

Supporting Information

Further Verticillene Diterpenoids, Eudesmane Sesquiterpe-noids, and Hydroperoxysteroids from a Taiwanese Soft Coral *Cespitularia* sp.

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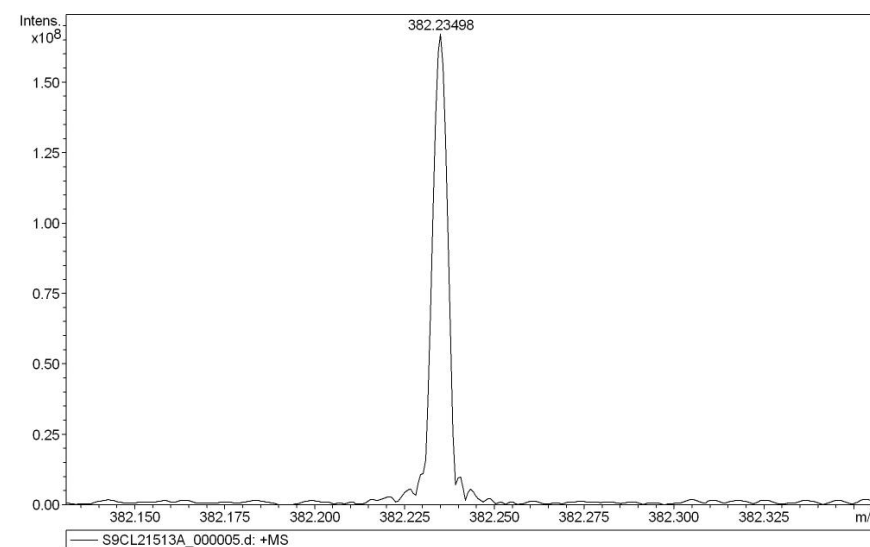
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Analysis Info

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 Sample Name S9-CL2-1-5-13A
 Comment ESI Positive

4/1/2021 3:18:11 PM
 Operator: YU HSIAO-CHING
 Instrument: BRUKER FT-MS solariX



Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	e ⁻ Conf	N-Rule
382.23498	1	C ₂₂ H ₃₃ NNaO ₃	100.00	382.23527	0.29	0.75	16.4	6.5	even	ok

Figure S1. HRESIMS of compound **1**

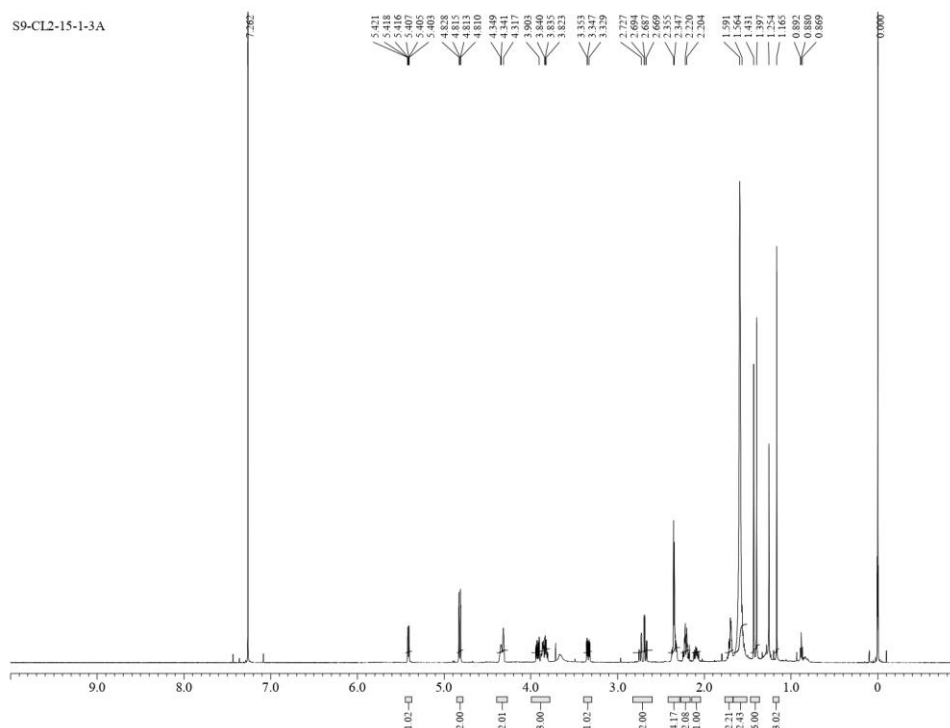


Figure S2. ¹H NMR spectrum (600MHz) of compound **1** in CDCl₃

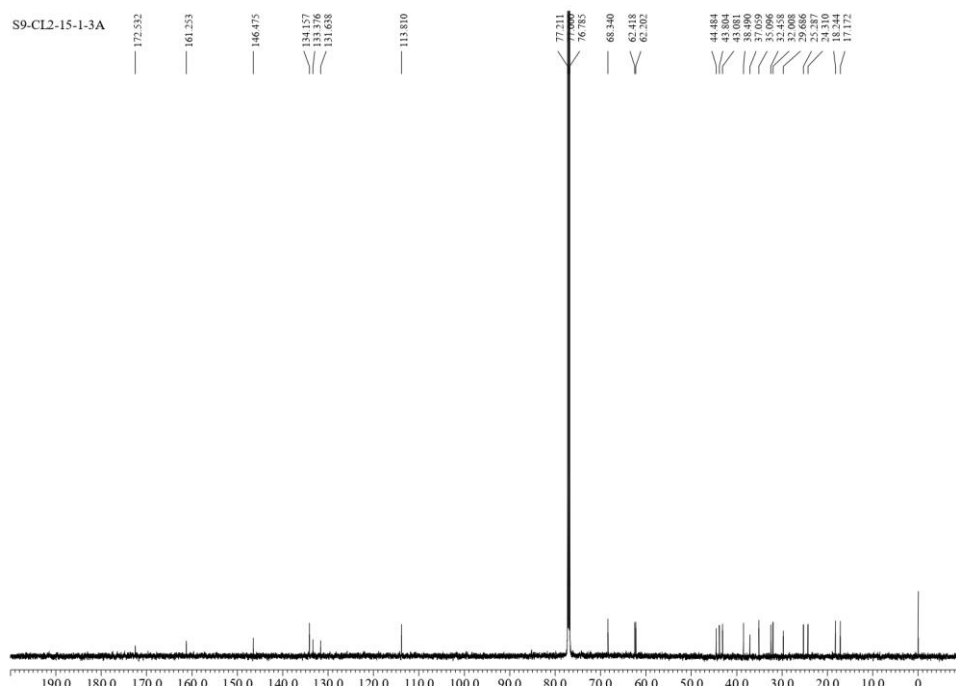


Figure S3. ^{13}C NMR spectrum (150 MHz) of compound **1** in CDCl_3

Y = 135[deg]
S9-CL2-15-1-3A

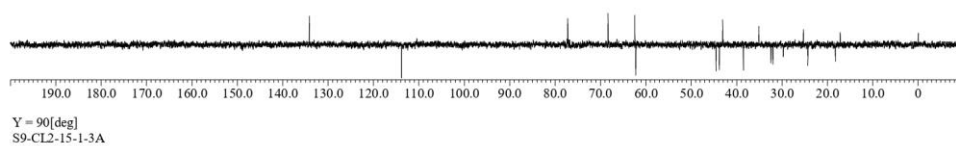


Figure S4. DEPT spectrum (150 MHz) of compound **1** in CDCl_3

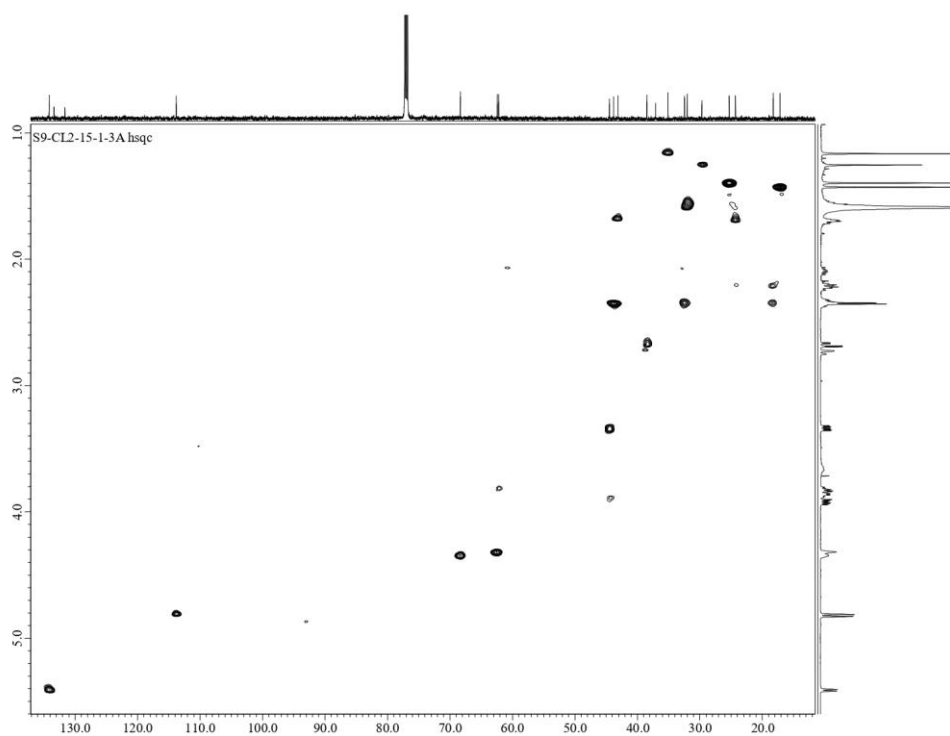


Figure S5. HSQC spectrum of compound **1** in CDCl₃

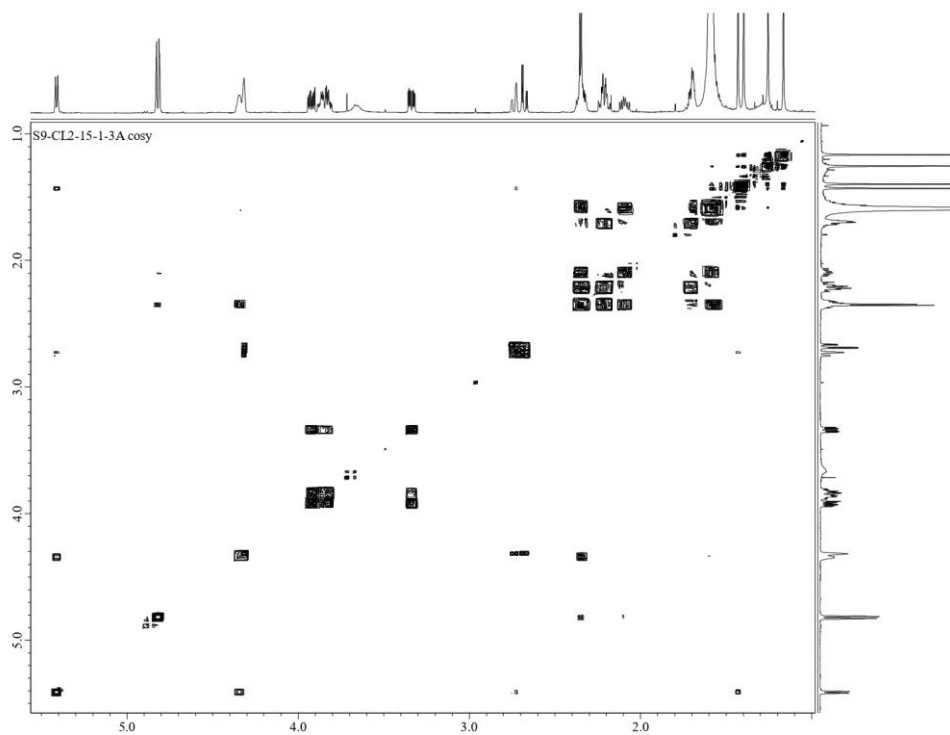


Figure S6. ¹H-¹H COSY spectrum of compound **1** in CDCl₃

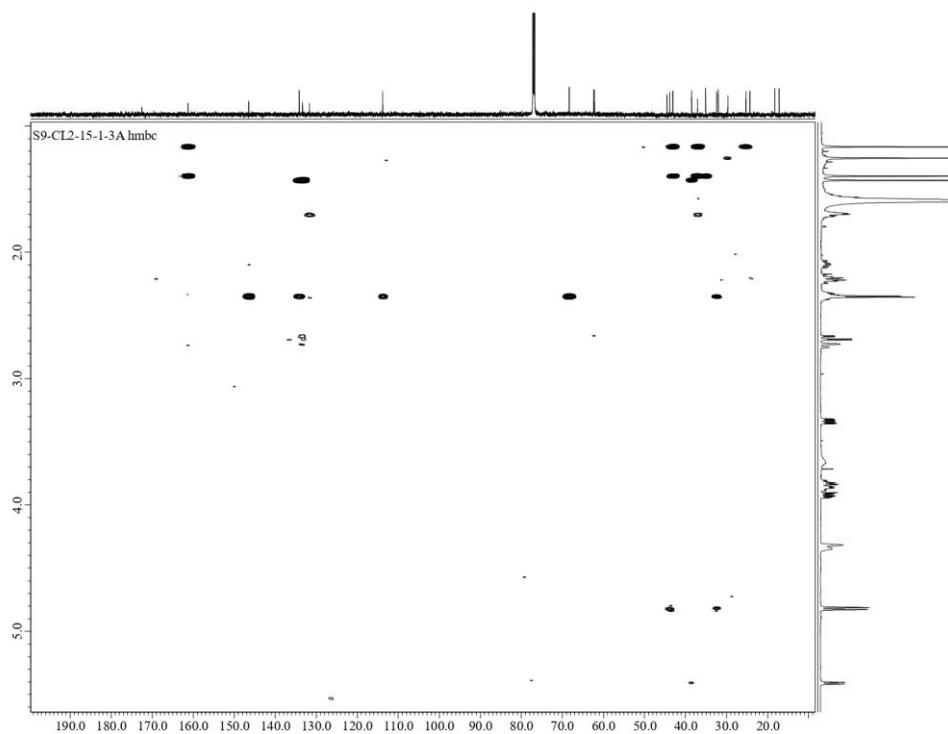


Figure S7. HMBC spectrum of compound **1** in CDCl_3

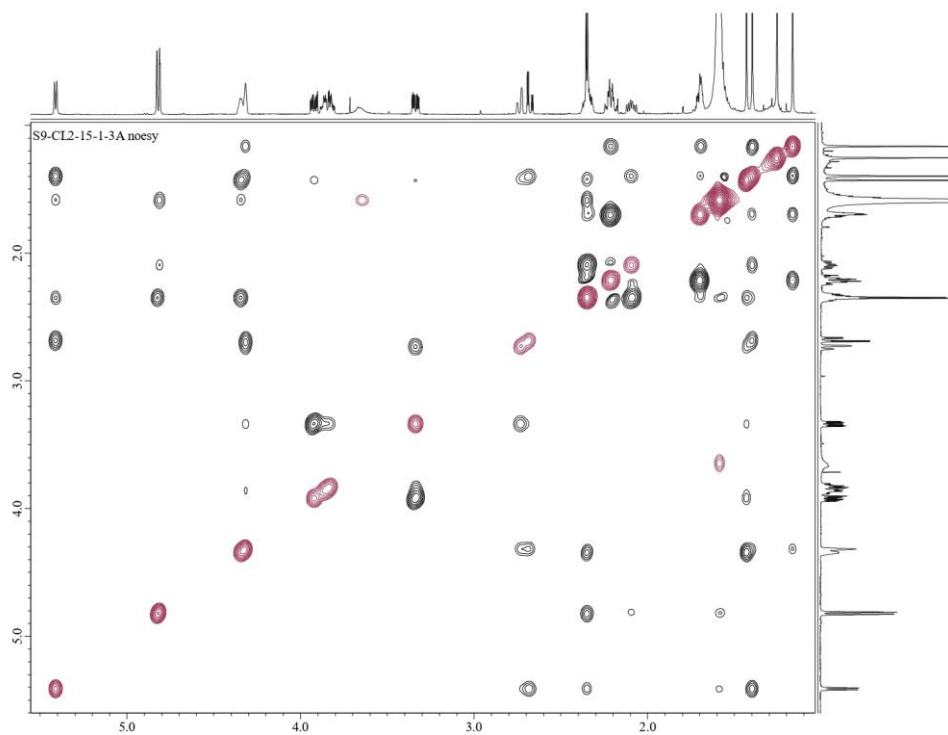


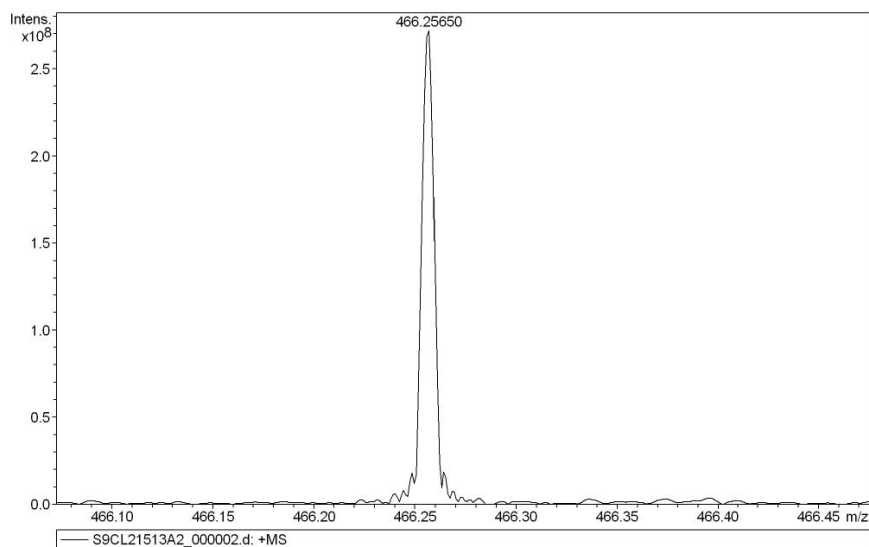
Figure S8. NOESY spectrum of compound **1** in CDCl_3

Mass Spectrum SmartFormula Report

Analysis Info

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 Sample Name S9-CL2-15-1-3A-2
 Comment ESI Positive

9/9/2021 12:39:49 PM
 Operator: YU HSIAO-CHING
 Instrument: BRUKER FT-MS solariX



Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	e ⁻ Conf	N-Rule
466.25650	1	C ₂₆ H ₃₇ NNaO ₅	100.00	466.25639	-0.11	-0.23	13.6	8.5	even	ok

