

Experimental Supporting Information

Synthesis and Biological Evaluation of N-Alkyl Sulfonamides Derived from Polycyclic Hydrocarbon Scaffolds Using a Nitrogen-Centered Radical Approach

Megan D. Hopkins, Ryan C. Witt, Ann Marie E. Flusche, John E. Philo, Garrett L. Ozmer, Gordon H. Purser, Robert J. Sheaff*, and Angus A. Lamar*

Department of Chemistry and Biochemistry, The University of Tulsa, 800 S. Tucker Dr., Tulsa, OK 74104, USA.

robert-sheaff@utulsa.edu

angus-lamar@utulsa.edu

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Construction of LED Chambers:

Visible-light photocatalytic reactions were set up in a light bath which was constructed in our laboratory by coiling LED strips around an evaporating dish according to our previous reports:¹⁻⁴

Waterproof 5050 LED strips (12V with power adapter, 18 LEDs/foot, approximately 0.24 Watt per LED – 72 Watt per strip) are coiled around the interior of evaporating dish (170mm x 90mm) using the adhesive backing of the LED strip. A Petri dish (150 x 20 mm) is placed upside down at the bottom of the dish to serve as an elevated glass “floor” to ensure that a round-bottom flask receives maximum light exposure. The ambient temperature inside the dish is monitored and is generally maintained (air-cooled or fan) between 19-22 °C (the temperature has not been observed above 25 °C).

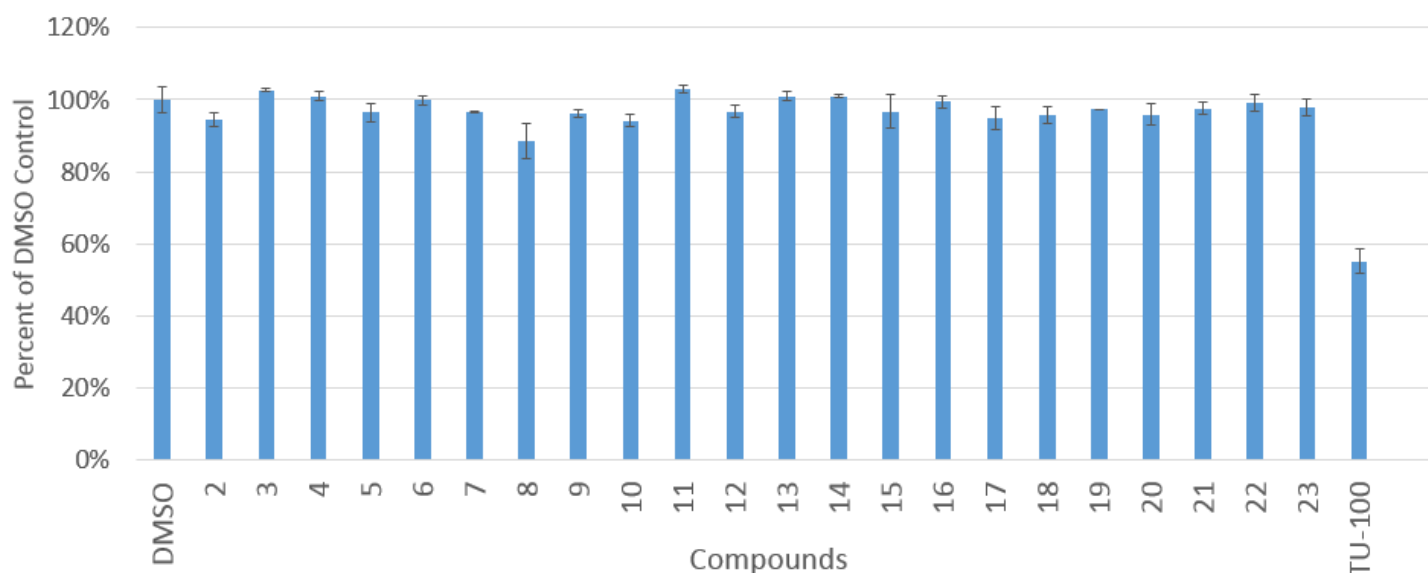
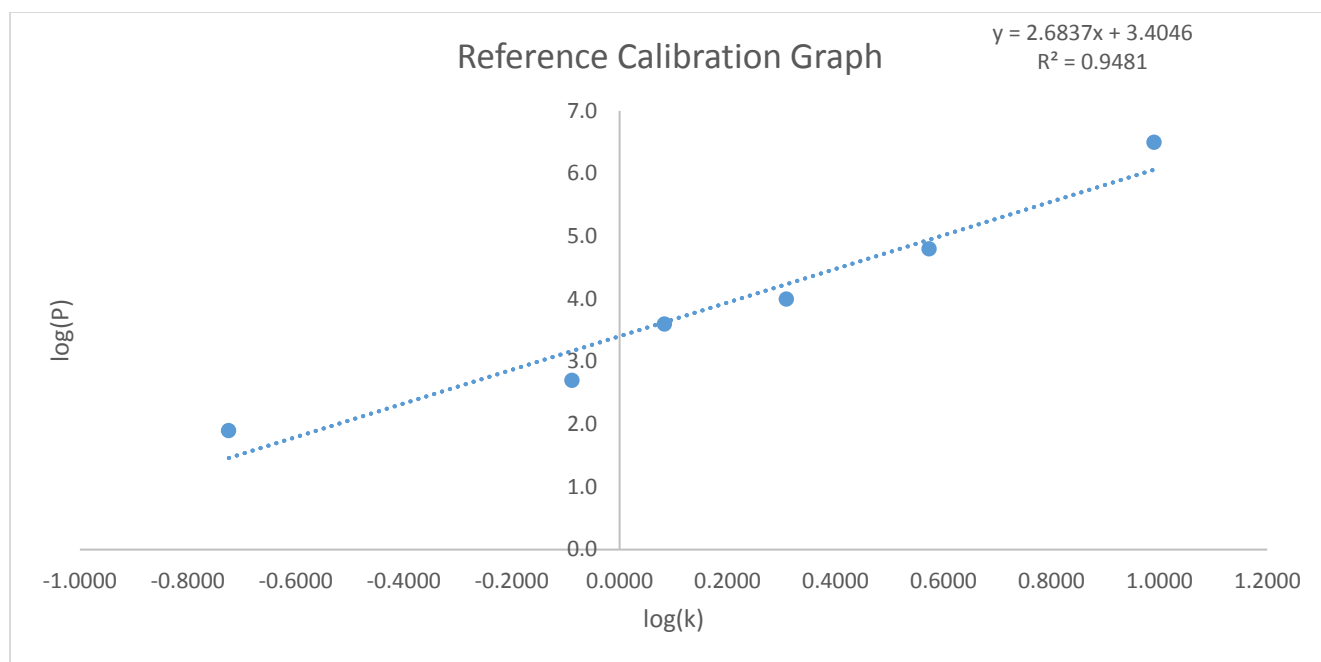


Figure S1 - Control experiments performed at 5 h exposure of 50 μ M of compounds using CellTiter-Glo assay with exogenous ATP added. No inhibition of the luciferase-producing assay itself was observed by the compounds (**2-23**). TU-100,⁵ a known inhibitor of the luciferase assay, was used as a positive control.

Compounds	Cell Lines (Values are shown as Percent of DMSO Control)				
	HDF	H293	HeLa	PC3	BxPC3
DMSO	100 ± 3.4%	100.0 ± 4.7%	100.0 ± 10.8%	100.0 ± 1.0%	100.0 ± 8.2%
2	54.3 ± 2.2%	80.9 ± 2.8%	57.6 ± 21.1%	55.3 ± 1.3%	55.5 ± 3.1%
3	10.1 ± 0.1%	67.9 ± 0.2%	27.0 ± 2.4%	46.4 ± 1.7%	38.0 ± 1.8%
4	97.7 ± 0.3%	86.0 ± 3.9%	92.8 ± 4.6%	73.9 ± 7.3%	90.5 ± 0.7%
5	20.8 ± 1.7%	76.0 ± 1.5%	40.5 ± 0.4%	43.9 ± 9.0%	52.3 ± 0.2%
6	39.8 ± 3.9%	63.6 ± 3.0%	54.9 ± 2.7%	41.7 ± 0.7%	46.5 ± 4.1%
7	67.8 ± 11.7%	94.0 ± 1.8%	79.9 ± 0.0%	67.5 ± 4.2%	68.7 ± 1.3%
8	25.6 ± 12.8%	61.1 ± 0.6%	46.8 ± 9.3%	74.9 ± 8.4%	56.7 ± 8.7%
9	1.1 ± 0.1%	41.0 ± 4.9%	4.6 ± 0.6%	17.5 ± 7.8%	1.9 ± 1.2%
10	20.6 ± 1.1%	80.0 ± 4.4%	40.4 ± 10.8%	37.1 ± 2.4%	50.8 ± 0.4%
11	77.0 ± 1.6%	68.1 ± 1.3%	71.1 ± 9.7%	53.9 ± 3.3%	75.5 ± 2.2%
12	61.0 ± 11.1%	82.3 ± 11.6%	59.2 ± 3.2%	63.2 ± 5.0%	69.5 ± 3.9%
13	107.3 ± 1.1%	77.4 ± 2.4%	81.9 ± 4.8%	76.8 ± 4.9%	80.8 ± 2.9%
14	86.6 ± 2.4%	76.0 ± 6.8%	81.8 ± 4.7%	47.3 ± 8.3%	76.7 ± 6.7%
15	13.4 ± 0.5%	46.7 ± 7.0%	22.1 ± 3.0%	44.5 ± 6.1%	42.5 ± 0.5%
16	19.2 ± 0.5%	51.9 ± 1.9%	15.8 ± 0.6%	9.9 ± 0.5%	7.9 ± 0.2%
17	1.2 ± 0.1%	7.8 ± 4.4%	1.5 ± 0.3%	5.6 ± 1.6%	1.0 ± 0.3%
18	58.3 ± 1.6%	67.0 ± 2.3%	79.5 ± 14.2%	50.1 ± 1.3%	48.4 ± 2.0%

Table S1. Cell viability results from screening of compounds **2-18** (50 μ M), 24 h incubation, CellTiter-Glo assay (Promega). Compound “hits” (<50% percent of DMSO control) are shown in red.



Compound	T_r	T_o (ethyl acetate)	k	Log(k)	Log(P)
Nitrobenzene	4.528	3.809	0.1888	-0.7240	1.9
Toluene	6.93	3.814	0.8170	-0.0878	2.7
Naphthalene	8.466	3.827	1.2122	0.0836	3.6
Biphenyl	11.637	3.83	2.0384	0.3093	4.0
Bibenzyl	18.179	3.831	3.7452	0.5735	4.8
DDT	41.243	3.827	9.7768	0.9902	6.5

Figure S2. Calibration curve and raw data for calculation of LogP values of known calibration compounds. This data was obtained by our research group and reported previously.⁶

Product Characterization:

All products were isolated according to general procedure unless otherwise noted and display the characterizational data shown below.

