

Triterpene Glycosides from *Curculigo orchoides* and their Cytotoxic Activity

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Table S1. ¹H NMR Chemical Shift Assignments for **1**, **1a**, and **2-6** in C₅D₅N.^a

position	1	1a	2	3	4	5	6
1 eq	1.18 m	1.30 m	1.12 m	1.15 m	1.31 m	1.12 m	1.16 m
ax	1.51 m	1.61 m	1.48 m	1.47 m	1.67 m	1.47 m	1.46 m
2 eq	2.38 m	1.96 m	2.36 m	2.21 m	2.52 m	2.33 m	2.32 m
ax	1.88 m	1.87 m	1.87 m	1.89 m	1.95 m	1.86 m	1.87 m
3	3.52 dd (11.7, 4.4)	3.54 dd (11.6, 4.6)	3.45 dd (11.6, 4.2)	3.44 dd (11.6, 4.3)	3.51 dd (11.6, 4.4)	3.45 dd (11.6, 4.2)	3.41 dd (11.6, 4.4)
4	—	—	—	—	—	—	—
5	1.32 dd (13.5, 4.3)	1.36 m	1.23 dd (12.9, 4.0)	1.26 dd (12.7, 3.7)	1.26 dd (12.0, 4.6)	1.24 dd (12.4, 3.9)	1.25 dd (12.3, 3.8)
6 α	1.58 m	1.62 m	1.54 m	1.56 m	1.54 m	1.57 m	1.54 m
β	0.78 m	0.78 m	0.73 m	0.70 m	0.69 m	0.66 m	0.70 m
7 α	1.14 m	1.17 m	1.12 m	1.12 m	1.10 m	1.10 m	1.12 m
β	1.38 m	1.38 m	1.36 m	1.34 m	1.39 m	1.28 m	1.35 m
8	1.58 dd (12.3, 4.0)	1.62 m	1.58 dd (12.6, 4.4)	1.58 dd (12.7, 3.7)	1.56 dd (12.9, 4.4)	1.54 dd (12.5, 4.6)	1.58 m
9	—	—	—	—	—	—	—
10	—	—	—	—	—	—	—
11 α	2.35 dd (14.5, 5.5)	2.41 dd (14.6, 5.7)	2.33 dd (15.3, 5.7)	2.34 dd (14.6, 5.5)	2.36 dd (14.9, 5.6)	2.32 dd (14.9, 5.8)	2.34 dd (14.9, 5.9)
β	1.86 dd (14.5, 9.3)	1.93 dd (14.6, 9.4)	1.87 dd (15.3, 9.3)	1.85 dd (14.6, 9.0)	1.85 dd (14.9, 9.4)	1.82 dd (14.9, 9.4)	1.85 dd (14.9, 9.1)
12	4.13 dd (9.3, 5.5)	4.16 dd (9.4, 5.7)	4.12 dd (9.3, 5.7)	4.13 dd (9.0, 5.5)	4.10 dd (9.4, 5.6)	4.11 dd (9.4, 5.8)	4.12 dd (9.1, 5.9)
13	—	—	—	—	—	—	—
14	—	—	—	—	—	—	—
15 α	2.21 dd (13.0, 8.1)	2.24 dd (12.9, 8.0)	2.20 dd (13.0, 8.1)	2.21 dd (12.9, 8.0)	2.21 dd (12.8, 8.0)	2.21 dd (13.0, 8.0)	2.21 dd (12.9, 8.0)
β	1.82 dd (13.0, 4.5)	1.85 dd (12.9, 4.8)	1.81 m	1.82 dd (12.9, 5.2)	1.82 dd (12.8, 4.5)	1.81 m	1.82 dd (12.9, 4.7)
16	4.82 ddd (8.1, 7.5, 4.5)	4.84 ddd (8.0, 7.5, 4.8)	4.81 m	4.82 ddd (8.0, 7.6, 5.2)	4.83 ddd (8.0, 7.6, 4.5)	4.81 m	4.82 ddd (8.0, 7.6, 4.7)
17	2.77 dd (10.9, 7.5)	2.78 dd (10.9, 7.5)	2.76 dd (10.9, 7.6)	2.76 dd (10.8, 7.6)	2.77 dd (10.9, 7.6)	2.76 dd (10.8, 7.7)	2.76 dd (10.9, 7.6)
18	1.40 s	1.43 s	1.38 s	1.41 s	1.39 s	1.38 s	1.40 s
19 a	0.48 d (3.8)	0.54 d (4.0)	0.49 d (3.6)	0.48 d (3.8)	0.45 d (3.9)	0.45 d (3.5)	0.48 d (3.9)
b	0.32 d (3.8)	0.41 d (4.0)	0.30 d (3.6)	0.31 d (3.8)	0.32 d (3.9)	0.29 d (3.5)	0.30 d (3.9)
20	2.25 m	2.27 m	2.23 m	2.26 m	2.25 m	2.24 m	2.25 m
21	1.39 d (7.5)	1.41 d (6.7)	1.38 d (6.7)	1.39 d (6.8)	1.40 d (6.7)	1.38 d (6.7)	1.39 d (6.9)
