Supporting Information

Rec. Nat. Prod. X:X (202X) XX-XX

A New *ent*-Atisane Diterpenoid from the Stems of *Euphorbia royleana*

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1. General experimental procedures

Optical rotations was measured on a Rudolph Research Autopol III automatic polarimeter. IR spectrum was obtained using a Thermo-Nicolet-6700 FT-IR microscopic spectroscopy. ECD spectrum was measured on an Applied Photophysics Chirascan spectrometer. NMR spectra were measured on a Bruker AM-400 spectrometer. HRESIMS were performed on an Agilent-6210-LC/TOF mass spectrometer. TLC was performed on precoated silica gel GF 254 plates (Marine Chemical Ltd., Qingdao, China). Silica gel (200–300 mesh, Qingdao Haiyang Chemical Co., Ltd.) and Sephadex LH-20 (25−100 μm, Pharmacia Biotech) were used for column chromatography (CC).

2. Extraction, isolation, and purification

The dried and powdered plant material of *Euphorbia royleana* (5 kg) was extracted three times with 95% EtOH (30 L) at room temperature, and yield a crude extract (288 g) after evaporation of the solvent in vacuo. Then, the crude extract was suspended in warm water and partitioned with EtOAc to get an EtOAc-soluble (115 g). The EtOAc-soluble fraction was separated by silica gel CC (CH_2Cl_2-MeOH, 200:1 → 1:1) to obtain eight fractions (L1–L8). Compounds **1** (22 mg), **3** (13 mg) and **4** (30 mg) were obtained from fraction L4 (2.9 g) by repeated Sephadex LH-20 and silica gel CC. Fraction L6 (3.6 g) was subjected to silica gel column (petroleum ether-EtOAc, 10:1 → 0:1) to obtain five subfractions (L6a–L6e). Fraction L6b (330 mg) and L6c (160 mg) was subjected to repeated silica gel CC to afford compounds **2** (8 mg) and **5** (15 mg), respectively. Fraction L6e (600 mg) was purified by Sephadex LH-20 CC to obtain compound **6** (36 mg).

Table S1: Energy (298.15 K) analysis for 3*S*,5*S*,8*S*,9*S*,10*R*,12*S*,13*R*-**1**

Conf.	**G (Hartree)**	**ΔG (Kcal/mol)**	**Boltzmann Distribution**
C1	-1232.074486	1.20356418	0.115840824
C2	-1232.076404	0	0.884159176

Table S2: Calculated ECD data for 3*S*,5*S*,8*S*,9*S*,10*R*,12*S*,13*R*-**1**

State	**C1**		**C2**	
	Excitation energies(eV)	**Rotatory Strengths***	**Excitation energies(eV)**	**Rotatory Strengths***
1	3.9612	7.5584	3.9744	11.9831
2	4.3334	-2.844	4.3343	-2.9525
3	5.8226	2.9783	5.8311	0.8258
4	5.9661	-10.9452	5.987	-14.8491
5	6.5294	12.5411	6.5313	11.4832
6	6.6849	28.2399	6.7015	29.3786
7	6.8682	52.145	6.8854	62.087
8	6.8875	-57.6043	6.9107	-68.3077
9	7.0409	-2.8289	7.0359	-4.0254
10	7.1327	18.9343	7.1411	20.1411
11	7.2553	-4.0164	7.2636	-18.531
12	7.3635	-16.8892	7.3697	14.4339
13	7.4538	12.266	7.425	16.6958
14	7.5333	6.9209	7.5057	-12.3458
15	7.577	-20.6688	7.5359	-1.4285
16	7.604	-2.2861	7.5938	-3.1082
17	7.6829	4.8218	7.71	-46.2817
18	7.7052	-44.4243	7.7402	23.4139
19	7.7474	-23.4273	7.7497	-9.5952
20	7.7778	-23.7584	7.7735	-63.529
21	7.8477	-22.9562	7.8203	12.8292
22	7.8828	15.1135	7.8939	9.1881
23	7.964	27.7288	7.9725	23.5217
24	8.0034	-18.2577	8.0152	-7.1854
25	8.0324	21.1693	8.037	20.5445
26	8.0783	-10.5271	8.1092	-7.3227
27	8.1738	10.7671	8.1547	-14.132
28	8.2204	-1.4907	8.191	27.8496
29	8.2335	-35.3406	8.2825	-17.0253
30	8.2836	-15.5594	8.2939	-4.5065

Conformational analyses of **1** were carried out via Monte Carlo searching using molecular mechanism with MMFF force field in the *Spartan 18* program with an energy cutoff of 5.0 Kcal/mol. The results showed three lowest energy conformers for **1**. These conformers were re-optimized using DFT at the b3lyp/6-31g(d) level in gas phase using the Gaussian 09 program. Two conformers of **1** (Figure S1) whose relative Gibbs free energies in the range of 0−1.5 Kcal/mol were refined and considered for next step. All the re-optimized conformers mentioned above were applied for theoretical ECD calculation.

