

Anti-inflammatory withanolides from *physalis minima*

Jiangping Wu,[†] Tao Zhang,[†] Meng Yu,[†] Hongmei Jia,[†] Hongwu Zhang,[†]

Qiongming Xu,[‡] Yucheng Gu,^{*,§} and Zhongmei Zou^{*,†}

[†]Institute of Medicinal Plant Development, Chinese Academy of Medical Sciences

and Peking Union Medical College, Beijing 100193, China;

[‡]College of Pharmaceutical Science, Soochow University, Suzhou 215123, China;

[§]Syngenta, Jealott's Hill International Research Centre, Berkshire RE42 6EY, UK.

* Corresponding author: Zhongmei Zou, Yucheng Gu, PhD;

Tel/Fax: 86-1057833290

E-mail address: zmzou@implad.ac.cn; yucheng.gu@syngenta.com

Index

page	contents
S4	Table S1 ^{13}C -NMR (150 MHz) spectroscopic data for compounds 6–10 in CD_3OD .
S5	Table S2 ^1H -NMR (600 MHz) spectroscopic data for compounds 6–10 in CD_3OD .
S6	HR-ESI-MS spectrum of compound 1 , ^1H NMR spectrum of compound 1 in CD_3OD at 600 MHz.
S7	Detailed ^1H NMR spectrum of compound 1 in CD_3OD at 600 MHz, Detailed ^1H NMR spectrum of compound 1 in CD_3OD at 600 MHz.
S8	^{13}C -NMR spectrum of compound 1 in CD_3OD at 150 MHz, Detailed ^{13}C NMR spectrum of compound 1 in CD_3OD at 150 MHz.
S9	Detailed ^{13}C NMR spectrum of compound 1 in CD_3OD at 150 MHz, HSQC spectrum of compound 1 in CD_3OD .
S10	HMBC spectrum of compound 1 in CD_3OD , COSY spectrum of compound 1 in CD_3OD .
S11	NOESY spectrum of compound 1 in CD_3OD .
S12	ECD spectrum of 1
S13	HR-ESI-MS spectrum of compound 2 , ^1H NMR spectrum of compound 2 in CD_3OD at 600 MHz.
S14	Detailed ^1H NMR spectrum of compound 2 in CD_3OD at 600 MHz, Detailed ^1H NMR spectrum of compound 2 in CD_3OD at 600 MHz.
S15	^{13}C NMR spectrum of compound 2 in CD_3OD at 150 MHz, Detailed ^{13}C NMR spectrum of compound 2 in CD_3OD at 150 MHz.
S16	Detailed ^{13}C NMR spectrum of compound 2 in CD_3OD at 150 MHz, HSQC spectrum of compound 2 in CD_3OD .
S17	HMBC spectrum of compound 2 in CD_3OD , COSY spectrum of compound 2 in CD_3OD .
S18	NOESY spectrum of compound 2 in CD_3OD .
S19	HR-ESI-MS spectrum of compound 3 , ^1H NMR spectrum of compound 3 in CD_3OD at 600 MHz.
S20	Detailed ^1H NMR spectrum of compound 3 in CD_3OD at 600 MHz, Detailed ^1H NMR spectrum of compound 3 in CD_3OD at 600 MHz.

S21	¹³ C NMR spectrum of compound 3 in CD ₃ OD at 150 MHz, Detailed ¹³ C NMR spectrum of compound 3 in CD ₃ OD at 150 MHz.
S22	Detailed ¹³ C NMR spectrum of compound 3 in CD ₃ OD at 150 MHz, HSQC spectrum of compound 3 in CD ₃ OD.
S23	HMBC spectrum of compound 3 in CD ₃ OD, COSY spectrum of compound 3 in CD ₃ OD.
S24	NOESY spectrum of compound 3 in CD ₃ OD.
S25	HR-ESI-MS spectrum of compound 4 , ¹ H NMR spectrum of compound 4 in CD ₃ OD at 600 MHz.
S26	Detailed ¹ H NMR spectrum of compound 4 in CD ₃ OD at 600 MHz, Detailed ¹ H NMR spectrum of compound 4 in CD ₃ OD at 600 MHz.
S27	¹³ C NMR spectrum of compound 4 in CD ₃ OD at 150 MHz, Detailed ¹³ C NMR spectrum of compound 4 in CD ₃ OD at 150 MHz.
S28	Detailed ¹³ C NMR spectrum of compound 4 in CD ₃ OD at 150 MHz, HSQC spectrum of compound 4 in CD ₃ OD.
S29	HMBC spectrum of compound 4 in CD ₃ OD, COSY spectrum of compound 4 in CD ₃ OD.
S30	NOESY spectrum of compound 4 in CD ₃ OD.
S31	HR-ESI-MS spectrum of compound 5 , ¹ H NMR spectrum of compound 5 in CD ₃ OD at 600 MHz.
S32	Detailed ¹ H NMR spectrum of compound 5 in CD ₃ OD at 600 MHz, Detailed ¹ H NMR spectrum of compound 5 in CD ₃ OD at 600 MHz.
S33	¹³ C NMR spectrum of compound 5 in CD ₃ OD at 150 MHz, Detailed ¹³ C NMR spectrum of compound 5 in CD ₃ OD at 125 MHz.
S34	Detailed ¹³ C NMR spectrum of compound 5 in CD ₃ OD at 150 MHz, HSQC spectrum of compound 5 in CD ₃ OD.
S35	HMBC spectrum of compound 5 in CD ₃ OD, COSY spectrum of compound 5 in CD ₃ OD.
S36	NOESY spectrum of compound 5 in CD ₃ OD.

Table S1 ^{13}C -NMR (150 MHz) spectroscopic data for compounds **6–10** in CD_3OD .

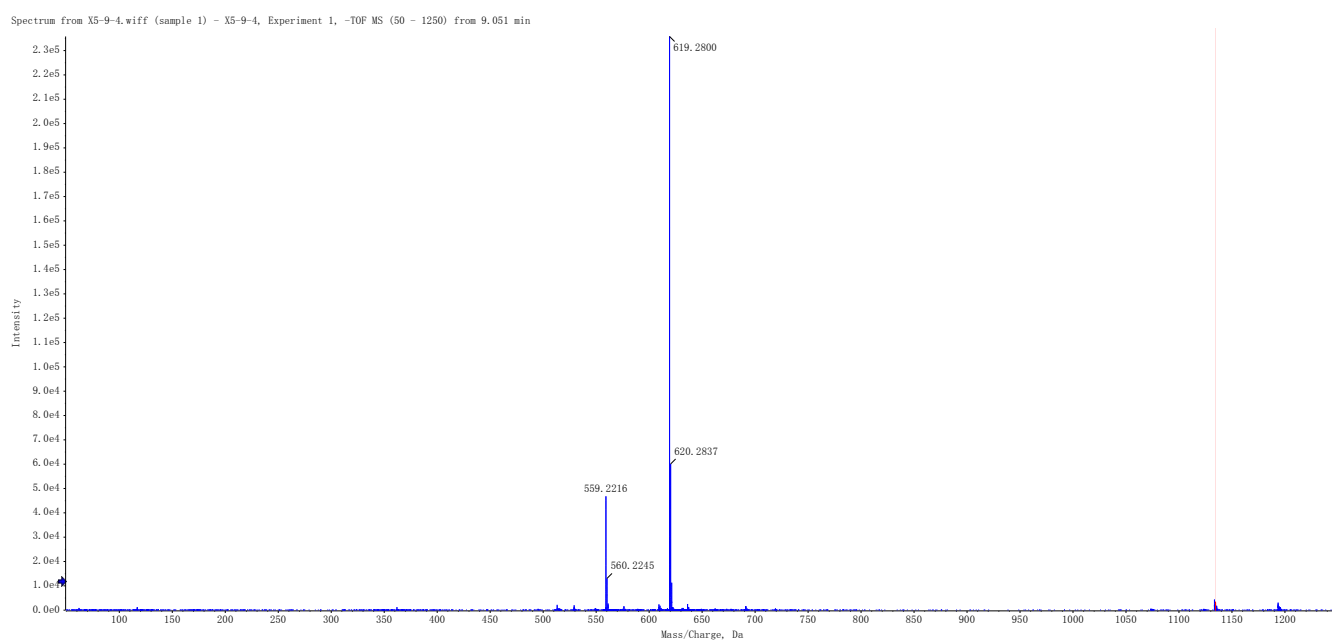
NO.	6	7	8	9	10
1	210.3	212.5	204.1	205.6	206.0
2	41.3	42.2	128.7	129.6	128.7
3	73.6	70.5	141.7	142.3	142.5
4	36.5	78.6	36.0	37.2	37.1
5	61.5	65.4	77.2	78.5	78.1
6	62.4	59.7	73.4	75.1	75.4
7	25.1	26.7	26.8	28.8	30.8
8	35.7	34.8	35.4	37.5	37.7
9	38.6	39.8	36.3	37.4	38.2
10	52.1	52.0	52.6	52.6	52.5
11	22.2	21.8	23.2	24.1	22.9
12	37.9	30.3	38.3	40.8	40.7
13	52.3	55.0	52.2	53.2	54.0
14	81.3	81.2	82.4	82.4	84.0
15	84.4	33.0	83.6	83.4	76.5
16	122.1	37.9	120.4	127.3	127.8
17	162.6	86.7	162.2	158.9	154.6
18	16.8	20.8	16.1	17.9	19.9
19	14.3	15.2	15.0	16.0	16.7
20	35.0	79.3	36.1	35.5	35.2
21	17.8	19.0	17.2	19.8	18.6
22	78.9	81.6	78.6	79.7	79.4
23	32.1	33.9	32.4	32.9	32.7
24	150.8	151.0	150.8	150.1	150.5
25	121.8	121.7	121.2	122.2	122.0
26	167.7	169.6	167.4	166.8	166.7
27	12.5	11.2	11.6	13.0	13.0
28	19.7	20.4	20.6	18.5	20.4
CH ₃ CO-1'	170.1	-	170.6	-	-
CH ₃ CO-2'	20.9	-	21.9	-	-
OMe	53.7	-	-	-	-

Table S2 ¹H-NMR (600 MHz) spectroscopic data for compounds **6–10** in CD₃OD.

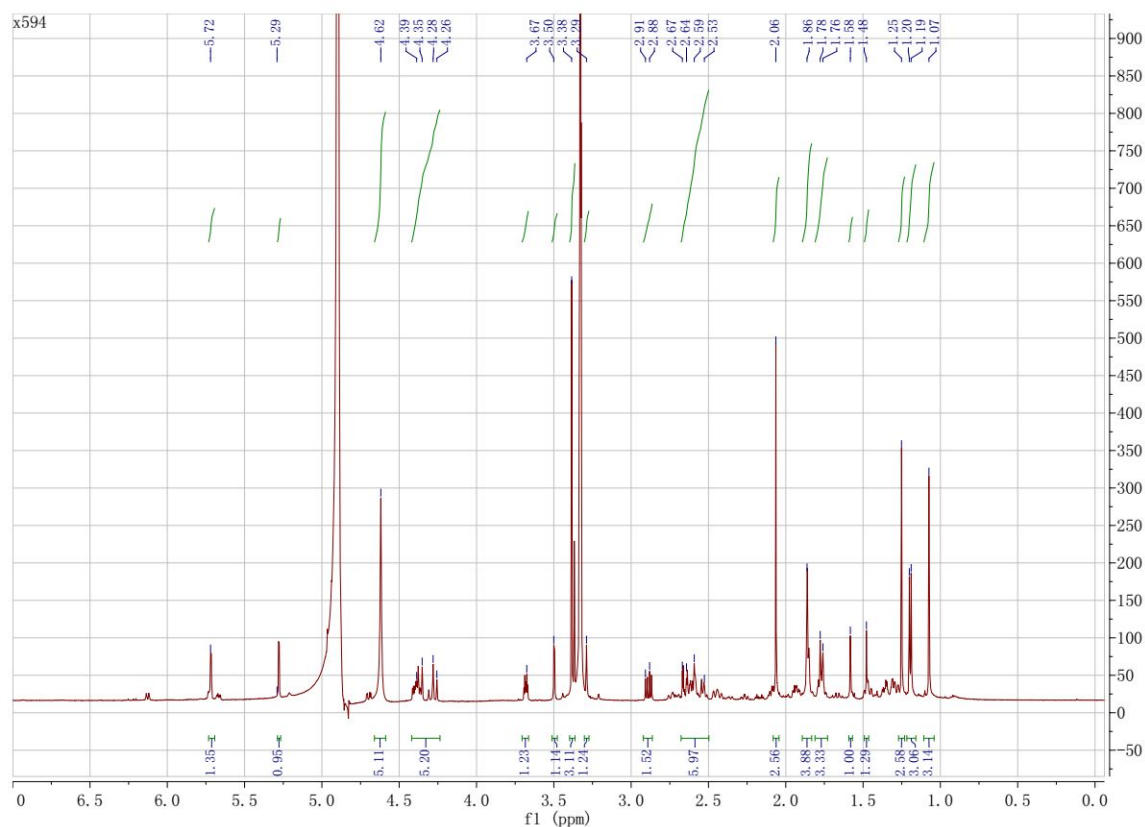
NO.	6	7	8	9	10
2	α : 2.88 dd (16.2, 3.0) β : 2.94 dd (16.2, 7.8)	α : 2.55 dd (15.6, 3.2) β : 2.91 dd (15.6, 7.2)	5.87 dd (10.2, 3.6)	6.15 dd (10.2, 3.6)	6.18 dd (10.2, 3.6)
3	3.79 m	4.08 m	6.57 ddd (10.2, 5.4, 3.6)	6.67 ddd (10.2, 5.4, 3.6)	6.69 ddd (10.2, 5.4, 3.6)
4	α : 1.68 (br d, 16.2) β : 3.66 d (3.0)	3.21 d (3.2)	α : 2.10 dd (10.2, 5.4) β : 3.36 dt (16.2, 3.6)	α : 2.45 dd (10.2, 5.4) β : 3.76 dt (16.2, 3.6)	α : 2.45 dd (10.2, 5.4) β : 3.74 dt (16.2, 3.6)
6	3.40 br s	3.30 br s	3.60 t (3.6)	4.30 br s	4.32 br s
7	α : 1.62 m β : 2.49 m	α : 2.49 m β : 2.60 m	α : 1.97 m, β : 2.21 m	α : 2.97 m, β : 3.47 m	α : 2.77 m, β : 3.57 m
8	2.59 m	2.63 m	2.30 m	2.86 m	2.76 m
9	1.93 m	2.57 m	2.12 m	3.79 m	3.69 m
11	1.25 m, 1.38 m	1.48 m, 1.70 m	2.16 m, 2.12 m	1.64 m, 2.82 m	1.42 m, 2.62 m
12	1.74 m, 1.47 m	1.51 m, 2.21 m	1.82 m, 2.31 m	1.95 m, 2.34 m	1.95 m, 2.34 m
15	5.82 d (3.0)	5.04 d (3.2)	5.37 d (2.4)	5.10 d (2.4)	5.25 d (2.4)
16	5.78 d (3.0)	3.67 m, 3.76 m	5.60 d (2.4)	6.13 d (2.4)	5.71 br s
18	1.37 s	1.21 s	1.16 s	1.43 s	1.39 s
19	1.35 s	1.26 s	1.21 s	1.72 s	1.70 s
20	2.57 m	-	2.53	2.64 m	2.62 m
21	1.21 d (7.2)	1.07 s	1.12 d (7.2)	1.22 d (7.2)	1.26 d (7.2)
22	4.39 dd (12.0, 3.6)	5.08 dd (12.0, 3.6)	4.34 dd (12.0, 3.6)	4.45 dd (12.0, 3.6)	4.43 dd (12.0, 3.6)
23	2.29 m, 2.59 m	1.86 m, 1.96 m	2.28 m, 2.39 m	2.08 m, 2.49 m	2.06 m, 2.52 m
27	1.84 s	1.86 s	1.86 s	1.85 s	1.85 s
28	1.53 (br s)	1.99 s	1.99 s	1.52 s	1.50 s
CH ₃ CO	2.09 s	2.10 s	2.03 s	-	-
OMe	3.21 s	-	-	-	-

Chemical shifts are in ppm, and coupling constants (*J*) in Hz are given in parentheses.

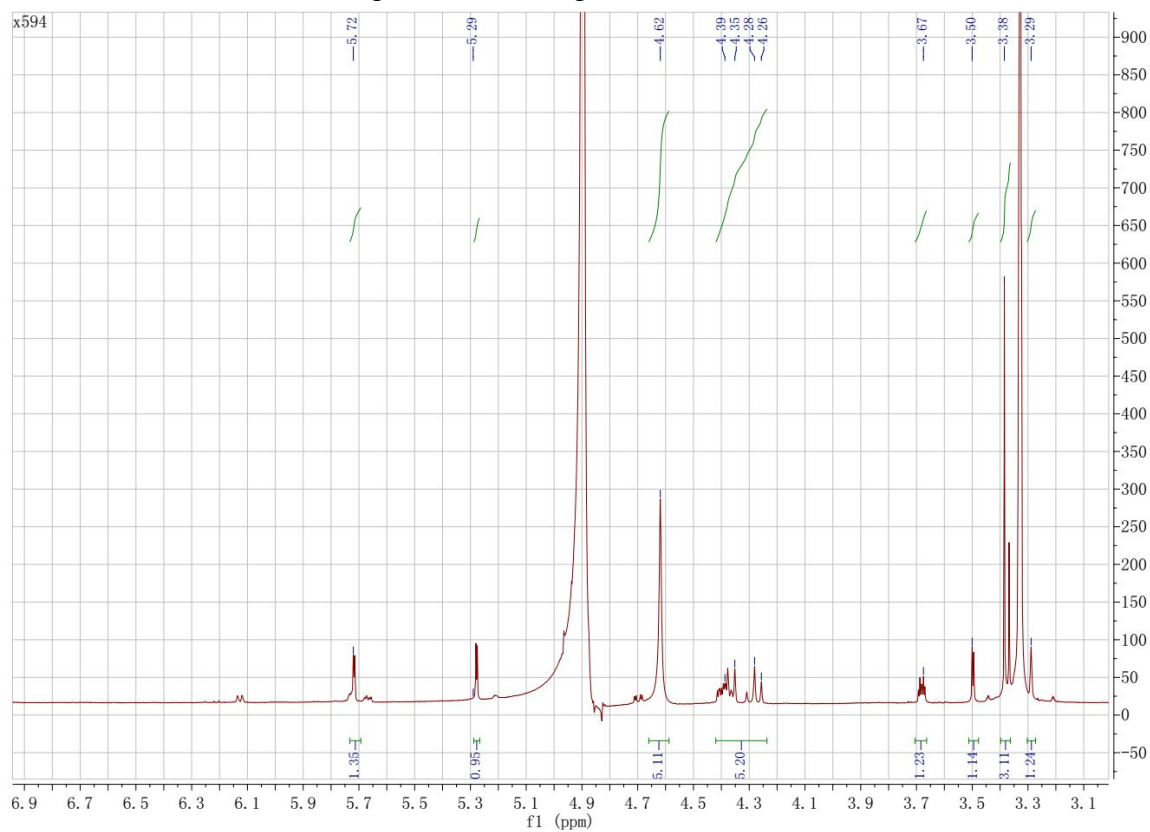
HR-ESI-MS spectrum of compound 1



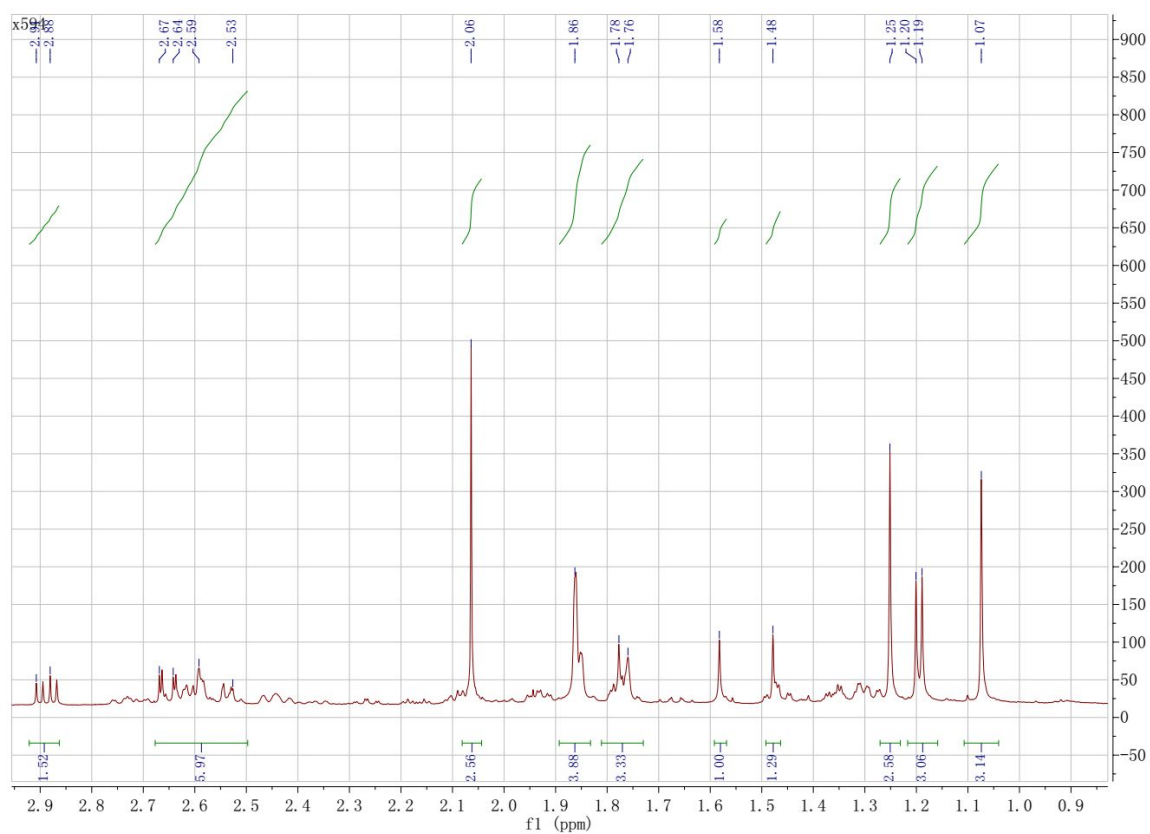
¹H NMR spectrum of compound 1 CD₃OD at 600 MHz