Figure S9. HRESIMS of compound 1a

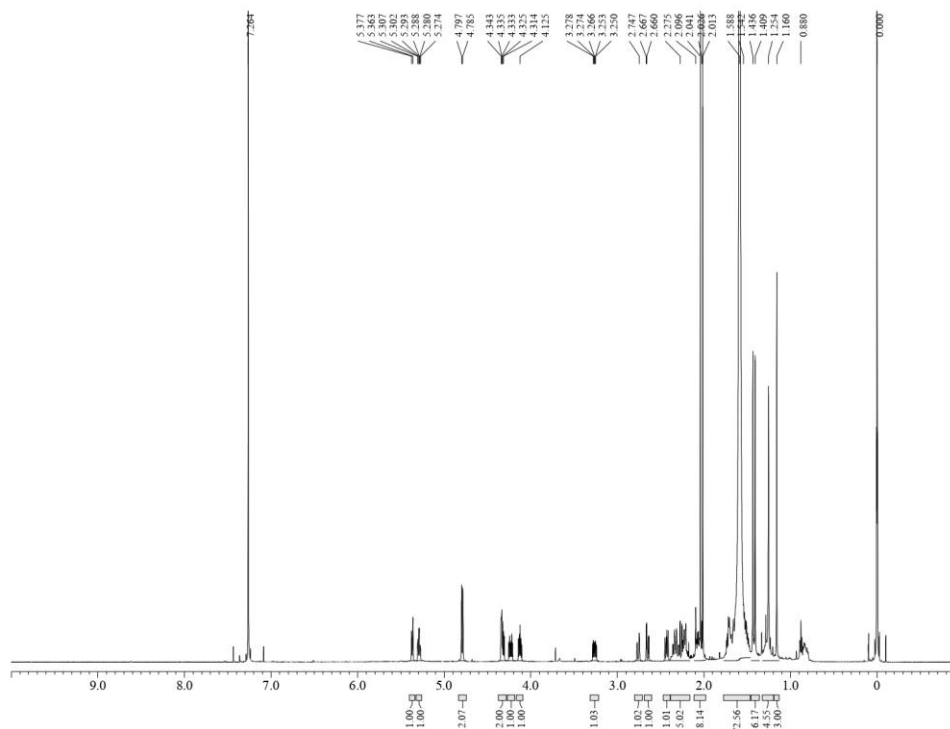


Figure S10. ¹H NMR spectrum (600MHz) of compound 1a in CDCl₃

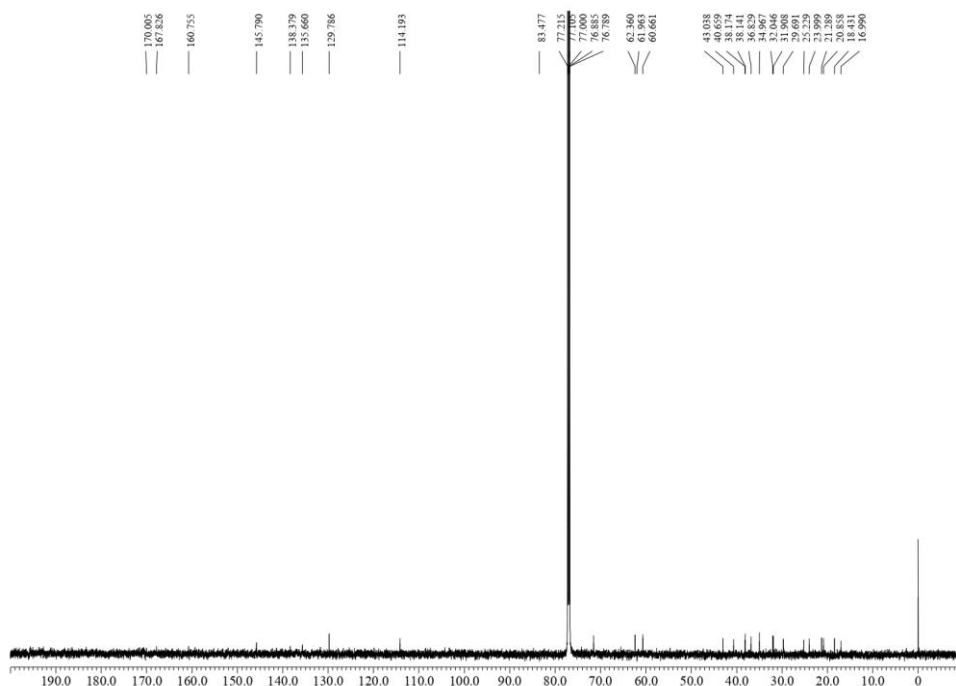
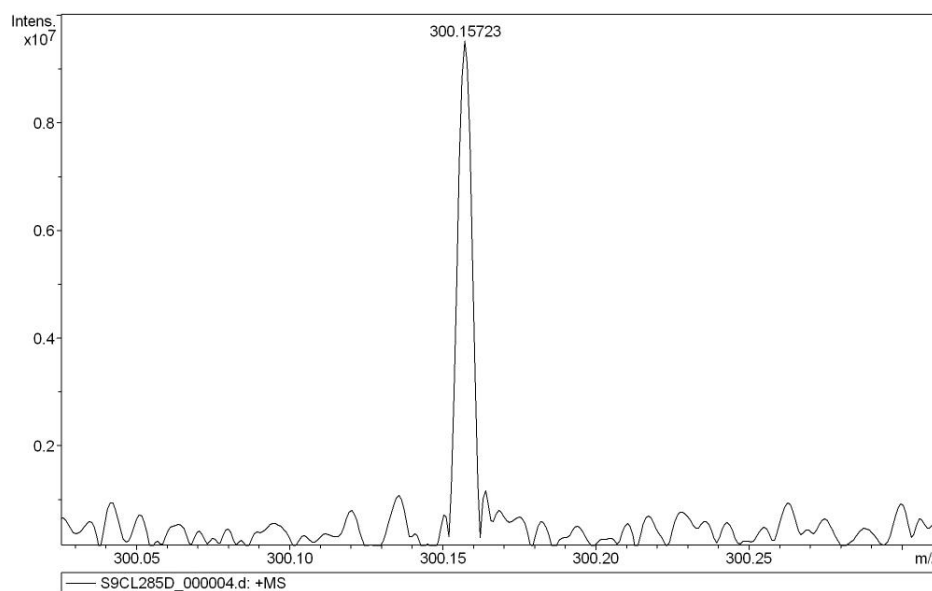


Figure S11. ^{13}C NMR spectrum (150 MHz) of compound **1a** in CDCl_3

Analysis Info

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 Method broadband first signal
 Sample Name S9-CL2-8-5D
 Comment ESI Positive

4/12/2021 5:11:11 PM
 Operator: YU HSIAO-CHING
 Instrument: BRUKER FT-MS solariX



Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	e ⁻ Conf	N-Rule
300.15723	1	C ₁₆ H ₂₃ NNaO ₃	100.00	300.15701	-0.22	-0.72	16.8	5.5	even	ok

Figure S12. HRESIMS of compound **2**

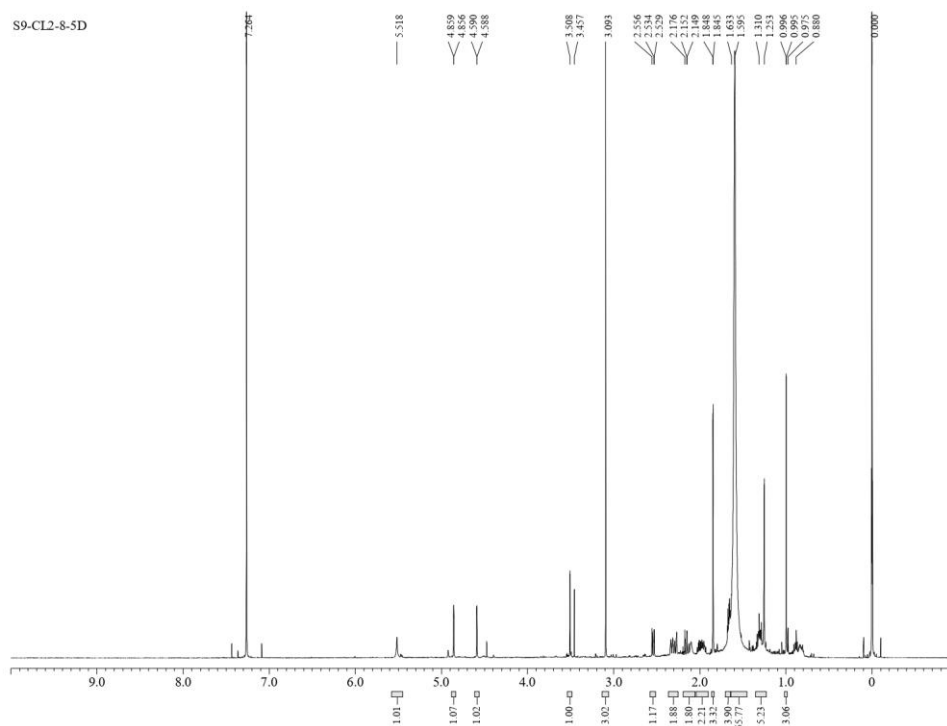


Figure S13. ^1H NMR spectrum (600MHz) of compound **2** in CDCl_3

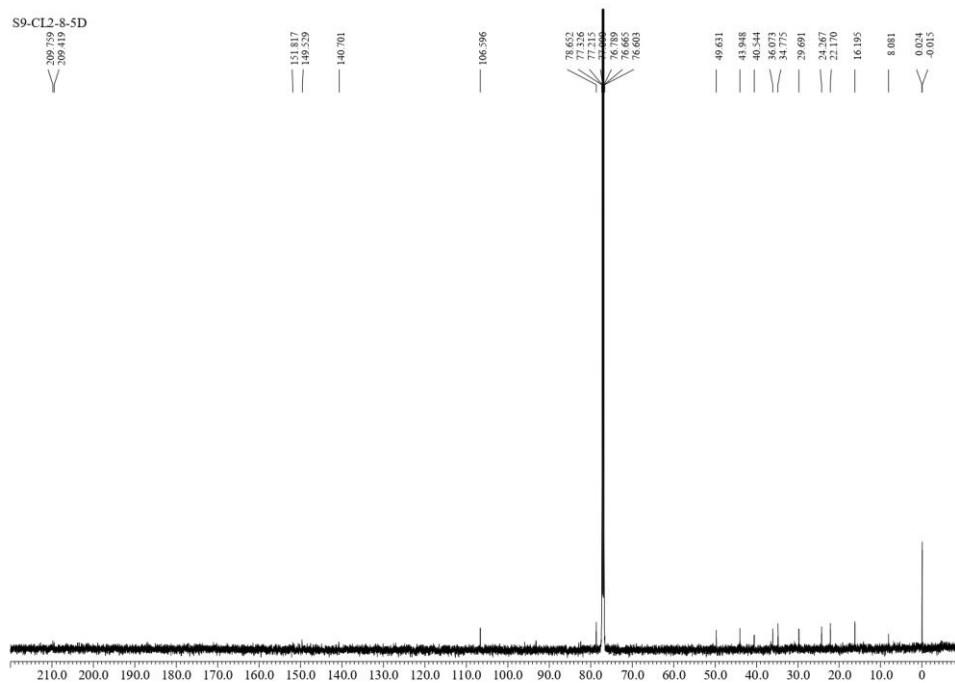
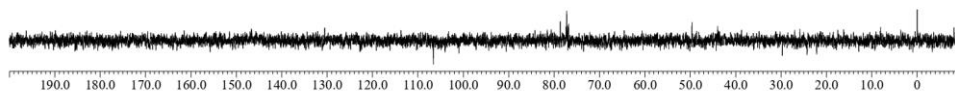