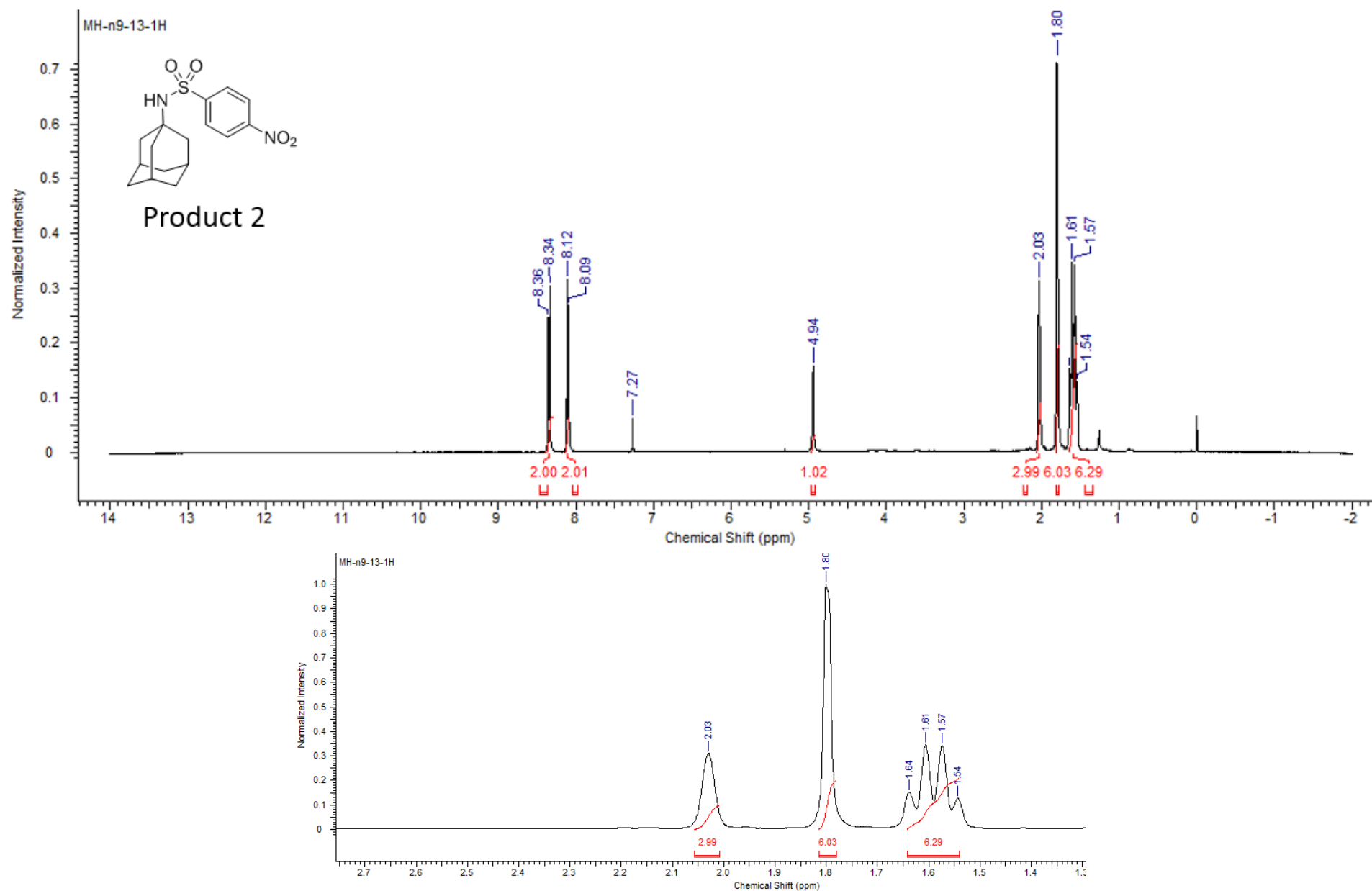
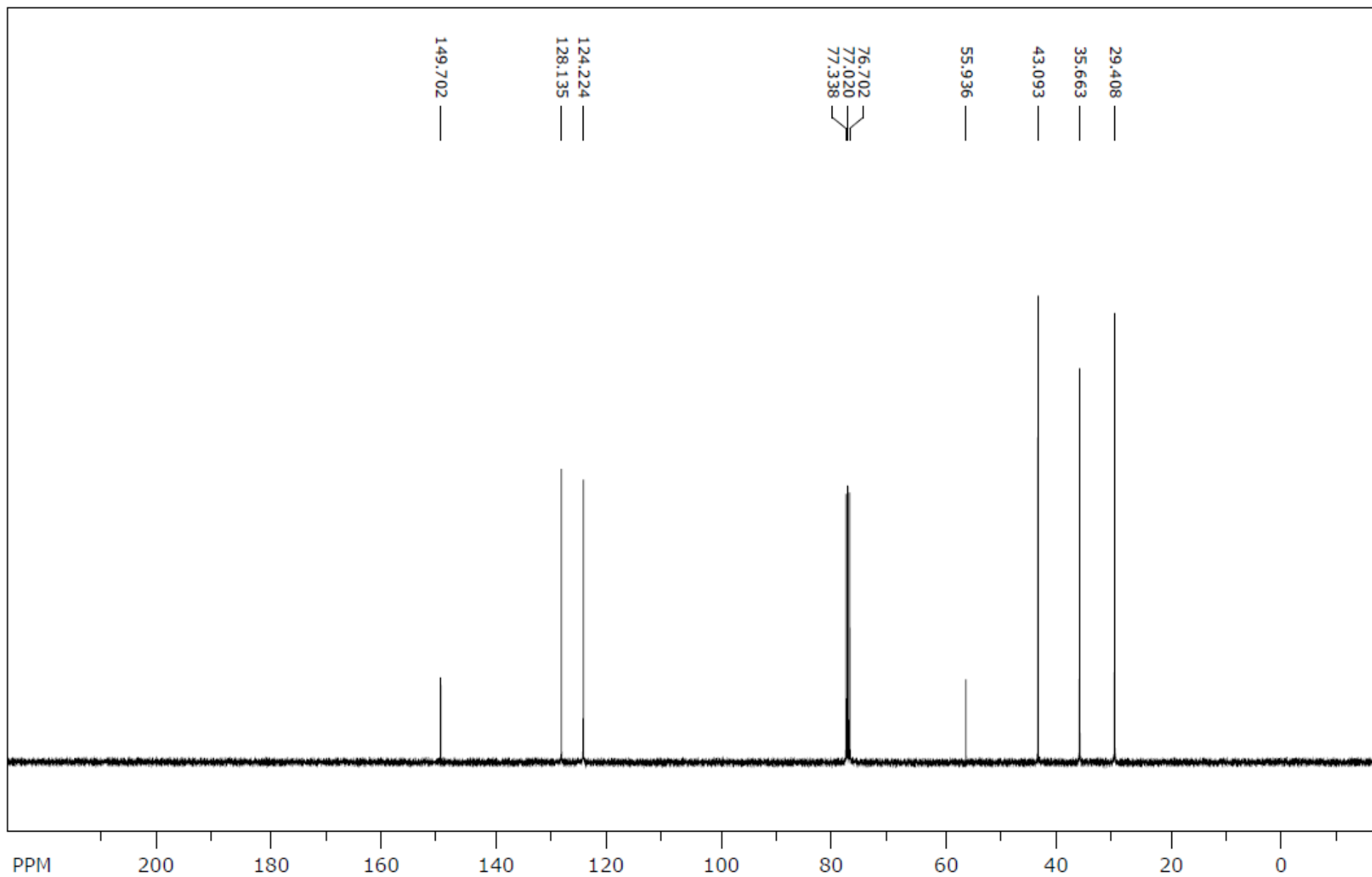


Figure S3. ¹H NMR of Product 2.



file: ...tane, 4-NO₂Ph\MH-n9-13-13C.fid\fid block# 1 expt: "s2pul"
transmitter freq.: 100.511715 MHz
time domain size: 63750 points
width: 24509.80 Hz = 243.8502 ppm = 0.384468 Hz/pt
number of scans: 1488

freq. of 0 ppm: 100.501162 MHz
processed size: 65536 complex points
LB: 0.500 GF: 0.0000
Hz/cm: 980.392 ppm/cm: 9.75401

Figure S4. ¹³C NMR of Product 2.