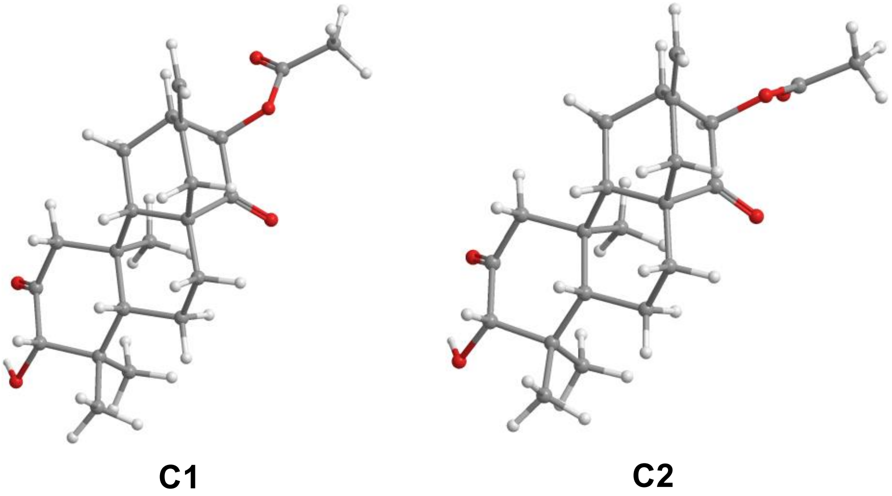
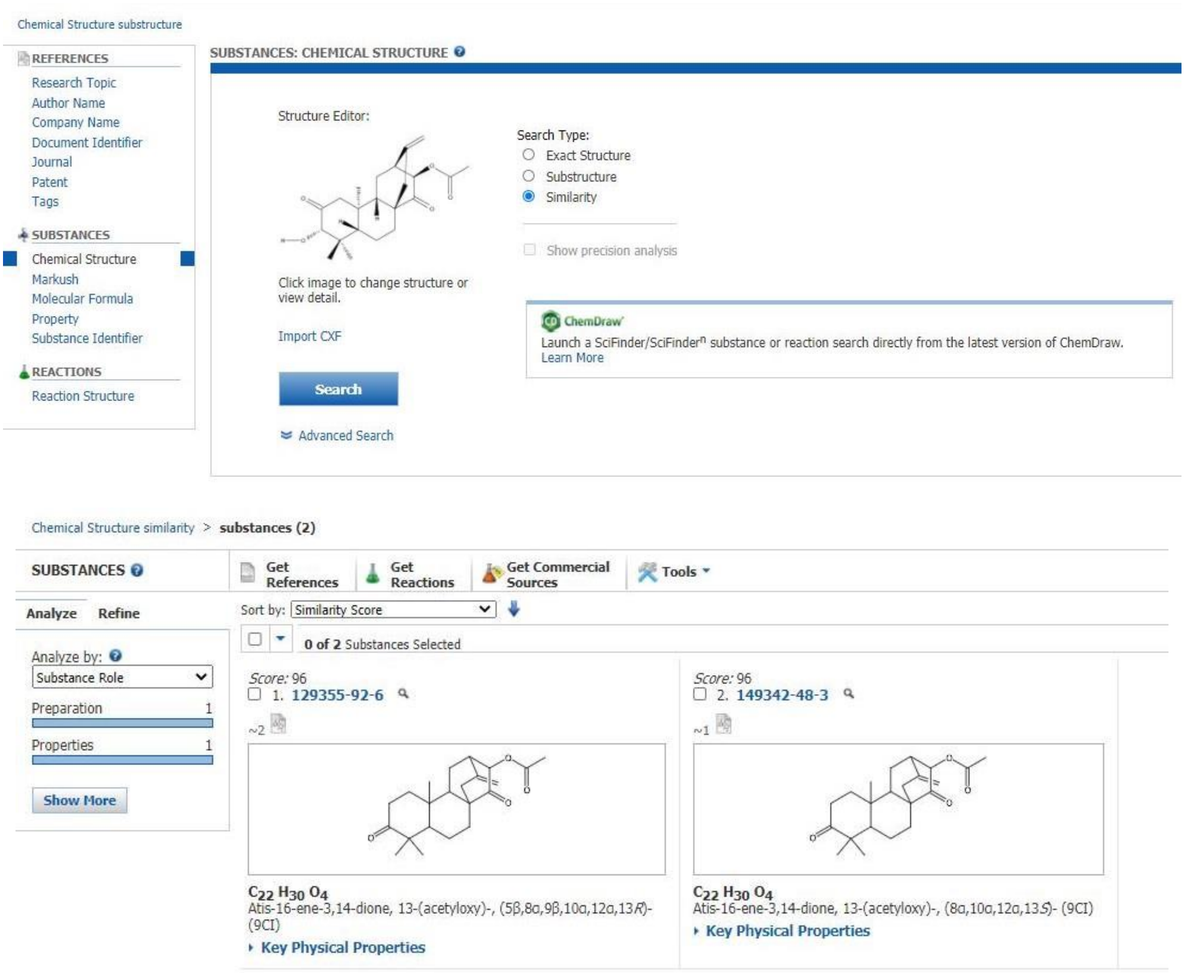