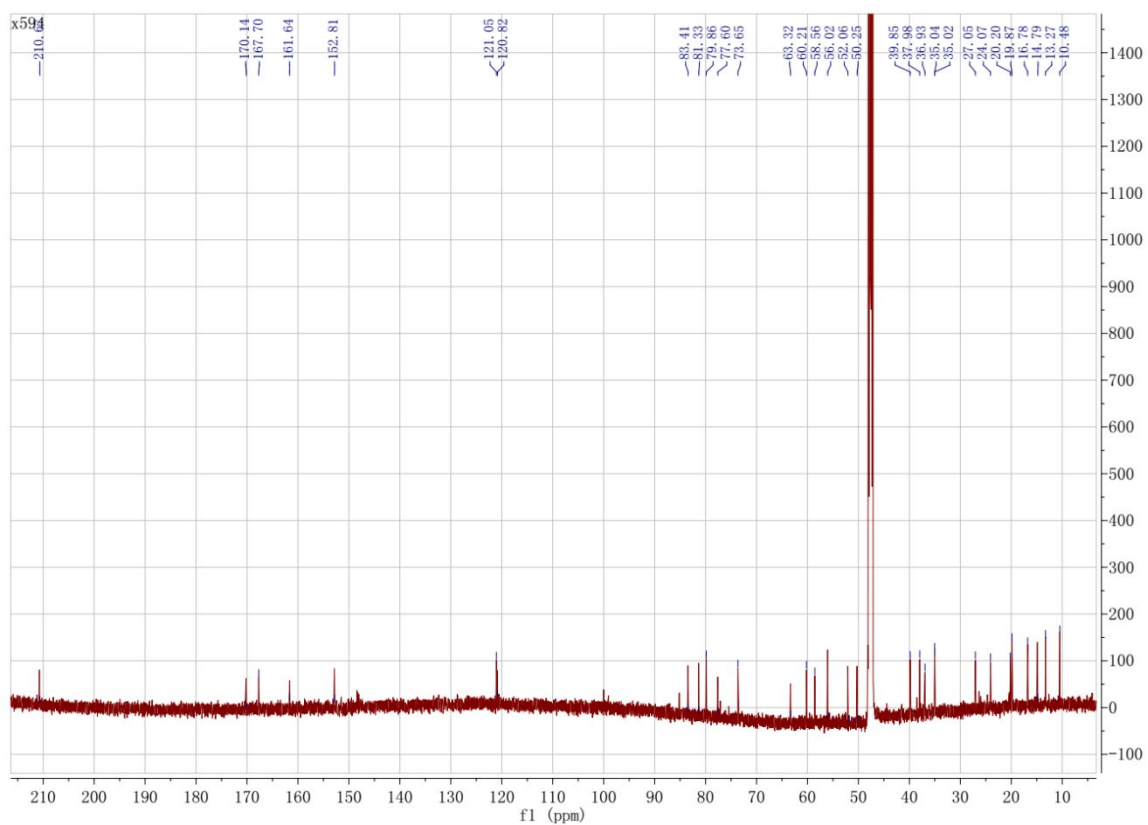
Detailed ¹H NMR spectrum of compound **1** in CD₃OD at 600 MHz



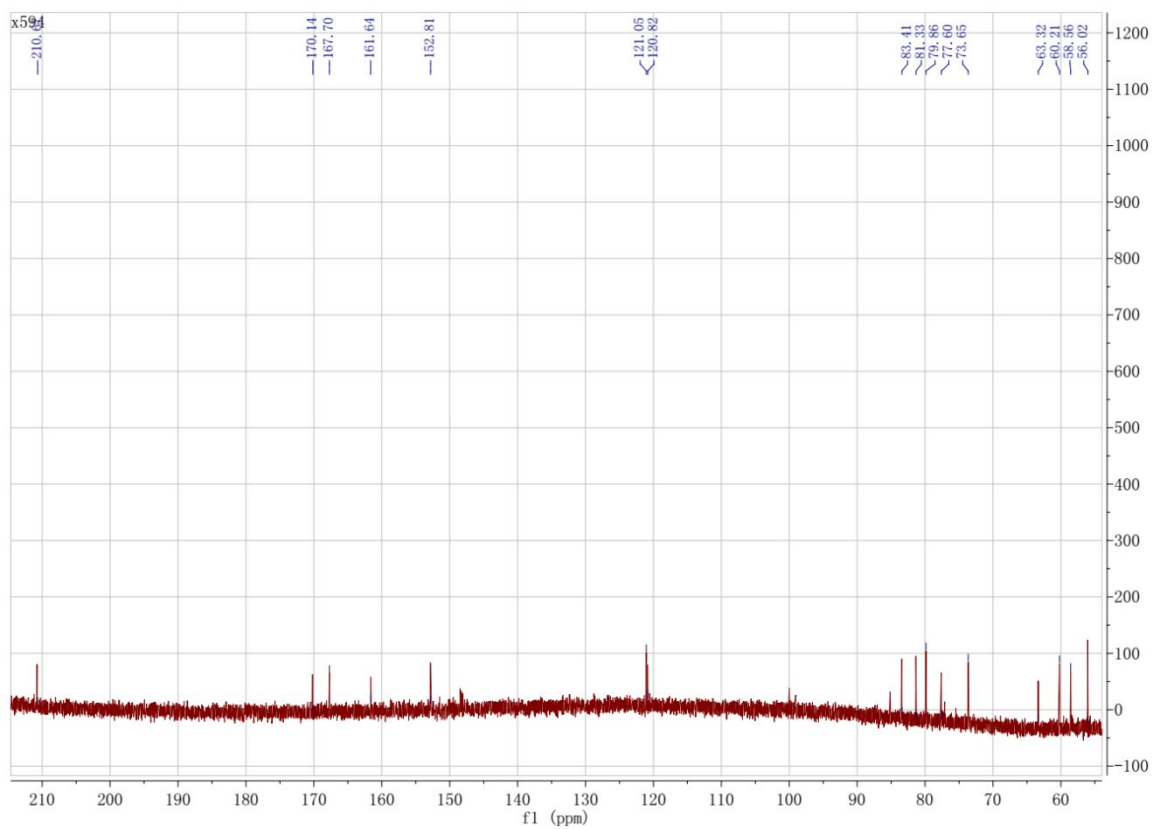
Detailed ¹H NMR spectrum of compound **1** in CD₃OD at 600 MHz



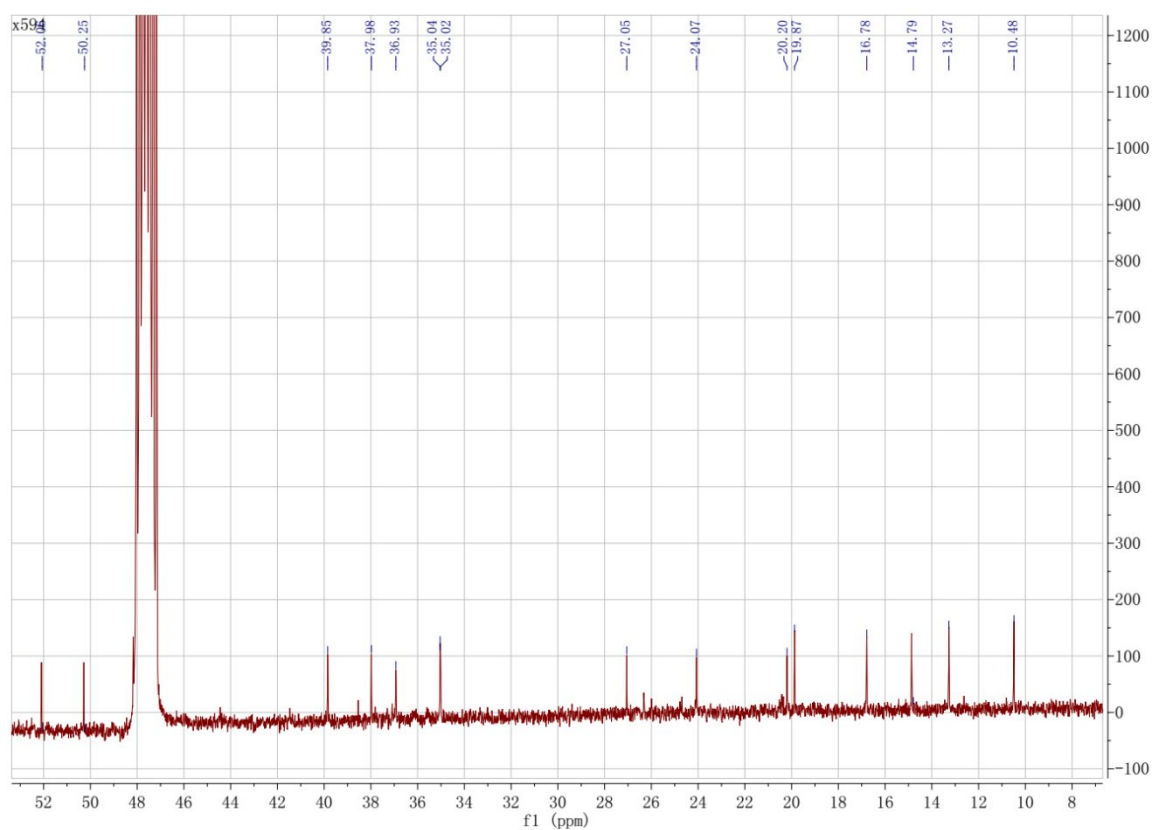
^{13}C NMR spectrum of compound **1** in CD_3OD at 150 MHz



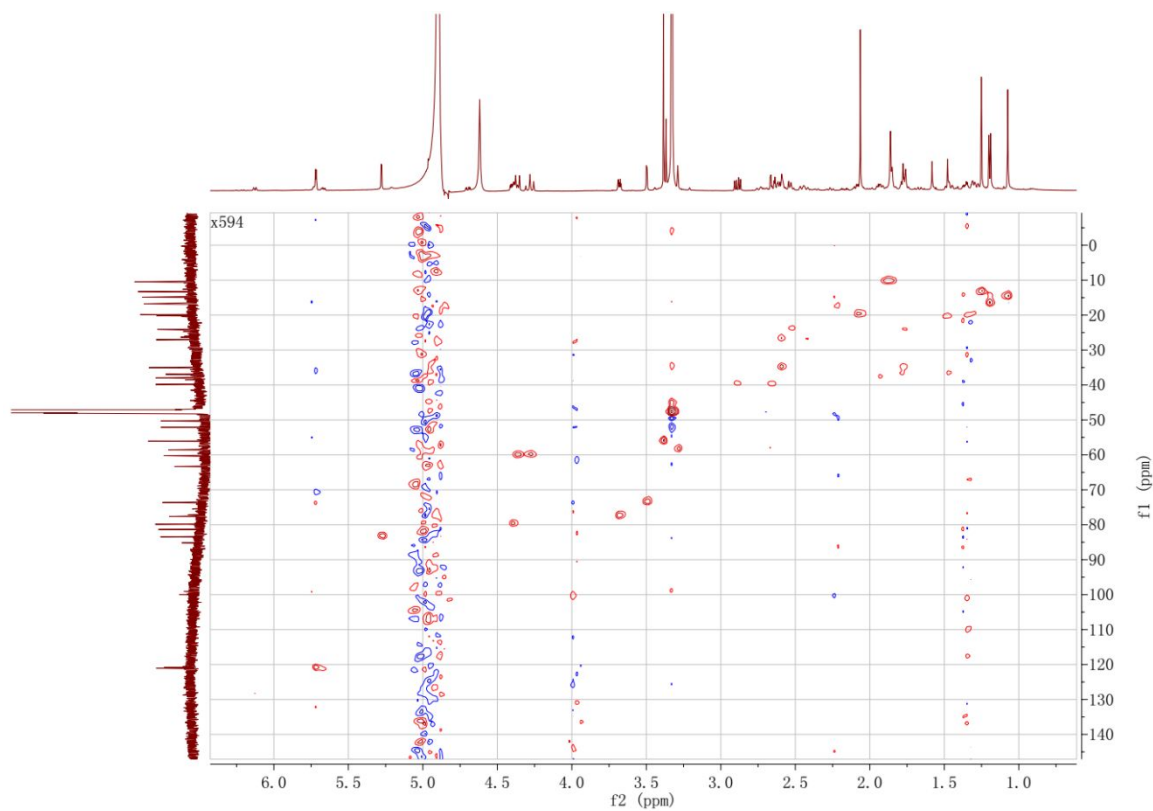
Detailed ^{13}C NMR spectrum of compound **1** in CD_3OD at 150 MHz



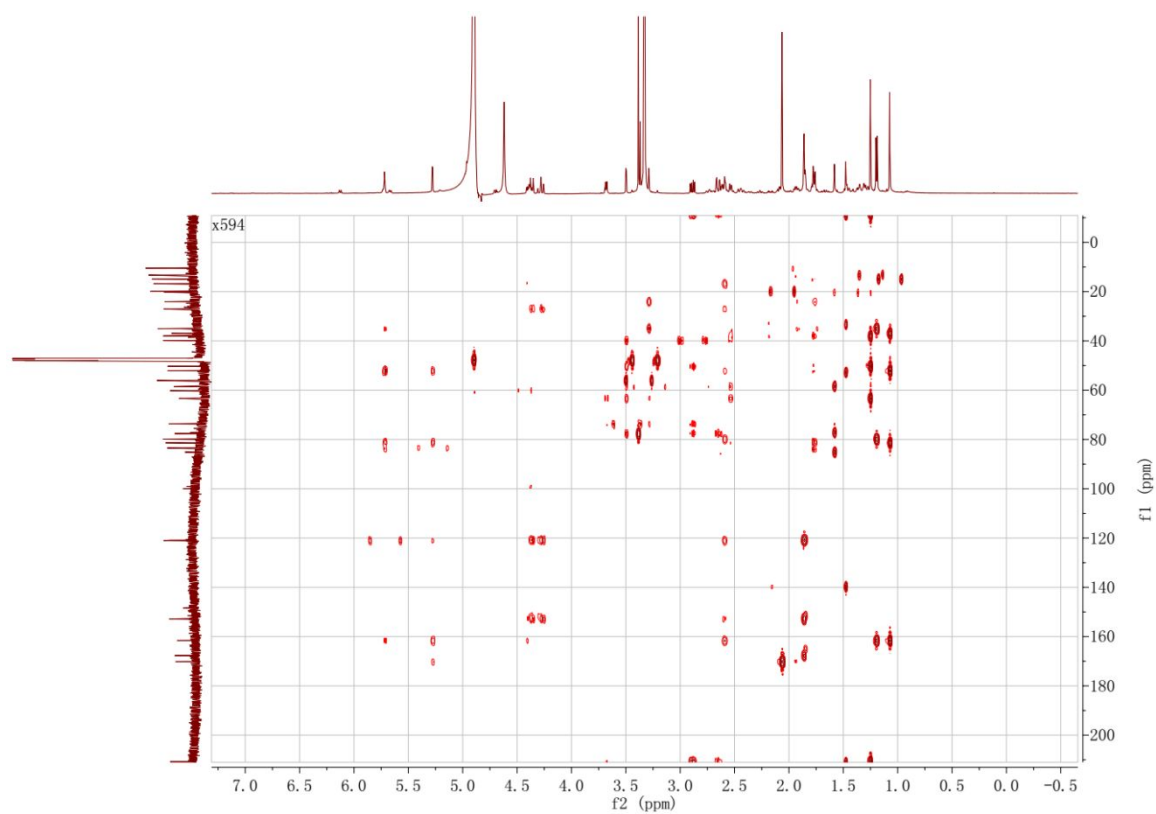
Detailed ^{13}C NMR spectrum of compound **1** in CD_3OD at 150 MHz



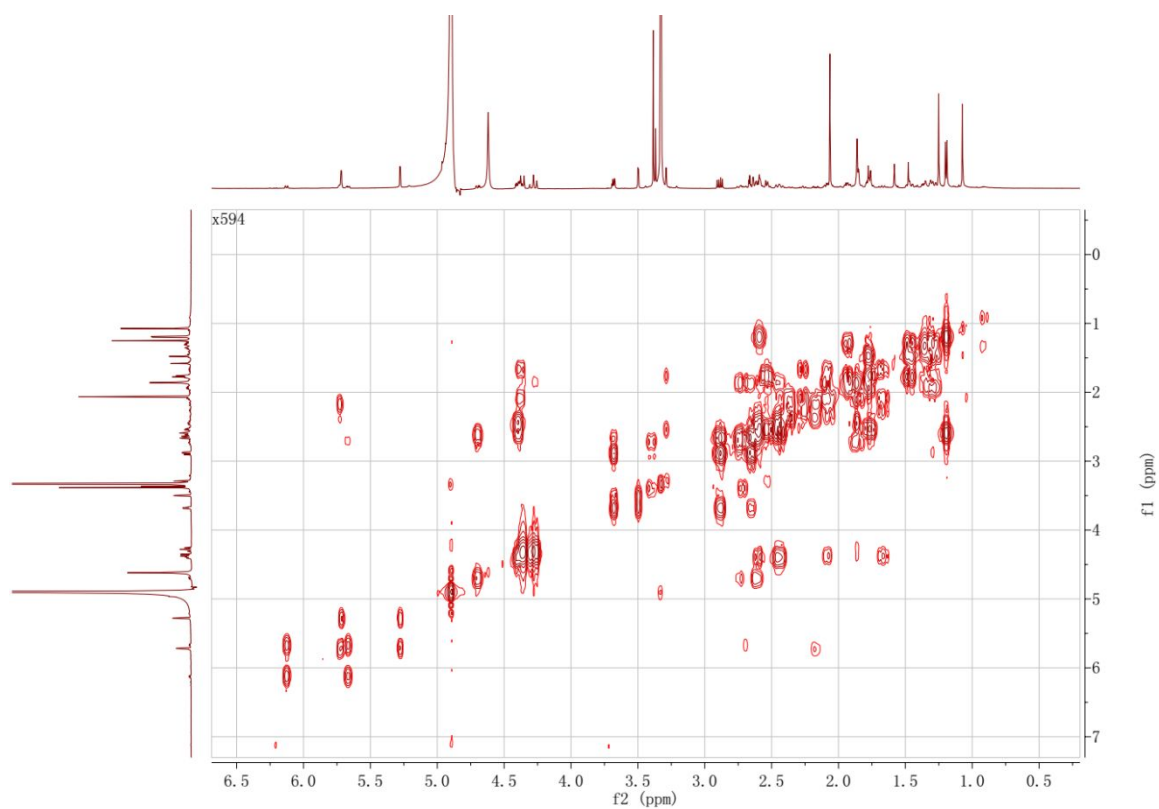
HSQC spectrum of compound **1** in CD_3OD



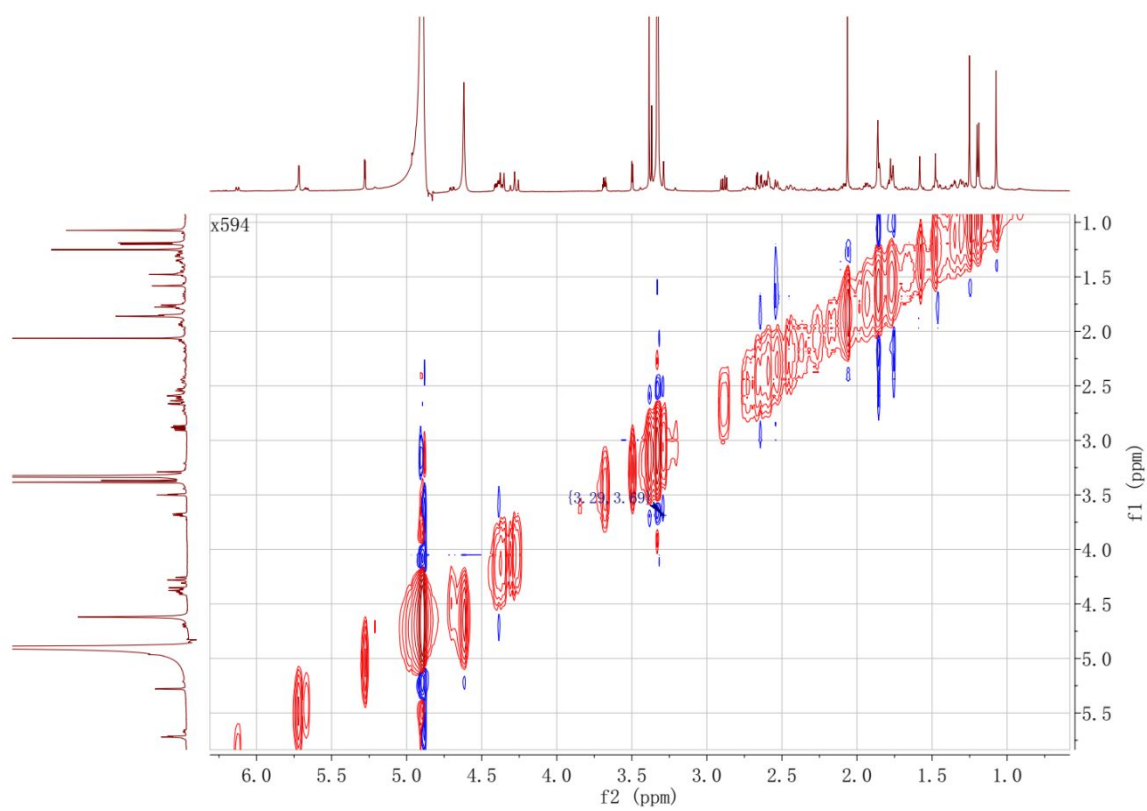
HMBC spectrum of compound **1** in CD₃OD