22 a	2.38 m	2.38 m	2.36 m	2.38 m	2.39 m	2.37 m	2.38 m
b	1.55 m	1.56 m	1.57 m	1.55 m	1.56 m	1.55 m	1.56 m
23 a	2.68 m	2.70 m	2.67 m	2.68 m	2.69 m	2.68 m	2.68 m
b	2.68 m	2.70 m	2.67 m	2.68 m	2.69 m	2.68 m	2.68 m
24	—	—	—	—	—	—	—
25	2.57 septet (6.9)	2.56 septet (6.9)	2.57 septet (6.9)	2.57 septet (6.9)	2.57 septet (6.9)	2.57 septet (6.9)	2.57 septet (6.9)
26	1.00 d (6.9)	1.01 d (6.9)	1.00 d (6.9)	1.01 d (6.9)	1.01 d (6.9)	1.00 d (6.9)	1.00 d (6.9)
27	1.03 d (6.9)	1.03 d (6.9)	1.02 d (6.9)	1.03 d (6.9)	1.03 d (6.9)	1.02 d (6.9)	1.03 d (6.9)
28	1.33 s	1.34 s	1.33 s	1.32 s	1.30 s	1.32 s	1.32 s
29	1.36 s	1.23 s	1.28 s	1.34 s	1.32 s	1.43 s	1.30 s
30	1.10 s	1.11 s	1.25 s	1.18 s	1.08 s	1.28 s	1.16 s
1'	4.96 d (7.8)	—	4.95 d (7.2)	4.94 d (7.6)	4.91 d (7.7)	4.94 d (7.0)	4.90 d (7.5)
2'	4.05 dd (8.9, 7.8)	—	4.30 dd (9.3, 7.2)	4.24 dd (9.0, 7.6)	4.00 dd (9.2, 7.7)	4.25 dd (9.1, 7.0)	4.22 dd (9.1, 7.5)
3'	4.25 dd (8.9, 8.9)	—	4.28 dd (9.3, 9.3)	4.32 dd (9.0, 9.0)	4.20 dd (9.2, 9.2)	4.23 dd (9.1, 9.1)	4.27 dd (9.1, 9.1)
4'	4.24 dd (8.9, 8.9)	—	4.15 dd (9.3, 9.3)	4.17 dd (9.0, 9.0)	4.13 dd (9.2, 9.2)	4.16 dd (9.1, 9.1)	4.14 dd (9.1, 9.1)
5'	3.97 ddd (8.9, 5.3, 2.5)	—	3.92 ddd (9.3, 5.3, 2.3)	3.88 ddd (9.0, 4.8, 2.3)	4.12 m	3.91 ddd (9.1, 5.0, 2.2)	3.86 ddd (9.1, 5.5, 2.2)
6' a	4.55 dd (11.7, 2.5)	—	4.53 dd (11.7, 2.3)	4.48 dd (12.0, 2.3)	4.86 m	4.52 dd (11.8, 2.2)	4.50 dd (11.7, 2.2)
b	4.40 dd (11.7, 5.3)	—	4.35 dd (11.7, 5.3)	4.35 dd (12.0, 5.3)	4.34 dd (11.1, 4.2)	4.35 dd (11.8, 5.0)	4.34 dd (11.7, 5.5)
1"	—	—	6.57 br s	5.39 d (7.6)	5.14 d (7.8)	6.53 br s	5.40 d (7.6)
2"	—	—	4.86 br d (3.4)	4.14 dd (9.1, 7.6)	4.06 dd (8.8, 7.8)	5.04 m	4.13 dd (9.3, 7.6)
3"	—	—	4.71 dd (9.3, 3.4)	4.25 dd (9.1, 9.1)	4.24 dd (8.8, 8.8)	4.89 dd (9.5, 2.9)	4.21 dd (9.3, 9.3)
4"	—	—	4.35 dd (9.3, 9.3)	4.34 dd (9.1, 9.1)	4.25 dd (8.8, 8.8)	4.53 dd (9.5, 9.5)	4.21 dd (9.3, 9.3)
5"	—	—	4.83 dq (9.3, 6.2)	3.94 ddd (9.1, 4.0, 3.6)	3.94 ddd (8.8, 5.1, 1.9)	4.81 dq (9.5, 6.1)	3.85 ddd (9.3, 4.5, 2.5)
6" a	—	—	1.72 d (6.2)	4.51 dd (11.5, 3.6)	4.52 dd (11.8, 1.9)	1.64 d (6.1)	4.43 dd (11.5, 2.5)
b	—	—	—	4.47 dd (11.5, 4.0)	4.38 dd (11.8, 5.1)	—	4.35 dd (11.5, 4.5)
1'''	—	—	—	—	—	5.51 d (7.9)	5.28 d (7.9)
2'''	—	—	—	—	—	4.12 dd (9.2, 7.9)	4.07 dd (9.3, 7.9)
3'''	—	—	—	—	—	4.28 dd (9.2, 9.2)	4.25 dd (9.3, 9.3)
4'''	—	—	—	—	—	4.30 dd (9.2, 9.2)	4.19 dd (9.3, 9.3)
5'''	—	—	—	—	—	3.99 ddd (9.2, 4.6, 2.2)	4.03 ddd (9.3, 5.8, 2.6)
6''' a	—	—	—	—	—	4.48 dd (11.7, 2.2)	4.54 dd (11.6, 2.6)
b	—	—	—	—	—	4.39 dd (11.7, 4.6)	4.31 dd (11.6, 5.8)

