Supplementary Data

***Rec. Nat. Prod*. 14:6 (2020) 405-409**

A New Lignan Glycoside from the Roots of *Silene tatarinowii* Regel

Xiaofei Liang[#], Yuze Li[1#], Yuwen Cui[2], Zhuofei Liang[1], Wenli Huang[1], Yi Jiang[1], Huawei Zhang[1] and Xiaomei Song[1*]

[1] School of Pharmacy, Shaanxi University of Chinese Medicine, Xianyang 712046, P.R. China
[2] Department of Pharmacy, Xi'an Medical University, Xi'an 710021, P.R. China

Table of Contents **page**

[*] Corresponding authors: E-Mail: songxiaom@126.com; Phone:+86-13636733632
[#]These authors contributed equally to this work

Experimental

General procedures

Optical rotation indices were determined in methanol on a Rudolph Autopol II digital polarimeter (Rudolph, Hackettstown, NJ, USA). UV spectra were recorded on a Shimadzu-2201 (Kyoto, Japan). The IR spectra were recorded on a Bruker TENSOR-27 instrument. The HR-ESI-MS spectra was taken on an Agilent Technologies 6550 Q-TOF. 1D and 2D NMR spectra were recorded on a Bruker-AVANCE400 instrument with TMS as an internal standard. The analytical HPLC was performed on a Waters 2695 Separations Module coupled with a 2996 Photodiode Array Detector and a Accurasil C_{18} column (4.6 mm × 250 mm, 5 mm particles, Ameritech, America). Semipreparative HPLC was performed on a system comprising a Shimadzu LC-6AD pump equipped with a SPD-20A UV detector and a Ultimate XB-C_{18} (10 mm × 250 mm, 5 mm particles) or YMC-Pack-ODS-A (10 mm × 250 mm, 5 mm particles). Silica gel was purchased from Qingdao Haiyang Chemical Group Corporation (Qingdao, China).

Cytotoxicity Assay

The cytotoxic activity assay toward the HCT116, HT29, A549 and H1299 tumor cell lines were measured by the MTT method *in vitro*, using 5-fluorouracil as positive control. Briefly, 1 × 10^4 mL^{-1} cells were seeded into 96-well plates and allowed to adhere for 24 h. Compounds **1** - **4** were dissolved in DMSO and diluted with complete medium to six degrees of concentration (from 0.001 mmol·L^{-1} to 0.4 mmol·L^{-1}) for inhibition rate determination. After incubation at 37 °C for 4 h, the supernatant fraction was removed before adding DMSO (100 μL) to each well. The inhibition rate (IR) and IC_{50} were calculated (see Table 1). Values are mean ± SD, n = 3.

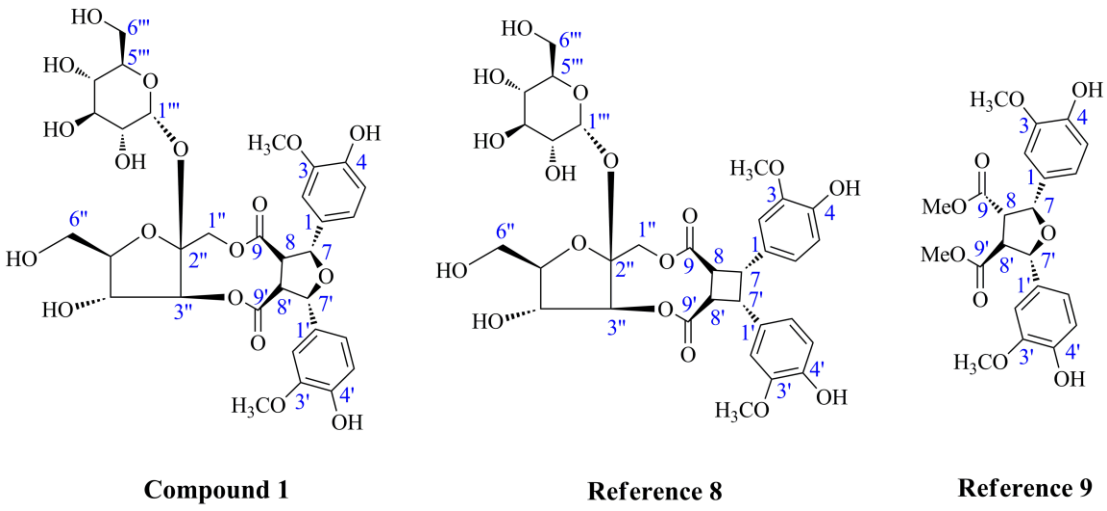


Figure 1. Comparison of the structure of compound **1** and references **8**, **9**

(Figure 1 in main text)

Table S1. Comparison of the spectroscopic data of Compound **1** and references **8**, **9***

	Compound 1		**Reference 8**		**Reference 9**	
No.	δ_H	δ_C	δ_H	δ_C	δ_H	δ_C
1	-	131.6	-	130.7	-	130.1
2	7.46, s	112.4	6.77, s	112.9	7.02, s	111.3
3	-	149.4	-	148.4	-	147.9
4	-	149.1	-	146.7	-	147.3
5	7.20, d, (7.6)	117.0	6.96, d, (8.0)	116.1	6.80, d, (8.0)	115.2
6	7.38, d, (8.2)	121.2	6.92, d, (8.2)	121.8	6.88, d, (8.0)	120.5
7	5.78, d, (5.2)	84.0	4.48, d, (9.0)	44.8	4.95, d, (8.6)	83.4
8	4.48, m	56.8	4.79,dd,(11.0,4.5)	44.5	3.60,dd,(6.3,8.5)	56.2
9	-	174.6	-	174.7	-	172.5
10	3.78, s	56.4	3.72, s	55.9	3.84, s	52.4
1′	-	131.3	-	130.6		132.2
2′	7.48, s	111.9	6.91, s	112.9	7.59, s	111.3
3′	-	149.1	-	148.2	-	148.3
4′	-	148.8	-	146.6	-	147.5
5′	7.22, d, (7.6)	116.9	6.96, d, (8.0)	115.9	6.85, d, (8.0)	115.5
6′	7.31, d, (8.2)	121.1	6.75, d, (8.0)	121.2	7.04, d, (8.0)	120.7
7′	5.72, d, (3.7)	82.3	4.84, d, (9.0)	43.9	5.27, d, (8.3)	84.1
8′	4.48, m	56.7	4.46,dd,(11.0,4.5)	46.0	3.78,dd,(6.3,8.3)	55.8
9′	-	170.7	-	172.3	-	172.9
10′	3.78, s	56.3	3.61, s	55.8	3.89, s	56.3
1″α	5.12, d, (12.8)	64.4	5.22, d, (12.0)	64.8		
1″β	5.12, d, (12.7)		4.26, d, (12.0)			
2″	-	110.2	-	109.8		
3″	5.53, s	80.5	5.61, s	79.6		
4″	4.63, s	89.1	4.63, s	88.7		
5″	5.22, d, (3.4)	74.8	5.13, m	75.0		
6″α	4.32, dd, (9.6, 5.3)	65.6	4.56,dd,(11.2, 3.4)	63.9		
6″β	4.27, dd, (9.8, 5.0)		4.50,dd,(11.2, 5.0)			
1‴	6.18, d, (3.0)	94.9	6.21, d, (3.6)	94.4		
2‴	4.92, m	75.3	5.03, m	74.7		
3‴	4.63, m	76.0	4.75, d, (9.0)	75.7		
4‴	4.28, m	72.7	4.26, m	73.8		
5‴	4.19, m	74.2	4.32, m	72.3		
6‴α	4.56, m	63.7	4.65,dd,(14.6, 2.8)	63.4		
6‴β	4.45, m		4.57,dd,(14.6, 3.4)			

*Table 1 in main text

S1:Spectroscopic Data of 1-4

*(7S,8R,7`R,8`S)-1-[1,3-(7,7`-bis-(4-hydroxy-3-methoxyphenyl)-tetrahydrofuran-8,8`-dicarboxyl)-β-D-fructofuranosyl]-α-D-glucopyranoside, named siletatoside A (**1**):* yellow amorphous powder (MeOH); $[\alpha]_D^{25}$ +37.1 (*c* 0.01, MeOH); IR ν_{max} (in MeOH) cm^{-1} :3377, 2940, 1735, 1600, 1450, 1377; UV λ_{max} (MeOH): 232nm; HR-ESI-MS at *m/z* 733.1932 $[M+Na]^+$; ^{1}H-NMR (pyridine-d_5, 400 MHz): δ_H 7.46 (1H, s, H-2), 7.38 (1H, d, *J* = 8.2 Hz, H-6), 7.31 (1H, d, *J* = 8.2 Hz, H-6'), 7.22 (1H, d, *J* = 7.6 Hz, H-5'), 7.20 (1H, d, *J* = 7.6 Hz, H-5), 6.18 (1H, d, *J* = 3.0 Hz, H-1'"), 5.78 (1H, d, *J* = 5.2 Hz, H-7), 5.72 (1H, d, *J* = 3.7 Hz, H-7'), 5.53 (1H, m, H-3"), 5.22 (1H, d, *J* = 3.4 Hz, H-5"), 5.12 (1H, d, *J* = 12.8 Hz, H-1"*α*), 5.12 (1H, d, *J* = 12.8 Hz, H-1"*β*), 4.92 (1H, m, H-2'"), 4.63 (1H, m, H-4'"), 4.63 (1H, m, H-3'"), 4.56 (1H, m, H-6'"*α*), 4.48 (1H, m, H-8), 4.48 (1H, m, H-8'), 4.45 (1H, m, H-6'"*β*), 4.28 (1H, m, H-4'"), 4.19 (1H, m, H-5'"), 3.78 (3H, s, H-10), 3.78 (3H, s, H-10'); ^{13}C-NMR (pyridine-d_5, 100 MHz): δ_C 174.6 (C-9), 170.7 (C-9'), 149.3 (C-3), 149.1 (C-3'), 149.1 (C-4), 148.8 (C-4'), 131.6 (C-1), 131.3 (C-1'), 121.2 (C-6), 121.1 (C-6'), 117.0 (C-5), 116.9 (C-5'), 112.4 (C-2),111.9 (C-2'), 110.2 (C-2"), 94.9 (C-1'"), 89.1 (C-4"), 84.0 (C-7), 82.3 (C-7'), 80.5 (C-3"), 76.0 (C-3'"), 75.3 (C-2'"), 74.8 (C-5"), 74.2 (C-5'"), 72.7 (C-4'"), 65.6 (C-6"), 64.4 (C-1"), 63.7 (C-6'"), 56.8 (C-8), 56.7 (C-8'), 56.4 (C-10), 56.3 (C-10').

*(+)- Isolariciresinol (**2**):* colorless oil, ^{1}H-NMR (pyridine-d_5, 400 MHz): δ_H 7.08 (1H, s, H-5), 6.97 (1H, s, H-2), 4.36 (1H, d, *J* = 10.2 Hz, H-7'), 4.24 (2H, m, H-9'a, H-9'b), 3.62 (2H, m, H-9a, H-9b), 3.24 (2H, m, H-7a, H-7b), 3.82 (3H, s, H-3OMe), 3.57 (3H, s, H-3'OMe), 2.35 (1H, m, H-8'). ^{13}C-NMR (pyridine-d_5, 100 MHz): δ_C 128.7 (C-1), 113.2 (C-2), 147.1 (C-3), 146.7 (C-4), 118.4 (C-5), 134.8 (C-6), 34.2 (C-7), 40.9 (C-8), 66.2 (C-9), 138.5 (C-1'), 114. (C-2'), 147.6 (C-3'), 149.2 (C-4'), 116.9 (C-5'), 123.5 (C-6'), 48.6 (C-7'), 48.4 (C-8'), 62.4 (C-9'), 56.6 (C-3OMe), 56.3 (C-3'OMe).

