

**Myrtucomvalones A-C, three unusual triketone-sesquiterpene adducts from the leaves of *Myrtus communis* ‘Variegata’**

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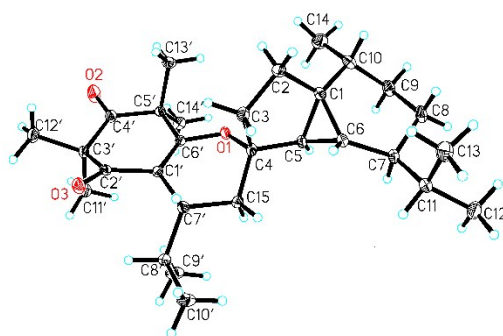
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## 1. Single crystal X-ray crystallographic analysis of 1 and 2



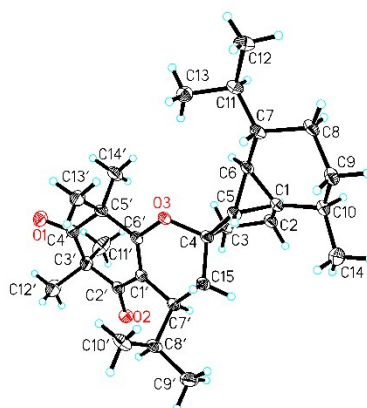
**Fig. S1** X-ray crystal structure of **1**

Crystallographic data for **1** have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication no. CCDC 1529330. Copies of the data can be obtained, free of charge, on application to the Director, CCDC, 12 Union Road, Cambridge CB2 IEZ, UK (fax: +44-(0)1223-336033 or email: [deposit@ccdc.cam.ac.uk](mailto:deposit@ccdc.cam.ac.uk)).

**Table S1** Crystal data and structure refinement for **1**

Identification code	20160910-o4-166-195-5-2
Empirical formula	C <sub>29</sub> H <sub>44</sub> O <sub>3</sub>
Formula weight	440.64
Temperature	293(2) K
Wavelength	1.54178 Å
Crystal system, space group	orthorhombic, <i>P</i> 2 <sub>1</sub> 2 <sub>1</sub> 2 <sub>1</sub>
Unit cell dimensions	<i>a</i> = 6.0056(10) Å, $\alpha$ = 90°. <i>b</i> = 18.0532(4) Å, $\beta$ = 90°. <i>c</i> = 23.3138(5) Å, $\gamma$ = 90°.
Volume	2527.69(9) Å <sup>3</sup>
<i>Z</i> , Calculated density	4, 1.158 Mg/m <sup>3</sup>
Absorption coefficient	0.561 mm <sup>-1</sup>
<i>F</i> (000)	968

Crystal size	0.45 × 0.33 × 0.23 mm
Theta range for data collection	4.500 to 62.854°
Limiting indices	-6 ≤ h ≤ 5, -20 ≤ k ≤ 20, -26 ≤ l ≤ 26
Reflections collected / unique	20350 / 4034 [R(int) = 0.0485]
Completeness to theta = 62.85	99.70%
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	1.000 and 0.563
Refinement method	Full-matrix least-squares on F <sup>2</sup>
Data / restraints / parameters	4034 / 0 / 299
Goodness-of-fit on F <sup>2</sup>	1.063
Final R indices [I > 2σ(I)]	R1 = 0.0299, wR2 = 0.0750
R indices (all data)	R1 = 0.0327, wR2 = 0.0779
Absolute structure parameter	-0.05(11)
Largest diff. peak and hole	0.134 and -0.130 e.Å



**Fig. S2** X-ray crystal structure of **2**

Crytallographic data for **2** have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication no. CCDC 1529333. Copies of the data can be obtained, free of charge, on application to the Director, CCDC, 12 Union Road, Cambridge CB2 IEZ, UK (fax: +44-(0)1223-336033 or email: [deposit@ccdc.cam.ac.uk](mailto:deposit@ccdc.cam.ac.uk)).

**Table S2** Crystal data and structure refinement for **2**

Identification code	exp_119
Empirical formula	C <sub>29</sub> H <sub>44</sub> O <sub>3</sub>
Formula weight	440.64
Temperature	100.01(10) K
Wavelength	1.54184 Å
Crystal system, space group	triclinic, <i>P</i> -1
Unit cell dimensions	a = 9.7468(7) Å, alpha = 107.318(7)°. b = 10.0413(9) Å, beta = 95.676(6)°. c = 14.7257(9) Å, gamma = 104.090(7)°.
Volume	1311.43(18) Å <sup>3</sup>
Z, Calculated density	2, 1.116 Mg/m <sup>3</sup>
Absorption coefficient	0.541 mm <sup>-1</sup>
F(000)	484
Crystal size	0.30 × 0.20 × 0.20 mm
Theta range for data collection	4.760 to 73.610°
Limiting indices	-8 ≤ h ≤ 11, -12 ≤ k ≤ 11, -18 ≤ l ≤ 18
Reflections collected / unique	8553 / 5070 [R(int) = 0.0556]
Completeness to theta = 66.97	99.66 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	1.000 and 0.703
Refinement method	Full-matrix least-squares on F <sup>2</sup>
Data / restraints / parameters	5070 / 0 / 298
Goodness-of-fit on F <sup>2</sup>	1.032
Final R indices [I > 2σ(I)]	R1 = 0.0774, wR2 = 0.2304
R indices (all data)	R1 = 0.0985, wR2 = 0.2426
Largest diff. peak and hole	0.370 and -0.304 e.Å

## 2. UV, IR, HR-ESI-MS, CD and NMR spectra of 1-3

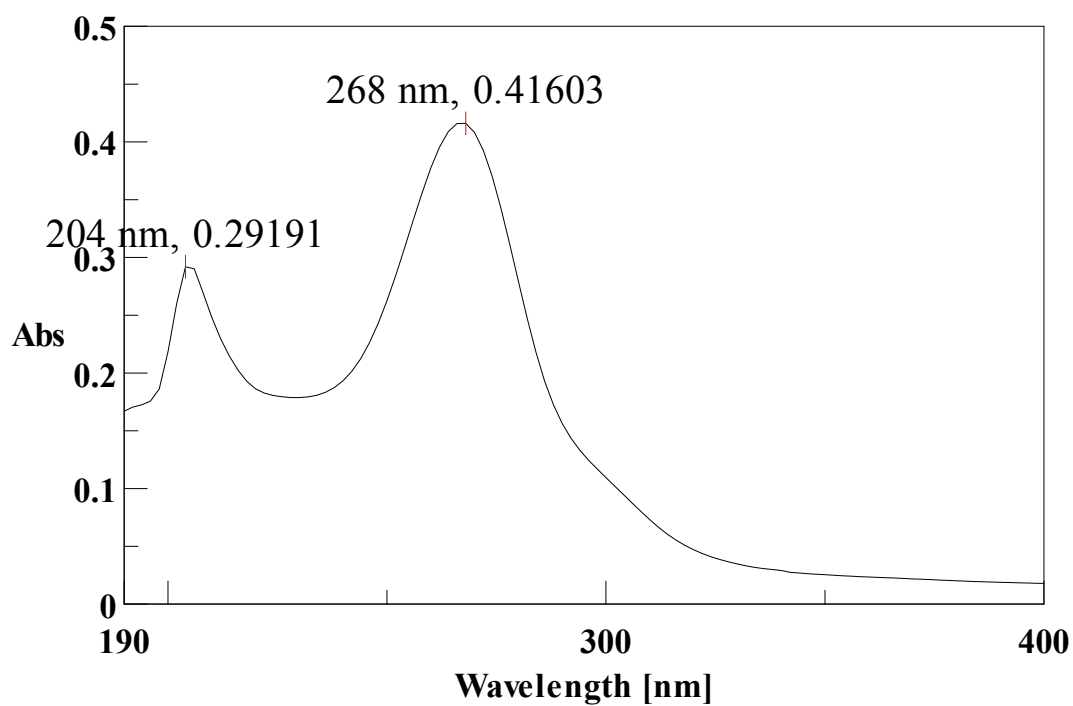


Fig. S3 UV spectrum of 1 in CH<sub>3</sub>OH

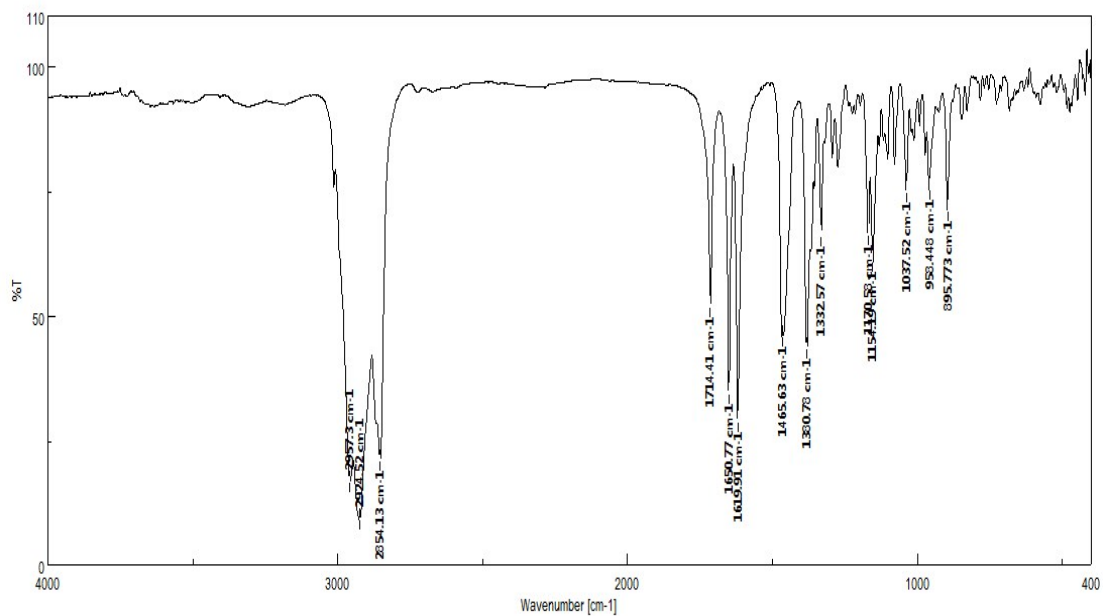


Fig. S4 IR (KBr disc) spectrum of 1

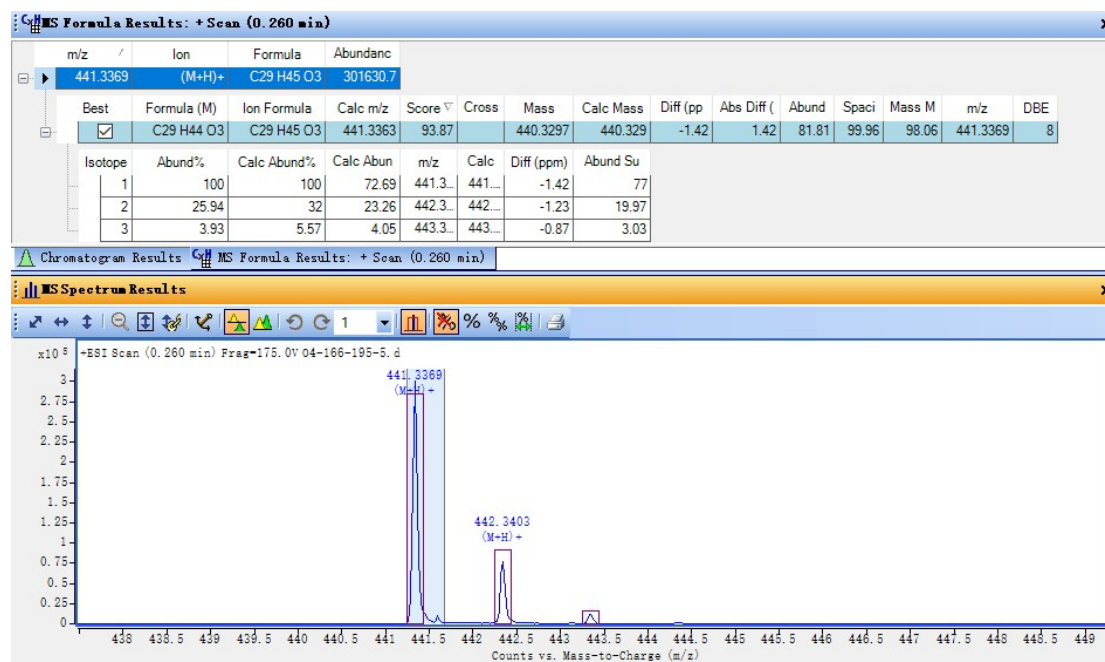


Fig. S5 HR-ESI-MS spectrum of 1

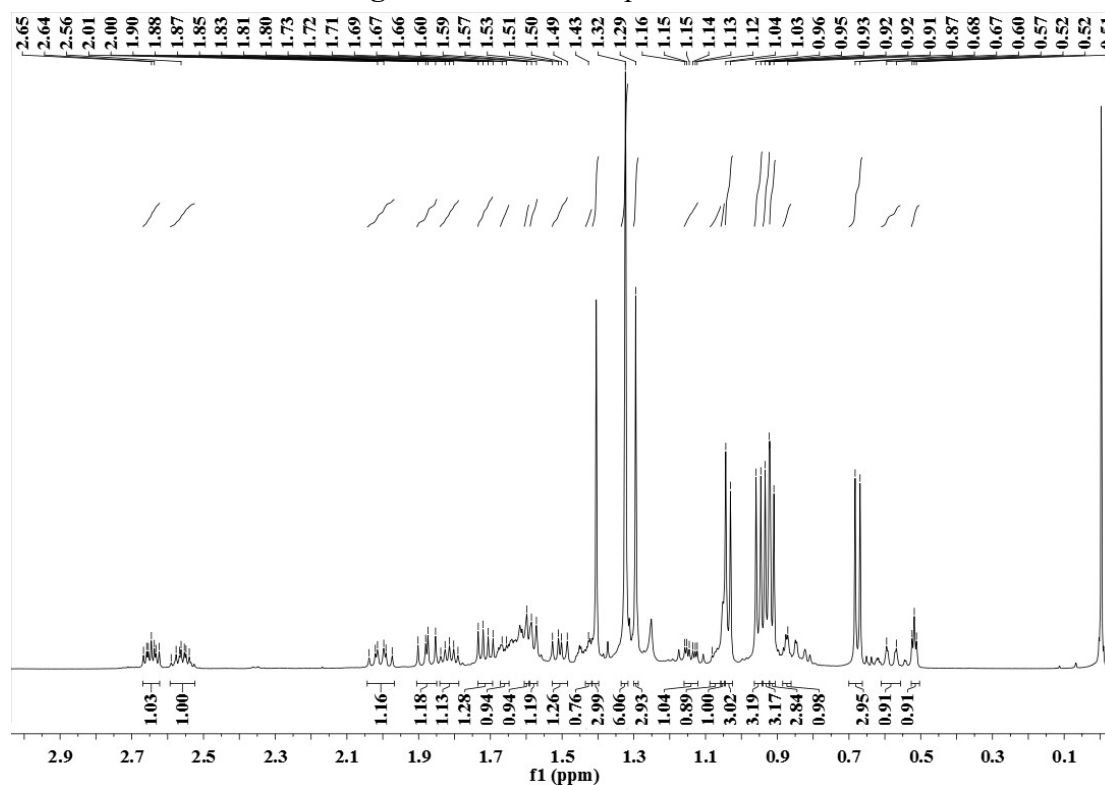


Fig. S6 <sup>1</sup>H NMR spectrum of 1 (500 MHz, CDCl<sub>3</sub>)



