

Supporting Information

Rec. Nat. Prod. 18:2 (2024) 296-301

A New Coumarin from the Roots of *Toddalia asiatica*

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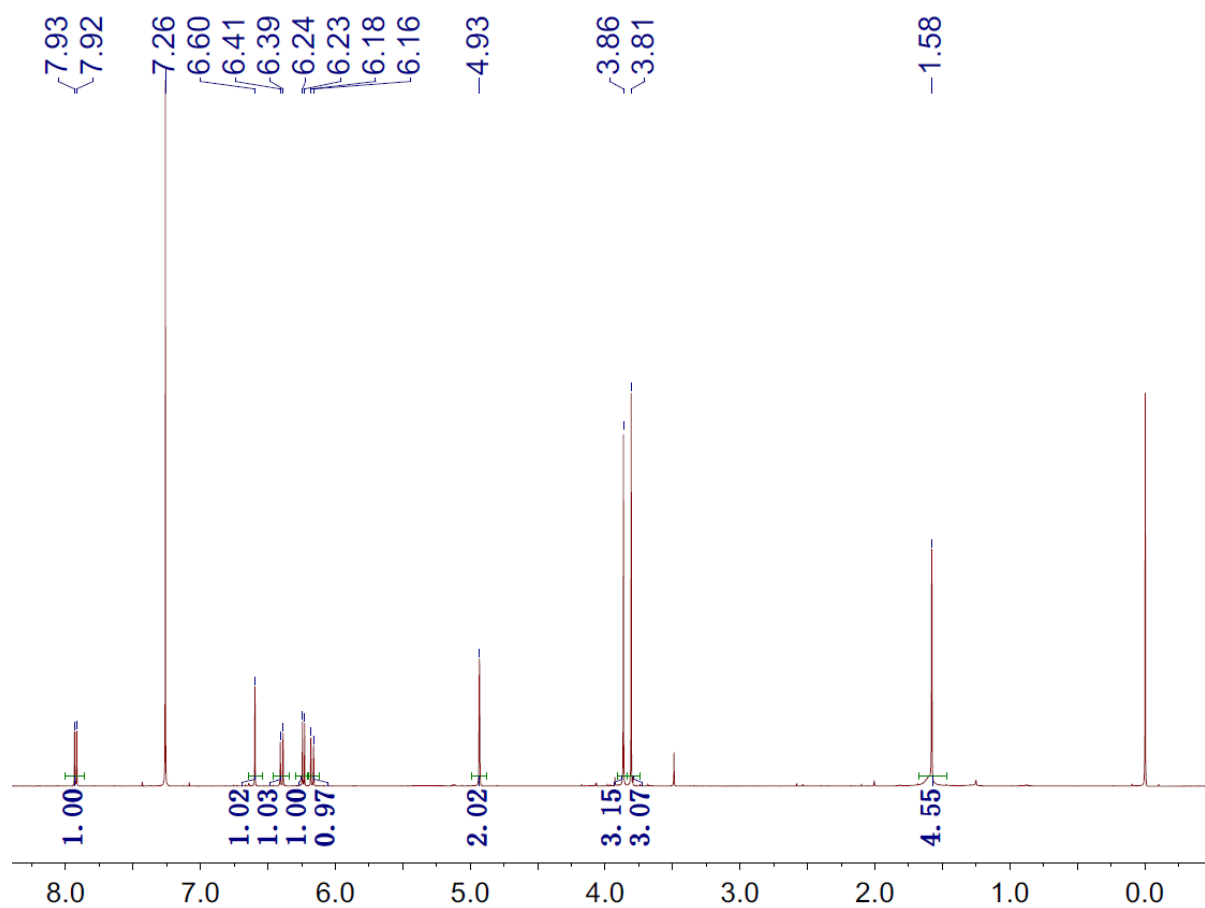


Figure S1: ^1H NMR spectrum of **1** in CDCl_3 at 600 MHz

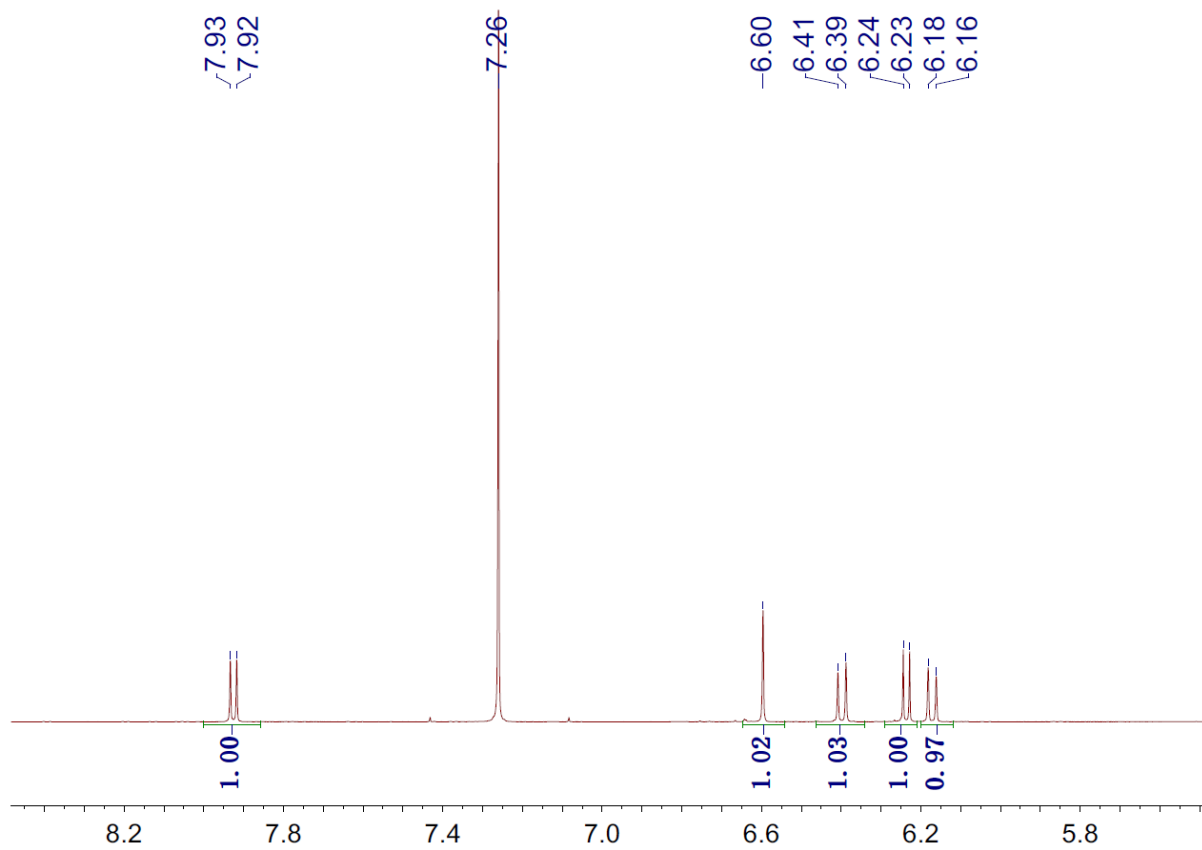


Figure S2: ^1H NMR spectrum of **1** in CDCl_3 at 600 MHz (From δ_{C} 5.5 ppm to 8.5 ppm)

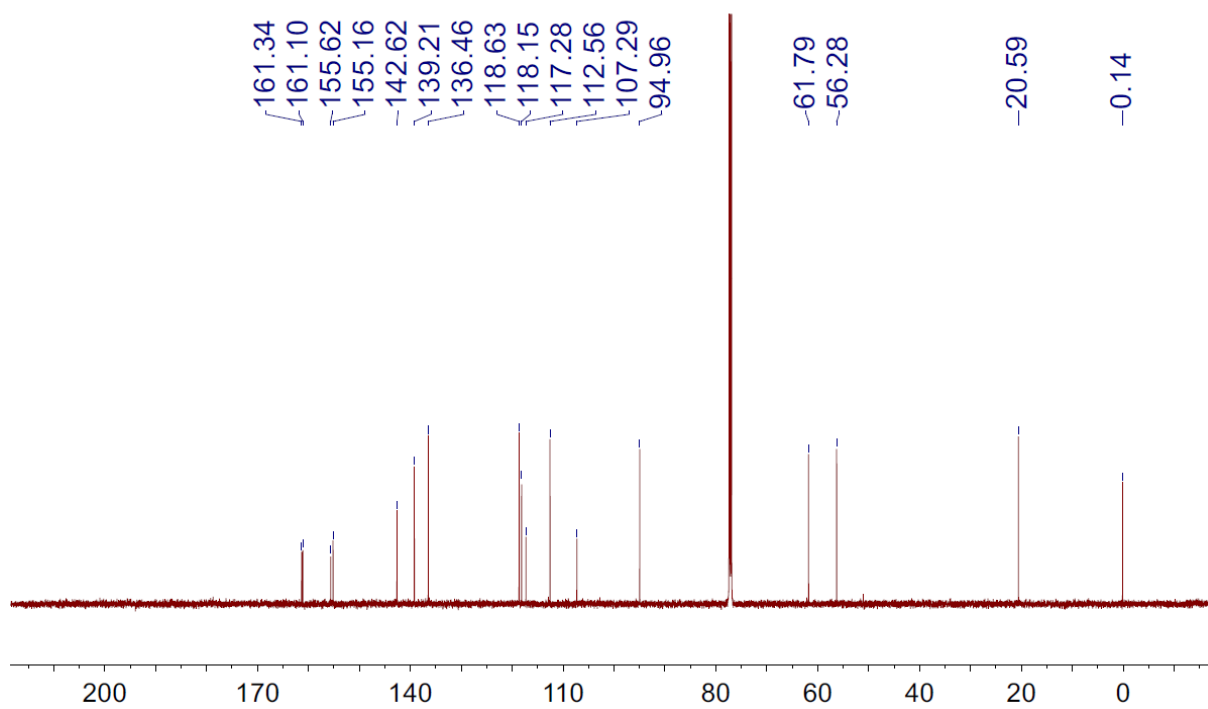


Figure S3: ^{13}C -NMR spectrum of **1** in CDCl_3 at 150 MHz

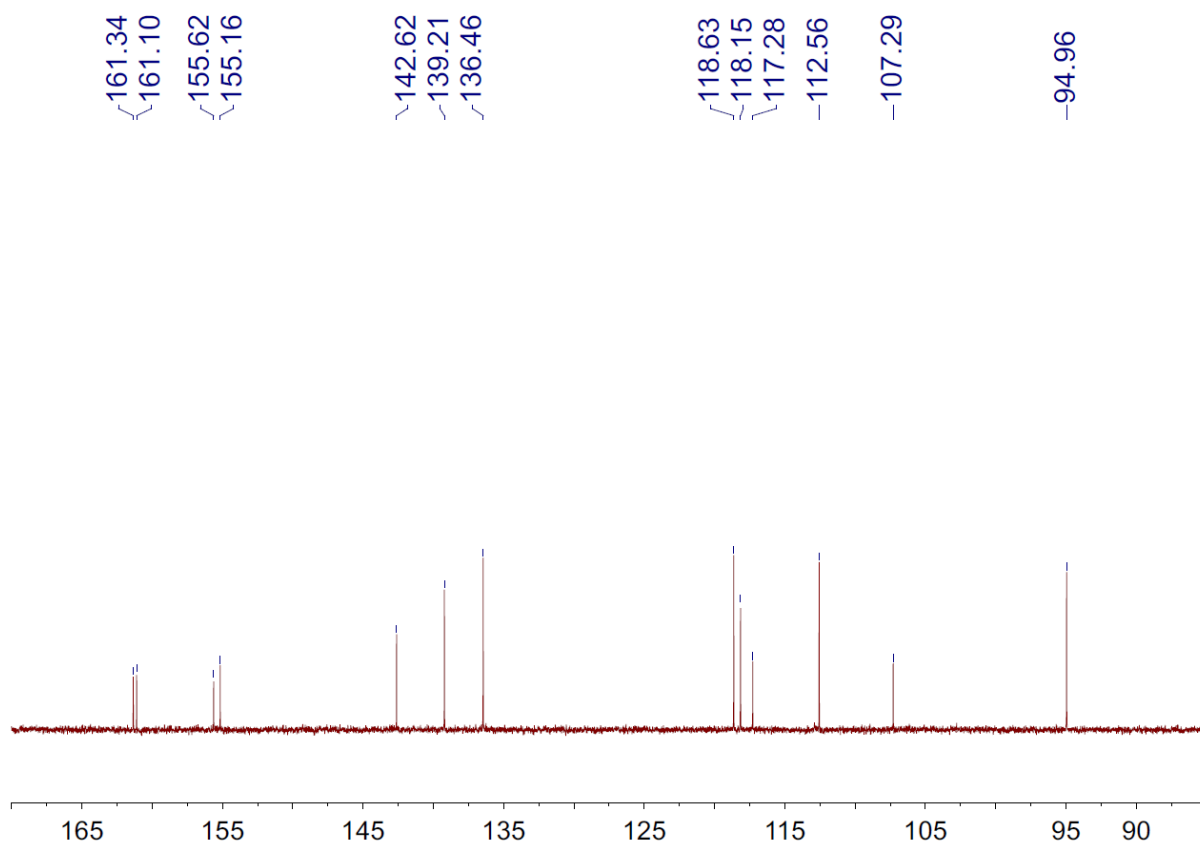


Figure S4: ^{13}C -NMR spectrum of **1** in CDCl_3 at 150 MHz (From δ_{C} 85 ppm to 170 ppm)

