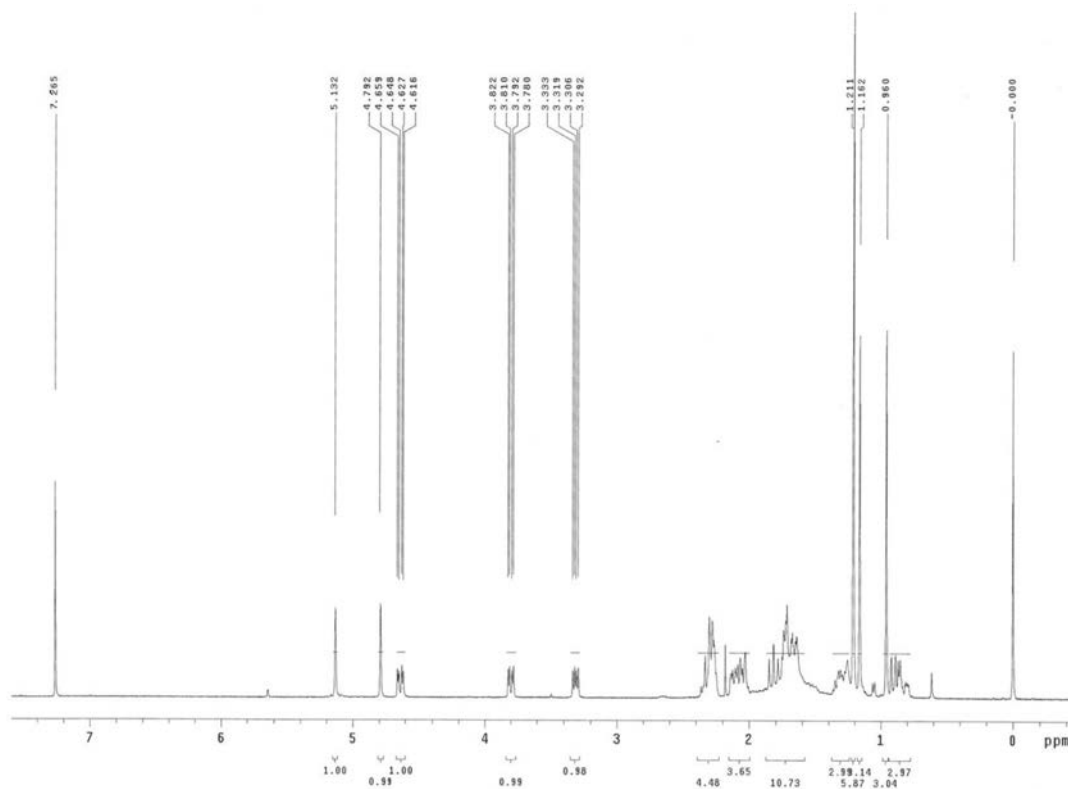


Figure S32. ¹H NMR spectrum of **5**.

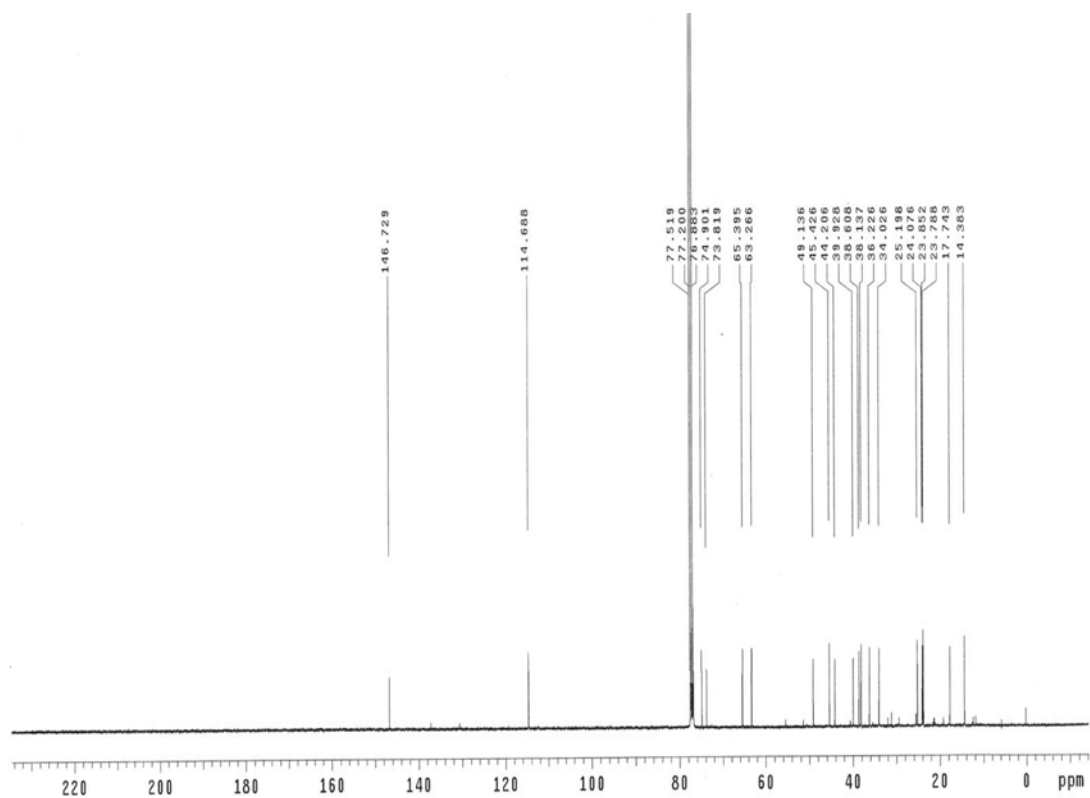


Figure S33. ^{13}C NMR spectrum of **5**.