*Balanophonin (**3**):* yellow powder, ^{1}H-NMR (pyridine-d_5, 400 MHz): δ_H 10.03 (1H, s, H-9'), 7.6 (1H, s, H-2'), 7.33 (1H, d, *J* = 1.5 Hz, H-2), 7.25 (1H, s, H-5), 7.23 (1H, m, H-6), 7.7 (1H, s, H-6'), 6.18 (1H, d, *J* = 7.0 Hz, H-7), 4.27 (2H, d, *J* = 5.6 Hz, H-9), 4.03 (1H, m, H-8), 3.81 (3H, s, H-3OMe), 3.69 (3H, s, H-3'OMe). ^{13}C-NMR (pyridine-d_5, 100 MHz): δ_C 132.4 (C-1), 111.5 (C-2), 149.1 (C-3), 145.9 (C-4), 117.1 (C-5), 120.4 (C-6), 90.55 (C-7), 54.2 (C-8), 64.2 (C-9), 131.6 (C-1'), 113.5 (C-2'), 145.2 (C-3'), 149.4 (C-4'), 133.0 (C-5'), 122.0 (C-6'), 155.2 (C-7'), 124.5 (C-8'), 191.4 (C-9'), 56.5 (C-3OMe), 56.4 (C-3'OMe).

*(+)- Lariciresinol (**4**):* colorless oil, ^{1}H-NMR (pyridine-d_5, 400 MHz): δ_H 7.35 (1H, d, *J* = 2.2 Hz, H-2), 7.27 (1H, d, *J* = 8.1 Hz, H-5'), 7.23 (1H, dd, *J* = 8.1 Hz, *J* = 2.2 Hz, H-6'), 7.21 (1H, d, *J* = 8.1 Hz, H-5), 7.02 (1H, d, *J* = 2.1 Hz, H-2'), 6.93 (1H, dd, *J* = 8.1 Hz, *J* = 2.1 Hz, H-6), 5.35 (1H, d, *J* = 5.8 Hz, H-7), 4.32 (1H, dd, *J* = 8.0 Hz, *J* = 6.8 Hz, H-9'a), 4.27 (1H, dd, *J* = 8.0 Hz, *J* = 6.8 Hz, H-9a), 4.15 (1H, dd, *J* = 8.0 Hz, *J* = 7.6 Hz, H-9b), 4.08 (1H, dd, *J* = 8.0 Hz, *J* = 7.6 Hz, H-9'b), 3.76 (6H, s, H-10, H-10'), 3.27 (1H, m, H-7'a), 3.18 (1H, m, H-8'), 2.86 (1H, m, H-7'b), 2.80 (1H, m, H-8). ^{13}C-NMR (pyridine-d_5, 100 MHz): δ_C 133.2 (C-1), 111.2 (C-2), 149.3 (C-3), 147.9 (C-4), 116.9 (C-5), 120.0 (C-6), 34.0 (C-7), 43.9 (C-8), 60.6 (C-9), 135.6 (C-1'), 113.8 (C-2'), 147.0 (C-3'), 149.3 (C-4'), 117.1 (C-5'), 122.4 (C-6'), 83.9 (C-7'), 54.4 (C-8'), 73.7 (C-9'), 56.5 (C-3OMe), 56.5 (C-3'OMe).

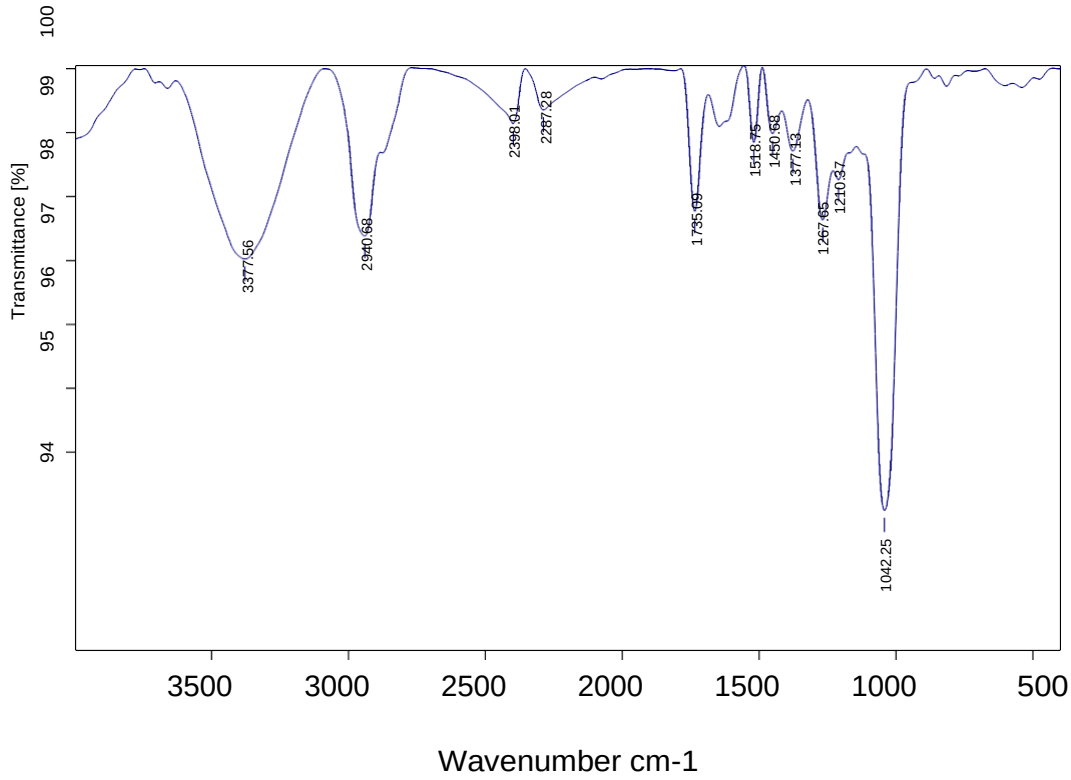


Figure S1: The IR spectrum of **1** (in KBr)