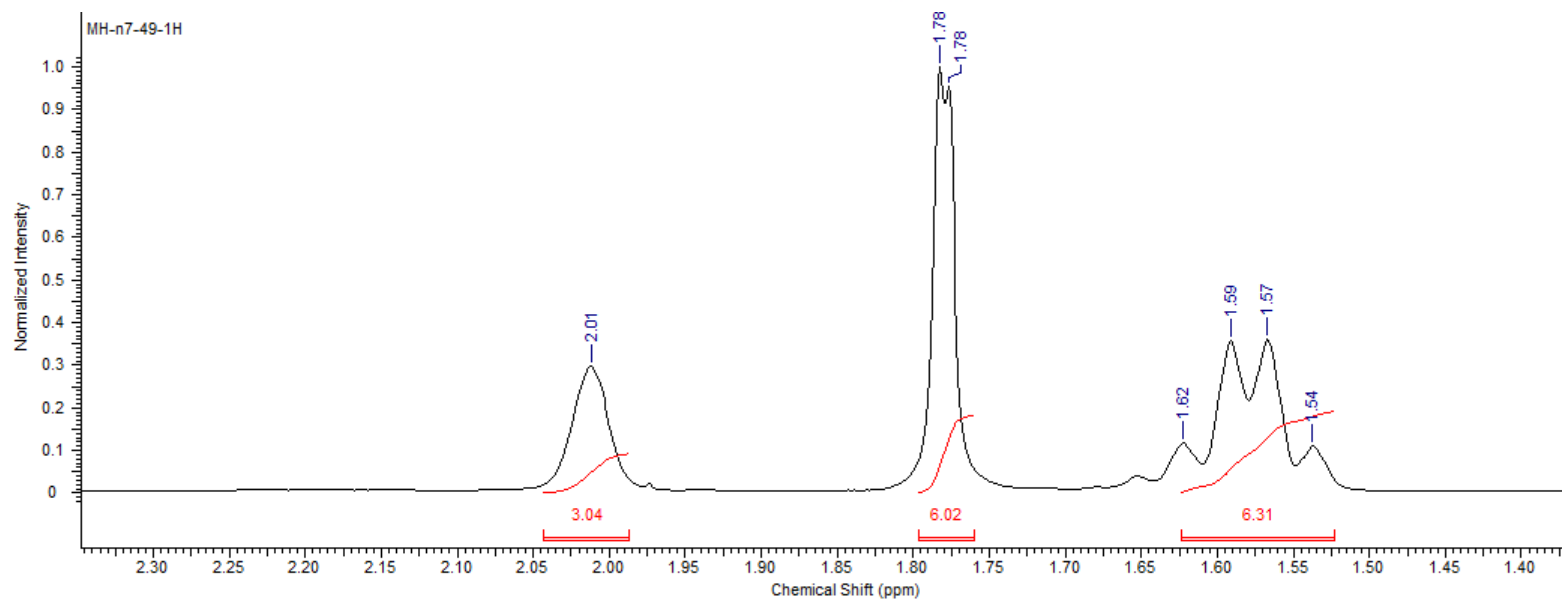
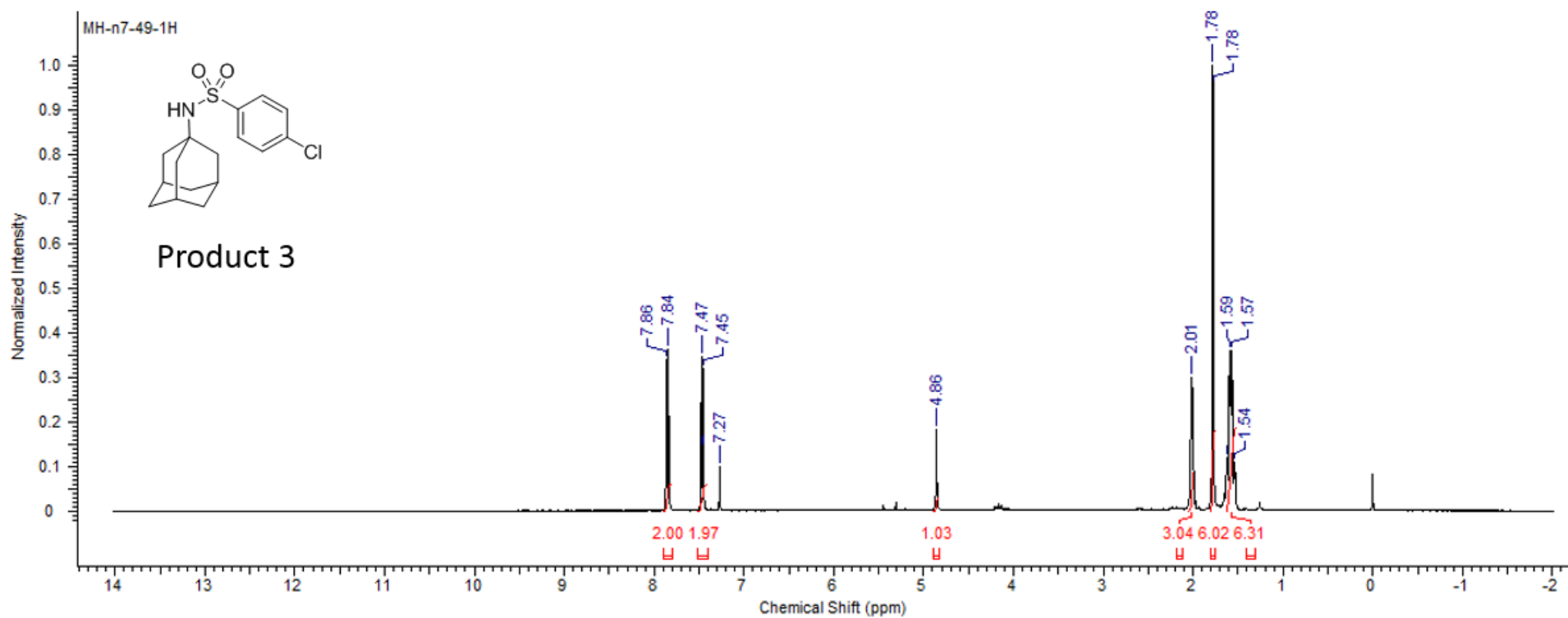
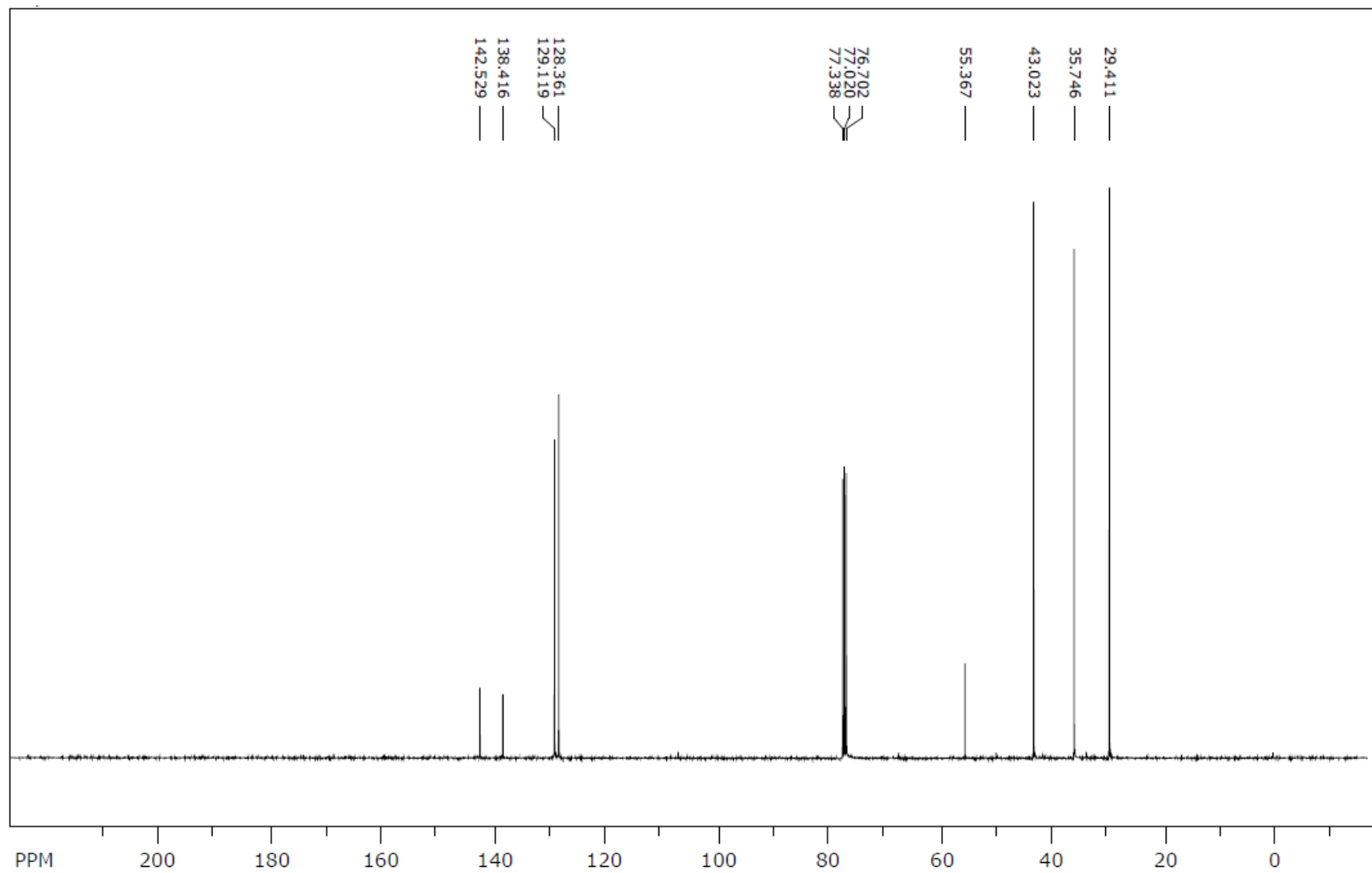


Figure S5. ^1H NMR of Product 3.



file: ...ntane, 4-ClPh\MH-n7-49-13C.fid\fid_block# 1 expt: "s2pul"
transmitter freq.: 100.511715 MHz
time domain size: 63750 points
width: 24509.80 Hz = 243.8502 ppm = 0.384468 Hz/pt
number of scans: 17000

freq. of 0 ppm: 100.501162 MHz
processed size: 65536 complex points
LB: 0.500 GF: 0.0000
Hz/cm: 980.392 ppm/cm: 9.75401

Figure S6. ¹³C NMR of Product 3.

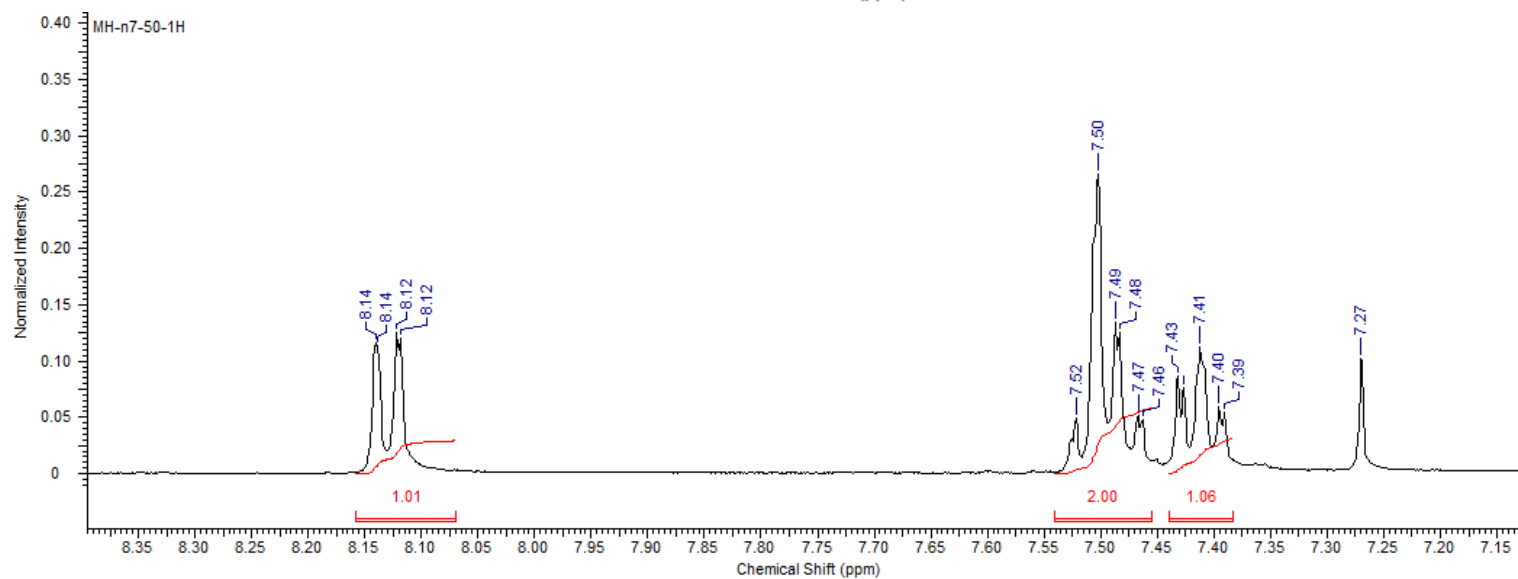
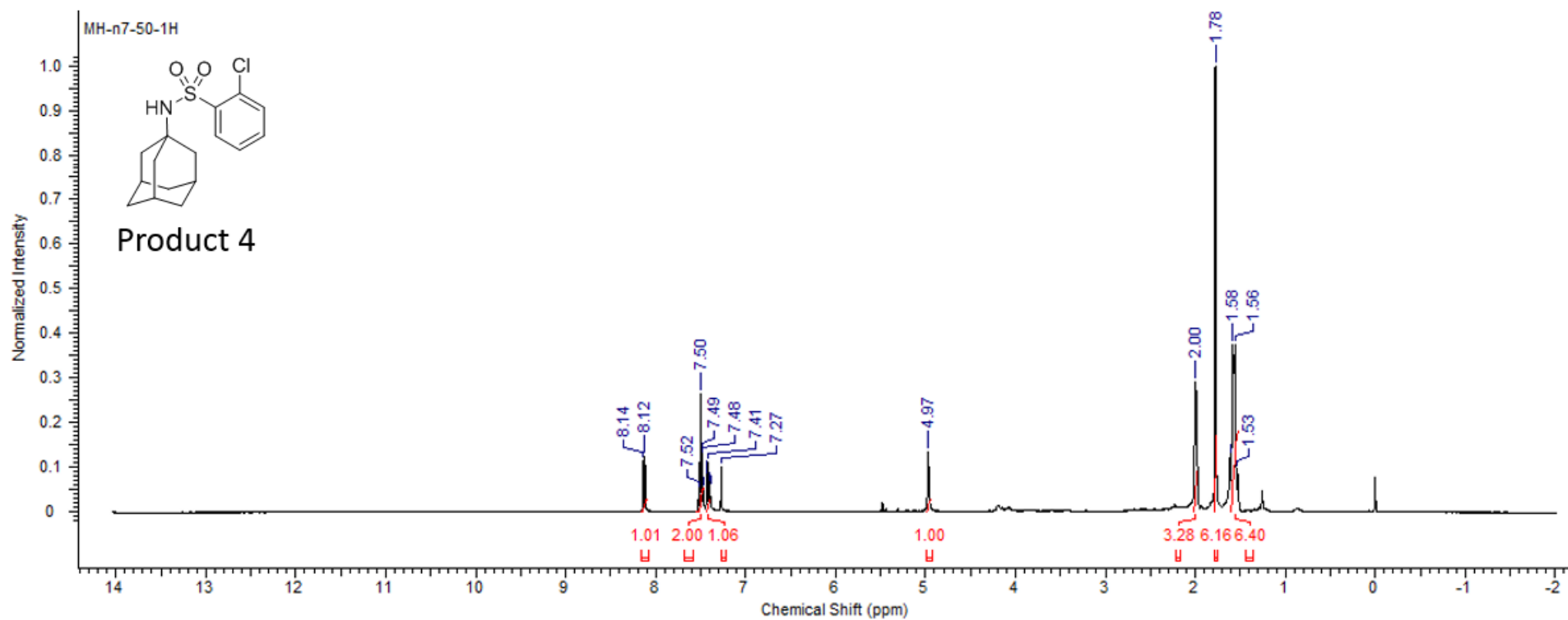
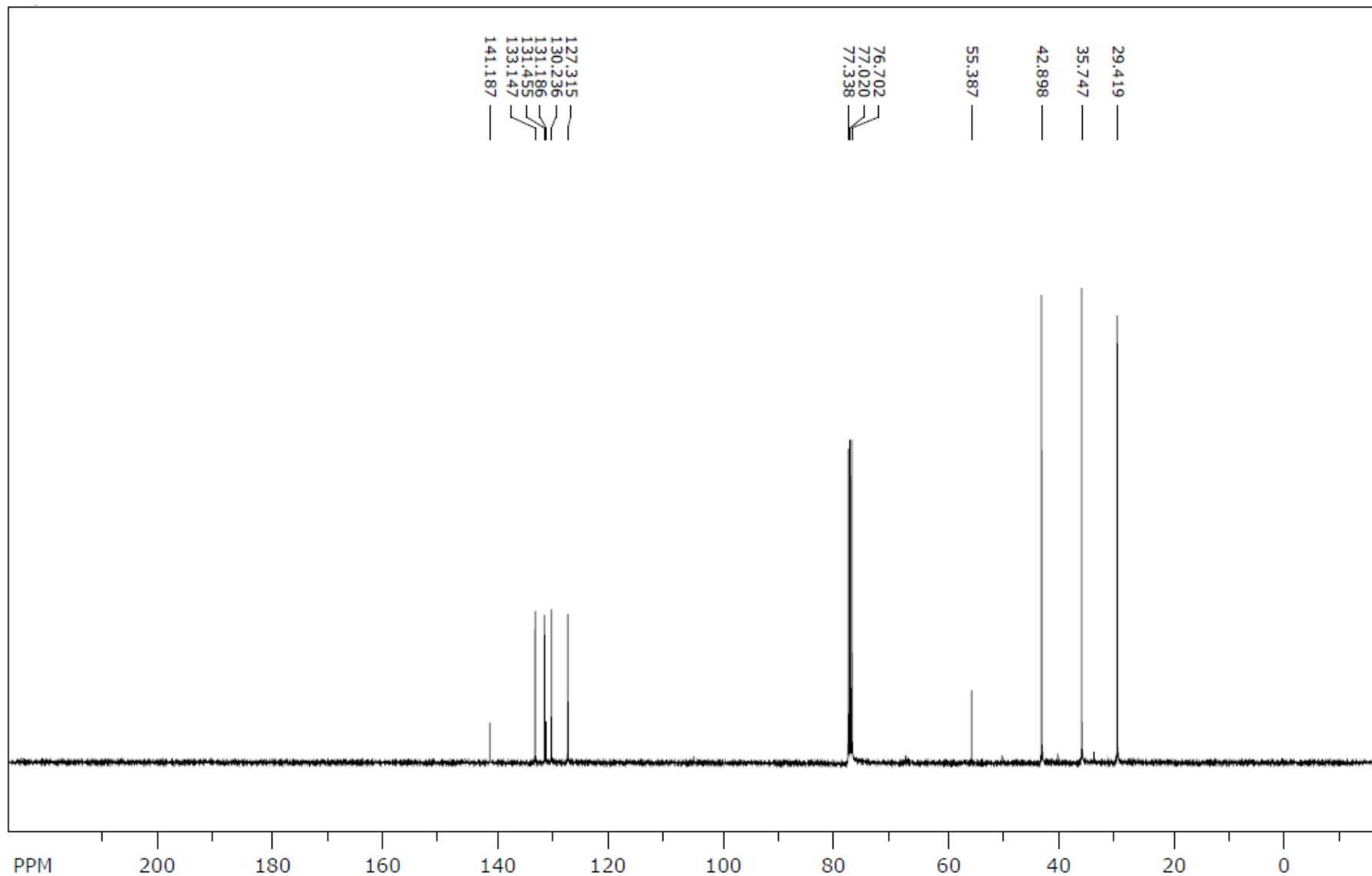