Figure S14. ^{13}C NMR spectrum (150 MHz) of compound **2** in CDCl_3

Y = 135[deg]
S9-CL2-8-5D



Y = 90[deg]
S9-CL2-8-5D

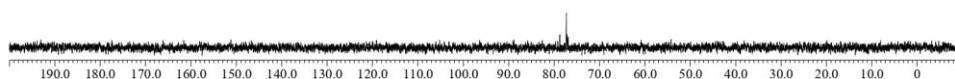


Figure S15. DEPT spectrum (150 MHz) of compound **2** in CDCl₃

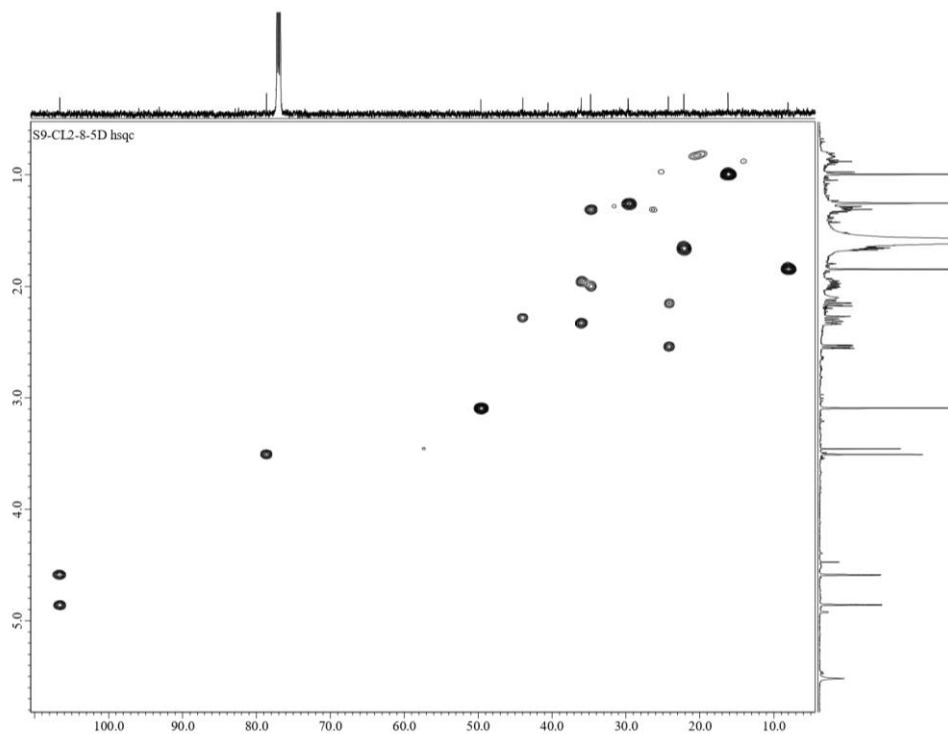


Figure S16. HSQC spectrum of compound **2** in CDCl₃

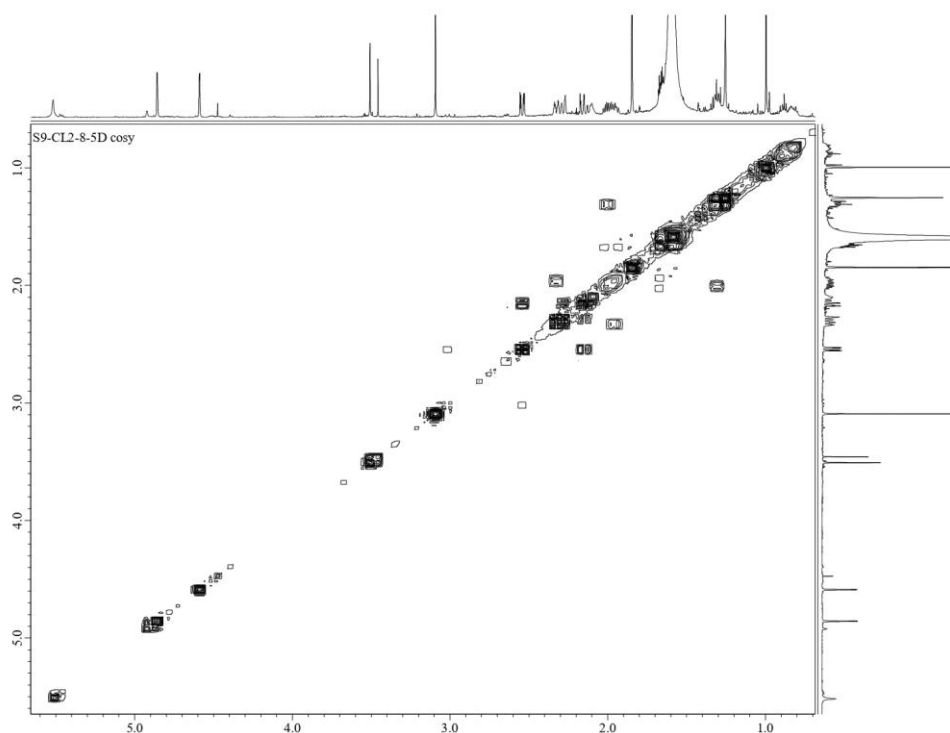


Figure S17. ^1H - ^1H COSY spectrum of compound **2** in CDCl_3

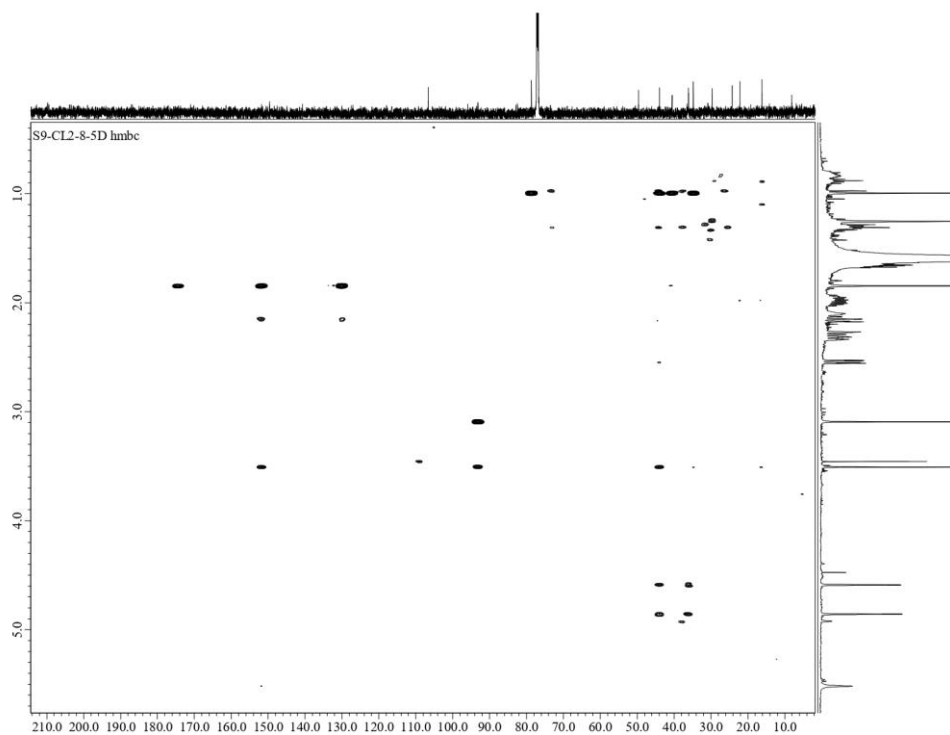


Figure S18. HMBC spectrum of compound **2** in CDCl_3

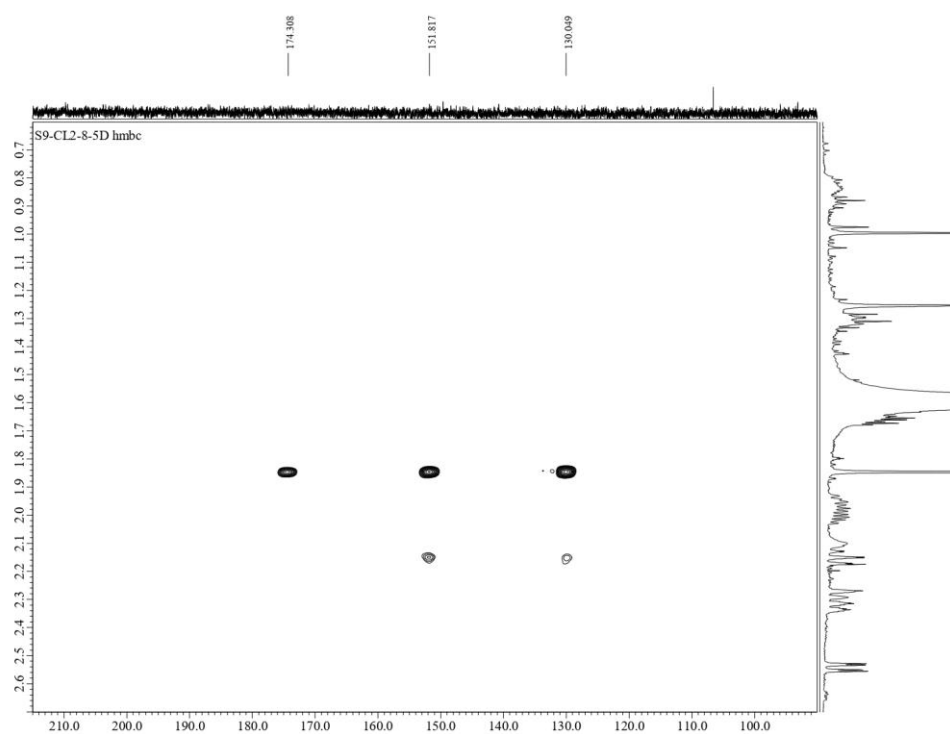


Figure S19. The amplificatory HMBC spectrum of compound **2** in CDCl_3

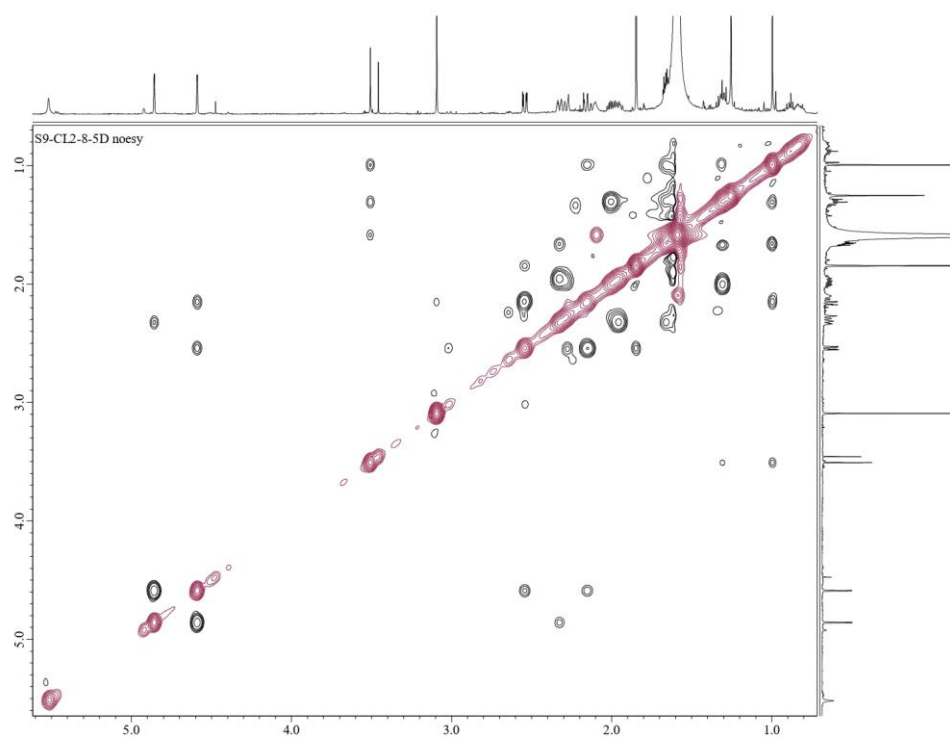
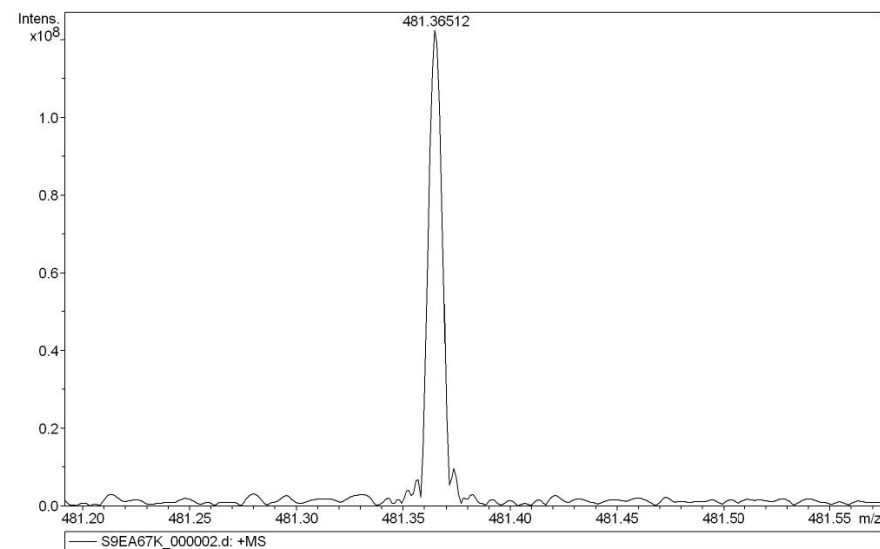


Figure S20. NOESY spectrum of compound **2** in CDCl_3

Analysis Info

Analysis Name D:\Data\1\S9EA67K_000002.d
 Method broadband first signal
 Sample Name S9-EA-6-7-K
 Comment ESI Positive

3/26/2021 11:55:00 AM
 Operator: YU HSIAO-CHING
 Instrument: BRUKER FT-MS solariX



Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	e ⁻ Conf	N-Rule
481.36512	1	C ₃₀ H ₅₀ NaO ₃	100.00	481.36522	0.09	0.20	7.9	5.5	even	ok

Figure S21. HRESIMS of compound 3

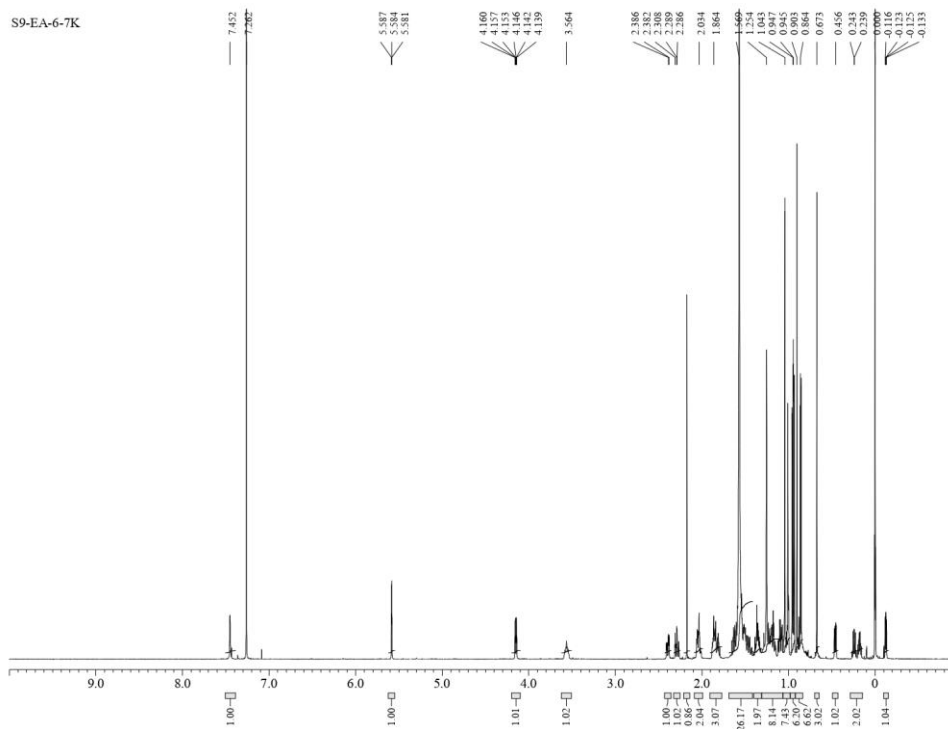


Figure S22. ¹H NMR spectrum (600MHz) of compound 3 in CDCl₃

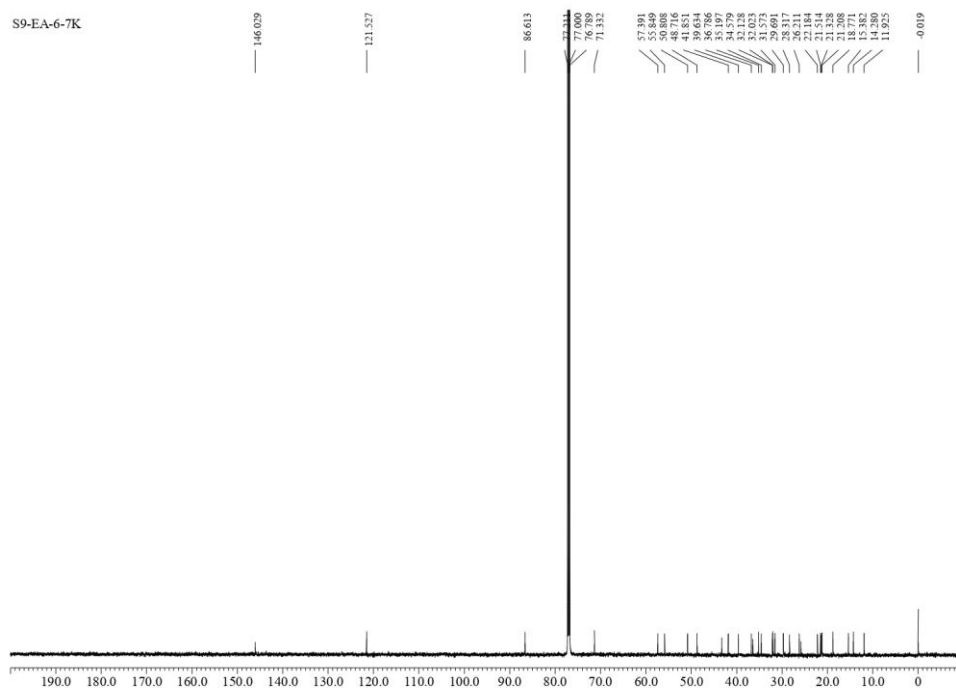


Figure S23. ^{13}C NMR spectrum (150 MHz) of compound **3** in CDCl_3

Y = 135[deg]
S9-EA-6-7K

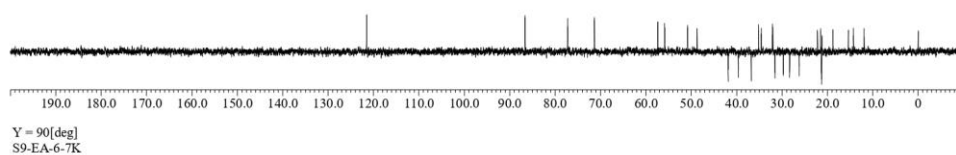


Figure S24. DEPT spectrum (150 MHz) of compound **3** in CDCl_3

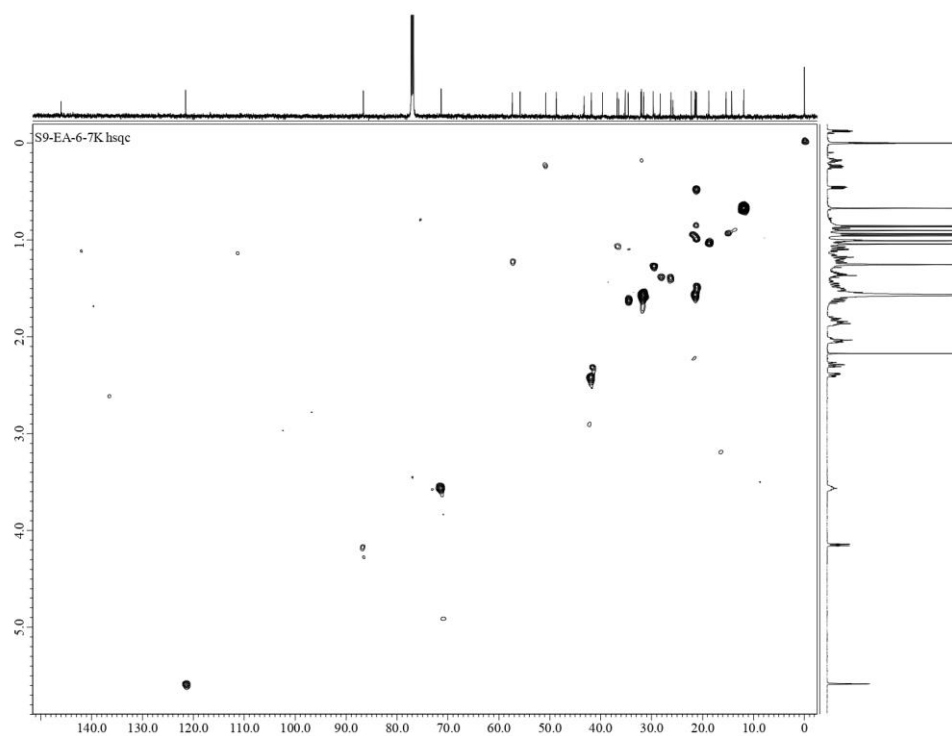


Figure S25. HSQC spectrum of compound **3** in CDCl₃

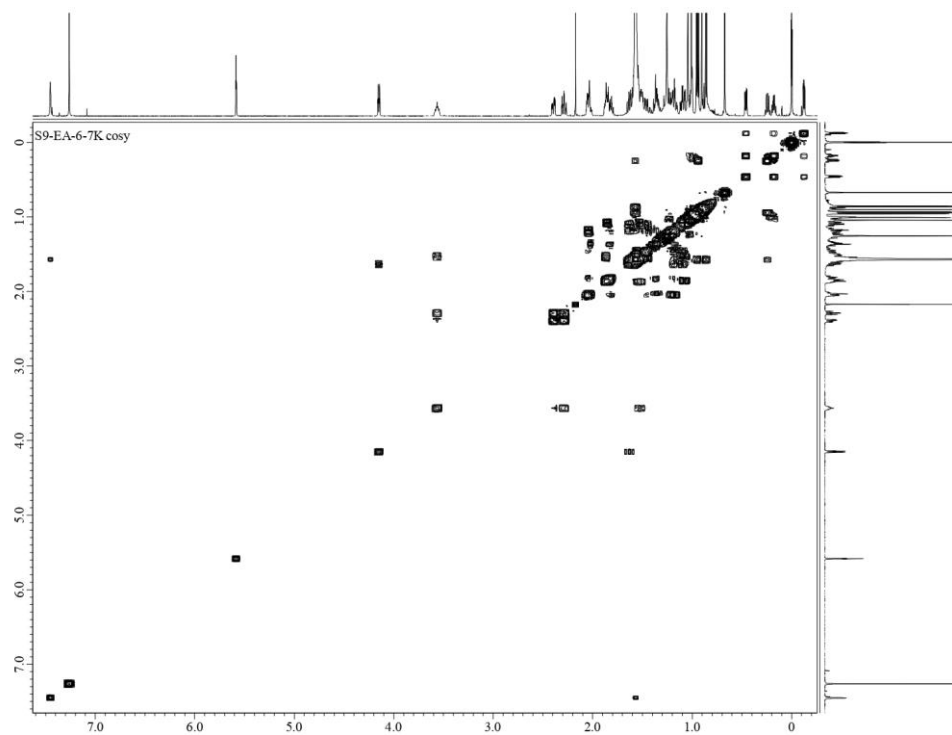


Figure S26. ¹H-¹H COSY spectrum of compound **3** in CDCl₃

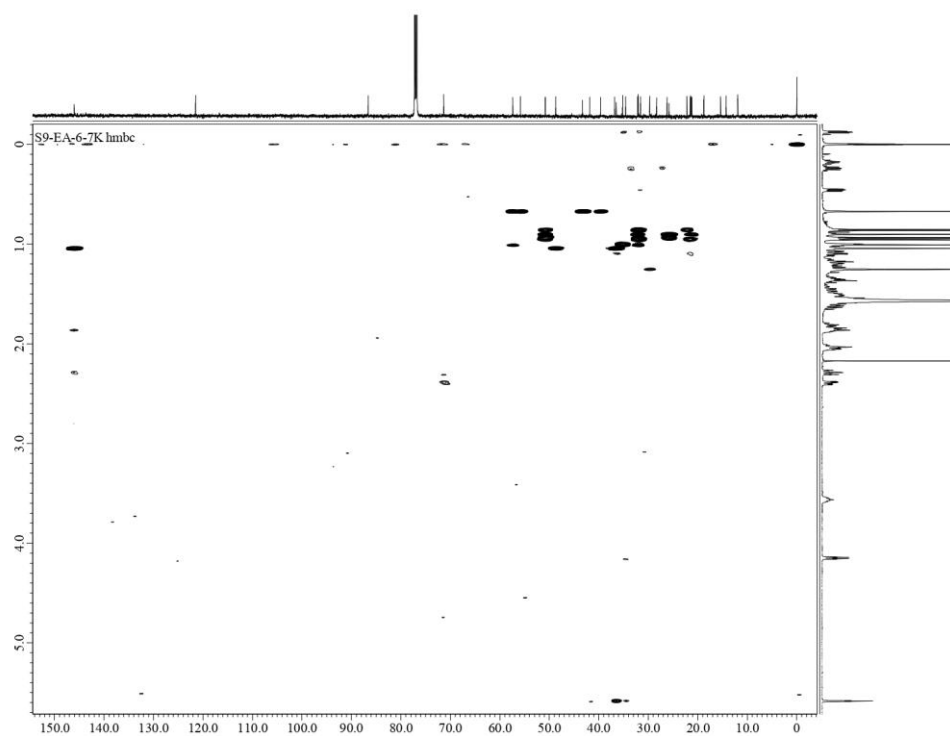


Figure S27. HMBC spectrum of compound **3** in CDCl_3

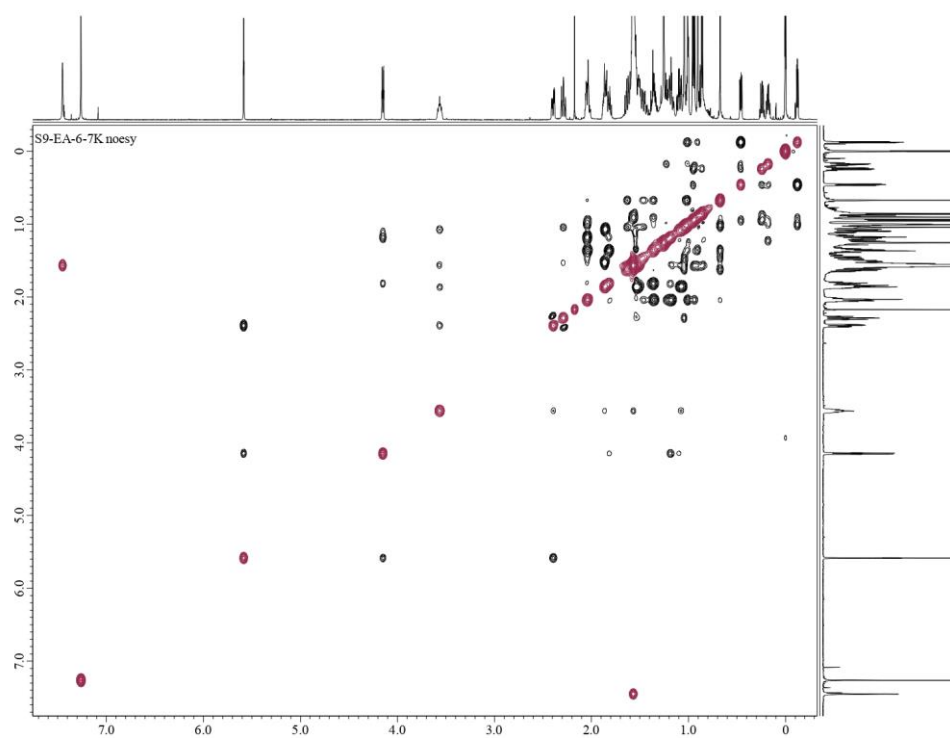
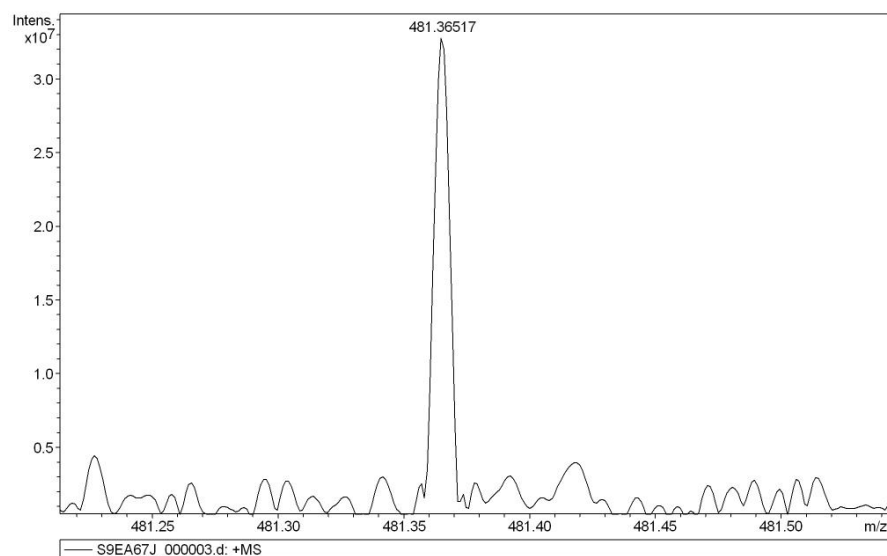