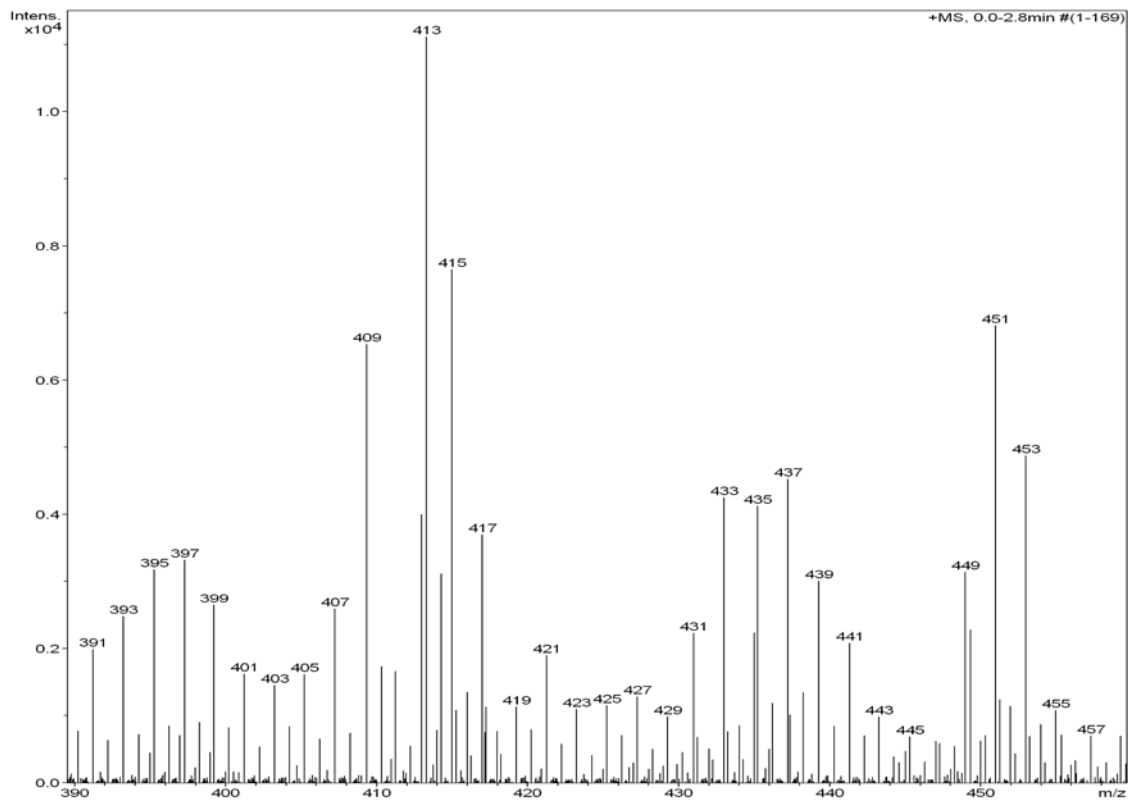


Figure S34. ESIMS spectrum of **6**.