Figure S7. ^1H NMR of Product 4.



file: ...ntane, 2-ClPh\MH-n7-50-13C.fid\fid block# 1 expt: "s2pul"
 transmitter freq.: 100.511715 MHz
 time domain size: 63750 points
 width: 24509.80 Hz = 243.8502 ppm = 0.384468 Hz/pt
 number of scans: 15000

freq. of 0 ppm: 100.501162 MHz
 processed size: 65536 complex points
 LB: 0.500 GF: 0.0000
 Hz/cm: 980.392 ppm/cm: 9.75401

Figure S8. ¹³C NMR of Product 4.

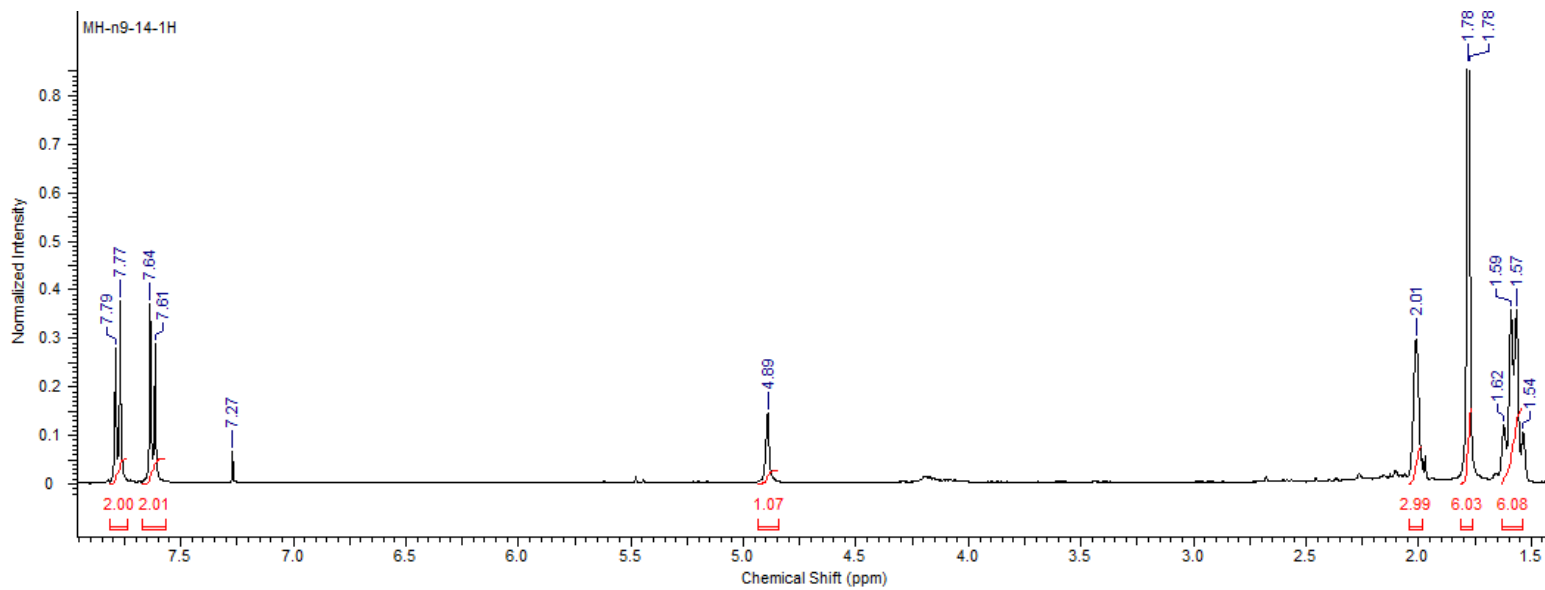
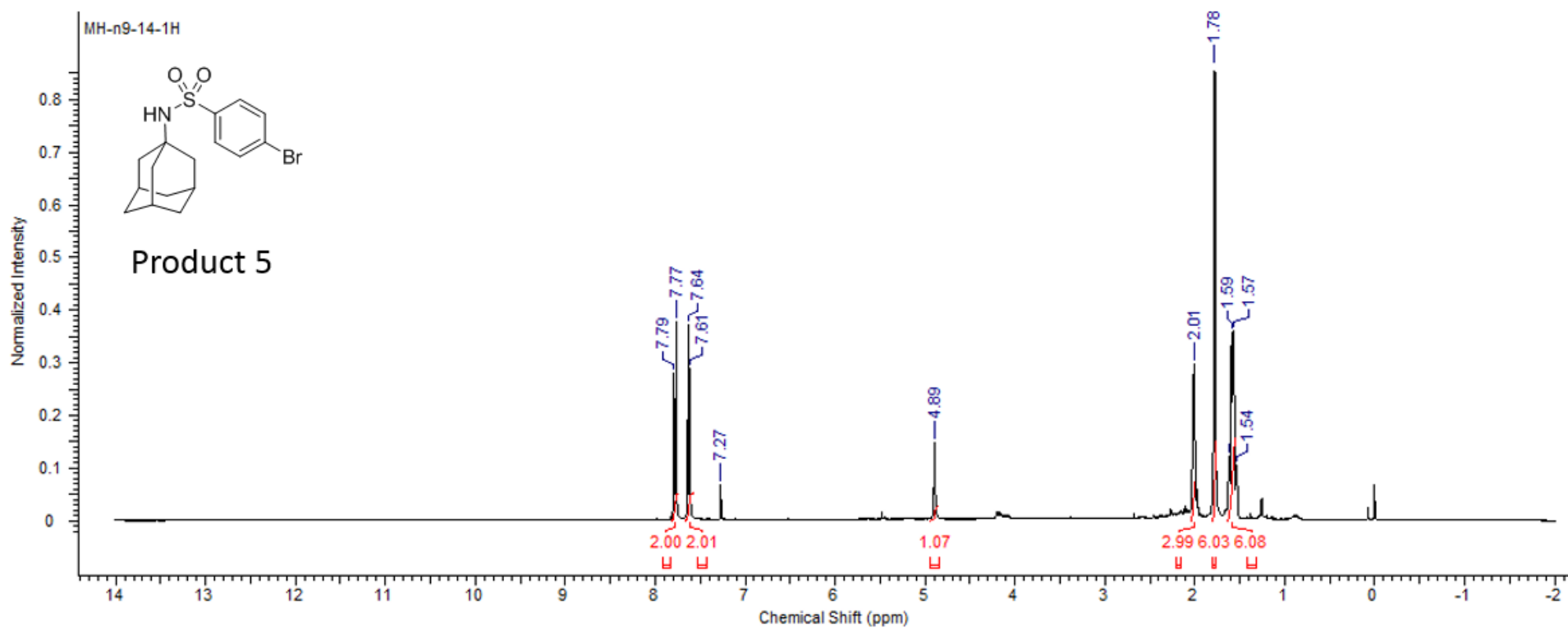
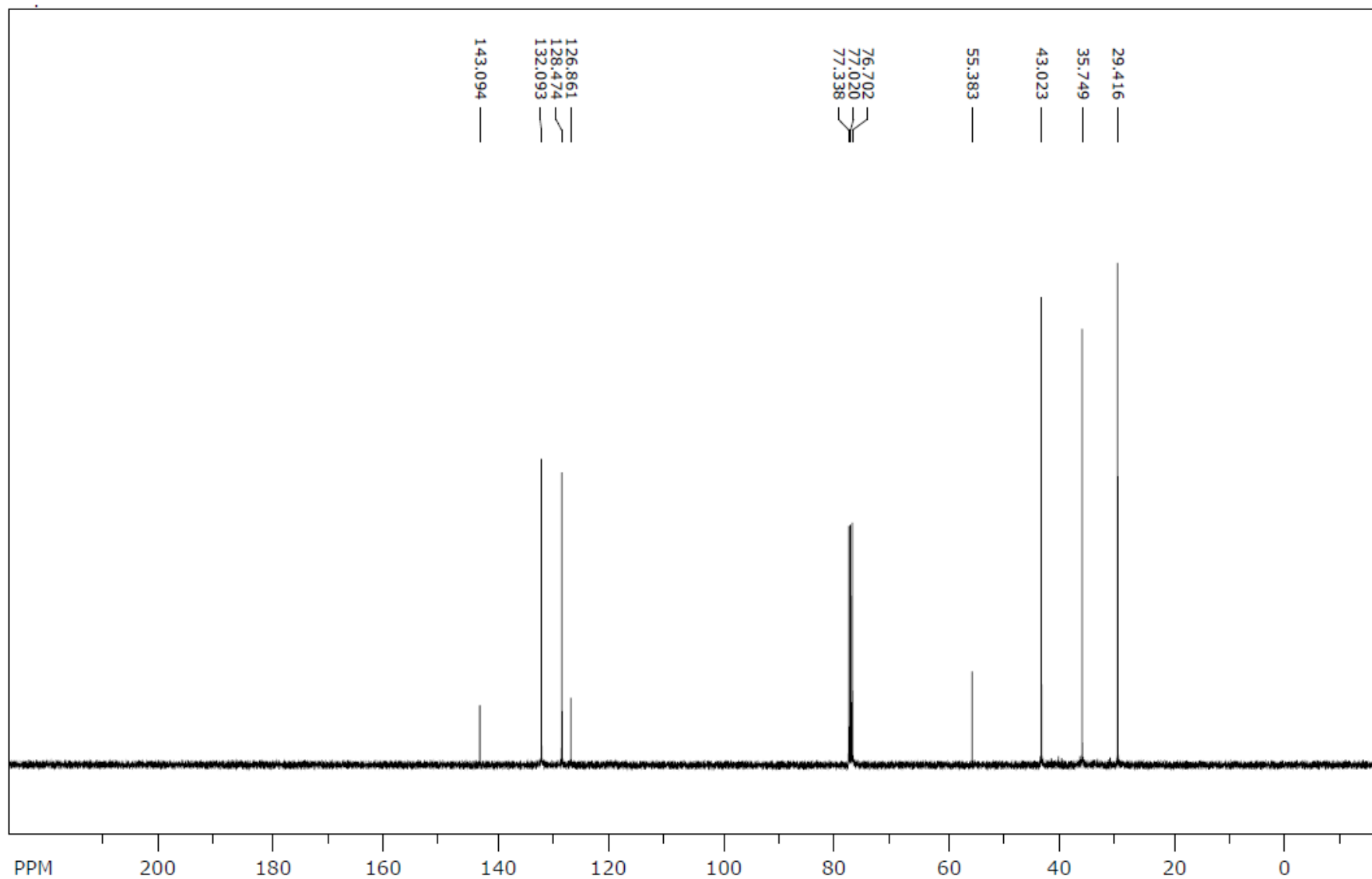