Figure S28. NOESY spectrum of compound **3** in CDCl_3

Analysis Info

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 Method broadband first signal
 Sample Name S9-EA-6-7-J
 Comment ESI Positive

3/26/2021 11:49:41 AM
 Operator: YU HSIAO-CHING
 Instrument: BRUKER FT-MS solariX



Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	e ⁻ Conf	N-Rule
481.36517	1	C ₃₀ H ₅₀ NaO ₃	100.00	481.36522	0.05	0.10	37.5	5.5	even	ok

Figure S29. HRESIMS of compound **4**

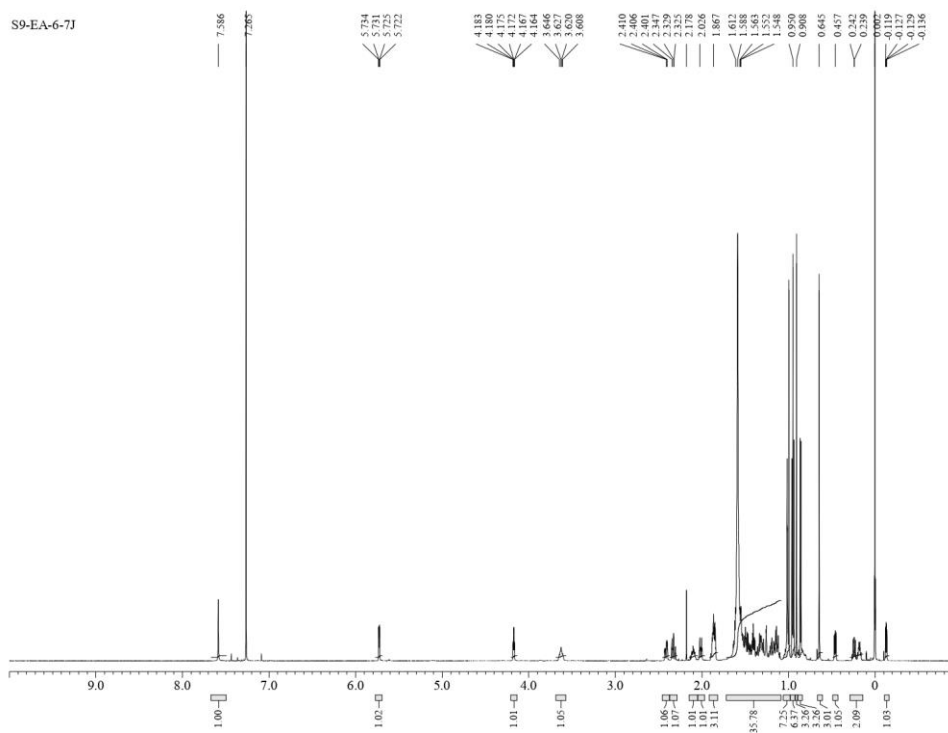


Figure S30. ¹H NMR spectrum (600MHz) of compound **4** in CDCl₃

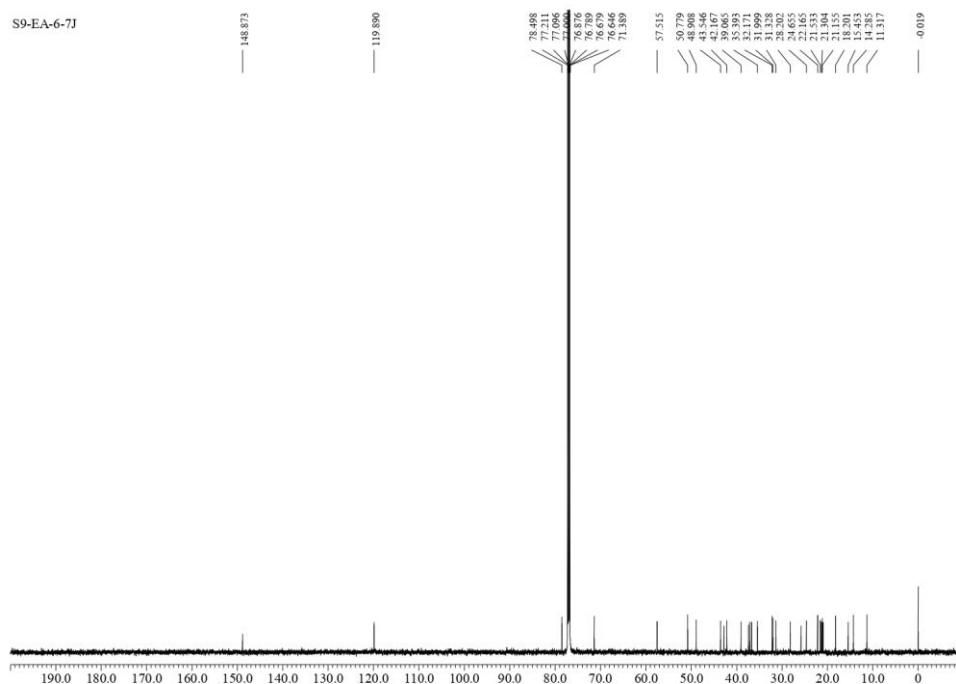


Figure S31. ^{13}C NMR spectrum (150 MHz) of compound **4** in CDCl_3

Y = 135[deg]
S9-EA-6-7J

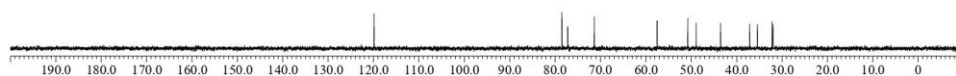
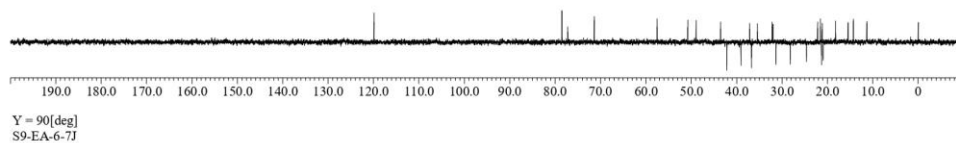


Figure S32. DEPT spectrum (150 MHz) of compound **4** in CDCl_3

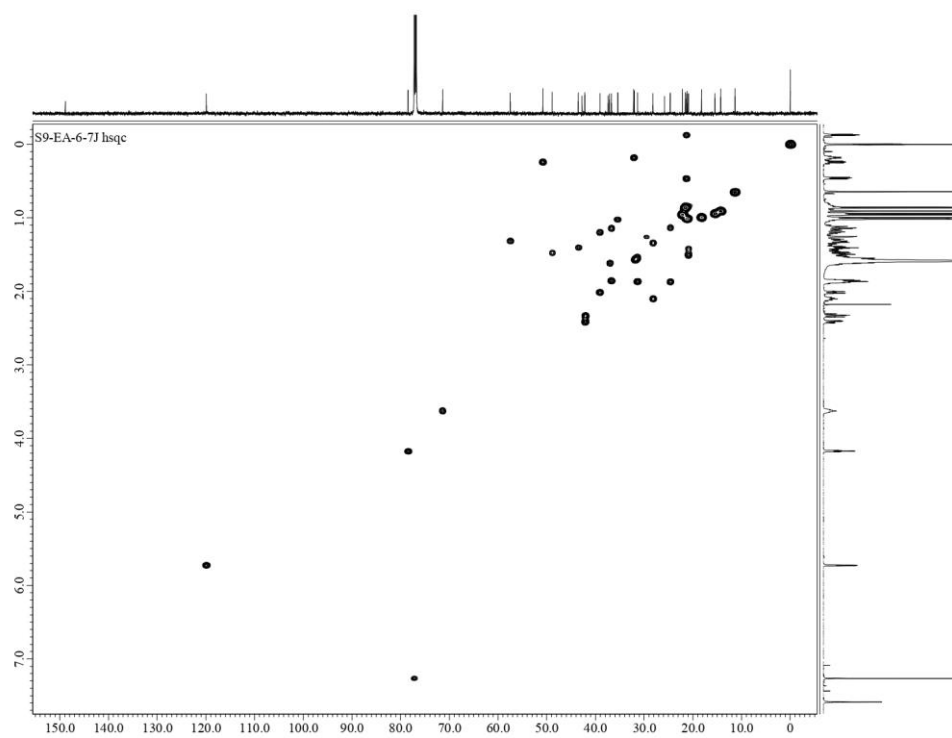


Figure S33. HSQC spectrum of compound **4** in CDCl₃

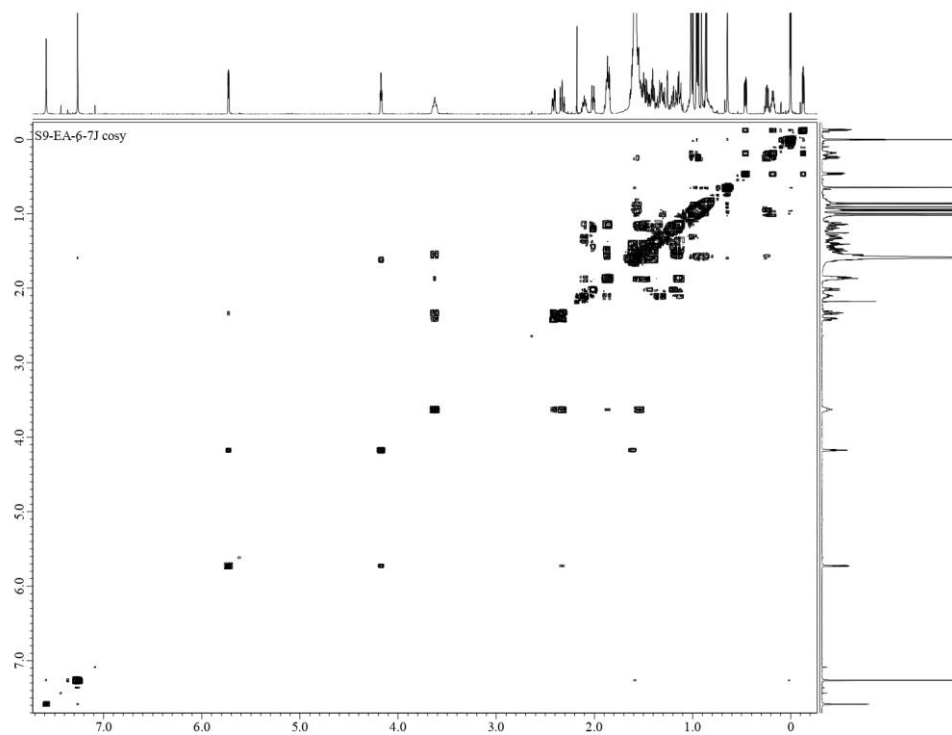


Figure S34. ¹H-¹H COSY spectrum of compound **4** in CDCl₃

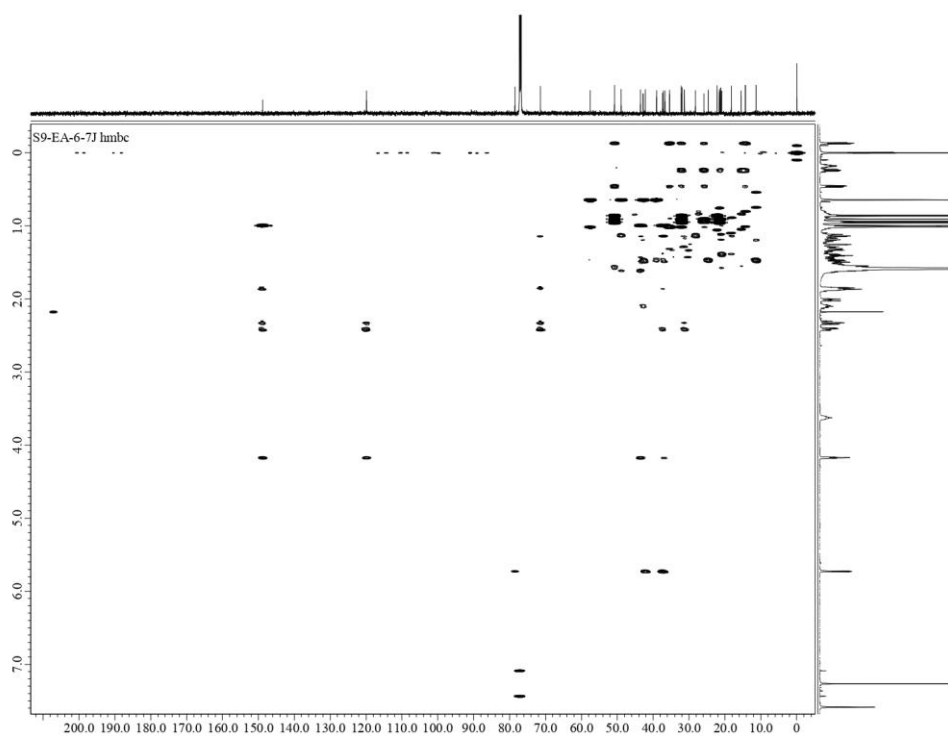


Figure S35. HMBC spectrum of compound **4** in CDCl₃

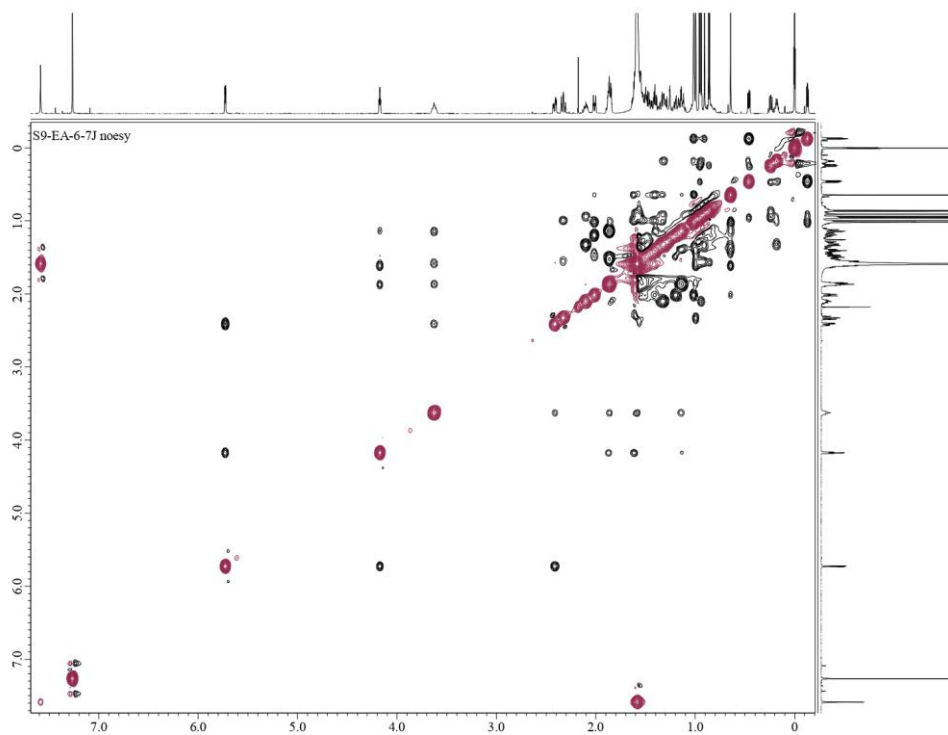
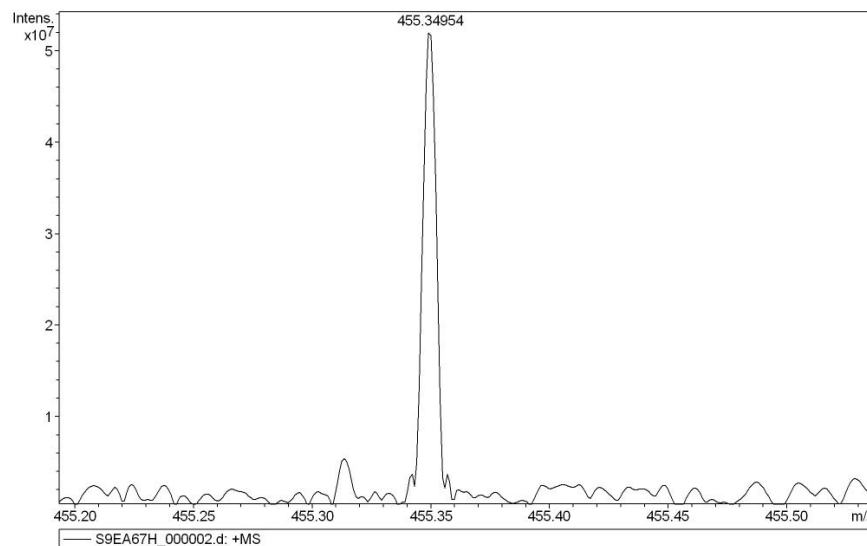


Figure S36. NOESY spectrum of compound **4** in CDCl₃

Analysis Info

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Method broadband first signal
Sample Name S9-EA-6-7-H
Comment ESI Positive

3/26/2021 11:35:26 AM
Operator: YU HSIAO-CHING
Instrument: BRUKER FT-MS solariX



Meas. m/z	#	Formula	Score	m/z	err [mDa]	err [ppm]	mSigma	rdb	e ⁻ Conf	N-Rule
455.34954	1	C ₂₈ H ₄₈ NaO ₃	100.00	455.34957	0.02	0.05	36.2	4.5	even	ok