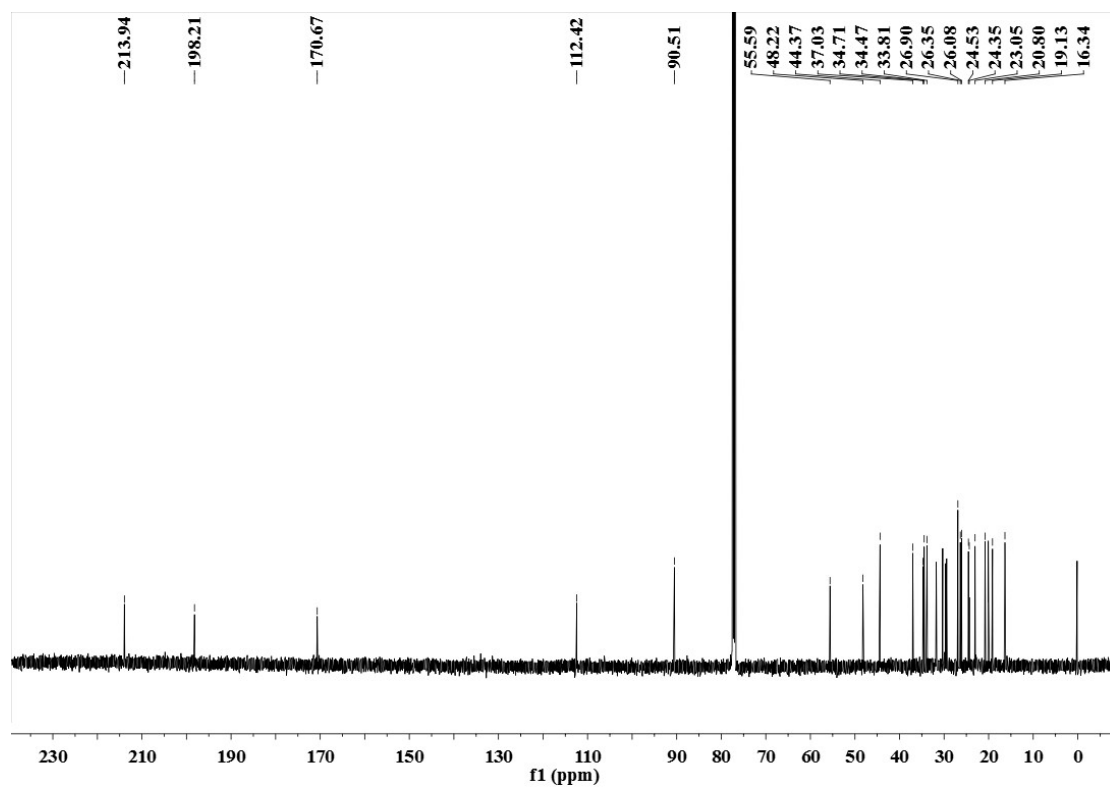


Fig. S7  $^{13}\text{C}$  NMR spectrum of **1** (125 MHz,  $\text{CDCl}_3$ )

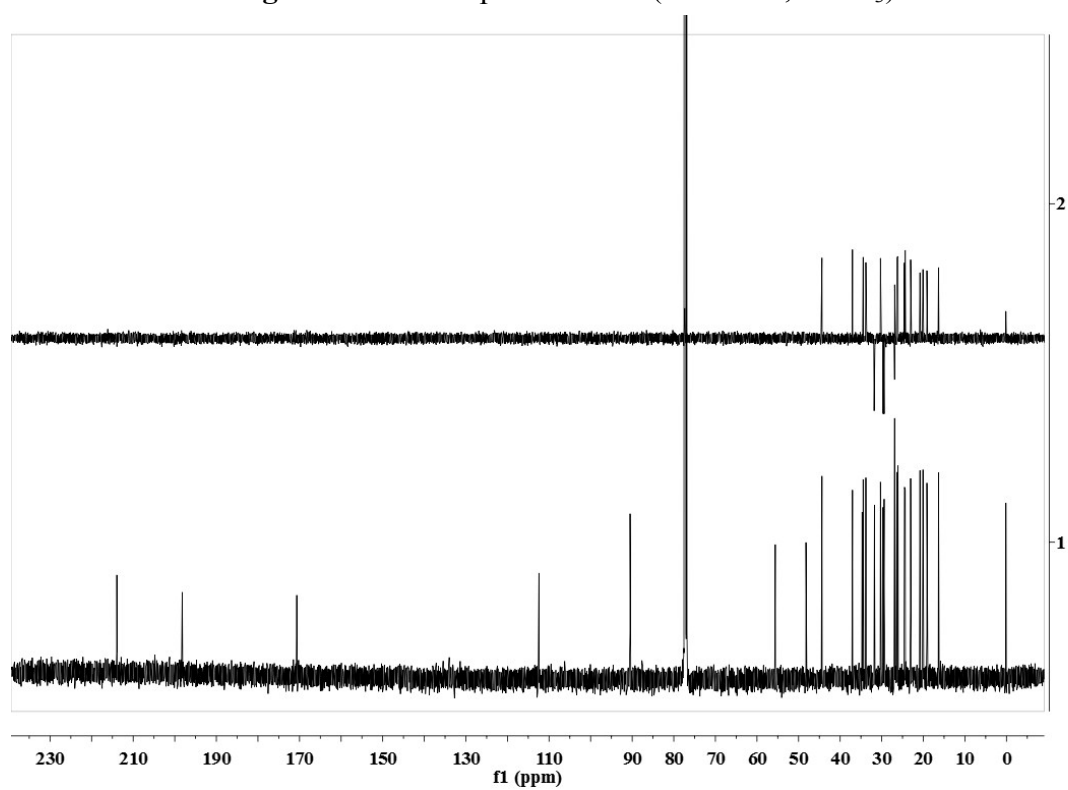
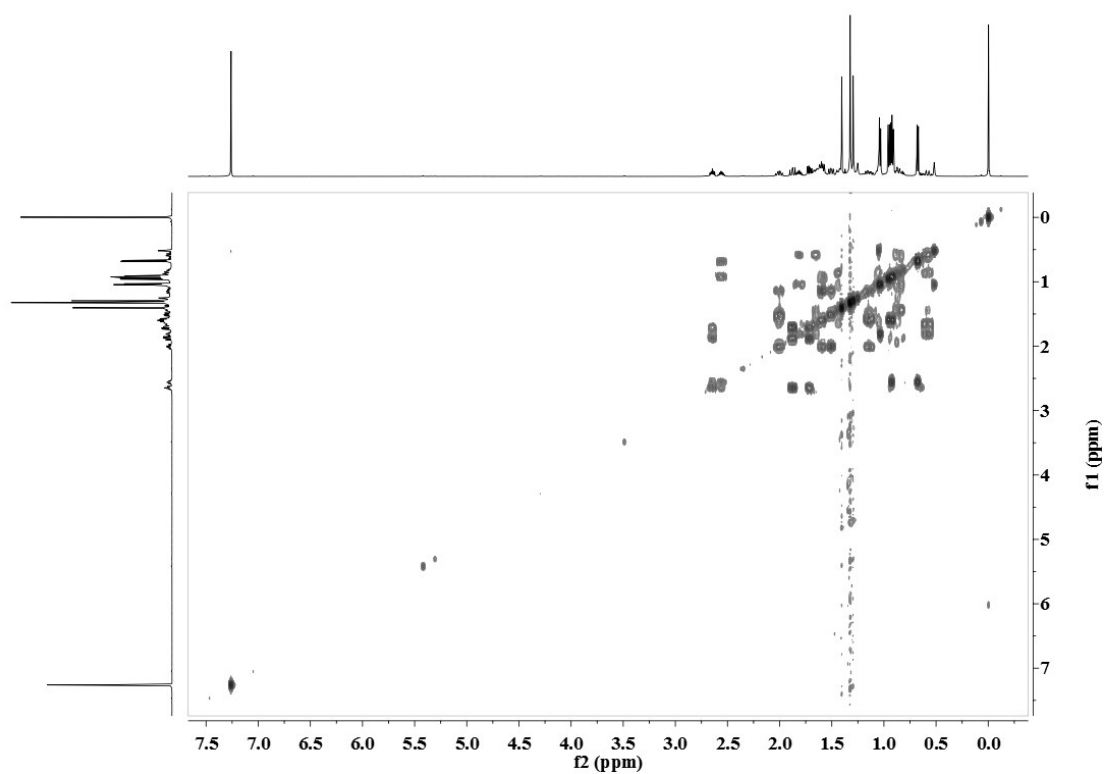
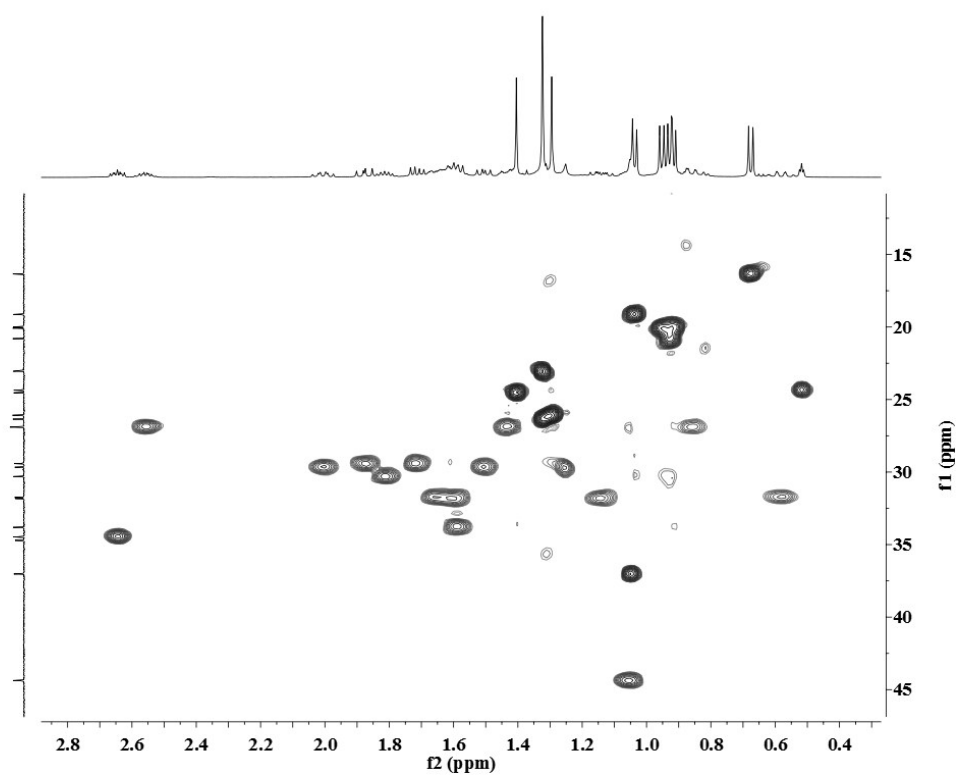


Fig. S8 DEPT-135 spectrum of **1** ( $\text{CDCl}_3$ )



**Fig. S9**  $^1\text{H}$ - $^1\text{H}$  COSY spectrum of **1** ( $\text{CDCl}_3$ )



**Fig. S10** HSQC spectrum of **1** ( $\text{CDCl}_3$ )

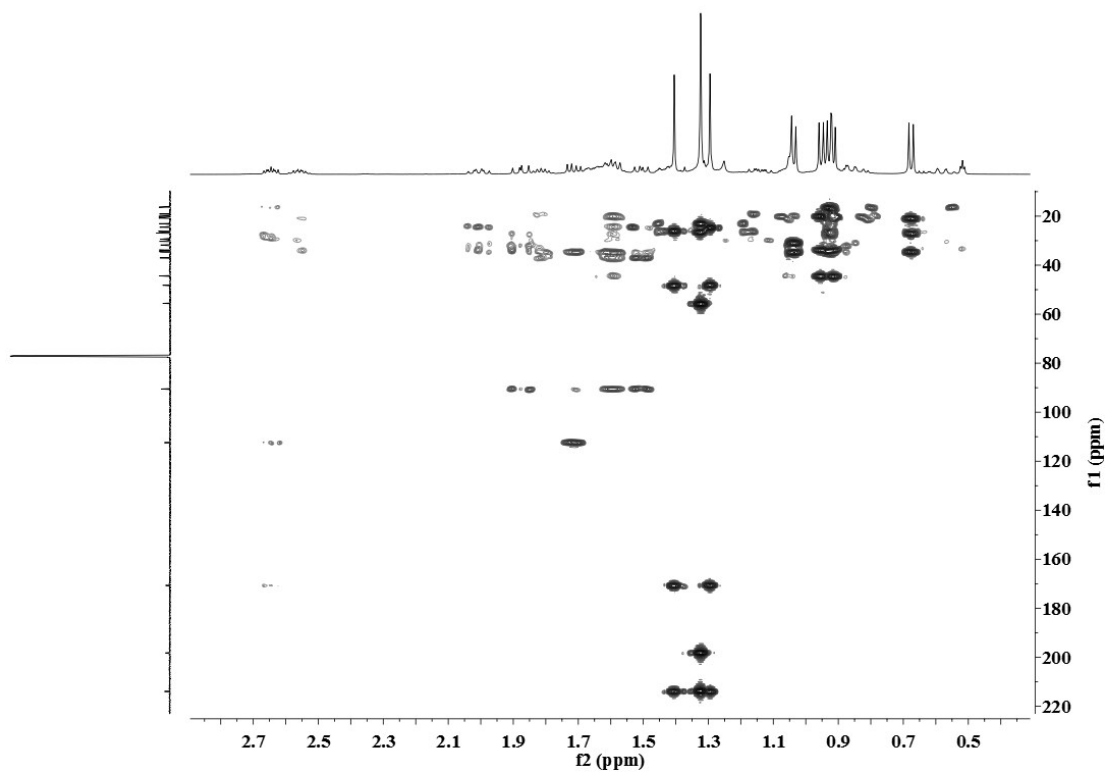


Fig. S11 HMBC spectrum of **1** (CDCl<sub>3</sub>)