^aValues in parentheses are coupling constants in Hz

Table S2. ^{13}C NMR Chemical Shift Assignments for **1**, **1a**, and **2-6** in $\text{C}_5\text{D}_5\text{N}$.

position	1	1a	2	3	4	5	6
1	32.4	32.7	32.5	32.4	32.4	32.5	32.4
2	29.9	31.3	30.0	30.0	30.1	30.1	30.0
3	88.7	78.0	88.4	88.7	88.7	88.5	88.8
4	41.3	41.1	41.2	41.3	41.3	41.3	41.3
5	47.8	47.7	48.0	47.8	47.7	47.9	47.8
6	21.4	21.7	21.3	21.4	21.4	21.2	21.3
7	26.7	26.8	26.7	26.7	26.7	26.7	26.7
8	49.3	49.3	49.2	49.3	49.3	49.1	49.3
9	20.0	20.1	19.8	20.0	20.1	19.8	20.0
10	26.1	26.4	26.0	26.1	26.1	26.0	26.1
11	40.1	40.3	40.1	40.2	40.1	40.1	40.2
12	72.5	72.5	72.5	72.5	72.5	72.5	72.5
13	50.0	50.1	50.0	50.1	50.0	50.0	50.0
14	47.1	47.1	47.0	47.1	47.1	47.0	47.0
15	50.3	50.3	50.2	50.3	50.3	50.2	50.3
16	71.7	71.7	71.7	71.7	71.7	71.7	71.7
17	49.3	49.3	49.3	49.3	49.4	49.3	49.3
18	18.5	18.5	18.5	18.5	18.5	18.5	18.5
19	30.1	30.2	30.1	30.1	30.1	30.1	29.9
20	30.2	30.3	30.2	30.3	30.3	30.2	30.2
21	17.1	17.1	17.1	17.1	17.0	17.1	17.1
22	30.8	30.8	30.8	30.8	30.8	30.8	30.8
23	38.1	38.2	38.1	38.2	38.2	38.1	38.2
24	216.0	216.0	216.0	215.9	216.0	216.0	216.0
25	40.7	40.7	40.7	40.7	40.7	40.7	40.7
26	18.4	18.4	18.4	18.4	18.4	18.4	18.4
27	18.4	18.4	18.4	18.4	18.4	18.4	18.4
28	22.1	22.1	22.1	22.1	22.1	22.0	22.1
29	25.8	26.2	25.6	25.8	25.7	25.8	25.7
30	15.5	14.9	15.6	15.4	15.1	15.8	15.4
1'	106.8		105.2	104.9	106.8	105.3	104.7
2'	75.8		77.7	83.5	75.6	76.9	82.8
3'	78.7		79.9	78.4	78.6	79.7	78.4
4'	71.8		72.1	71.6	71.7	72.1	71.5
5'	78.2		78.1	78.0	77.1	78.1	77.9
6'	63.0		62.8	62.8	70.4	62.8	62.7
1''			101.7	106.1	105.5	101.3	105.2
2''			72.5	77.7	75.2	71.7	75.7
3''			72.5	78.0	78.4	83.3	88.0
4''			74.1	71.7	71.7	73.0	69.7
5''			69.6	78.2	78.4	69.5	77.9
6''			18.7	62.8	62.8	18.6	62.4
1'''						106.8	105.7
2'''						75.9	75.7
3'''						78.5	78.2
4'''						71.3	71.6
5'''						78.6	78.6
6'''						62.4	62.5

Table S3. Log GI₅₀ Values of **1a** against 39 Human Cancer Cell Lines.