Figure S37. HRESIMS of compound **5**

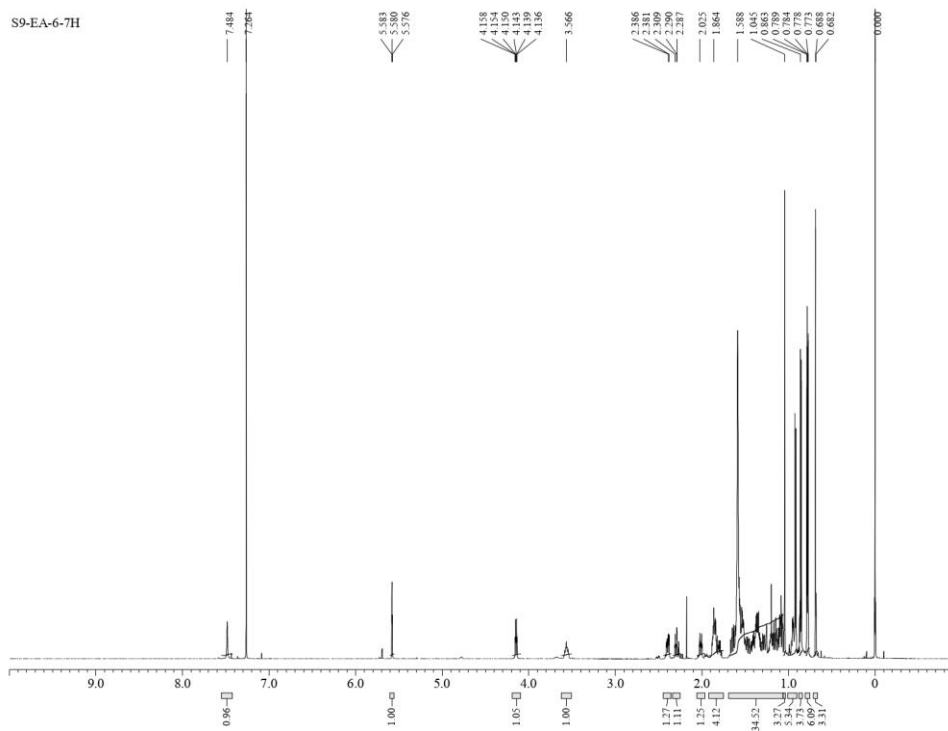


Figure S38. ¹H NMR spectrum (600MHz) of compound **5** in CDCl₃

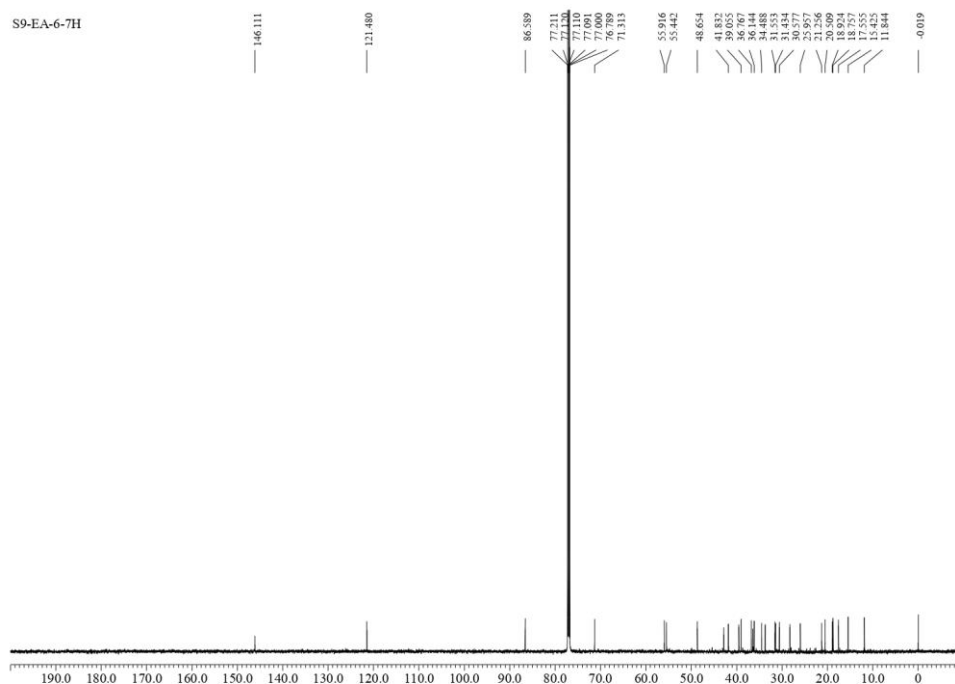


Figure S39. ^{13}C NMR spectrum (150 MHz) of compound **5** in CDCl_3

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S9-EA-6-7H

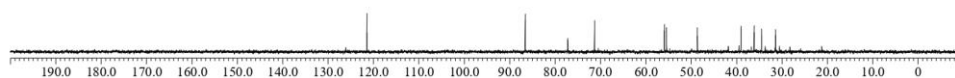
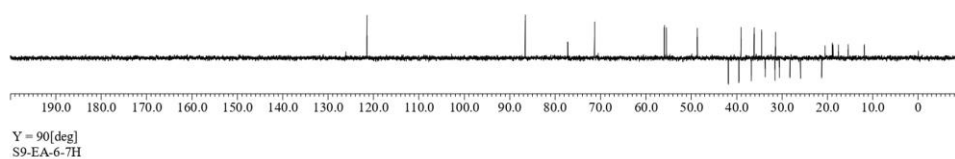