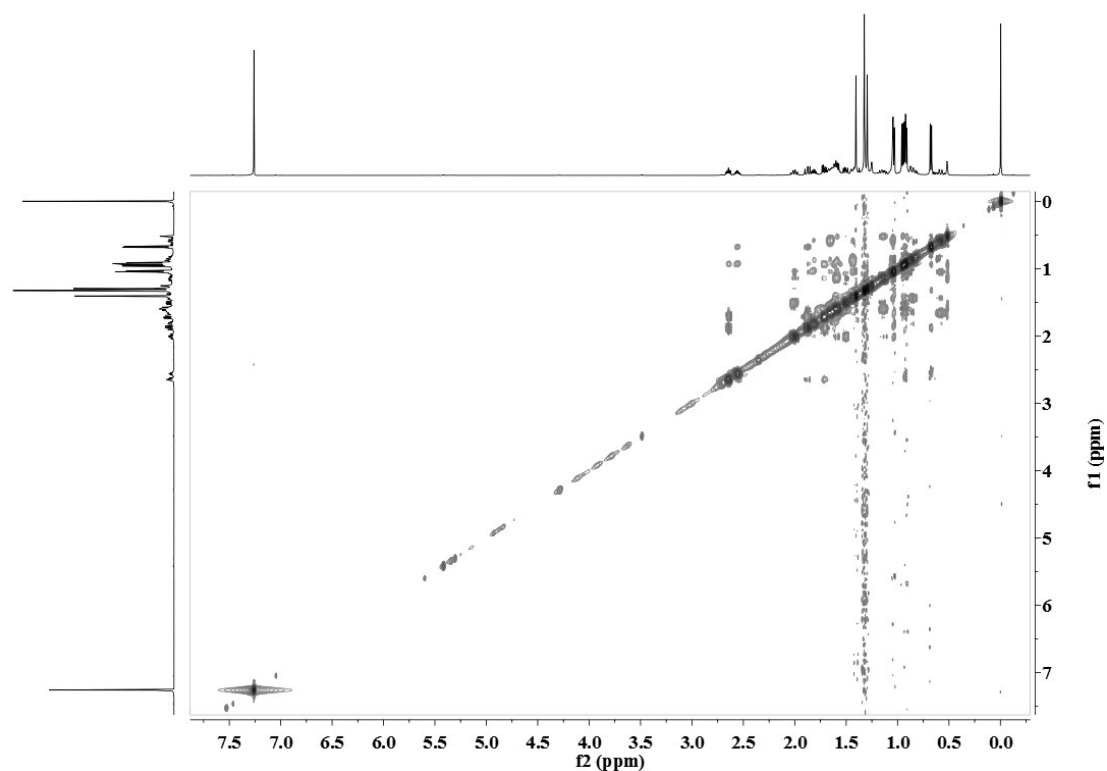


Fig. S12 NOESY spectrum of **1** (CDCl<sub>3</sub>)

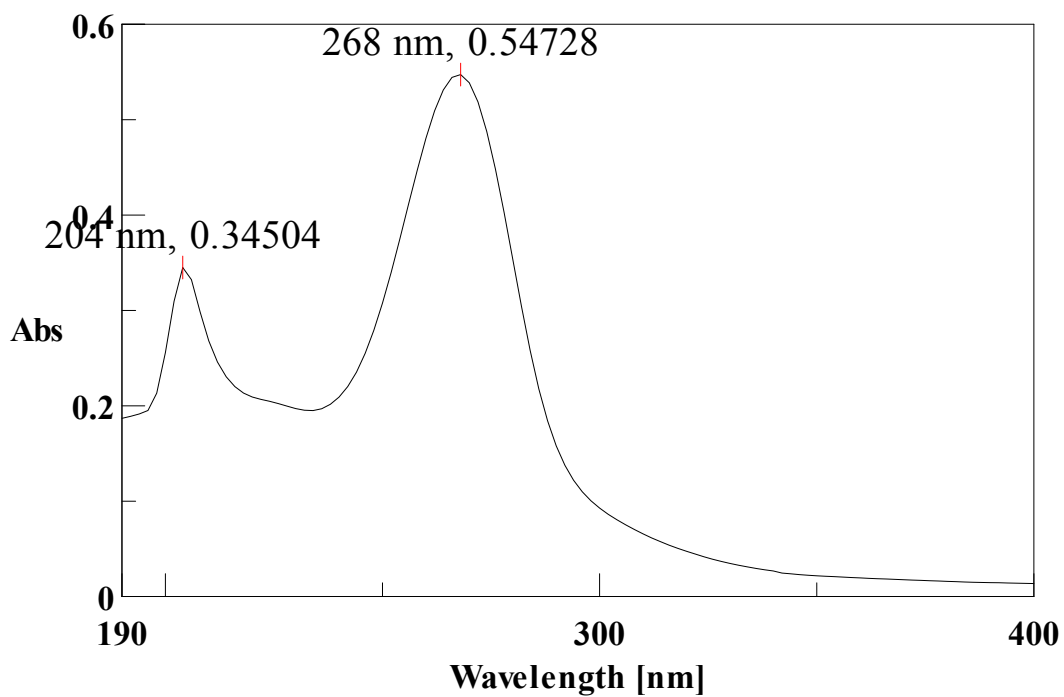


Fig. S13 UV spectrum of 2 in CH<sub>3</sub>OH

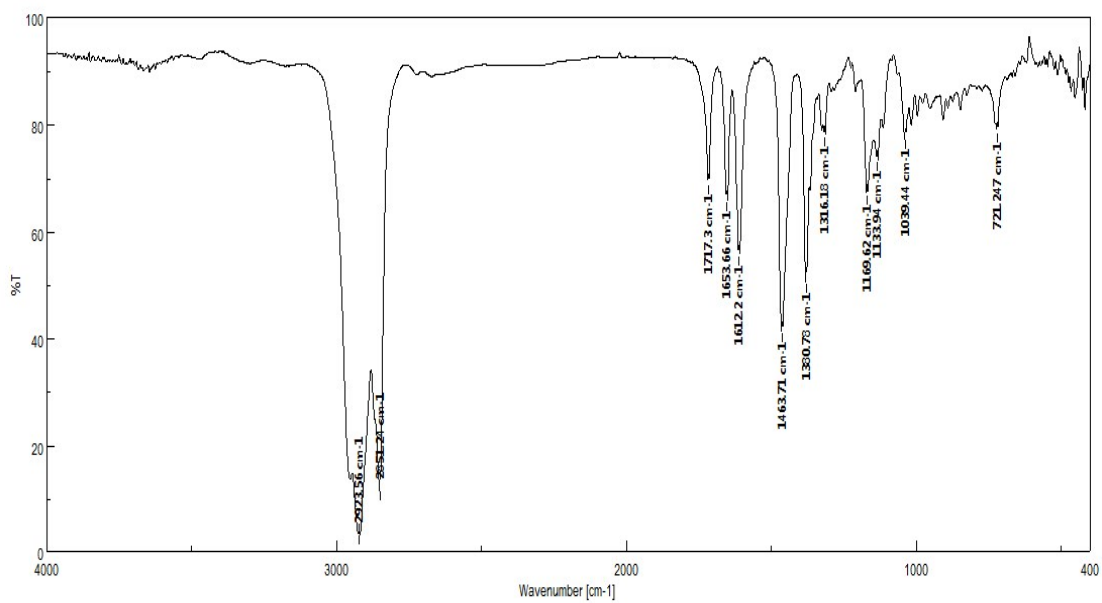


Fig. S14 IR (KBr disc) spectrum of 2

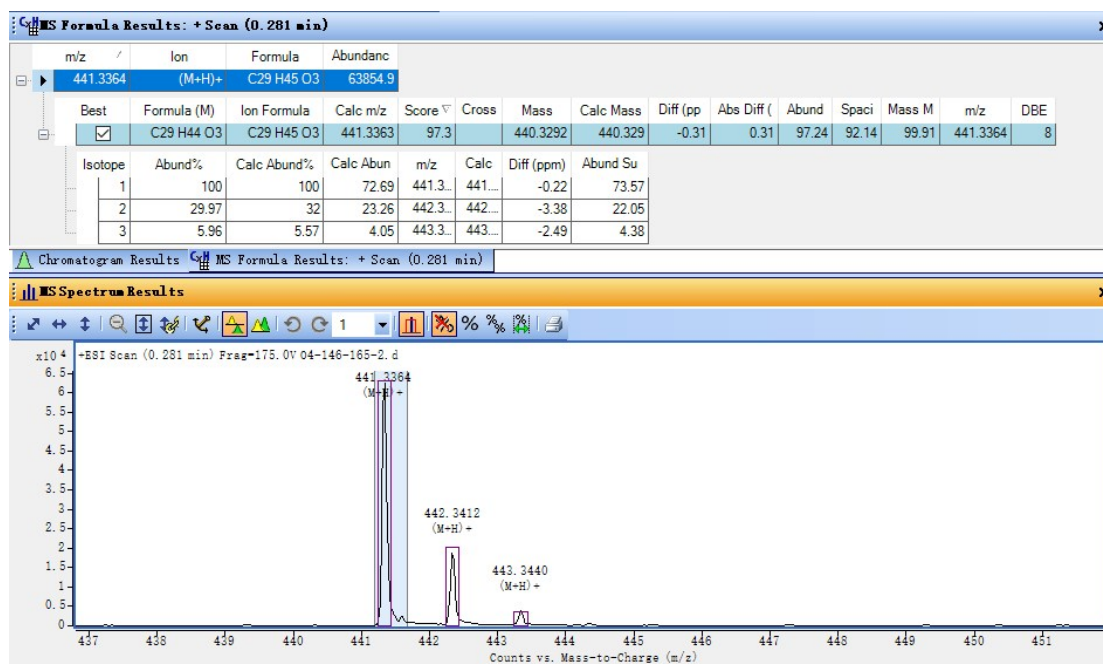


Fig. S15 HR-ESI-MS spectrum of **2**

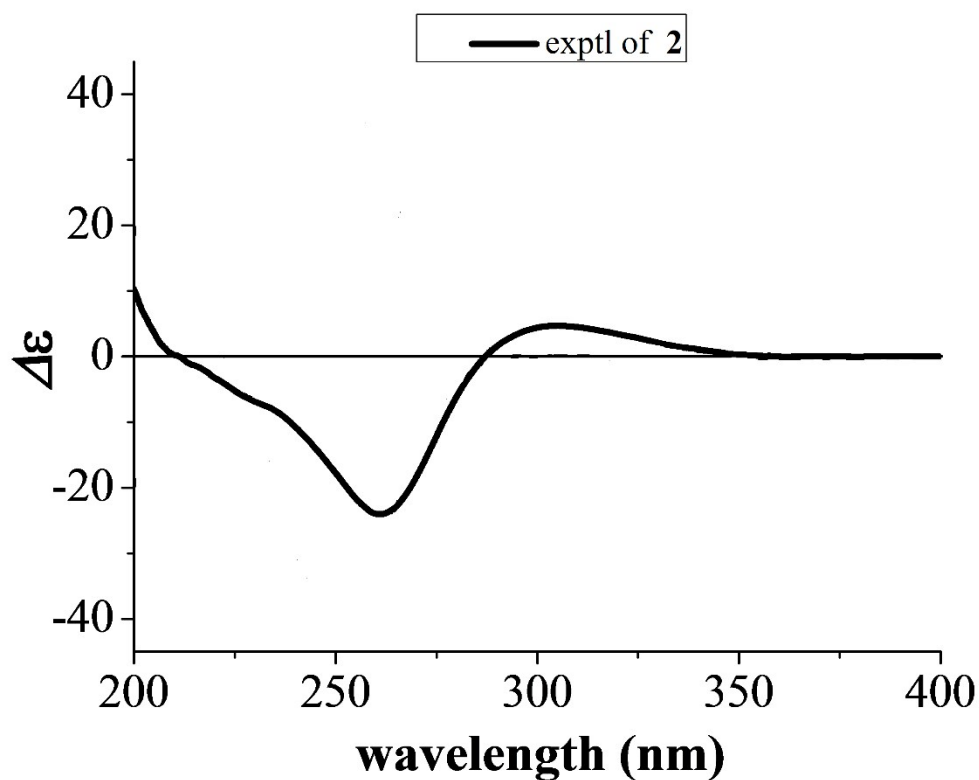


Fig. S16 CD spectrum of **2** in CH<sub>3</sub>CN

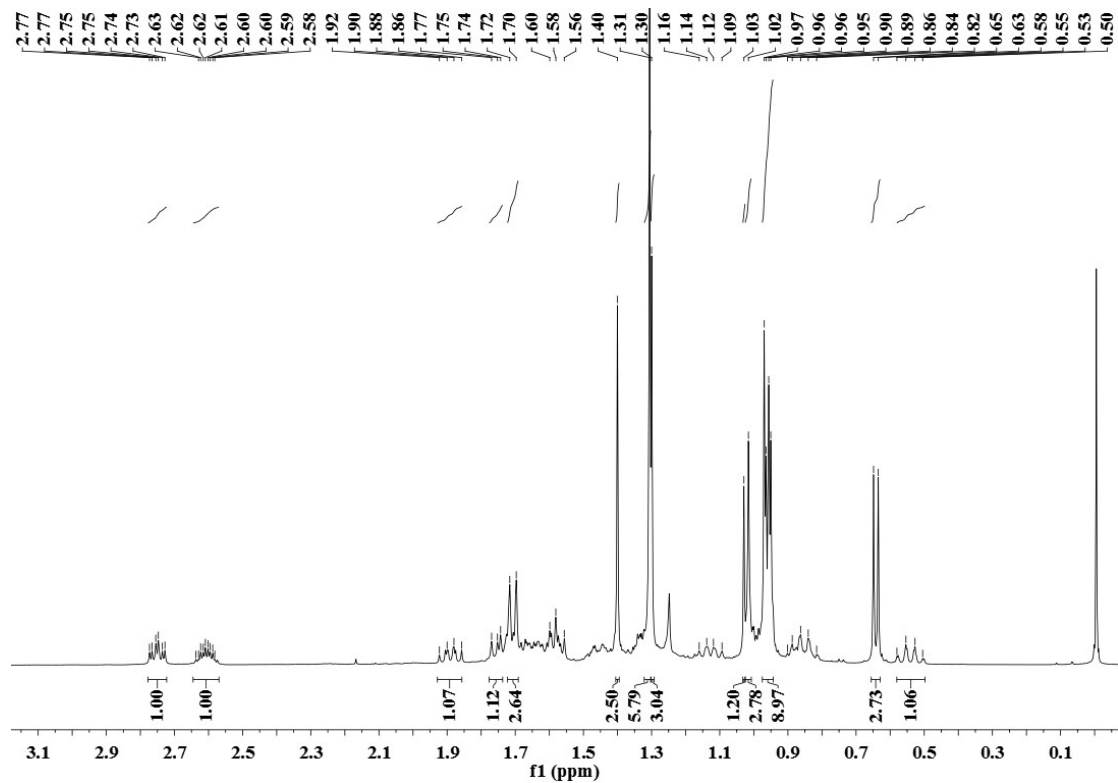


Fig. S17  $^1\text{H}$  NMR spectrum of **2** (500 MHz,  $\text{CDCl}_3$ )