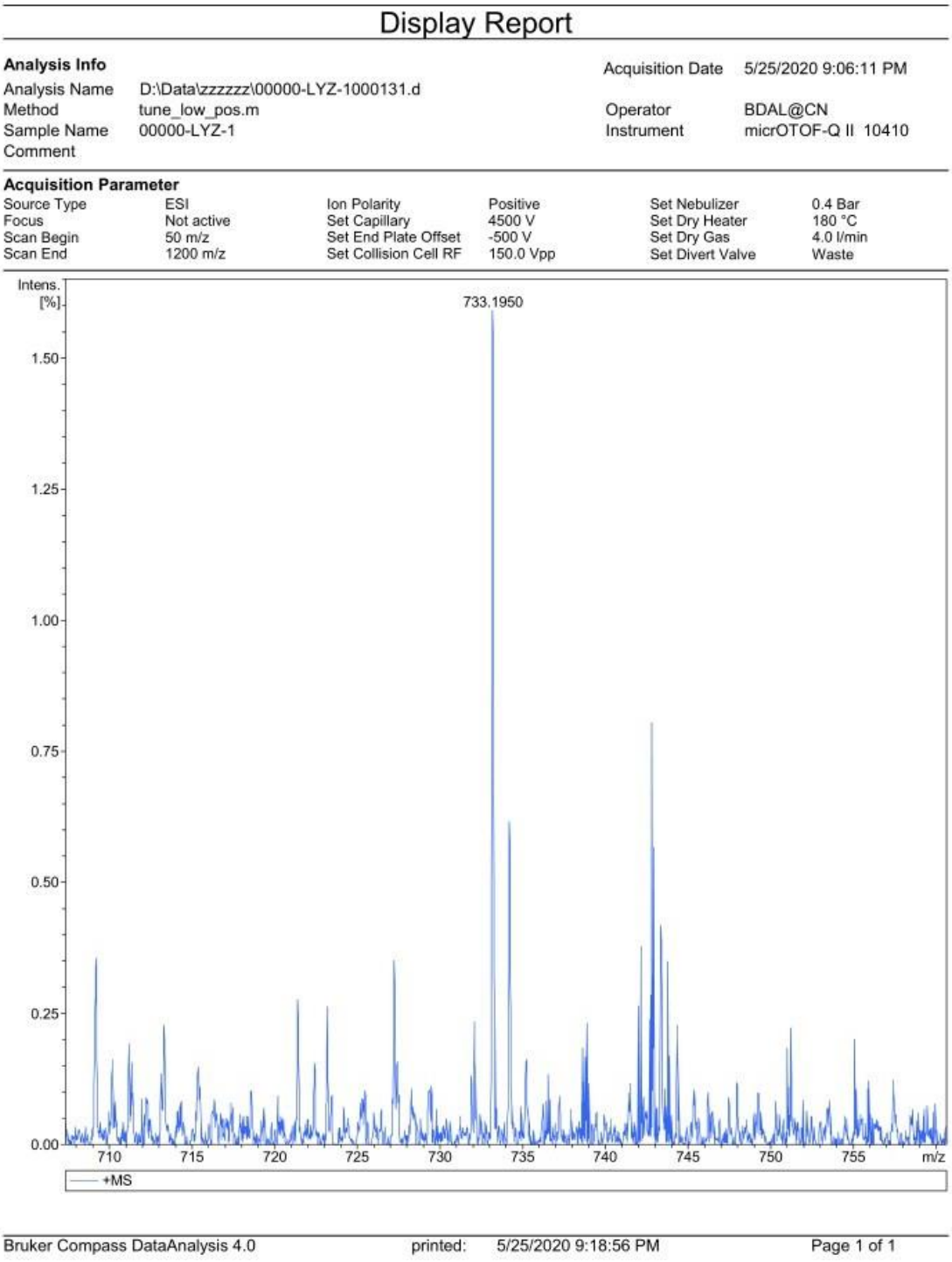


Figure S2: The HR-ESI-MS spectrum of **1** (in MeOH）

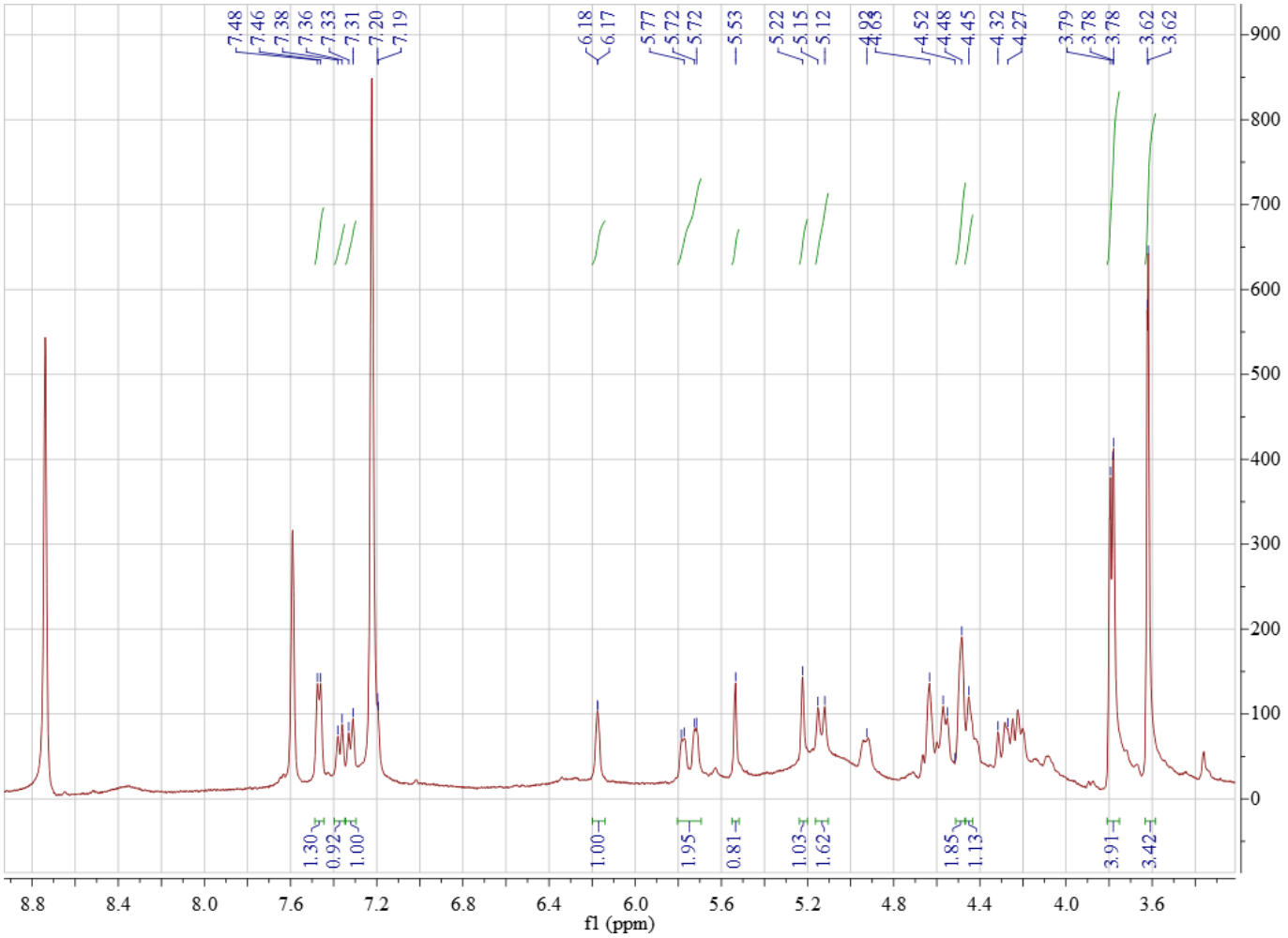


Figure S3: The ^{1}H NMR spectrum of **1** (in pyridine-d_5)

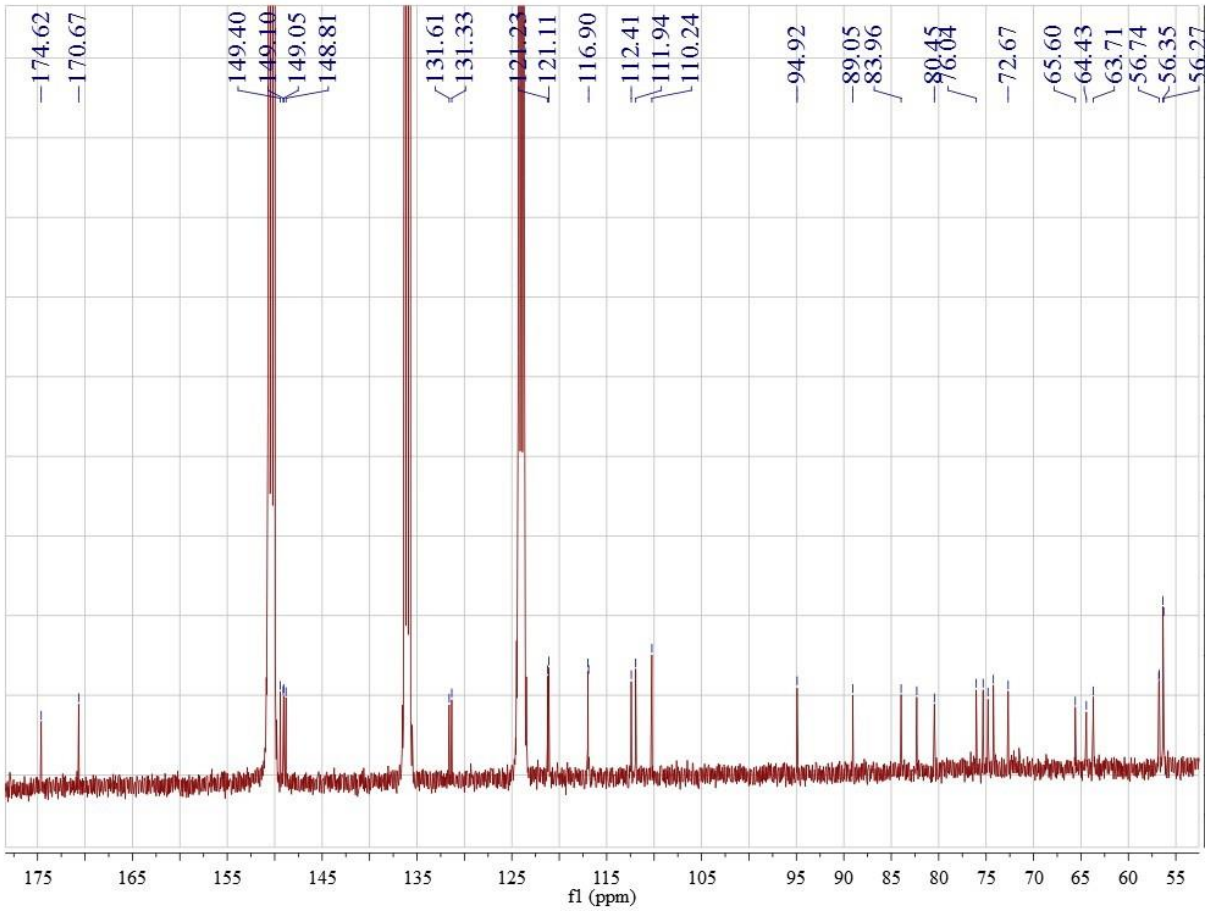


Figure S4: The ^{13}C NMR spectrum of **1** (in pyridine-d_5)

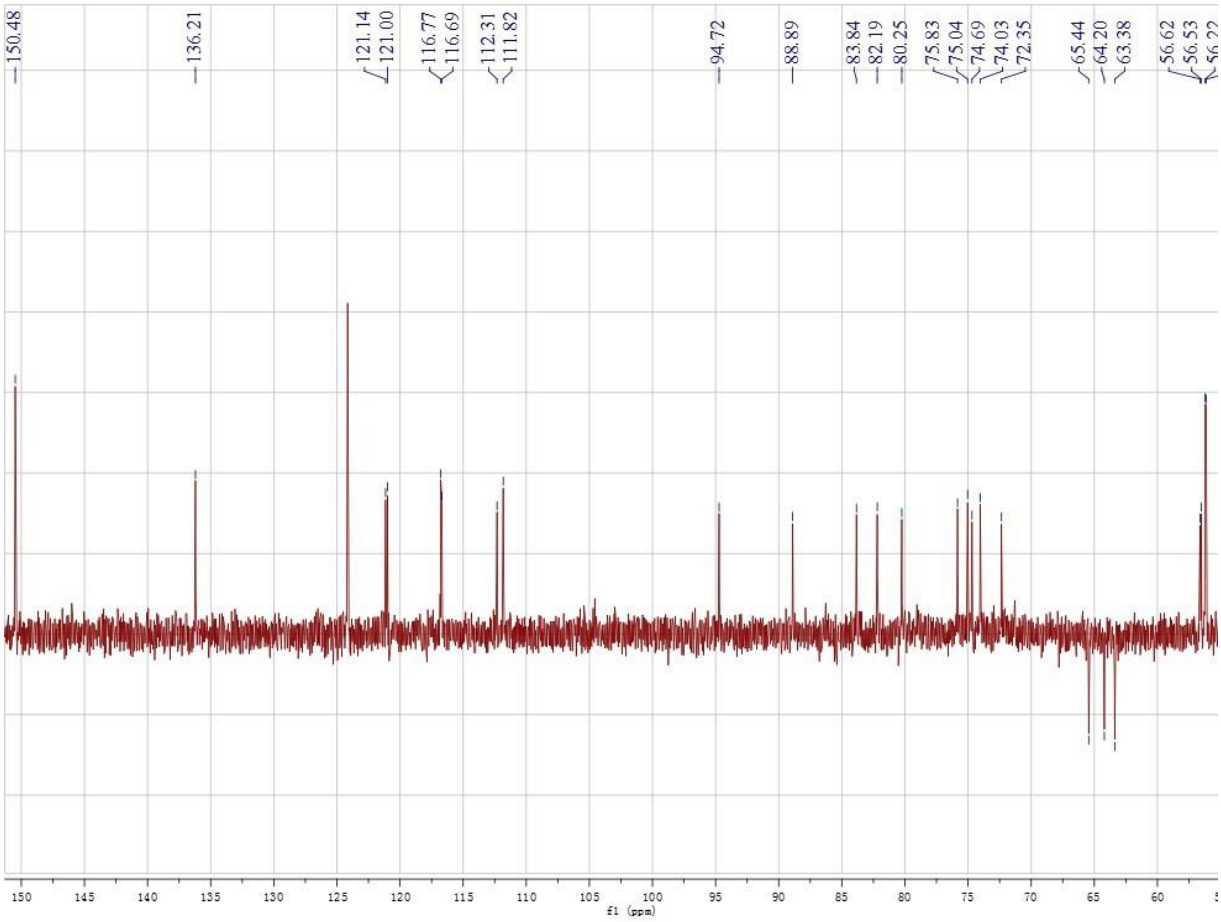
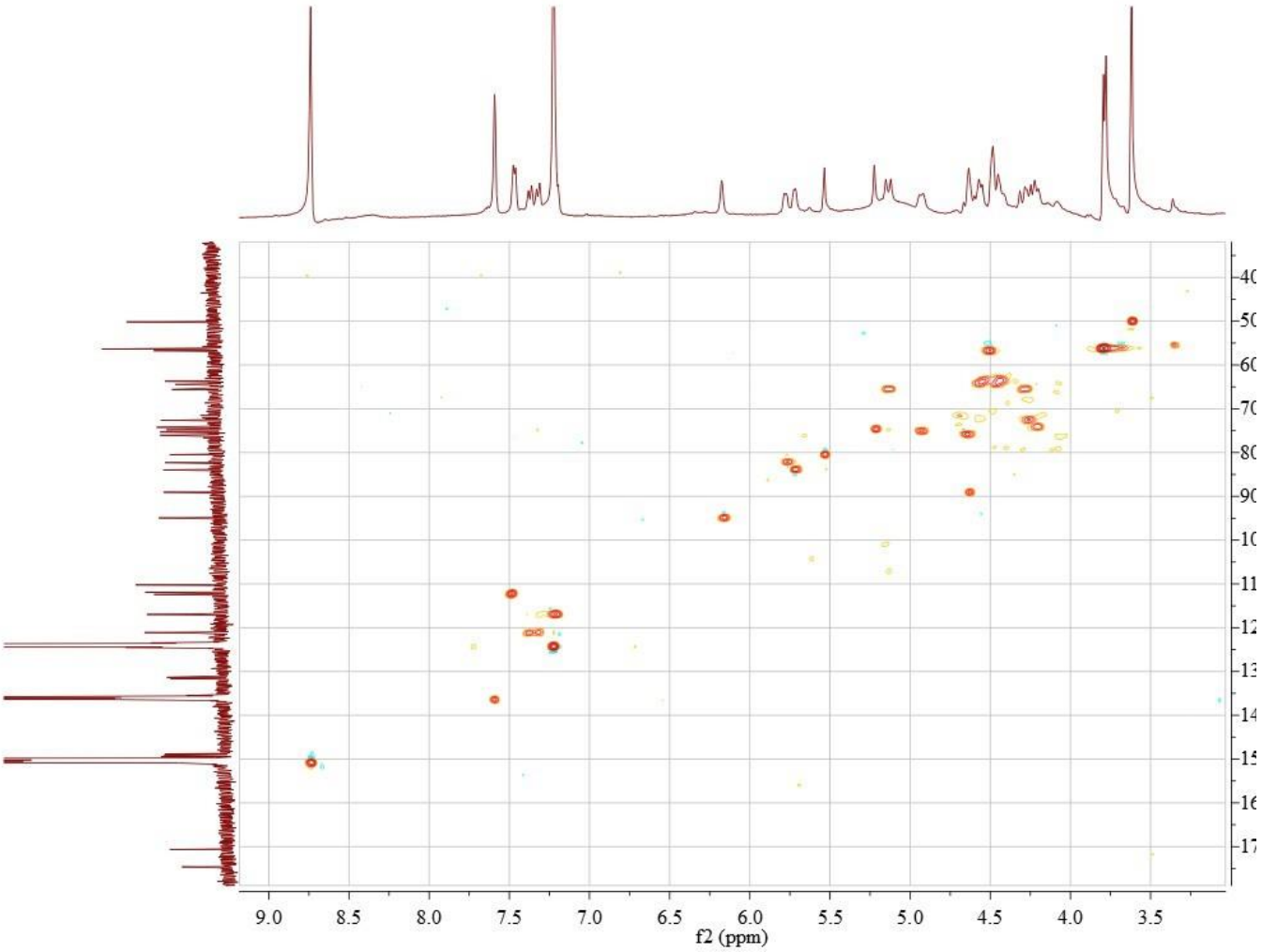