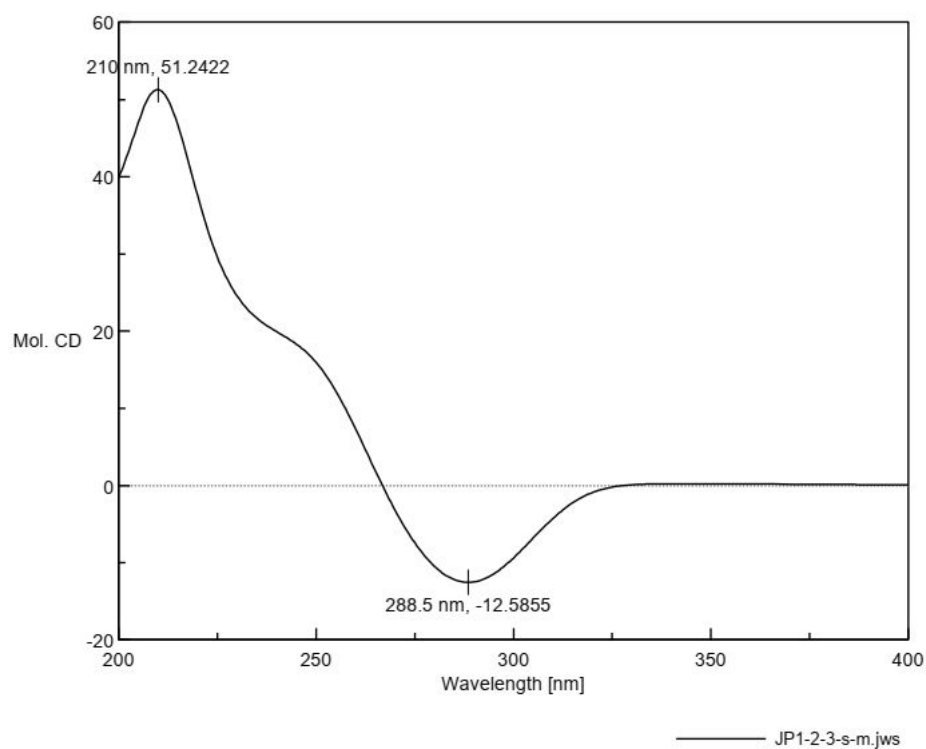
COSY spectrum of compound **1** in CD₃OD



NOESY spectrum of compound **1** in CD₃OD



ECD spectrum of 1



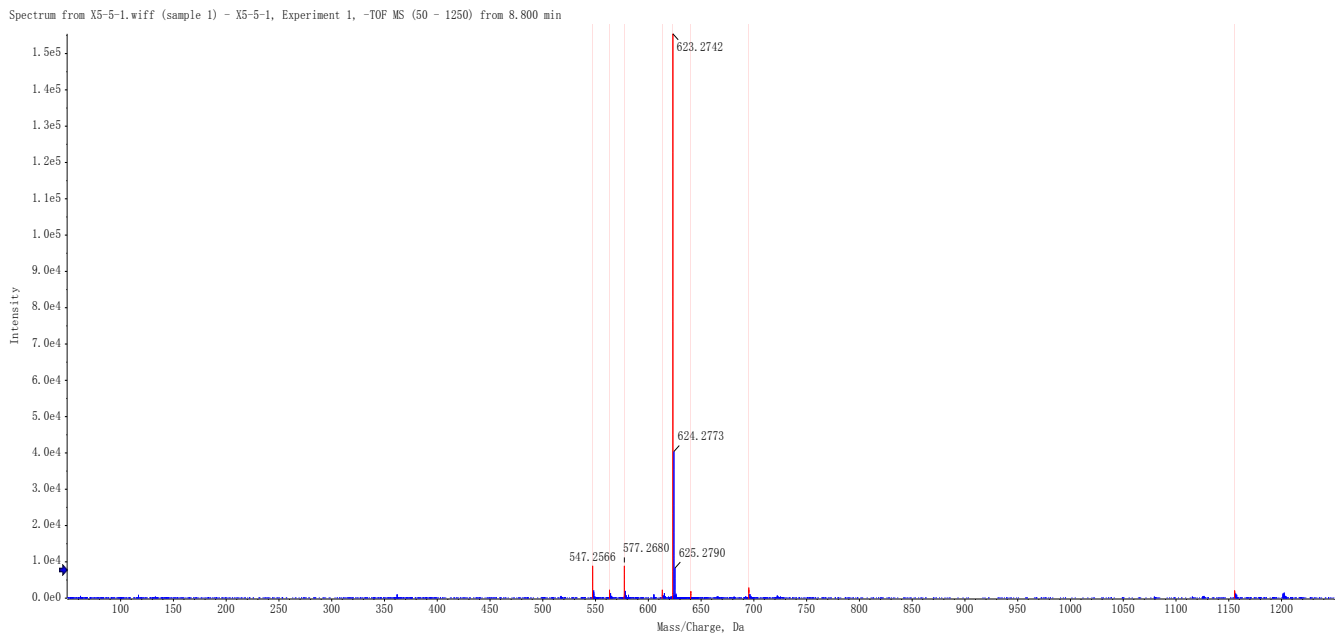
[Measurement Information]
Instrument Name J-815
Model Name J-815
Serial No. A024461168

Accessory Standard
Accessory S/N A024461168
Cell Length 1 mm

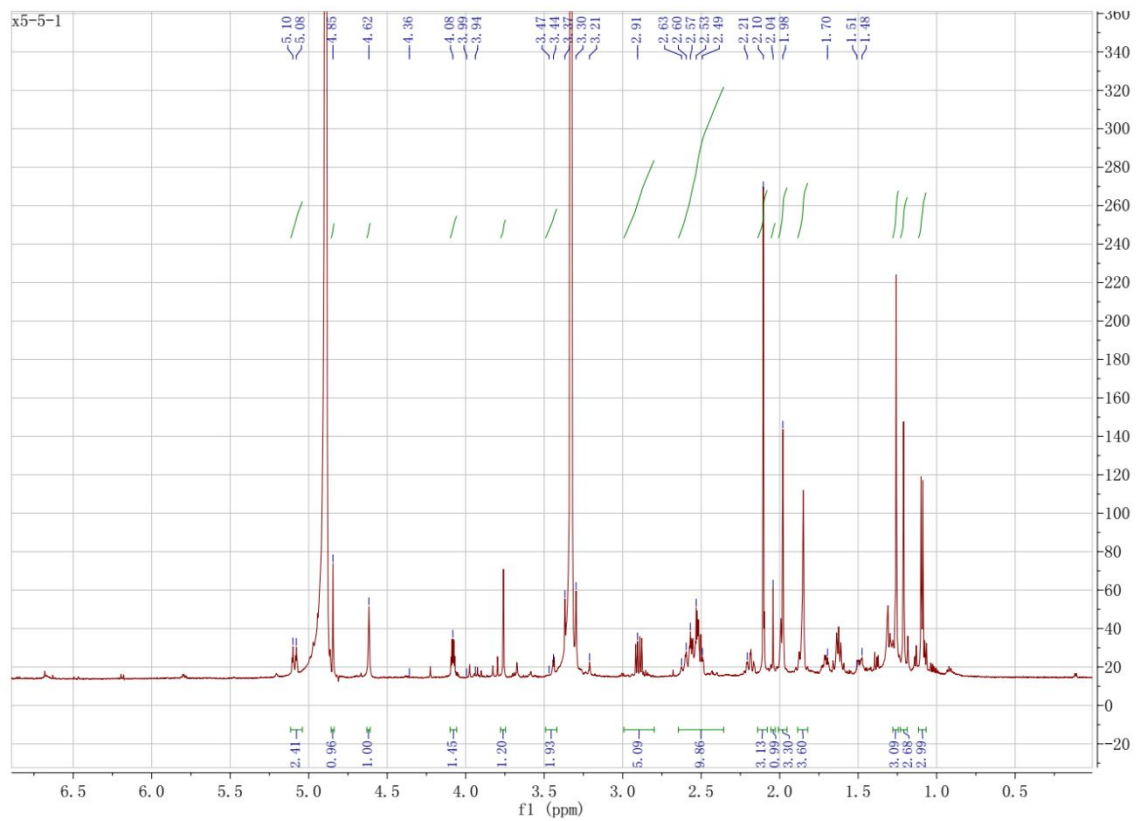
Measurement date 2020/3/19 11:25

Photometric Mode CD, HT, Abs
Measure Range 400 - 200 nm
Data pitch 0.5 nm
Sensitivity Standard
D.I.T. 1 sec
Bandwidth 1.00 nm
Start Mode Immediately
Scanning Speed 100 nm/min
Baseline Correction Baseline
Shutter Control Auto
CD Detector PMT
PMT Voltage Auto
Accumulations 2
Solvent MEOH
Concentration 0.4 (w/v)%

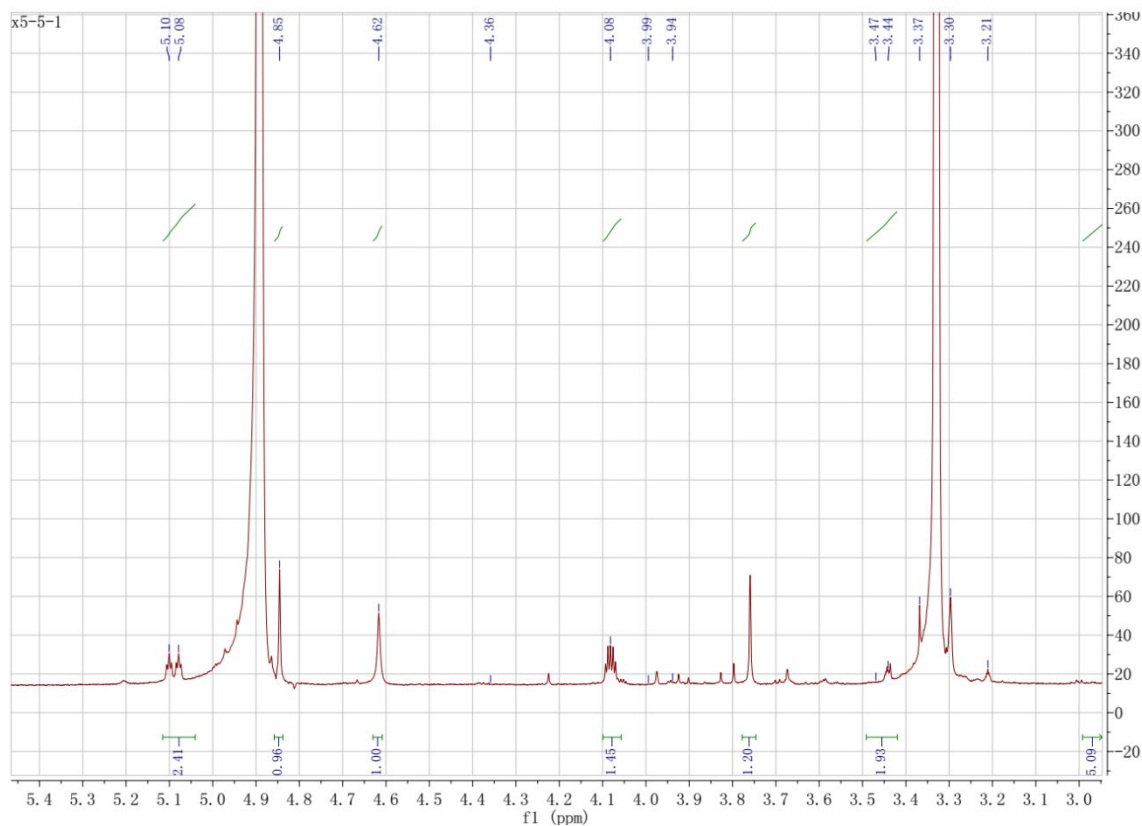
HR-ESI-MS spectrum of compound 2



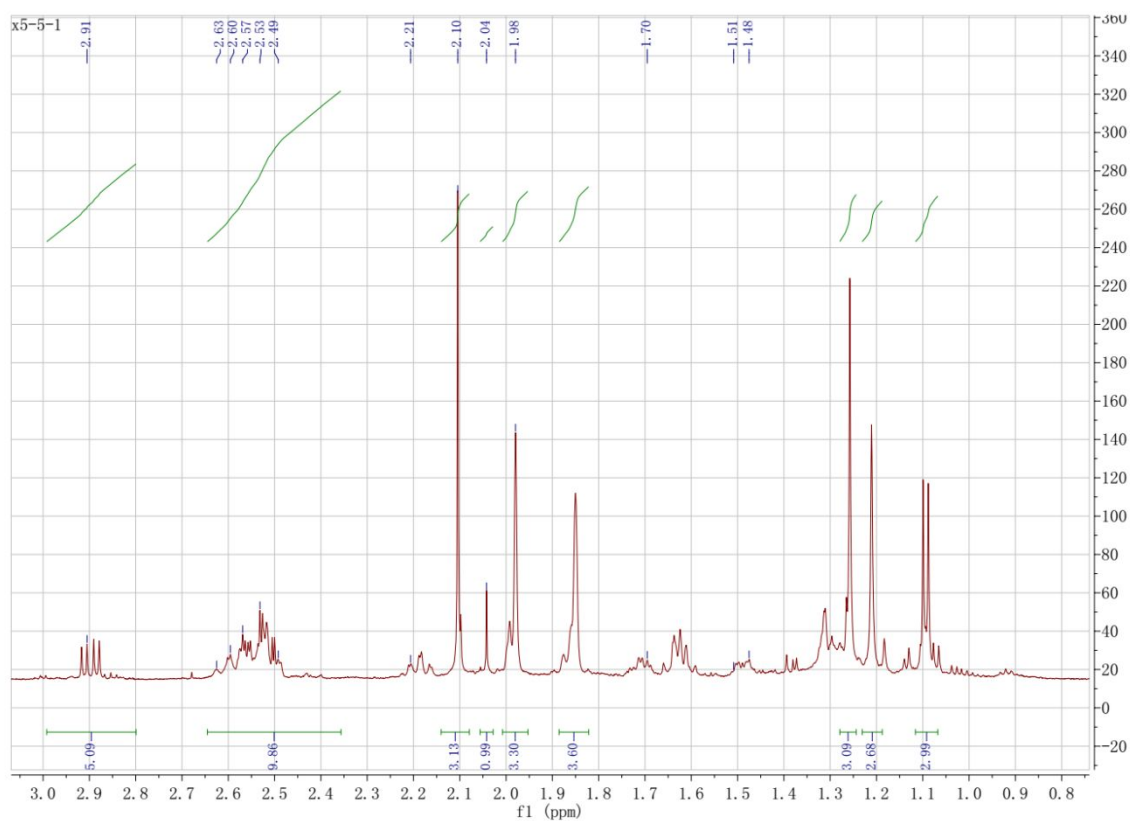
^1H NMR spectrum of compound 2 in CD_3OD at 600 MHz



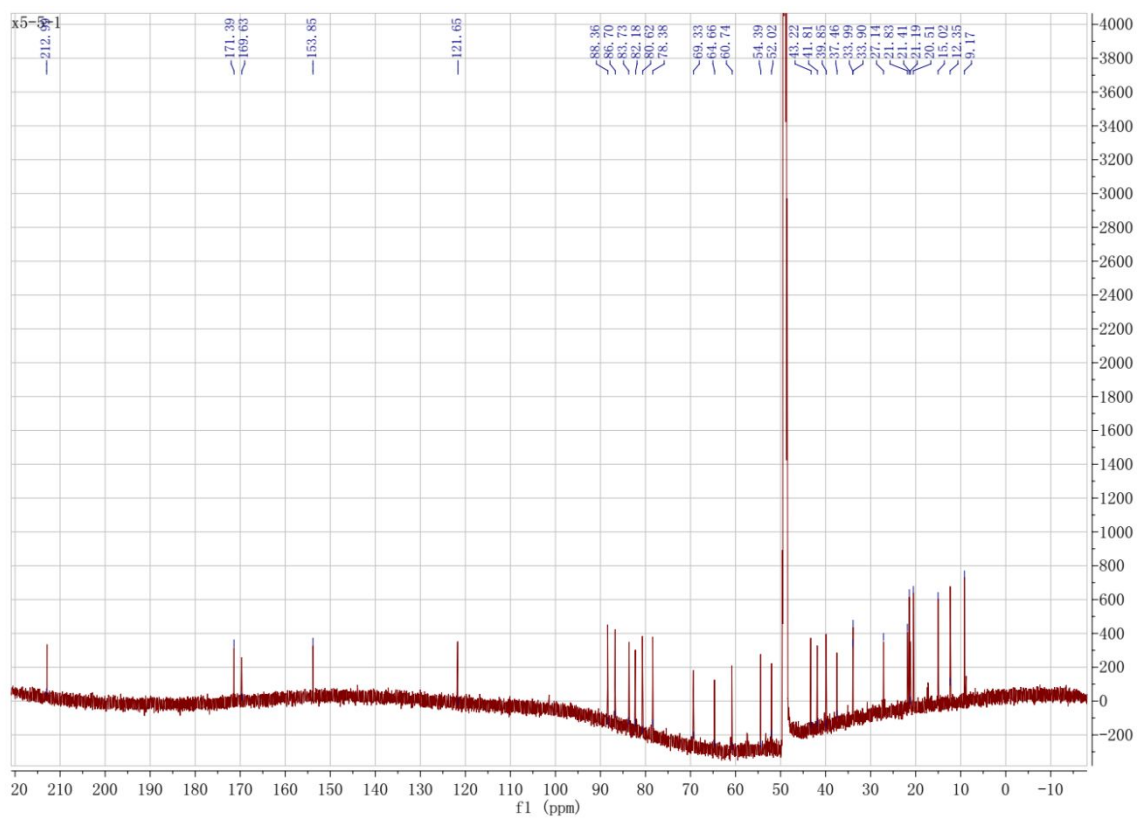
Detailed ^1H NMR spectrum of compound **2** in CD_3OD at 600 MHz



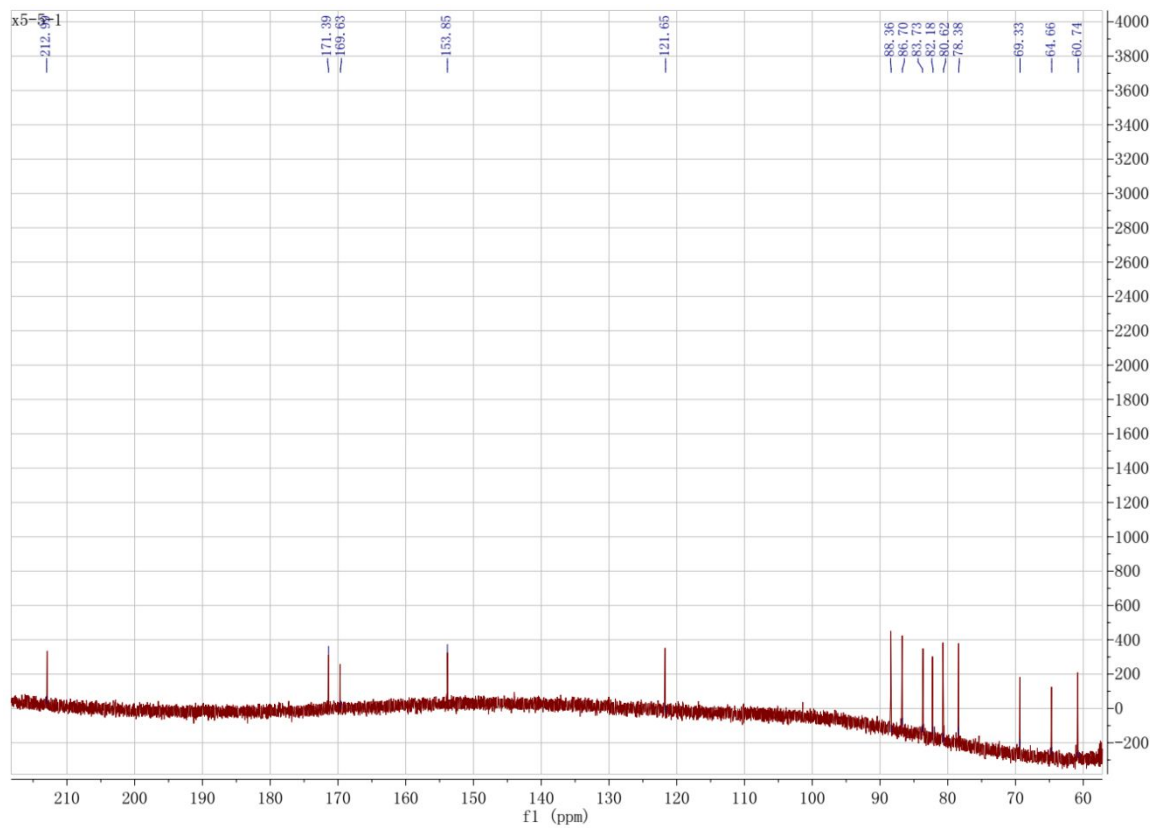
Detailed ^1H NMR spectrum of compound **2** in CD_3OD at 600 MHz



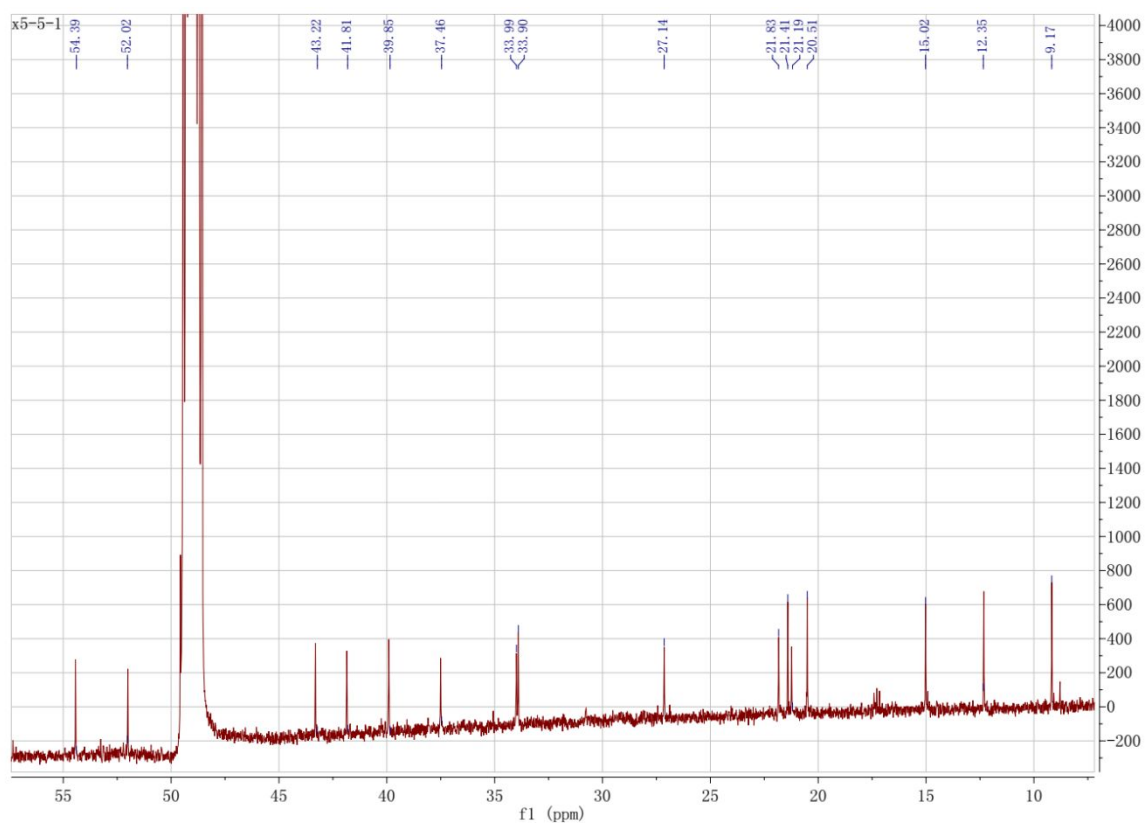
^{13}C NMR spectrum of compound **2** in CD_3OD at 150 MHz