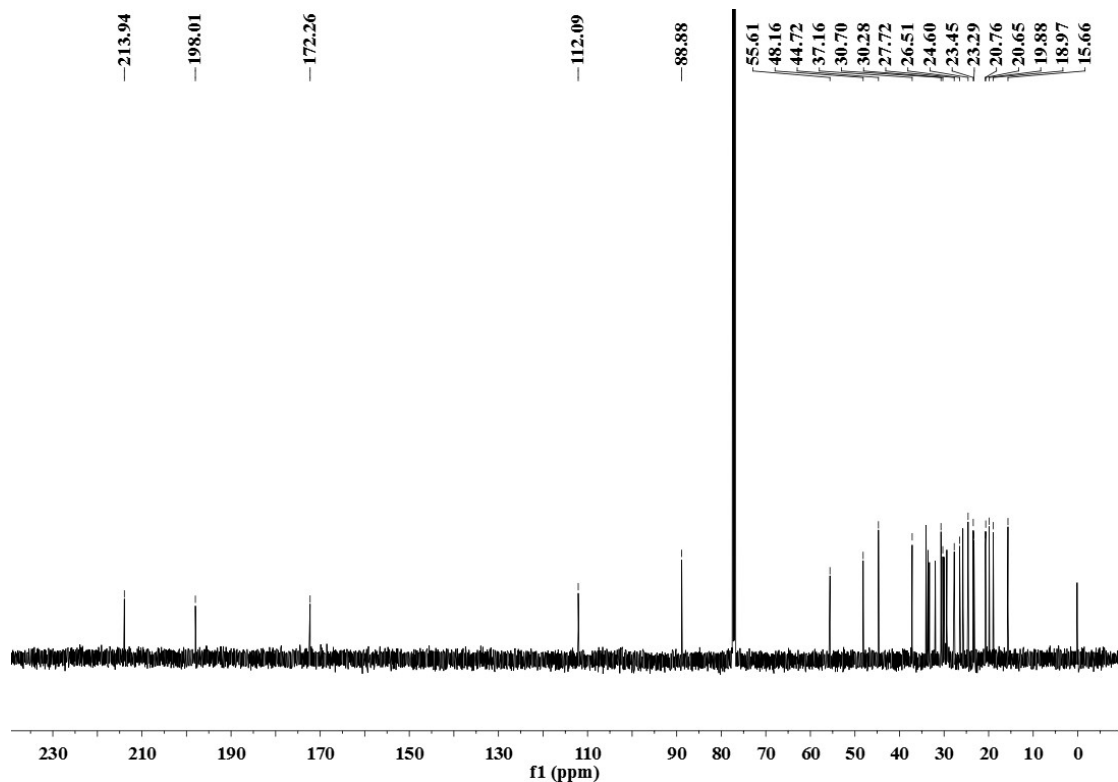


Fig. S18  $^{13}\text{C}$  NMR spectrum of **2** (125 MHz,  $\text{CDCl}_3$ )

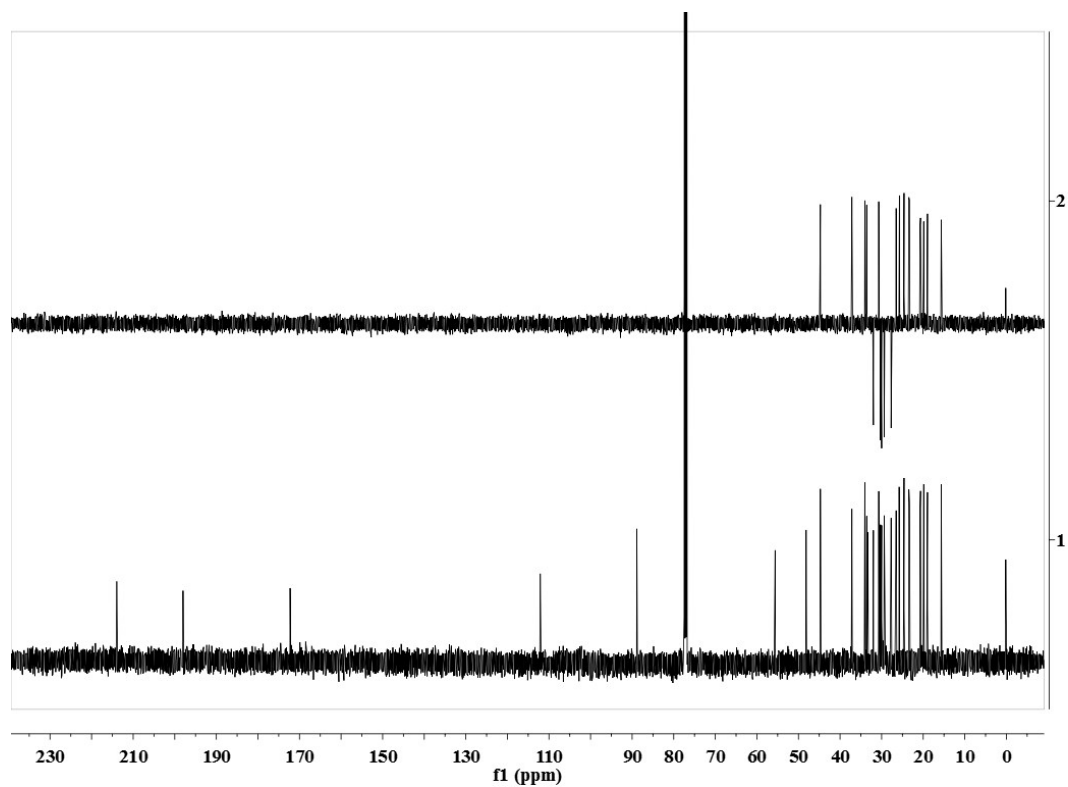


Fig. S19 DEPT-135 spectrum of **2** (CDCl<sub>3</sub>)

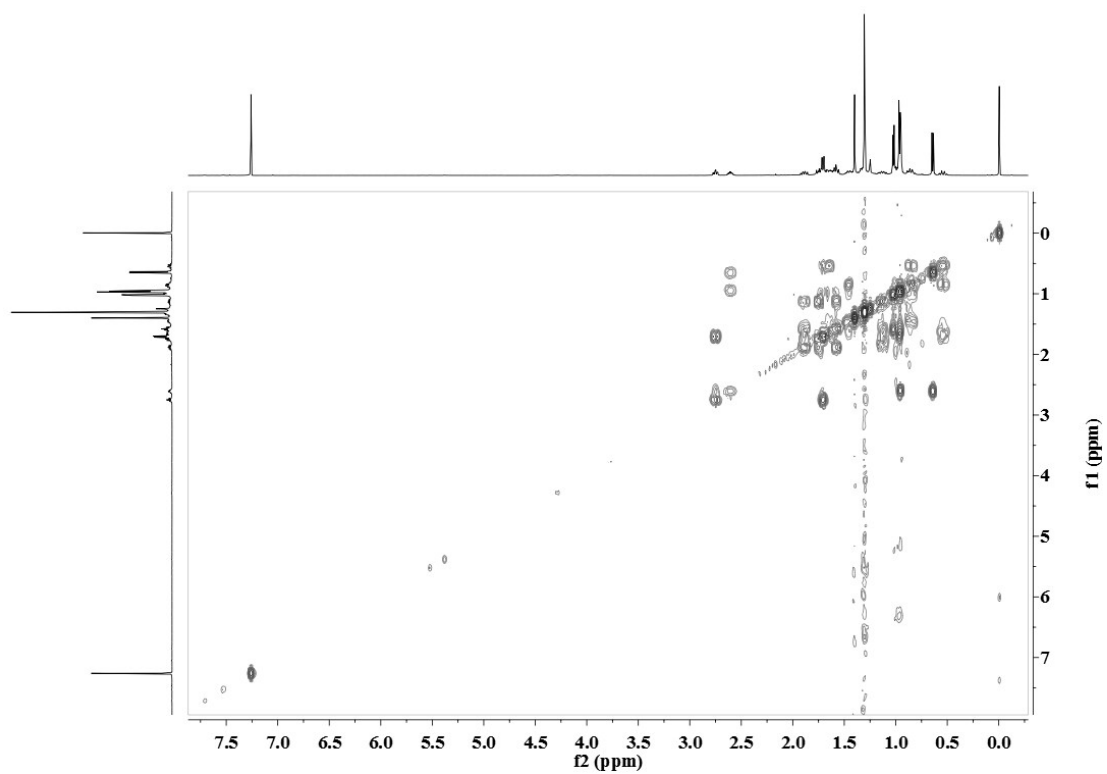
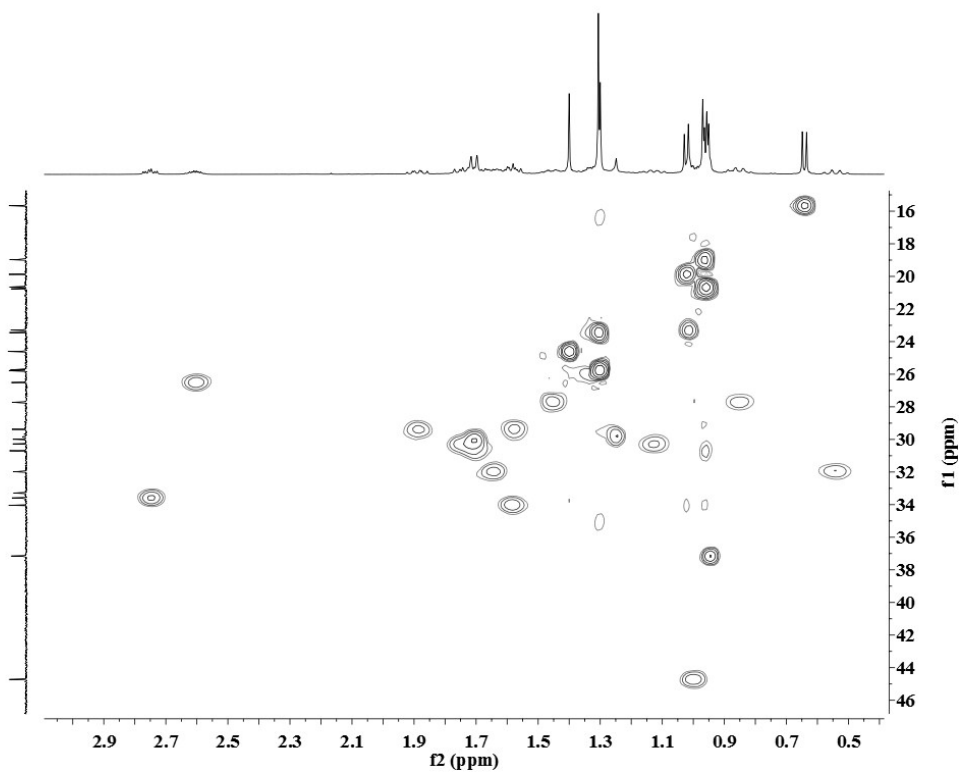
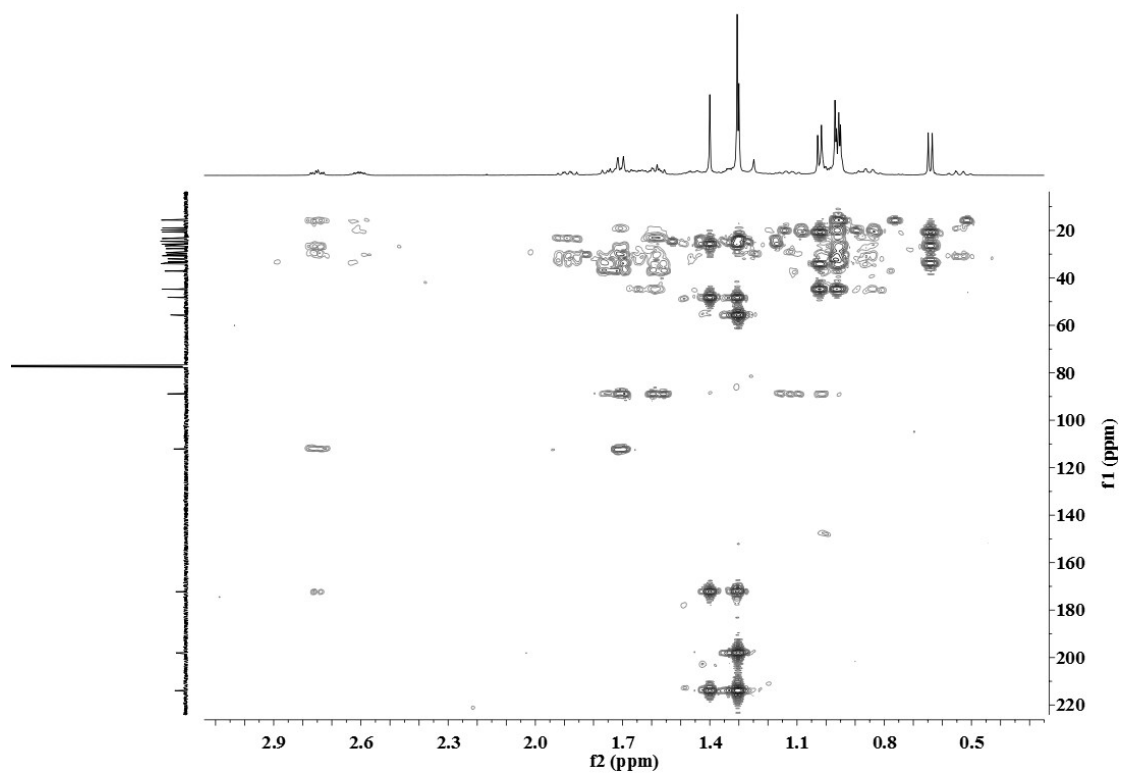


Fig. S20 <sup>1</sup>H-<sup>1</sup>H COSY spectrum of **2** (CDCl<sub>3</sub>)



**Fig. S21** HSQC spectrum of **2** (CDCl<sub>3</sub>)



**Fig. S22** HMBC spectrum of **2** (CDCl<sub>3</sub>)



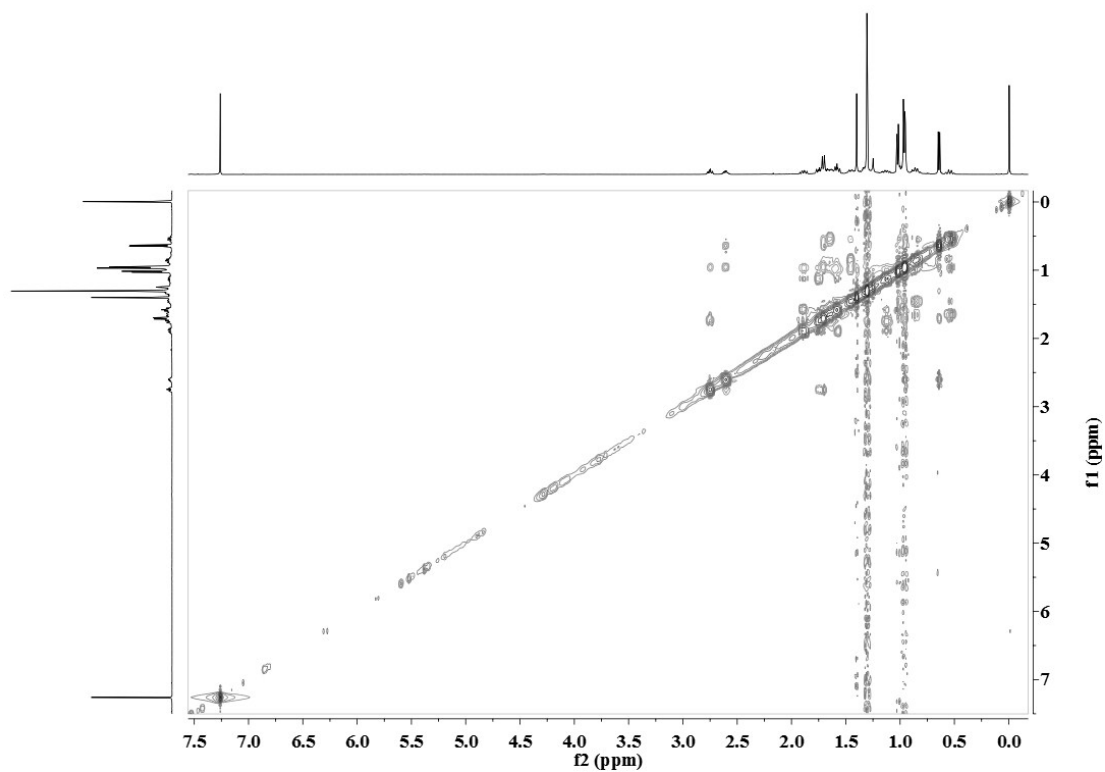


Fig. S23 NOESY spectrum of **2** ( $\text{CDCl}_3$ )