Figure S1: Optimized lowest energy conformers for 3*S*,5*S*,8*S*,9*S*,10*R*,12*S*,13*R*-**1**



Chemical Structure substructure

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Research Topic
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Company Name
Document Identifier
Journal
Patent
Tags

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Substance Identifier

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Score: 96
☐ 1. 129355-92-6
~2
$C_{22}H_{30}O_4$
Atis-16-ene-3,14-dione, 13-(acetyloxy)-, (5β,8α,9β,10α,12α,13*R*)-(9CI)
Key Physical Properties

Score: 96
☐ 2. 149342-48-3
~1
$C_{22}H_{30}O_4$
Atis-16-ene-3,14-dione, 13-(acetyloxy)-, (8α,10α,12α,13*S*)- (9CI)
Key Physical Properties

Figure S2: Scifinder search of new compound **1**

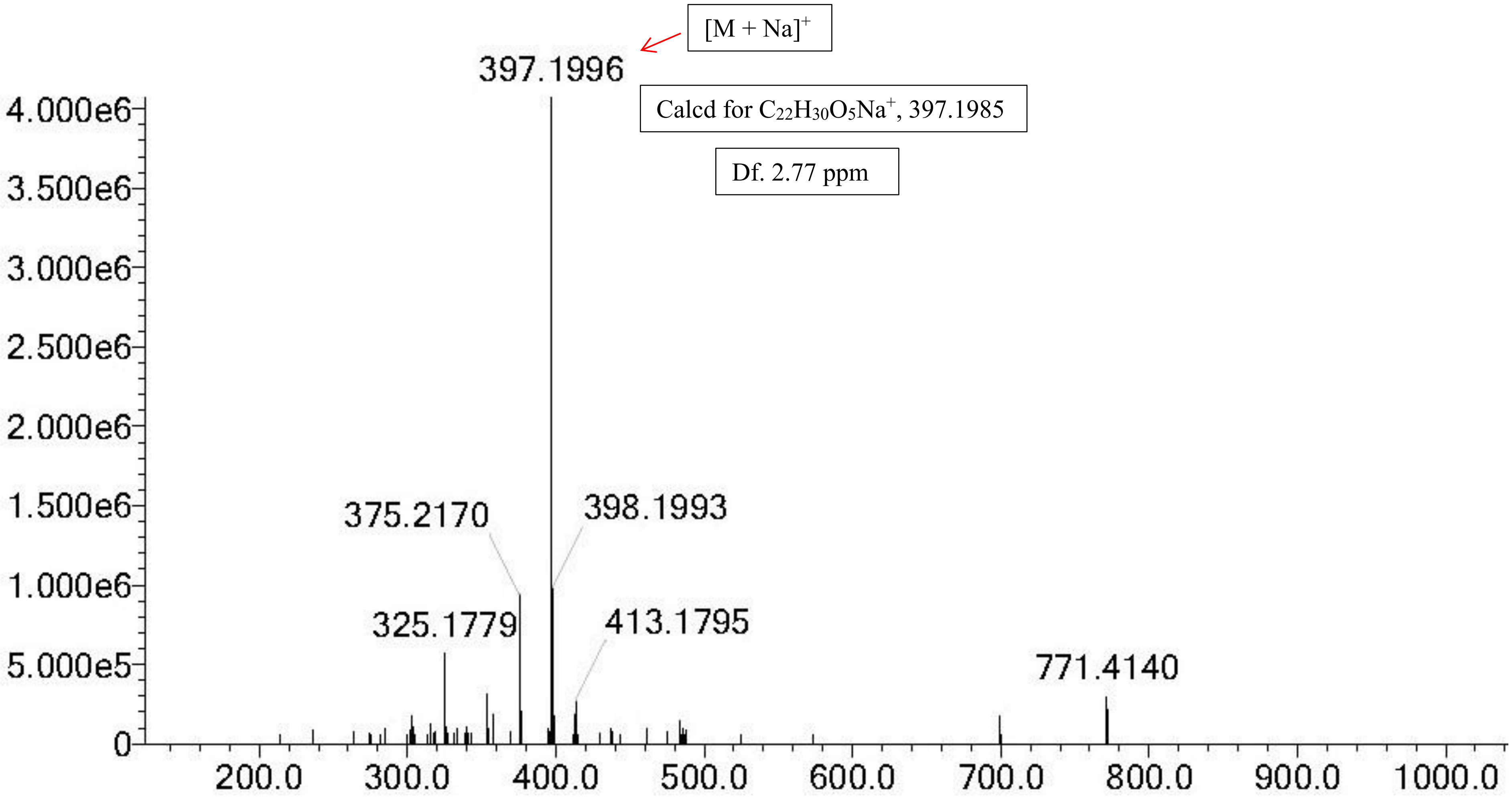

Figure S3: HR-ESI-MS spectrum of **1**

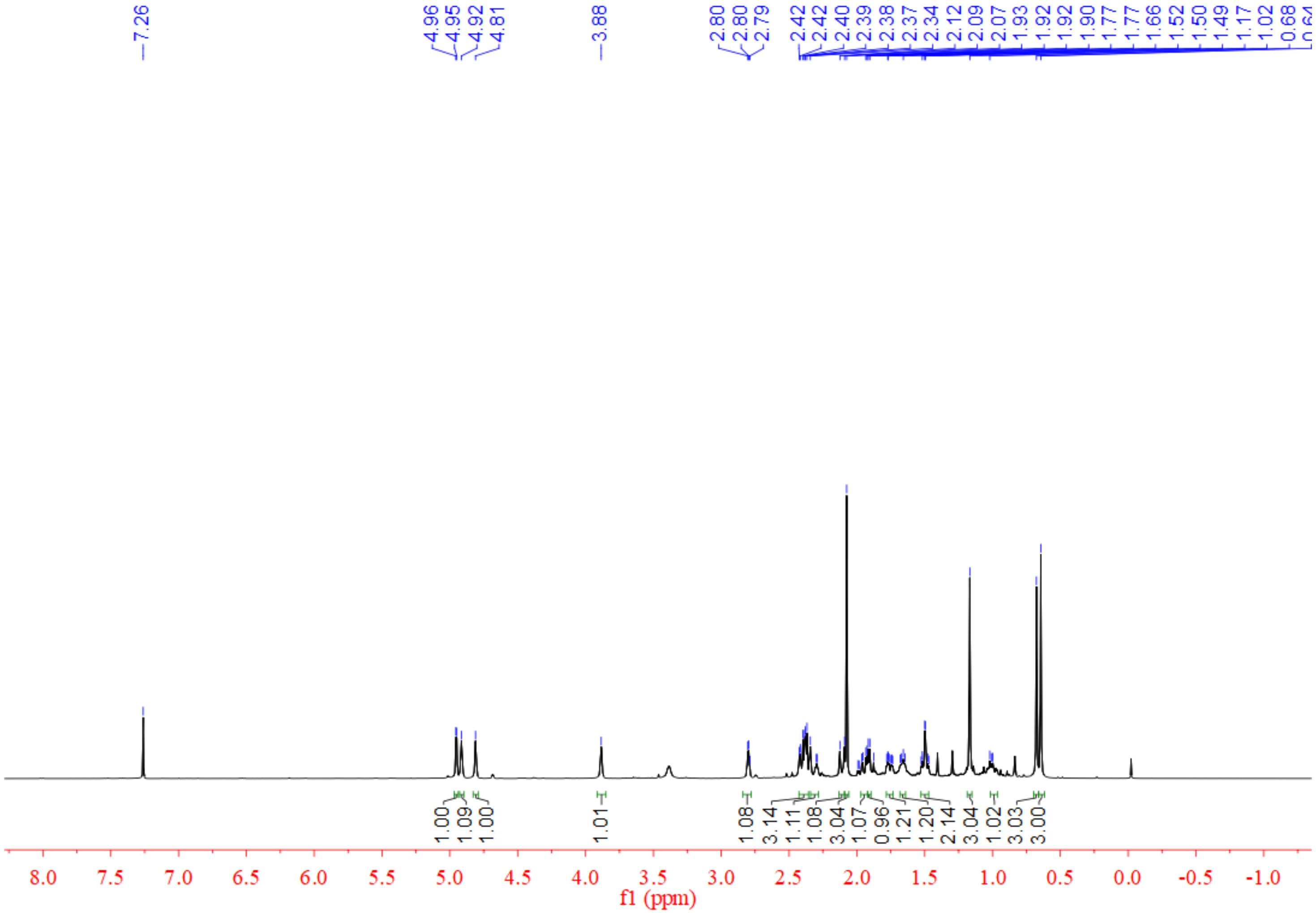

Figure S4: ^{1}H-NMR (400 MHz, $CDCl_3$) spectrum of **1**