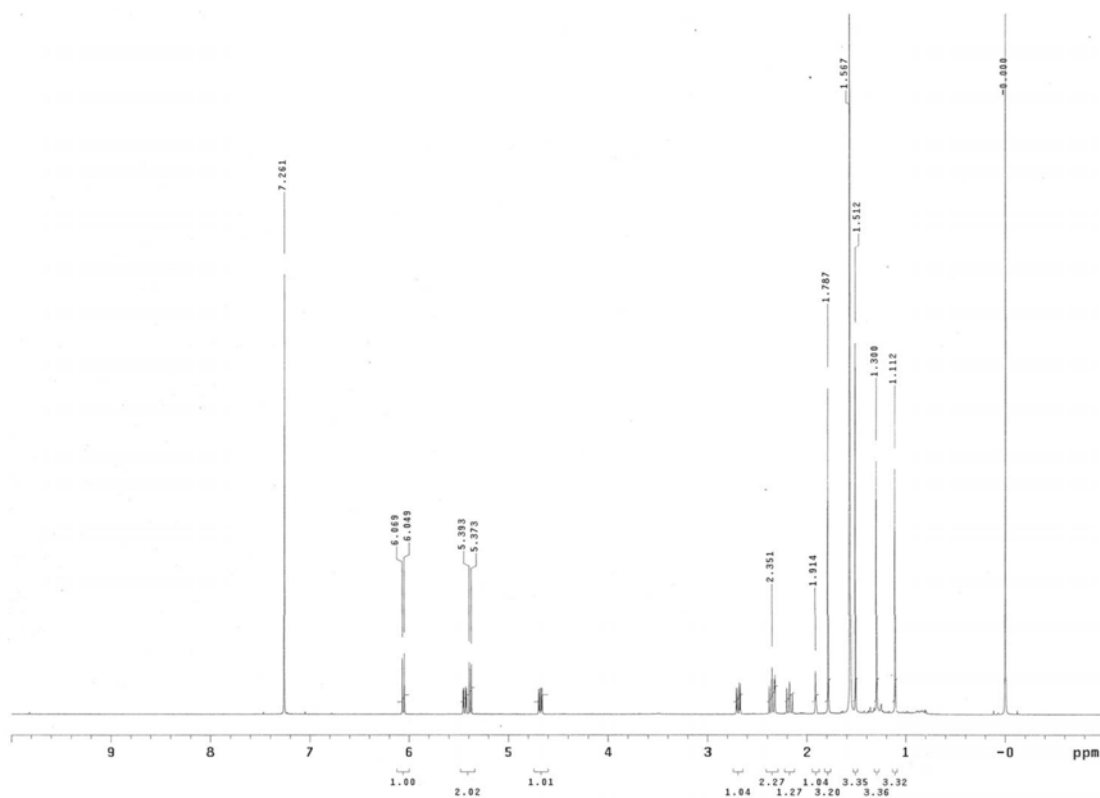


Figure S35. ¹H NMR spectrum of **6**.

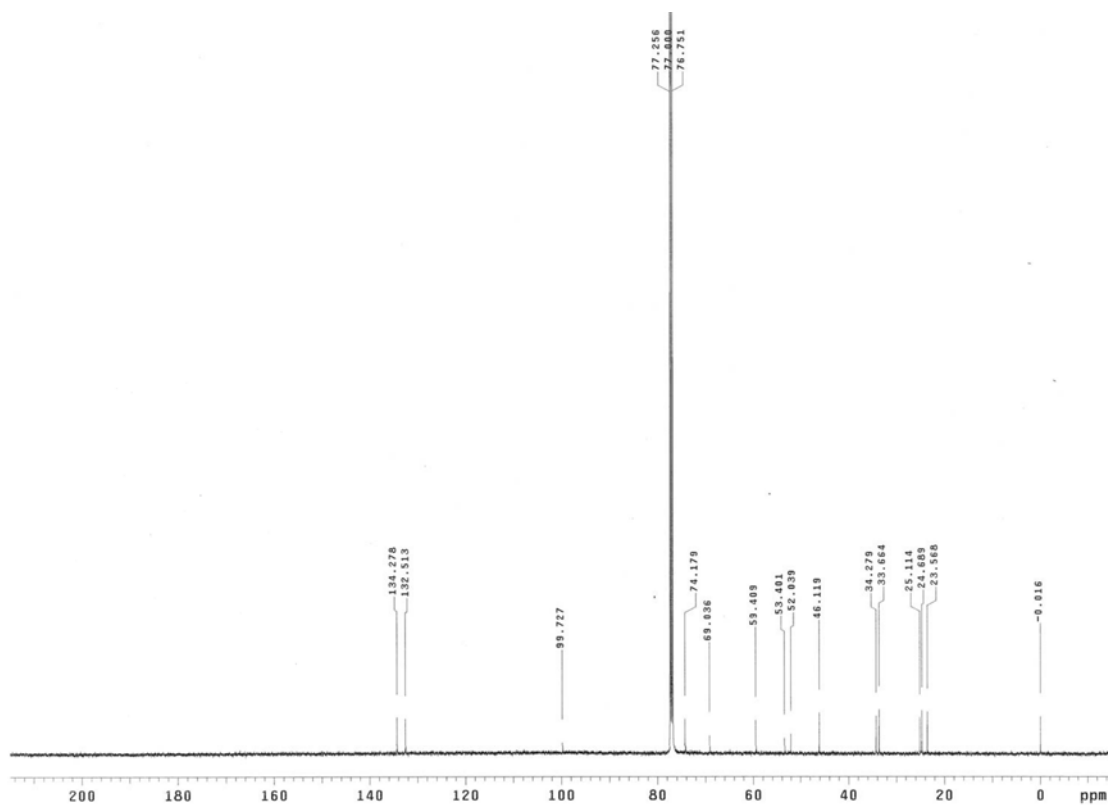


Figure S36. ¹³C NMR spectrum of **6**.