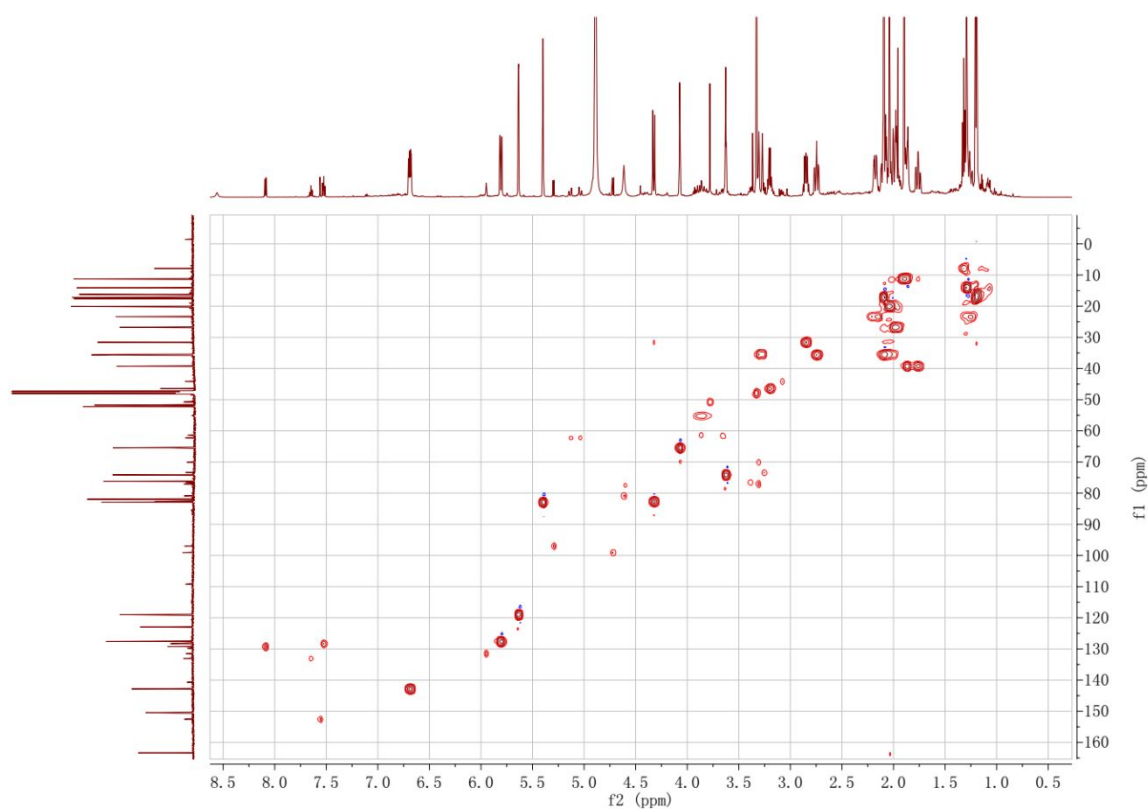
Detailed ^{13}C NMR spectrum of compound **2** in CD_3OD at 150 MHz



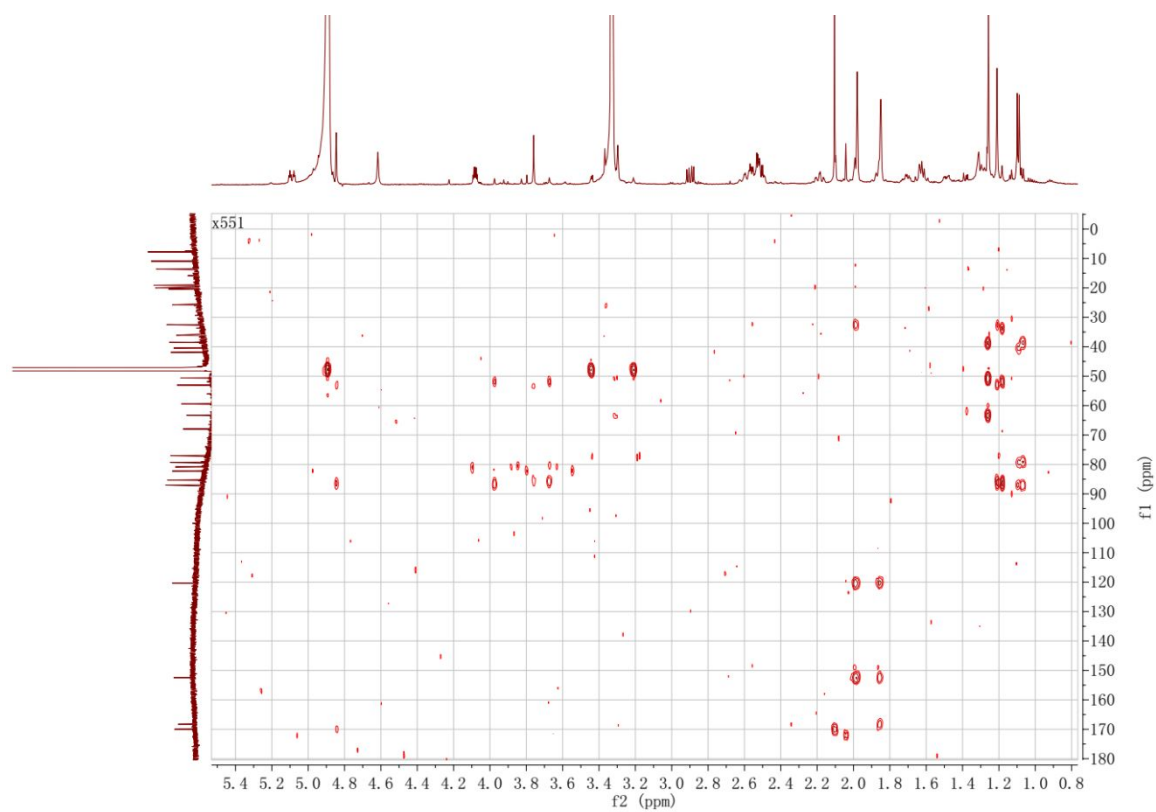
Detailed ^{13}C NMR spectrum of compound **2** in CD_3OD at 150 MHz



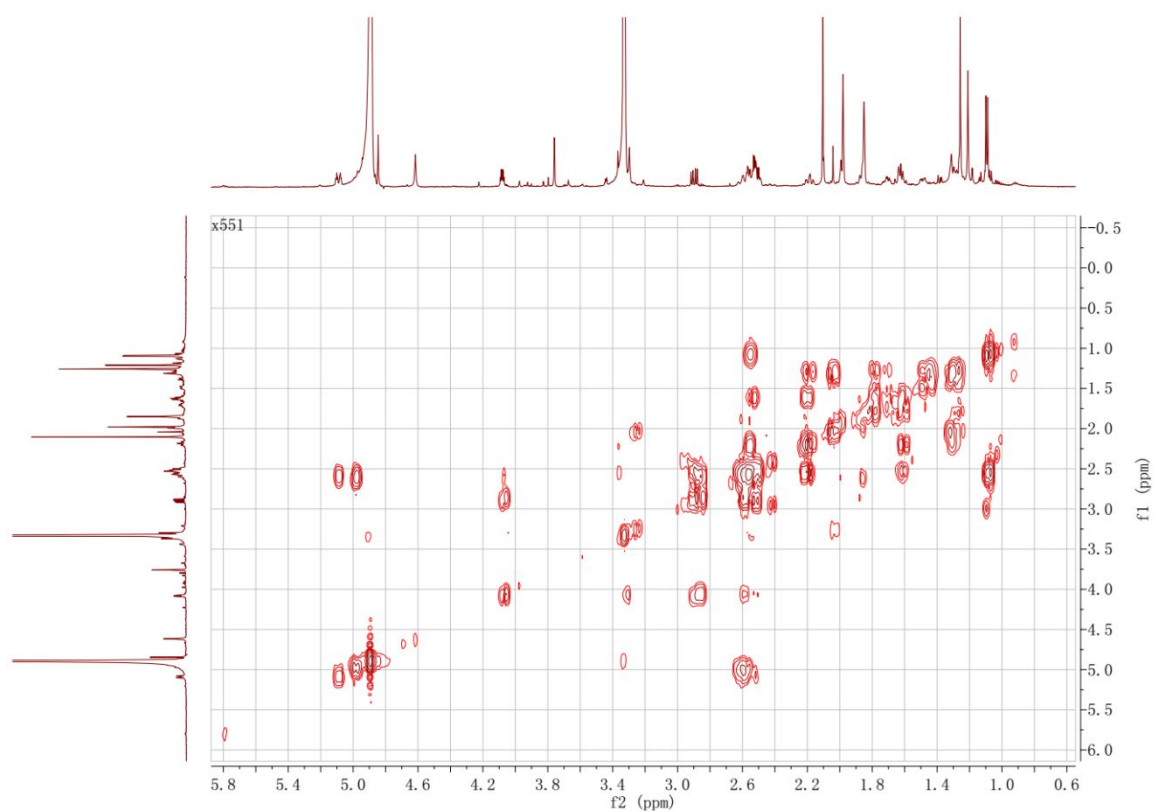
HSQC spectrum of compound **2** in CD_3OD



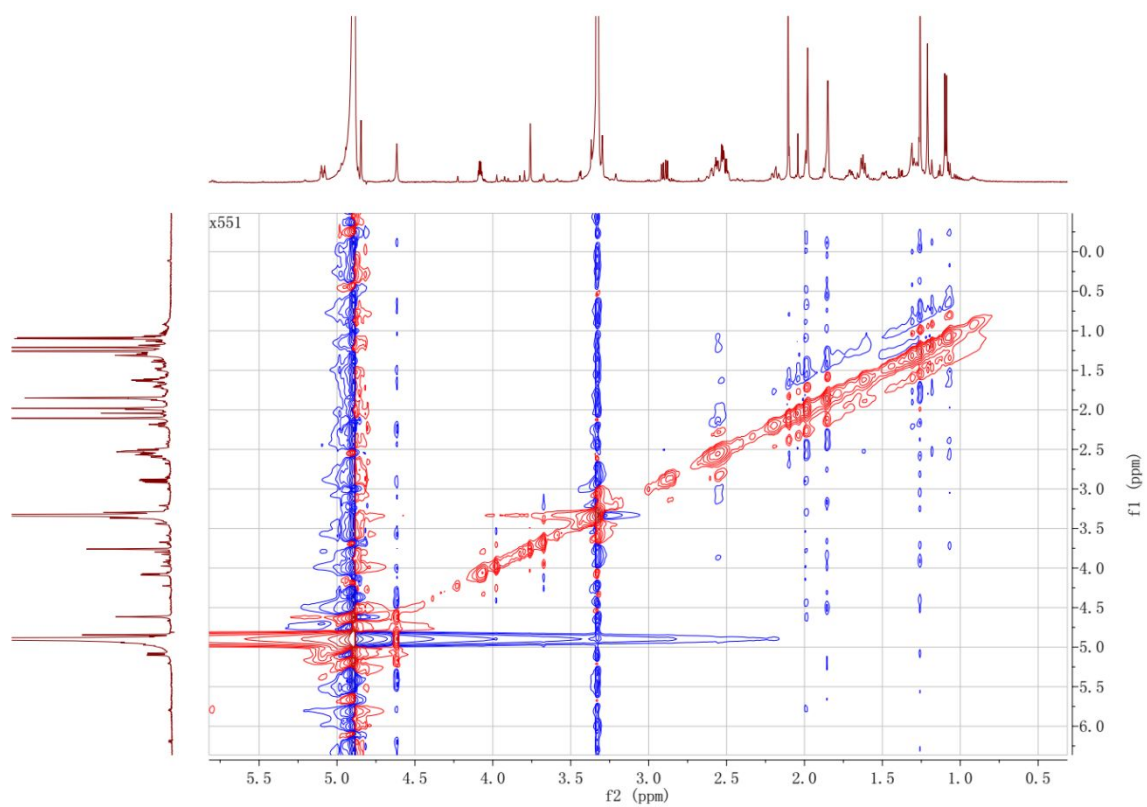
HMBC spectrum of compound **2** in CD₃OD



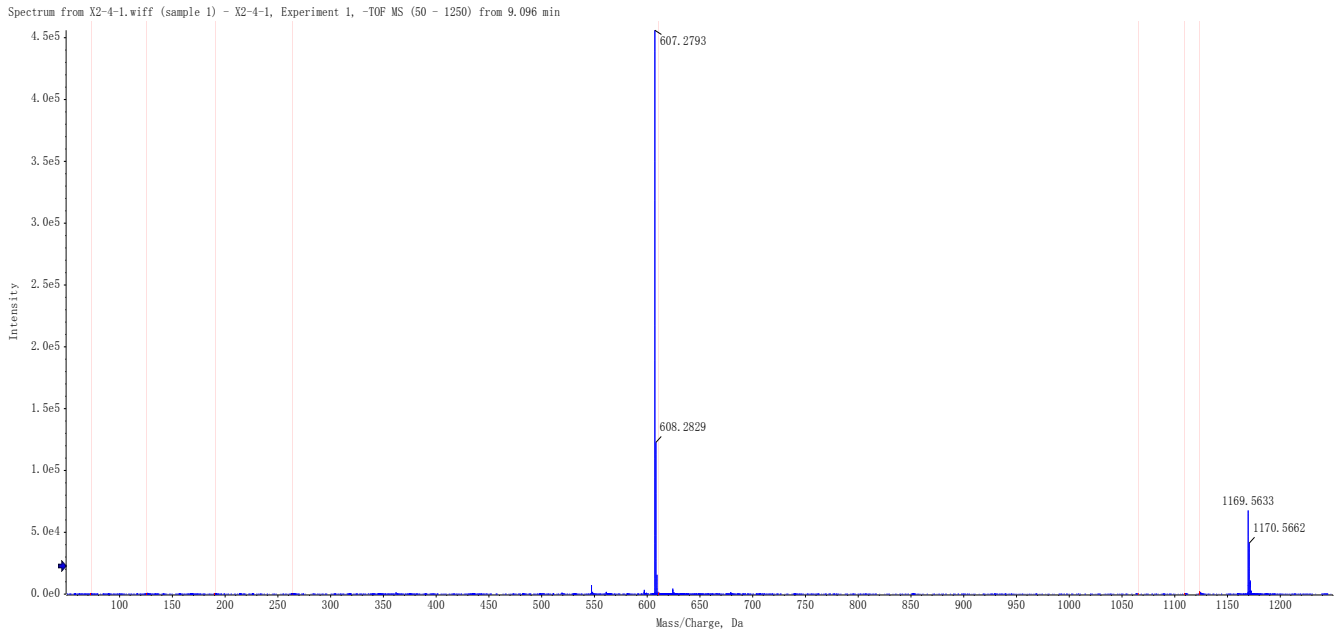
COSY spectrum of compound **2** in CD₃OD



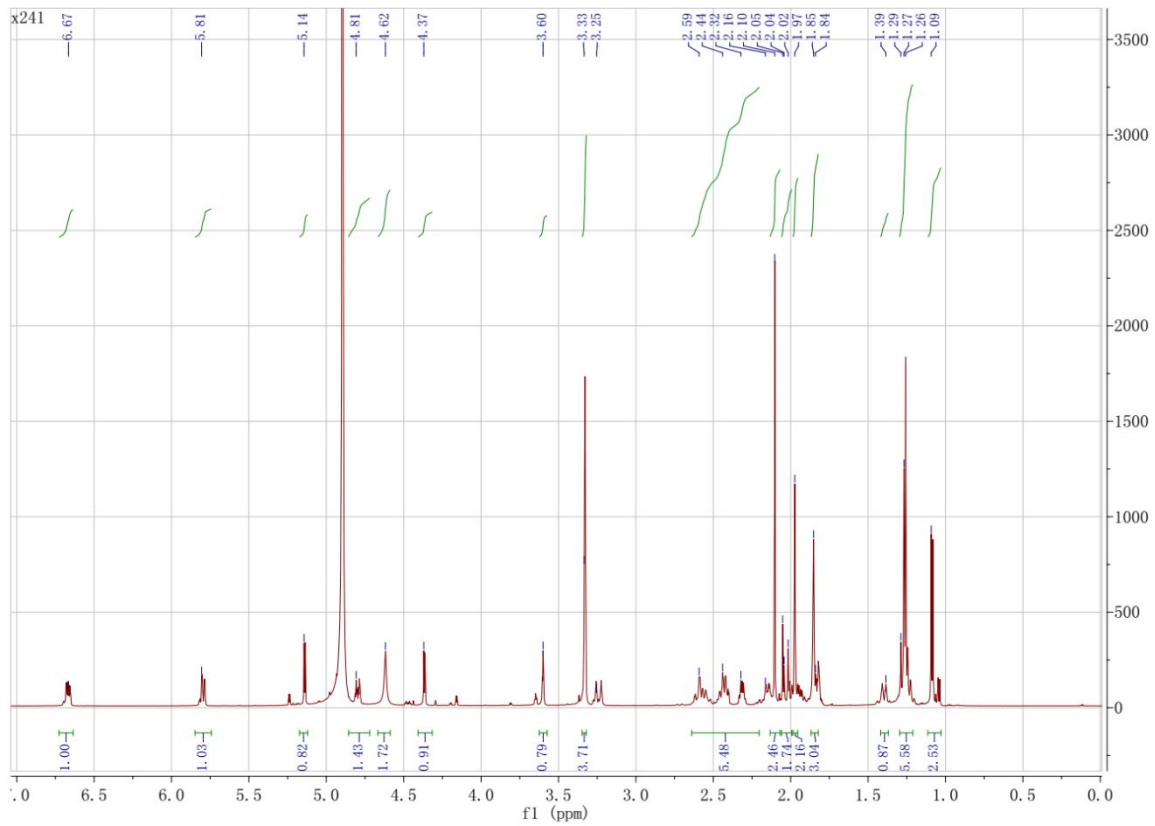
NOESY spectrum of compound **2** in CD₃OD