Figure S5: The DEPT spectrum of **1** (in pyridine-d_5)



F

Figure S6: The HMQC spectrum of **1** (in pyridine-d_5)

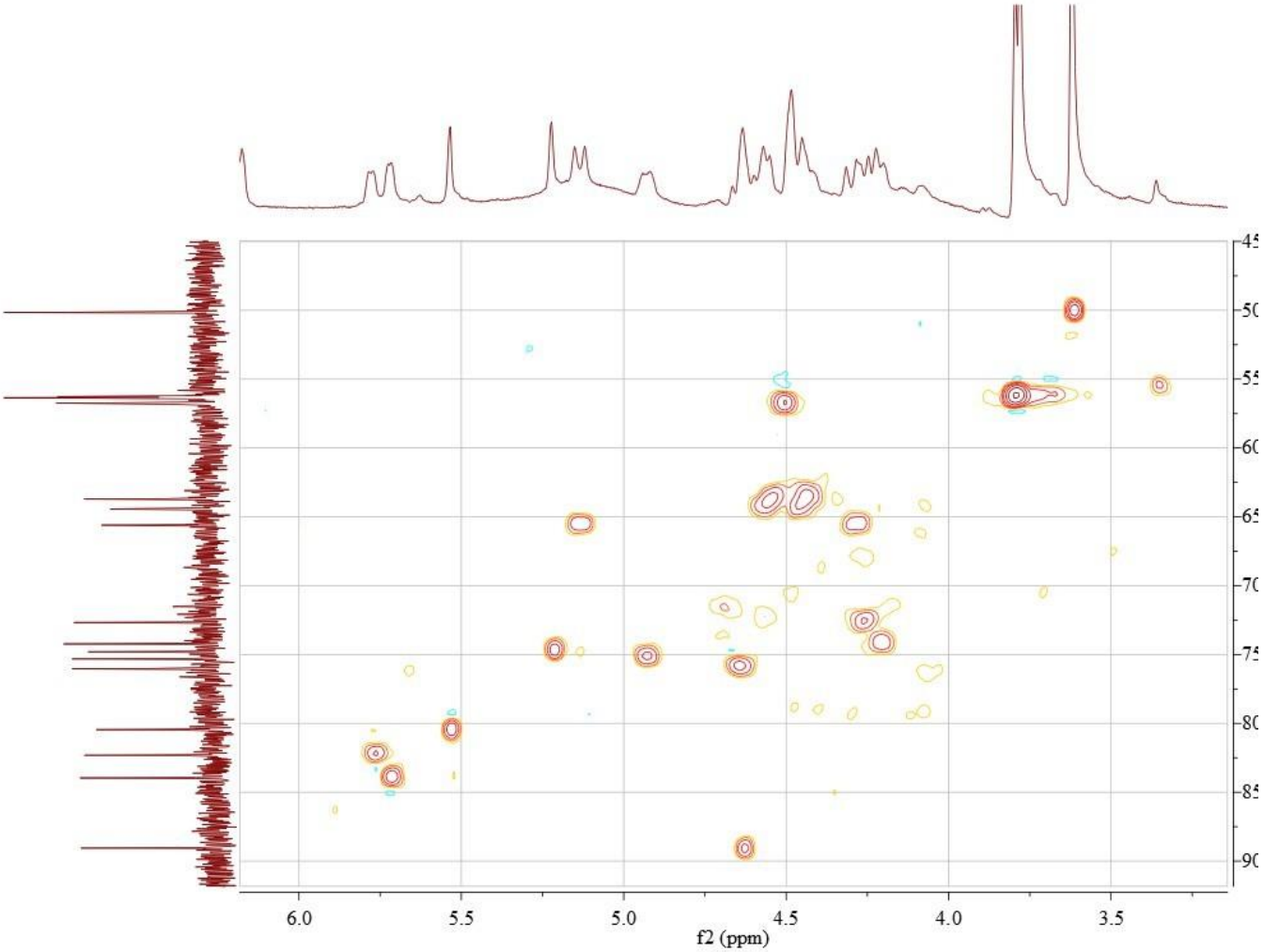


Figure S7: The HMQC spectrum of **1** (in pyridine-d_5) (From δ_C 45 ppm to δ_C 90 ppm)

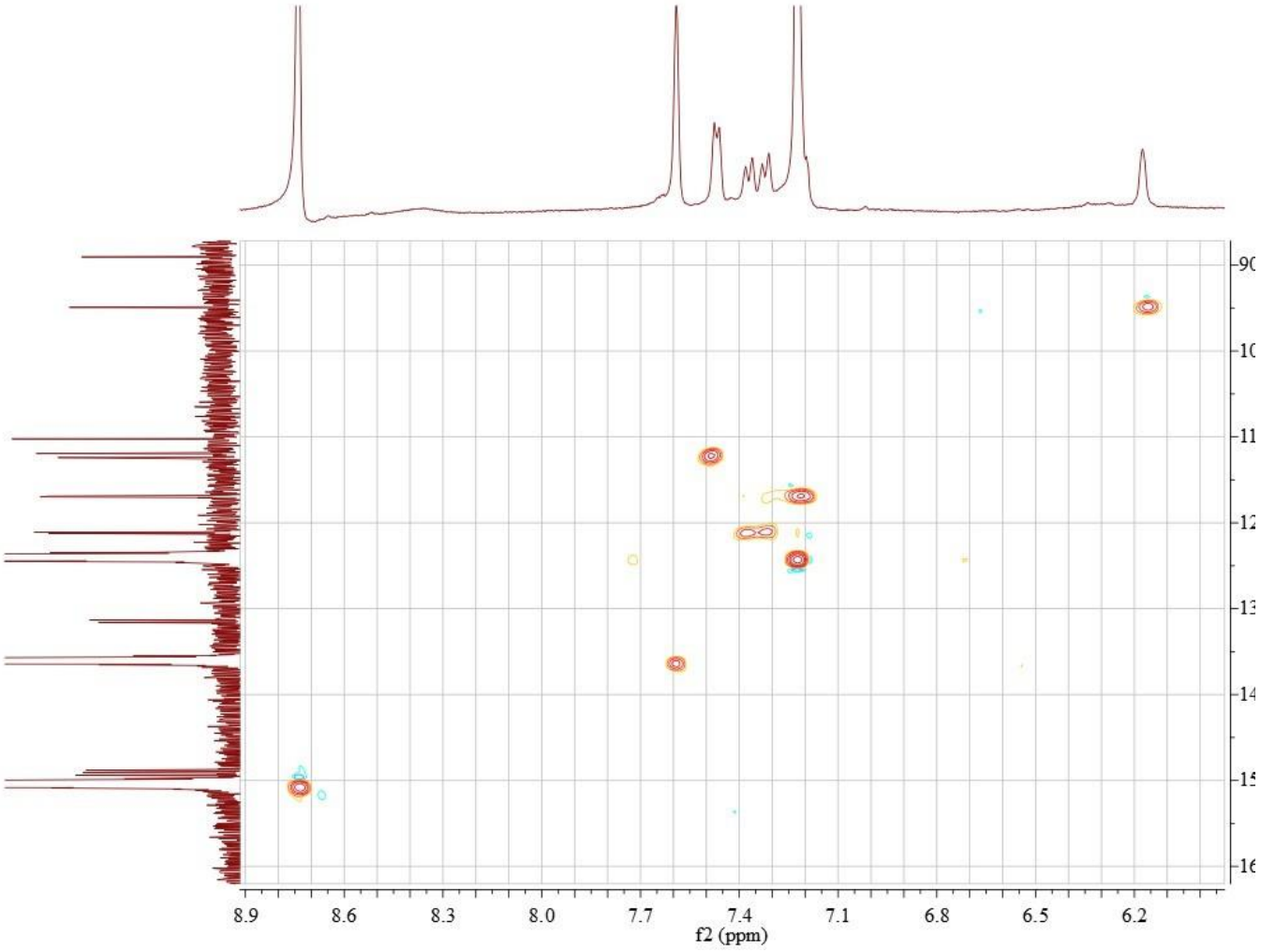


Figure S8: The HMQC spectrum of **1** (in pyridine-d_5) (From δ_C 90 ppm to δ_C 160 ppm)