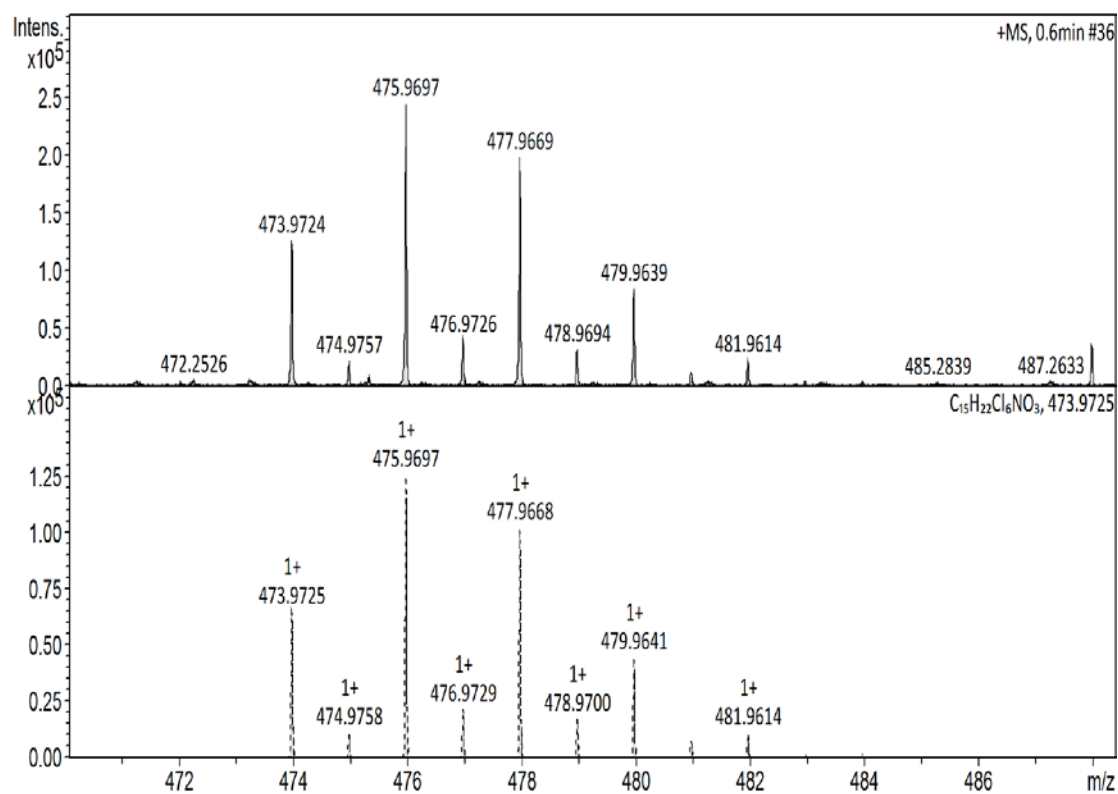


Figure S37. HRESIMS spectrum of 7.

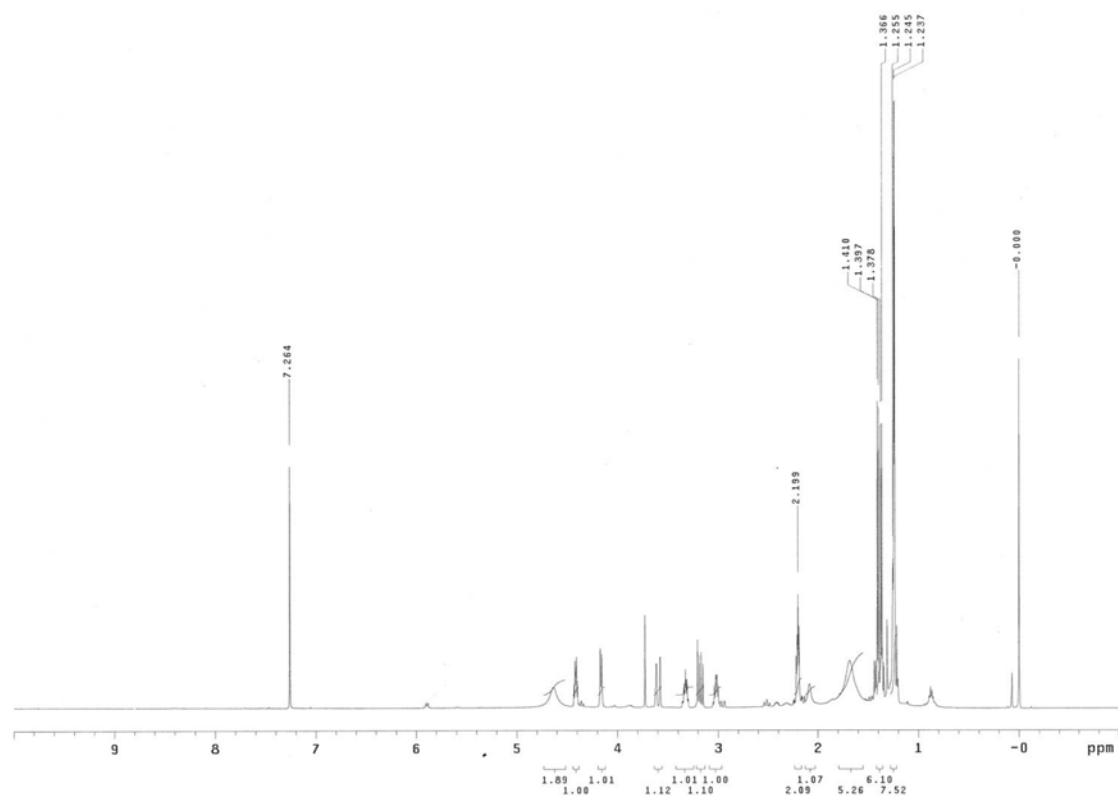


Figure S38. ¹H NMR spectrum of 7.