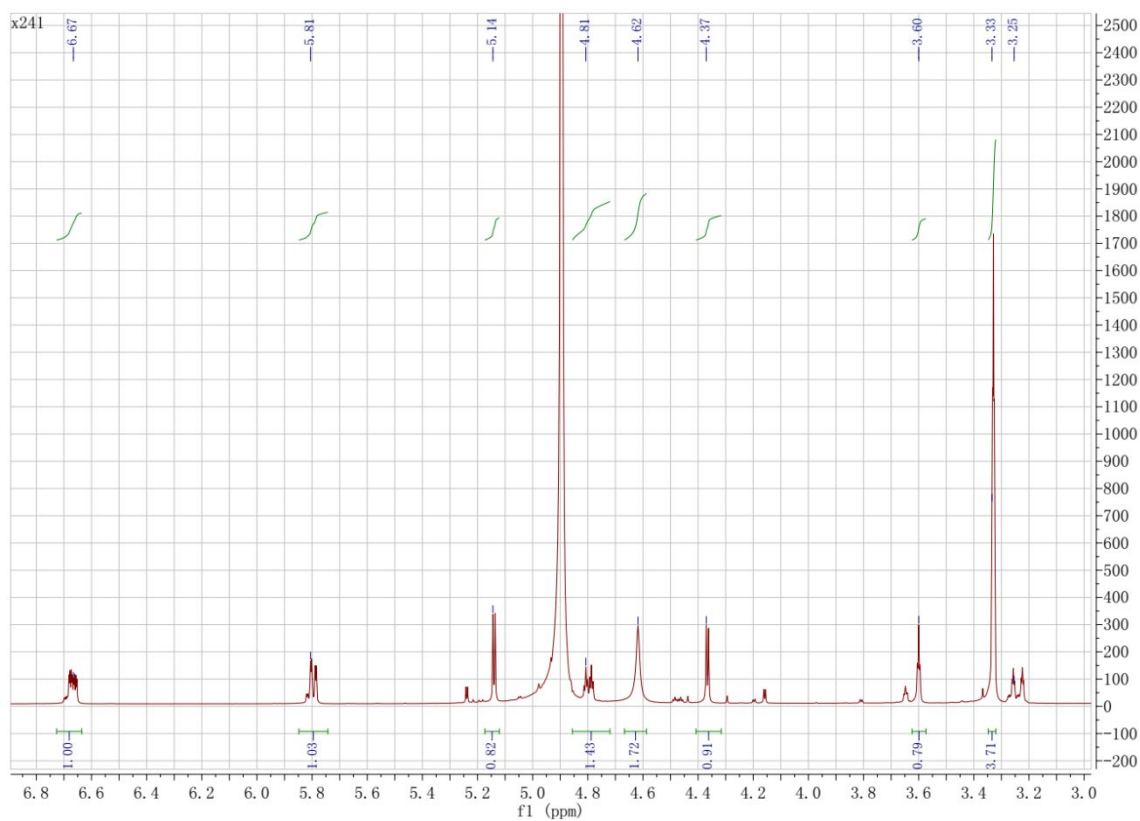
HR-ESI-MS spectrum of compound 3



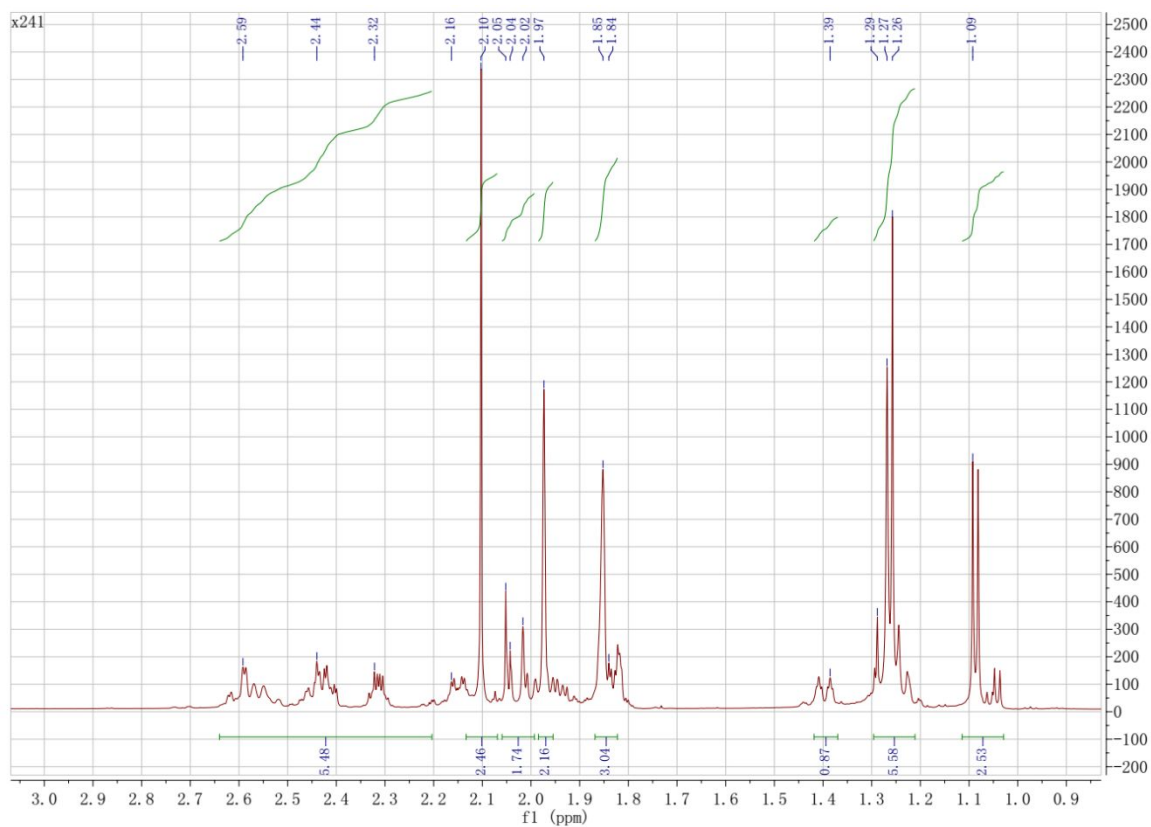
¹H NMR spectrum of compound 3 in CD₃OD at 600 MHz



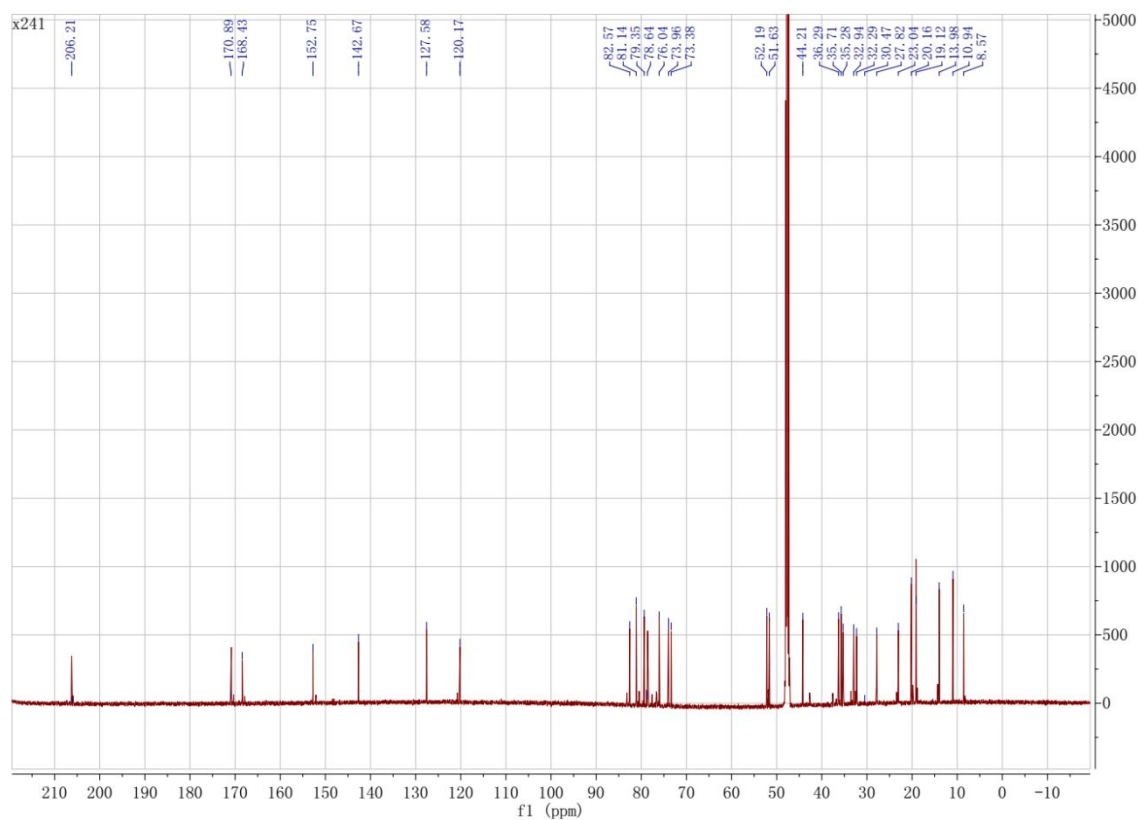
Detailed ^1H NMR spectrum of compound **3** in CD_3OD at 600 MHz



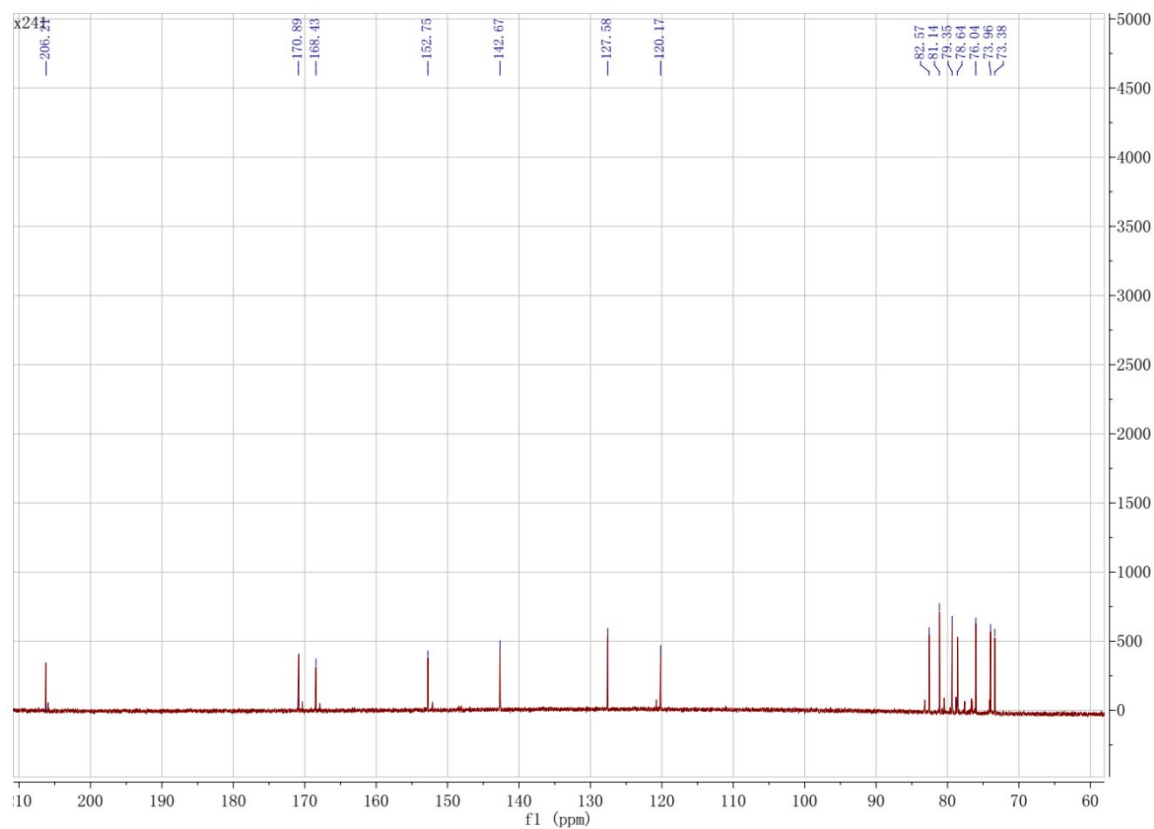
Detailed ^1H NMR spectrum of compound **3** in CD_3OD at 600 MHz



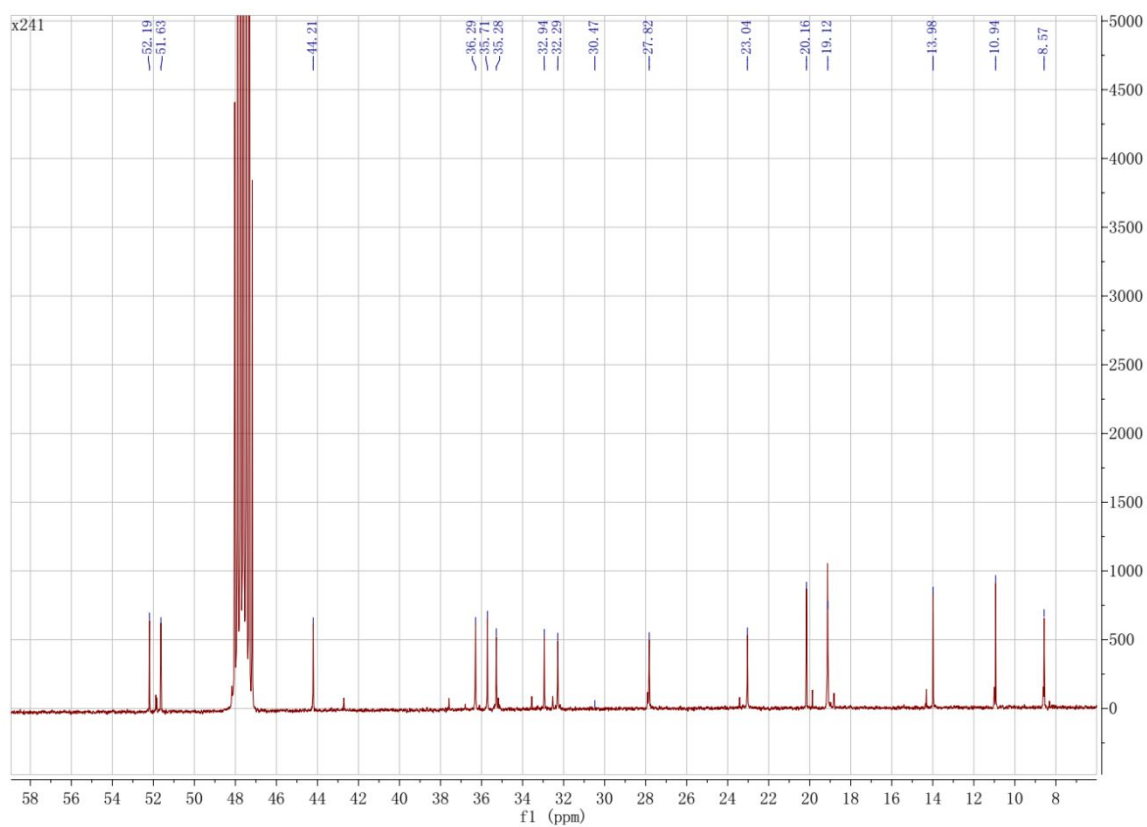
^{13}C NMR spectrum of compound **3** in CD_3OD at 150 MHz



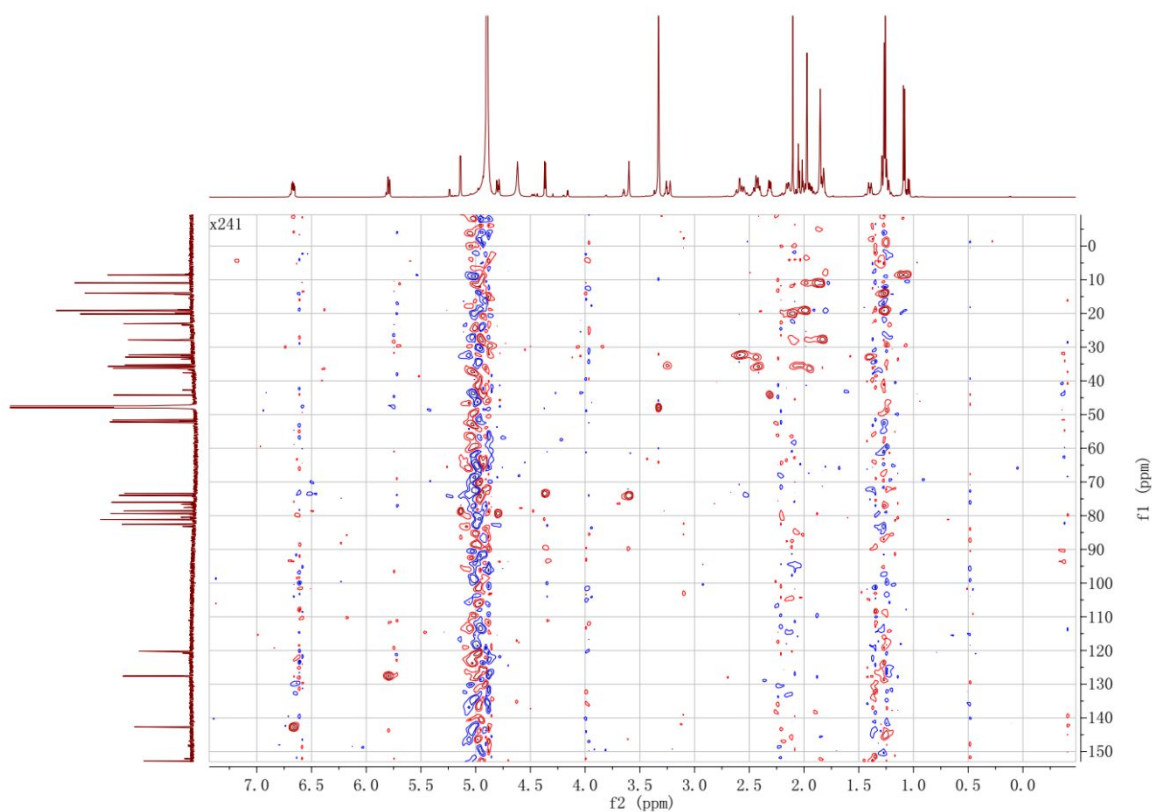
Detailed ^{13}C NMR spectrum of compound **3** in CD_3OD at 150 MHz



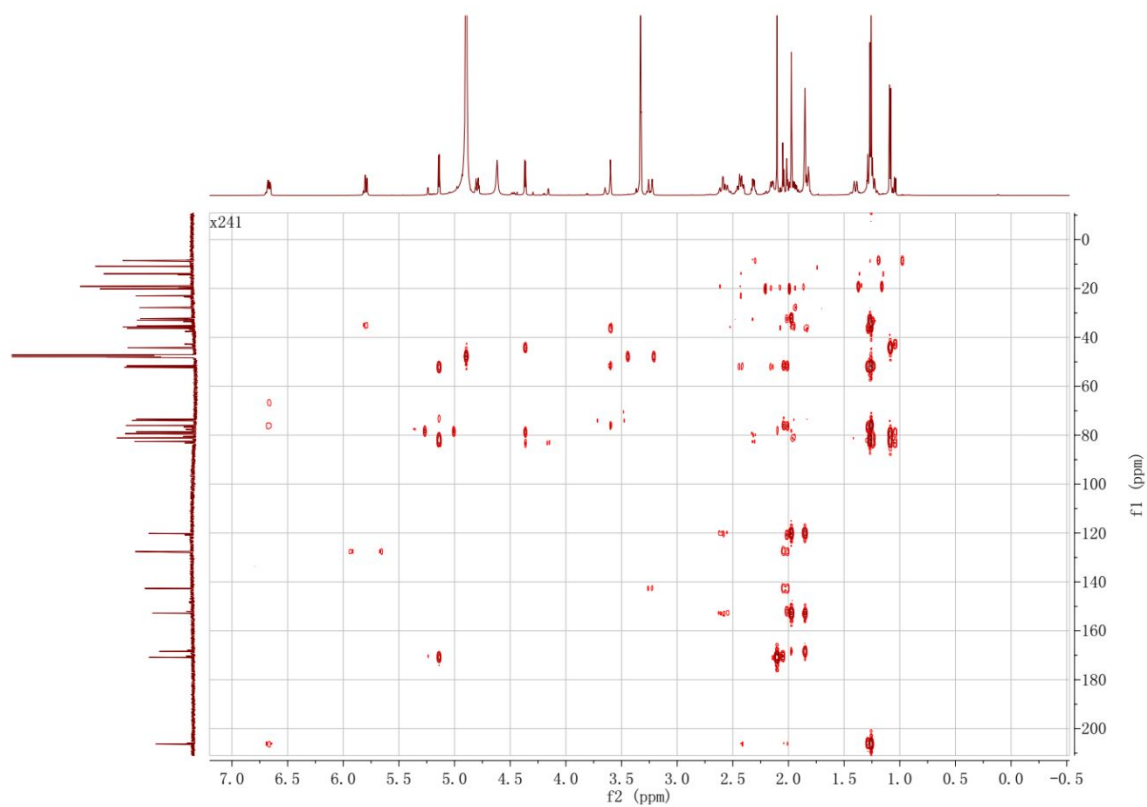
Detailed ^{13}C NMR spectrum of compound **3** in CD_3OD at 150 MHz



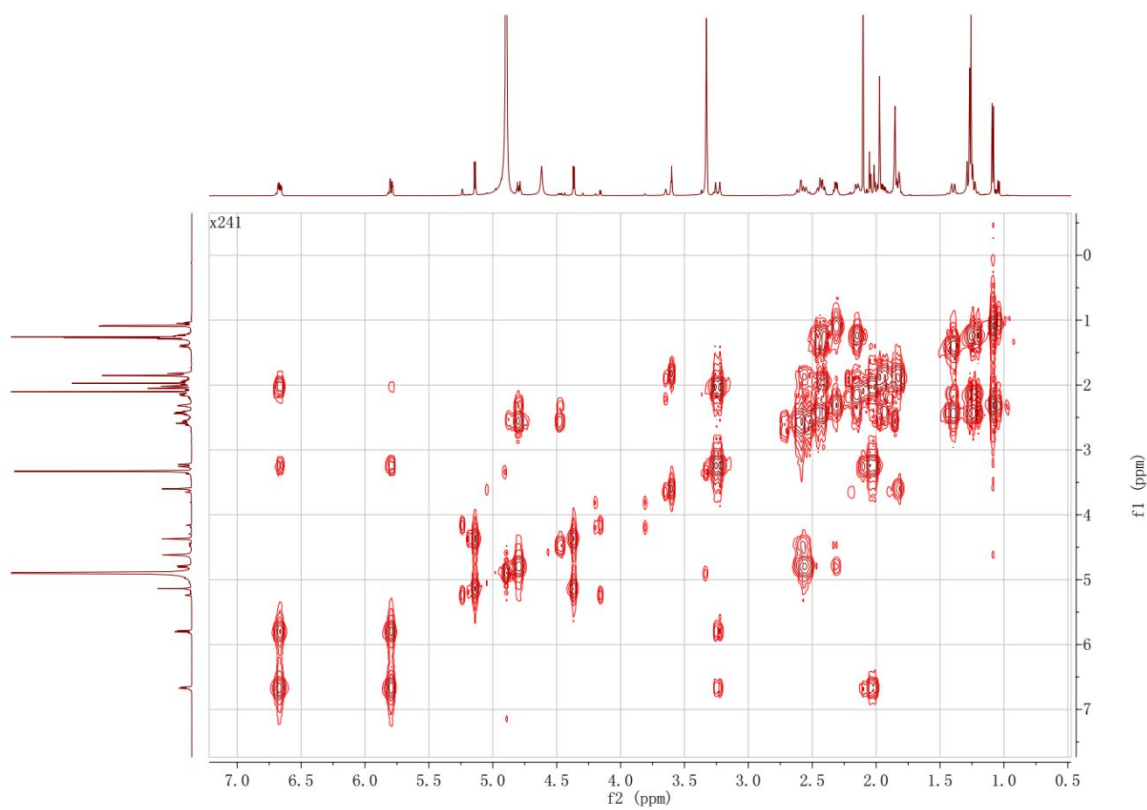
HSQC spectrum of compound **3** in CD_3OD