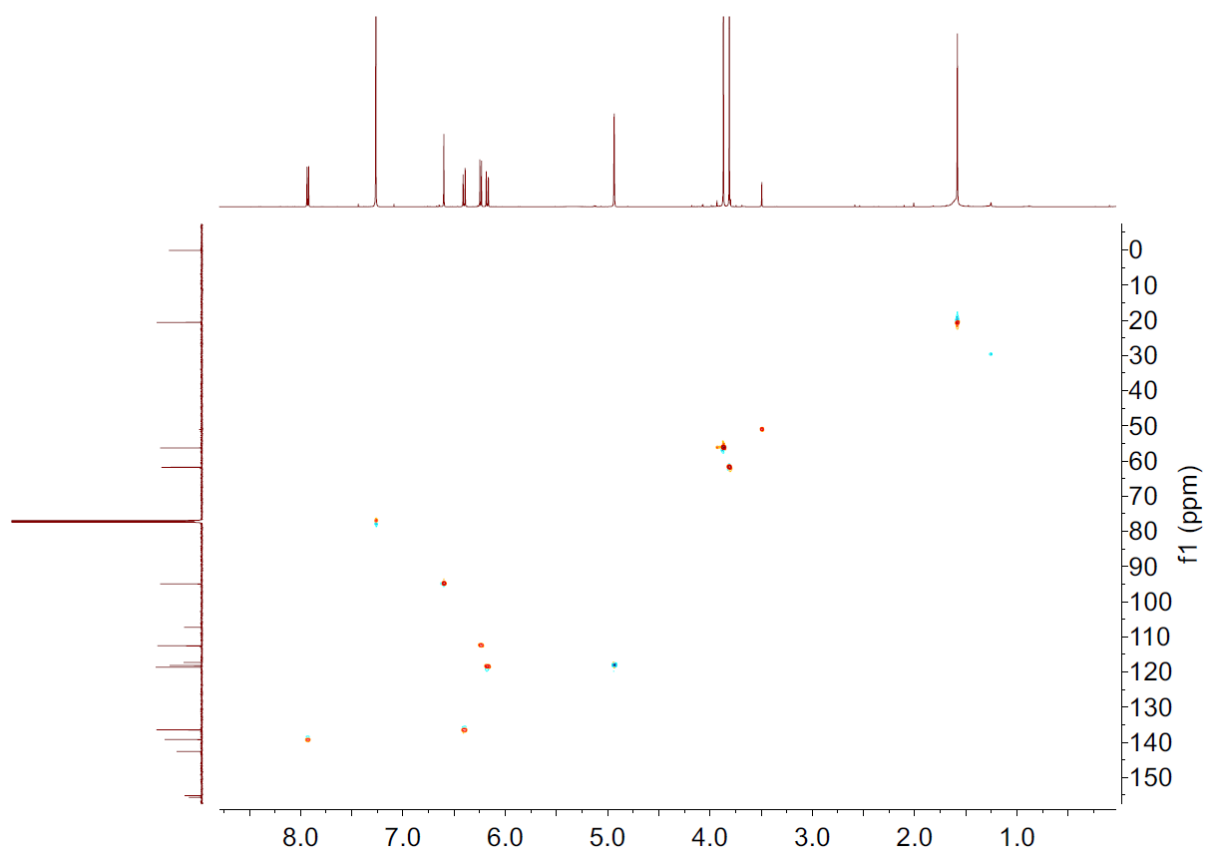


Figure S5: HSQC spectrum of **1**

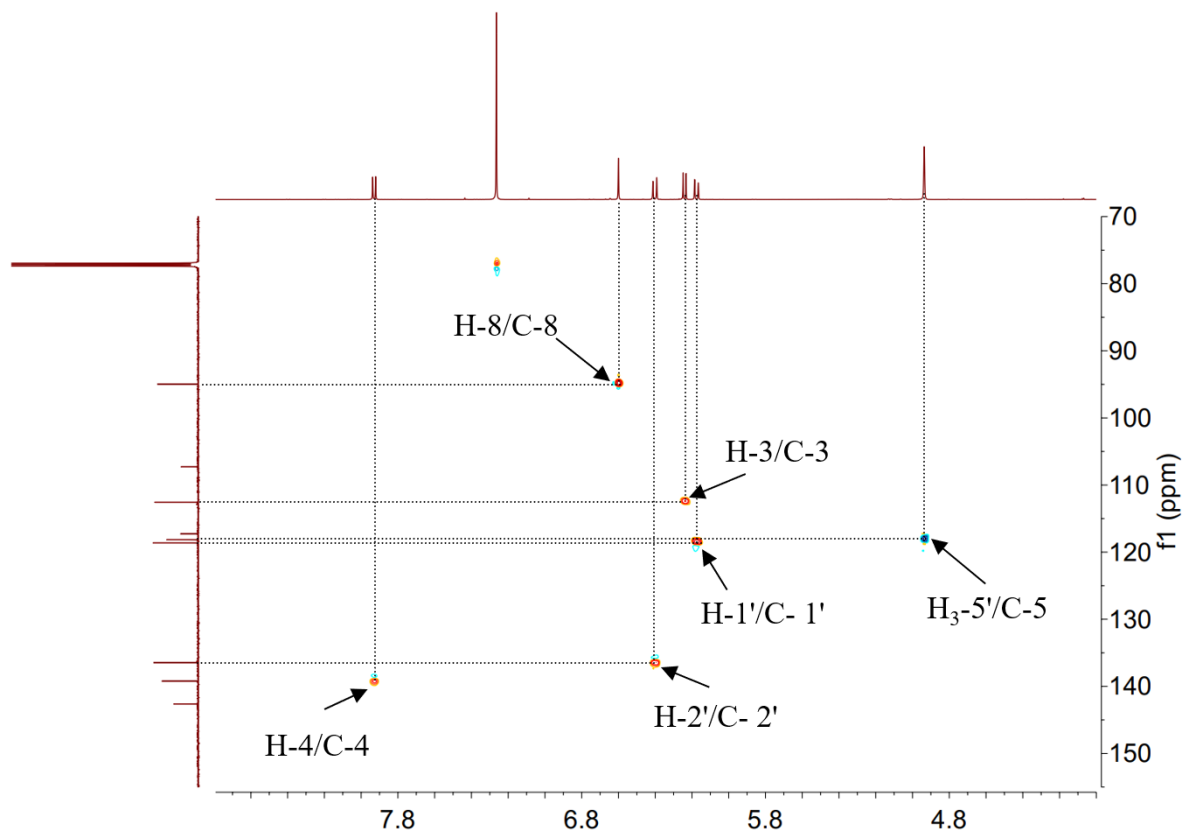


Figure S6: HSQC spectrum of **1** (From δ_{C} 70 ppm to 155 ppm)

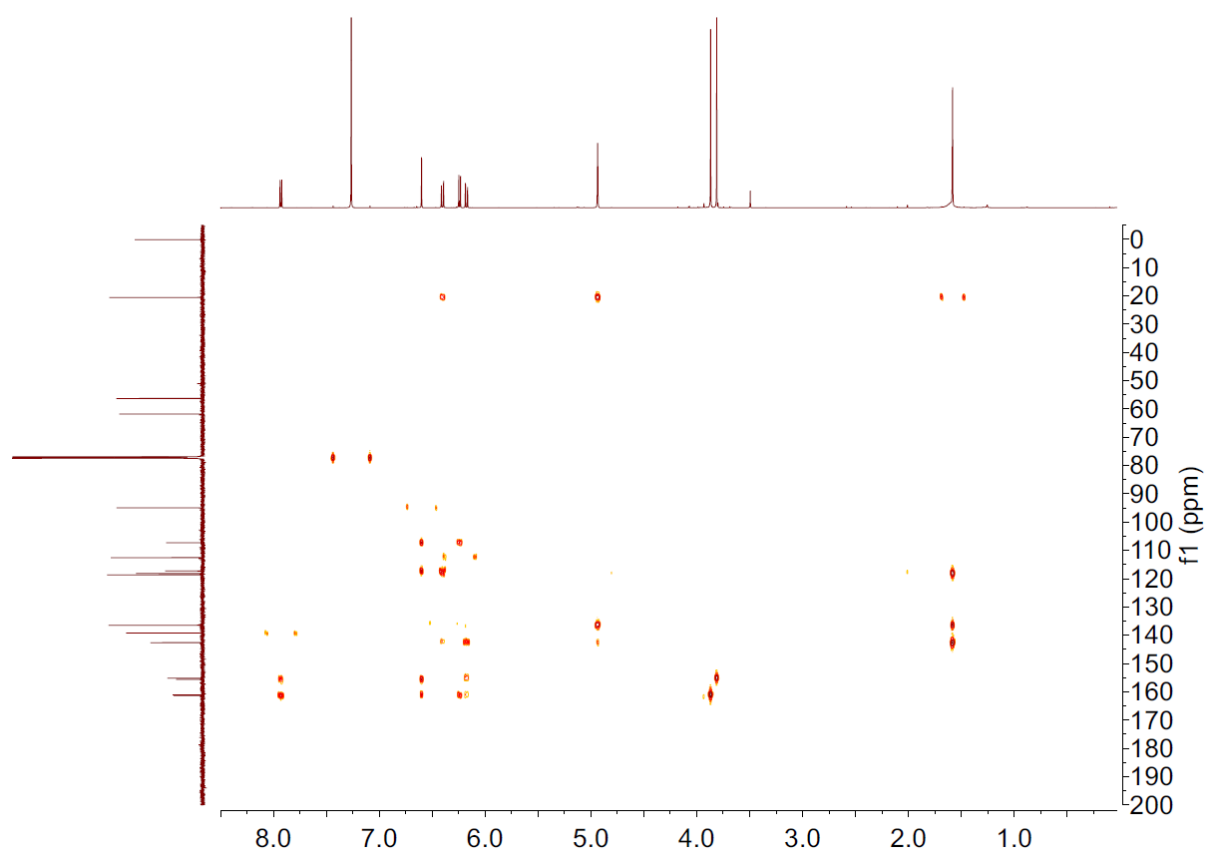


Figure S7: HMBC spectrum of **1**

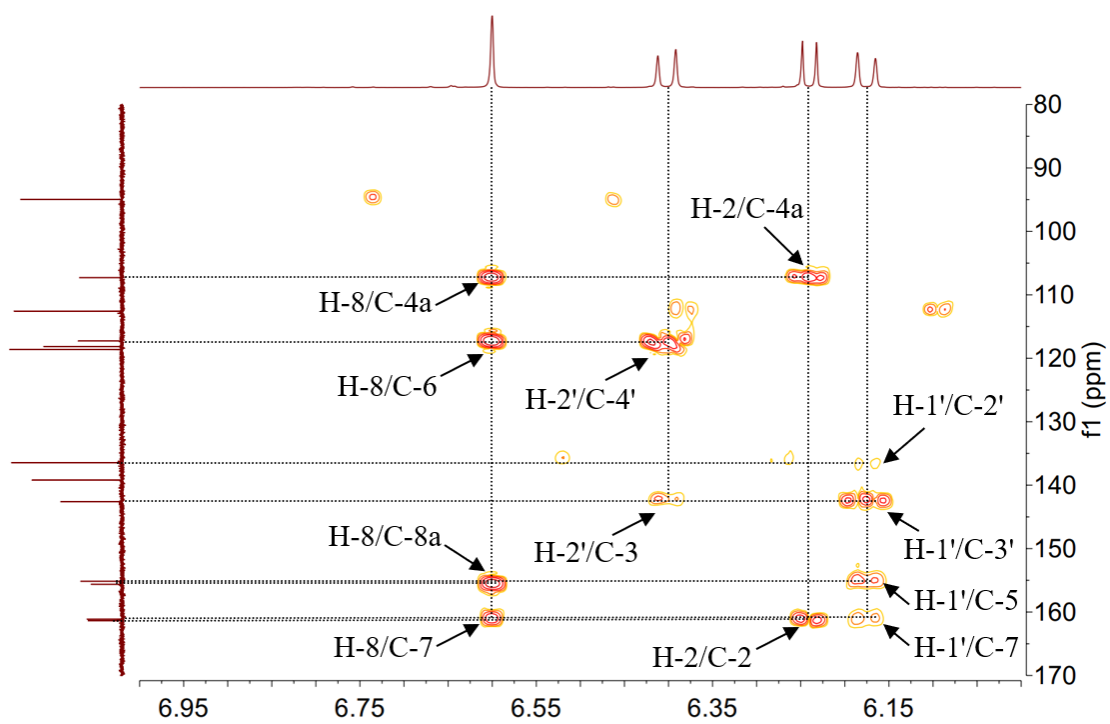


Figure S8: HMBC spectrum of **1** (From δ_C 80 ppm to 170 ppm)