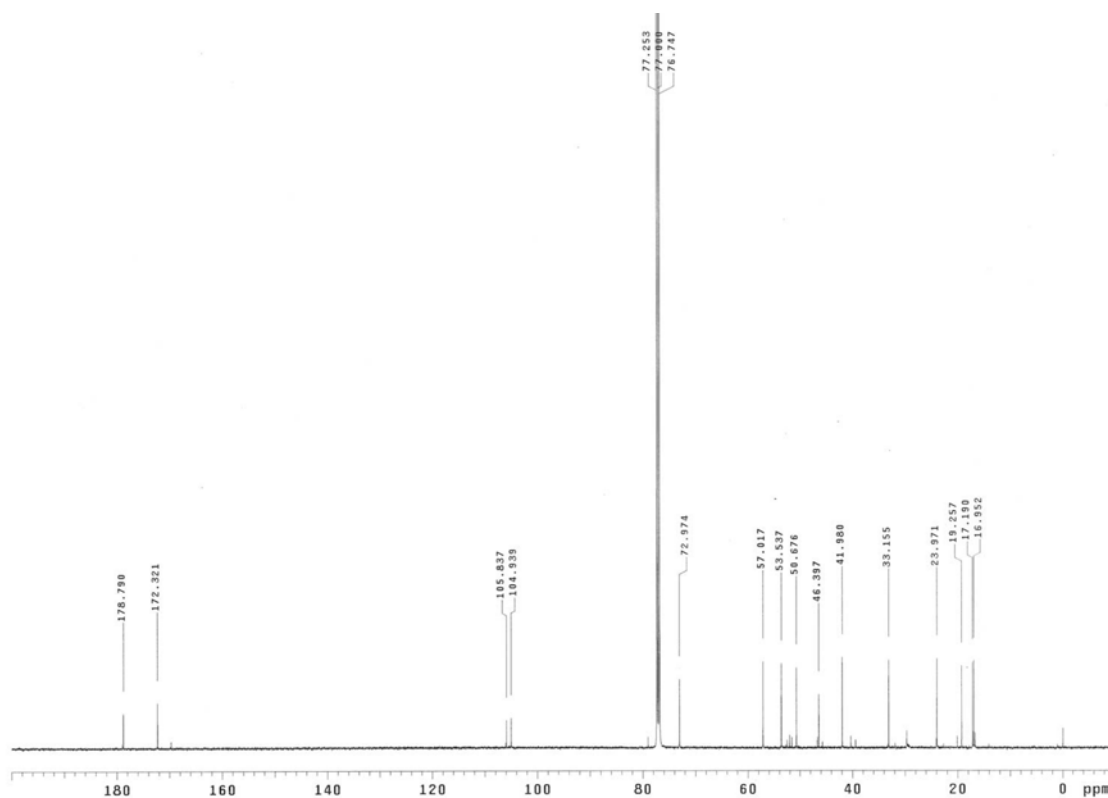
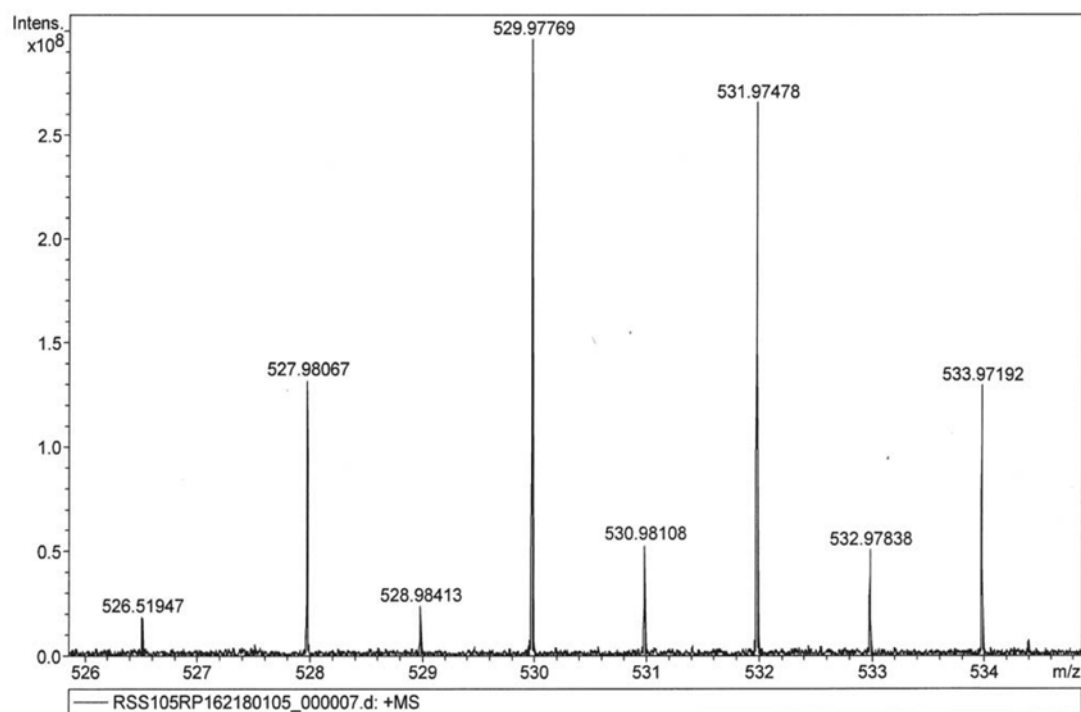


Figure S39. ^{13}C NMR spectrum of **7**.



Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	e ⁻	Conf	N-Rule
527.98067	1	C ₁₆ H ₂₅ Cl ₆ N ₄ NaO ₄	100.00	527.98070	0.02	0.05	43.8	1.5	even		ok

Figure S40. HRESIMS spectrum of **8**.

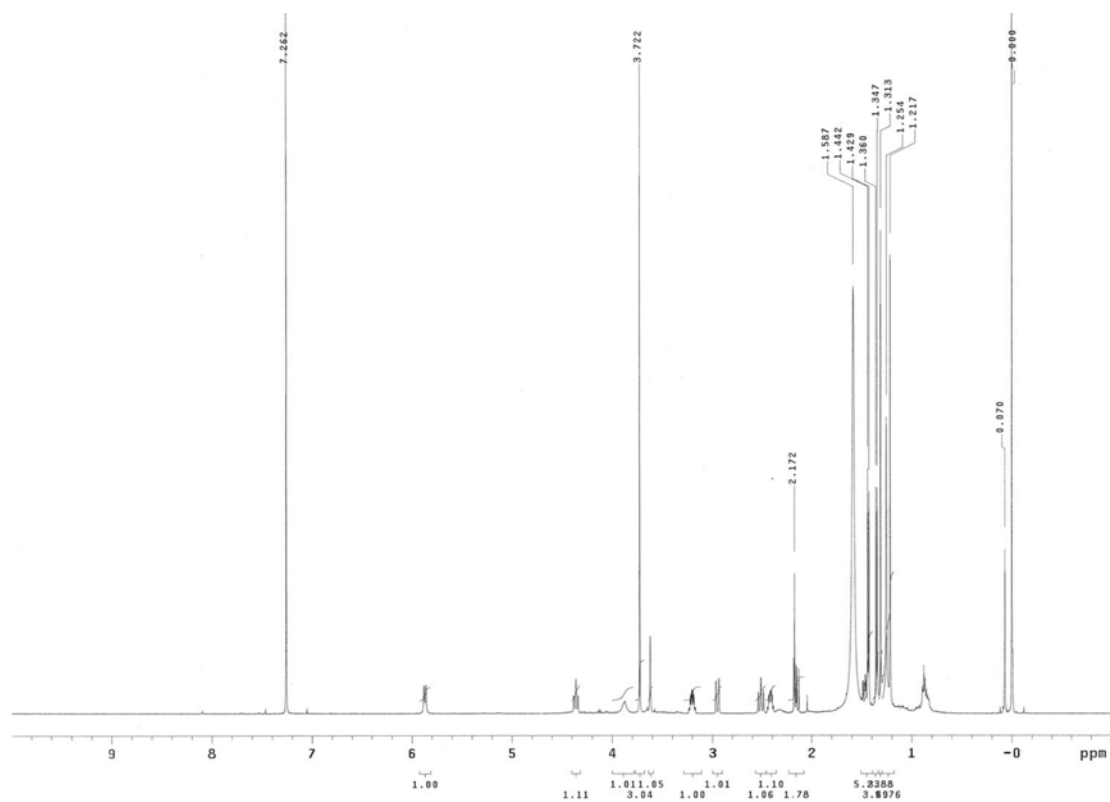


Figure S41. ¹H NMR spectrum of 8.