type of cancer	cell line	log GI ₅₀ ^{a,b}
Breast	HBC-4	-5.50
	BSY-1	-5.71
	HBC-5	-5.54
	MCF-7	-5.42
	MDA-MB-231	-5.70
Central nervous system	U251	-5.66
	SF-268	-5.86
	SF-295	-5.68
	SF-539	-5.67
	SNB-75	-5.42
	SNB-78	-5.30
Colon	HCC2998	-4.78
	KM-12	-5.21
	HT-29	-5.48
	HCT-15	-5.46
	HCT-116	-5.62
Lung	NCI-H23	-5.51
	HCI-H226	-5.56
	NCI-H522	-5.54
	NCI-H460	-5.65
	A549	-5.57
	DMS273	-5.68
	DMS114	-5.75
Melanoma	LOX-IMVI	-5.81
Ovary	OVCAR-3	-5.49
	OVCAR-4	-5.62
	OVCAR-5	-5.32
	OVCAR-8	-5.81
	SK-OV-3	-5.32
Kidney	RXF-631L	-5.75
	ACHN	-5.70
Stomach	St-4	-5.56
	MKN1	-5.59
	MKN7	-5.72
	MKN28	-5.69
	MKN45	-4.19
	MKN74	-5.66
Prostate	DU-145	-5.68
	PC-3	-4.83
MG-MID ^c		-5.51
Delta ^d		0.35
Range ^e		1.67

^a Log concentrations for inhibition of cell growth at 50% relative to control

^b The cell growth was determined according to the sulforhodamine B assay.

^c Mean log GI₅₀ value in all cell lines tested

^d Difference in the log GI₅₀ value between the most sensitive cells and the MG-MID value

^e Difference in the log GI₅₀ value between the most and least sensitive cells

Figure S1. ^1H NMR (500 MHz; $\text{C}_5\text{D}_5\text{N}$) of **1**.

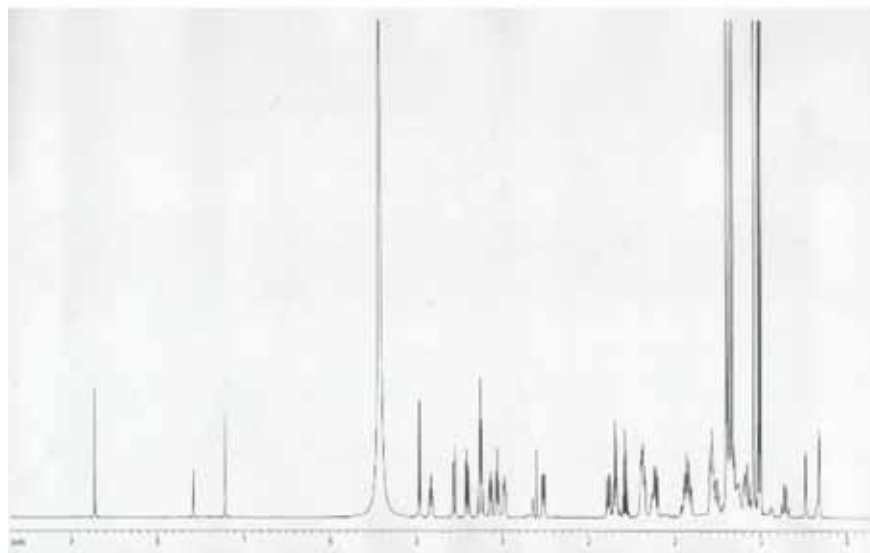


Figure S2. ^{13}C NMR (125 MHz; $\text{C}_5\text{D}_5\text{N}$) of **1**.

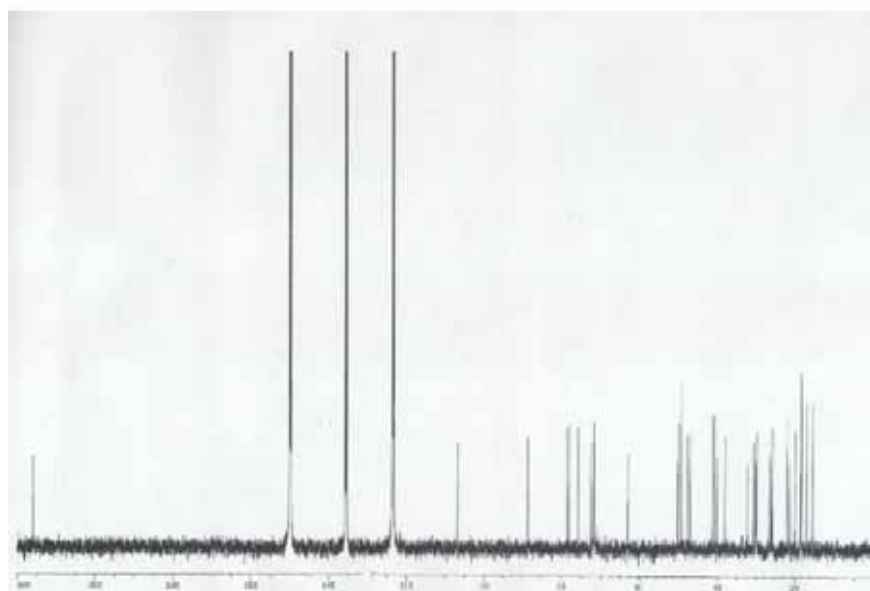


Figure S3. ^1H NMR (500 MHz; $\text{C}_5\text{D}_5\text{N}$) of **1a**.