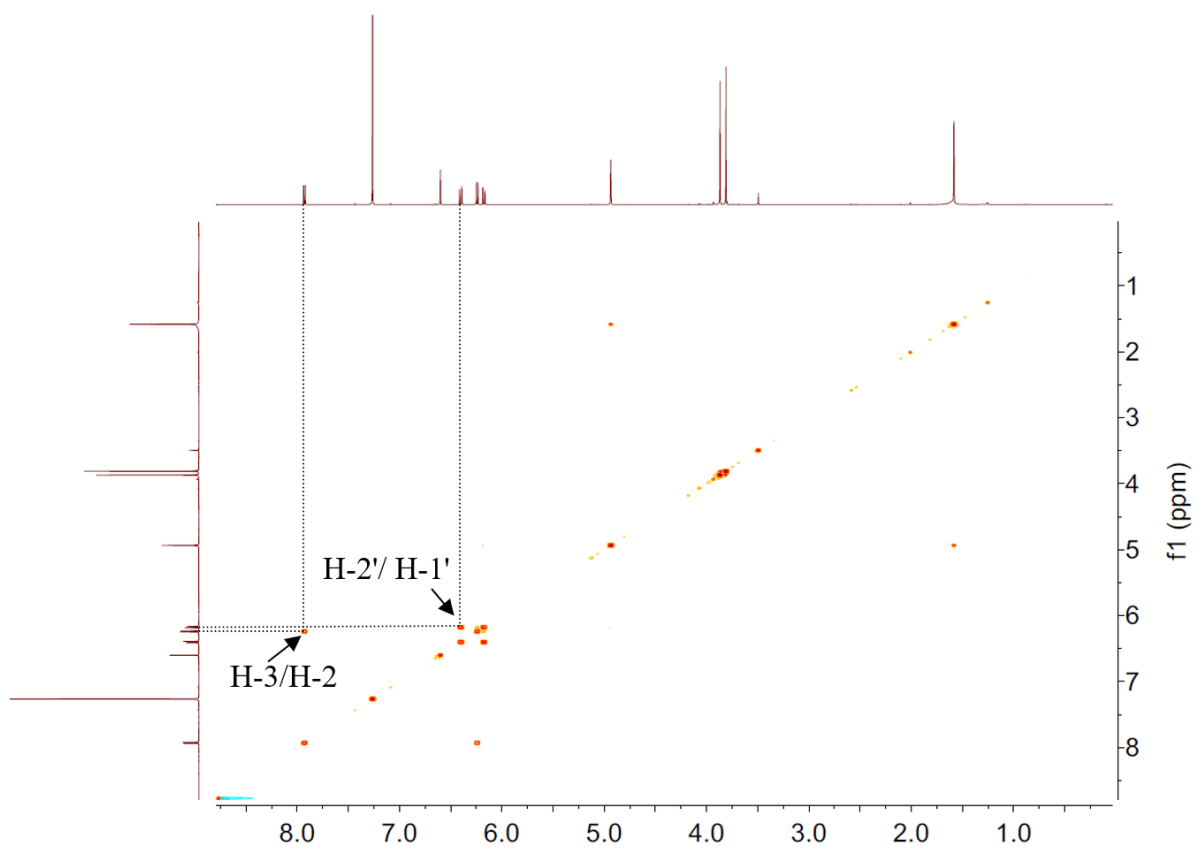


Figure S9: ^1H - ^1H COSY spectrum of **1**

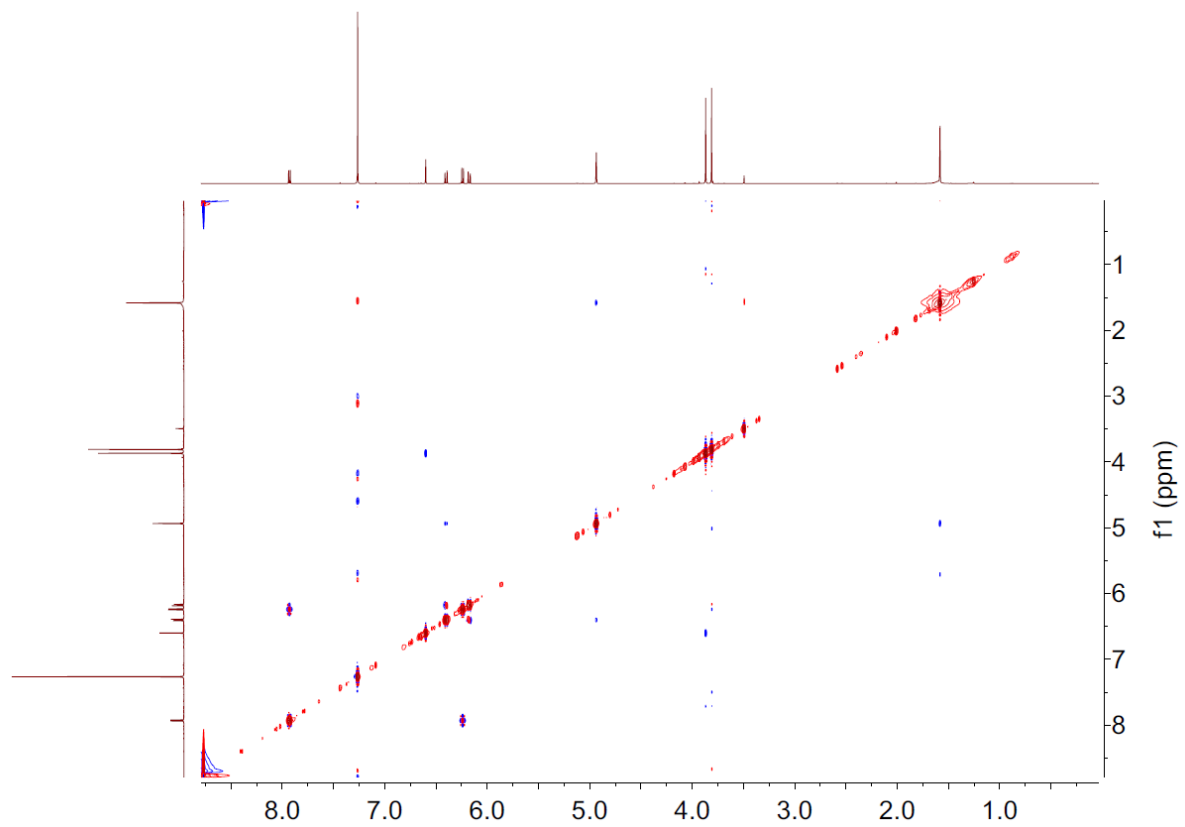


Figure S10: ROESY spectrum of **1**

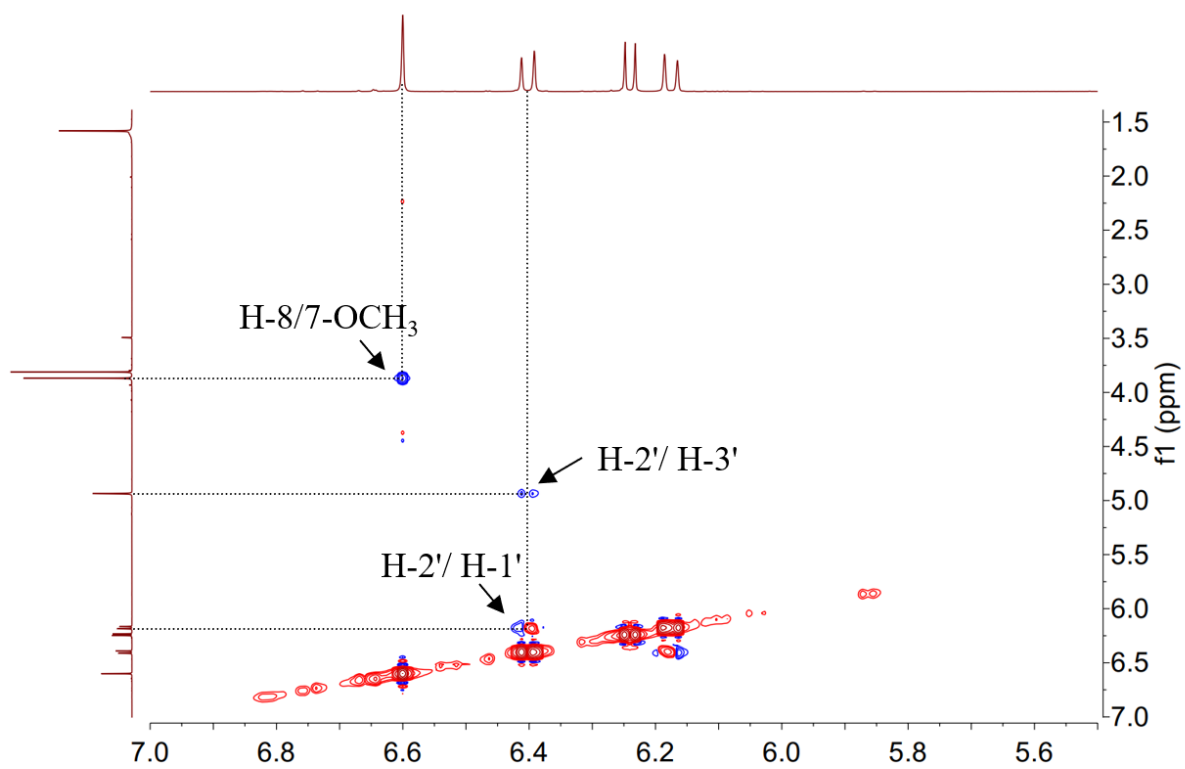


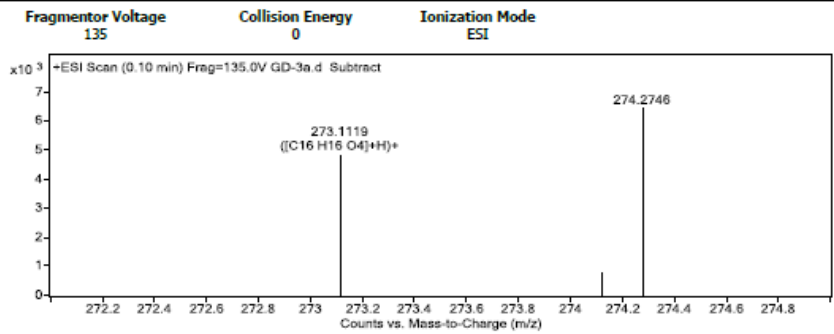
Figure S11: ROESY spectrum of **1** (From δ_c 5.5 ppm to 7.0 ppm)

Qualitative Analysis Report

Data Filename	GD-3a.d	Sample Name	GD-3a
Sample Type	Sample	Position	P1-A7
Instrument Name	Instrument 1	User Name	
Acq Method	s.m	Acquired Time	4/15/2022 4:29:18 PM
IRM Calibration Status	Success	DA Method	PCDL.m
Comment			

Sample Group	Info.
Acquisition SW	6200 series TOF/6500 series
Version	Q-TOF B.05.01 (B5125.2)

User Spectra



Peak List

m/z	z	Abund	Formula	Ion
81.5207	1	1527.6		
94.0454	1	1598.04		
241.0858	1	5223.42		
273.1119	1	4862.71	C16 H16 O4	(M+H)+
274.2746	1	6434.04		
275.0923	1	5187.49		
276.0955	1	1829.45		
318.2998	1	5097.9		
460.269	1	1489.63		
654.3335	1	3005.94		

Formula Calculator Element Limits

Element	Min	Max
C	3	60
H	0	120
O	0	30

Formula Calculator Results

Formula	CalculatedMass	CalculatedMz	Mz	Diff. (mDa)	Diff. (ppm)	DBE
C16 H16 O4	272.1049	273.1121	273.1119	0.20	0.73	9.0000

--- End Of Report ---

Figure S12: HR-ESI-MS spectrum of 1

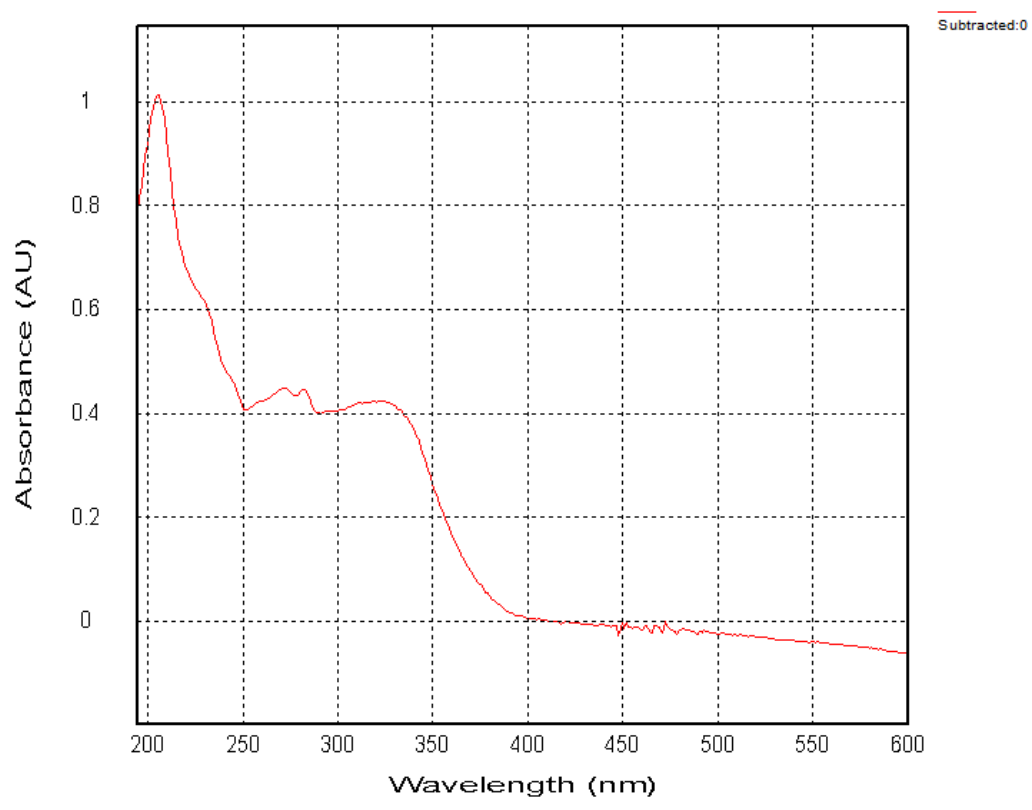
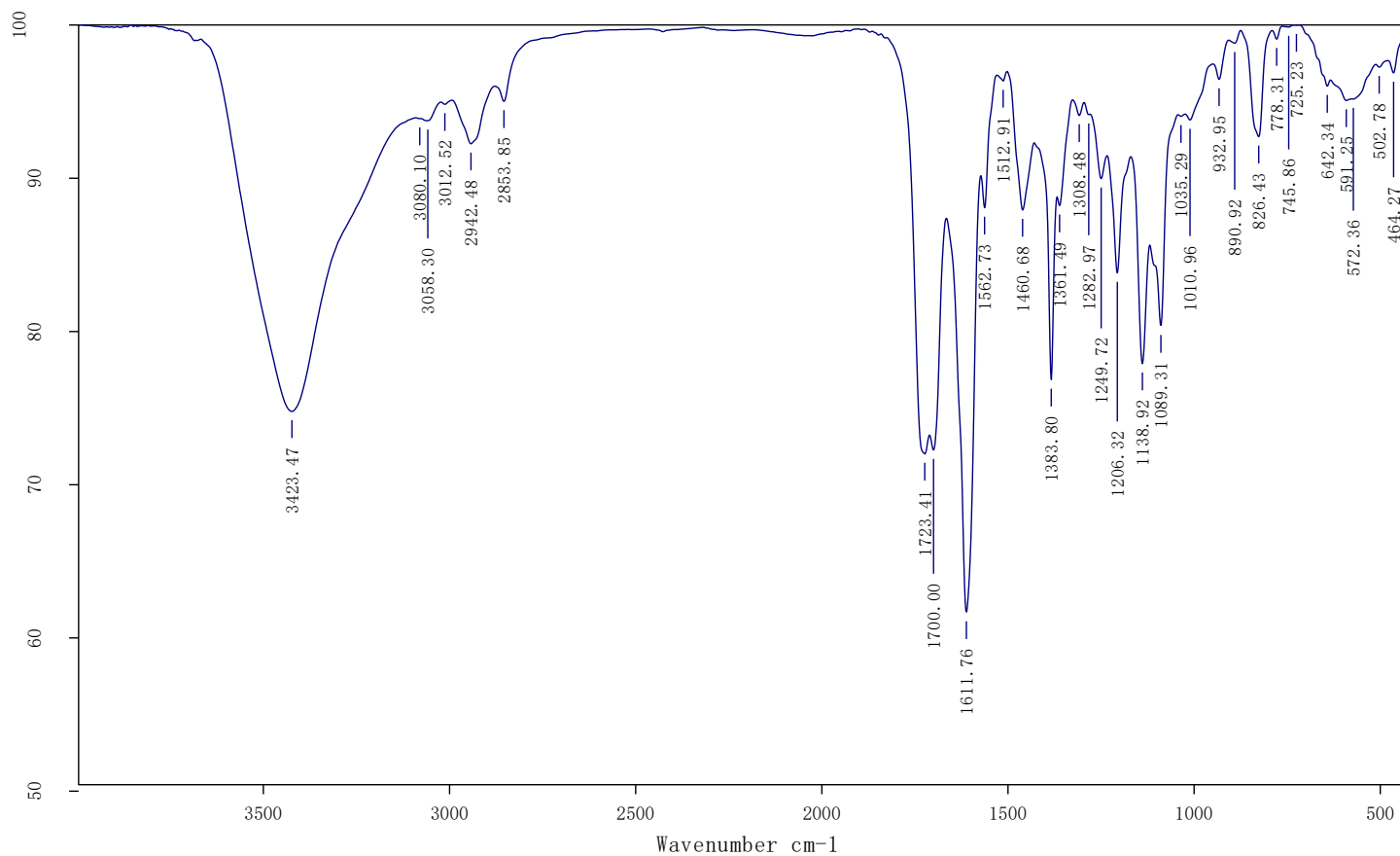


Figure S13: UV spectrum of **1**



Sample Name: GD-3a
 Sample Form: KBr
 Path of File: E:\data
 Date of Measurement: 2022/4/15

Resolution: 4
 Aperture Setting: 6 mm
 Number of Background Scans: 16
 Number of Sample Scans: 16

Beamsplitter Setting: KBr
 Source Setting: MIR
 Instrument Type: BRUKER VERTEX 70
 Soft Version: OPUS8.1

Figure S14: IR spectrum of 1

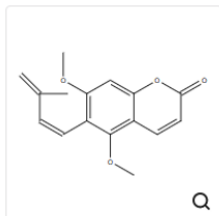
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667 Searches



December 25, 2023

🔍 Substances
11:21 PM



As Drawn (2)
Substructure (18)
Similarity (25K)

Rerun Search

Edit Search

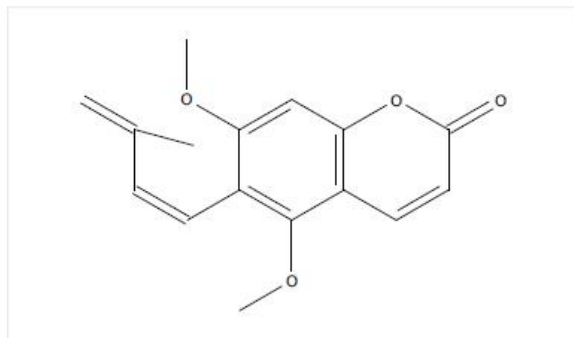
Figure S15: Scifinder search report of new compound

Initiating Search


January 5, 2024, 2:51PM

Substances:

Filtered By:

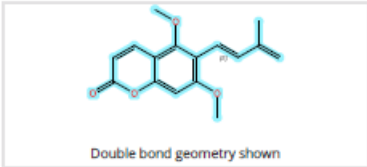



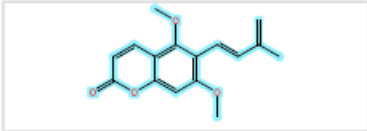



Structure Match: **As Drawn**

Search Tasks

Task	Search Type	View
Exported: Returned Substance Results + Filters (2)	 Substances	View Results

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Substances (2)[View in SciFinder®](#)

1													
<p>1810706-07-0</p>  <p>Double bond geometry shown</p> <p>C₁₆H₁₆O₄ 5,7-Dimethoxy-6-[(1E)-3-methyl-1,3-butadien-1-yl]-2H-1-benzopyran-2-one</p> <p>  2 References  0 Reactions  2 Suppliers </p>	<table border="1"> <thead> <tr> <th>Key Physical Properties</th> <th>Value</th> <th>Condition</th> </tr> </thead> <tbody> <tr> <td>Molecular Weight</td> <td>272.30</td> <td>-</td> </tr> <tr> <td>Boiling Point (Predicted)</td> <td>466.7±45.0 °C</td> <td>Press: 760 Torr</td> </tr> <tr> <td>Density (Predicted)</td> <td>1.163±0.06 g/cm³</td> <td>Temp: 20 °C; Press: 760 Torr</td> </tr> </tbody> </table>	Key Physical Properties	Value	Condition	Molecular Weight	272.30	-	Boiling Point (Predicted)	466.7±45.0 °C	Press: 760 Torr	Density (Predicted)	1.163±0.06 g/cm ³	Temp: 20 °C; Press: 760 Torr
Key Physical Properties	Value	Condition											
Molecular Weight	272.30	-											
Boiling Point (Predicted)	466.7±45.0 °C	Press: 760 Torr											
Density (Predicted)	1.163±0.06 g/cm ³	Temp: 20 °C; Press: 760 Torr											
2													
<p>2581827-30-5</p>  <p>C₁₆H₁₆O₄ 5,7-Dimethoxy-6-(3-methyl-1,3-butadien-1-yl)-2H-1-benzopyran-2-one</p> <p>  1 Reference  0 Reactions  0 Suppliers </p>	<table border="1"> <thead> <tr> <th>Key Physical Properties</th> <th>Value</th> <th>Condition</th> </tr> </thead> <tbody> <tr> <td>Molecular Weight</td> <td>272.30</td> <td>-</td> </tr> <tr> <td>Boiling Point (Predicted)</td> <td>466.7±45.0 °C</td> <td>Press: 760 Torr</td> </tr> <tr> <td>Density (Predicted)</td> <td>1.163±0.06 g/cm³</td> <td>Temp: 20 °C; Press: 760 Torr</td> </tr> </tbody> </table>	Key Physical Properties	Value	Condition	Molecular Weight	272.30	-	Boiling Point (Predicted)	466.7±45.0 °C	Press: 760 Torr	Density (Predicted)	1.163±0.06 g/cm ³	Temp: 20 °C; Press: 760 Torr
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Figure S16: Scifinder similarity report for new compound

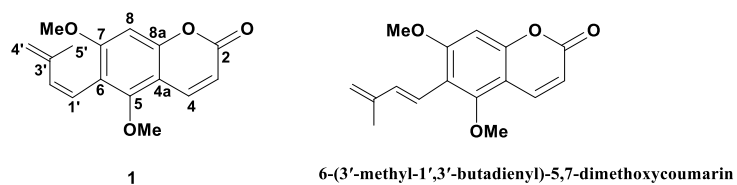


Figure S17: Structure of 6-(3'-methyl-1',3'-butadienyl)-5,7-dimethoxycoumarin which is the most similar to compound **1** according to Scifinder search

Table S1. ^1H and ^{13}C NMR data for **1** and similar compound 6-(3'-methyl-1',3'-butadienyl)-5,7-dimethoxycoumarin (**11**) (δ in ppm, J in Hz).