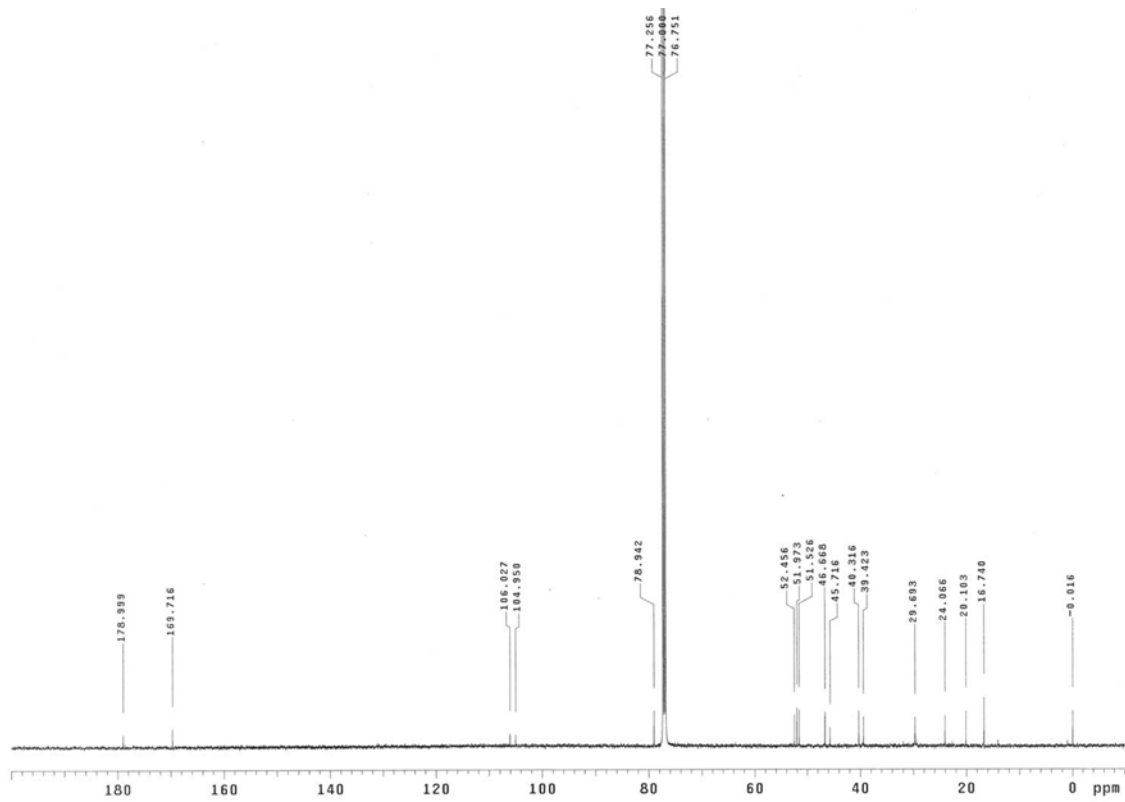


Figure S42. ¹³C NMR spectrum of 8.

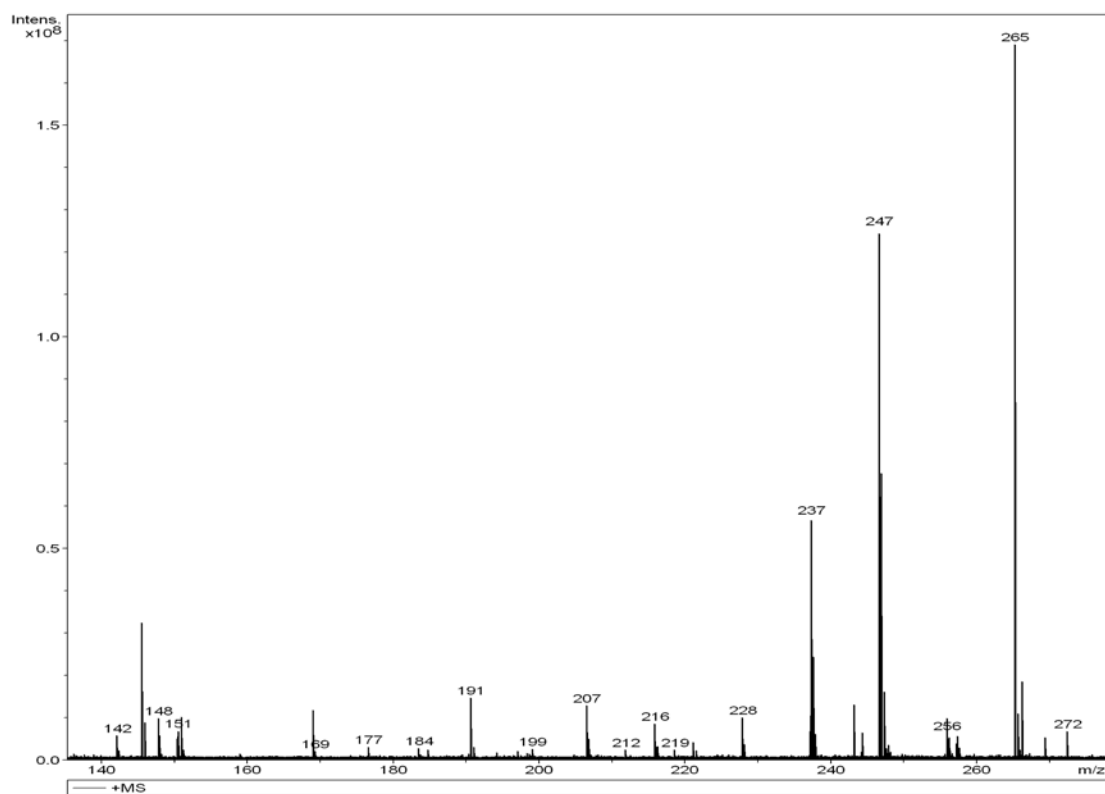


Figure S43. ESIMS spectrum of **9**.