Figure S9. ¹H NMR of Product 5.



file: ...ntane, 4-BrPh\MH-n9-14-13C.fid\fid_block# 1 expt: "s2pul"
 transmitter freq.: 100.511715 MHz
 time domain size: 63750 points
 width: 24509.80 Hz = 243.8502 ppm = 0.384468 Hz/pt
 number of scans: 1360

freq. of 0 ppm: 100.501163 MHz
 processed size: 65536 complex points
 LB: 0.500 GF: 0.0000
 Hz/cm: 980.392 ppm/cm: 9.75401

Figure S10. ^{13}C NMR of Product 5.

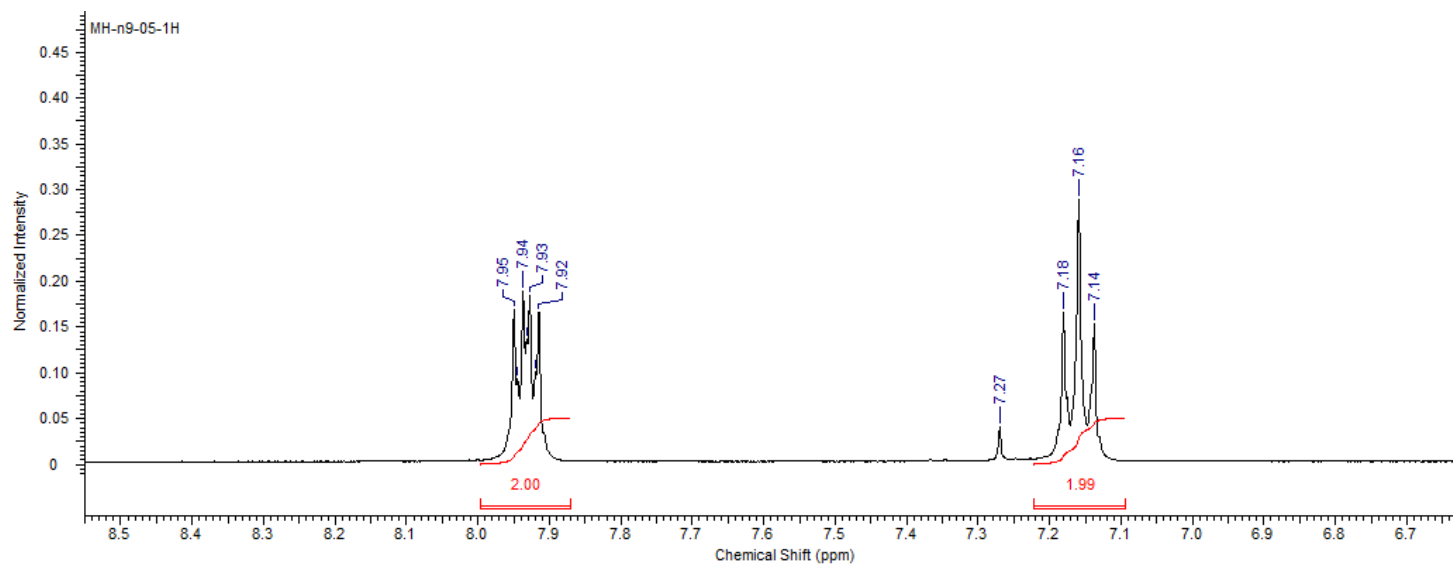
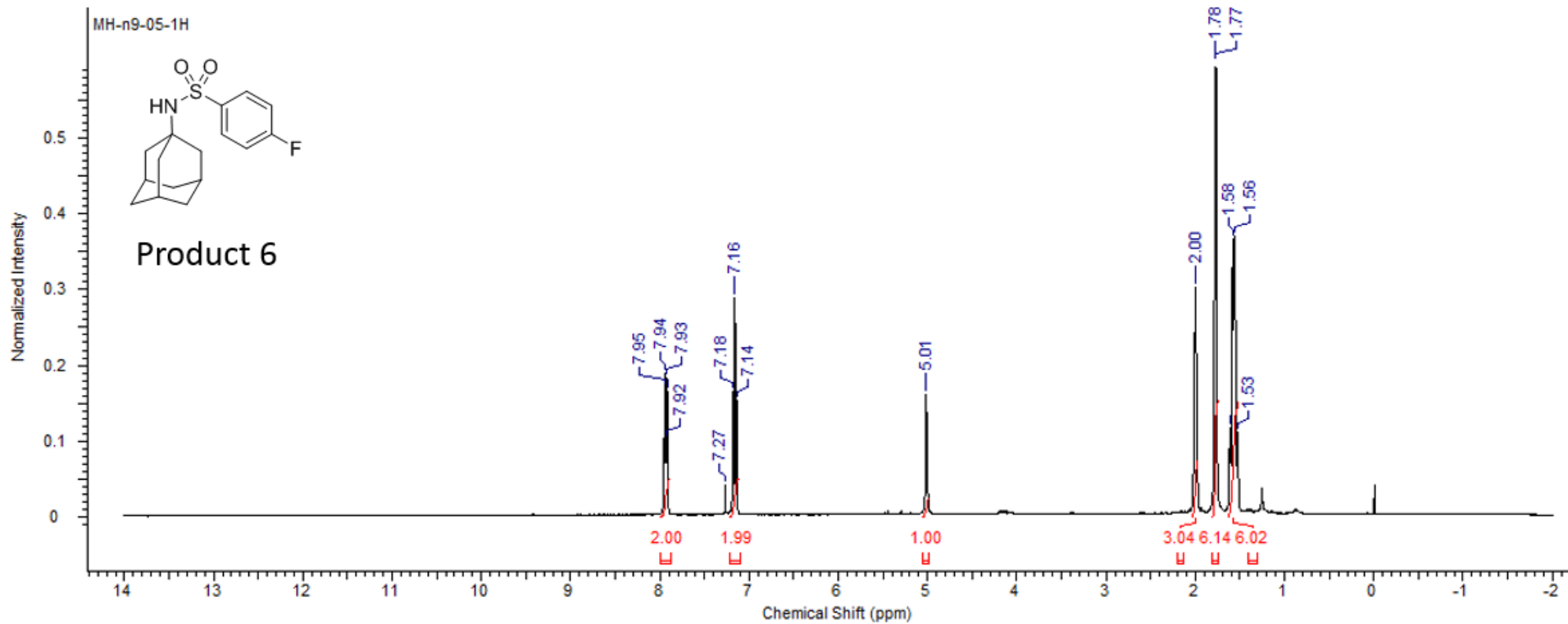
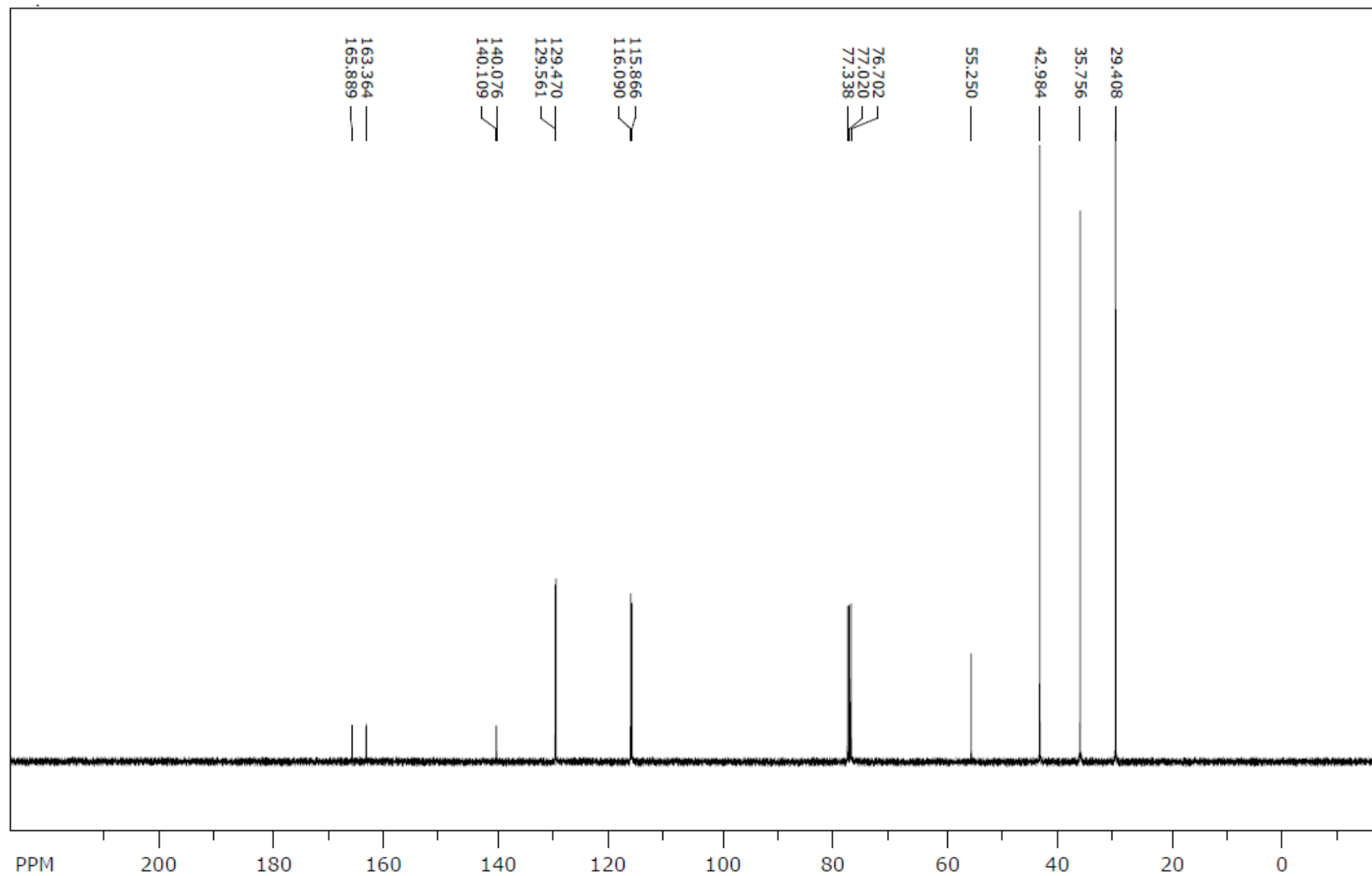