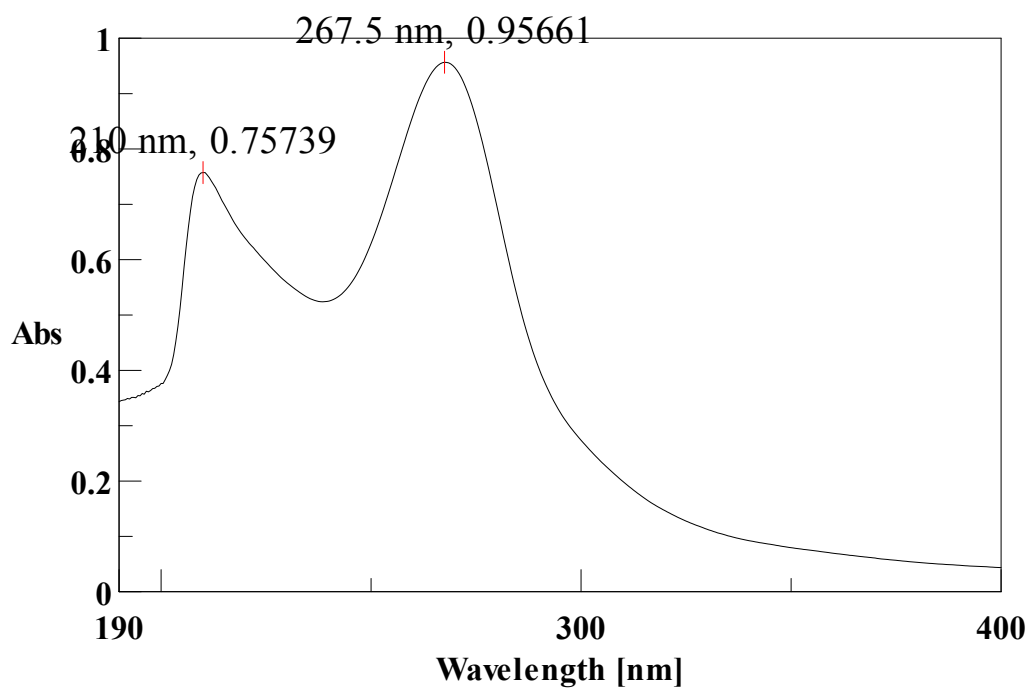


Fig. S24 UV spectrum of **3** in  $\text{CH}_3\text{OH}$

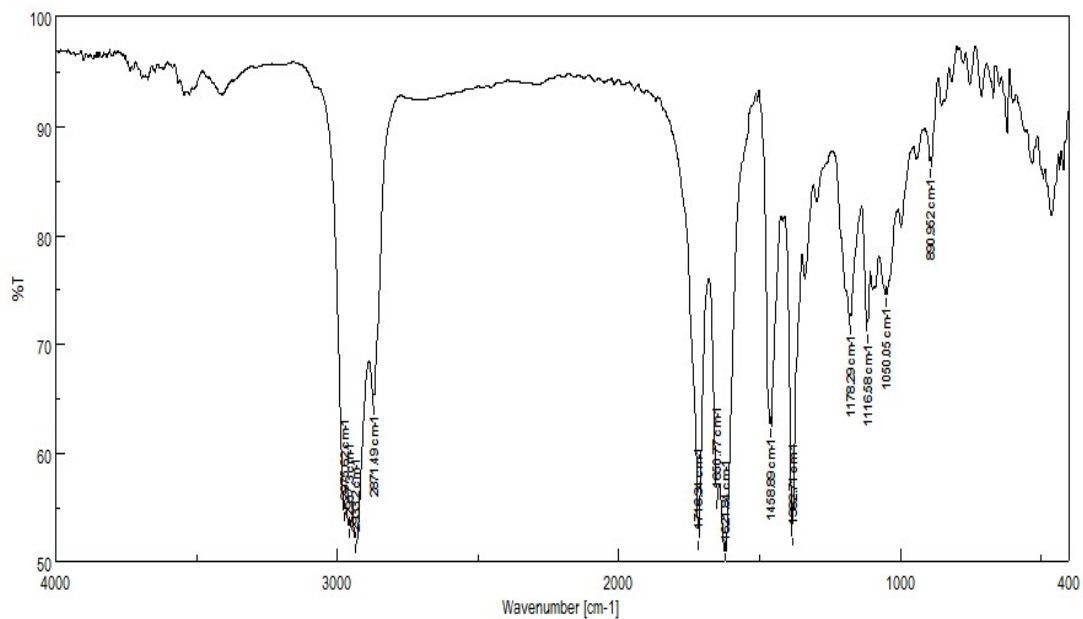


Fig. S25 IR (KBr disc) spectrum of 3

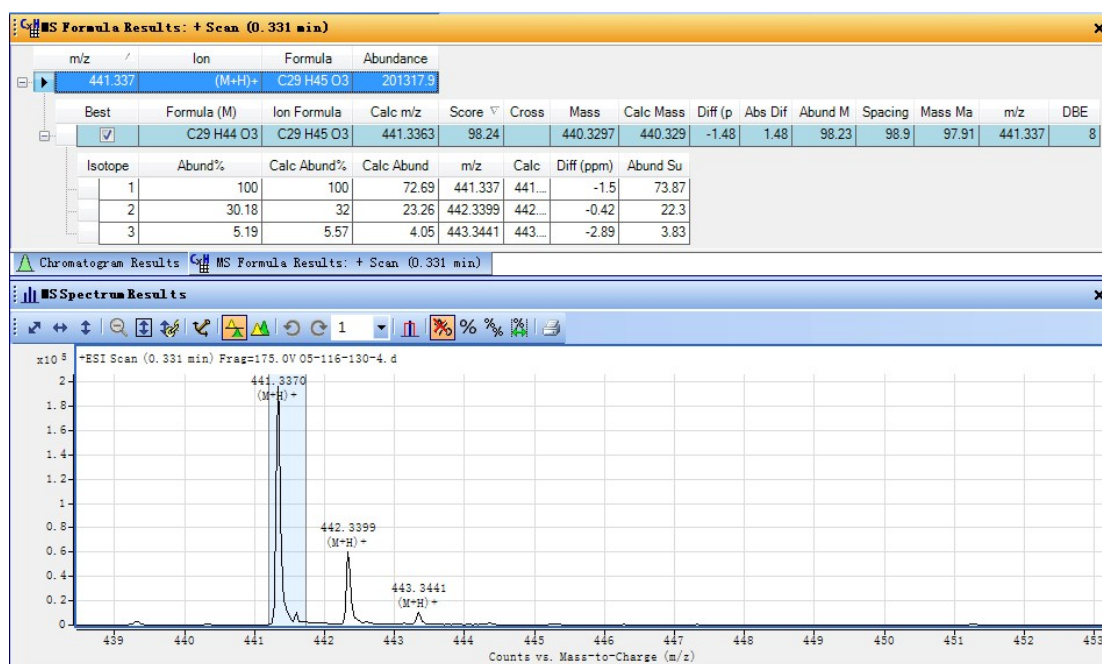


Fig. S26 HR-ESI-MS spectrum of 3

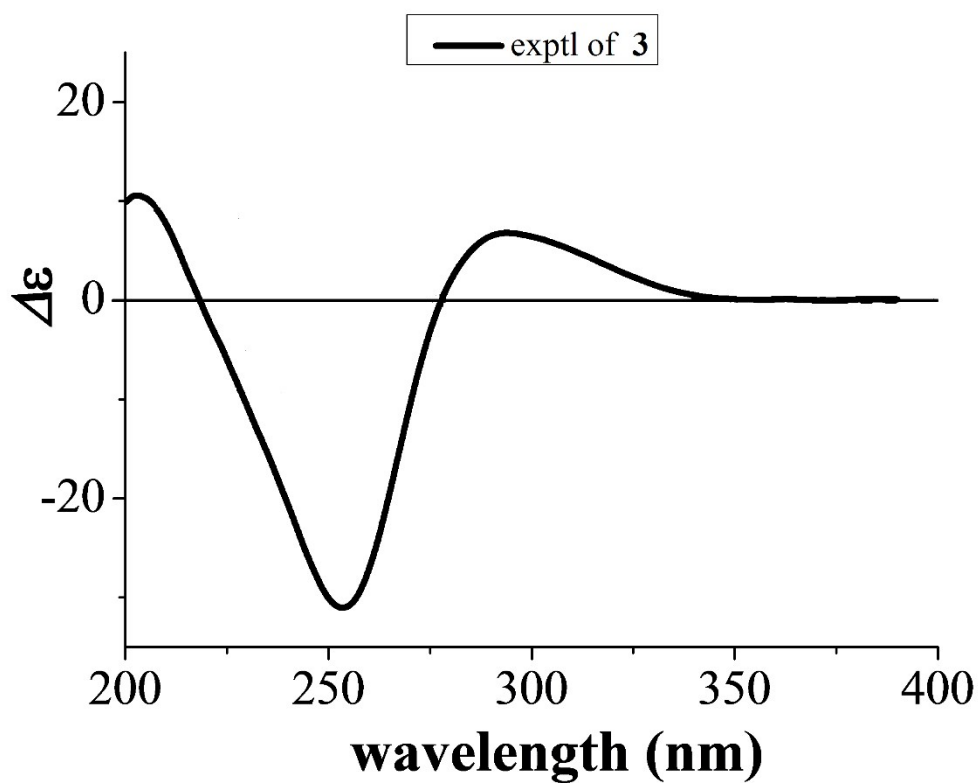


Fig. S27 CD spectrum of **3** in CH<sub>3</sub>CN

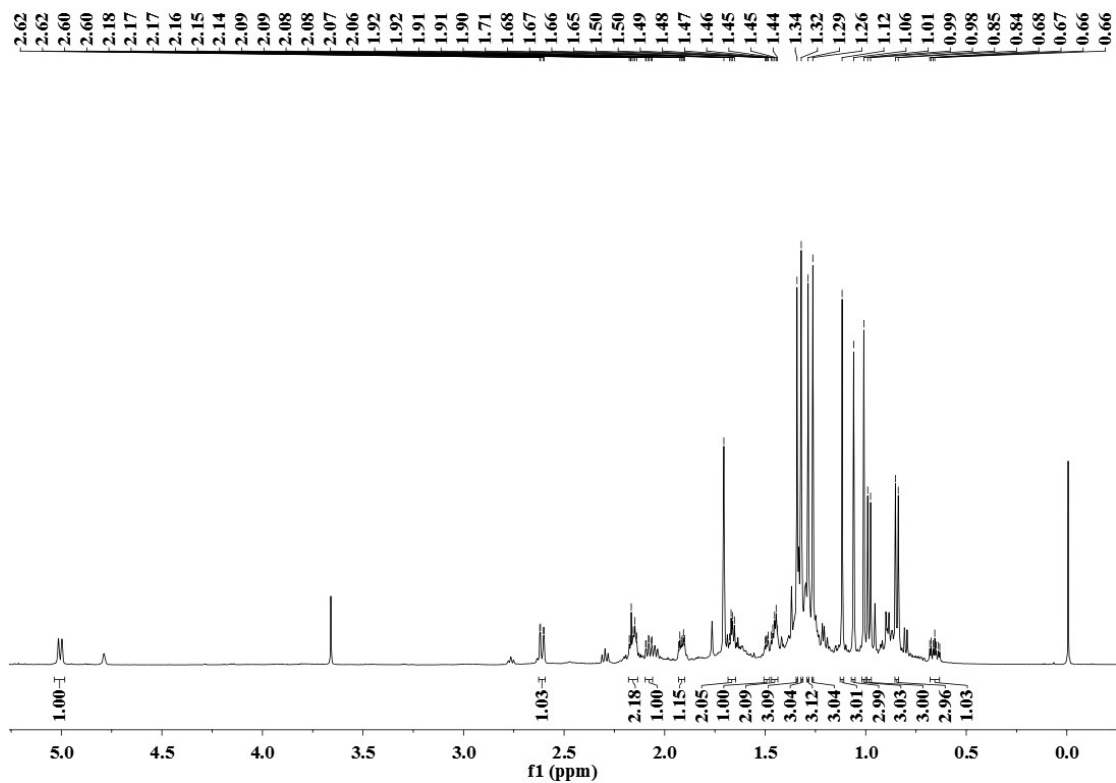


Fig. S28 <sup>1</sup>H NMR spectrum of **3** (500 MHz, CDCl<sub>3</sub>)

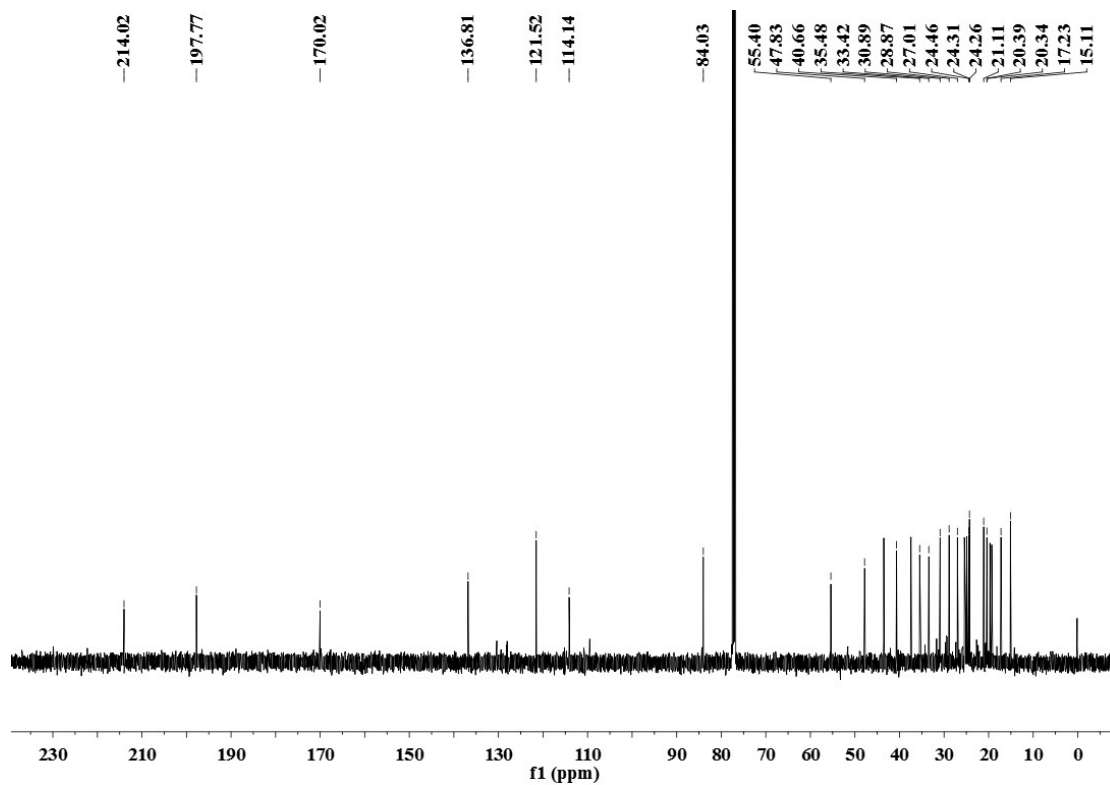


Fig. S29  $^{13}\text{C}$  NMR spectrum of **3** (125 MHz,  $\text{CDCl}_3$ )