Figure S40. DEPT spectrum (150 MHz) of compound **5** in CDCl_3

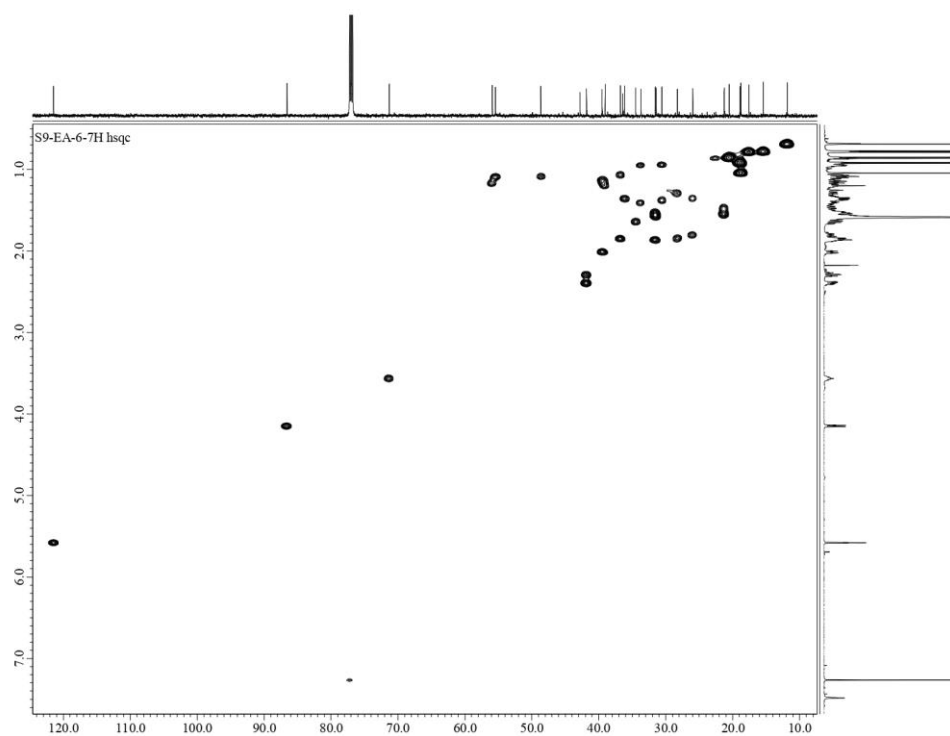


Figure S41. HSQC spectrum of compound **5** in CDCl₃

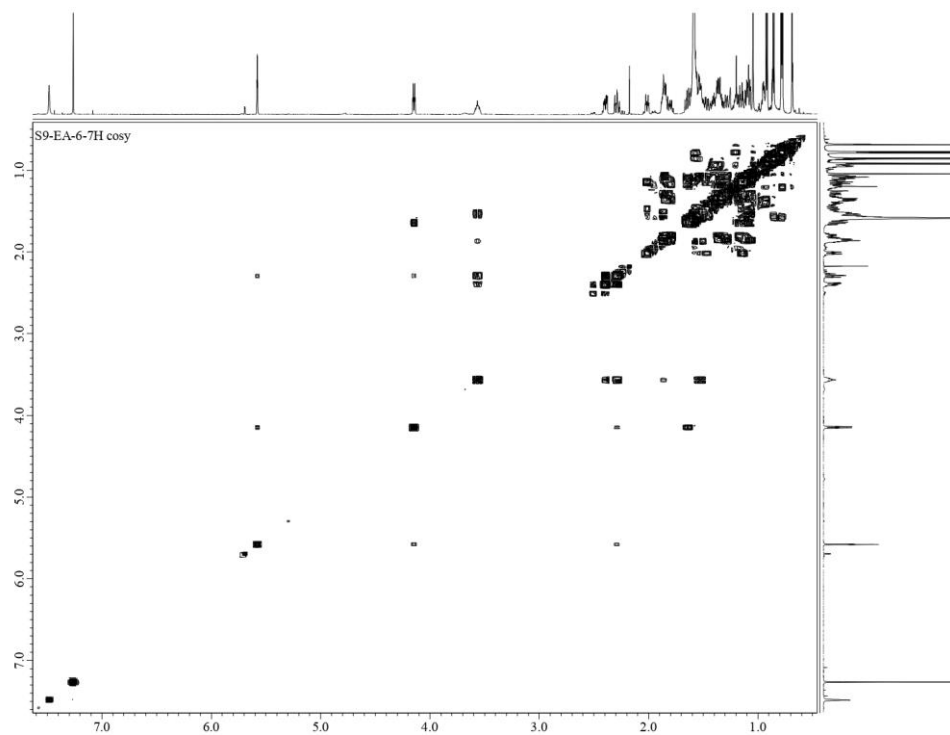


Figure S42. ¹H-¹H COSY spectrum of compound **5** in CDCl₃

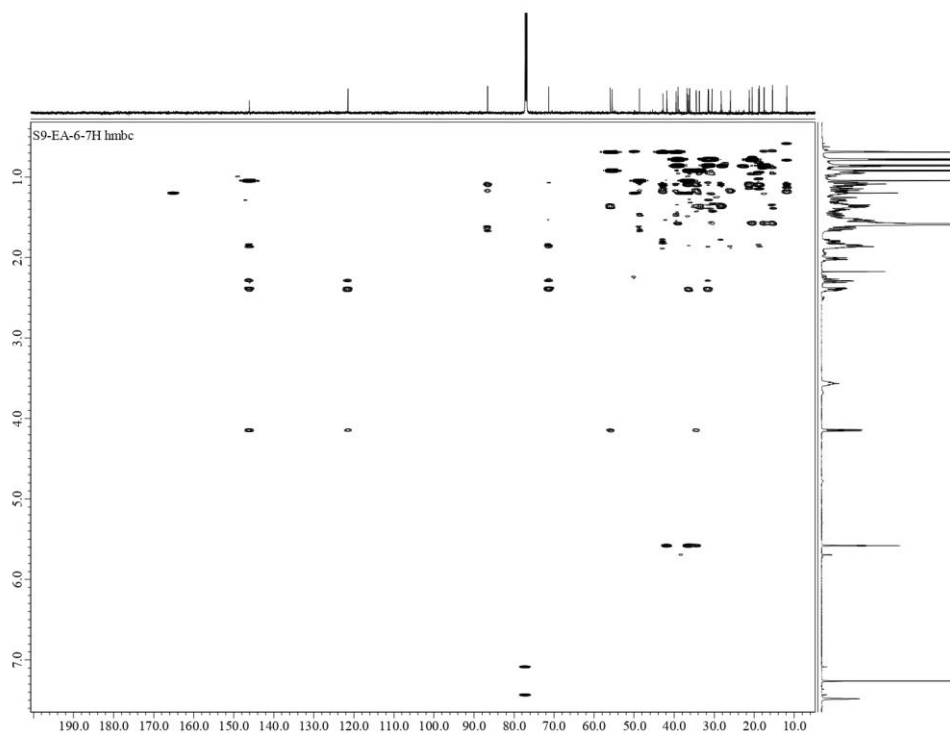


Figure S43. HMBC spectrum of compound **5** in CDCl_3

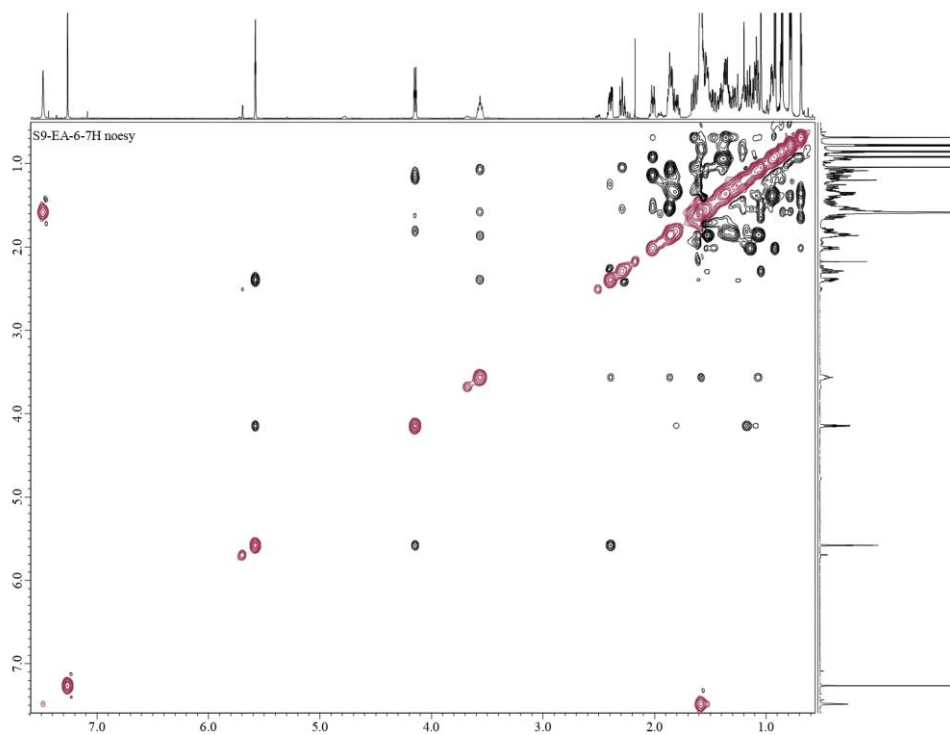


Figure S44. NOESY spectrum of compound **5** in CDCl_3

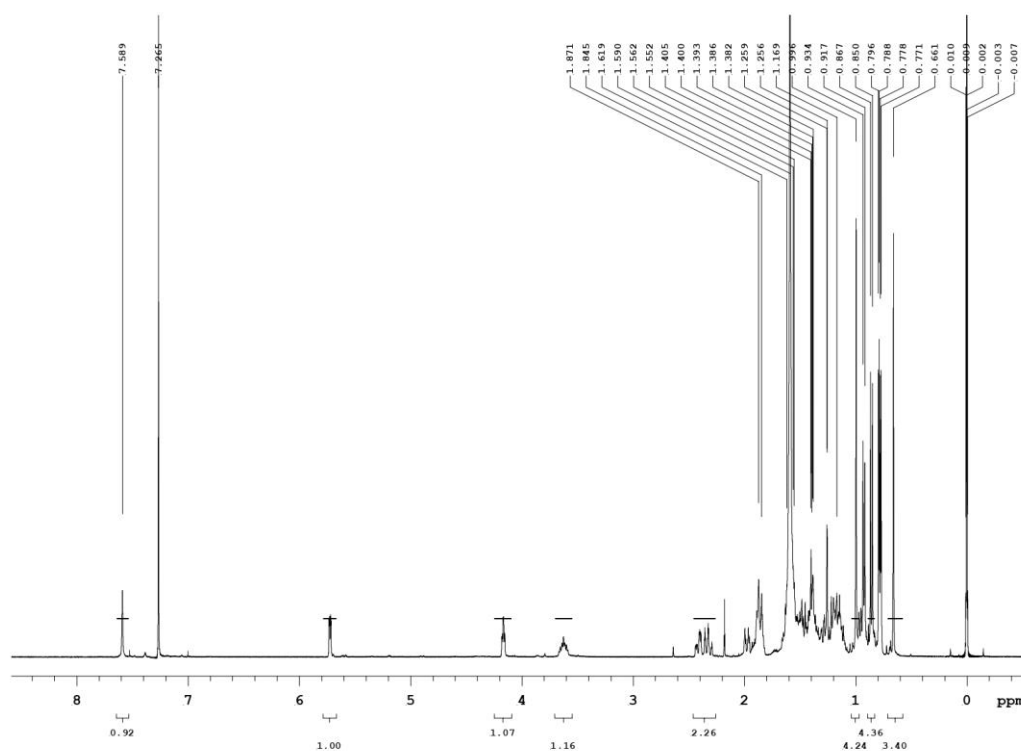


Figure S45. ^1H NMR spectrum (400MHz) of compound **6** in CDCl_3

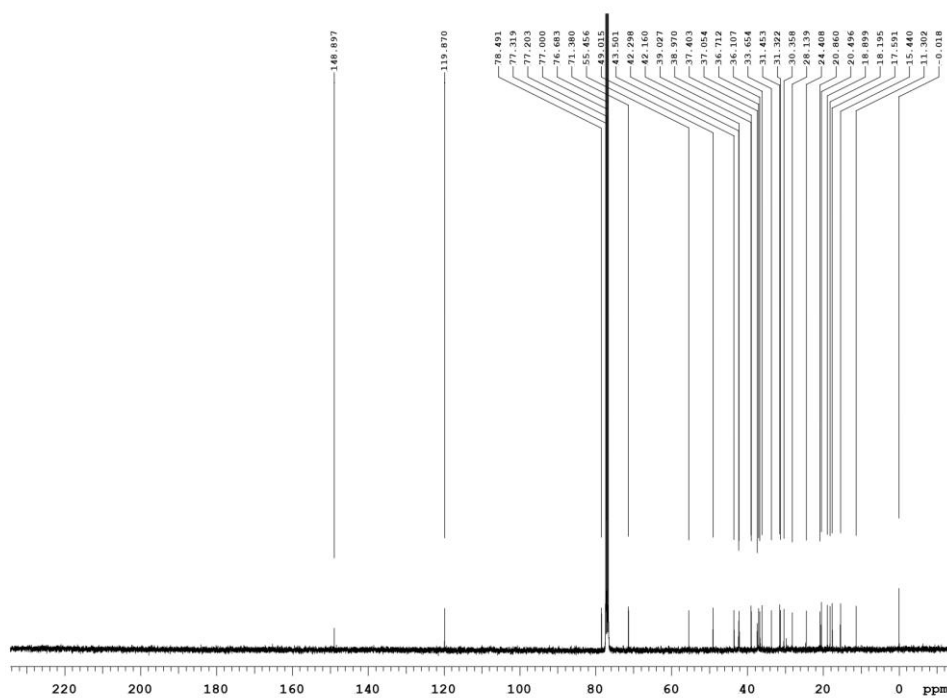


Figure S46. ^{13}C NMR spectrum (100 MHz) of compound **6** in CDCl_3

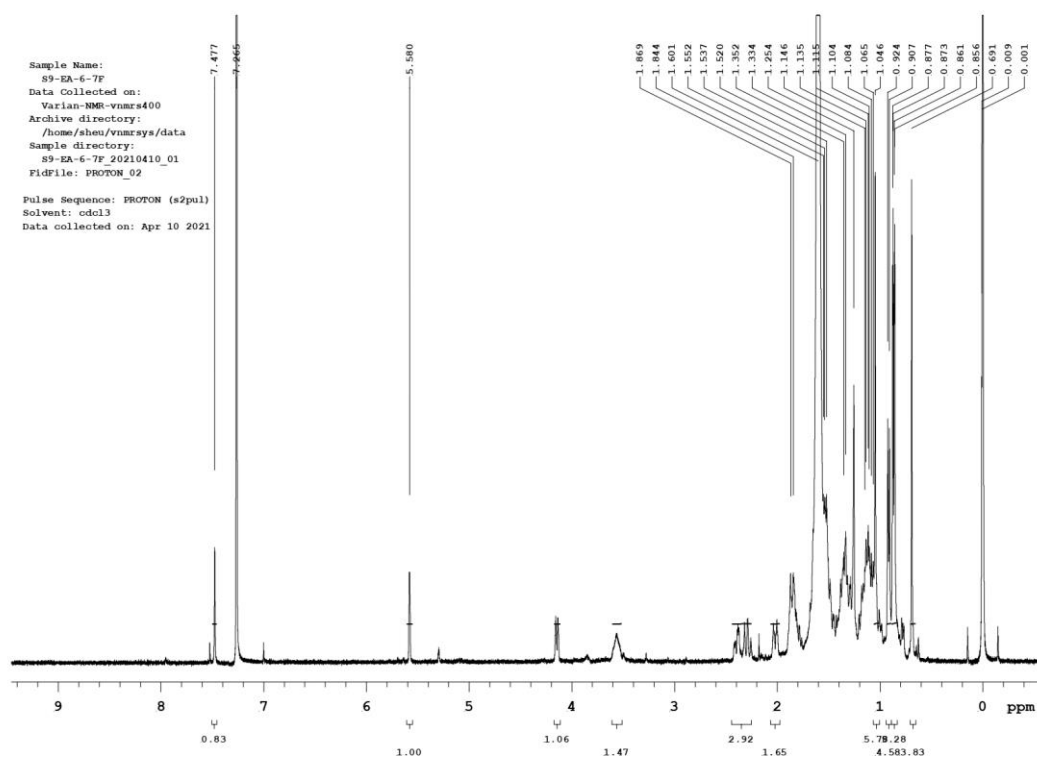


Figure S47. ^1H NMR spectrum (400MHz) of compound **7** in CDCl_3

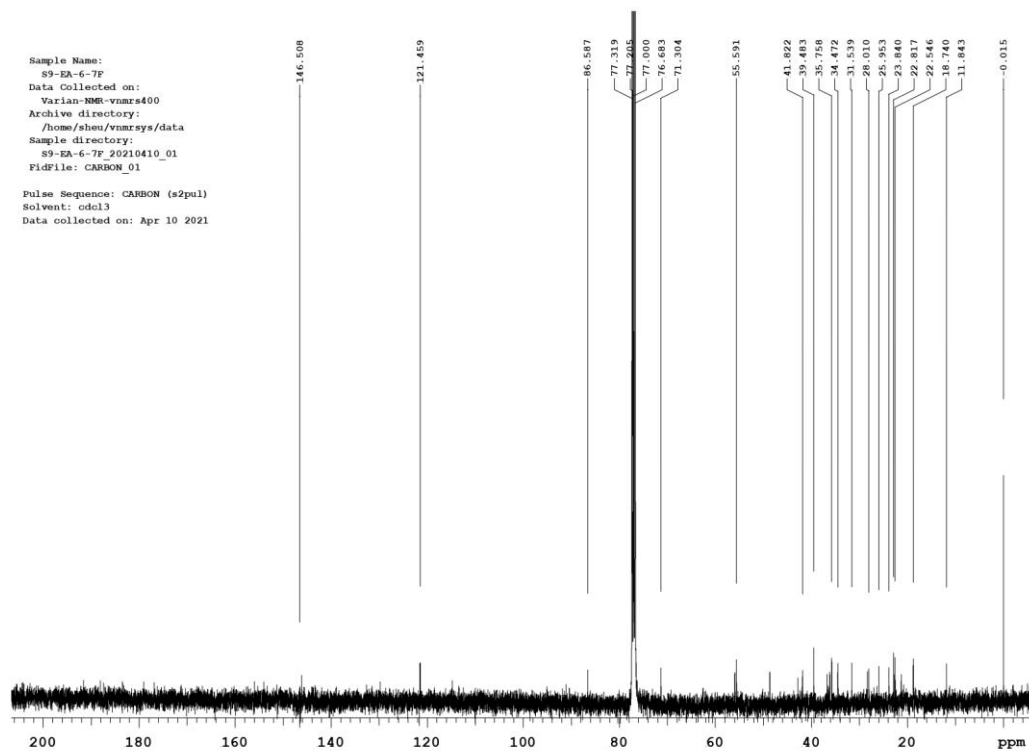


Figure S48. ^{13}C NMR spectrum (100 MHz) of compound **7** in CDCl_3

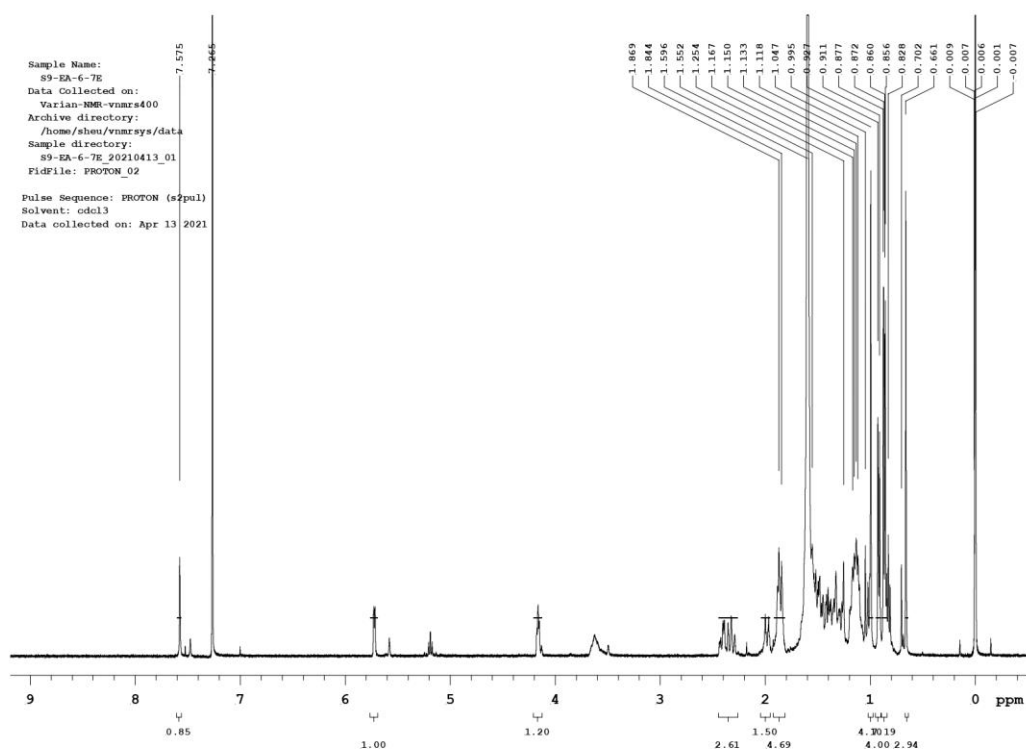


Figure S49. ^1H NMR spectrum (400MHz) of compound **8** in CDCl_3

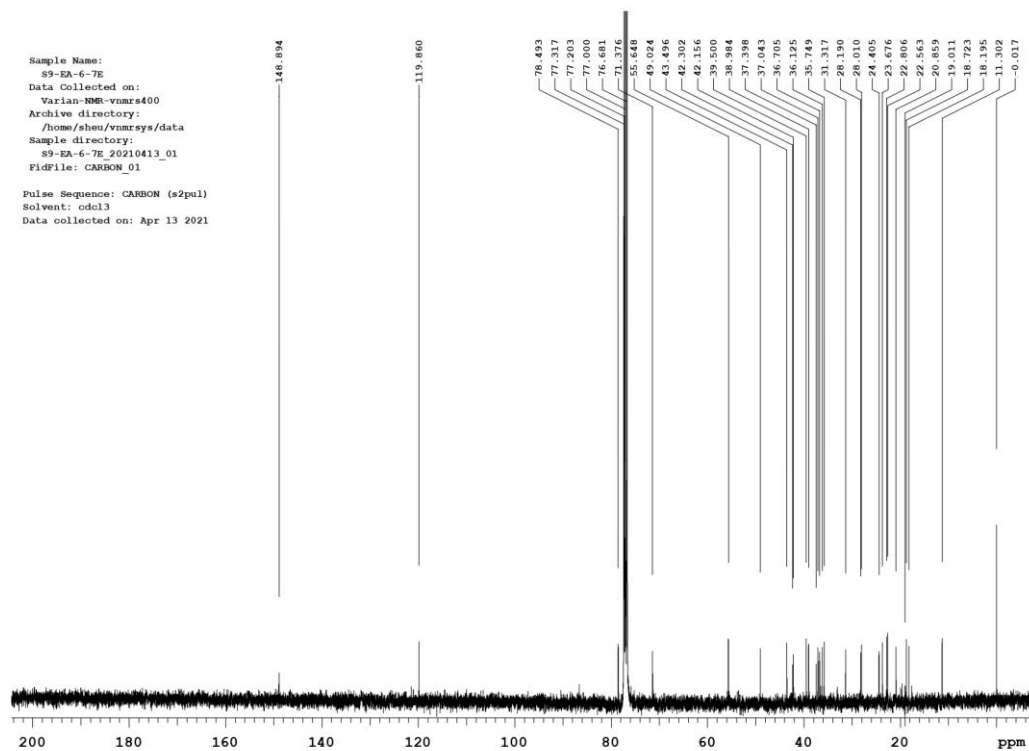


Figure S50. ^{13}C NMR spectrum (100 MHz) of compound **8** in CDCl_3

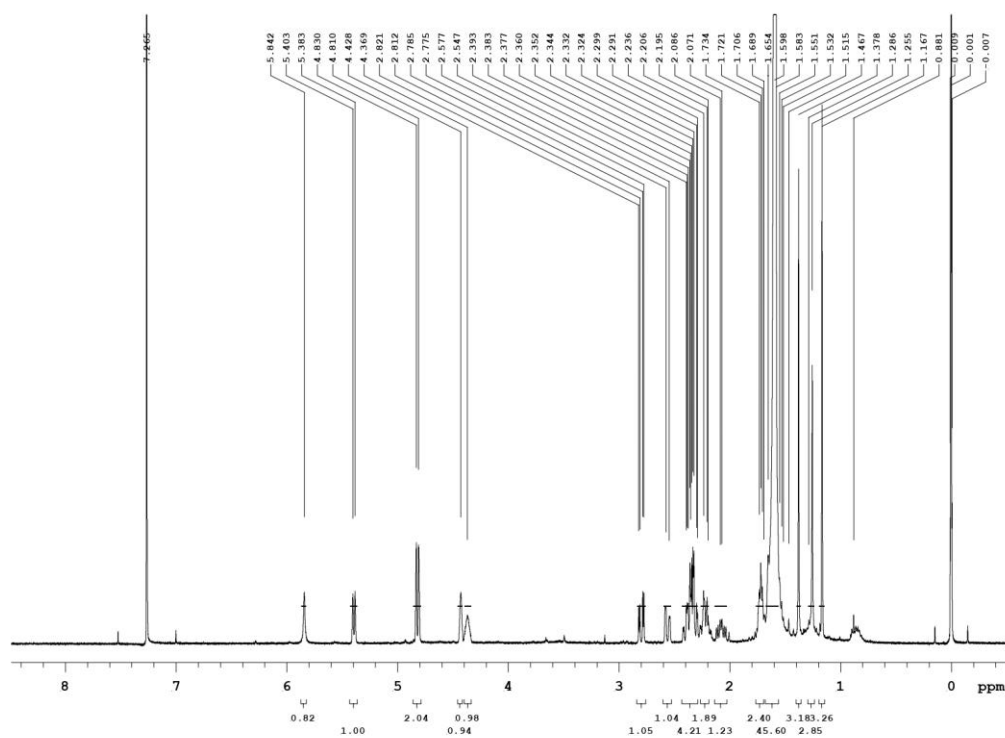


Figure S51. ^1H NMR spectrum (400MHz) of compound **9** in CDCl_3

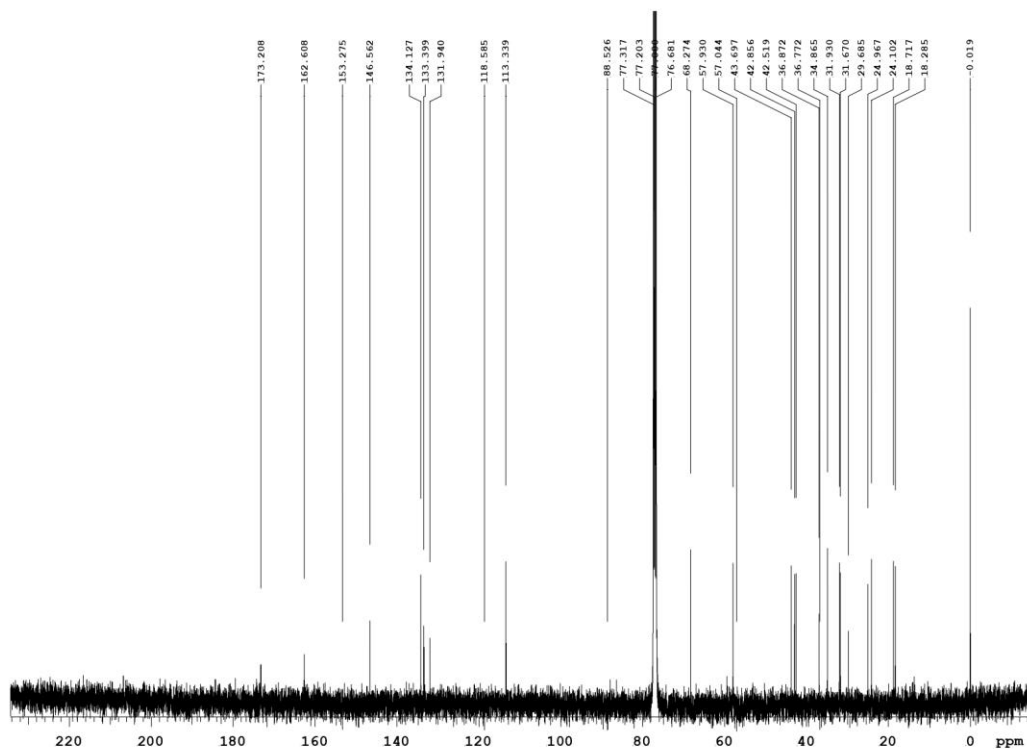


Figure S52. ^{13}C NMR spectrum (100 MHz) of compound **9** in CDCl_3

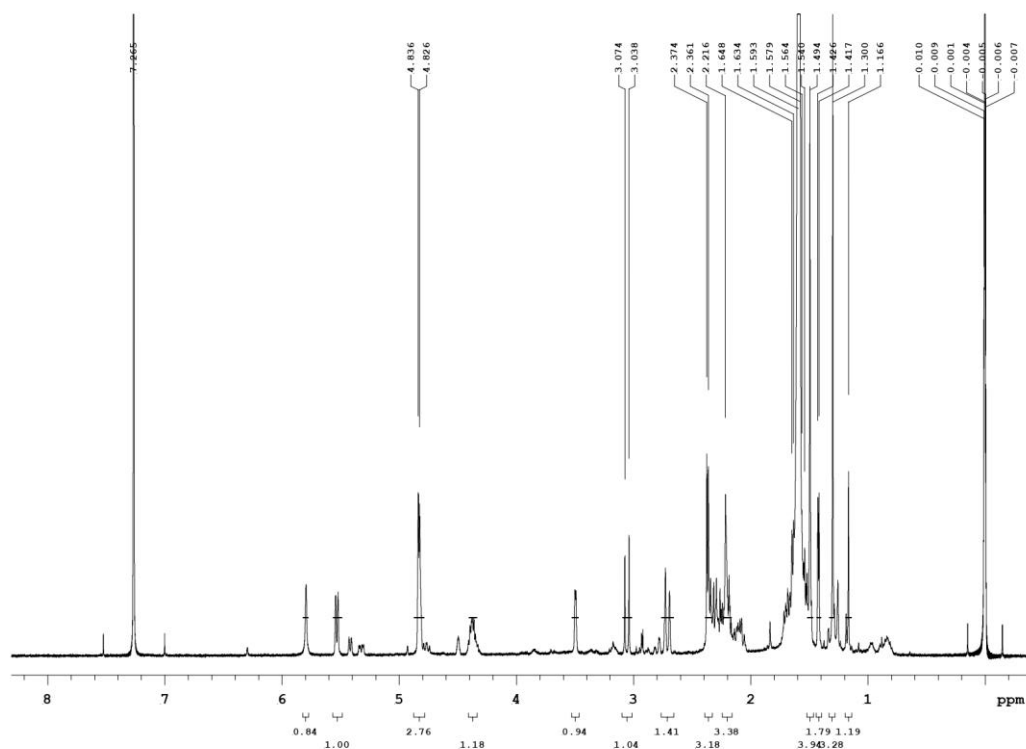


Figure S53. ¹H NMR spectrum (400MHz) of compound **10** in CDCl₃

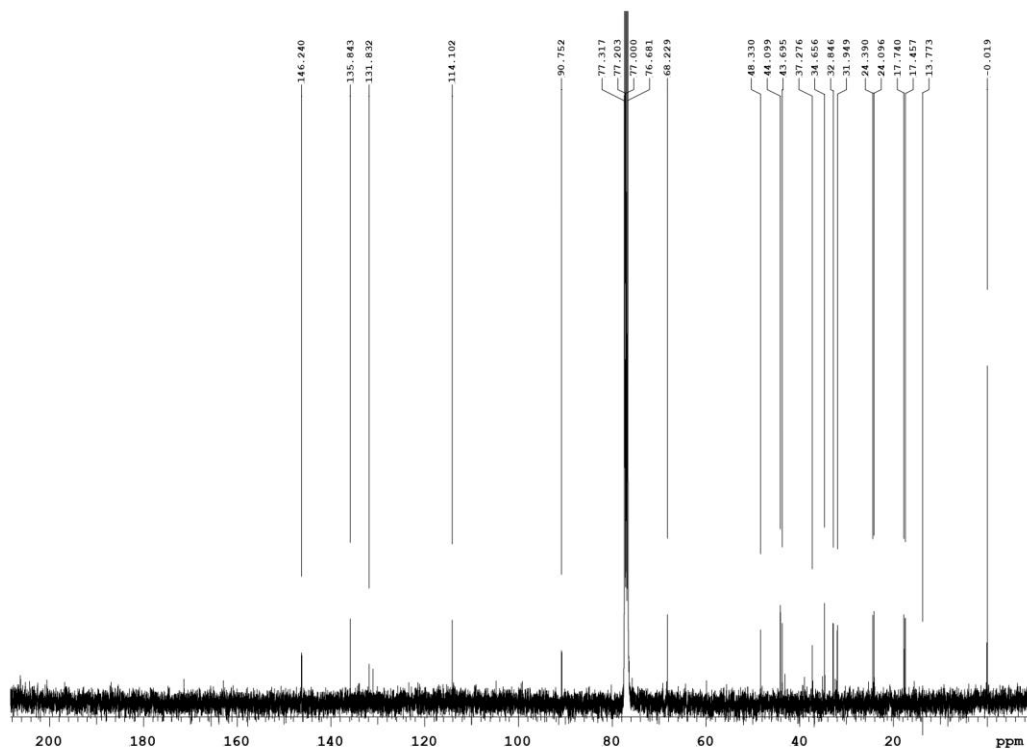


Figure S54. ¹³C NMR spectrum (100 MHz) of compound **10** in CDCl₃

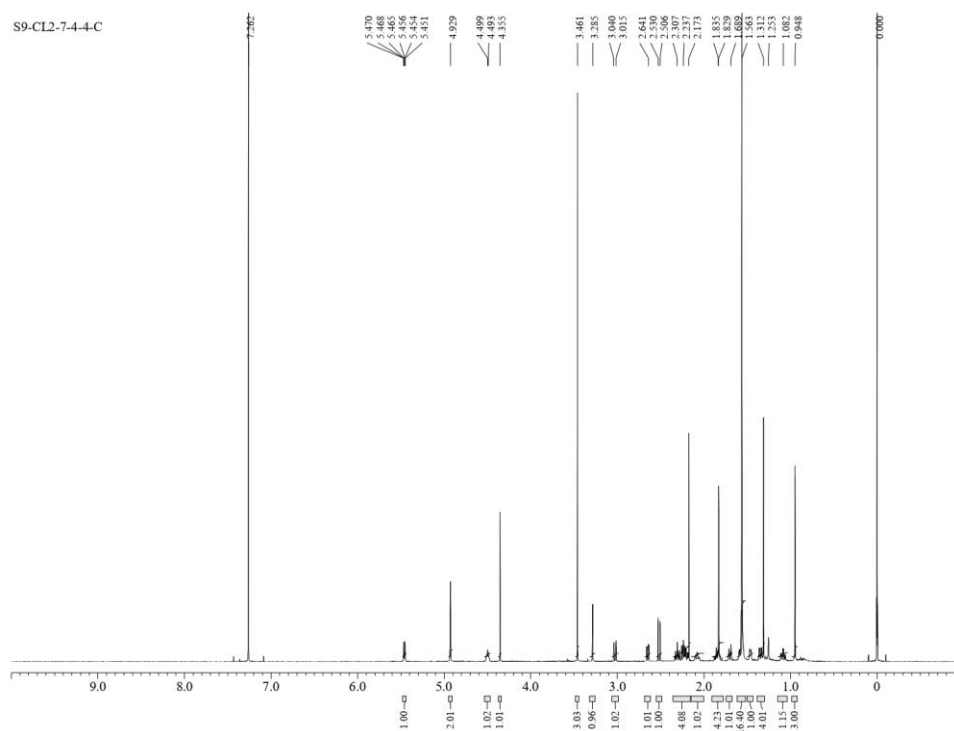


Figure S55. ^1H NMR spectrum (600MHz) of compound **11** in CDCl_3

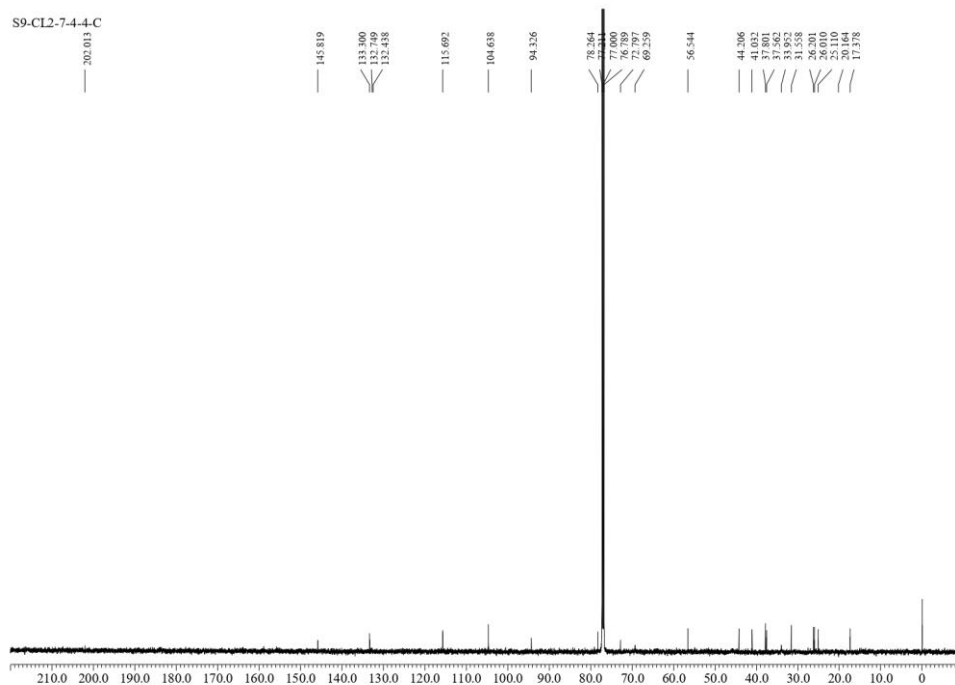


Figure S56. ^{13}C NMR spectrum (150 MHz) of compound **11** in CDCl_3

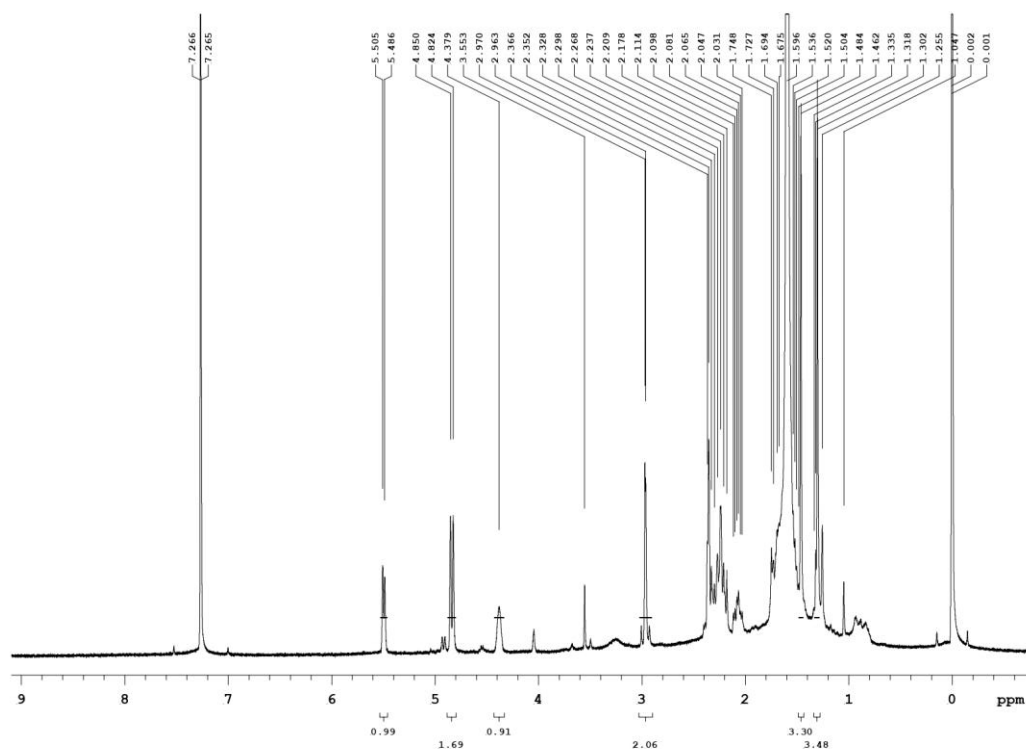


Figure S57. ¹H NMR spectrum (400MHz) of compound **12** in CDCl₃