Position	1		6-(3'-methyl-1',3'-butadienyl)-5,7-dimethoxycoumarin (11)	
	δ_{H}	δ_{C}	δ_{H}	δ_{C}
2		161.3		161.0
3	6.24 (d, $J = 9.6$ Hz, 1H)	112.6	6.22 (d, $J = 9.6$ Hz, 1H)	112.8
4	7.93 (d, $J = 9.6$ Hz, 1H)	139.2	7.88 (d, $J = 9.6$ Hz, 1H)	138.9
4a		107.3		107.6
5		155.2		155.7
6		117.3		116.6
7		161.1		161.6
8	6.60 (s, 1H)	95.0	6.59 (s, 1H)	95.6
8a		155.6		154.9
1'	6.17 (d, $J = 12.2$ Hz, 1H)	118.6	6.62 (d, $J = 16.8$ Hz, 1H)	118.3
2'	6.40 (d, $J = 12.2$ Hz, 1H)	136.5	7.23 (d, $J = 16.8$ Hz, 1H)	136.9
3'		142.6		143.0
4'	4.93 (s, 2H)	118.2	5.09 (s, 1H), 5.08 (s, 1H)	117.8
5'	1.58 (s, 3H)	20.6	1.97 (s, 3H)	18.3
5-OCH ₃	3.81 (s, 3H)	61.8	3.76 (s, 3H)	62.0
7-OCH ₃	3.86 (s, 3H)	56.3	3.89 (s, 3H)	56.3

The ^1H and ^{13}C NMR data of compound **1** were recorded at BRUKER AVANCE 600 MHz spectrometer, with CDCl_3 as solvent; the ^1H and ^{13}C NMR data of the compound **1'** reported in the literature were recorded on a Varian Mercury plus spectrometer operating at 400 MHz (^1H) and at 100 MHz (^{13}C), with CDCl_3 as solvent.