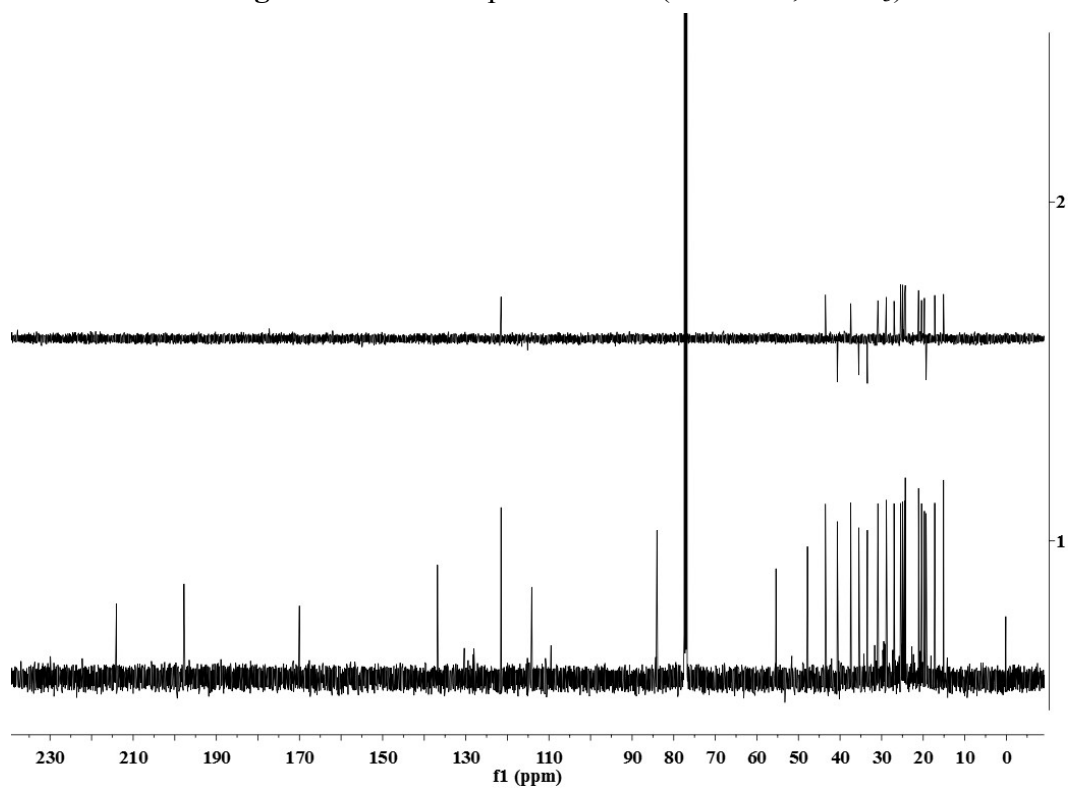


Fig. S30 DEPT-135 spectrum of **3** ( $\text{CDCl}_3$ )

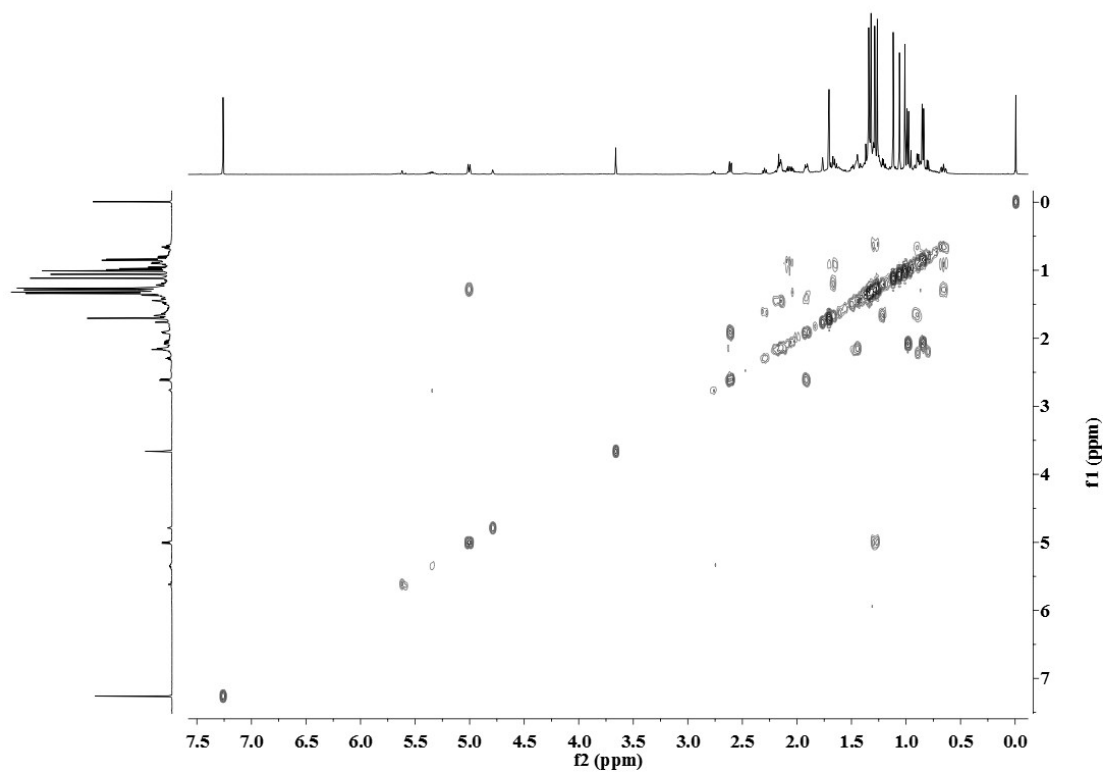


Fig. S31  $^1\text{H}$ - $^1\text{H}$  COSY spectrum of **3** ( $\text{CDCl}_3$ )

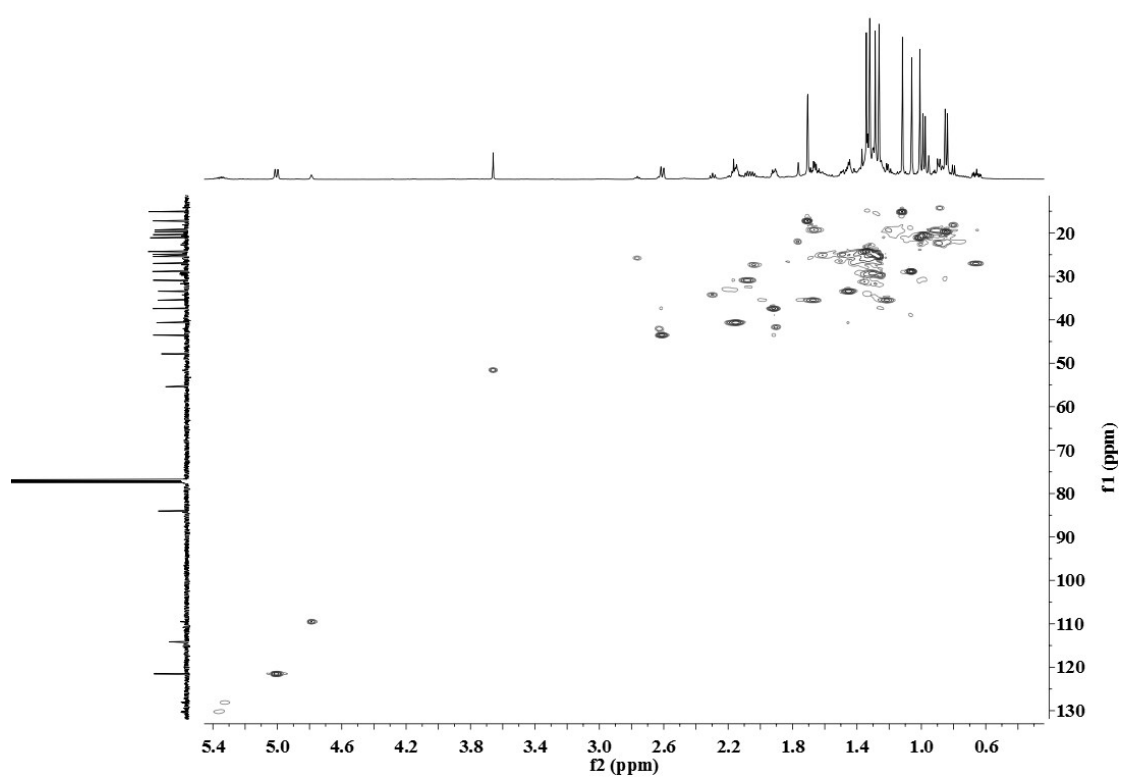


Fig. S32 HSQC spectrum of **3** ( $\text{CDCl}_3$ )

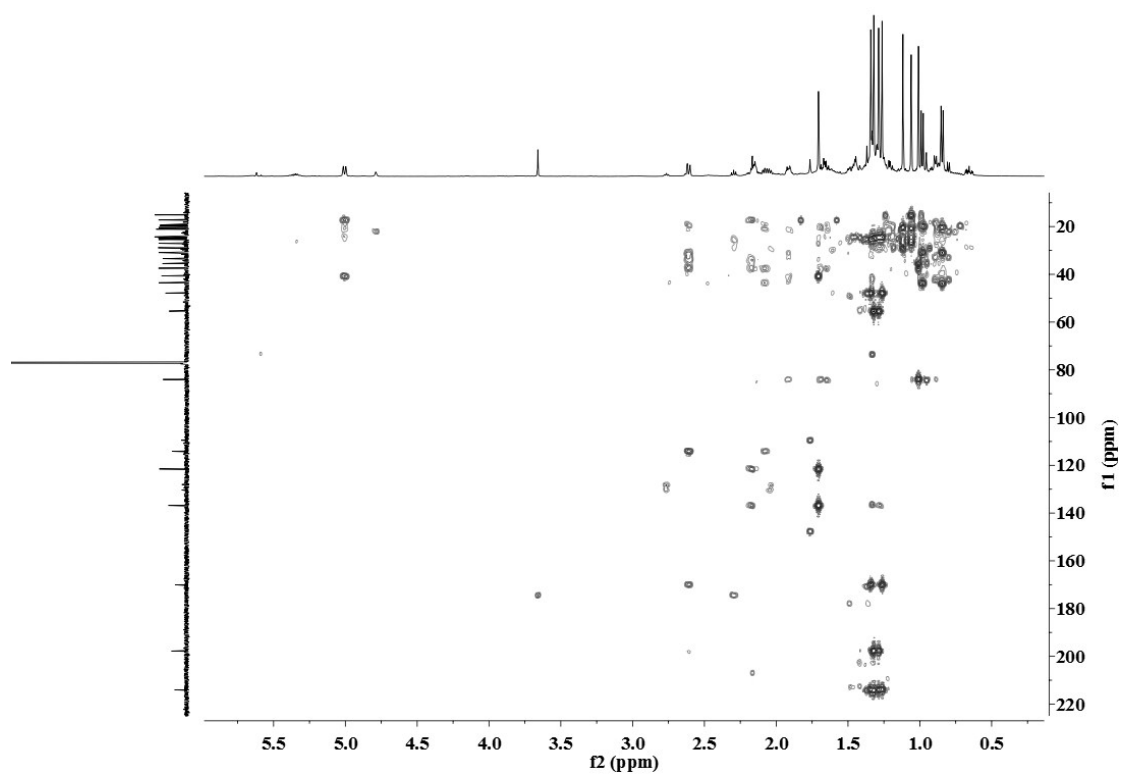


Fig. S33 HMBC spectrum of **3** (CDCl<sub>3</sub>)