Figure S11. ¹H NMR of Product 6.



file: ...antane, 4-FPh\MH-n9-05-13C.fid\fid block# 1 expt: "s2pul"
 transmitter freq.: 100.511715 MHz
 time domain size: 63750 points
 width: 24509.80 Hz = 243.8502 ppm = 0.384468 Hz/pt
 number of scans: 1088

freq. of 0 ppm: 100.501164 MHz
 processed size: 65536 complex points
 LB: 0.500 GF: 0.0000
 Hz/cm: 980.392 ppm/cm: 9.75401

Figure S12. ¹³C NMR of Product 6.

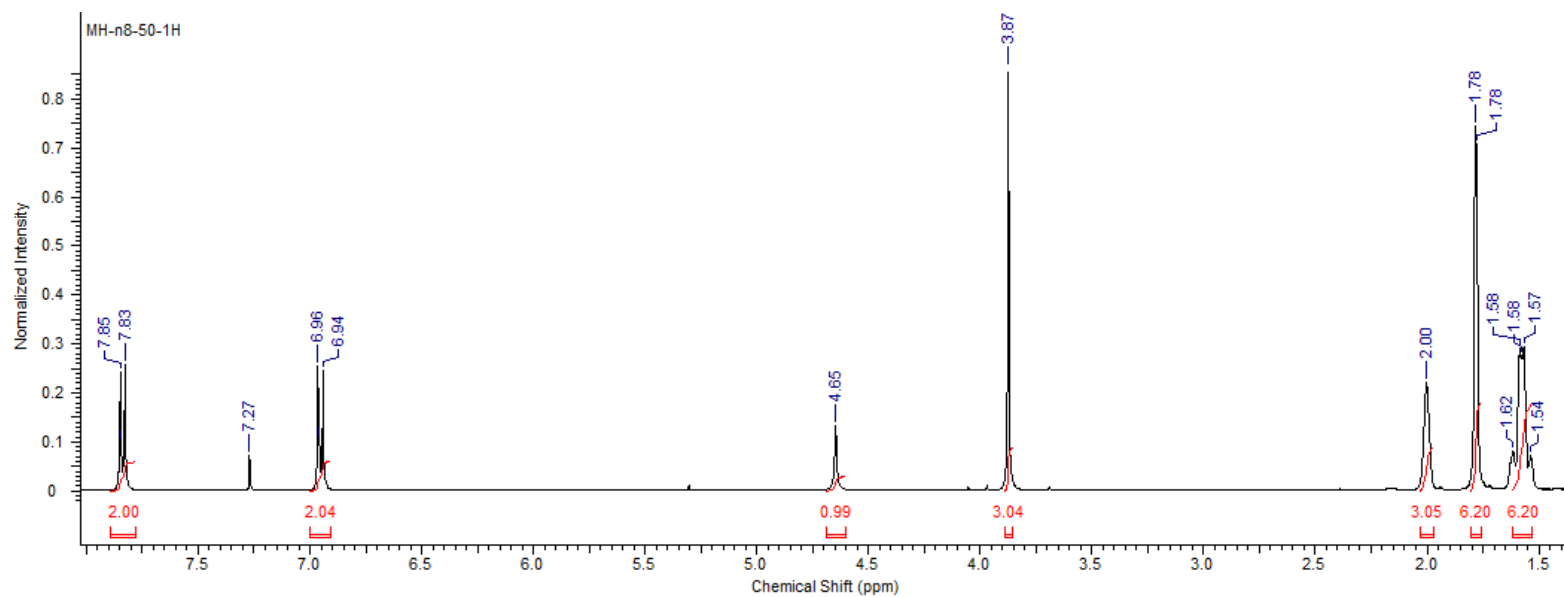
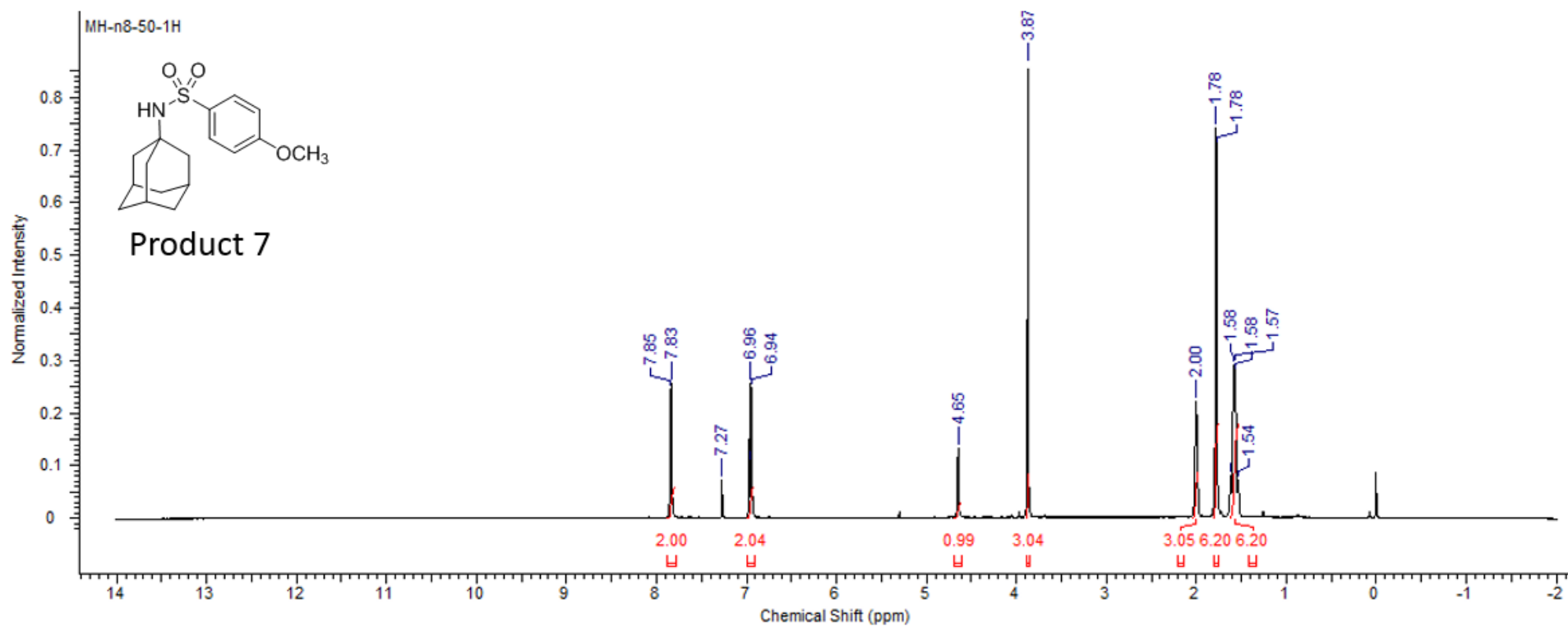
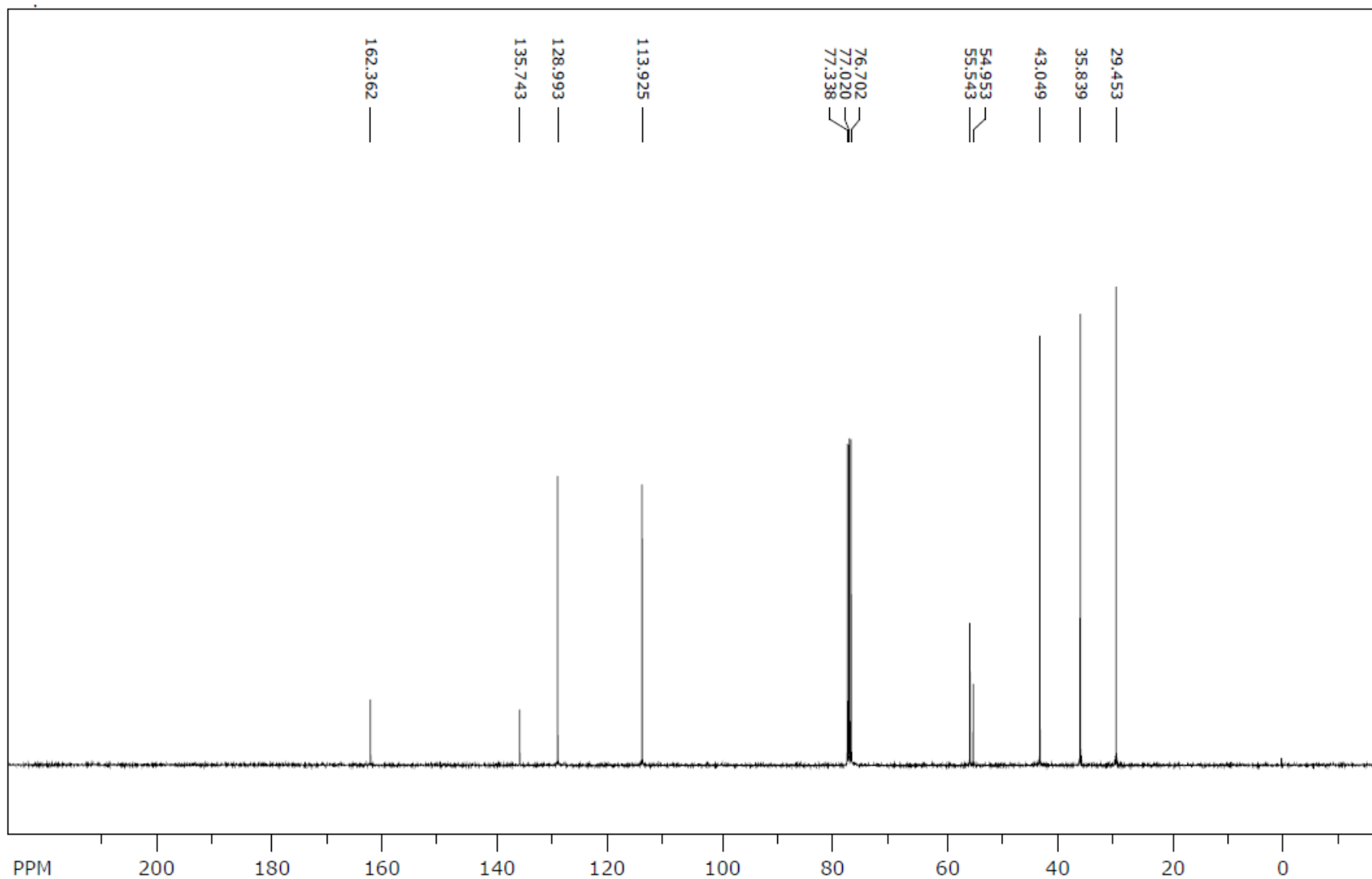