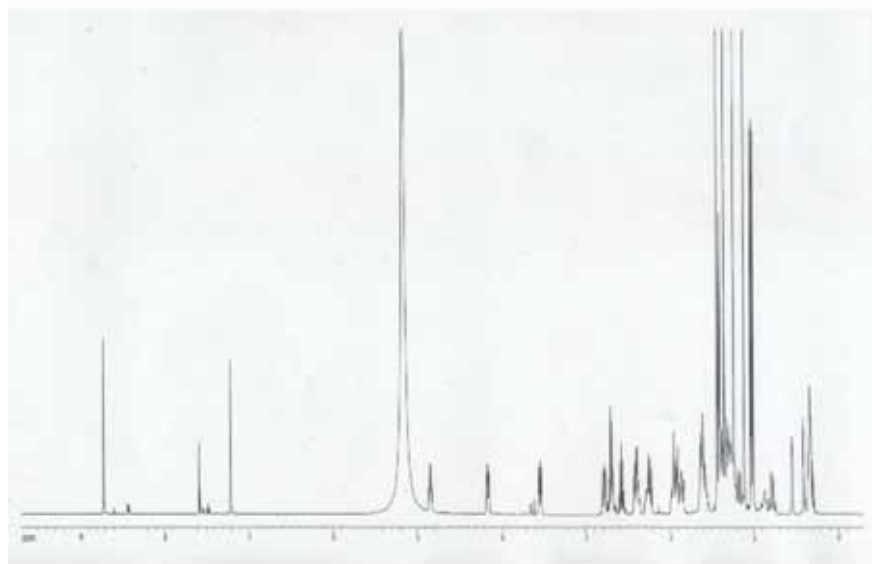


Figure S4. ^{13}C NMR (125 MHz; $\text{C}_5\text{D}_5\text{N}$) of **1a**.

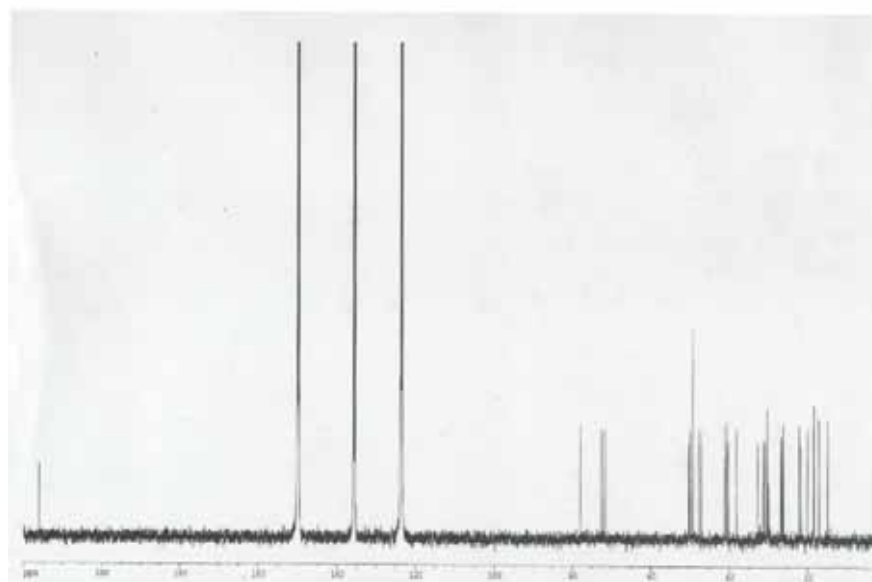


Figure S5. ^1H NMR (500 MHz; $\text{C}_5\text{D}_5\text{N}$) of **2**.

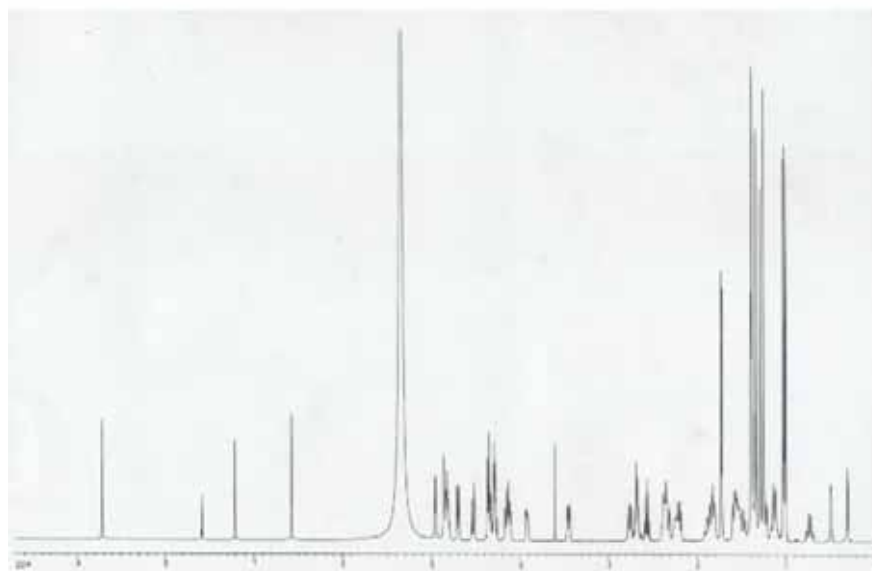


Figure S6. ^{13}C NMR (125 MHz; $\text{C}_5\text{D}_5\text{N}$) of **2**.