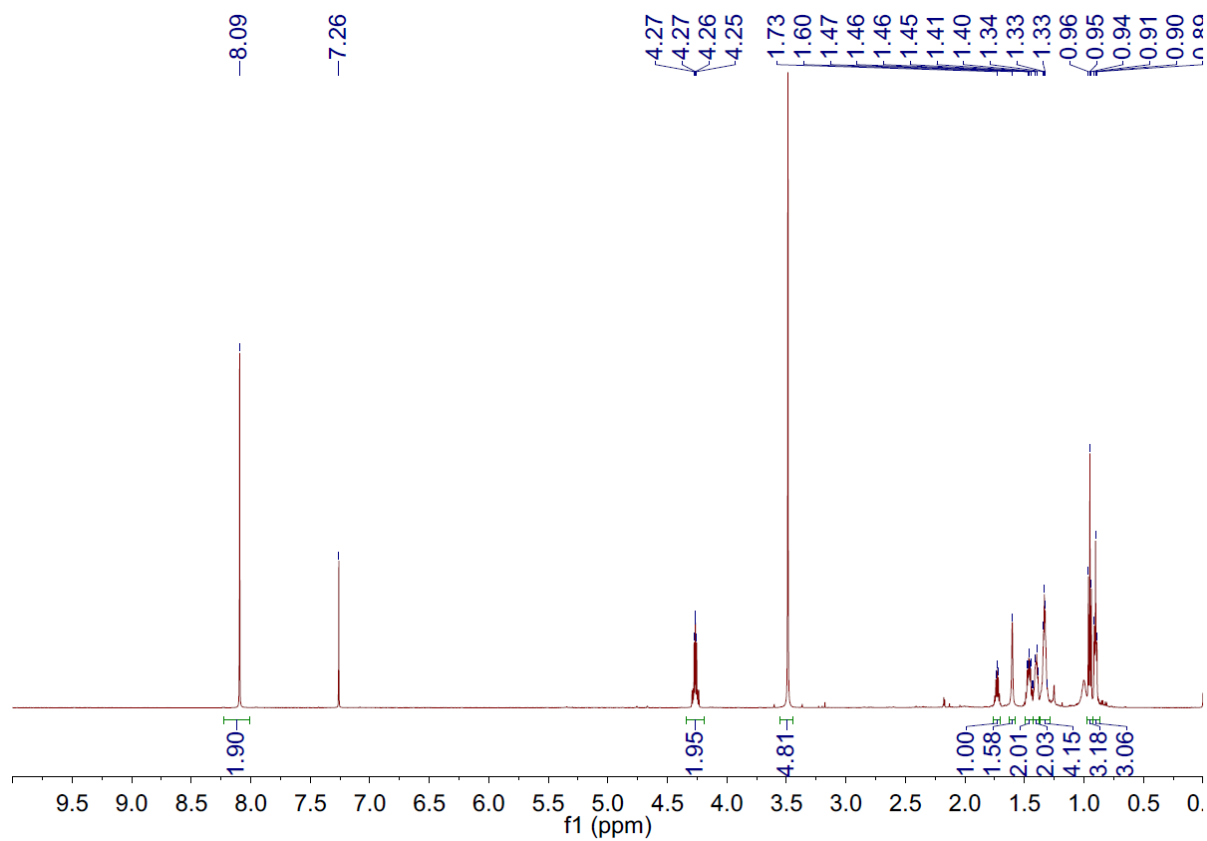


Figure S18: ^1H NMR spectrum of **2** in CDCl_3 at 600 MHz

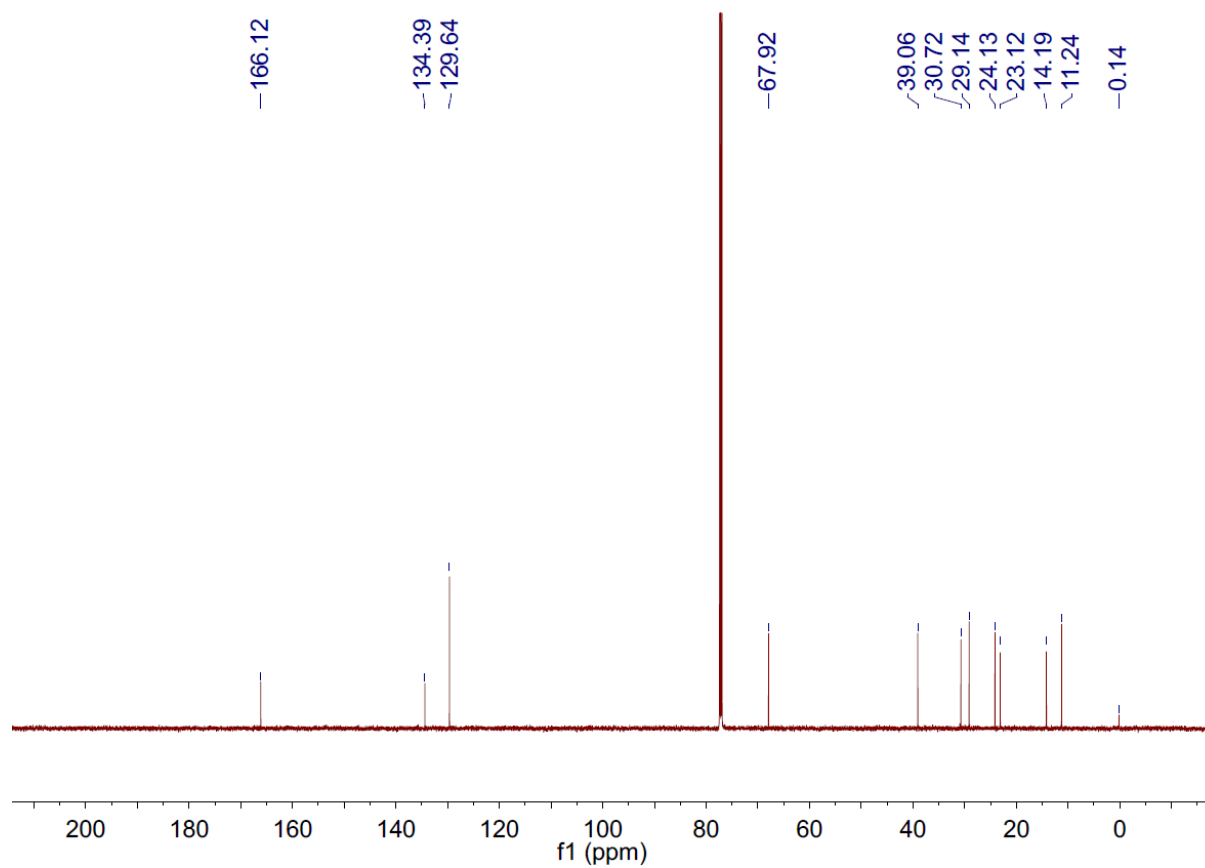


Figure S19: ^{13}C NMR spectrum of **2** in CDCl_3 at 150 MHz

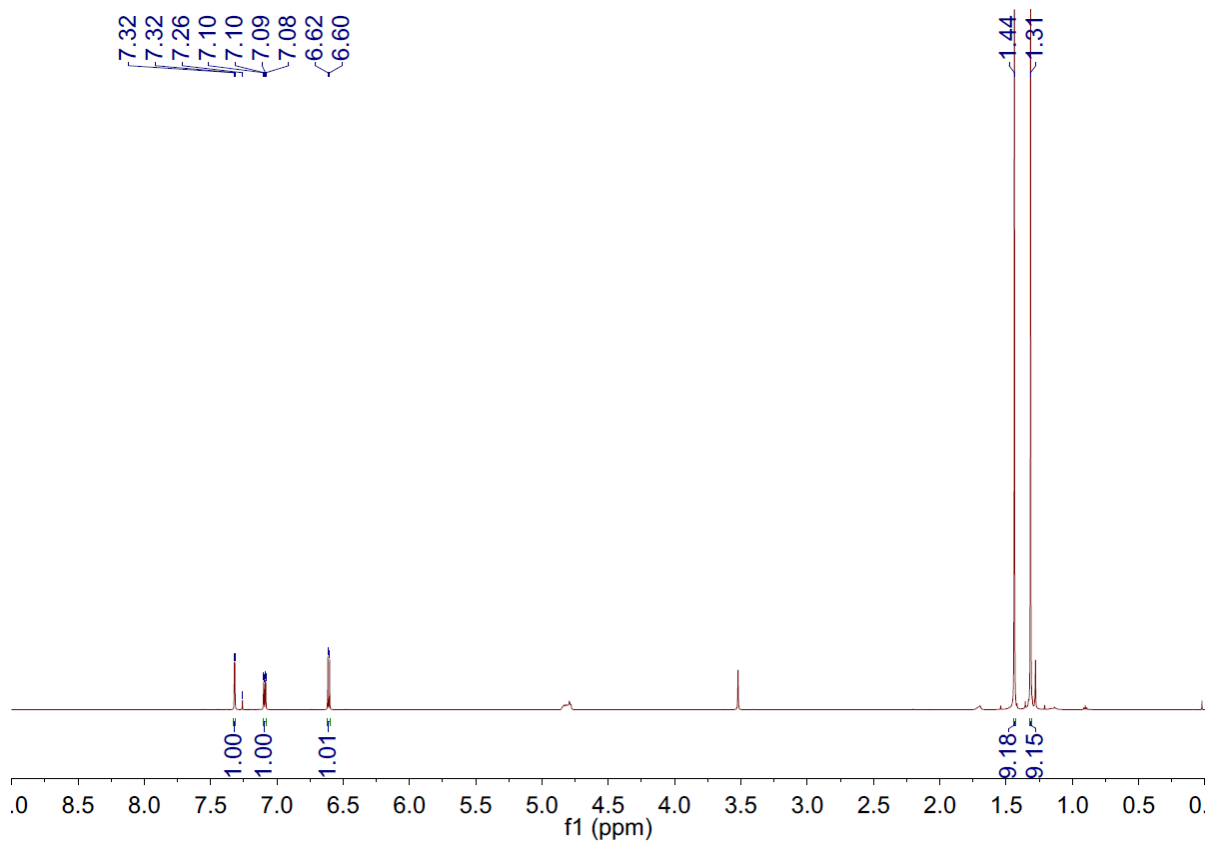


Figure S20: ^1H NMR spectrum of **3** in CDCl_3 at 600 MHz

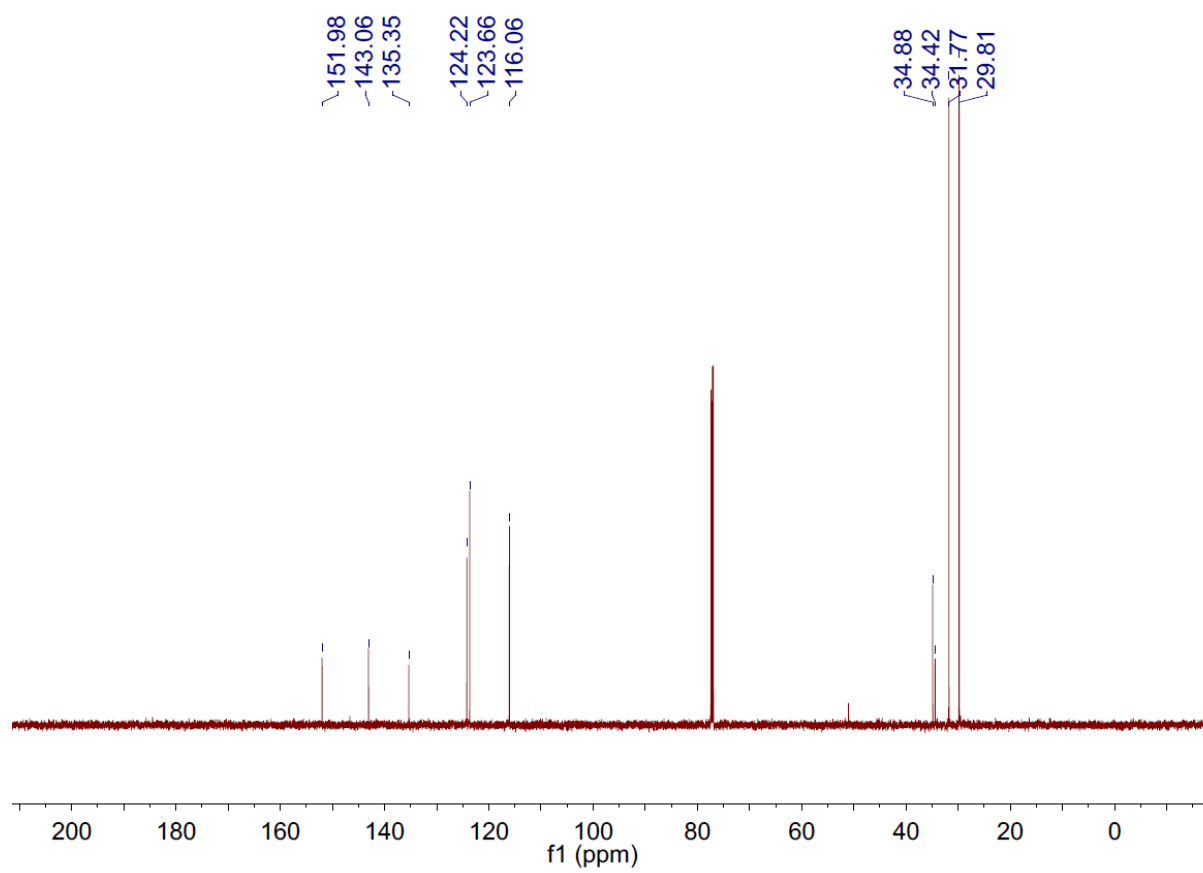


Figure S21: ^{13}C NMR spectrum of **3** in CDCl_3 at 150 MHz

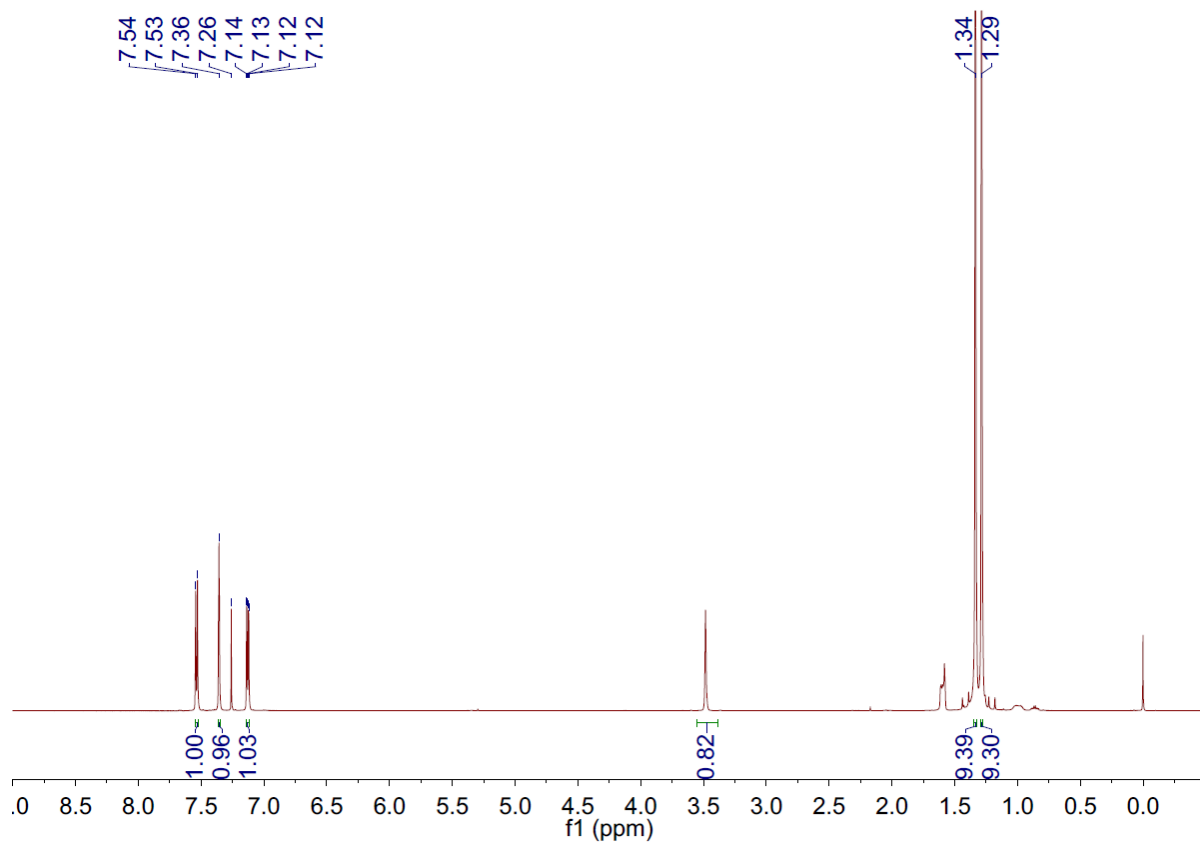


Figure S22: ^1H NMR spectrum of **4** in CDCl_3 at 600 MHz

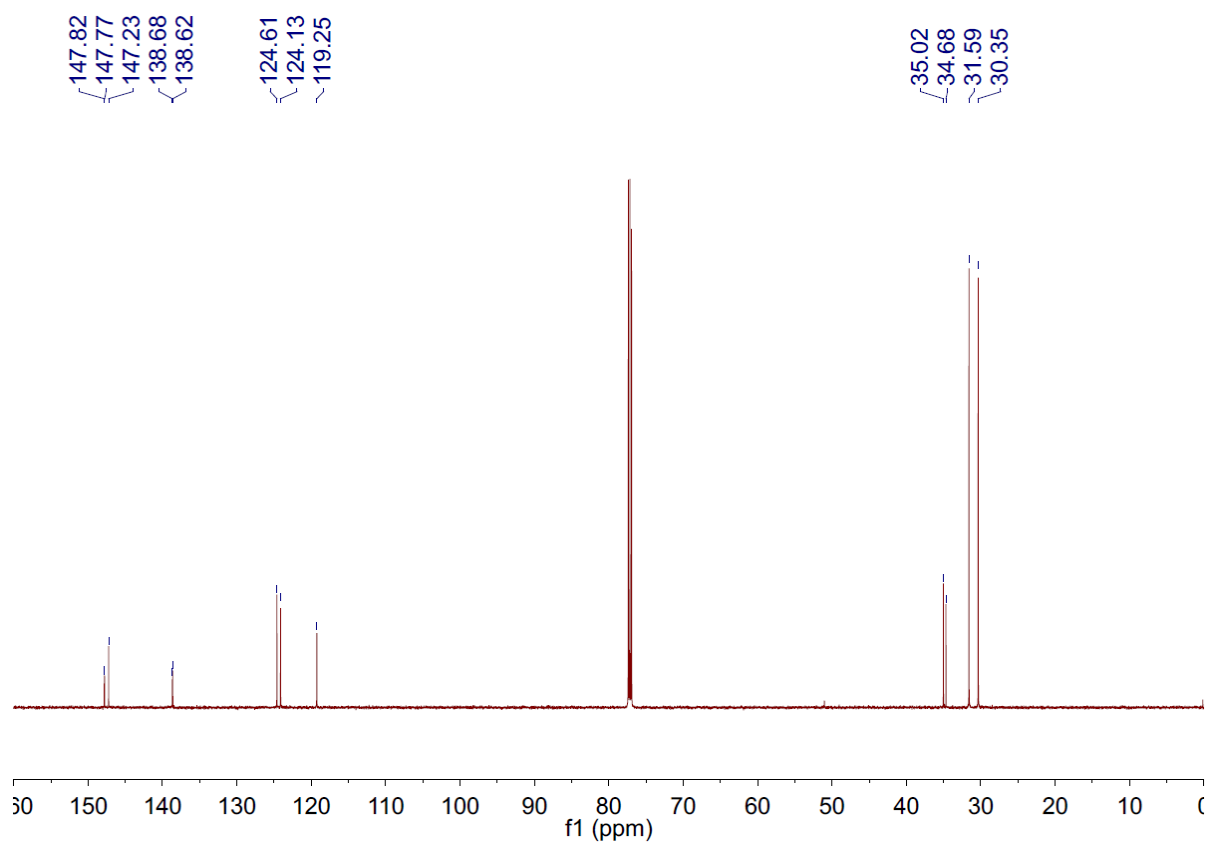


Figure S23: ^{13}C NMR spectrum of **4** in CDCl_3 at 150 MHz

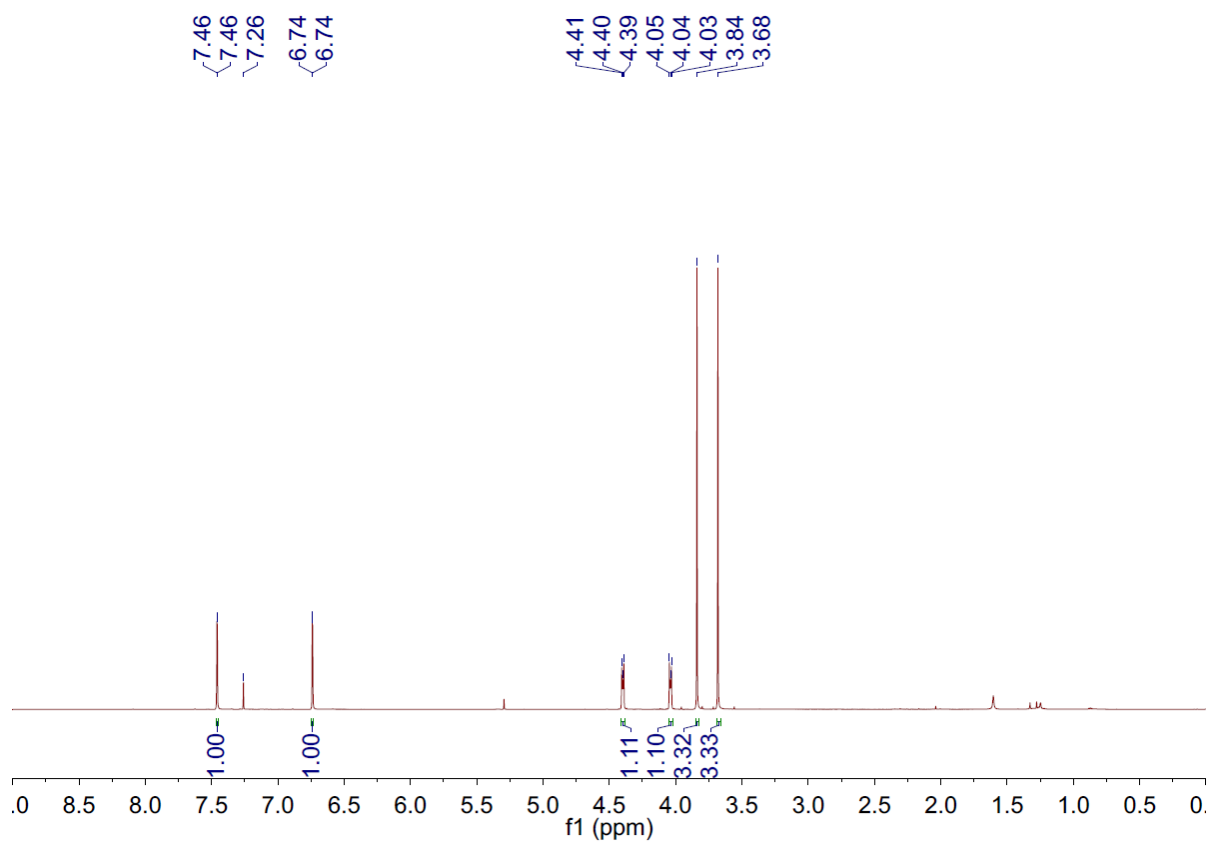


Figure S24: ^1H NMR spectrum of **5** in CDCl_3 at 600 MHz

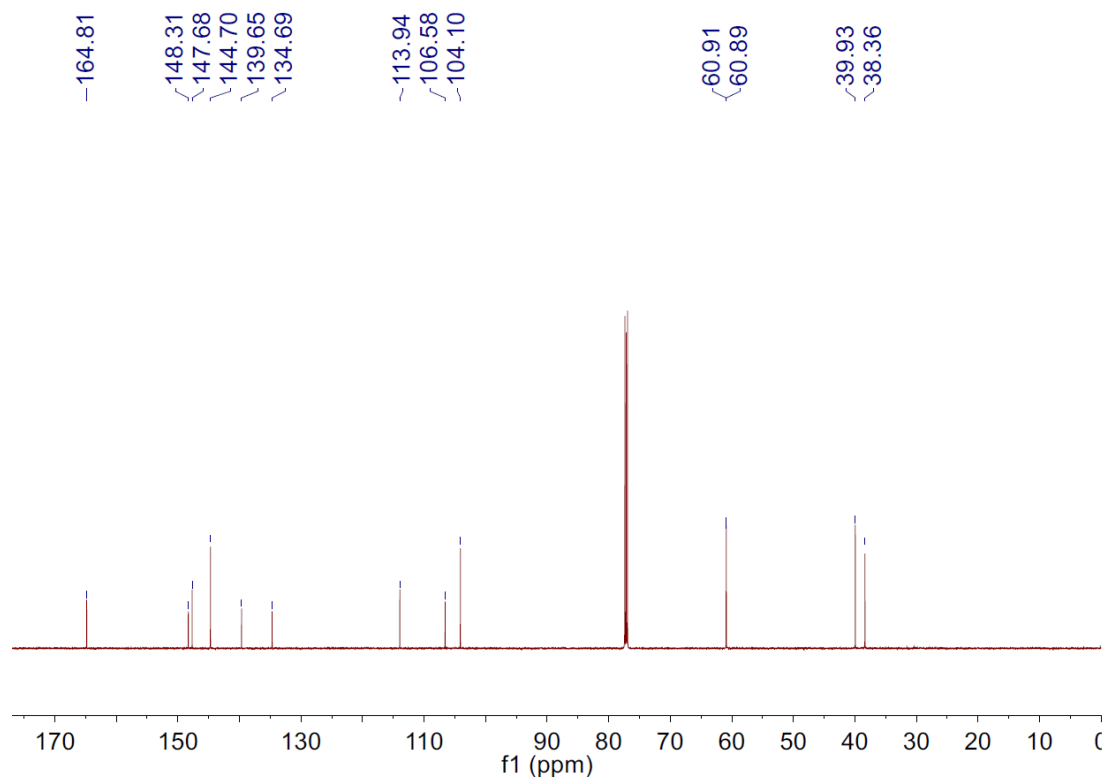


Figure S25: ^{13}C NMR spectrum of **5** in CDCl_3 at 150 MHz

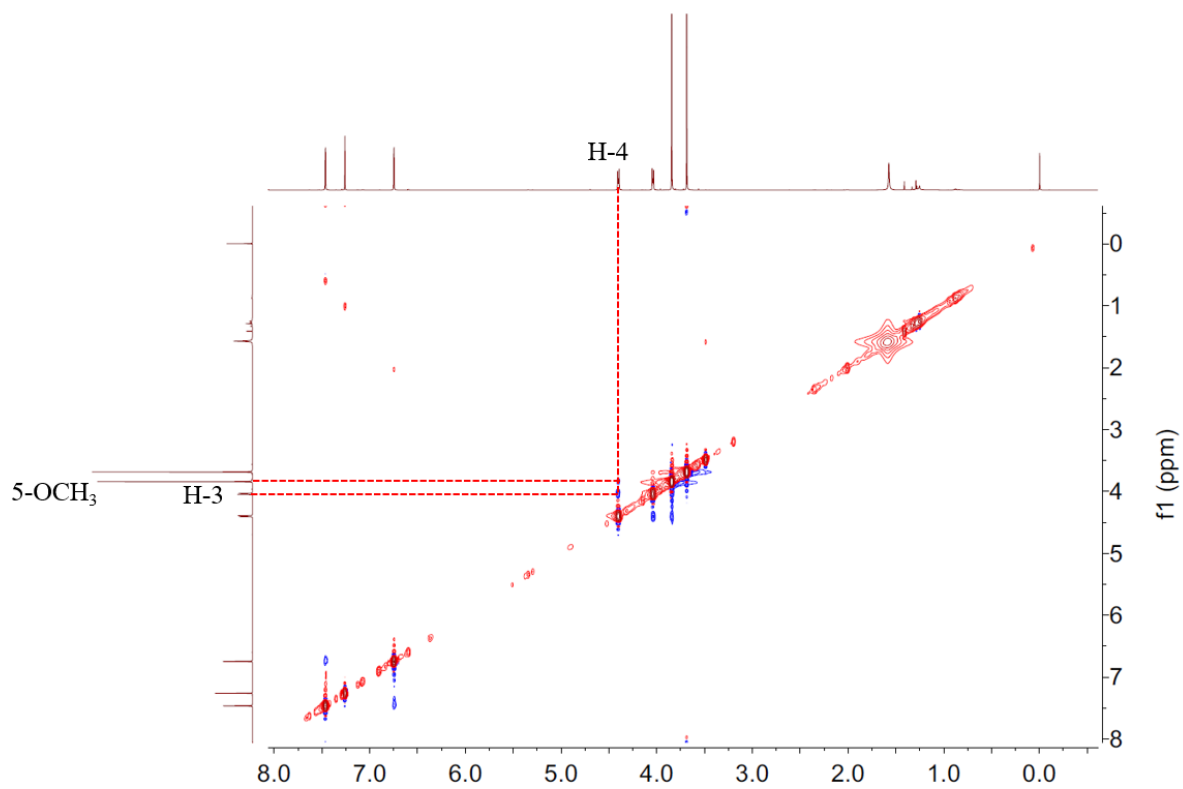


Figure S26: ROESY spectrum of **5**

Calculation details