Figure S13. ^1H NMR of Product 7.



file: ...tane, 4-OMePh\MH-n8-50-13C.fid\fid_block# 1 expt: "s2pul"
 transmitter freq.: 100.511715 MHz
 time domain size: 63750 points
 width: 24509.80 Hz = 243.8502 ppm = 0.384468 Hz/pt
 number of scans: 17000

freq. of 0 ppm: 100.501162 MHz
 processed size: 65536 complex points
 LB: 0.500 GF: 0.0000
 Hz/cm: 980.392 ppm/cm: 9.75401

Figure S14. ¹³C NMR of Product 7.

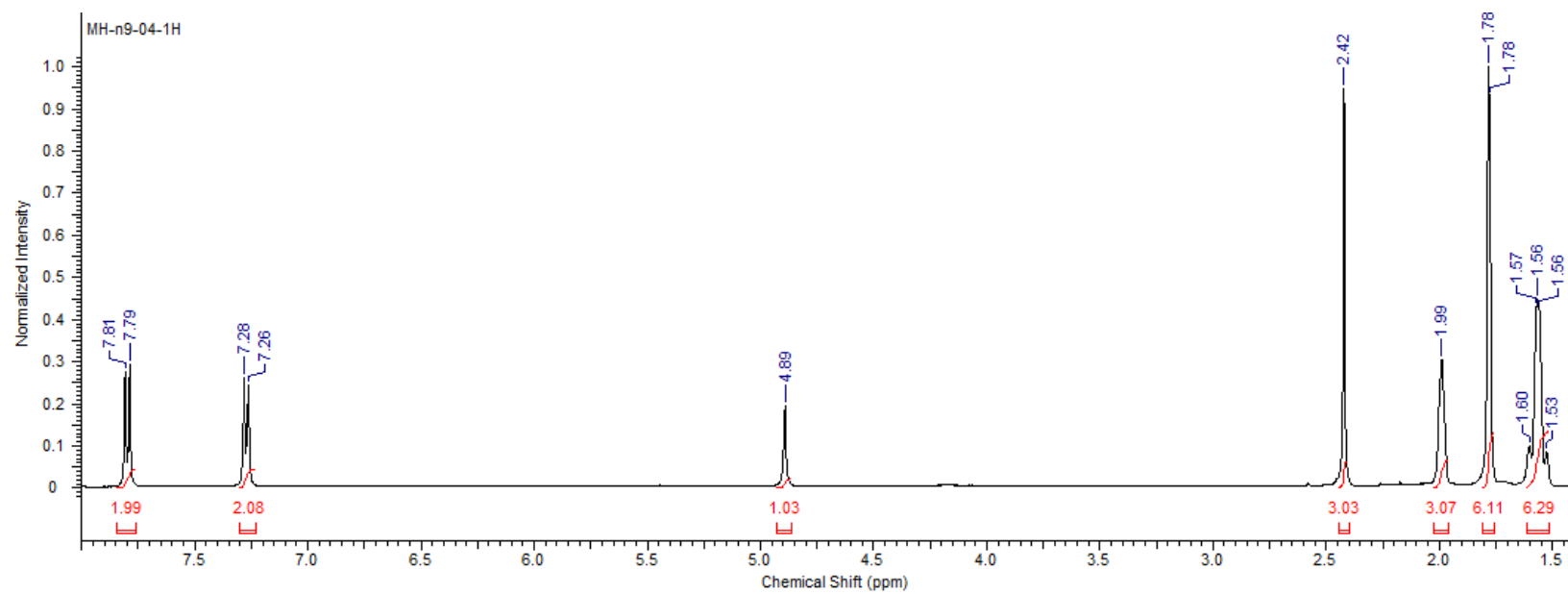
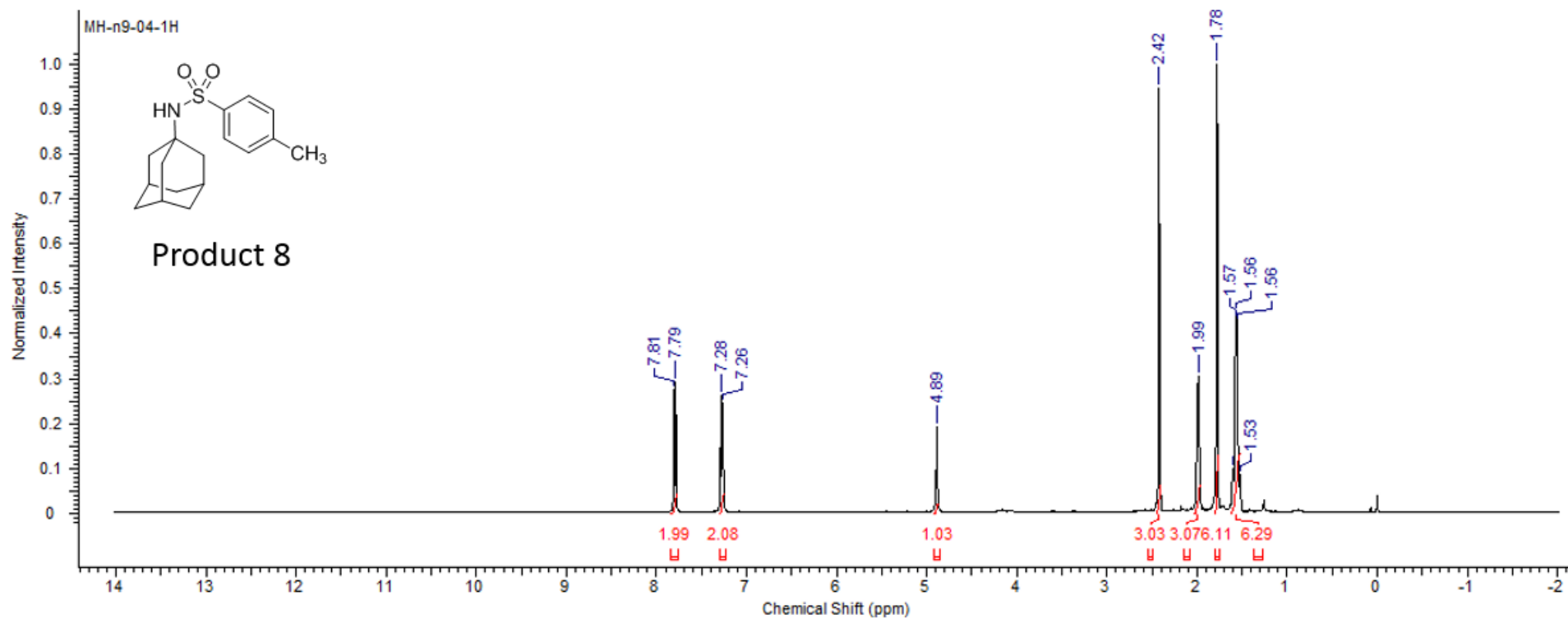
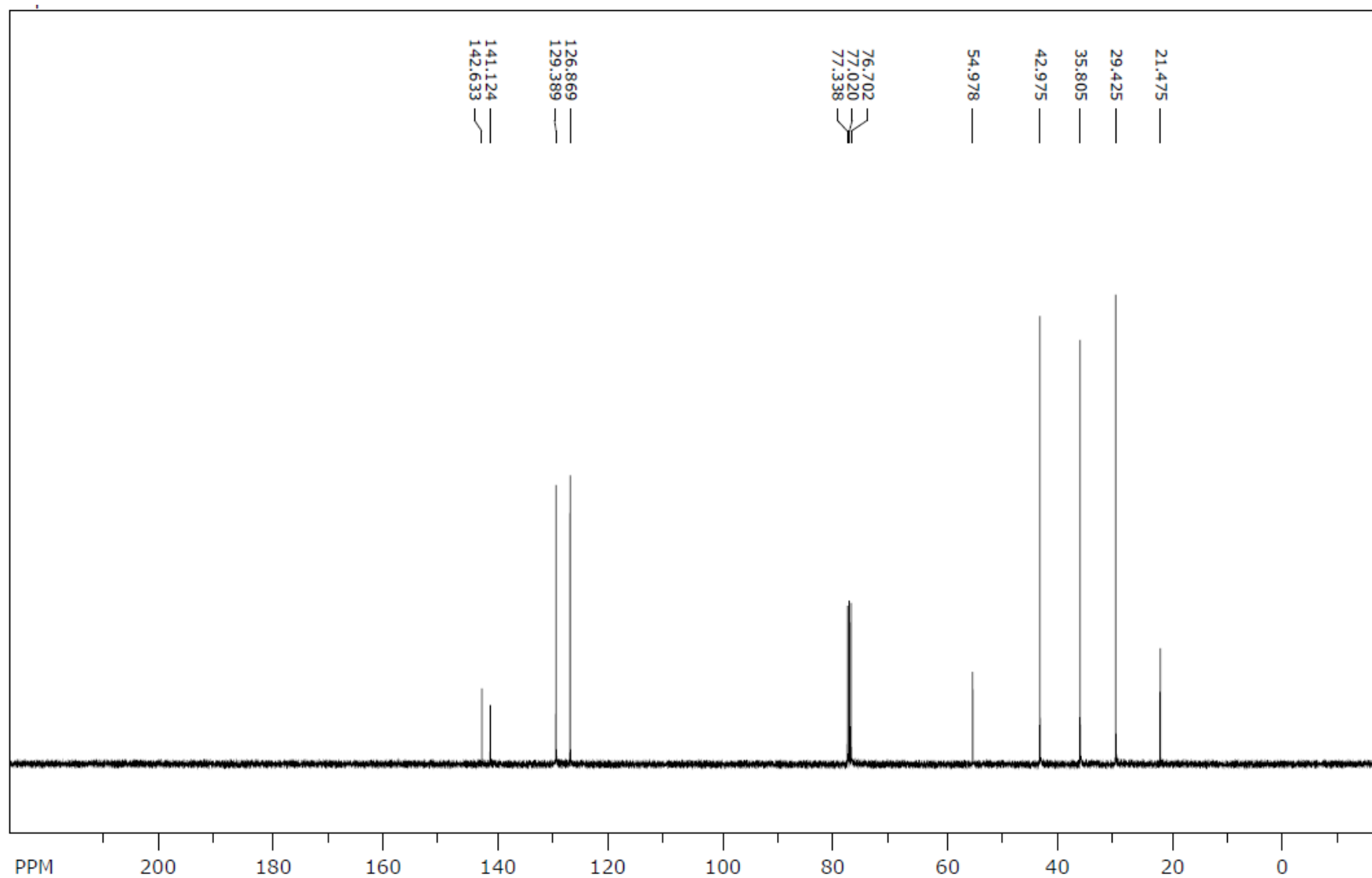


Figure S15. ^1H NMR of Product 8.



file: ...tane, 4-CH3Ph\MH-n9-04-13C.fid\fid block# 1 expt: "s2pul"
transmitter freq.: 100.511715 MHz
time domain size: 63750 points
width: 24509.80 Hz = 243.8502 ppm = 0.384468 Hz/pt
number of scans: 1120

freq. of 0 ppm: 100.501164 MHz
processed size: 65536 complex points
LB: 0.500 GF: 0.0000
Hz/cm: 980.392 ppm/cm: 9.75401

Figure S16. ¹³C NMR of Product 8.