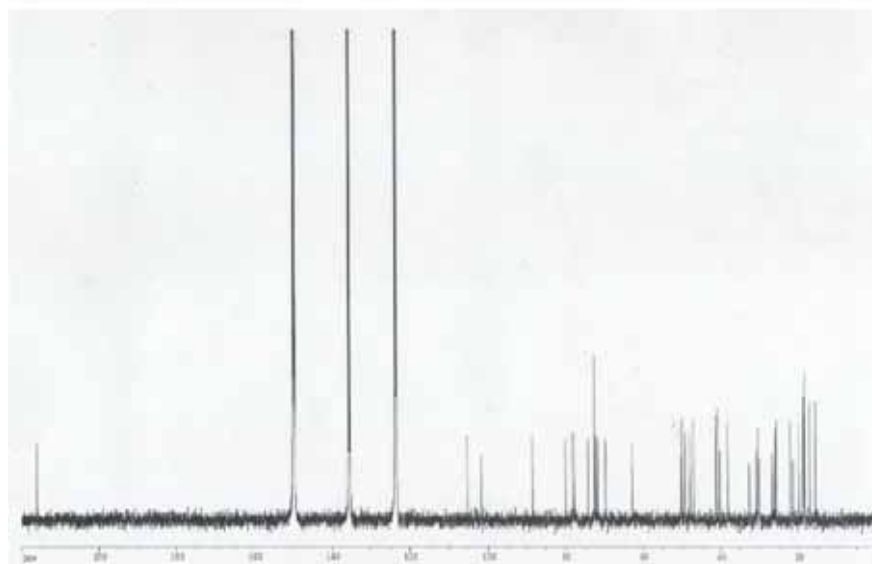


Figure S7. ^1H NMR (500 MHz; $\text{C}_5\text{D}_5\text{N}$) of **3**.

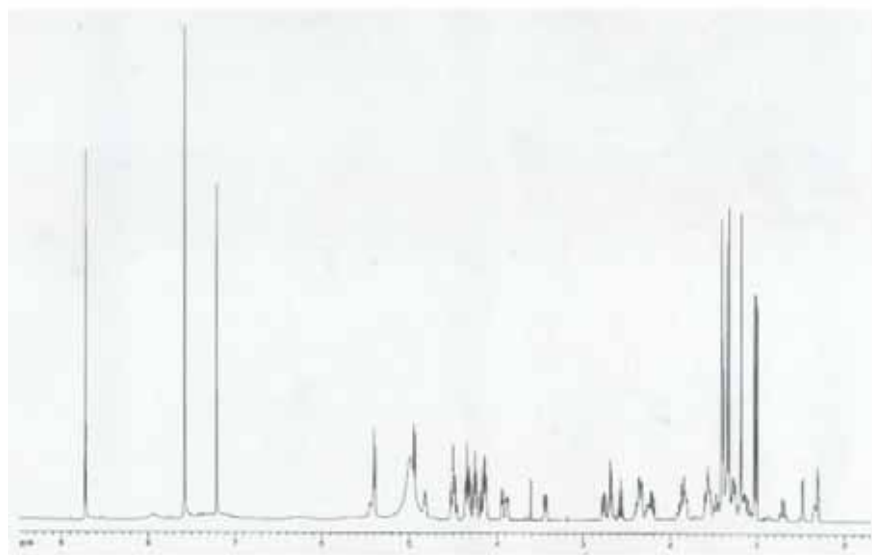


Figure S8. ^{13}C NMR (125 MHz; $\text{C}_5\text{D}_5\text{N}$) of **3**.