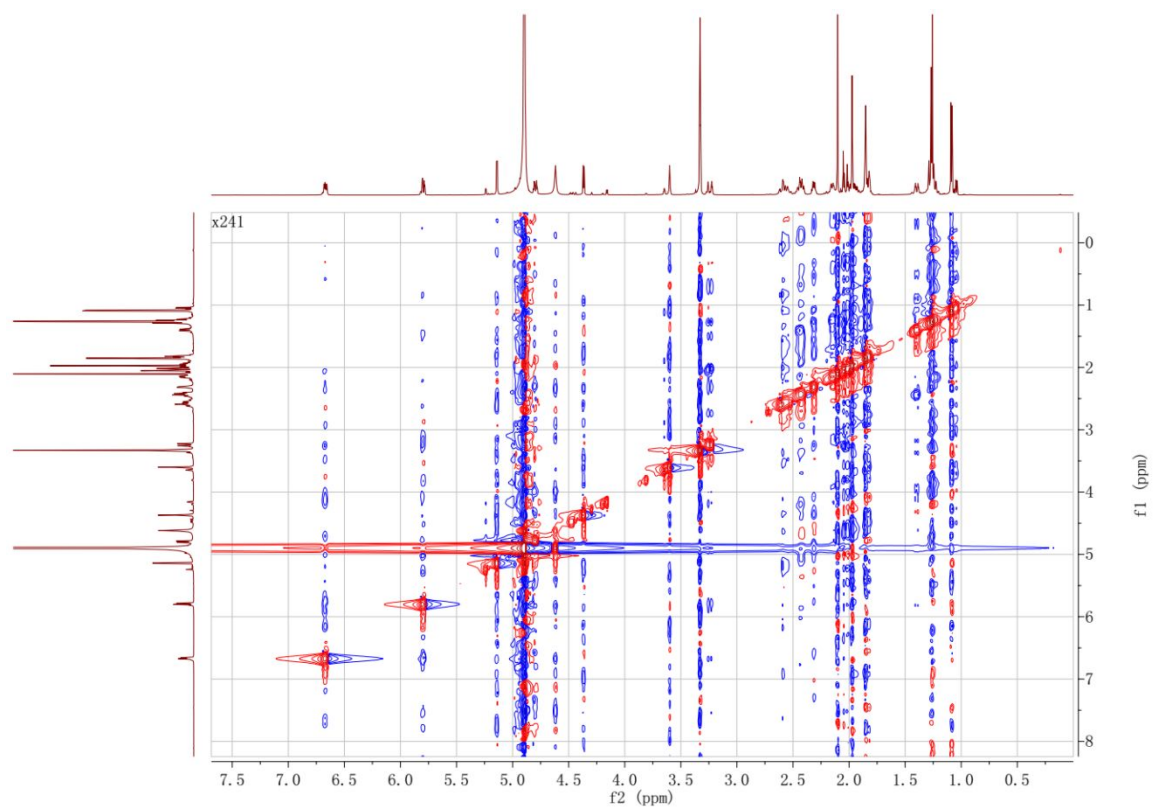
HMBC spectrum of compound **3** in CD₃OD



COSY spectrum of compound **3** in CD₃OD

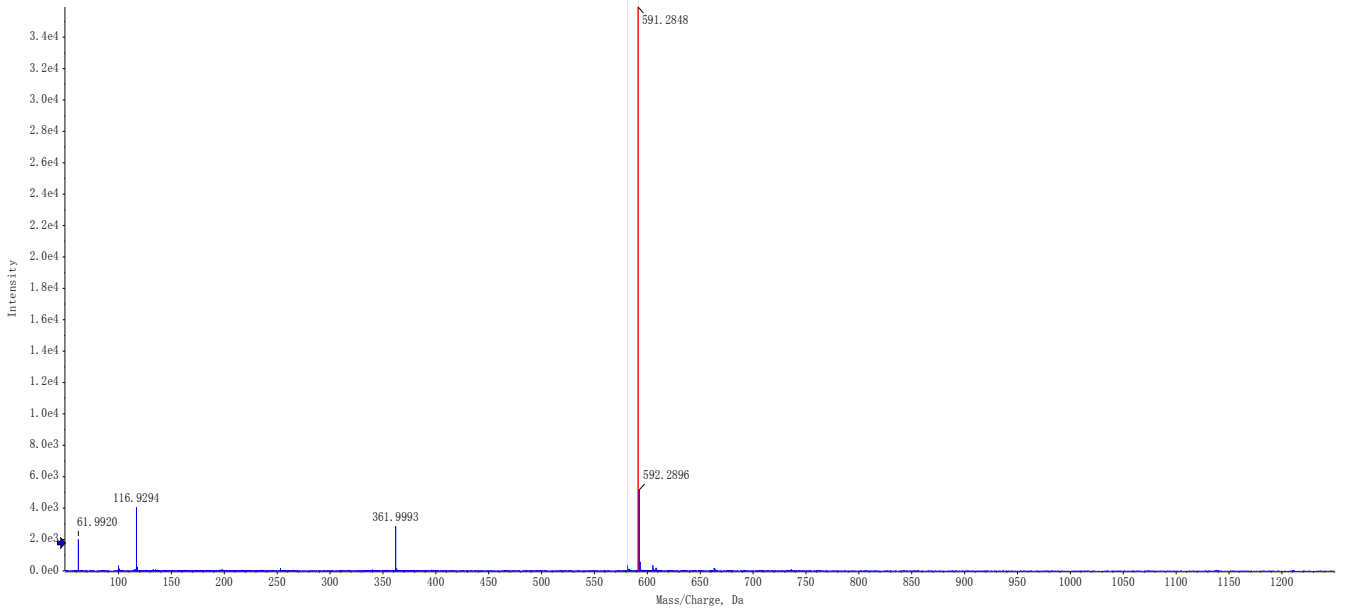


NOESY spectrum of compound **3** in CD₃OD

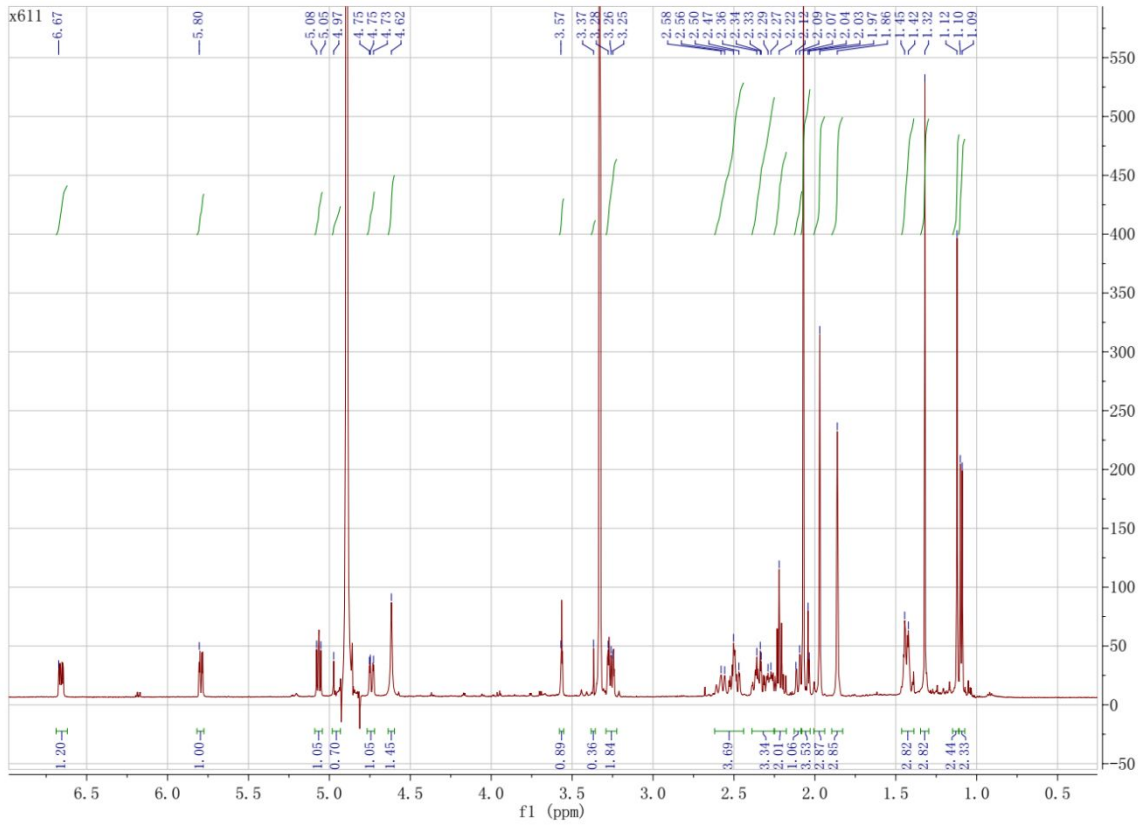


HR-ESI-MS spectrum of compound 4

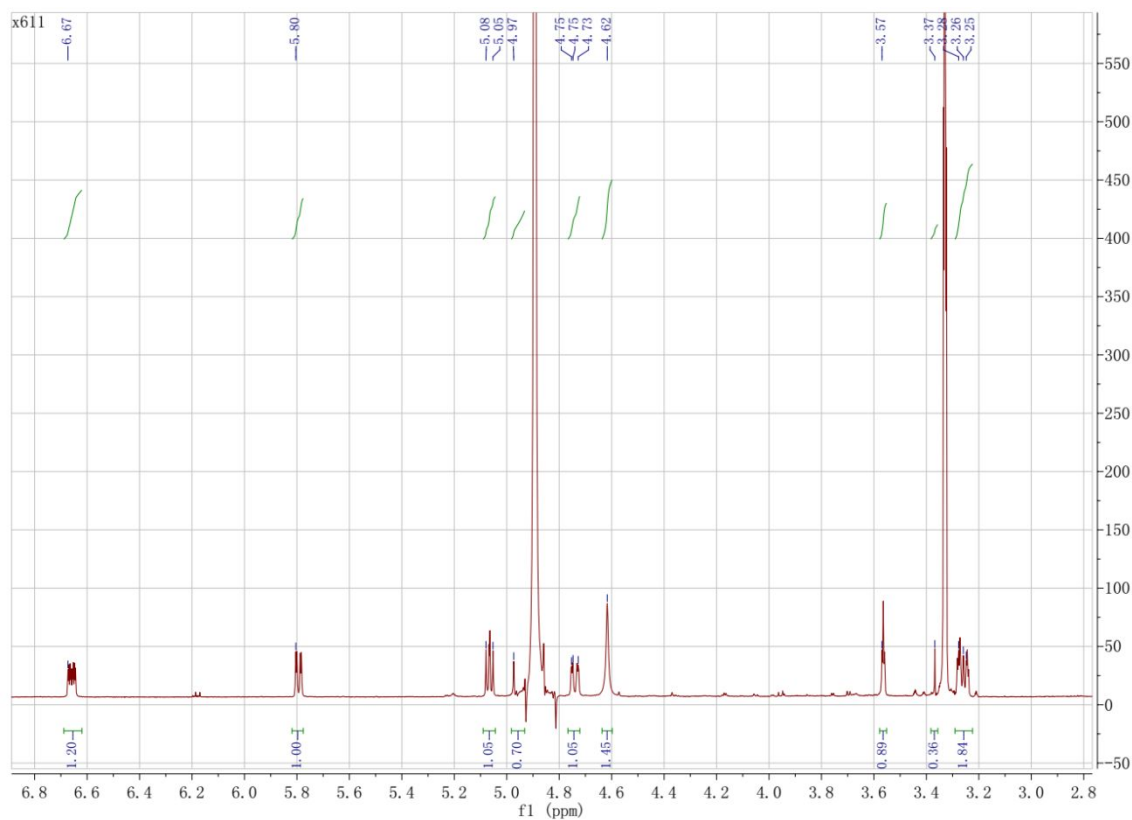
Spectrum from X6-1-1.wiff (sample 1) - X6-1-1, Experiment 1, -TOF MS (50 - 1250) from 9.297 min



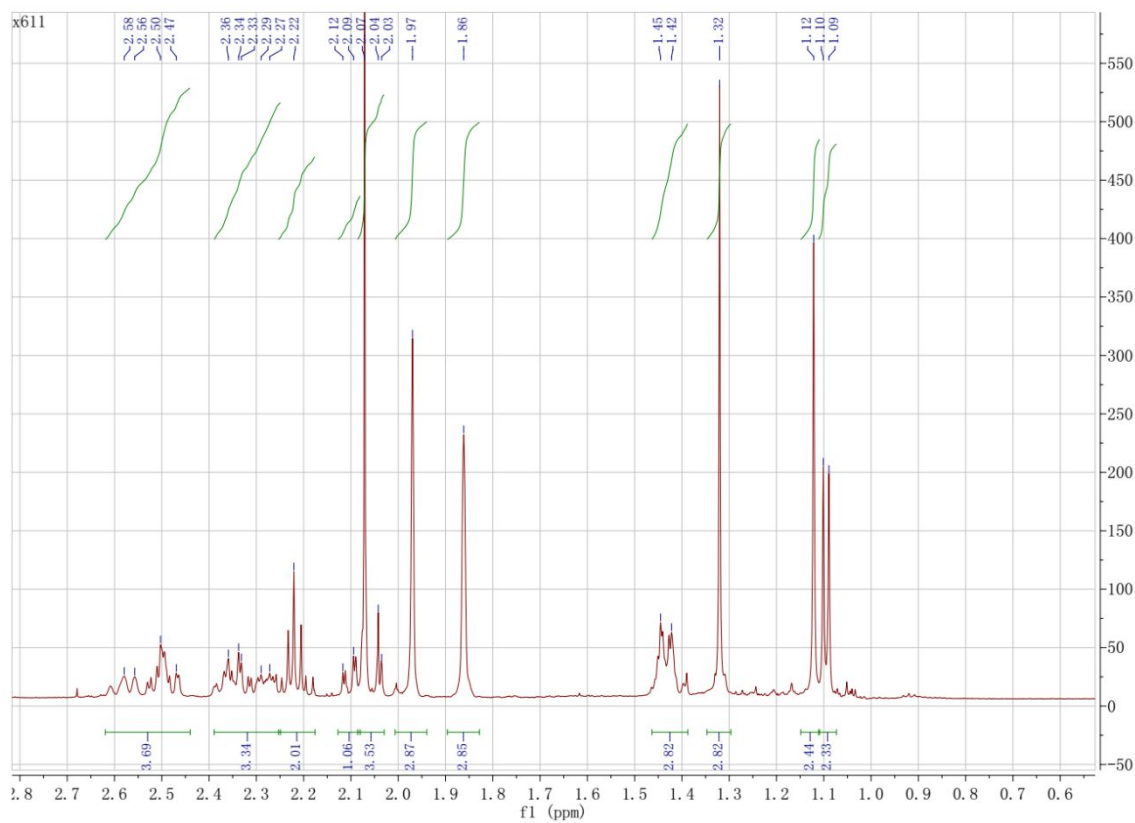
¹H NMR spectrum of compound 4 in CD₃OD at 600 MHz



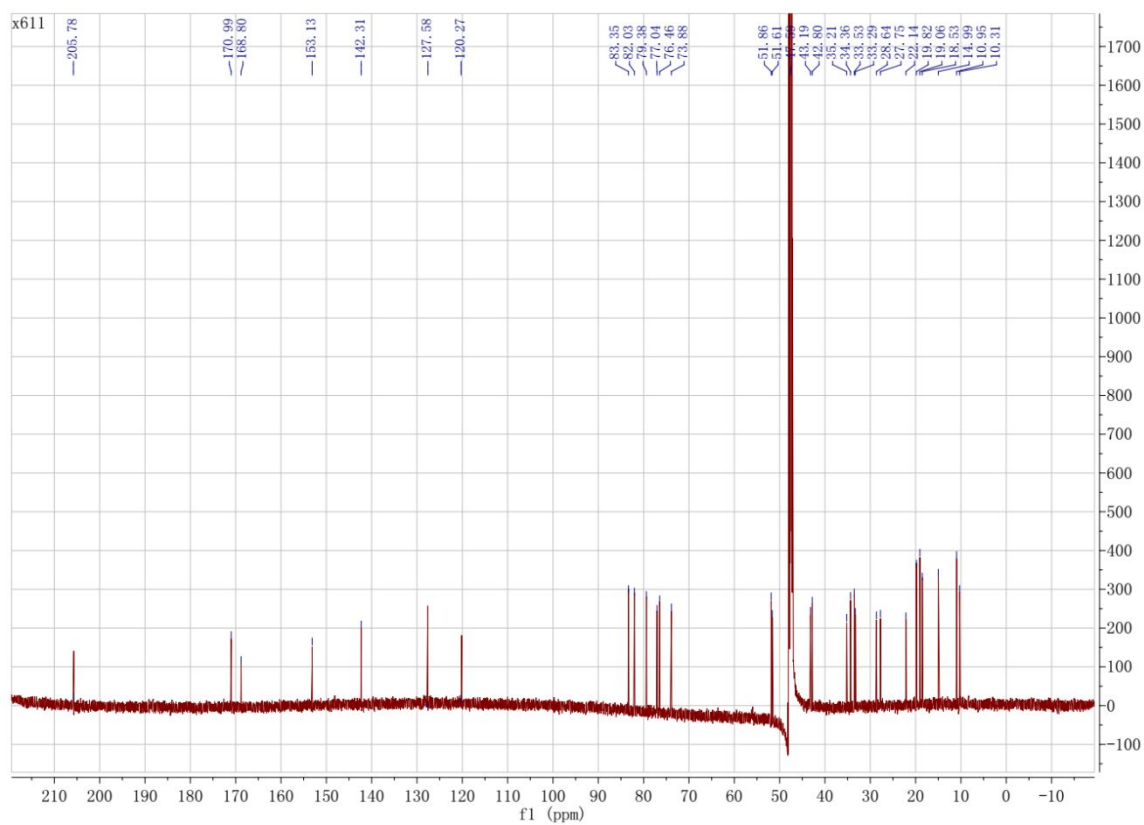
Detailed ^1H NMR spectrum of compound **4** in CD_3OD at 600 MHz



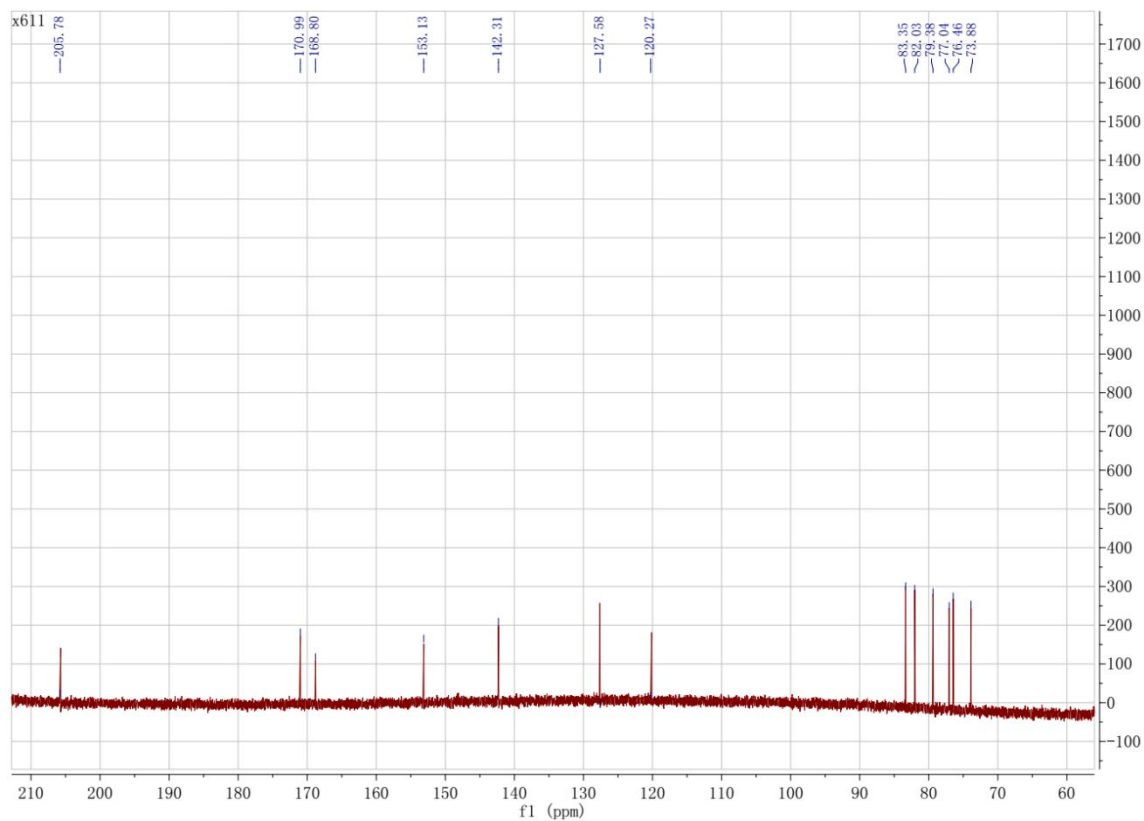
Detailed ^1H NMR spectrum of compound **4** in CD_3OD at 600 MHz