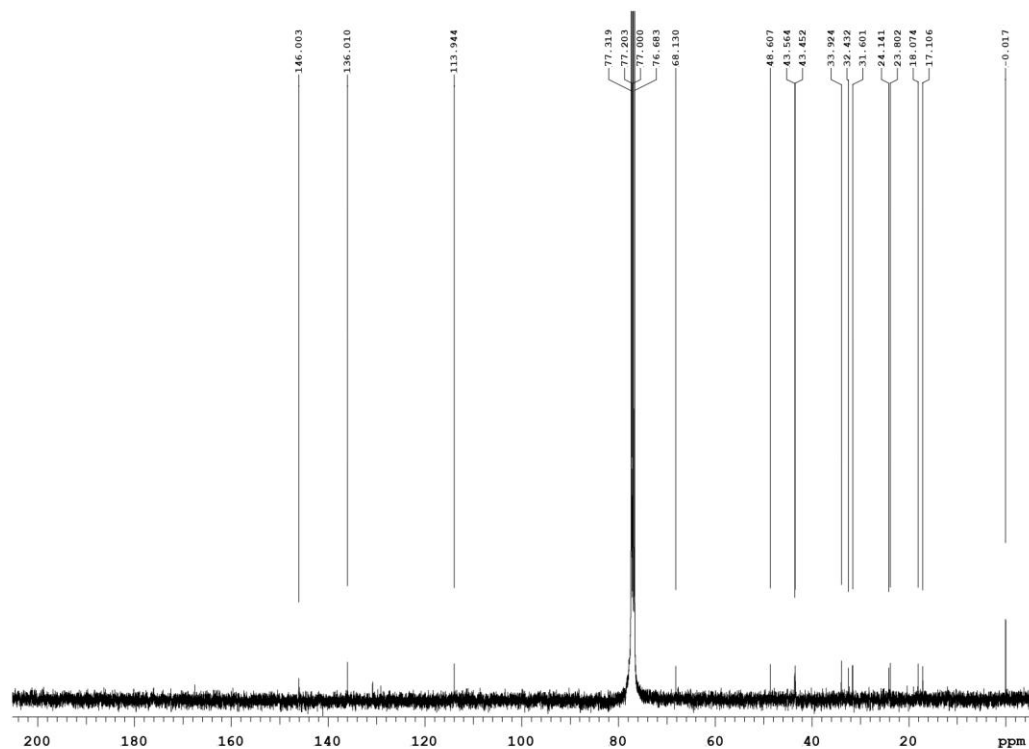


Figure S58. ¹³C NMR spectrum (100 MHz) of compound **12** in CDCl₃

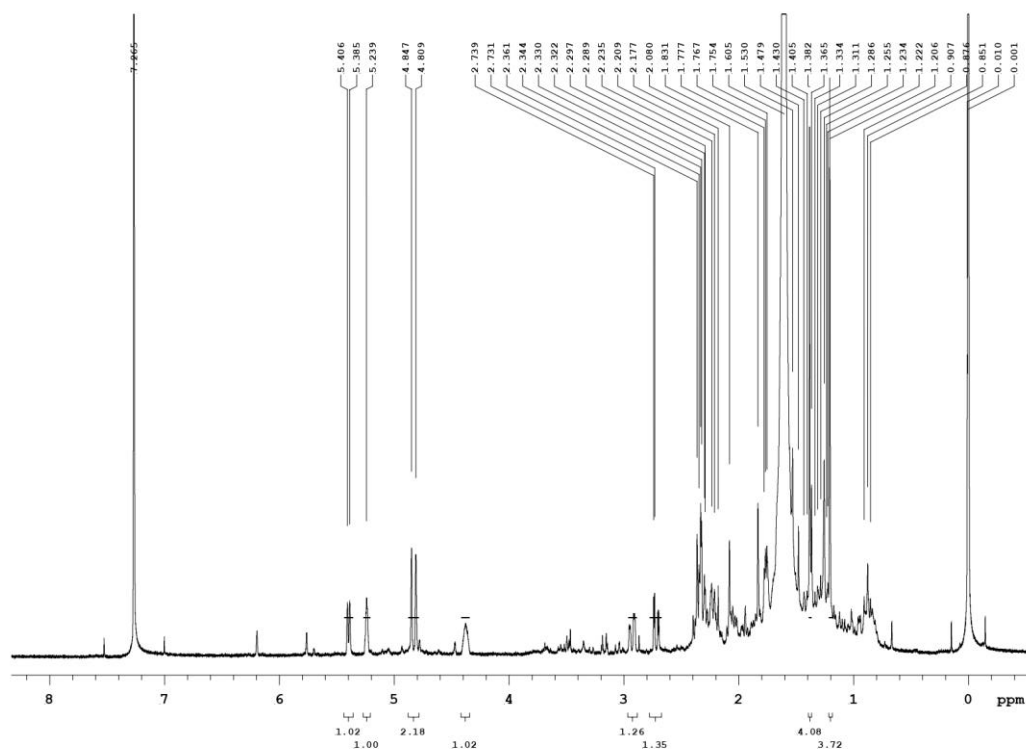


Figure S59. ^1H NMR spectrum (400MHz) of compound **13** in CDCl_3

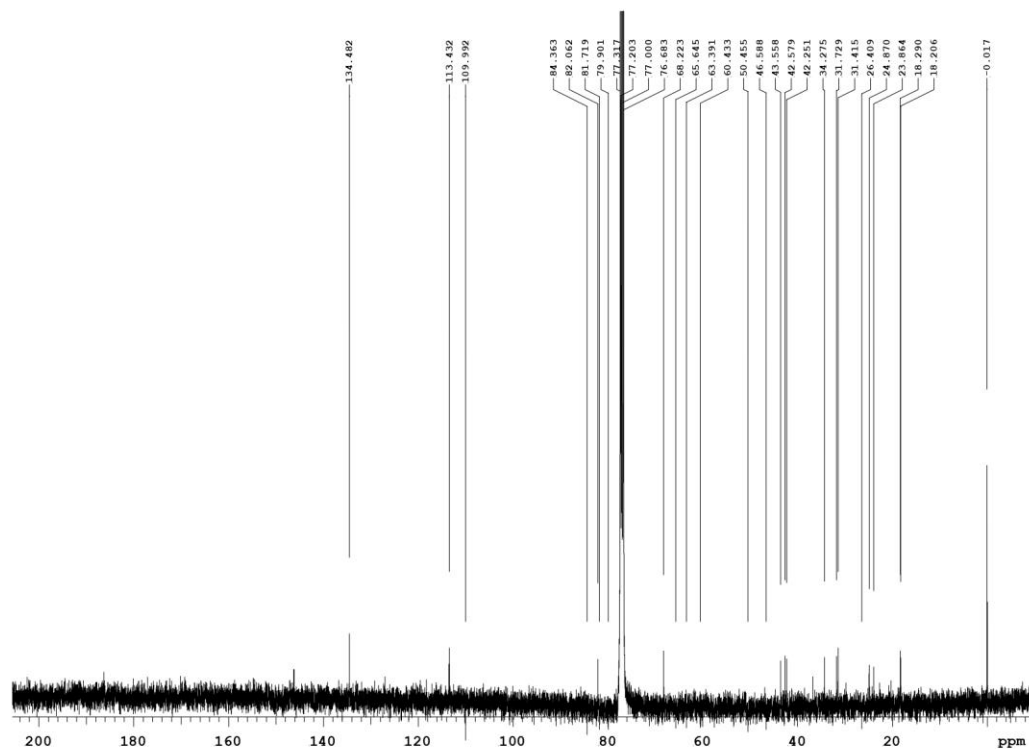


Figure S60. ^{13}C NMR spectrum (100 MHz) of compound **13** in CDCl_3

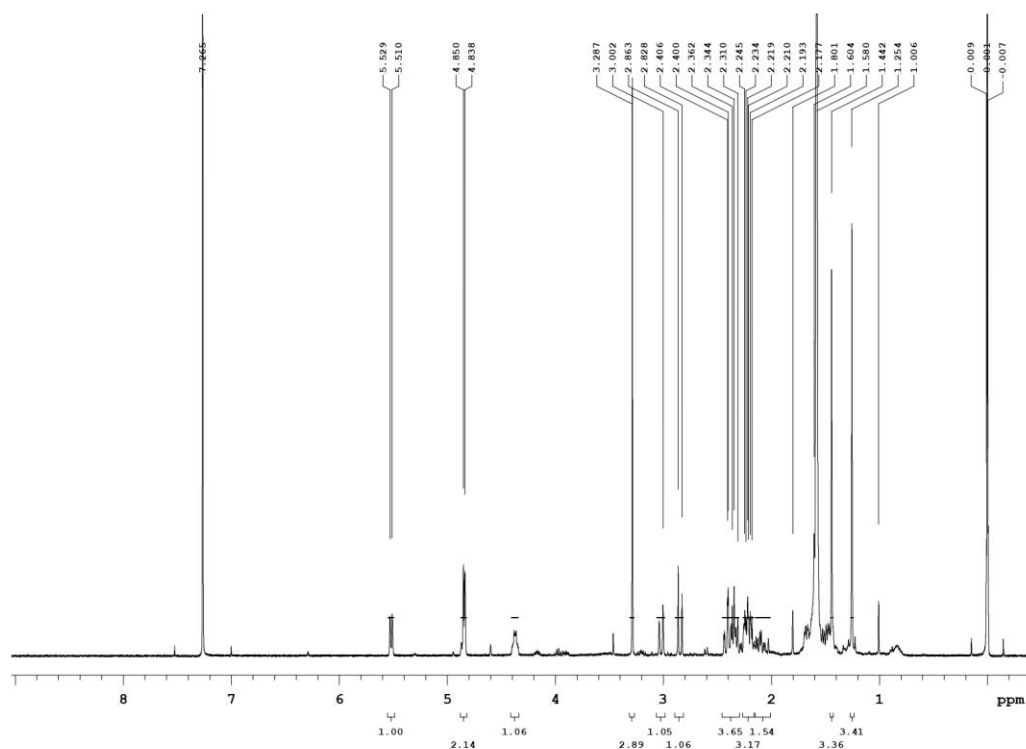


Figure S61. ¹H NMR spectrum (400MHz) of compound **14** in CDCl₃

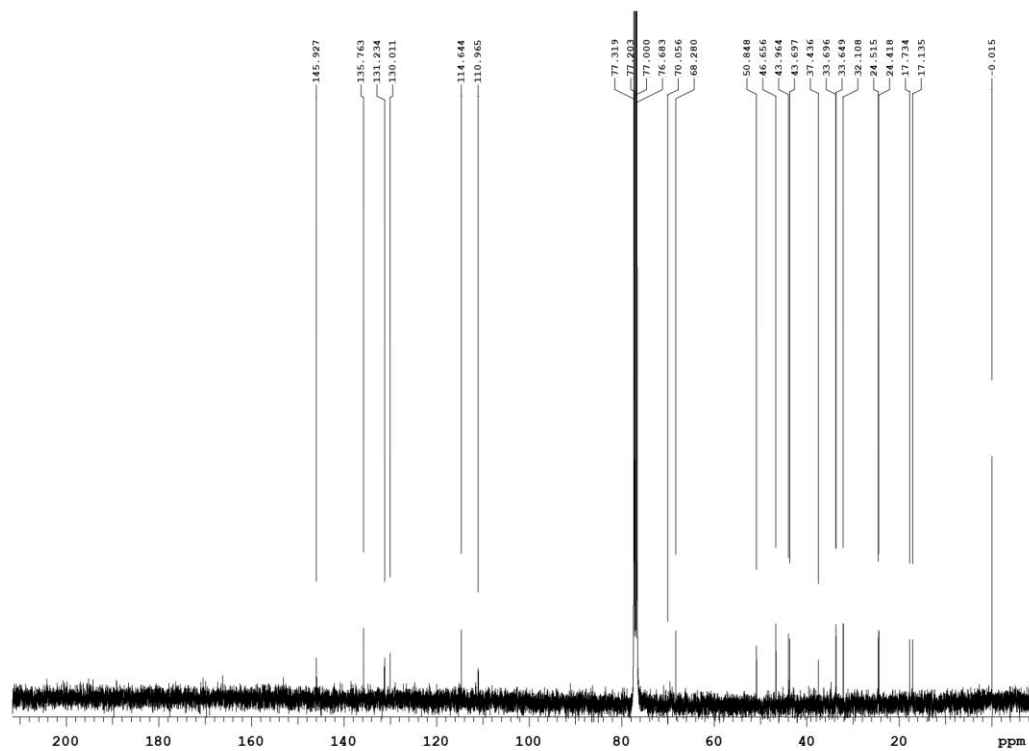


Figure S62. ¹³C NMR spectrum (100 MHz) of compound **14** in CDCl₃

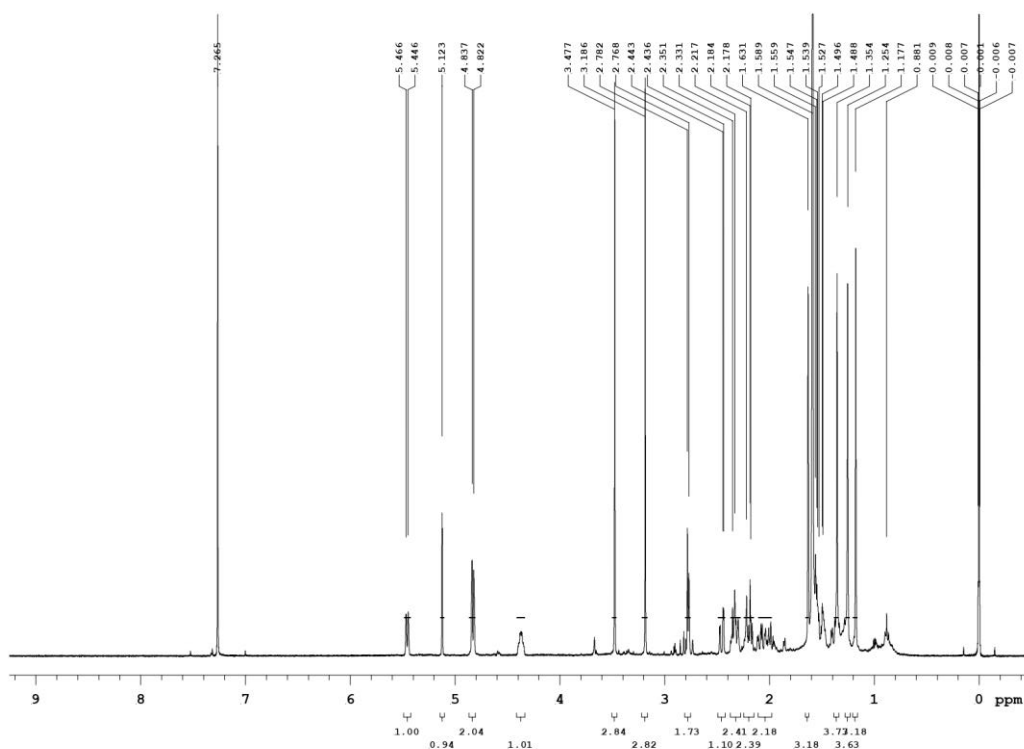


Figure S63. ^1H NMR spectrum (400MHz) of compound **15** in CDCl_3

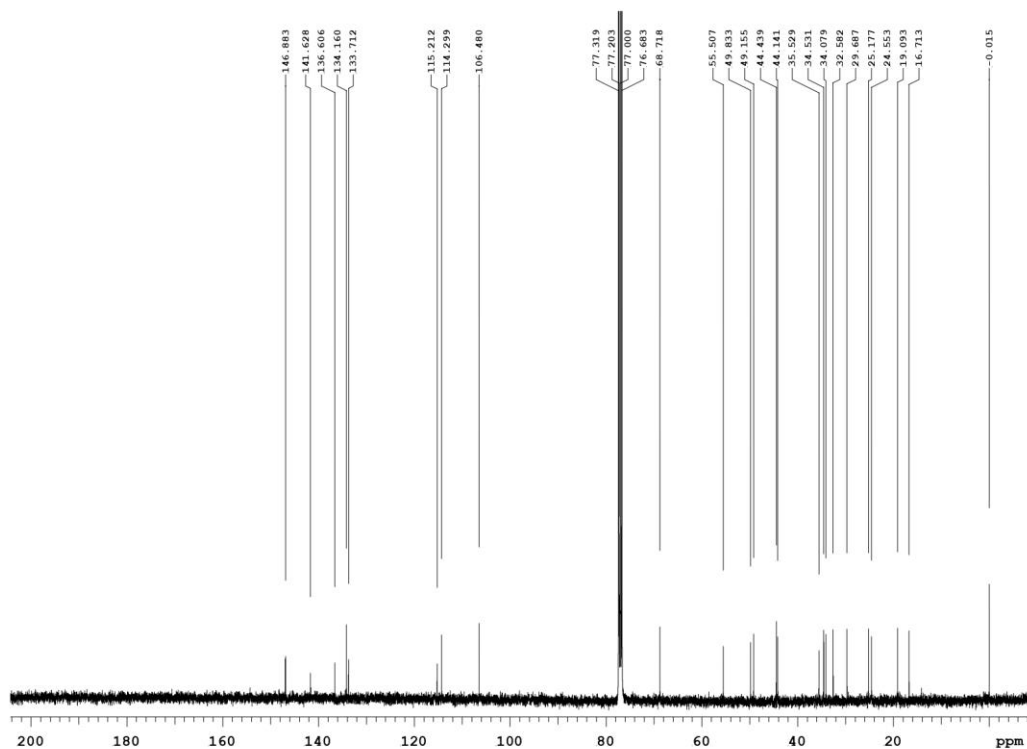


Figure S64. ^{13}C NMR spectrum (100 MHz) of compound **15** in CDCl_3

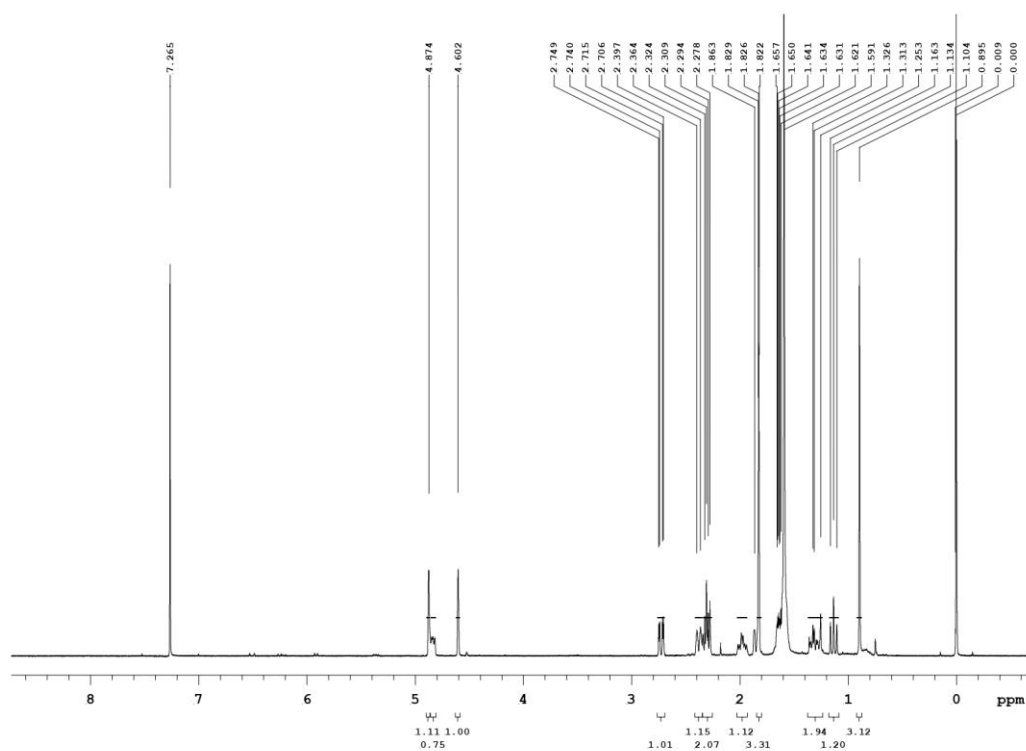


Figure S65. ^1H NMR spectrum (400MHz) of compound **16** in CDCl_3

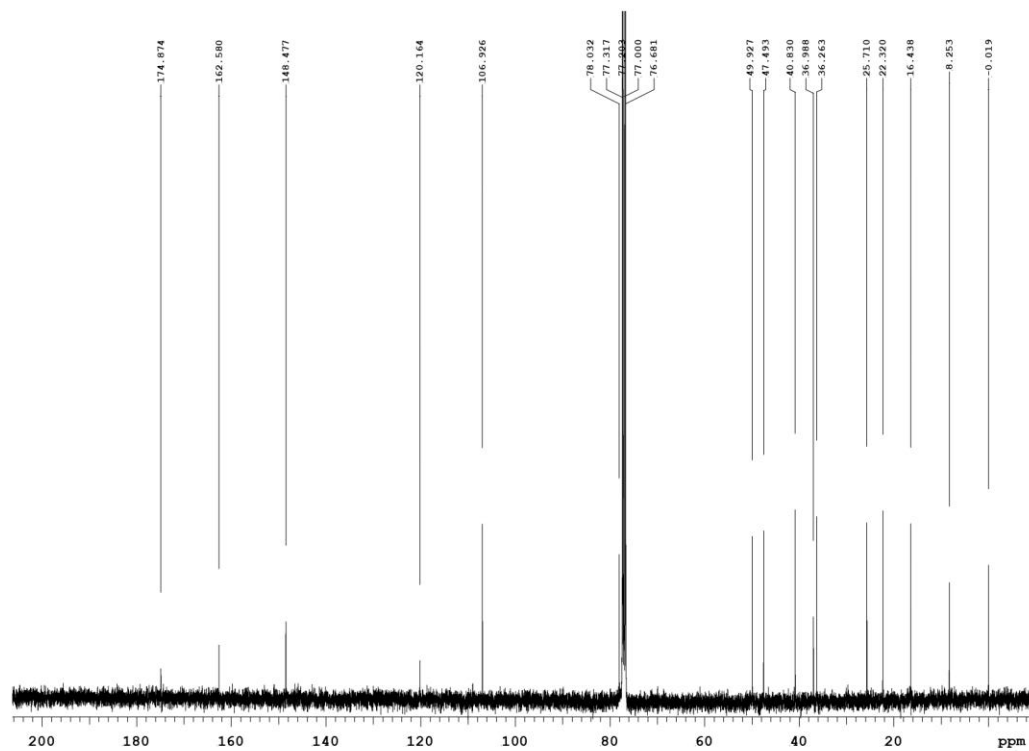


Figure S66. ^{13}C NMR spectrum (100 MHz) of compound **16** in CDCl_3

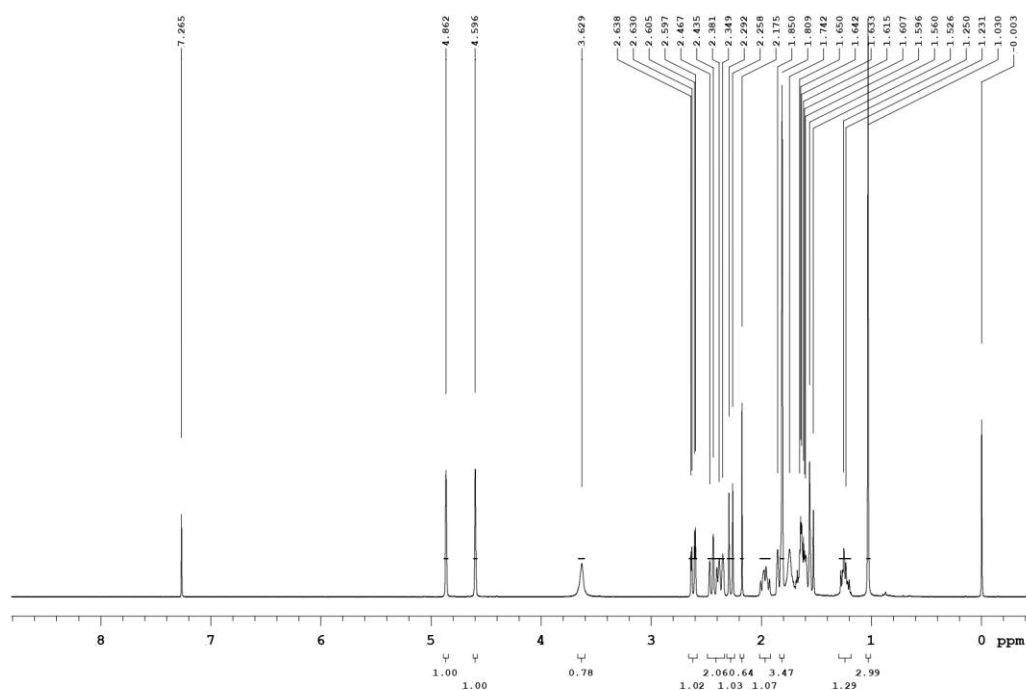


Figure S67. ¹H NMR spectrum (400MHz) of compound **17** in CDCl₃

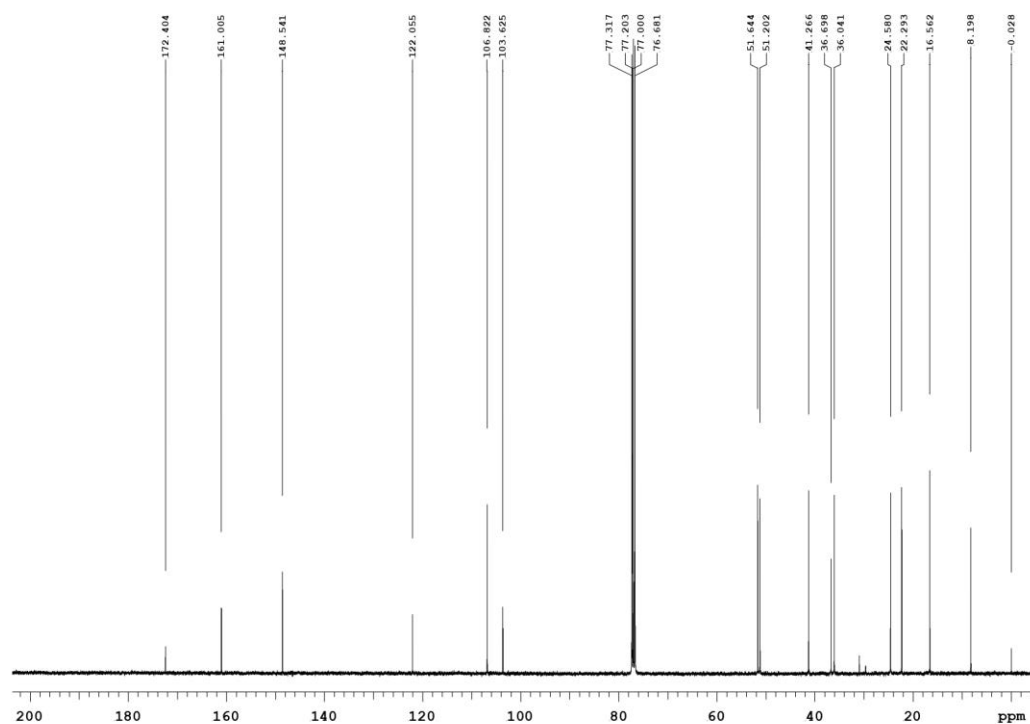


Figure S68. ¹³C NMR spectrum (100 MHz) of compound **17** in CDCl₃

Table S1. The cytotoxicity data of compounds **1–16** and **1a**.

Compounds	Cell lines IC ₅₀ (μg/mL)		
	HepG2	MDA-MB231	A549
1	> 20	> 20	> 20
1a	> 20	> 20	> 20
2	> 20	> 20	> 20
3	> 20	> 20	> 20
4	> 20	> 20	> 20
5	> 20	15.40	18.74
6	> 20	> 20	> 20
7	> 20	> 20	> 20
8	> 20	> 20	> 20
9	> 20	> 20	> 20
10	> 20	> 20	> 20
11	> 20	> 20	> 20
12	> 20	> 20	> 20
13	> 20	> 20	> 20
14	> 20	> 20	> 20
15	> 20	> 20	> 20
16	> 20	> 20	> 20
Doxorubicin	0.37	0.30	0.15

Table S2. Antibacterial activity (zone of inhibition in mm).

Compounds (25 µg/disk)	Inhibition zone (mm)									
	<i>B. s.</i>	<i>S. a.</i>	<i>S. e.</i>	<i>E. a.</i>	<i>E. c.</i>	<i>K. p.</i>	<i>S. m.</i>	<i>S. s.</i>	<i>S. t.</i>	<i>Y. e.</i>