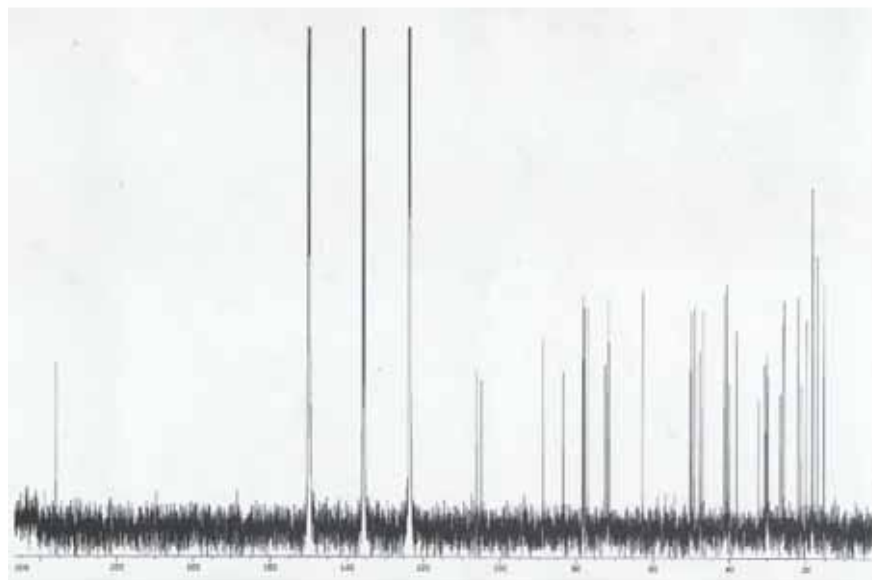


Figure S9. ^1H NMR (500 MHz; $\text{C}_5\text{D}_5\text{N}$) of **4**.

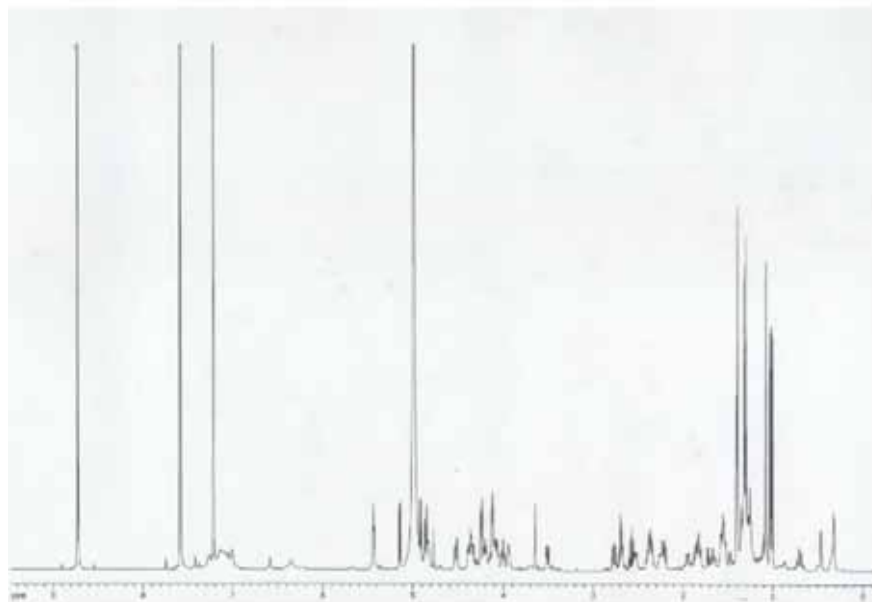


Figure S10. ^{13}C NMR (125 MHz; $\text{C}_5\text{D}_5\text{N}$) of **4**.

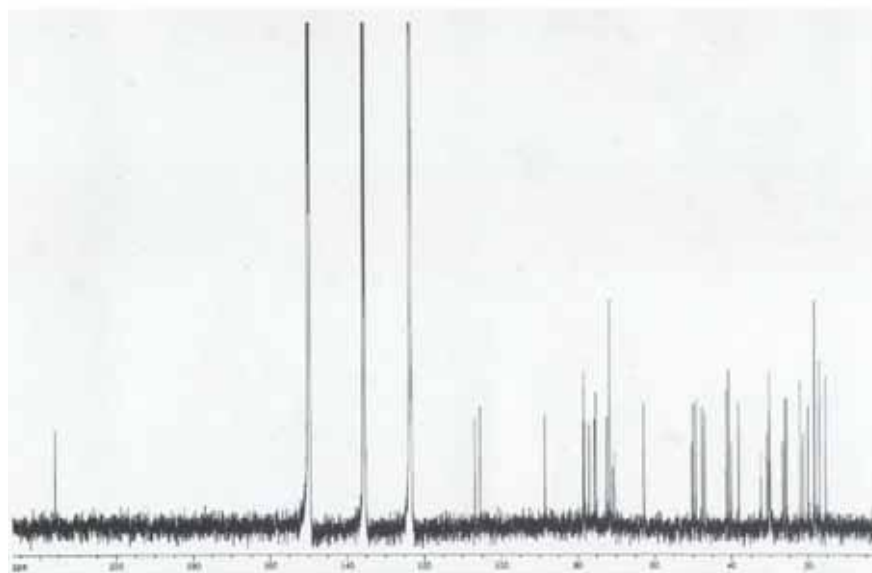


Figure S11. ^1H NMR (500 MHz; $\text{C}_5\text{D}_5\text{N}$) of **5**.