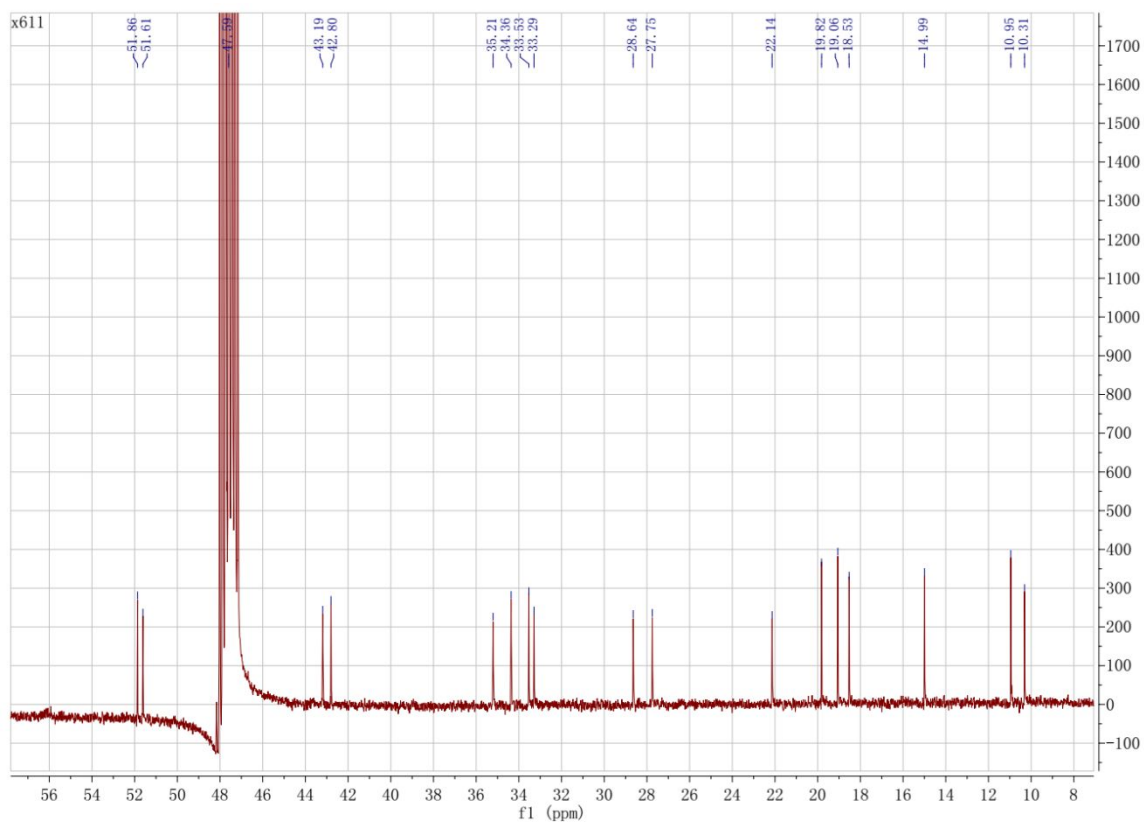
^{13}C NMR spectrum of compound **4** in CD_3OD at 150 MHz



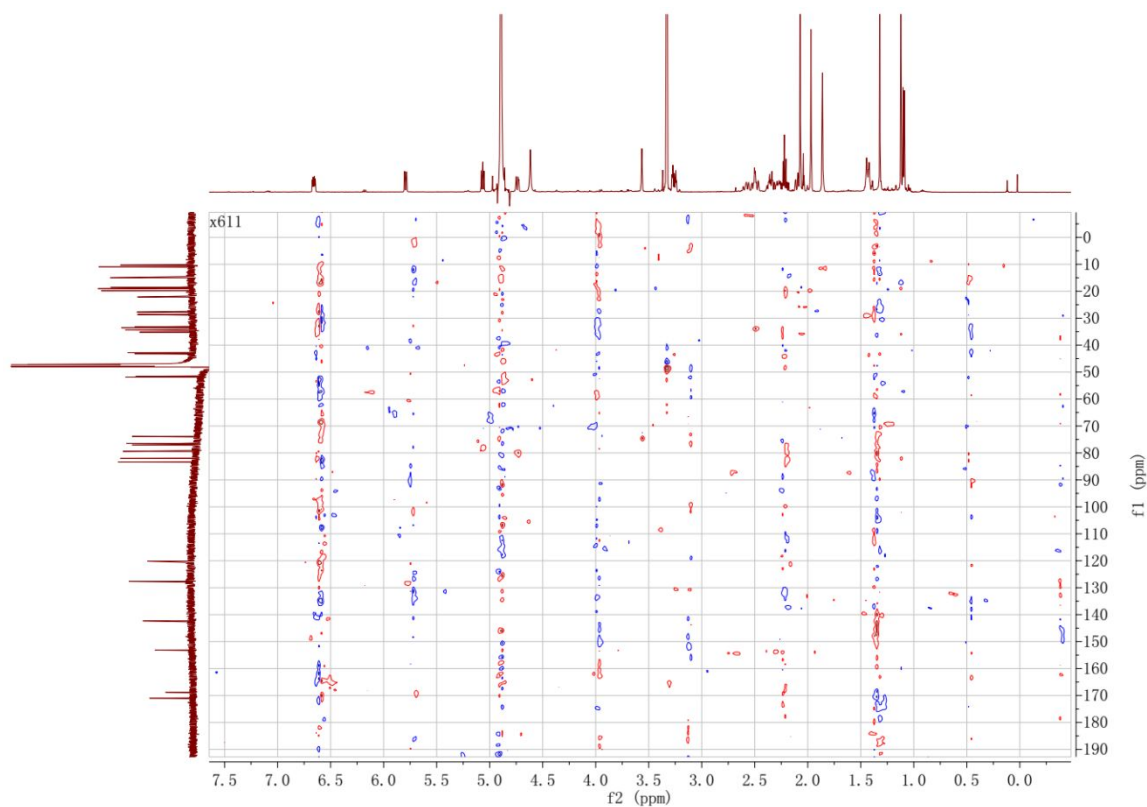
Detailed ^{13}C NMR spectrum of compound **4** in CD_3OD at 150 MHz



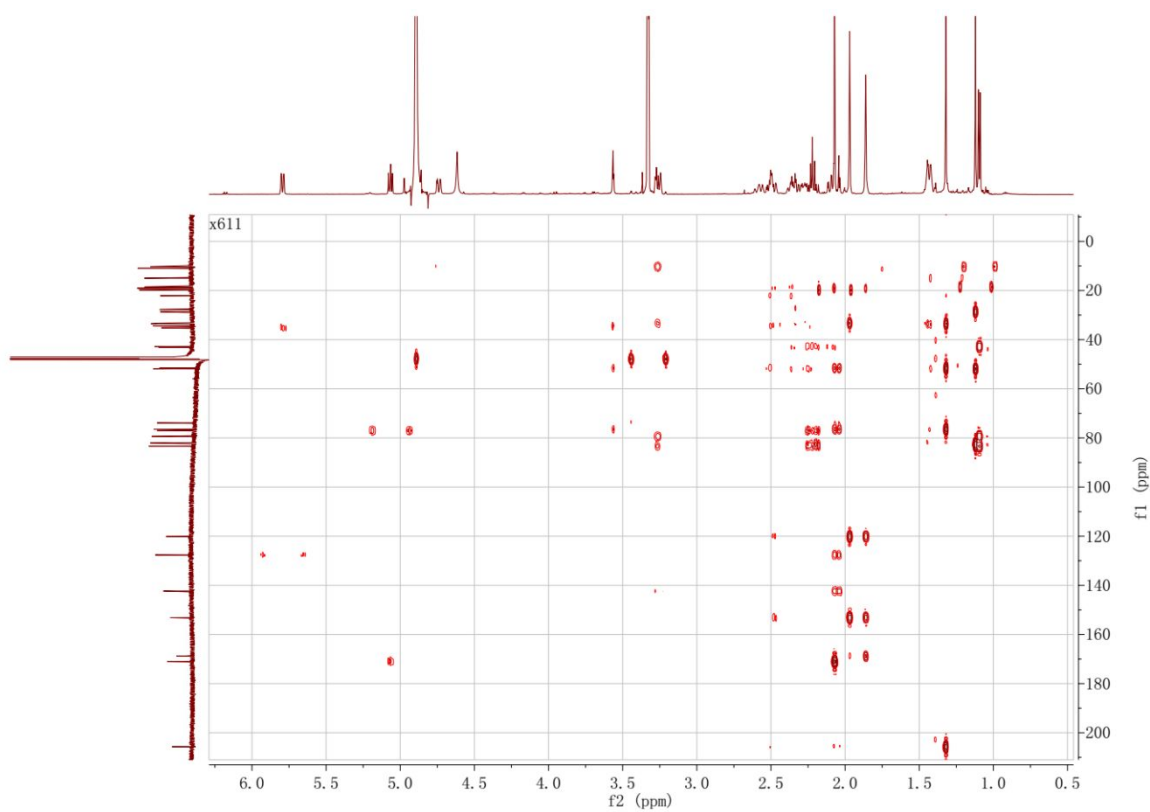
Detailed ^{13}C NMR spectrum of compound **4** in CD_3OD at 150 MHz



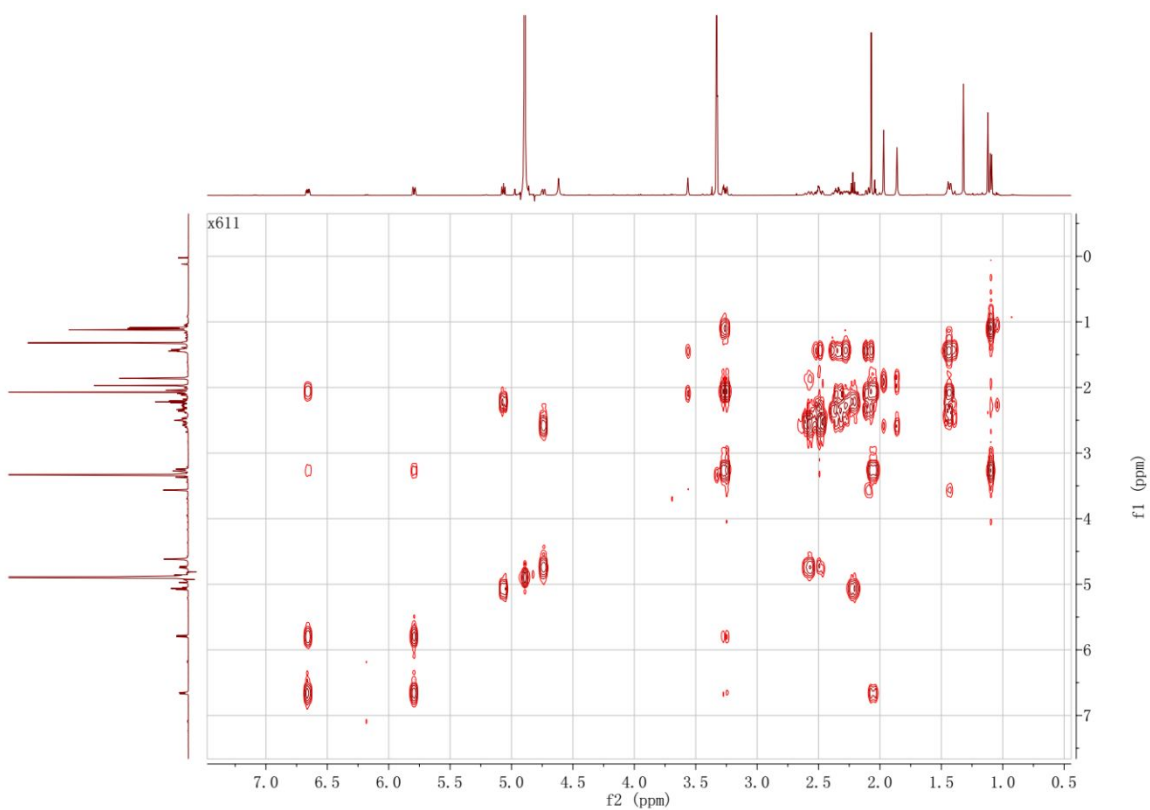
HSQC spectrum of compound **4** in CD_3OD



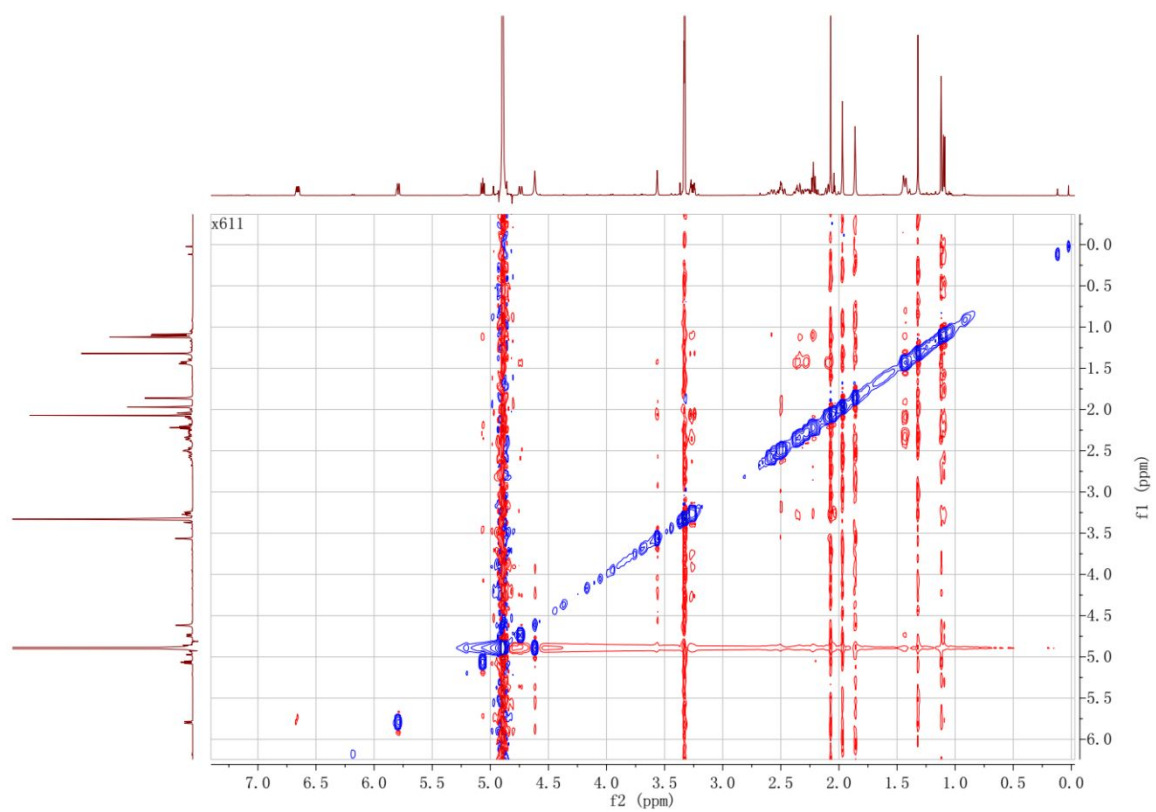
HMBC spectrum of compound **4** in CD₃OD



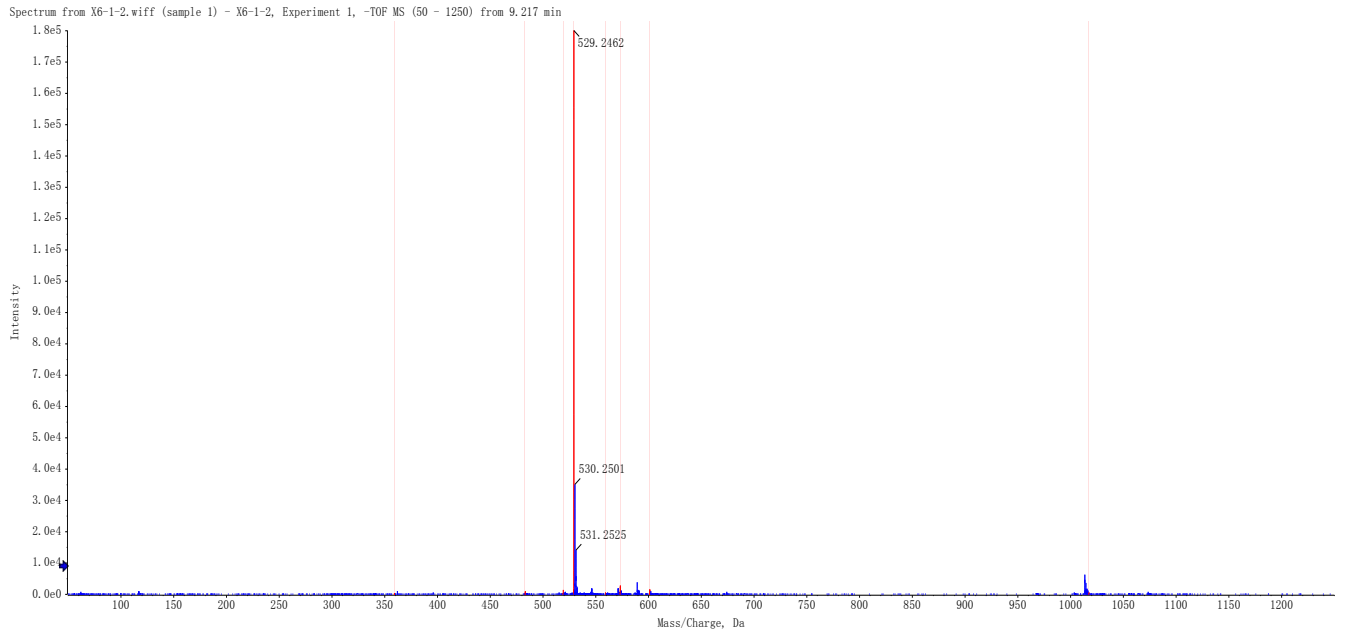
COSY spectrum of compound **4** in CD₃OD



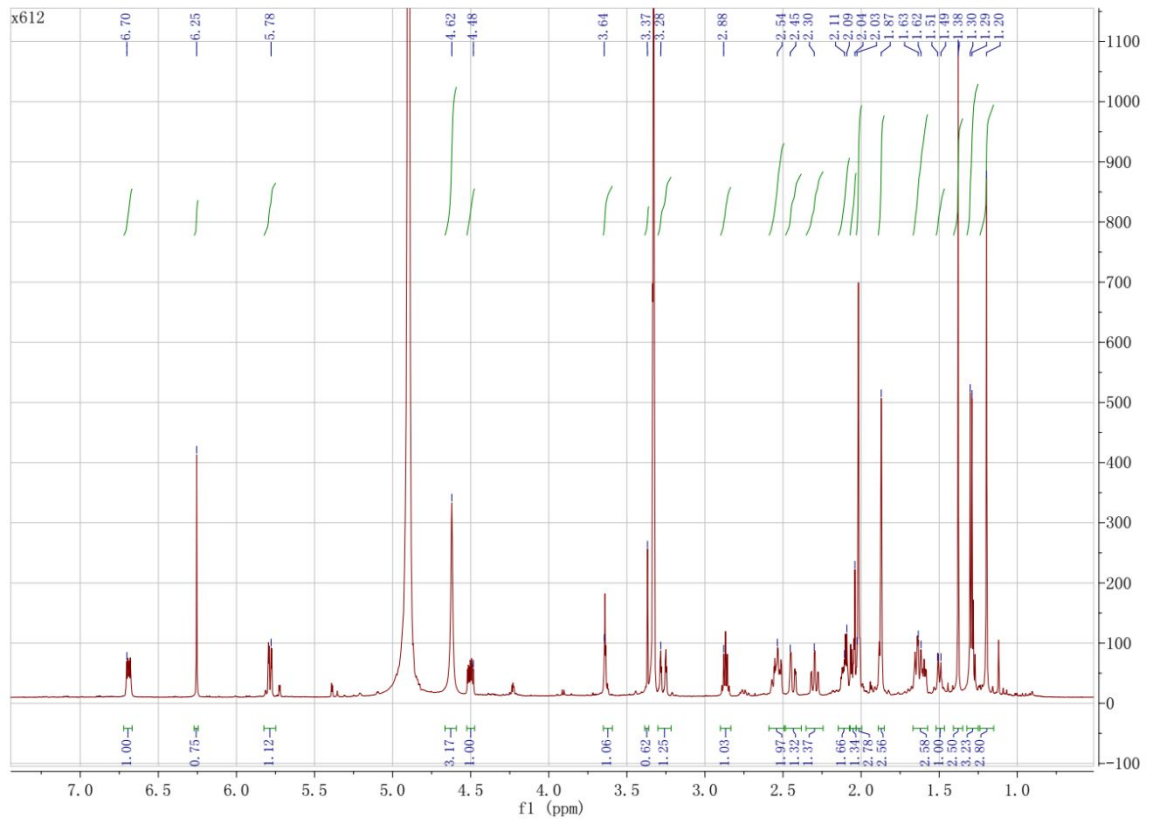
NOESY spectrum of compound **4** in CD₃OD