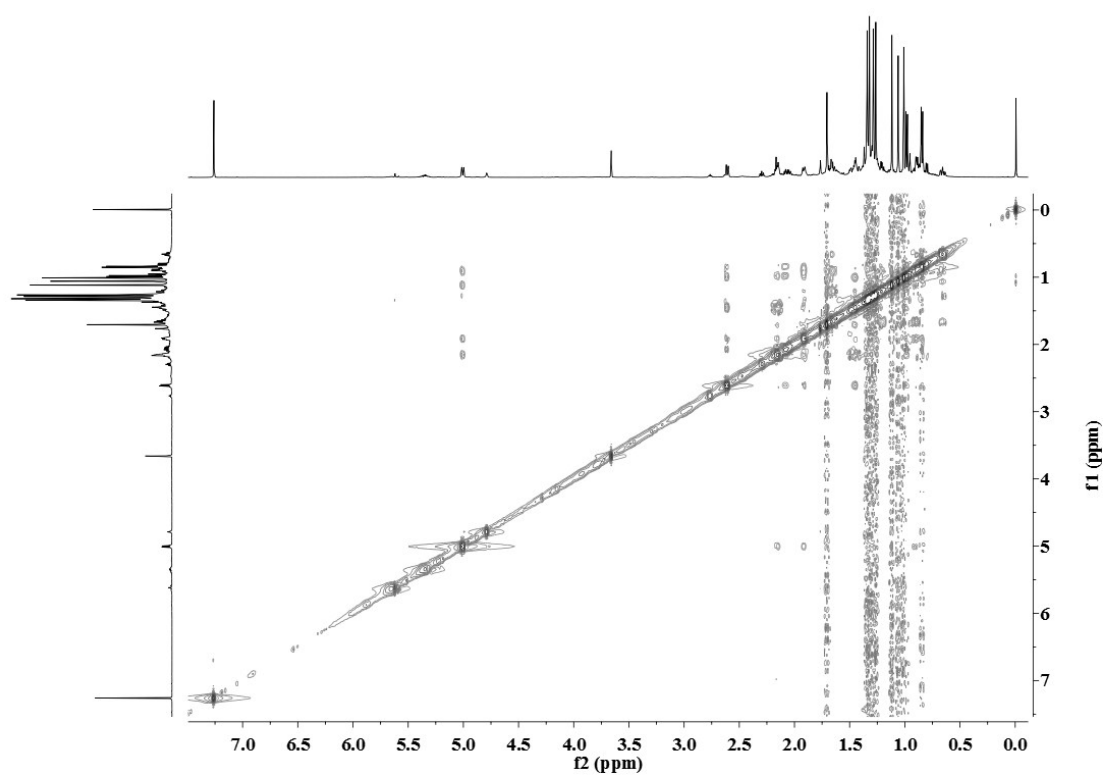
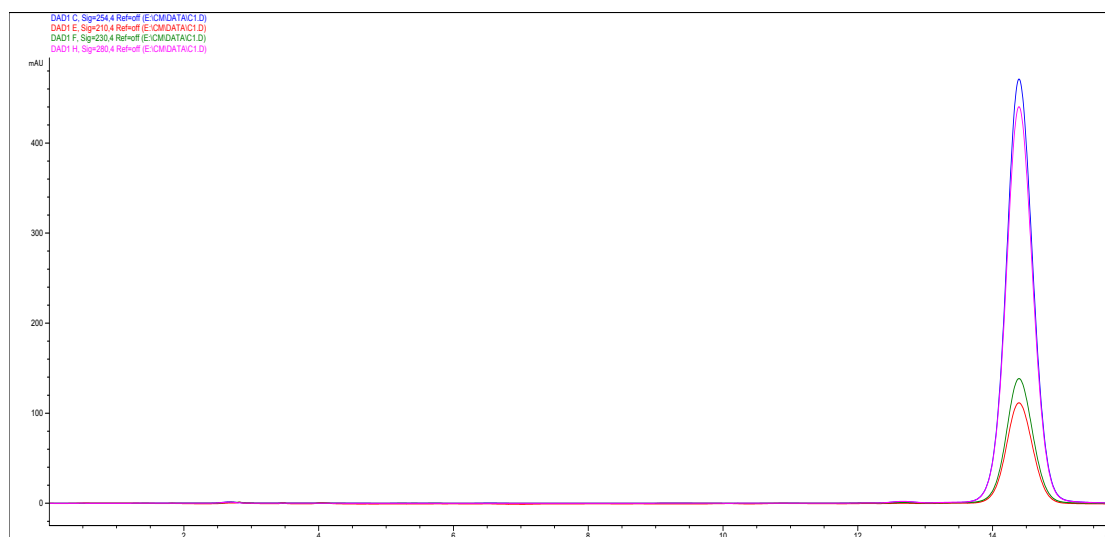


Fig. S34 NOESY spectrum of **3** (CDCl<sub>3</sub>)

### 3. Purity analysis of 1-7 for biological test

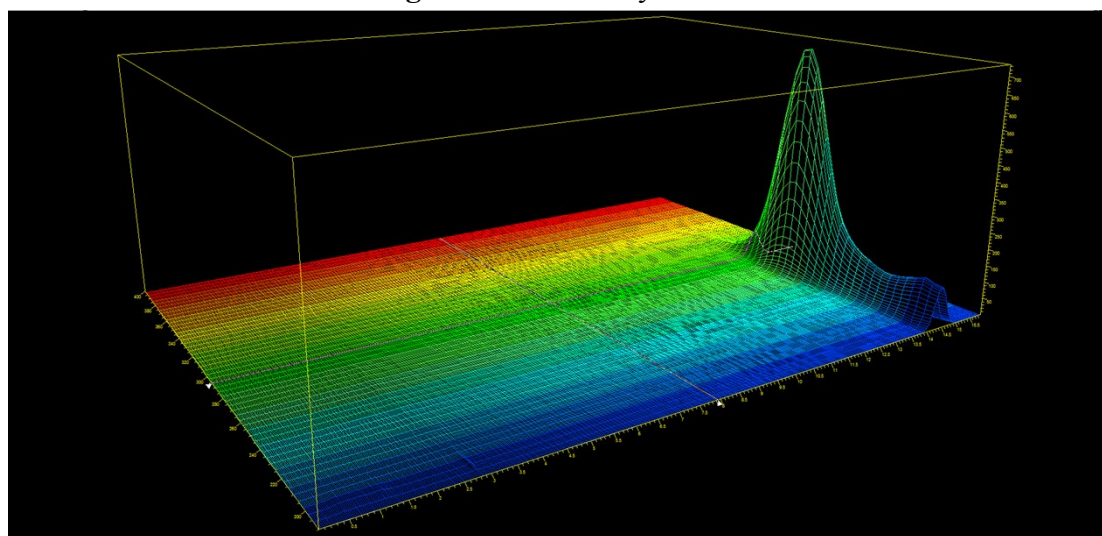
**Table S3** The purities of 1-7 calculated by area normalization method

Compounds	Wavelength (nm)			
	210	230	254	280
<b>1</b>	99.46%	99.42%	99.55%	99.57%
<b>2</b>	99.06%	99.50%	99.54%	99.55%
<b>3</b>	98.51%	98.64%	99.99%	99.99%
<b>4</b>	99.83%	99.99%	99.99%	99.99%
<b>5</b>	99.86%	99.99%	99.99%	99.99%
<b>6</b>	98.80%	96.76%	99.77%	99.70%
<b>7</b>	98.67%	96.22%	99.59%	99.42%

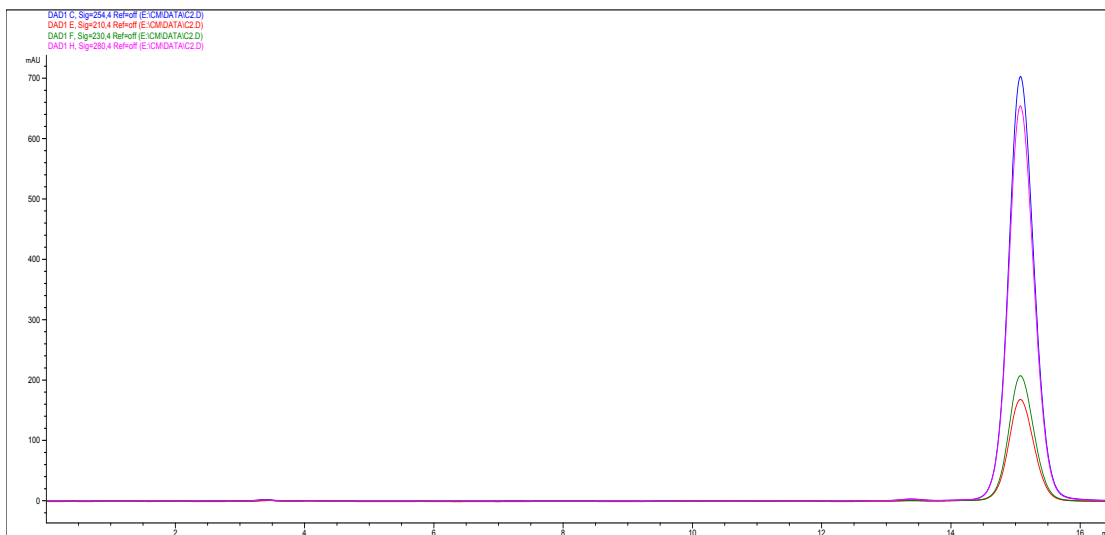


CH<sub>3</sub>OH-H<sub>2</sub>O, 95:5 Rt: 14.39 min

**Fig. S35** HPLC analysis of **1**

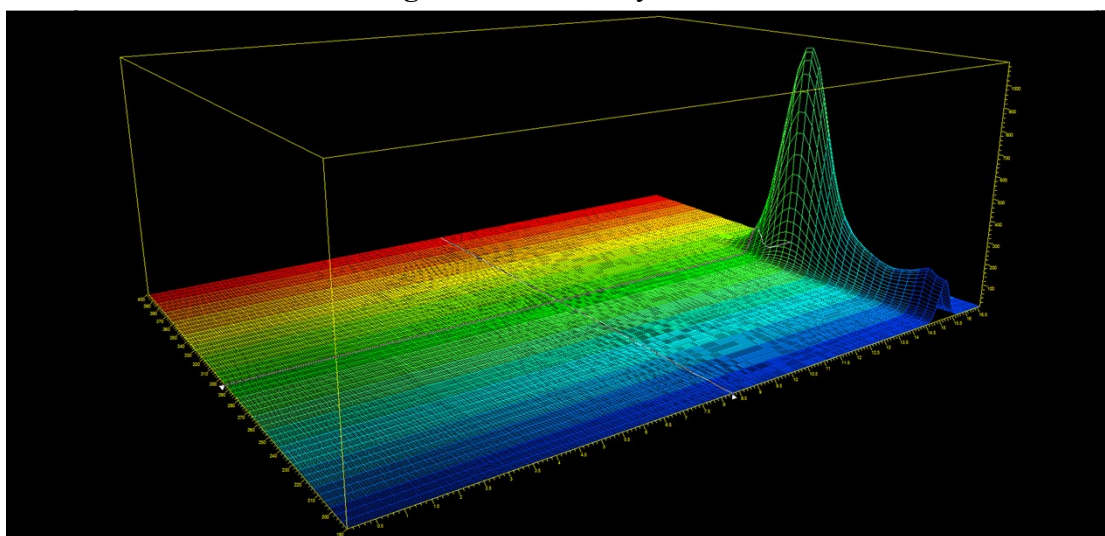


**Fig. S36** HPLC analysis 3D spectrum of **1**

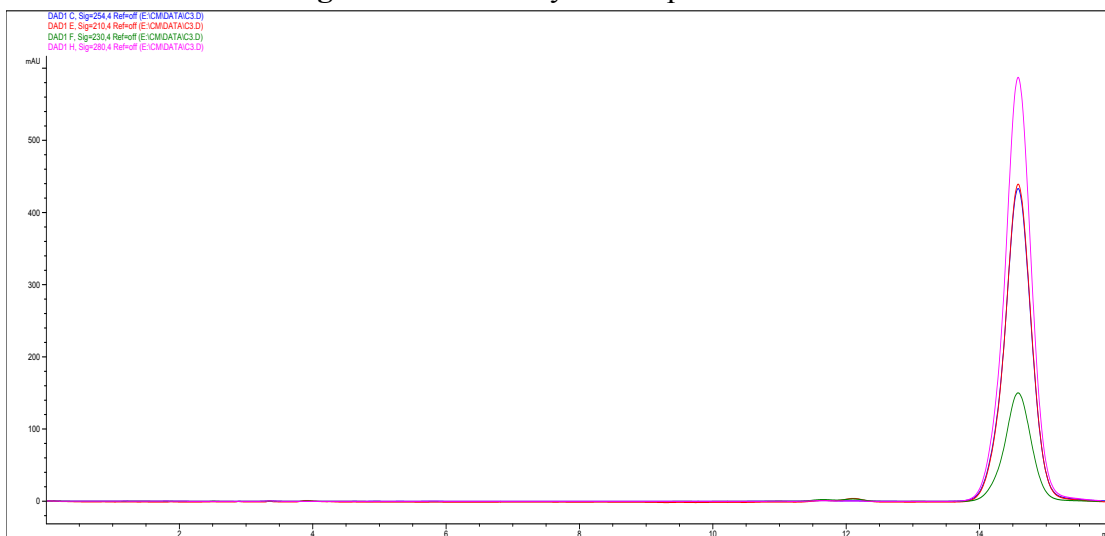


CH<sub>3</sub>OH-H<sub>2</sub>O, 95:5 Rt: 15.08 min

**Fig. S37** HPLC analysis of **2**



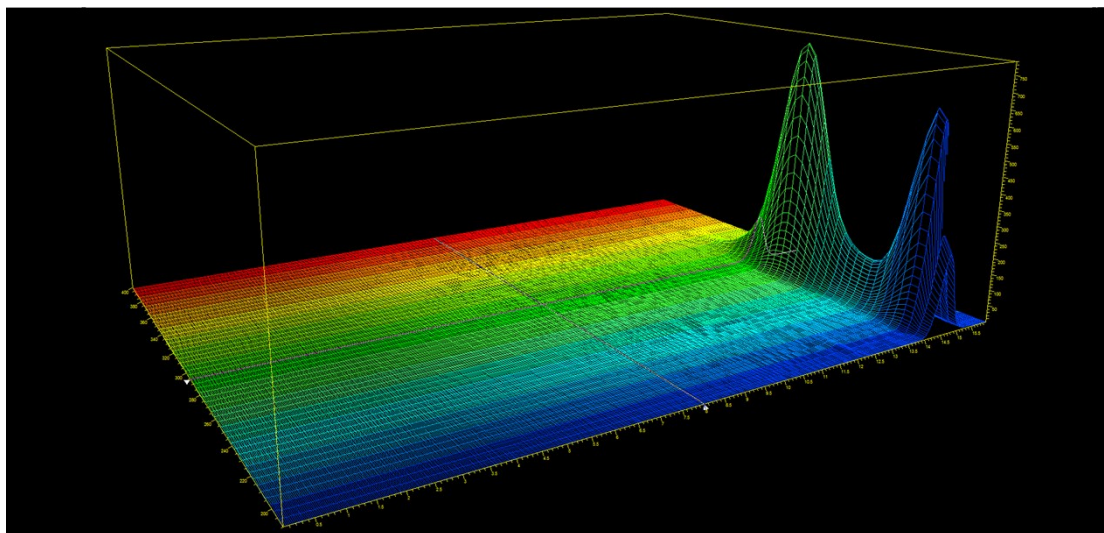
**Fig. S38** HPLC analysis 3D spectrum of **2**



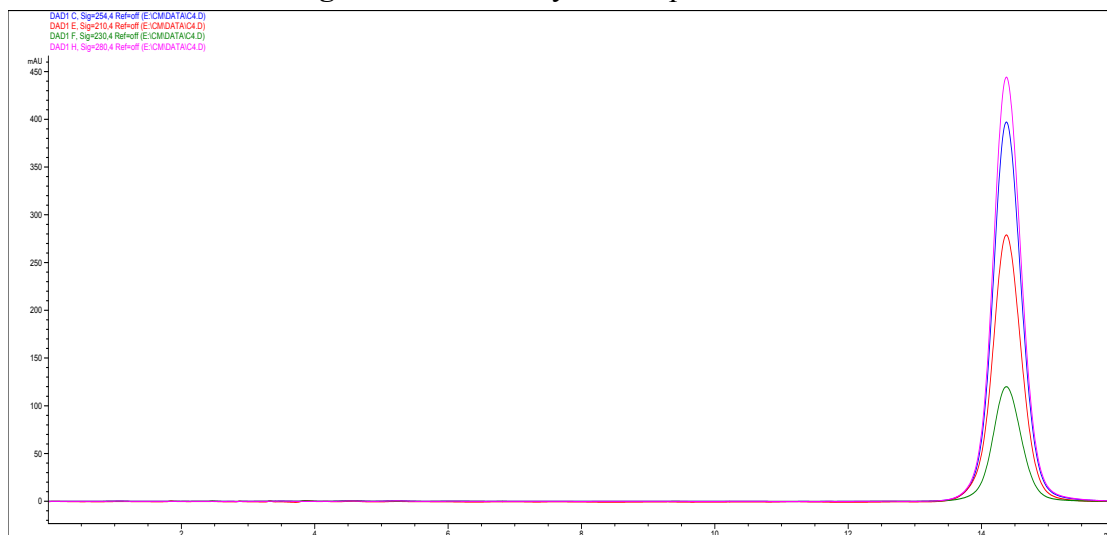
CH<sub>3</sub>OH-H<sub>2</sub>O, 95:5 Rt: 14.85 min