The all obtained stable conformers were subsequently optimized by using Gaussian16 software at the B3LYP/6-31G(d) level in PCM model. The optimized stable conformers were selected for further NMR calculations at the B3LYP/6-311+G(d,p) level in PCM model. The overall NMR were weighted by Boltzmann distribution.

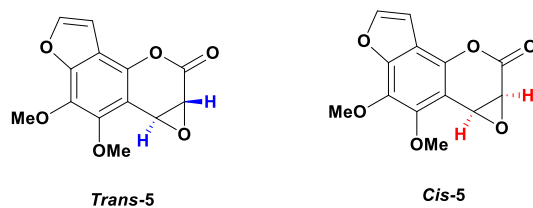


Figure S27: Structures of isomers *Trans-5* and *Cis-5*

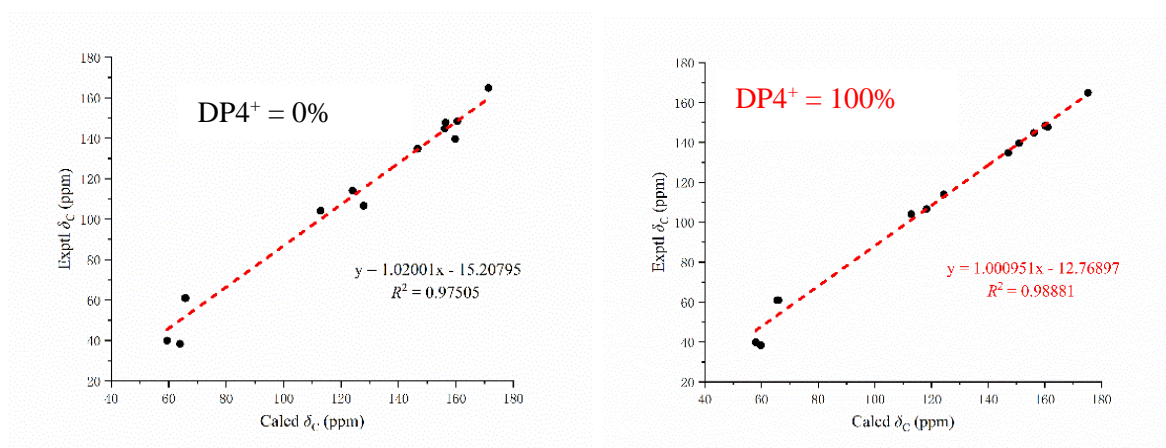




Figure S28: Key parameters of the calculated chemical shifts of *trans-5* and *cis-5* and including coefficient of determination (R^2) and DP4⁺ probability.

Table S2. DP4⁺ analysis of compound 5

		DP4+	 0.00%	 100.00%
Nuclei	sp2?	xperimenta	Isomer 1	Isomer 2
C	x	164.82	171.3782	175.28
C		39.94	59.4389	57.98
C		38.37	63.9823	59.79
C	x	147.68	156.4536	161.06
C	x	134.7	146.6901	147.18
C	x	148.32	160.5864	160.20
C	x	113.96	124.0551	124.36
C	x	144.73	156.1638	156.19
C	x	104.12	112.9216	112.88
C	x	106.56	127.9897	118.34
C		60.92	65.6252	66.02
C		60.89	65.8561	65.59
C	x	139.66	159.795	151.00