3	–	–	–	–	–	–	–	–	–	–
4	–	–	–	–	–	–	–	–	–	–
5	–	–	6	–	–	5	–	–	–	–
6	–	–	–	–	–	–	–	–	–	–
7	–	–	–	–	–	–	–	–	–	–
9	–	–	–	–	–	9	–	–	–	–
12	–	–	–	–	–	–	–	–	–	–
Ampicillin	9	12	10	5	5	5	8	9	8	18

–: Inactive

Ampicillin: Positive control

B. s.: *Bacillus subtilis* (ATCC6051)

S. a.: *Staphylococcus aureus* (ATCC9144)

S. e.: *Salmonella enterica* (ATCC13076)

E. a.: *Enterobacter aerogenes* (ATCC13048)

E. c.: *Escherichia coli* (ATCC25922)

K. p.: *Klebsiella pneumoniae* (ATCC10031)

S. m.: *Serratia marcescens* (ATCC25419)

S. s.: *Shigella sonnei* (ATCC11060)

S. t.: *Salmonella typhimurium* (ATCC14028)

Y. e.: *Yersinia enterocolitica* (ATCC23715)

Table S3. Inhibitory effects of compounds **1–10**, **1a**, **14**, and **15** on TNF- α expression, PGE₂, and NO production in LPS induced dendritic cells.

Compounds	Inh % ¹		
	TNF- α	PGE ₂	NO
1	2.3 \pm 2.0	2.9 \pm 1.5	11.0 \pm 2.4
1a	−0.1 \pm 1.6	−0.8 \pm 5.9	14.0 \pm 1.5 *
2	7.3 \pm 3.2	12.8 \pm 3.5	19.6 \pm 4.0 ***
3	8.0 \pm 3.0	11.4 \pm 2.1	33.8 \pm 1.5 ***
4	3.8 \pm 2.7	2.4 \pm 2.1	34.9 \pm 3.9 ***
5	0.8 \pm 1.8	3.5 \pm 4.2	16.0 \pm 3.7 **
6	5.0 \pm 2.9	−4.4 \pm 0.4	14.6 \pm 3.9 *
7	1.9 \pm 5.1	−7.6 \pm 2.8	3.7 \pm 5.7
8	0.2 \pm 0.5	−4.9 \pm 3.2	10.2 \pm 3.2
9	3.3 \pm 1.7	1.2 \pm 5.9	16.8 \pm 4.7 **
10	−3.9 \pm 1.1	−11.5 \pm 4.6	11.9 \pm 2.7
14	5.2 \pm 1.8	−8.8 \pm 6.3	24.8 \pm 1.4***
15	23.6 \pm 2.5 ***	21.2 \pm 0.9 *	35.0 \pm 3.7 ***
DEX ²	85.6 \pm 3.4 ****	–	73.4 \pm 1.3 ****

1. Percentage of inhibition (Inh %) at the concentration 100 μ M for **1–3**, **5**, **7**, **9**, **11**, **14** and **15** and 25 μ M for **4**, **6**, and **8** compared with the control group (100 % for stimulated LPS alone). Results are presented as mean \pm SEM. (n = 3). * p < 0.05, ** p < 0.01, *** p < 0.001, **** p < 0.0001.

2. Positive control: dexamethasone (DEX) at 100 μ M.