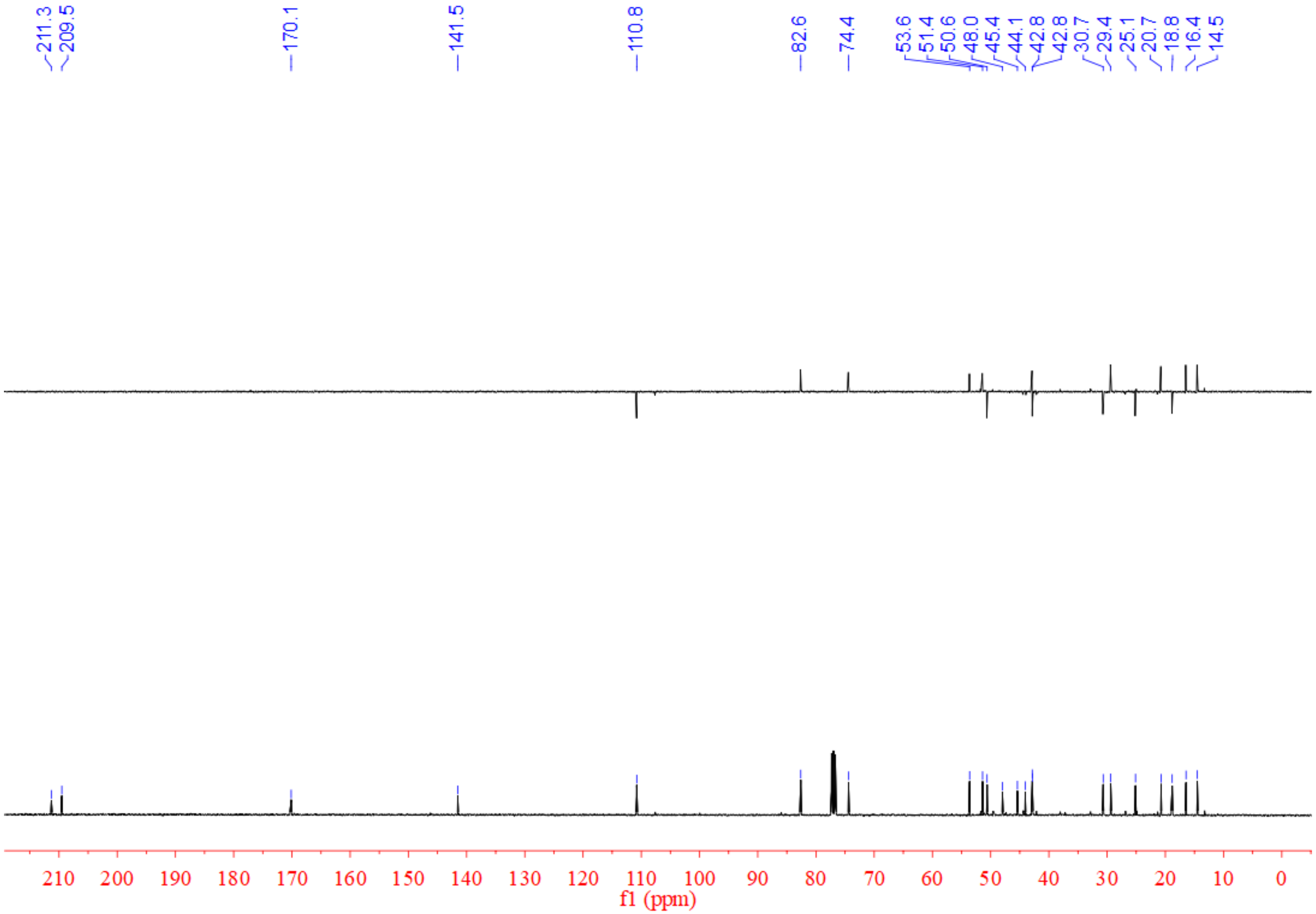

Figure S5: ^{13}C-NMR and DEPT 135 (100 MHz, $CDCl_3$) spectrum of **1**

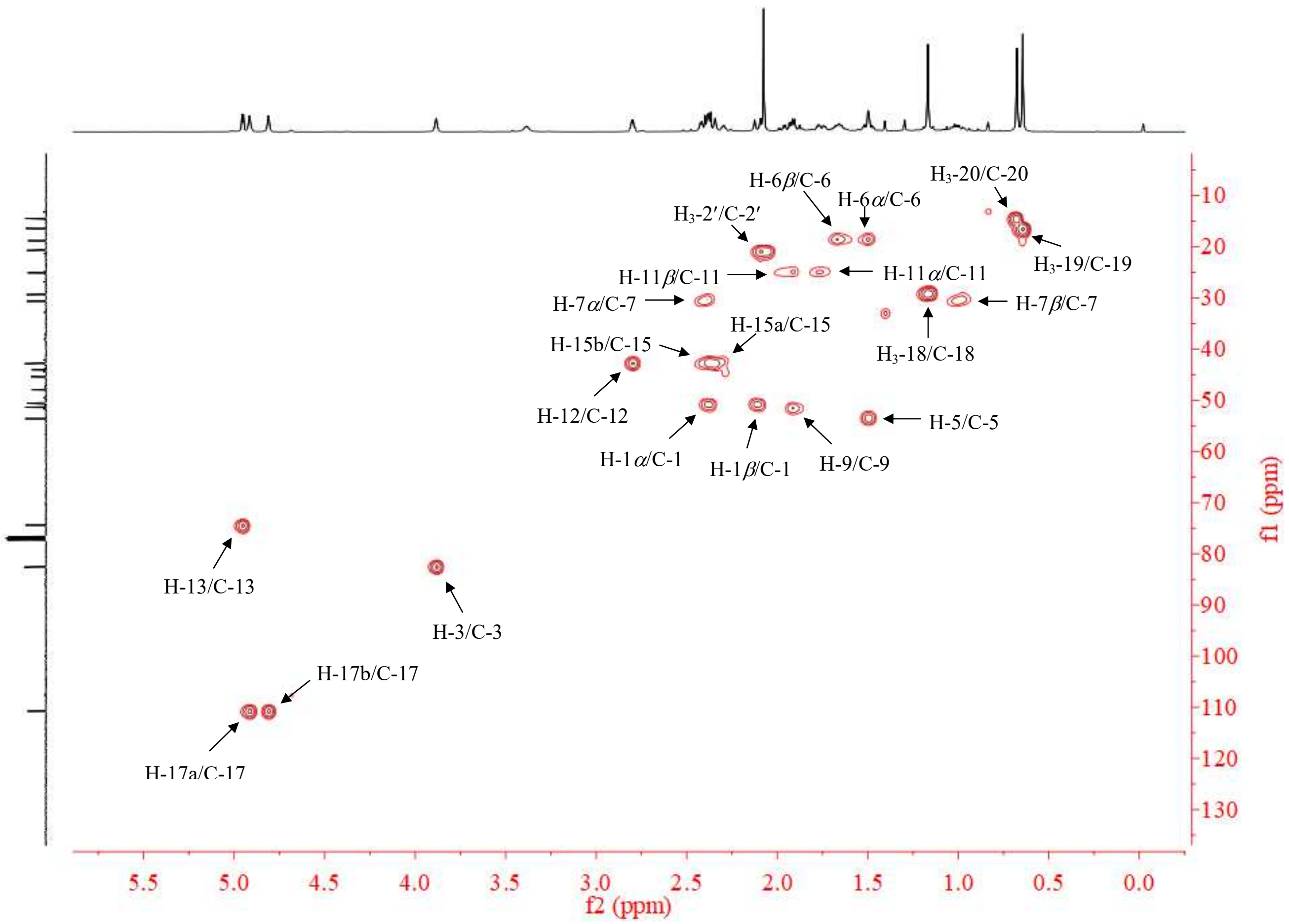

Figure S6: HSQC spectrum of **1**