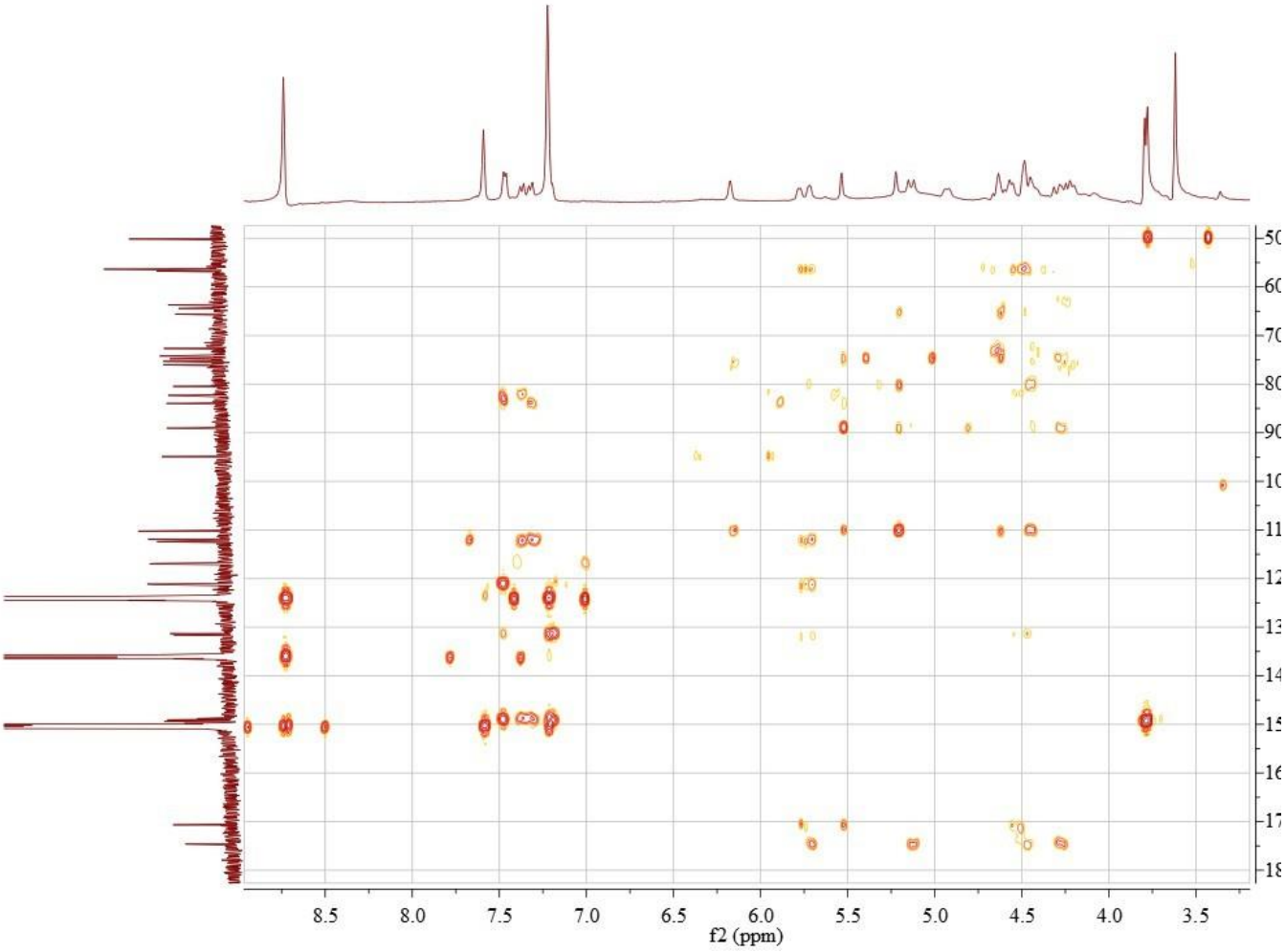


Figure S9: The HMBC spectrum of **1** (in pyridine-d_5)

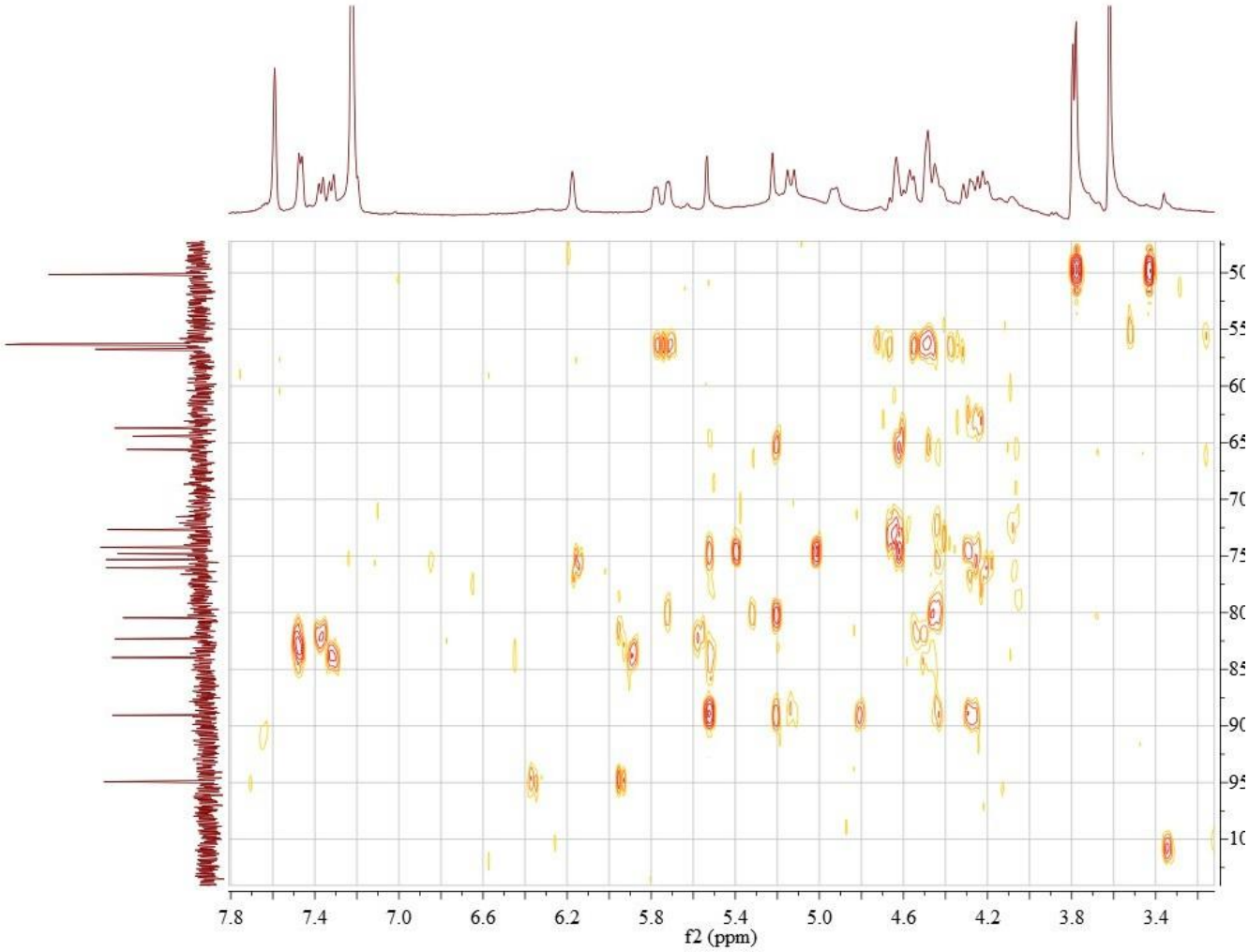


Figure S10: The HMBC spectrum of **1** (in pyridine-d_5) (From δ_C 50 ppm to δ_C 100 ppm)

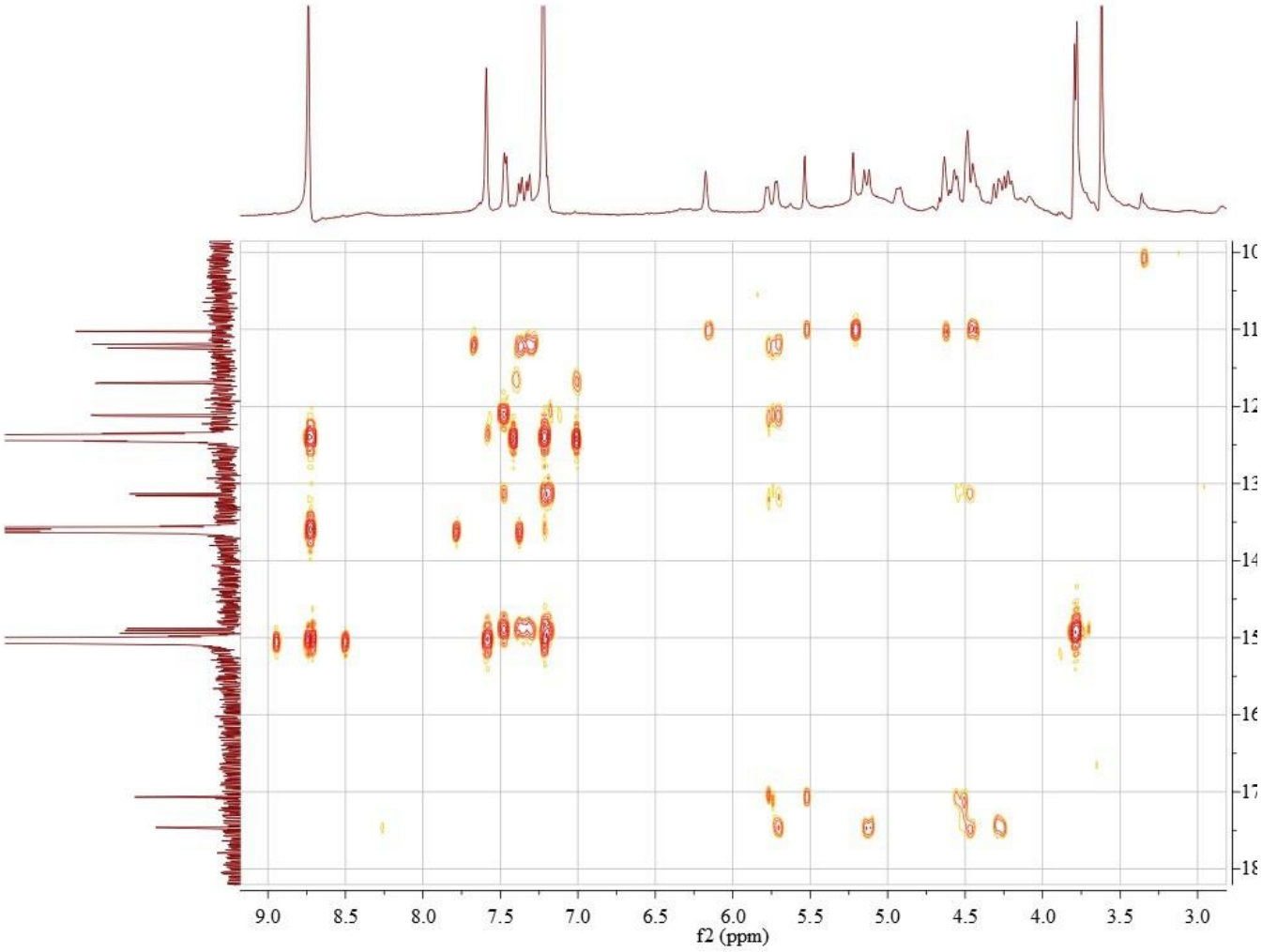


Figure S11: The HMBC spectrum of **1** (in pyridine-d_5) (From δ_C 100 ppm to δ_C 180 ppm)