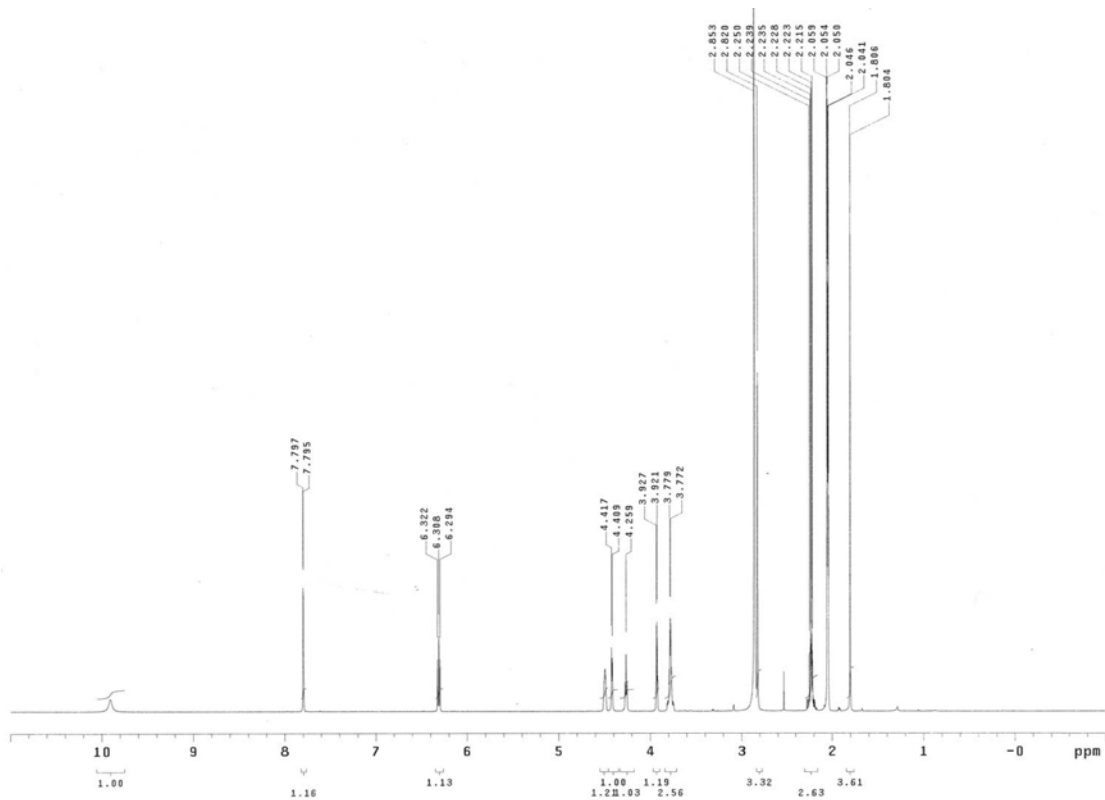


Figure S44. ¹H NMR spectrum of **9**.

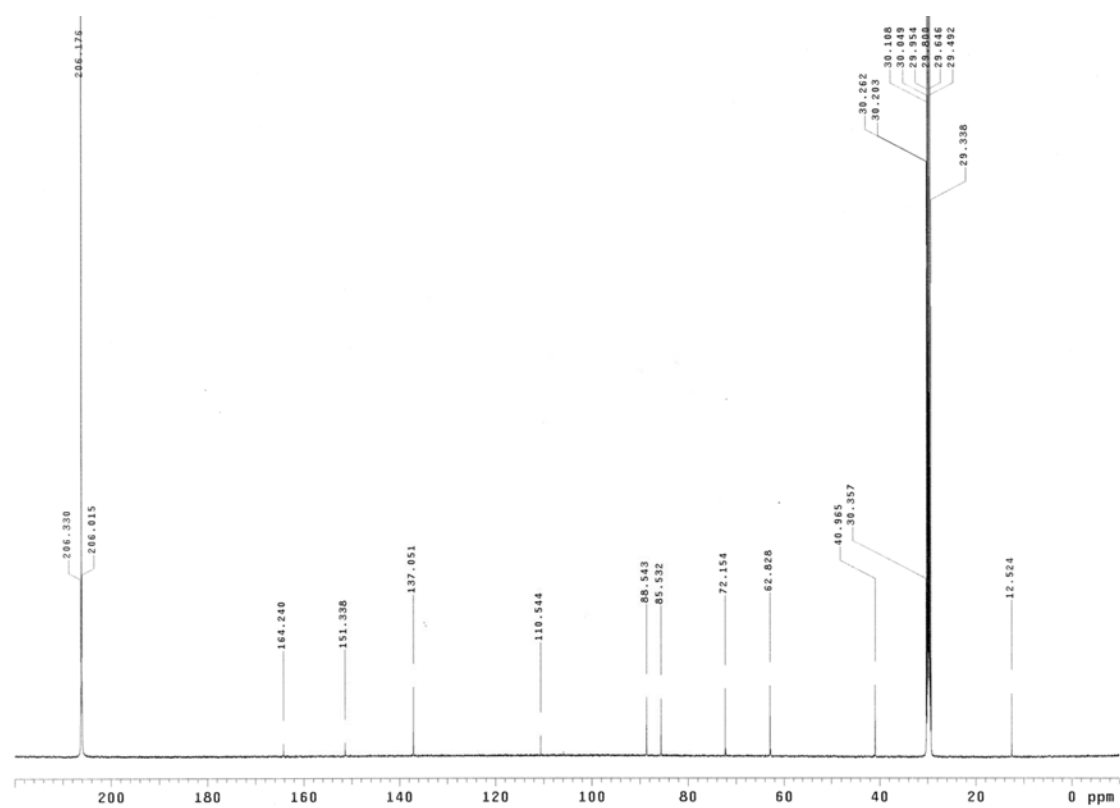


Figure S45. ¹³C NMR spectrum of **9**.