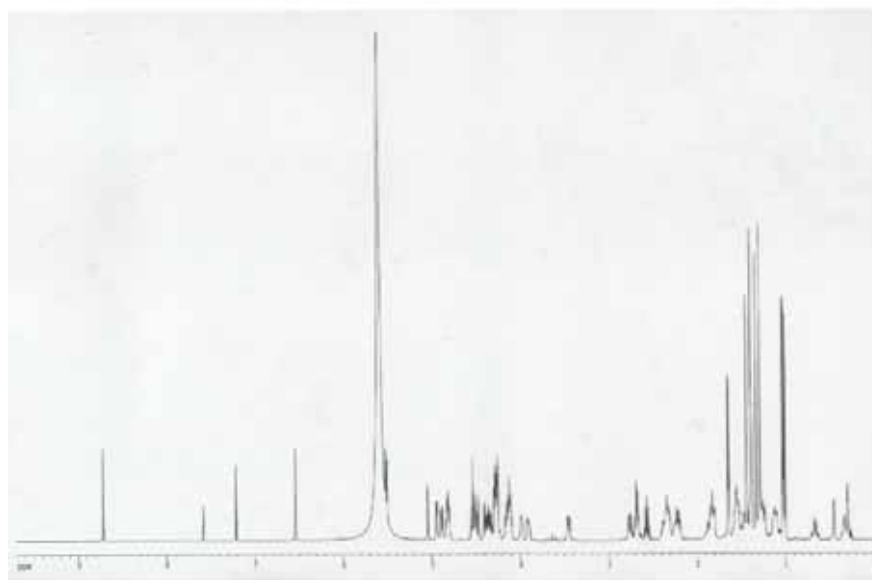


Figure S12. ^{13}C NMR (125 MHz; $\text{C}_5\text{D}_5\text{N}$) of **5**.

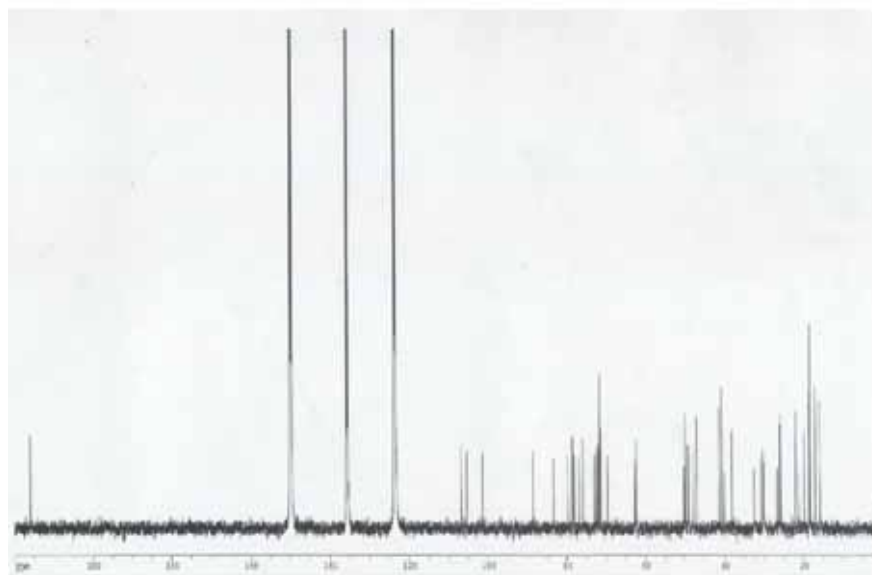


Figure S13. ^1H NMR (500 MHz; $\text{C}_5\text{D}_5\text{N}$) of **6**.

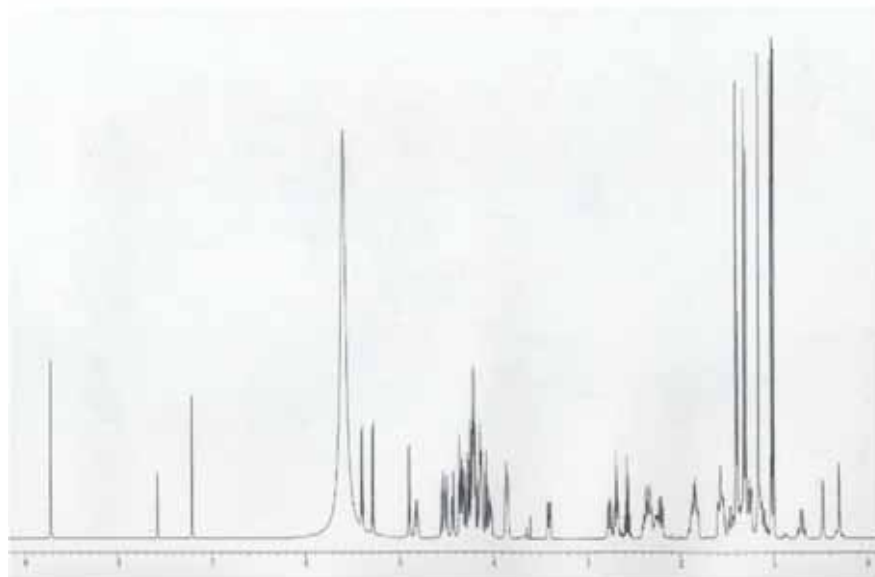


Figure S14. ^{13}C NMR (125 MHz; $\text{C}_5\text{D}_5\text{N}$) of **6**.