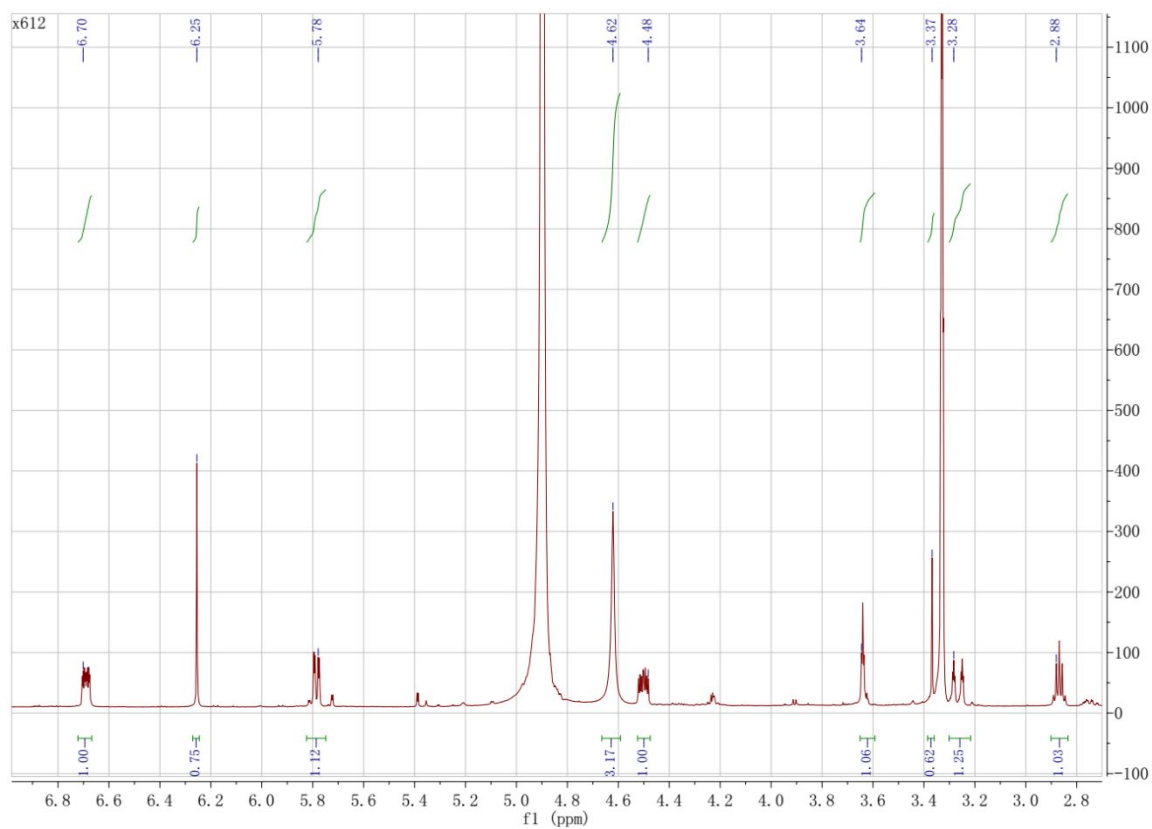
HR-ESI-MS spectrum of compound 5



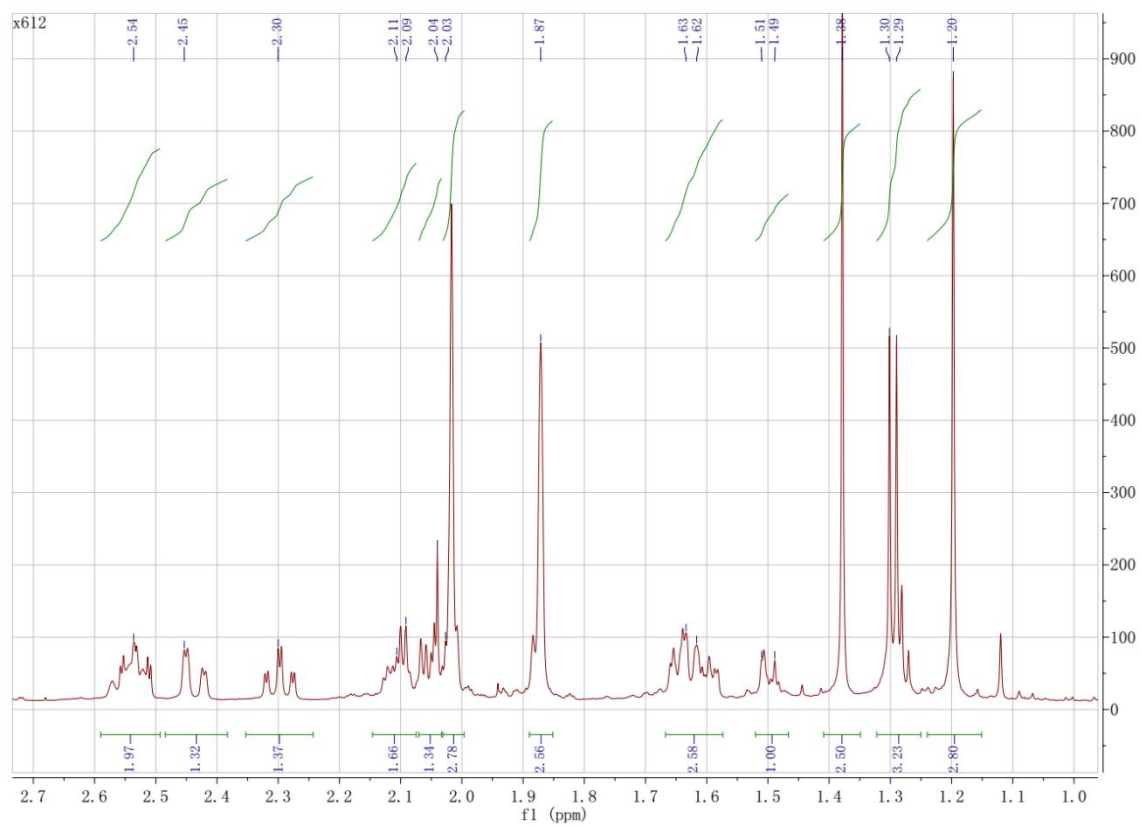
¹H NMR spectrum of compound 5 in CD₃OD at 600 MHz



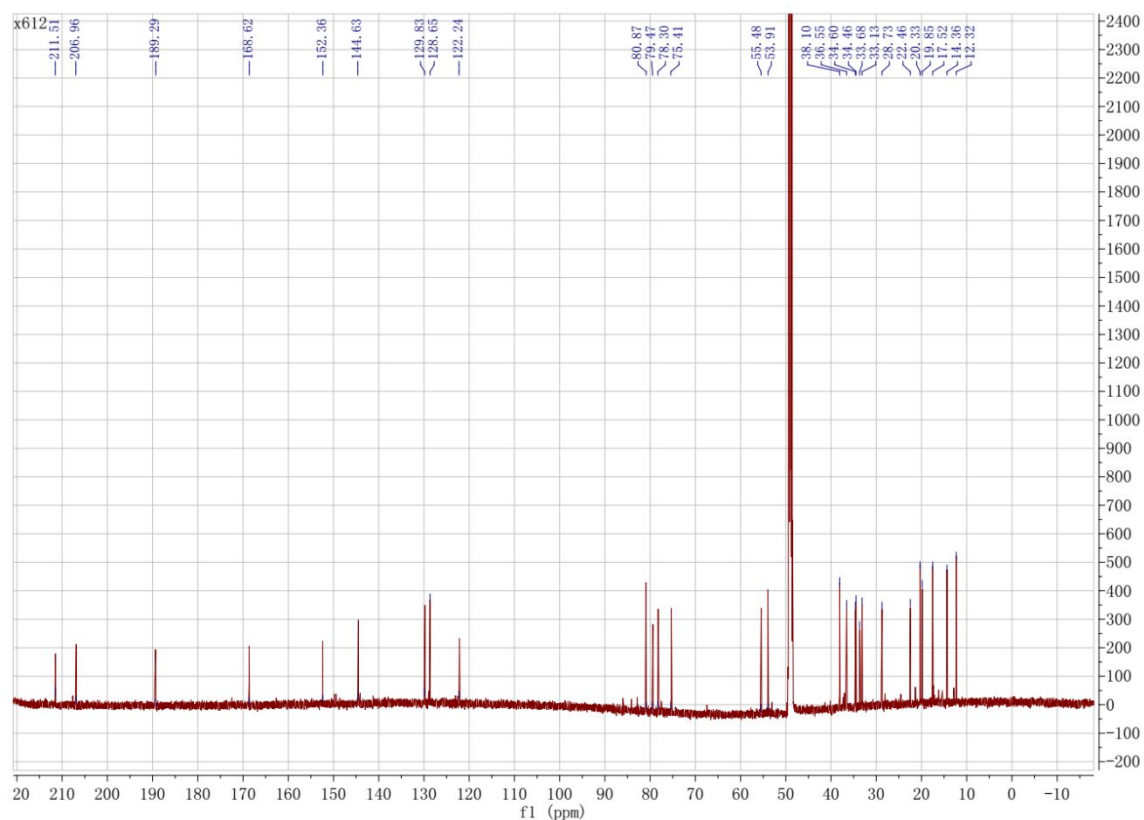
Detailed ^1H NMR spectrum of compound **5** in CD_3OD at 600 MHz



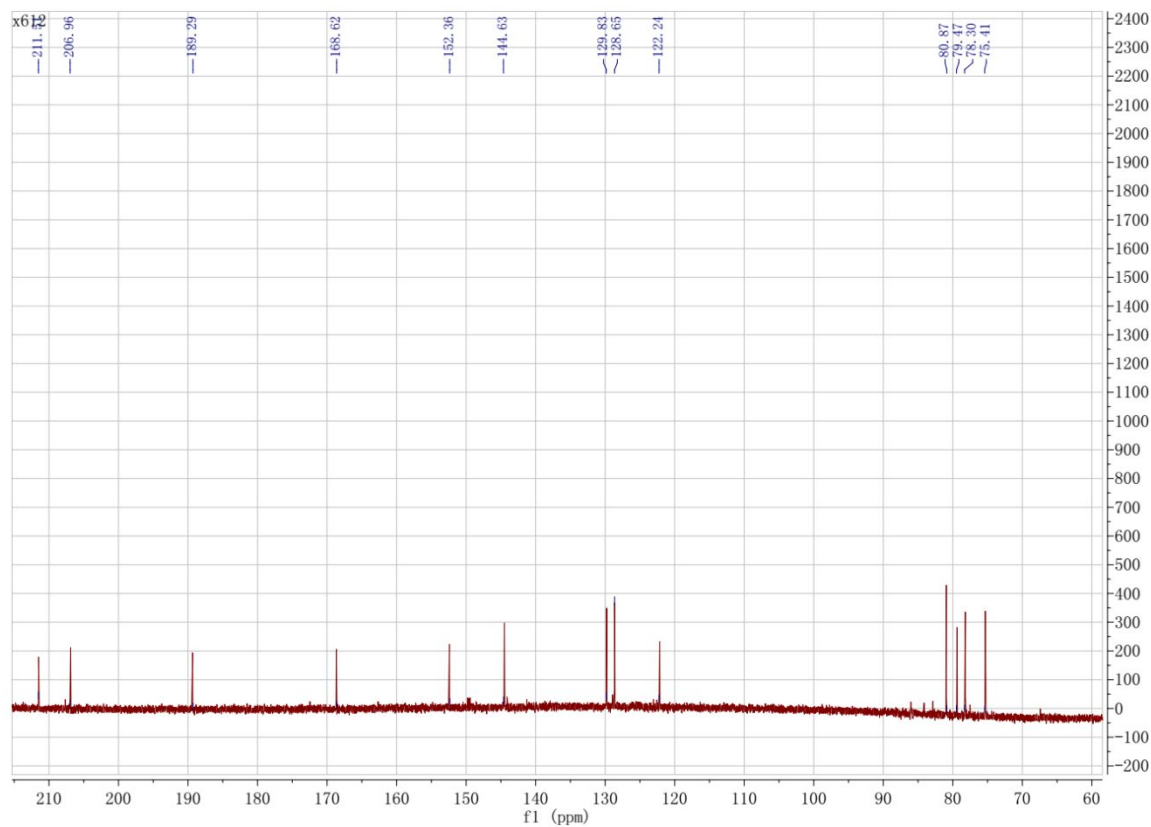
Detailed ^1H NMR spectrum of compound **5** in CD_3OD at 600 MHz



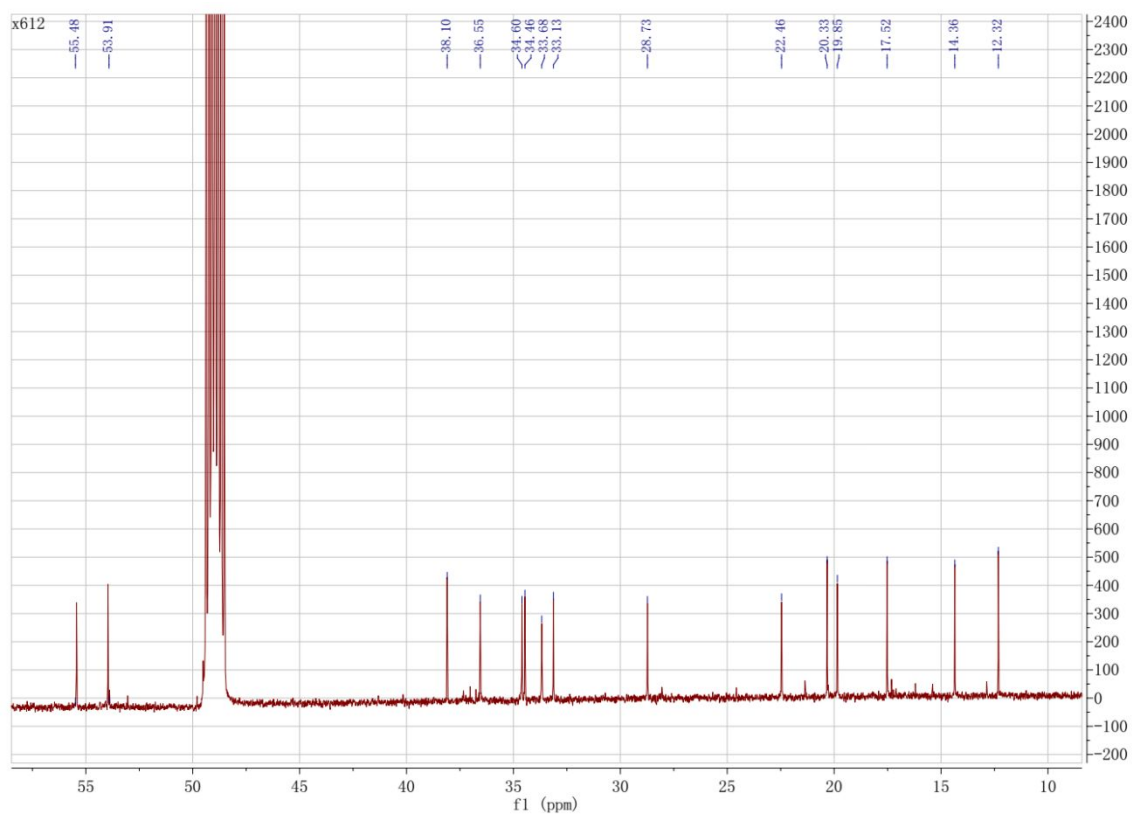
^{13}C NMR spectrum of compound **5** in CD_3OD at 150 MHz



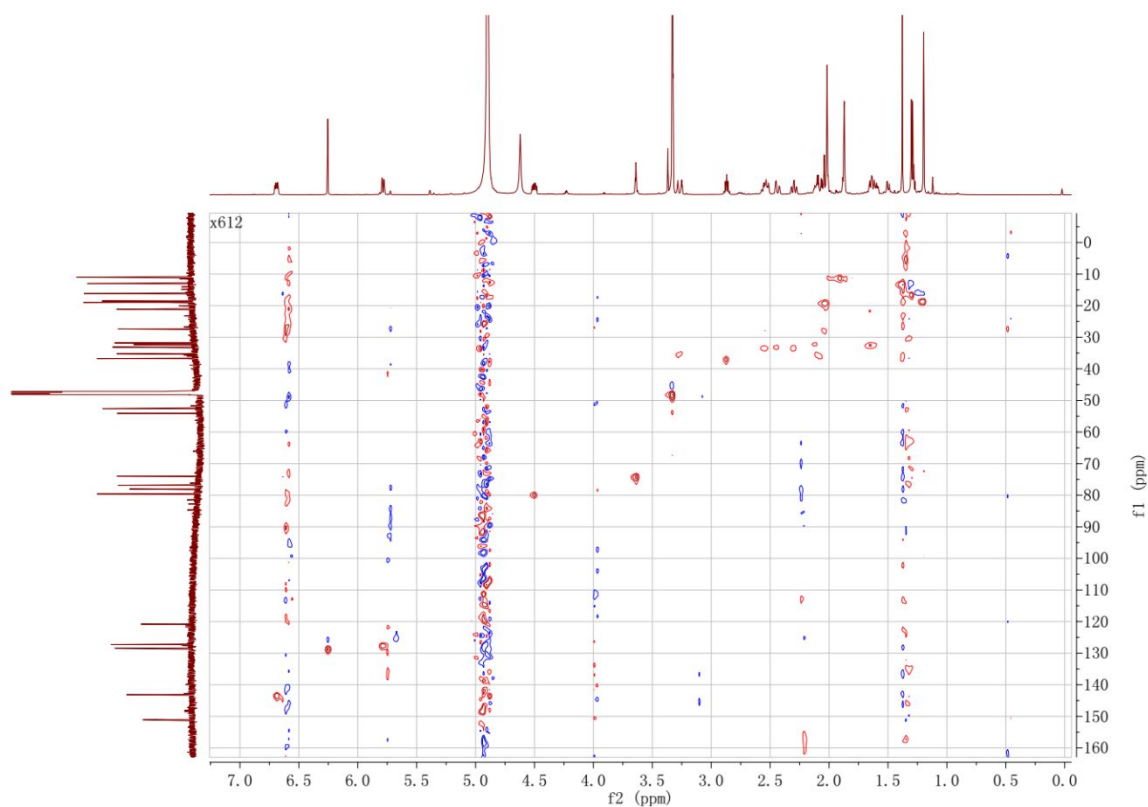
Detailed ^{13}C NMR spectrum of compound **5** in CD_3OD at 150 MHz



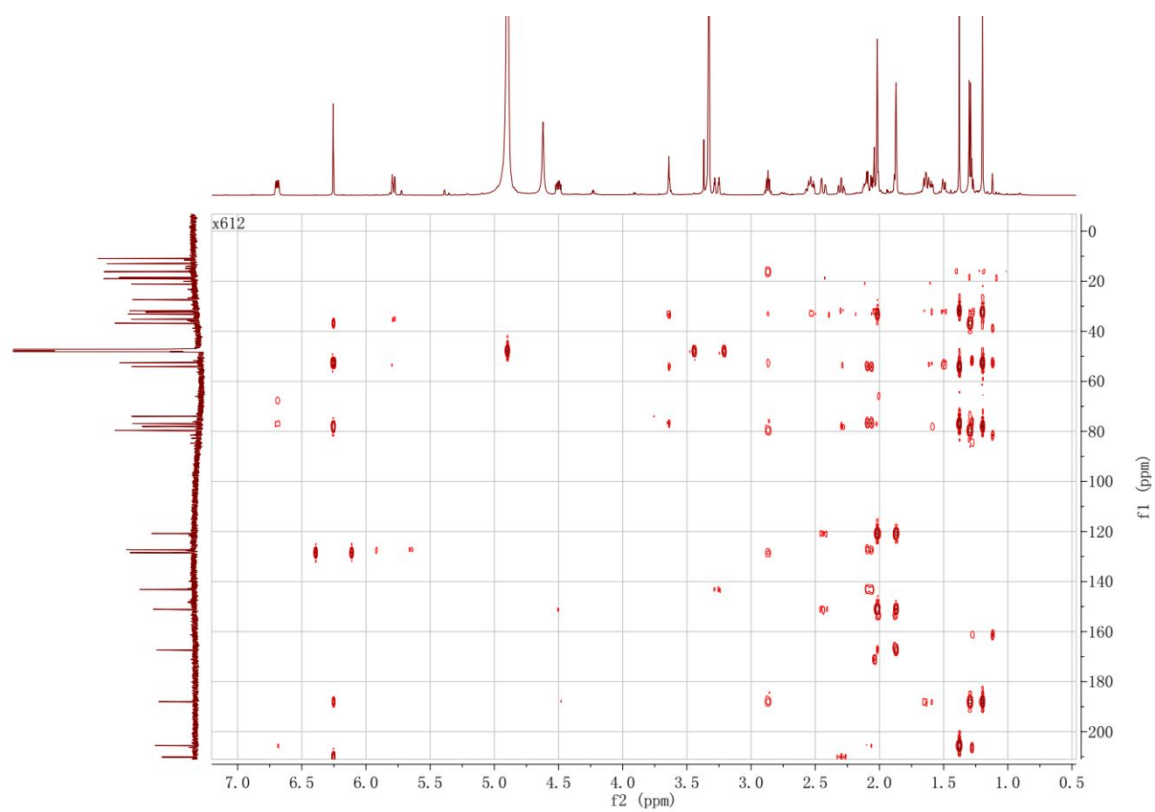
Detailed ^{13}C NMR spectrum of compound **5** in CD_3OD at 150 MHz



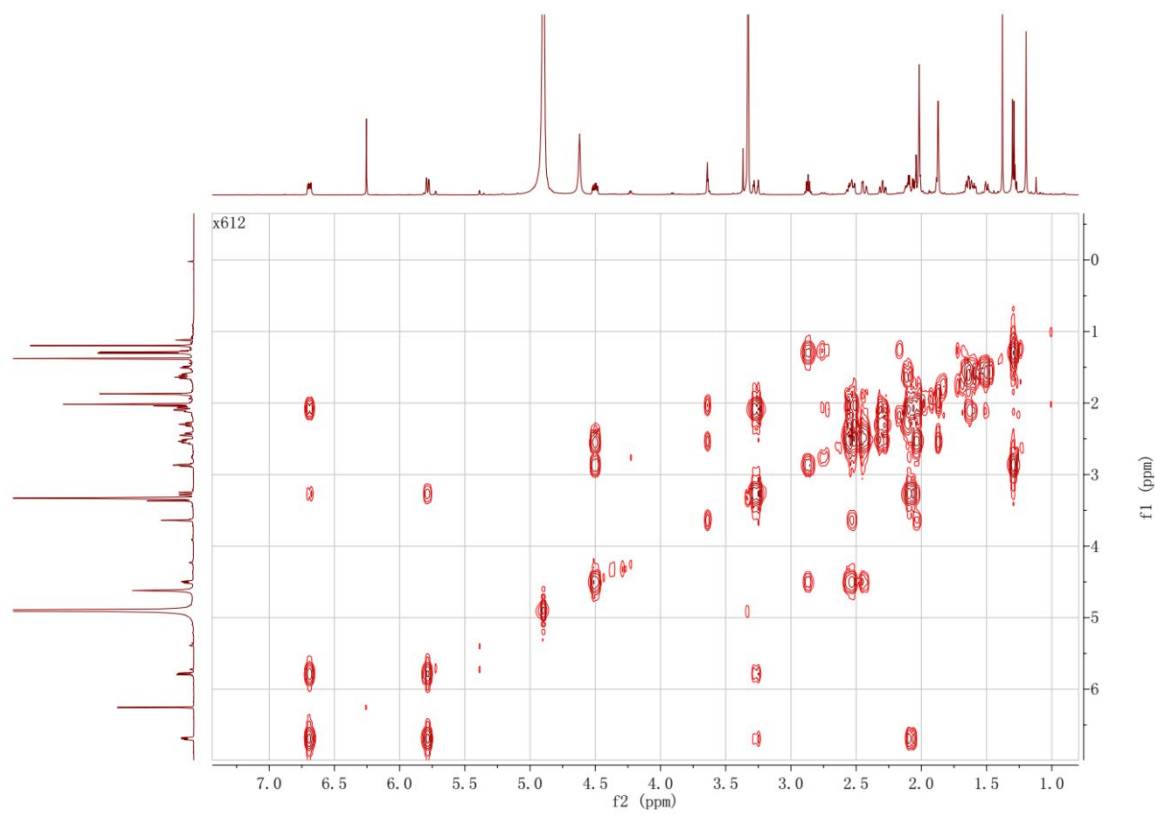
HSQC spectrum of compound **5** in CD_3OD



HMBC spectrum of compound **5** in CD₃OD



COSY spectrum of compound **5** in CD₃OD



NOESY spectrum of compound **5** in CD₃OD