**Fig. S39** HPLC analysis of **3**



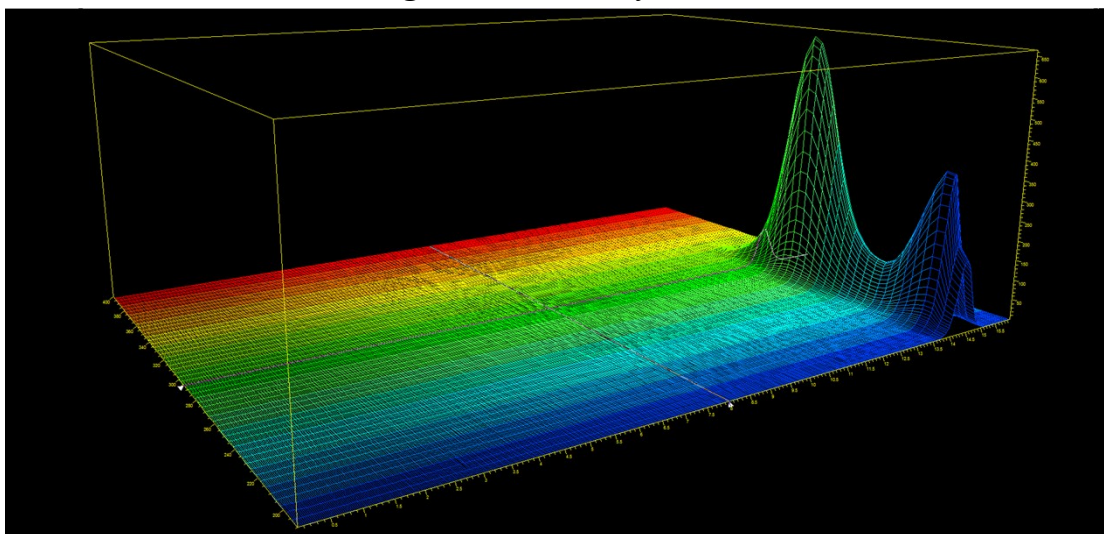


**Fig. S40** HPLC analysis 3D spectrum of **3**

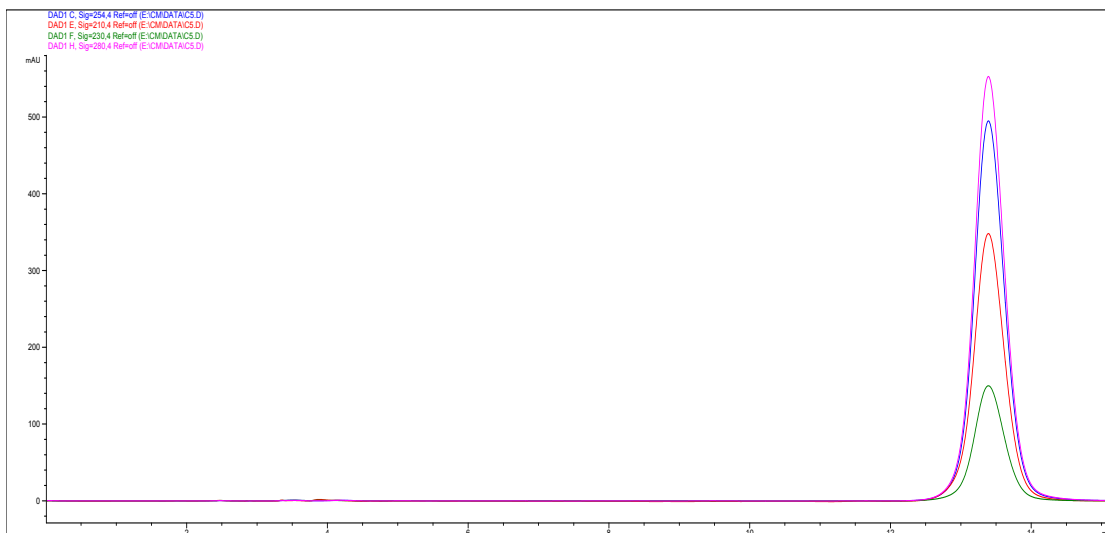


CH<sub>3</sub>OH-H<sub>2</sub>O, 90:10 Rt: 14.37 min

**Fig. S41** HPLC analysis of **4**

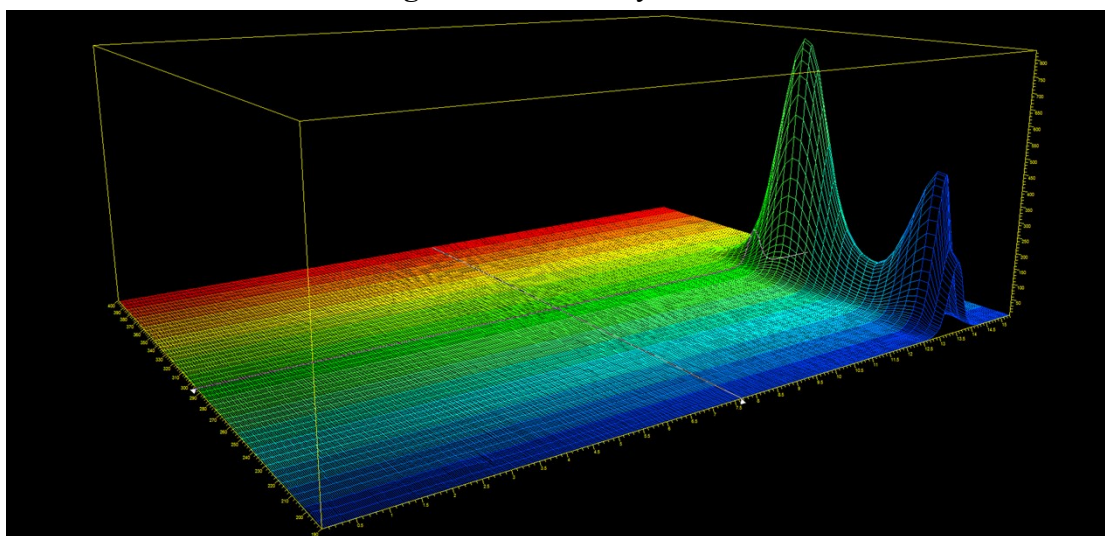


**Fig. S42** HPLC analysis 3D spectrum of **4**

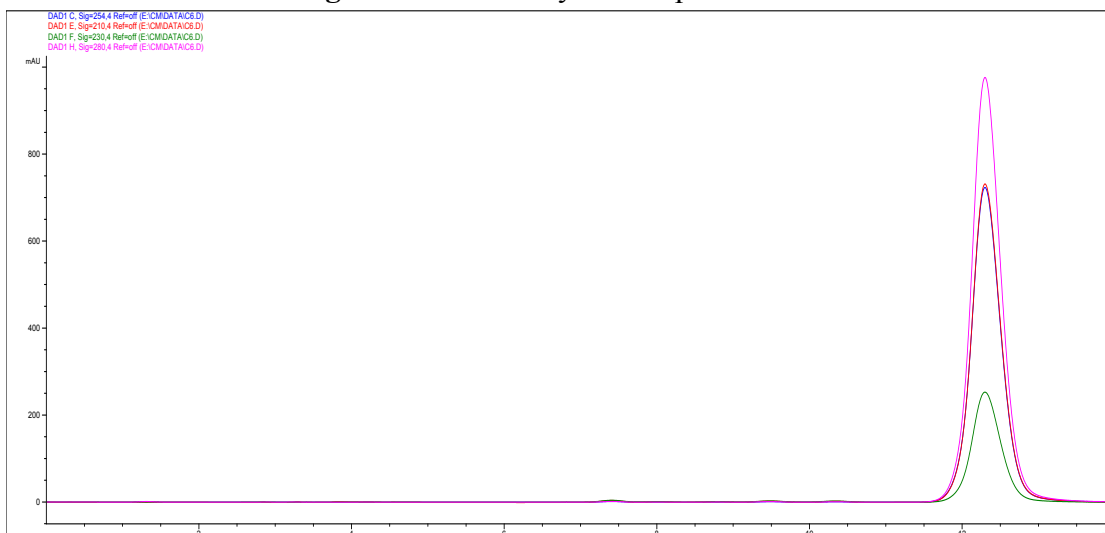


CH<sub>3</sub>OH-H<sub>2</sub>O, 92:8 Rt: 13.39 min

**Fig. S43 HPLC analysis of 5**

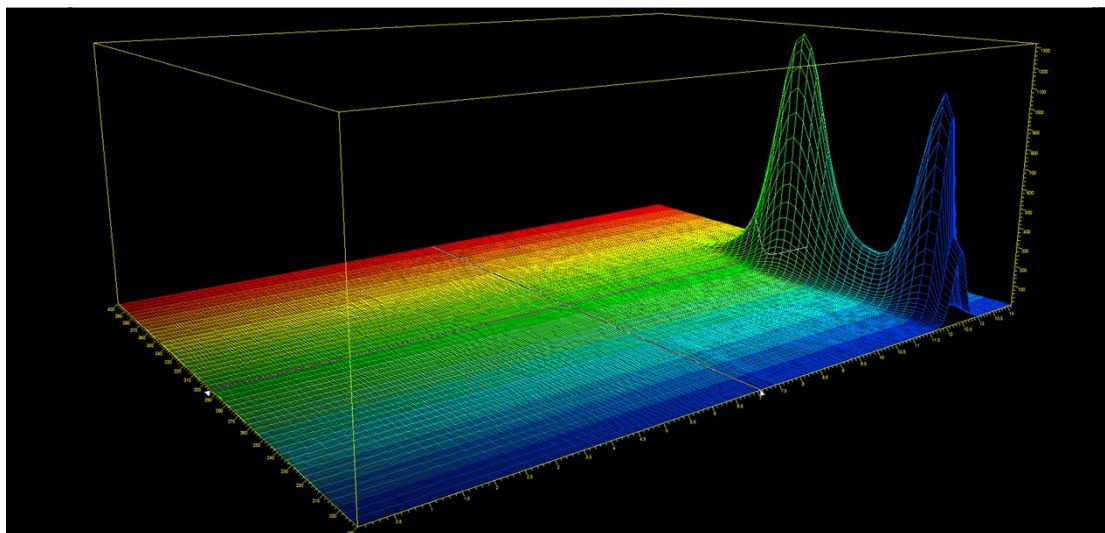


**Fig. S44 HPLC analysis 3D spectrum of 5**

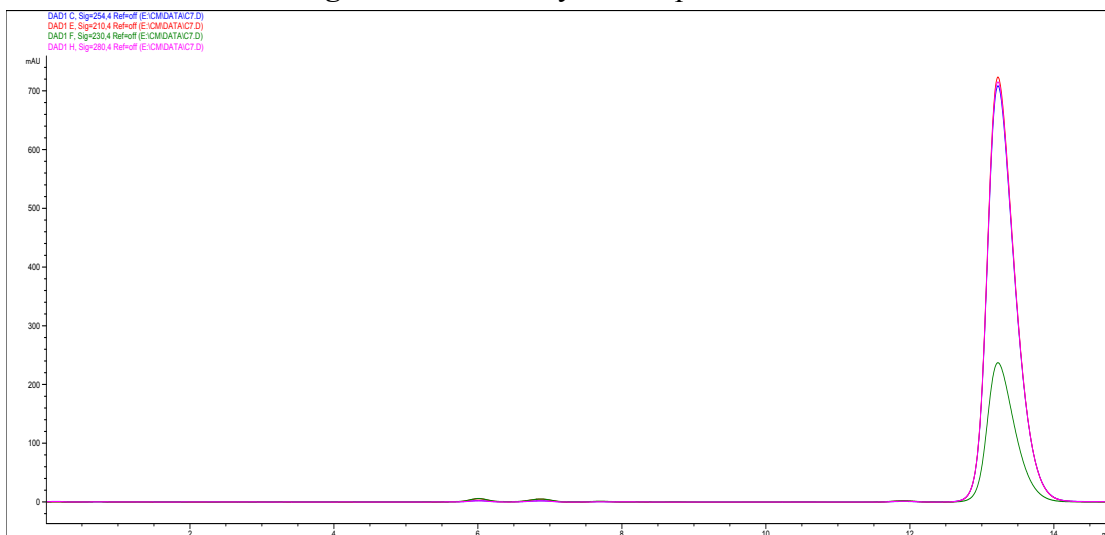


CH<sub>3</sub>OH-H<sub>2</sub>O, 90:10 Rt: 12.30 min

**Fig. S45 HPLC analysis of 6**

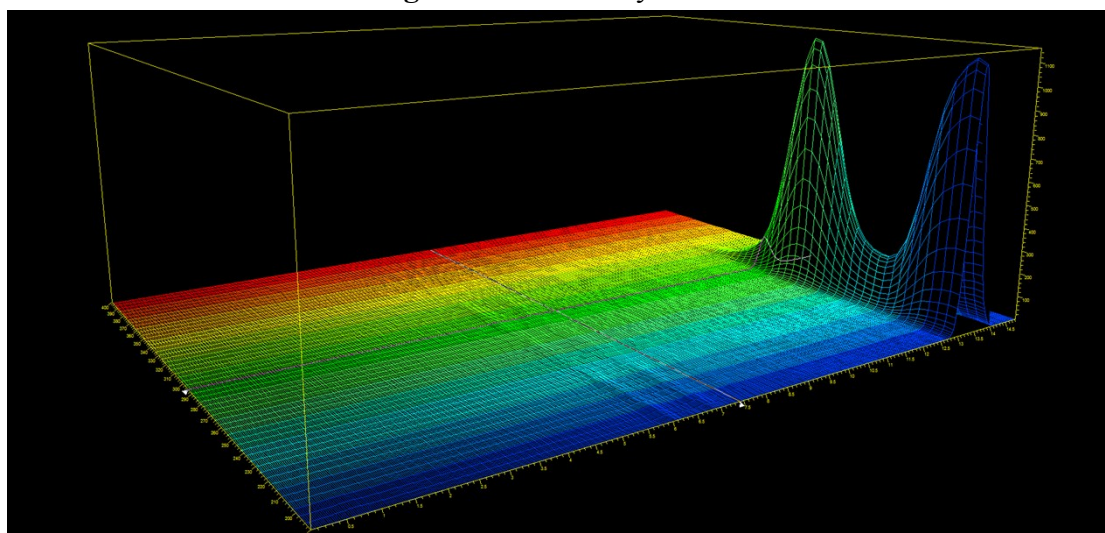


**Fig. S46** HPLC analysis 3D spectrum of **6**



CH<sub>3</sub>CN-H<sub>2</sub>O, 92:8 Rt: 13.22 min

**Fig. S47** HPLC analysis of **7**



**Fig. S48** HPLC analysis 3D spectrum of **7**