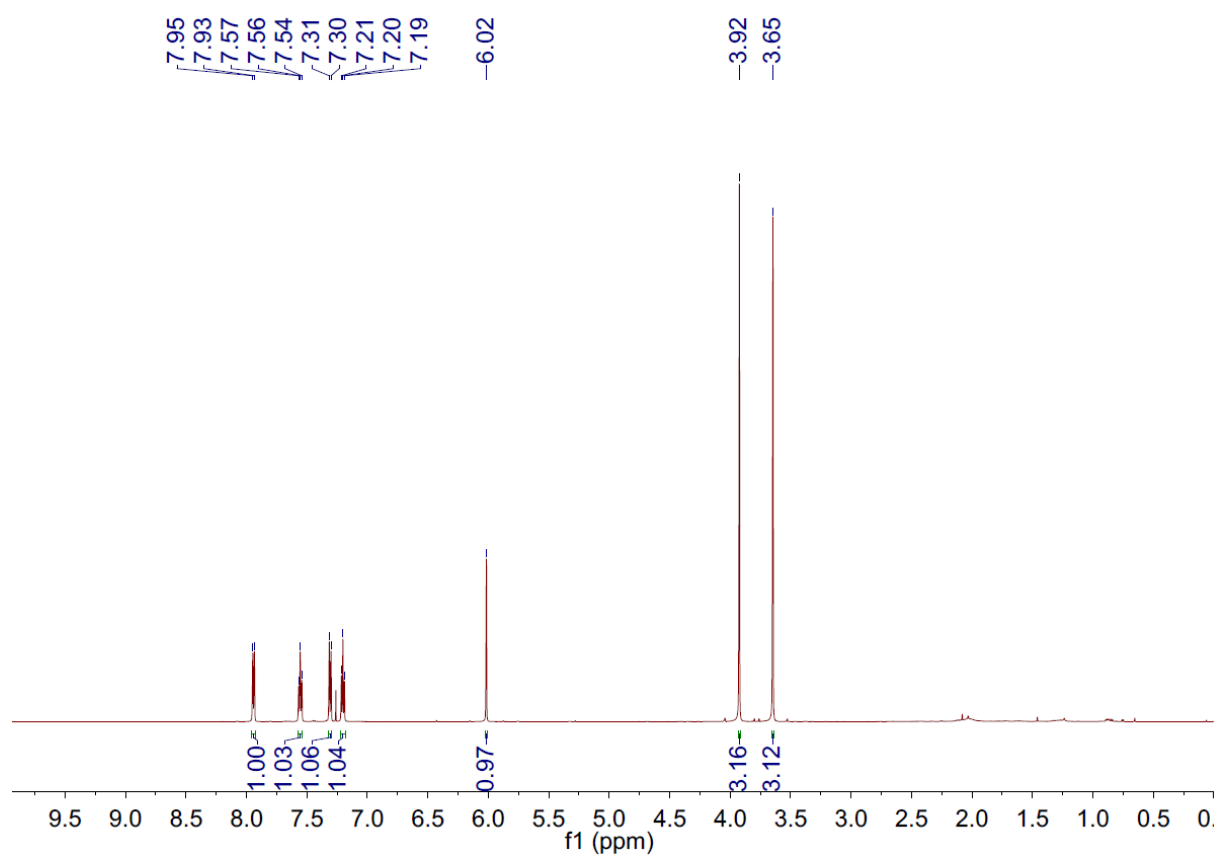


Figure S29: ^1H NMR spectrum of **6** in CDCl_3 at 600 MHz

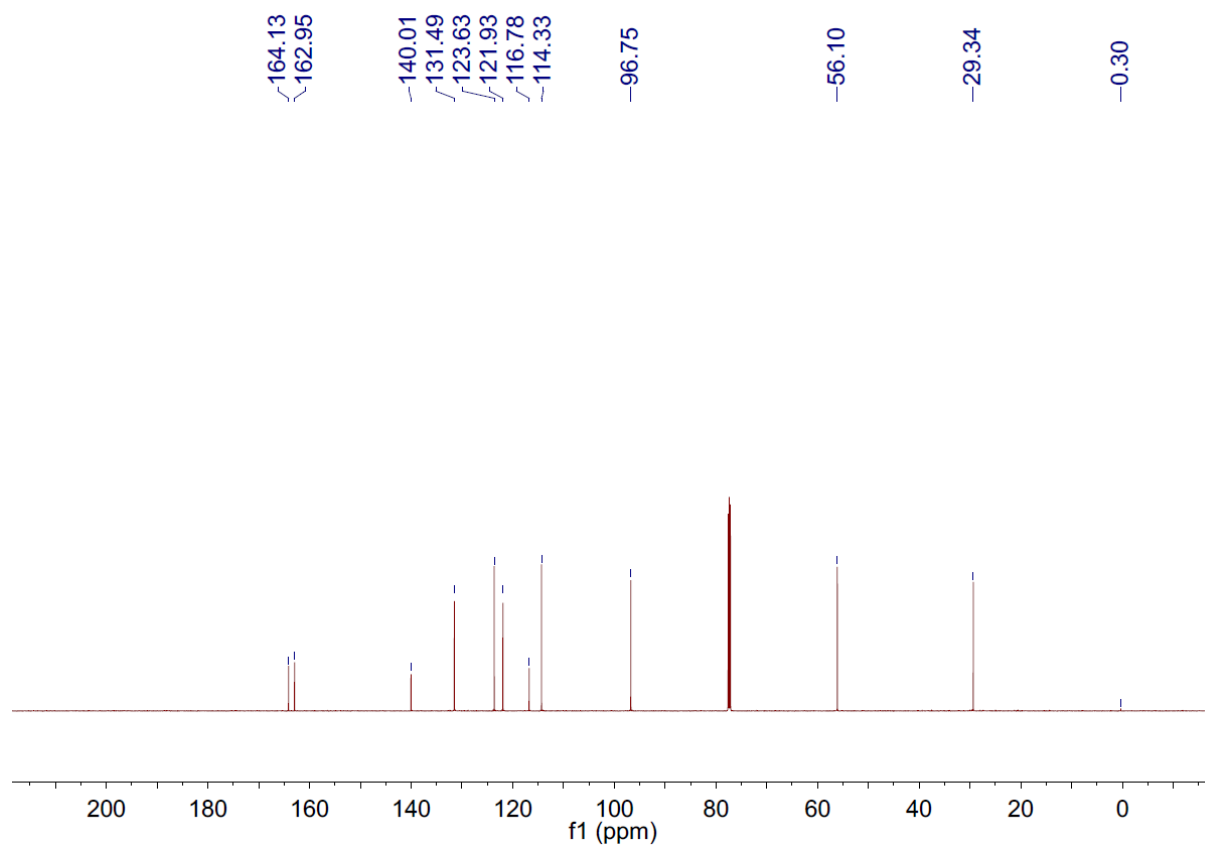


Figure S30: ^{13}C NMR spectrum of **6** in CDCl_3 at 600 MHz

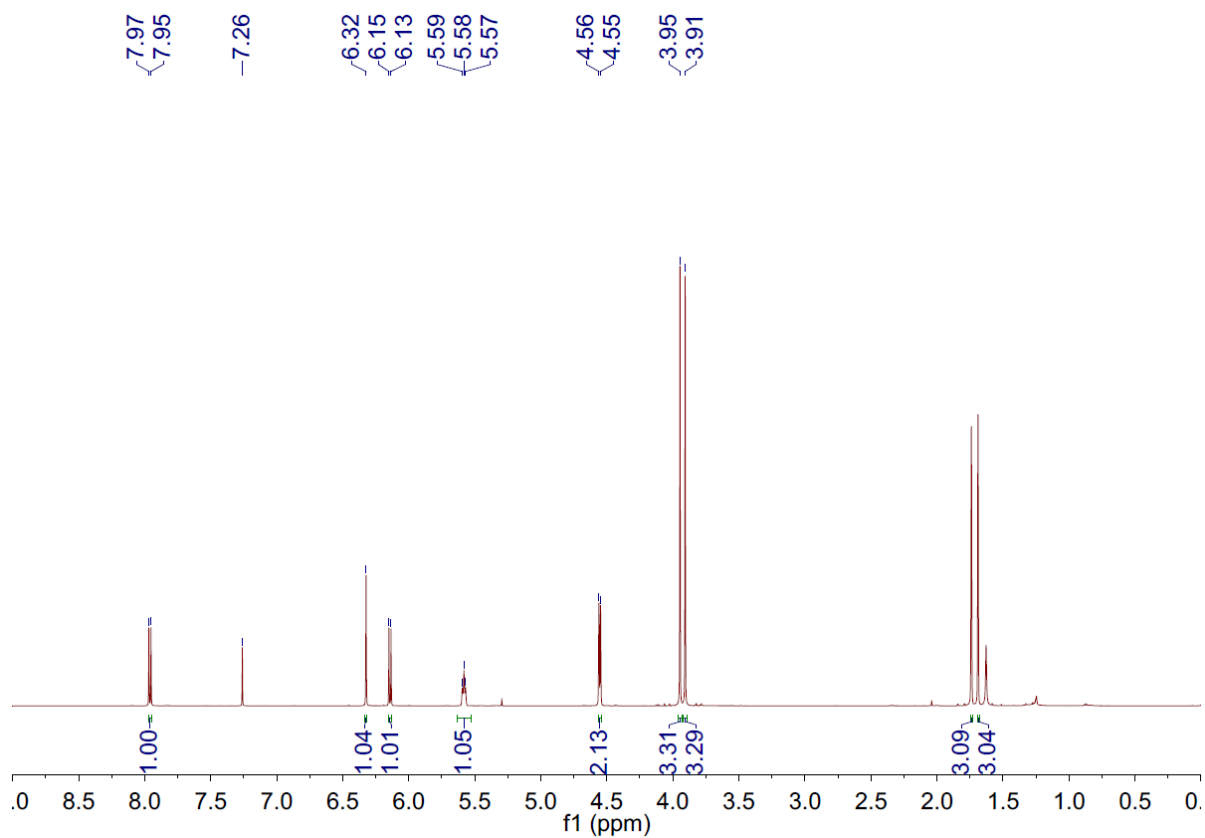


Figure S31: ^1H NMR spectrum of **7** in CDCl_3 at 600 MHz

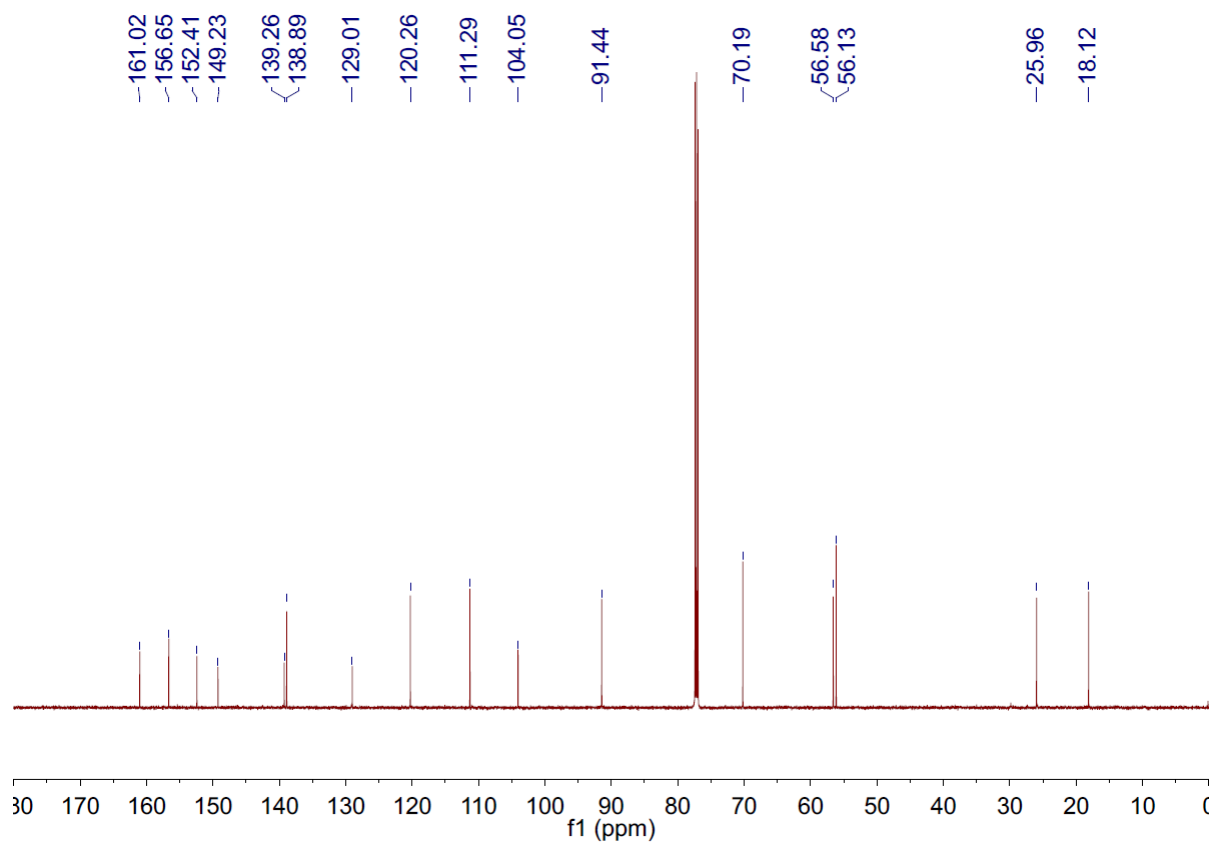


Figure S32: ^{13}C NMR spectrum of **7** in CDCl_3 at 150 MHz

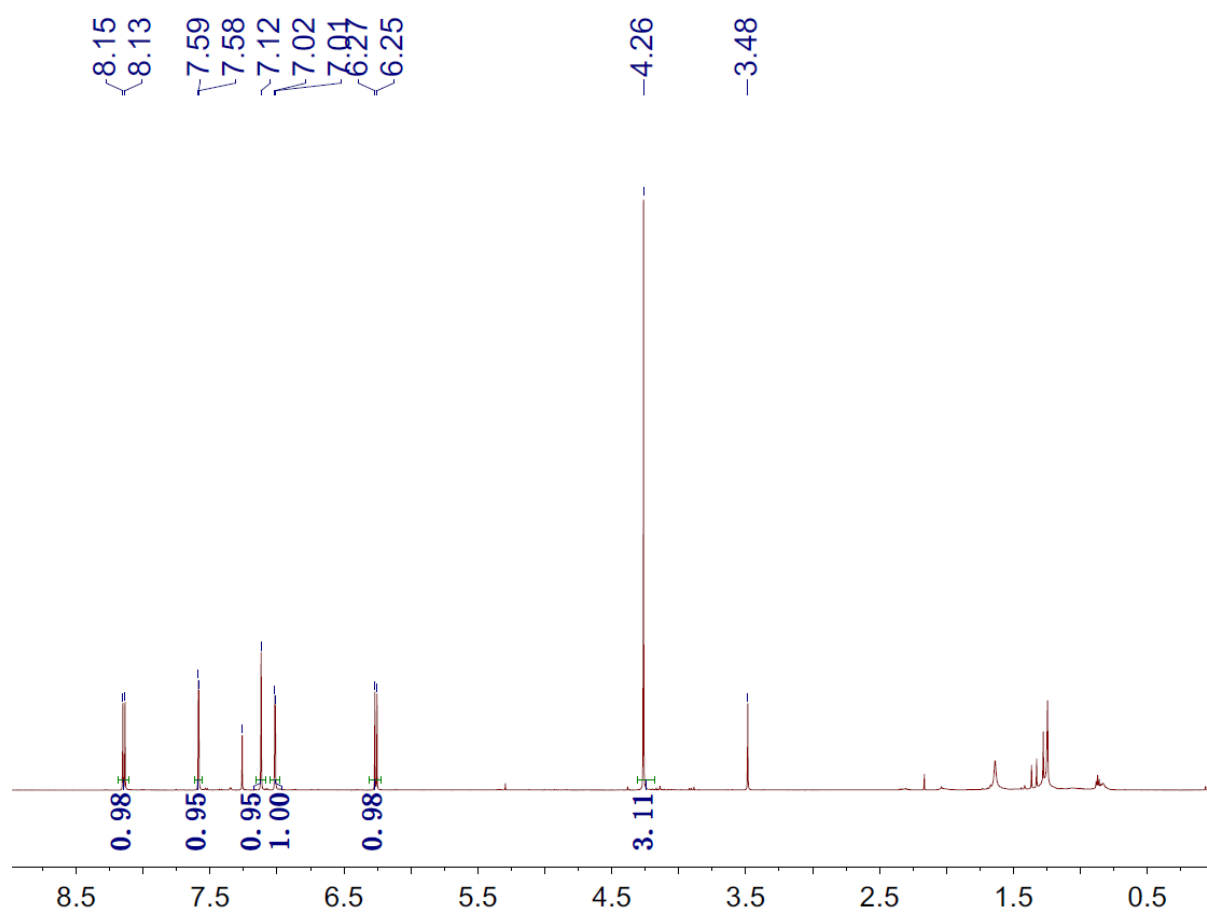


Figure S33: ¹H NMR spectrum of **8** in CDCl₃ at 600 MHz

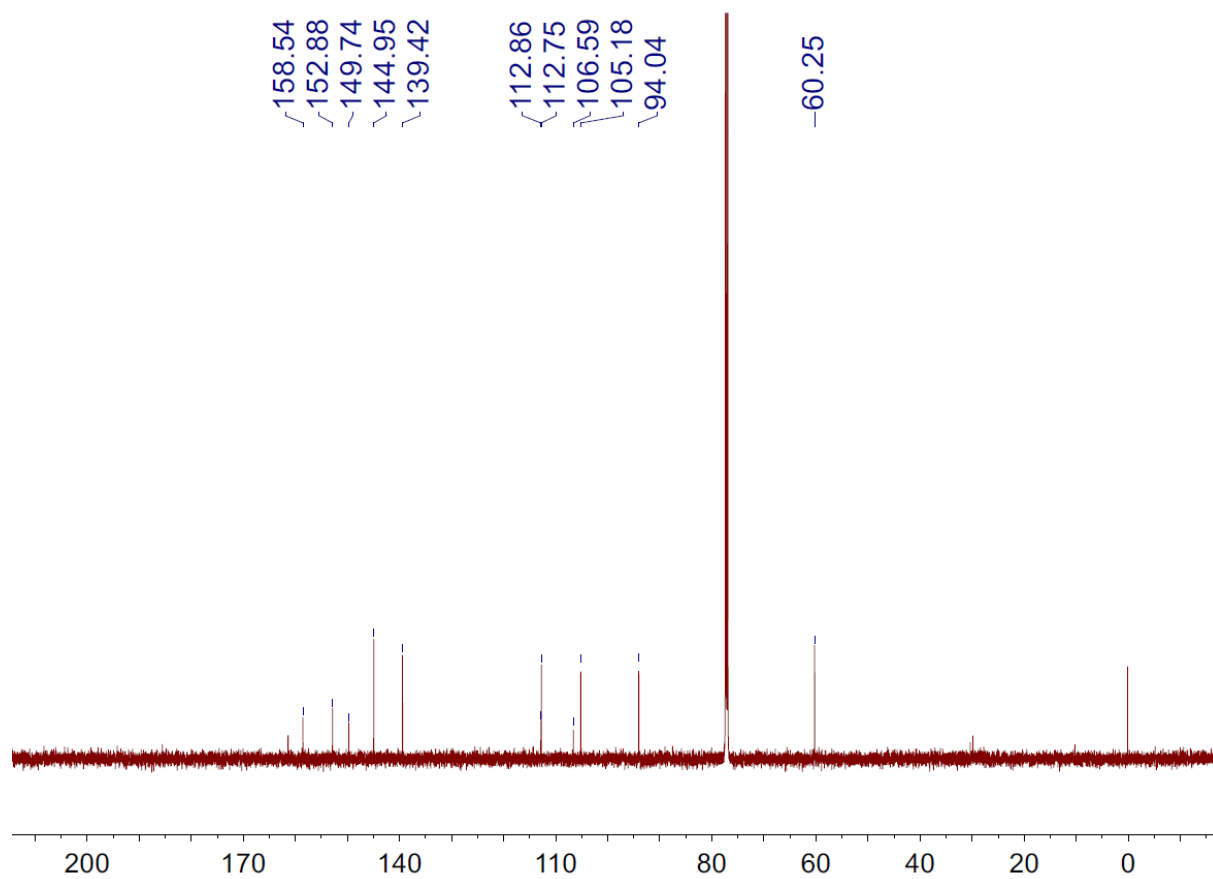


Figure S34: ^{13}C NMR spectrum of **8** in CDCl_3 at 150 MHz

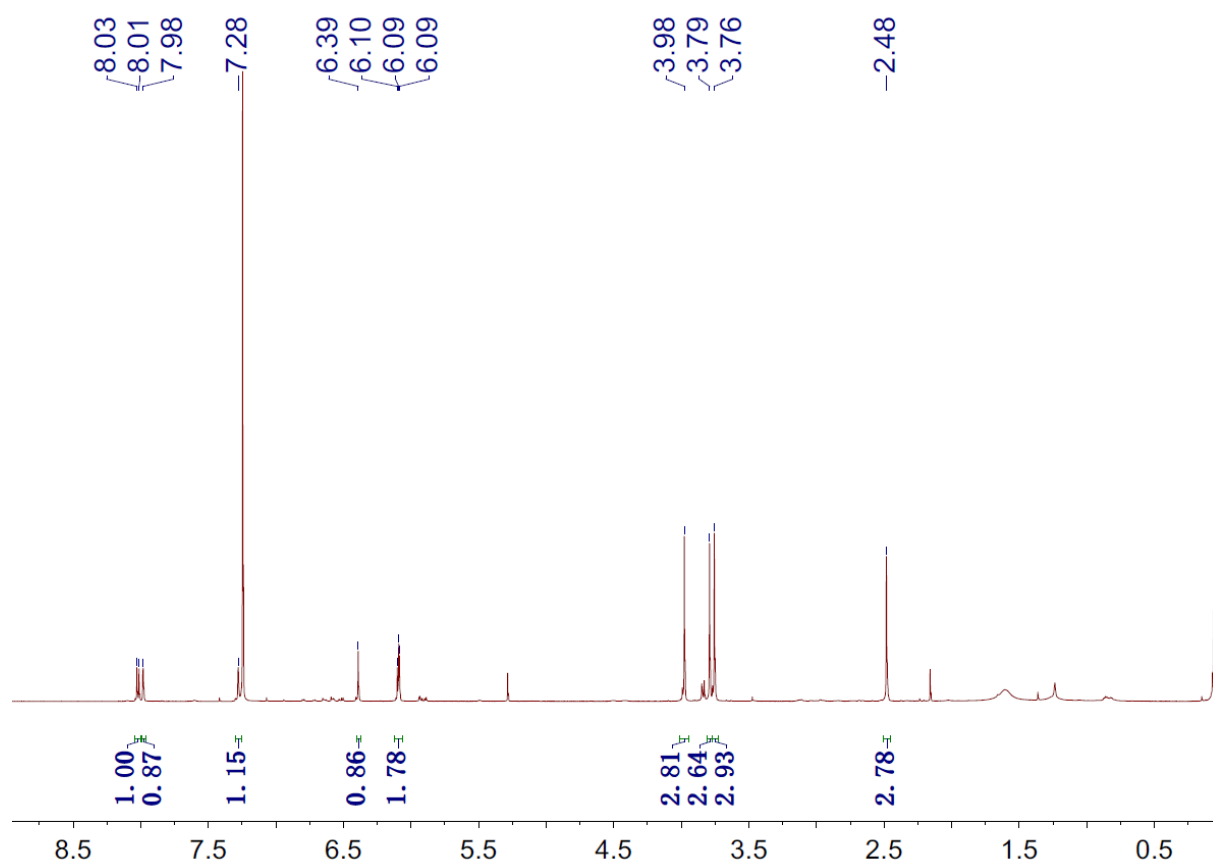


Figure S35: ^1H NMR spectrum of **9** in CDCl_3 at 600 MHz

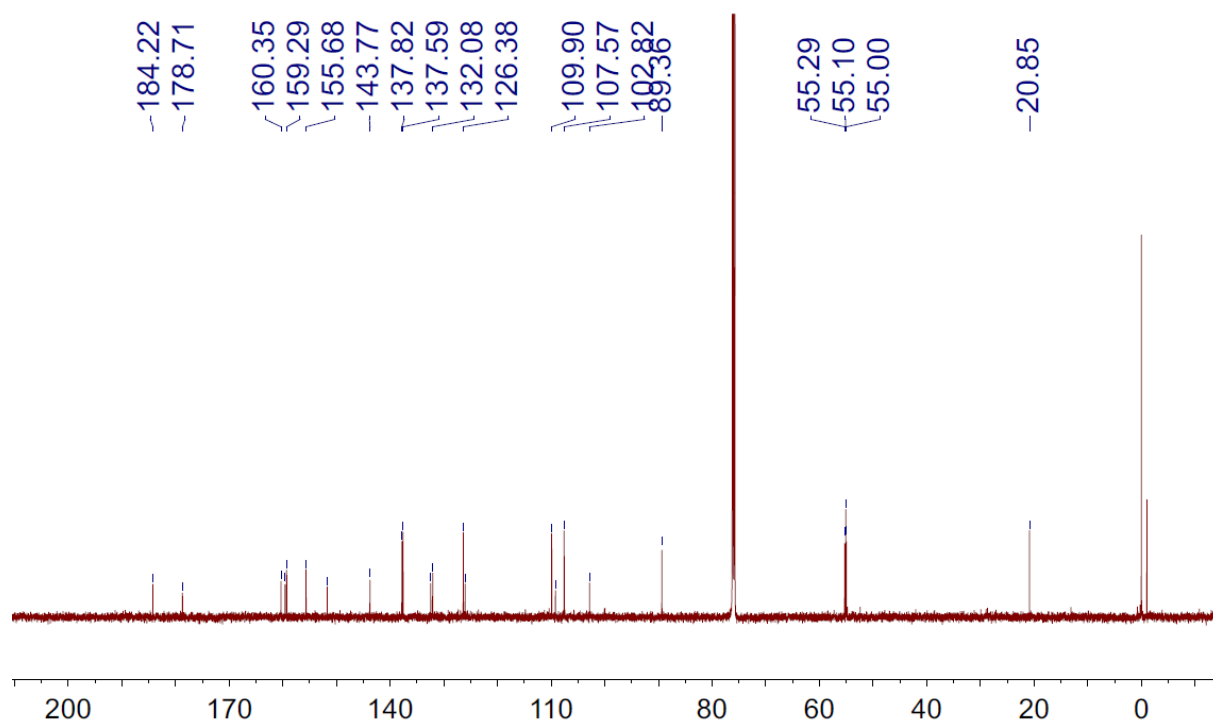


Figure S36: ¹³C NMR spectrum of **9** in CDCl₃ at 150 MHz

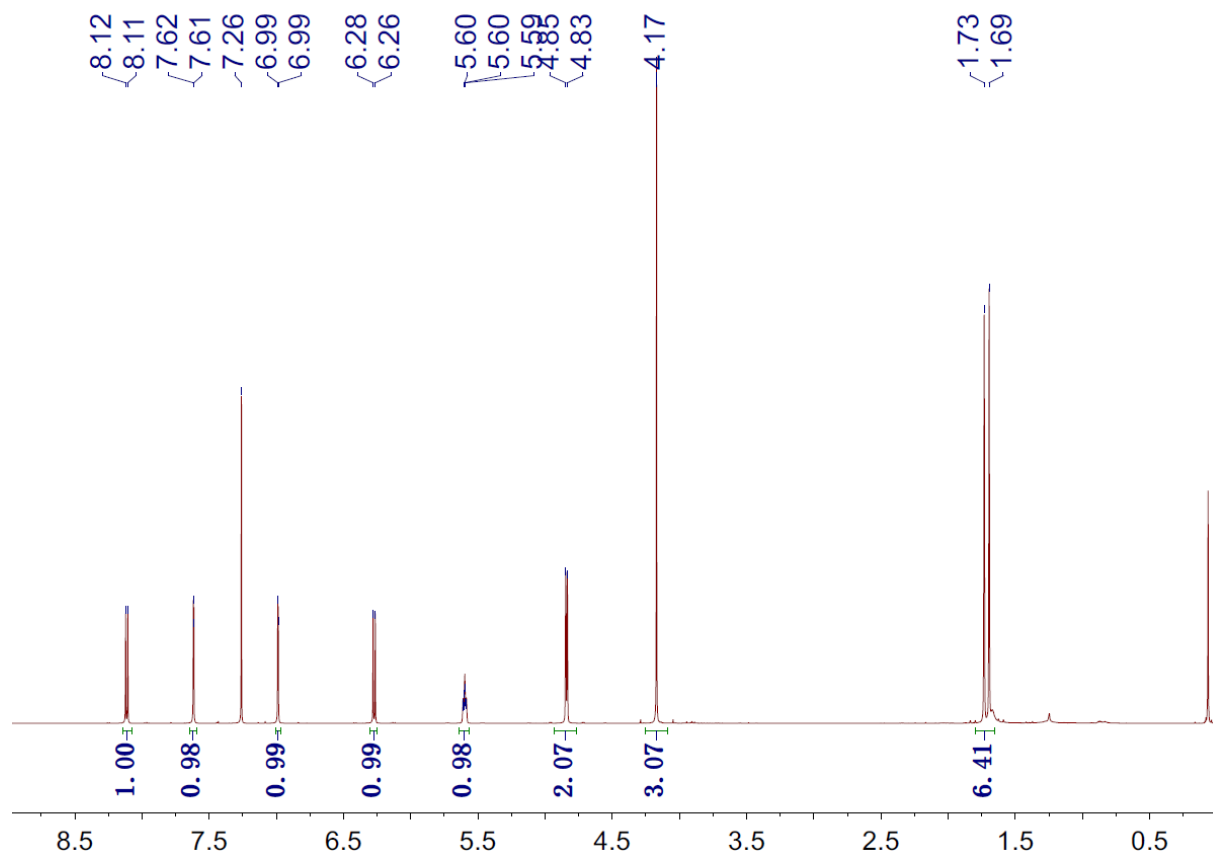


Figure S37: ^1H NMR spectrum of **10** in CDCl_3 at 600 MHz

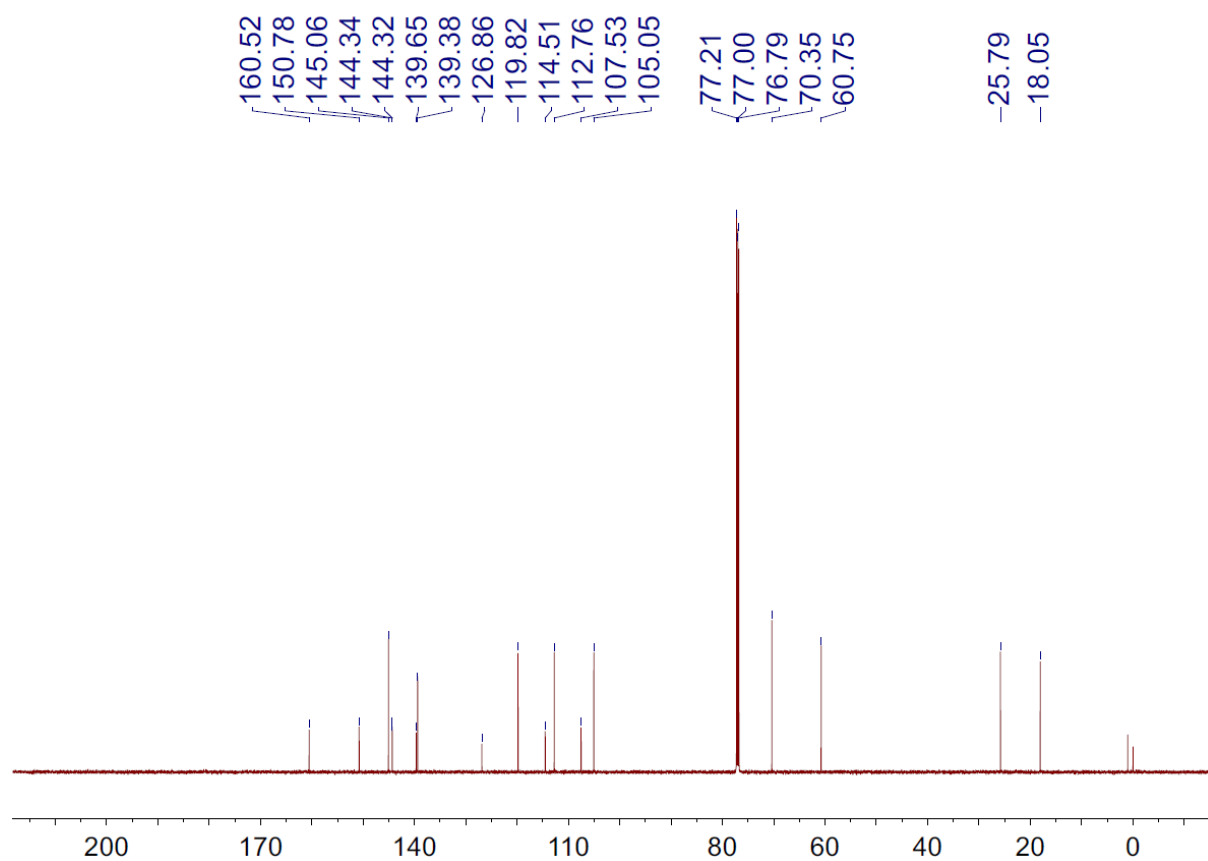


Figure S38: ^{13}C NMR spectrum of **10** in CDCl_3 at 150 MHz

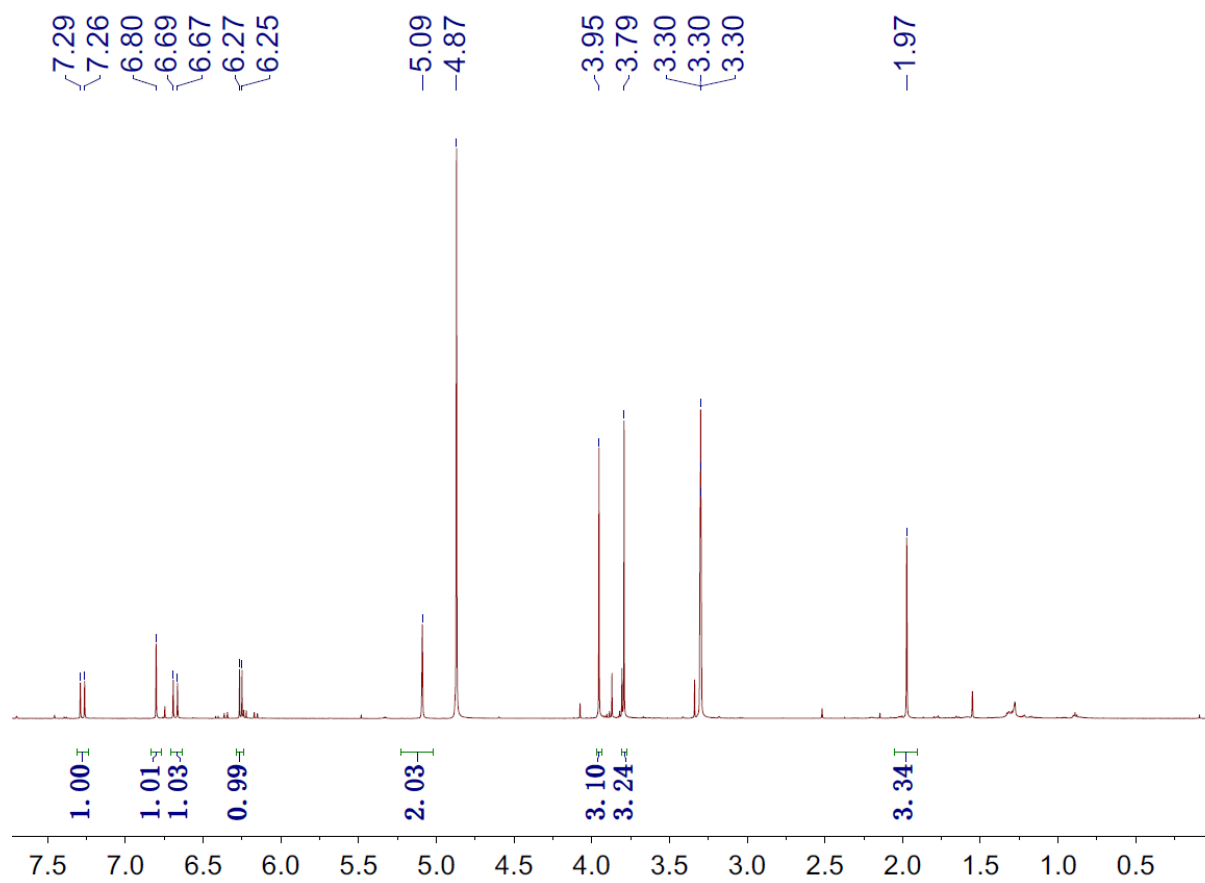


Figure S39: ^1H NMR spectrum of **11** in $\text{MeOH-}d_4$ (600 MHz)

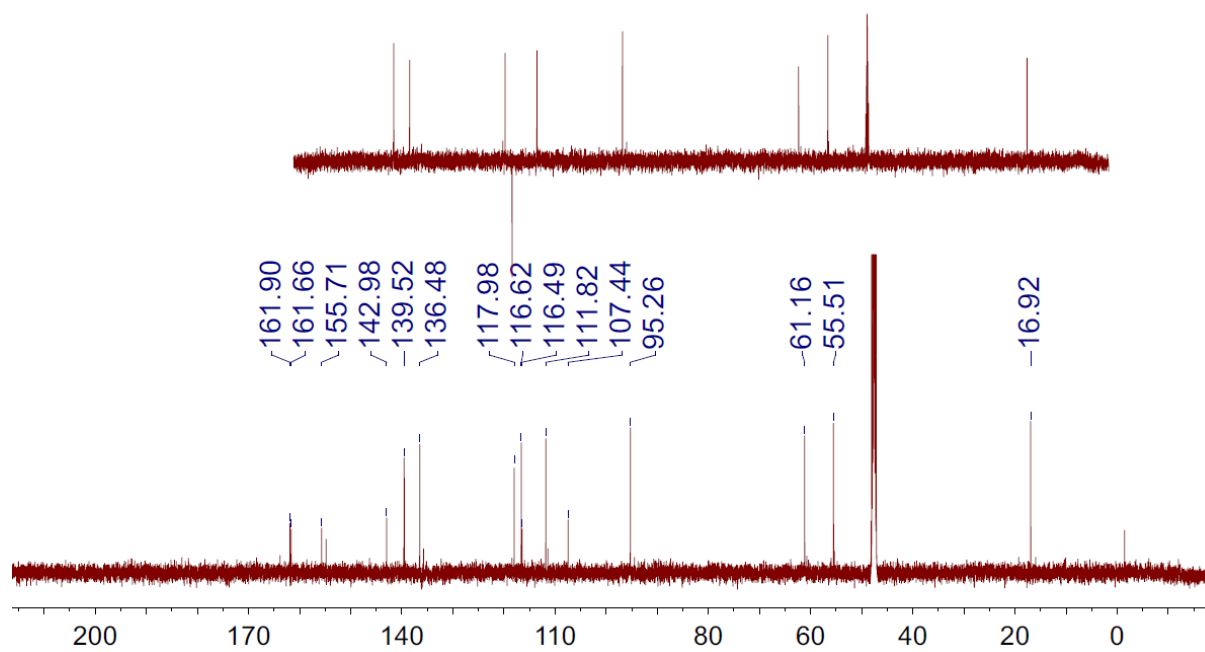


Figure S40: ^{13}C NMR spectrum of **11** $\text{MeOH-}d_4$ (150 MHz)

Table S3: Inhibitory effects of compounds **1**, **5-11** on four kinds of tumor cells