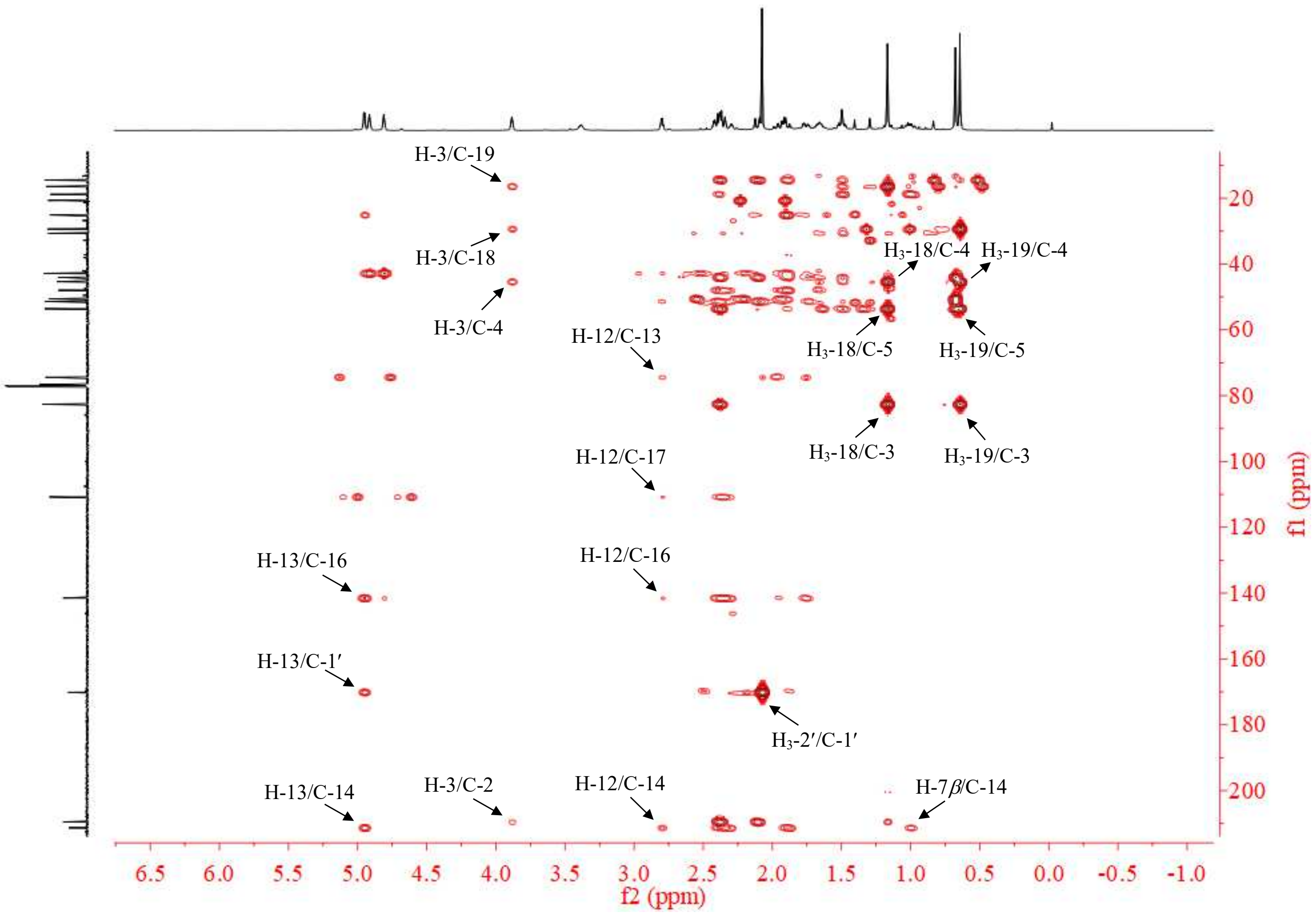

Figure S7: HMBC spectrum of **1**

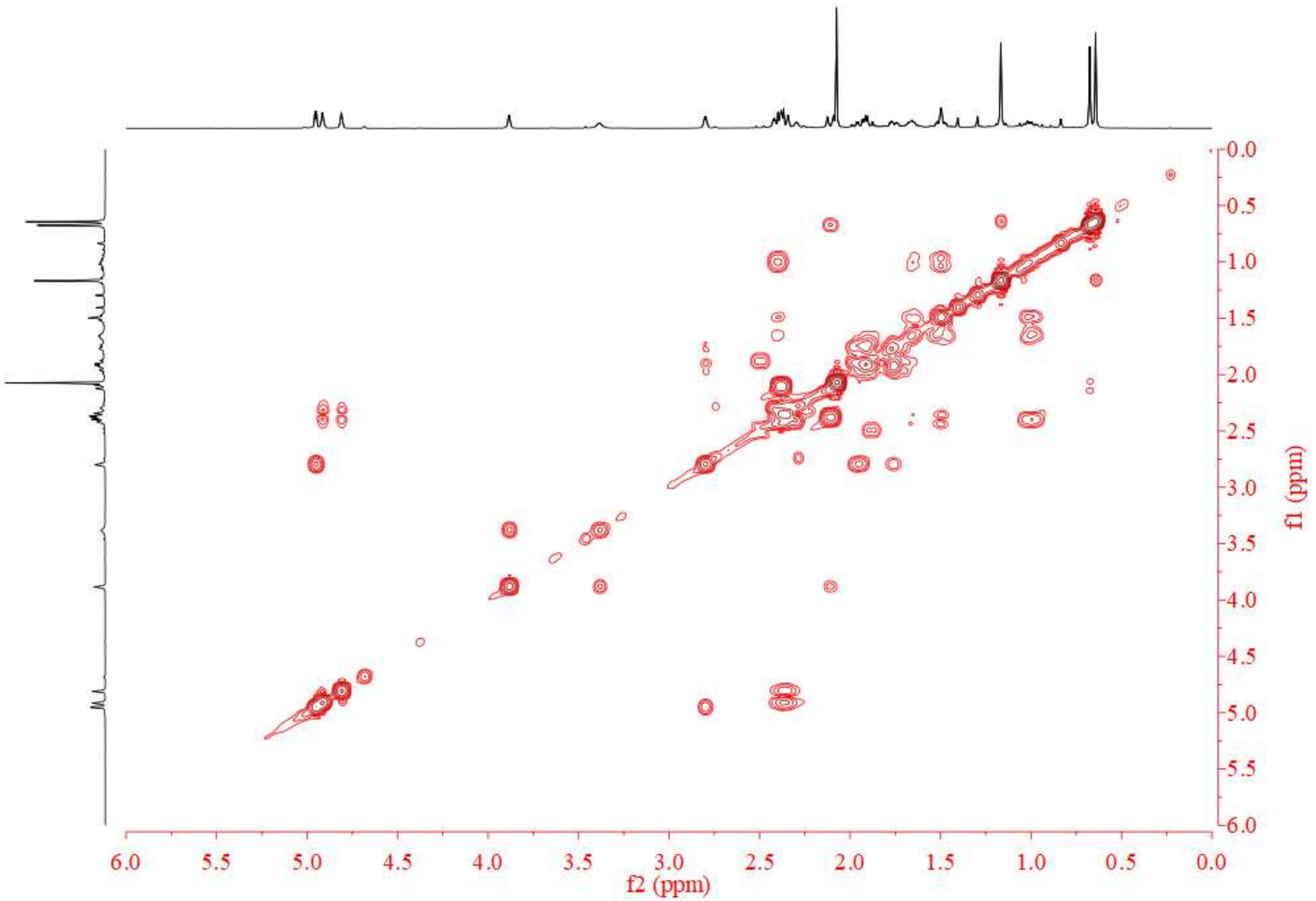

Figure S8: ^{1}H−^{1}H COSY spectrum of **1**

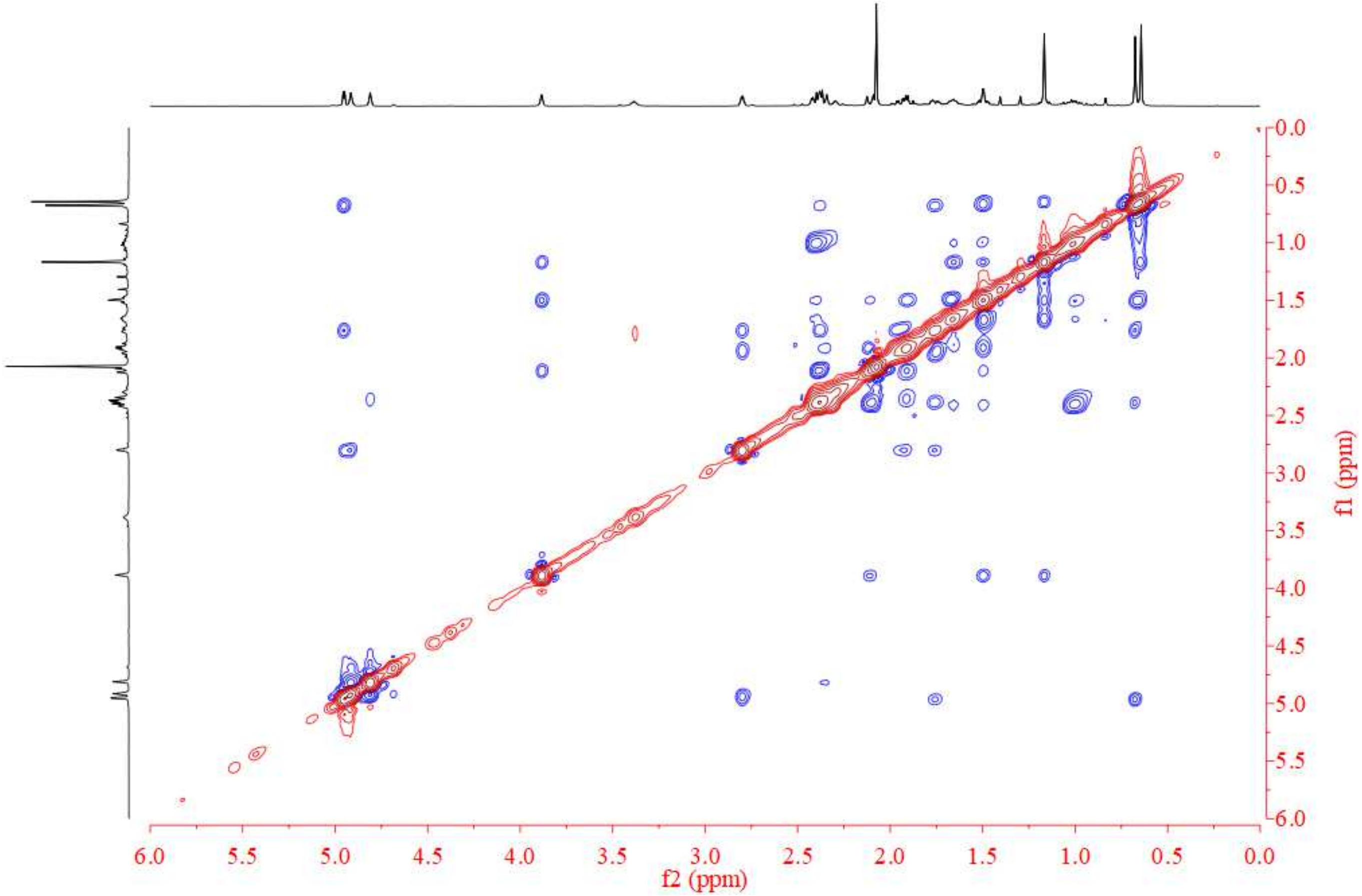

Figure S9: NOESY spectrum of **1**