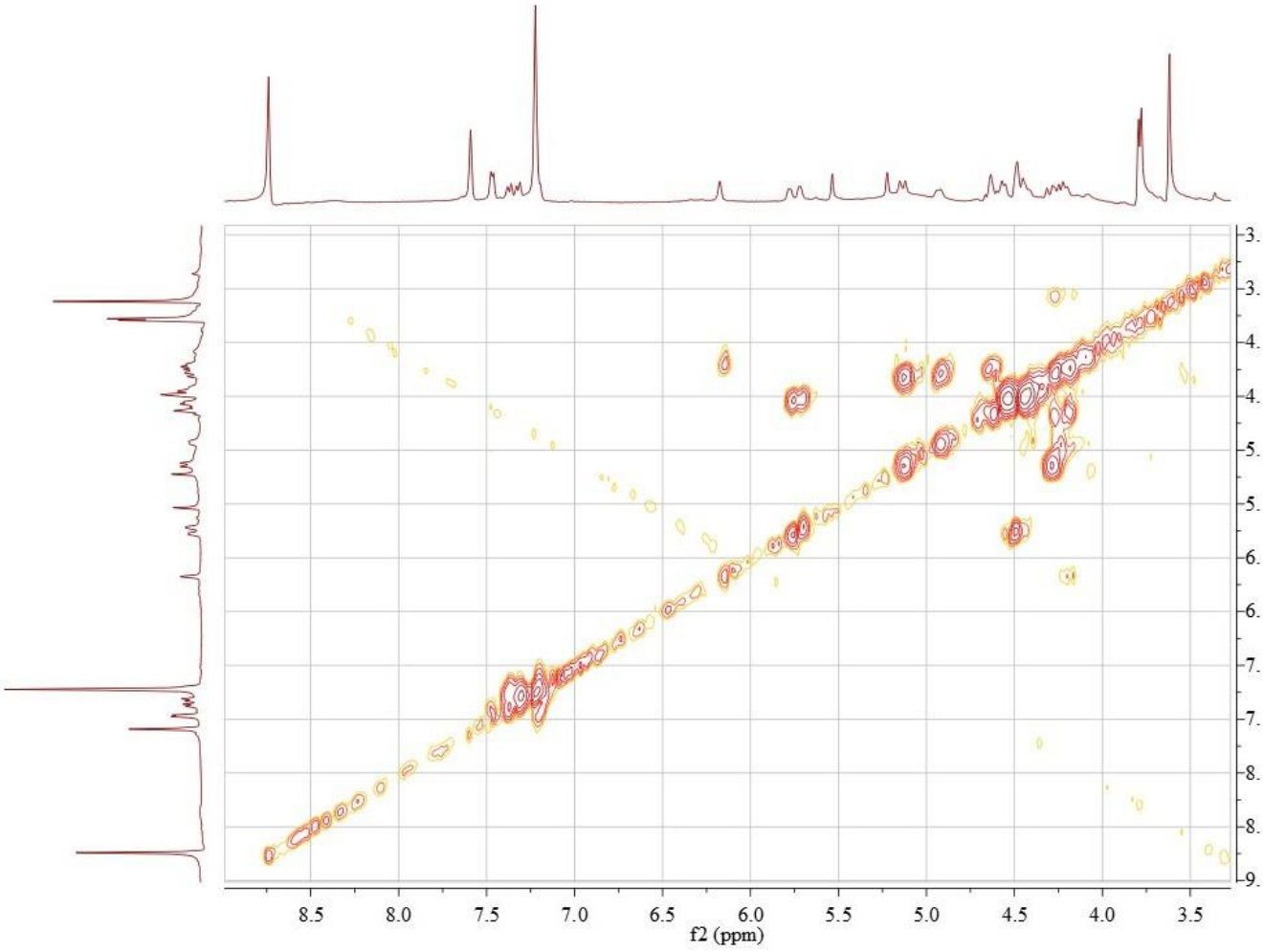


Figure S12. The ^{1}H-^{1}H COSY spectrum of **1** (in pyridine-d_5)

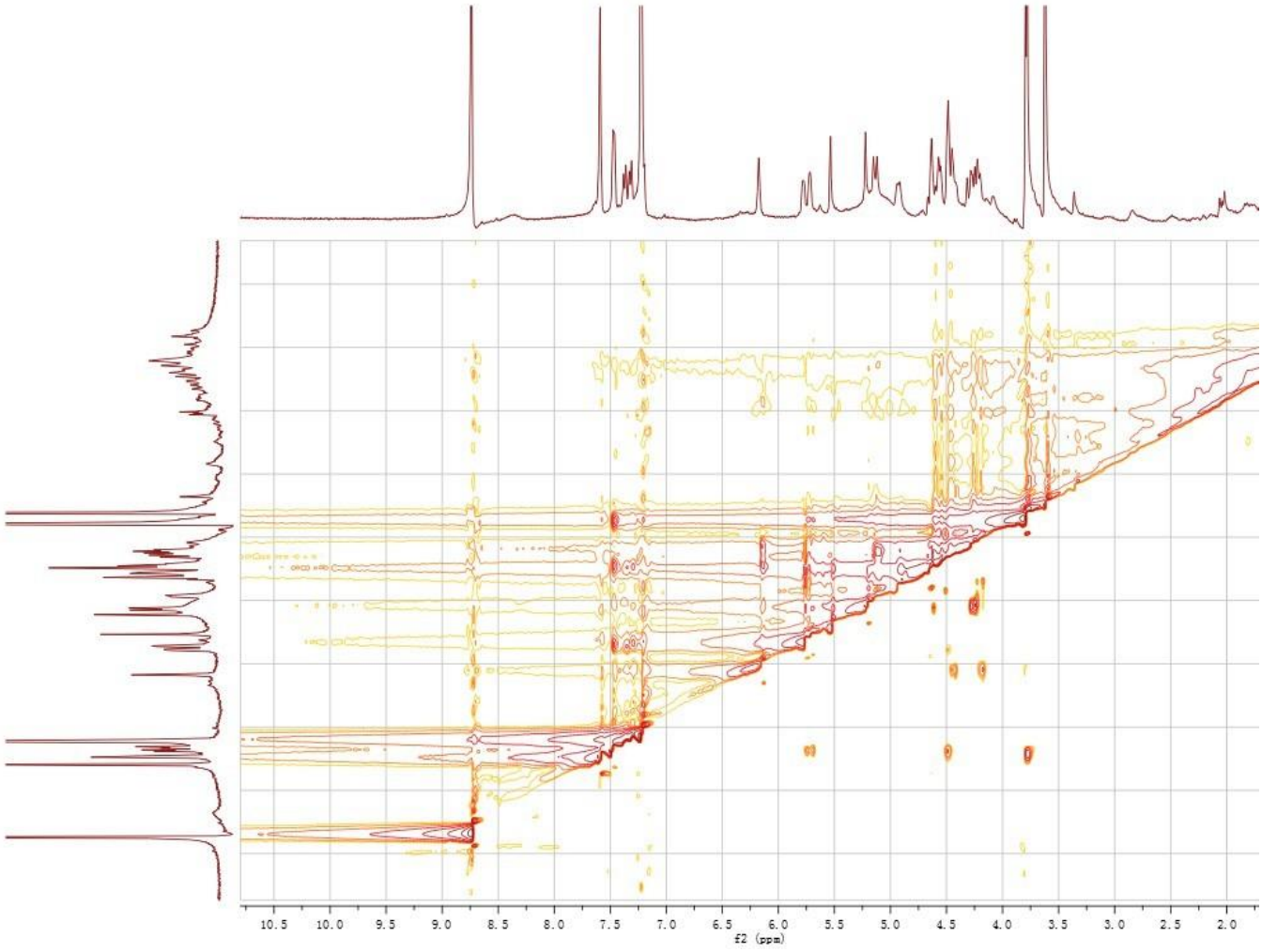
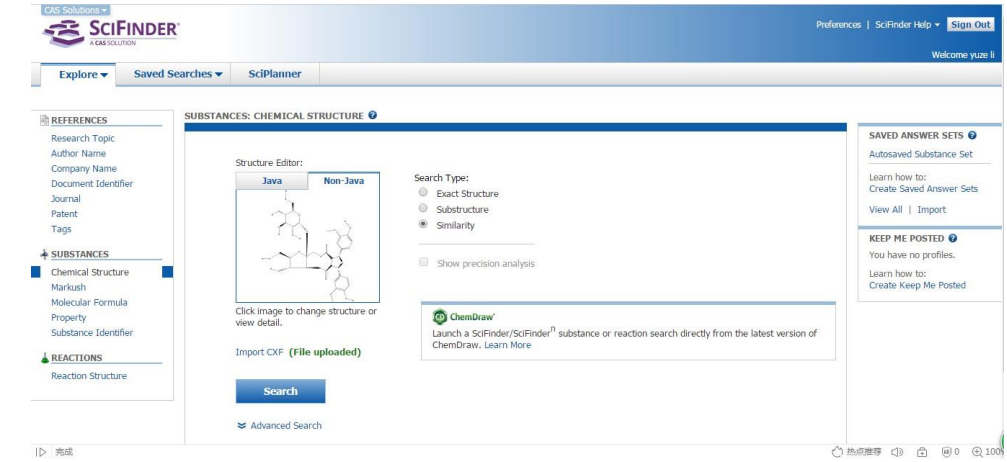
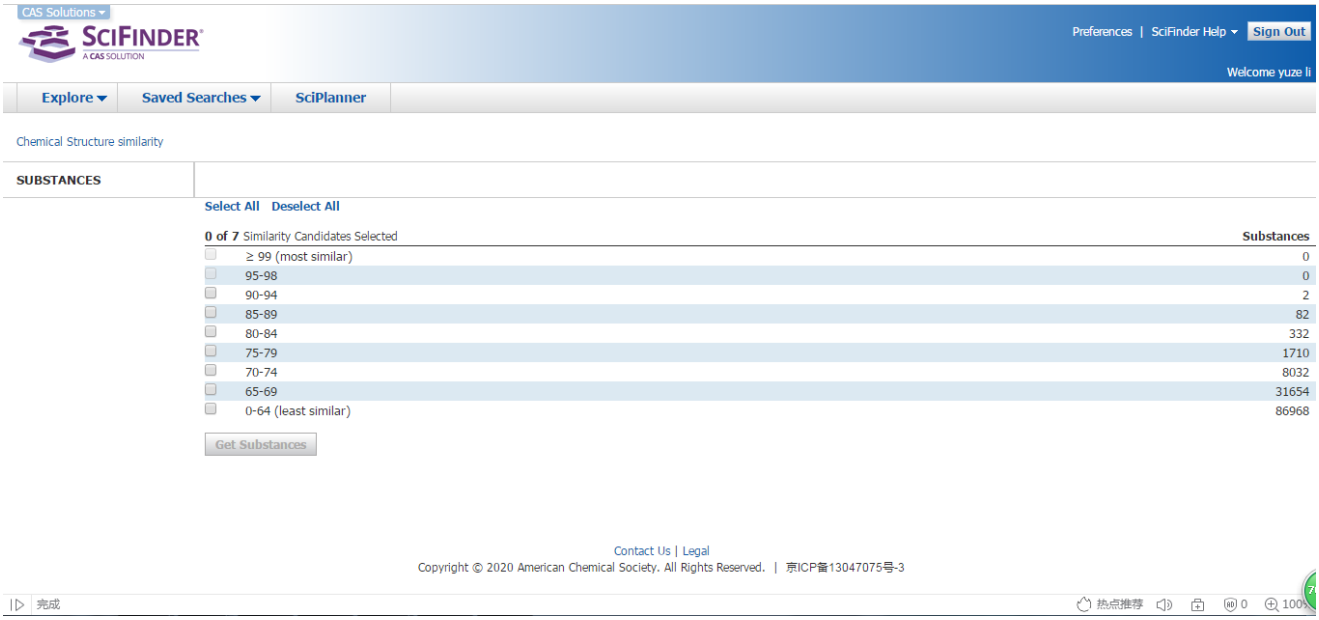


Figure S13: The NOESY spectrum of **1** (in pyridine-d_5)