compound	Concentration	Inhibition rate (%)			
	(μ M)	HEP-2	SGC7901	SW480	MDA-MB-231
1	100	7.93 \pm 1.19	9.67 \pm 0.75	-	3.19 \pm 2.36
5	100	13.32 \pm 2.70	45.15 \pm 0.85	19.48 \pm 3.77	26.48 \pm 3.90
6	100	7.94 \pm 0.78	8.18 \pm 2.62	-	17.57 \pm 3.61
7	100	17.32 \pm 2.03	17.70 \pm 0.85	16.42 \pm 3.49	27.90 \pm 1.96
8	100	24.09 \pm 2.74	28.14 \pm 1.62	42.72 \pm 1.34	20.93 \pm 3.29
9	100	21.92 \pm 3.85	35.62 \pm 1.32	6.53 \pm 2.50	30.66 \pm 2.98
10	100	87.59 \pm 1.19	40.46 \pm 2.12	49.14 \pm 0.76	32.78 \pm 4.79
5-FU	2 mM	33.05 \pm 0.74	31.73 \pm 0.45	24.80 \pm 1.09	36.77 \pm 0.18

The instruments and equipment

IR spectra were recorded on a Bruker Tensor 37 infrared spectrophotometer with KBr disk. NMR spectra were measured on AVANCE 600 MHz spectrometer with TMS as the internal standard. The UV spectra were measured with a Shimadzu UV-2450 spectrophotometer. HR-ESIMS data were determined using a Shimadzu UPLC-IT-TOF spectrometer. The semi-preparative HPLC was performed on a NS4205 pump operating system equipped with Nucifera Si column (9.4 mm × 25 cm, 5 μm). RP HPLC was performed on CXTH LC3050N with an Innoval ODS-2 column (10 μm, 10 × 250 mm, Agela, Tianjin, China). Column chromatography (CC) was used with silica gel (200-300 mesh, Qingdao Marine Chemical Factory, Qingdao, China).