Search report of SciFinder of 1

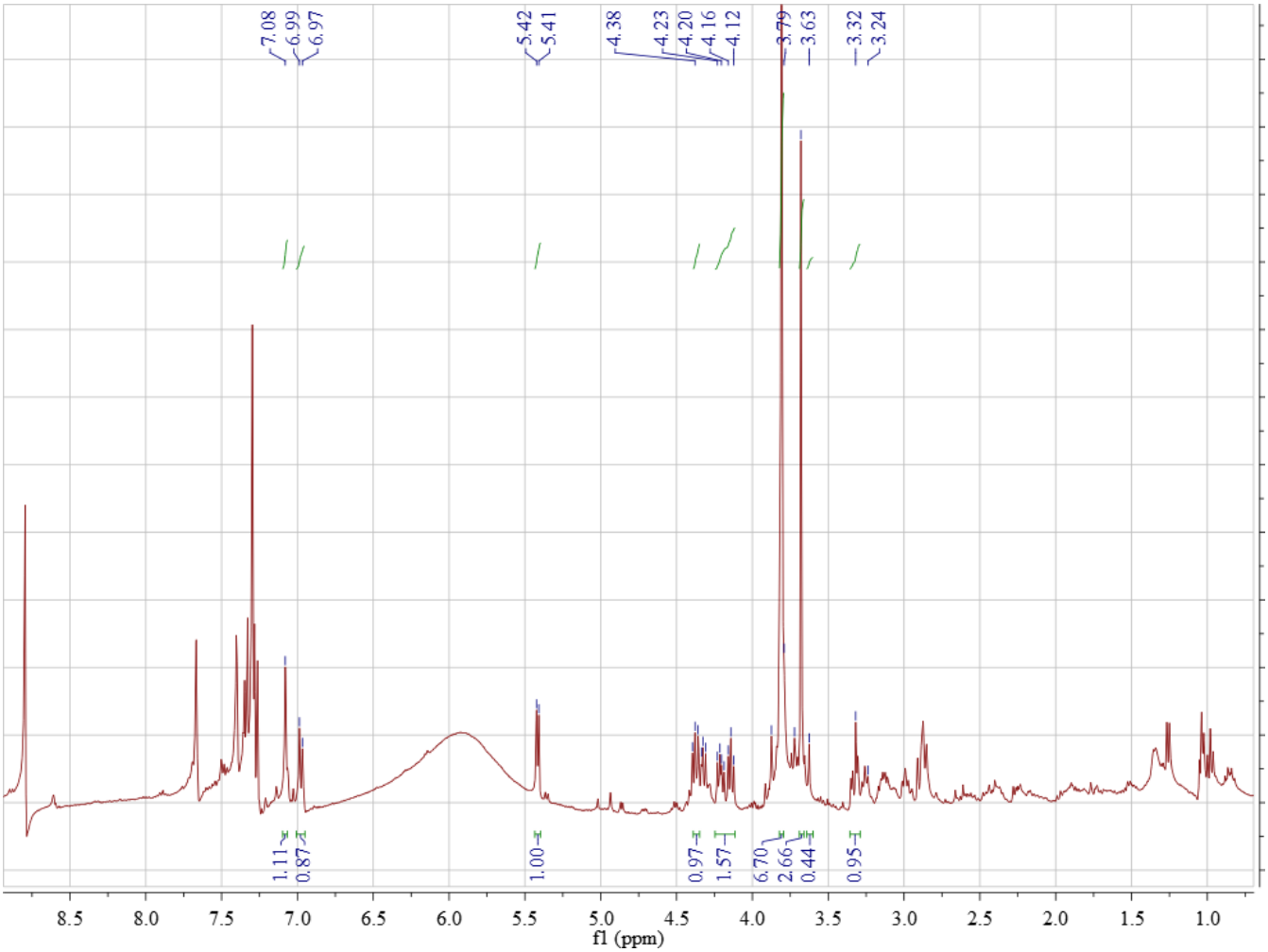


Figure S14: The ^{13}C NMR spectrum of **2** (in pyridine-d_5

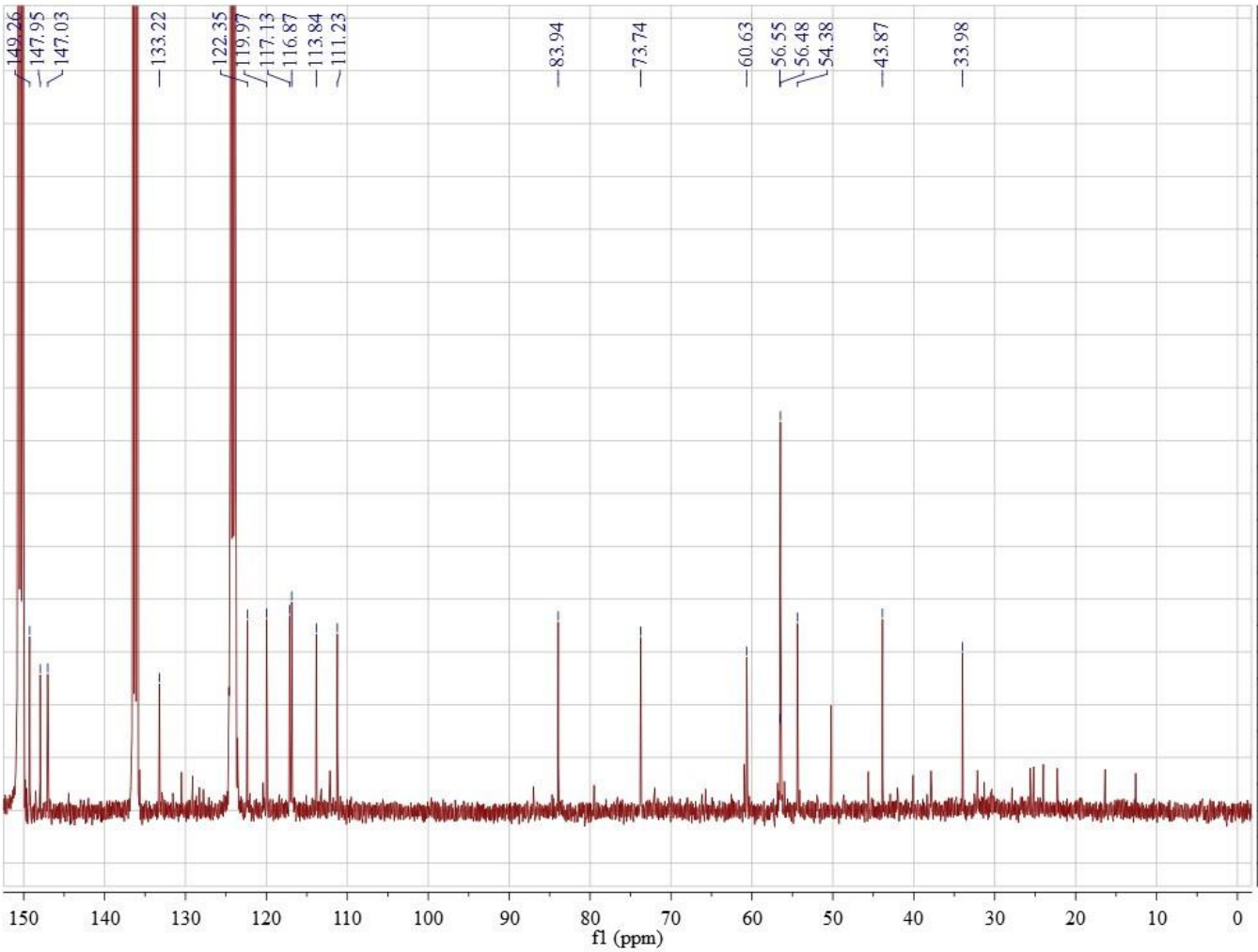


Figure S15: The ^{13}C NMR spectrum of **2** (in pyridine-d_5)

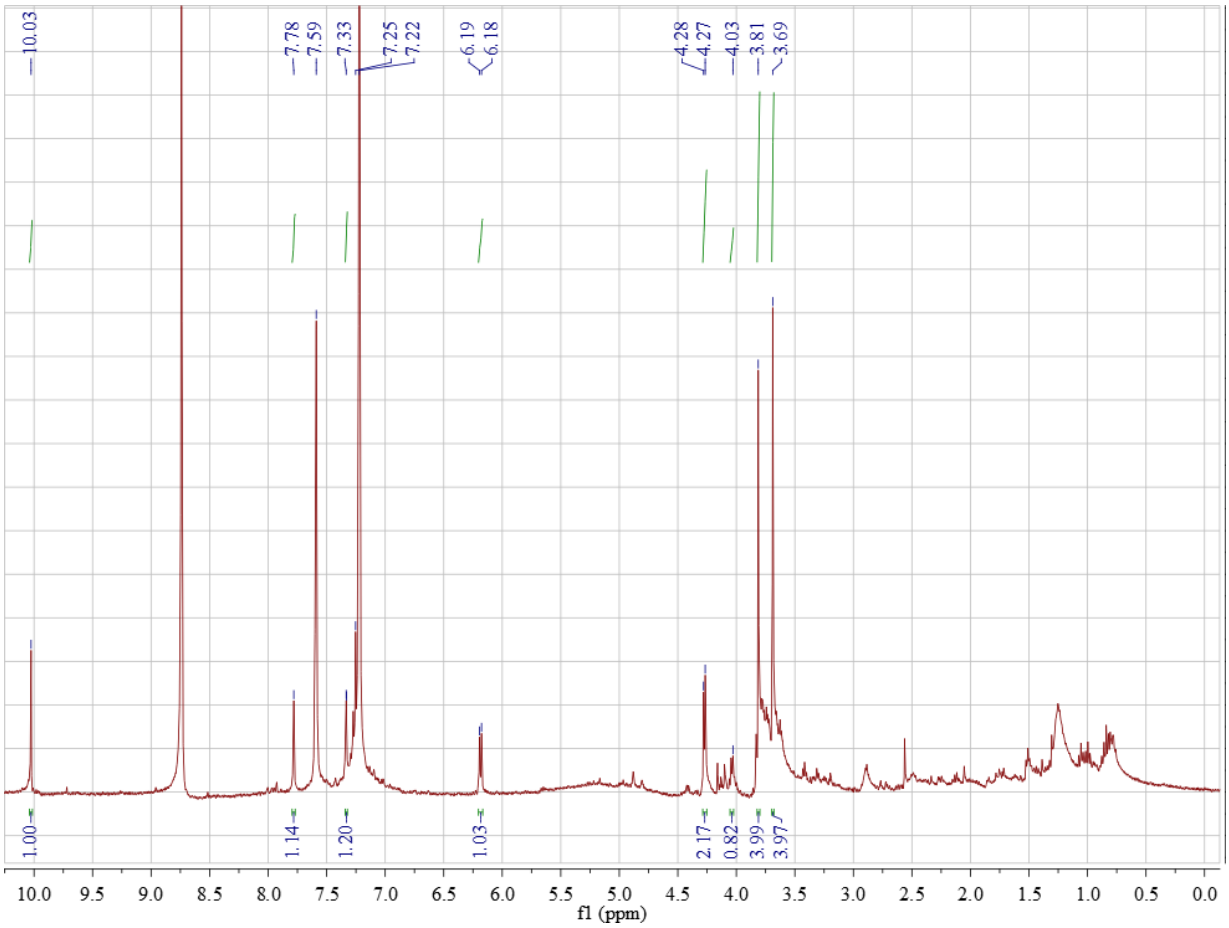


Figure S16: The ^{1}H NMR spectrum of **3** (in pyridine-d_5)

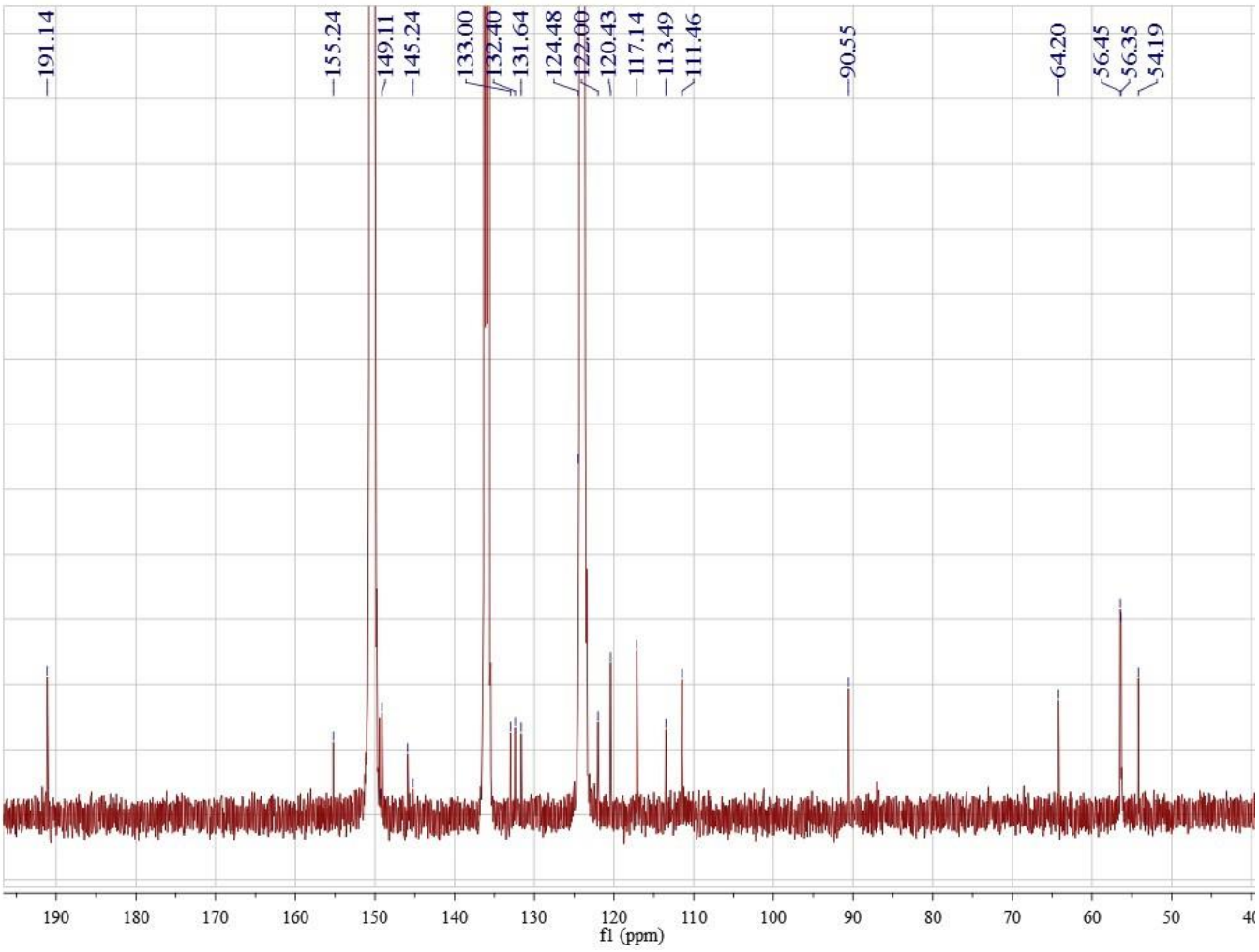


Figure S17: The ^{13}C NMR spectrum of **3** (in pyridine-d_5)

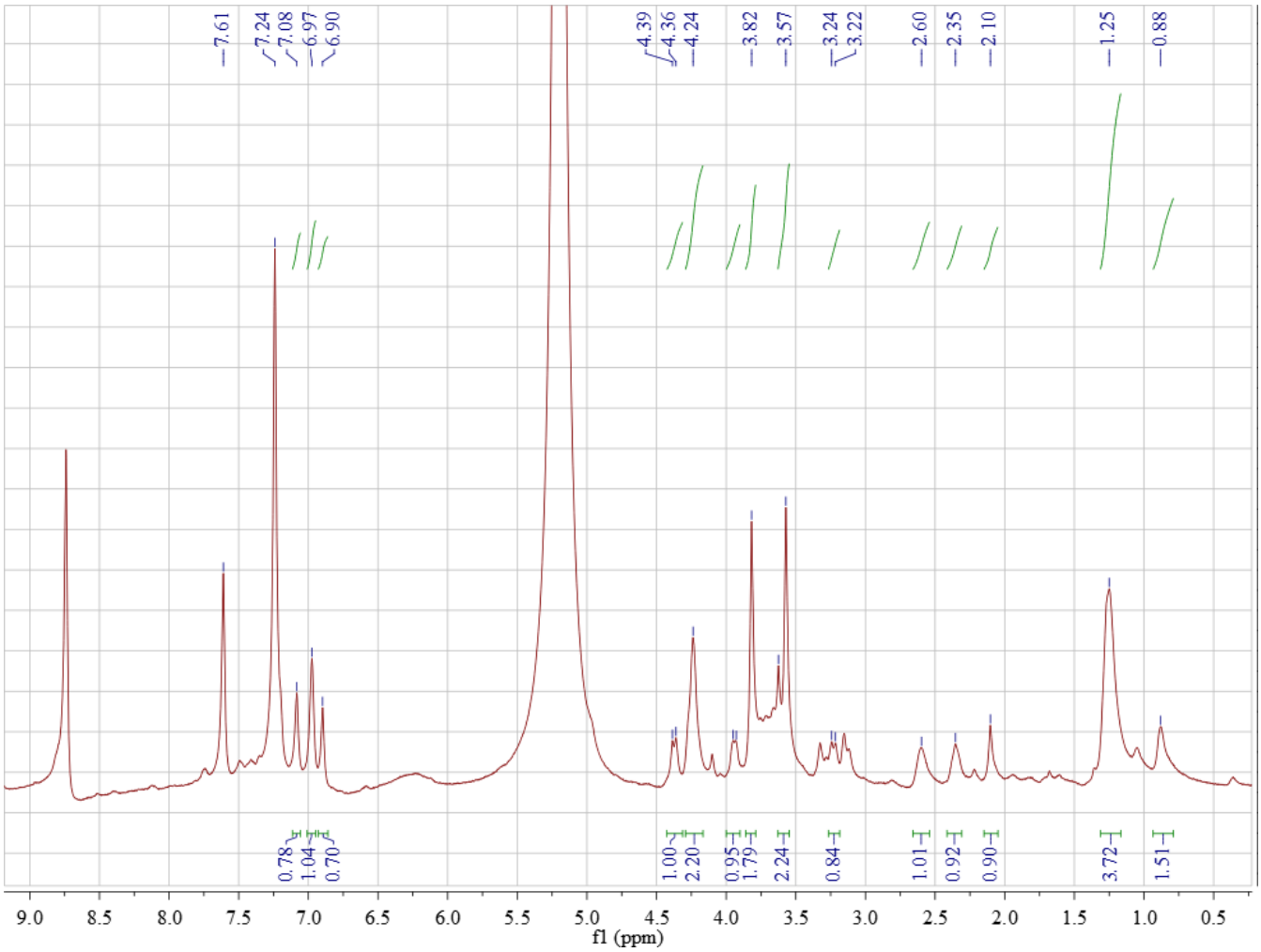


Figure S18: The ^{1}H NMR spectrum of **4** (in pyridine-d_5)

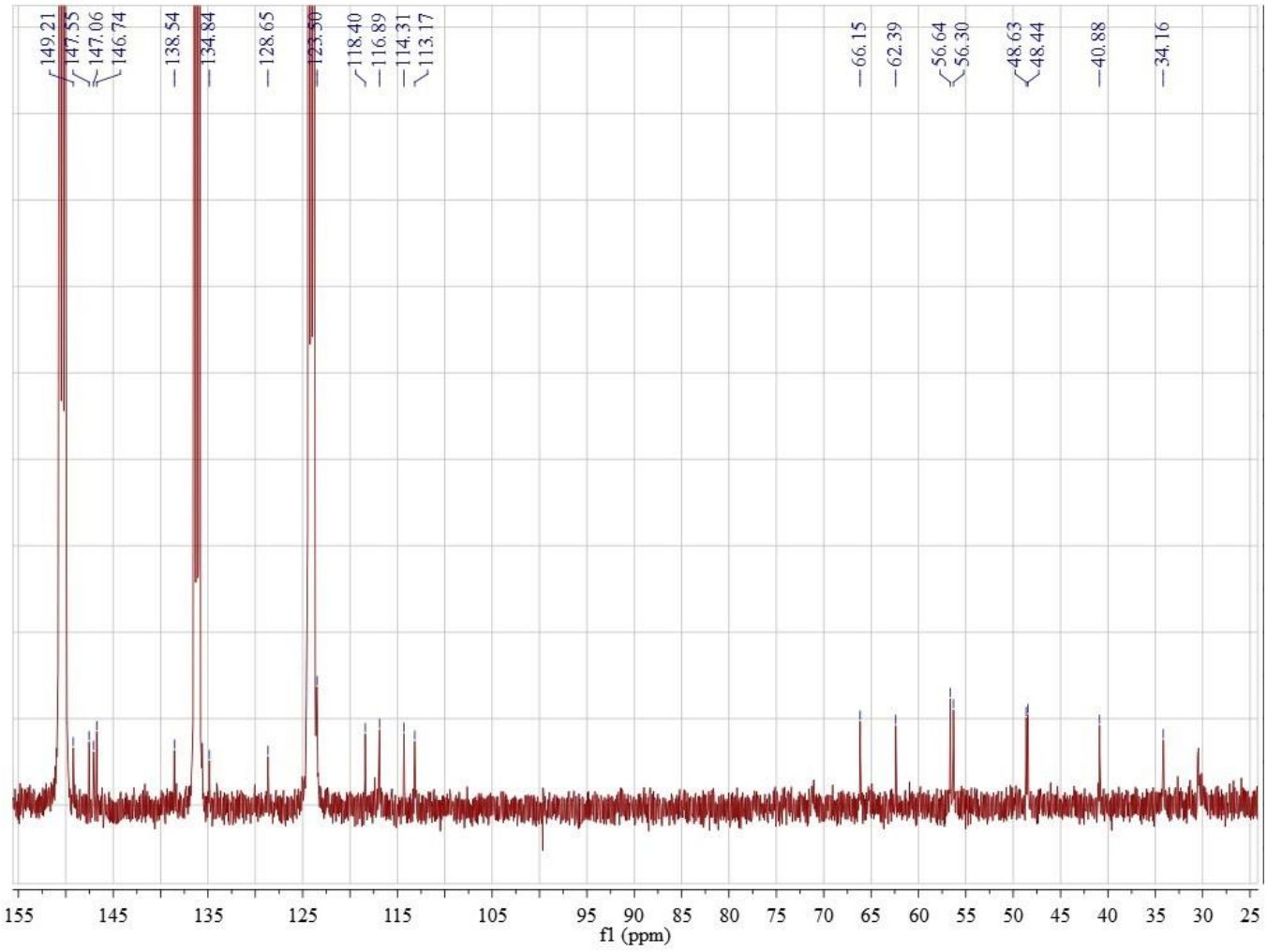


Figure S19: The ^{13}C NMR spectrum of **4** (in pyridine-d_5)