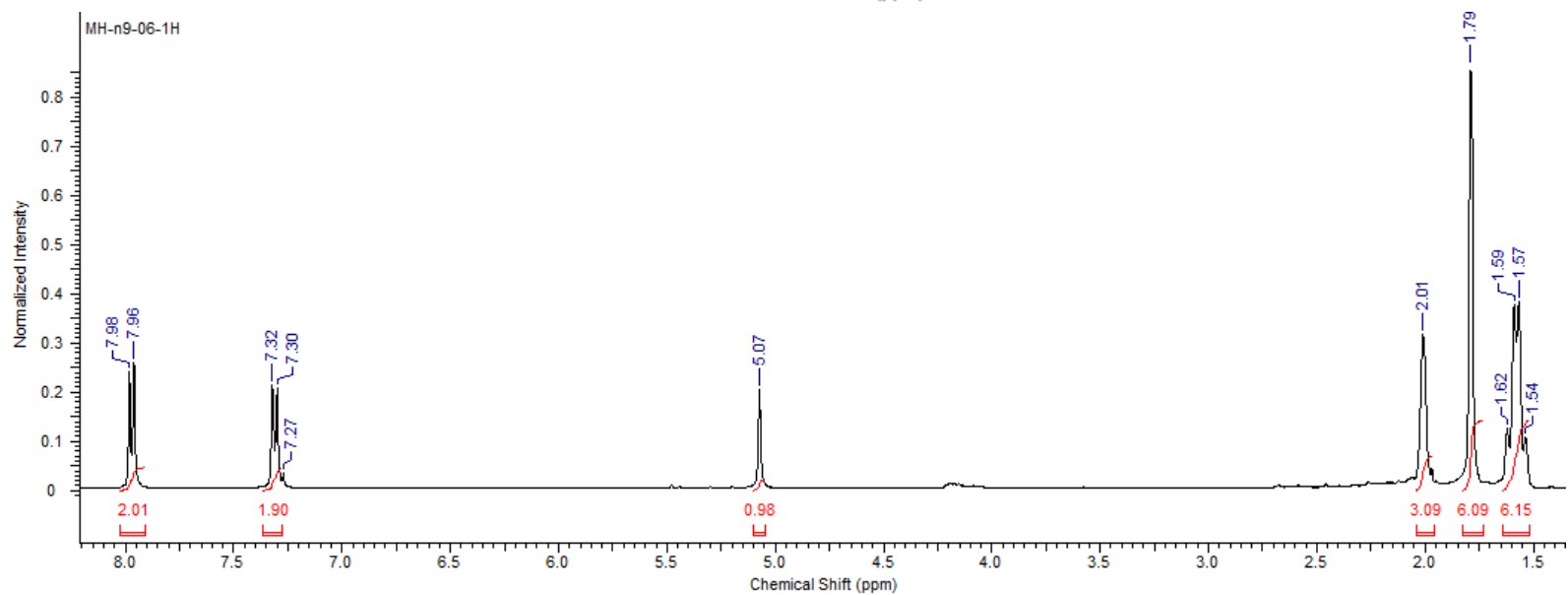
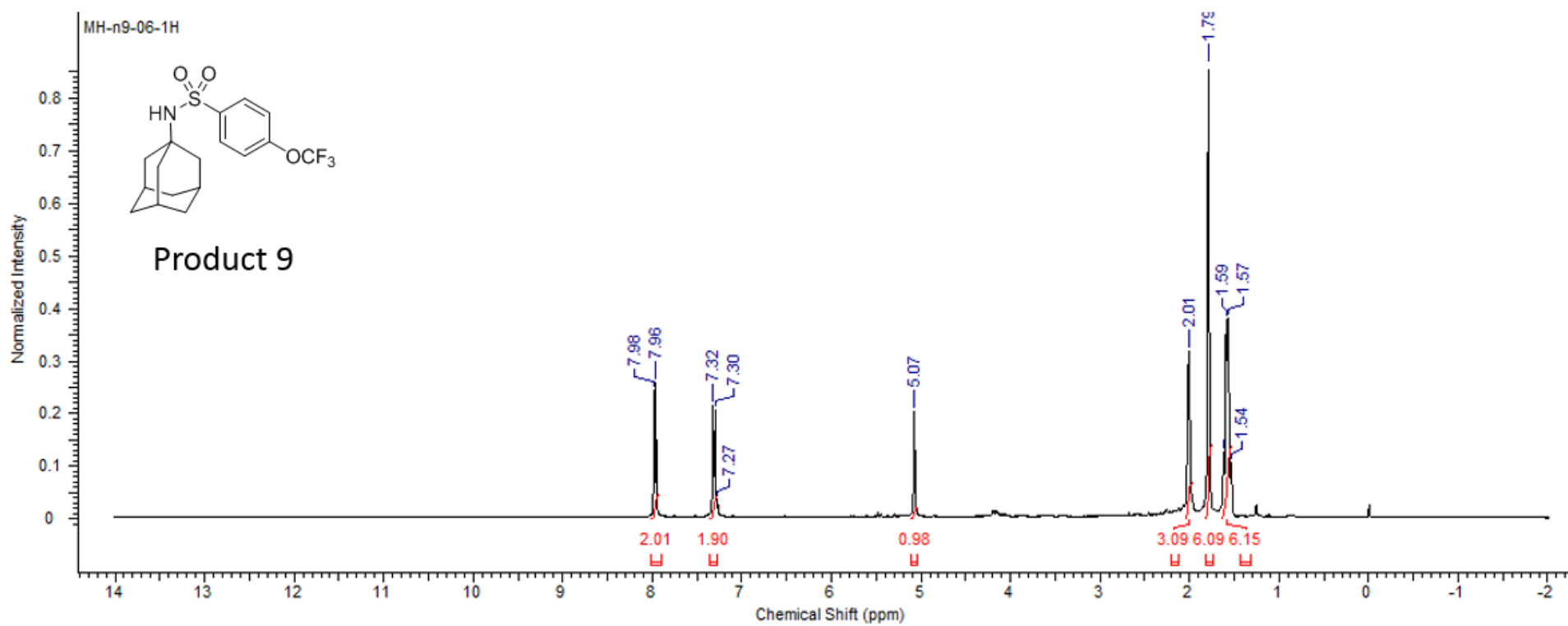
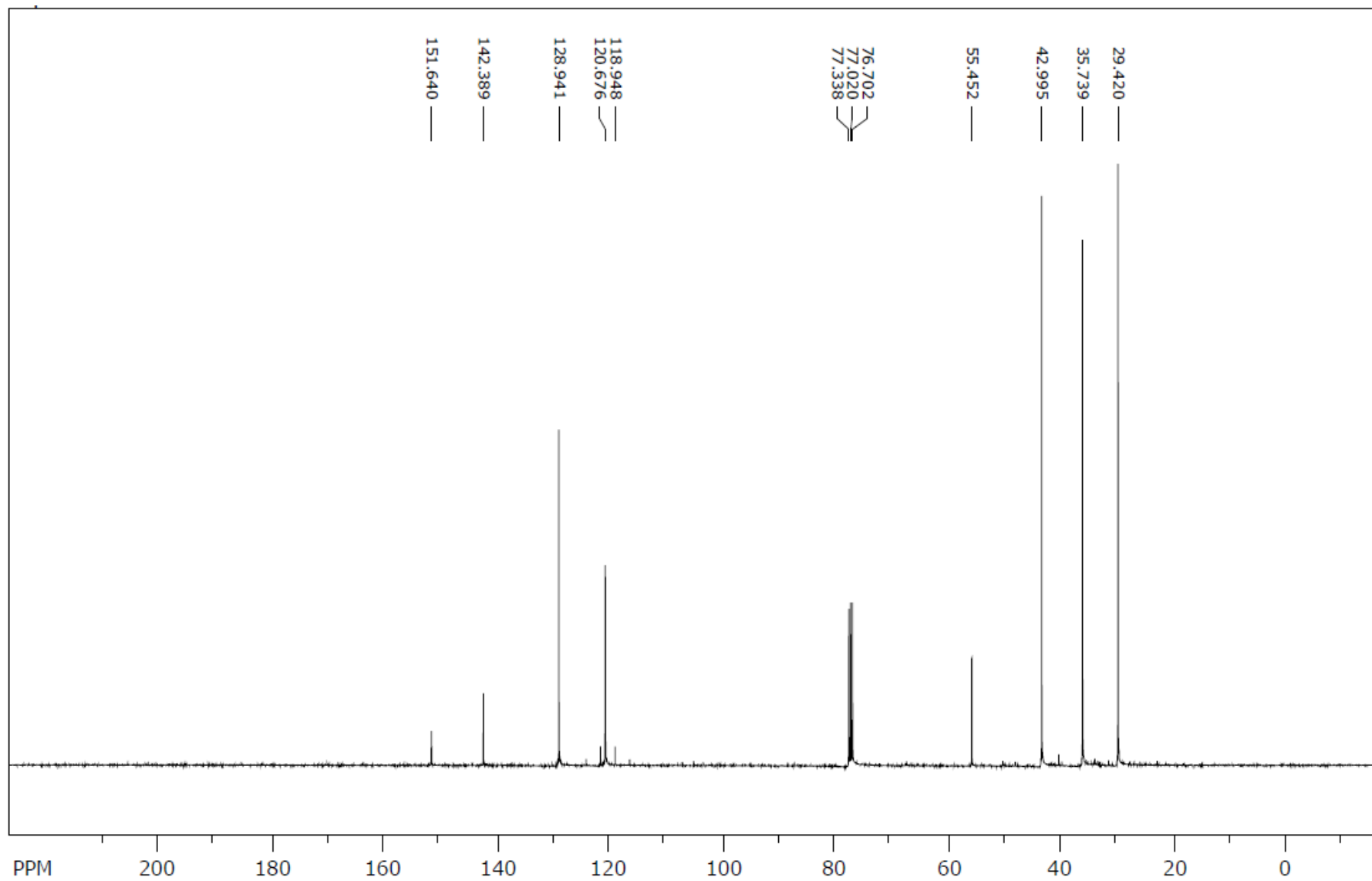


Figure S17. ^1H NMR of Product 9.



file: ...ane, 4-OCF3Ph\MH-n9-06-13C.fid\fid_block# 1 expt: "s2pul"
transmitter freq.: 100.511715 MHz
time domain size: 63750 points
width: 24509.80 Hz = 243.8502 ppm = 0.384468 Hz/pt
number of scans: 17000

freq. of 0 ppm: 100.501162 MHz
processed size: 65536 complex points
LB: 0.500 GF: 0.0000
Hz/cm: 980.392 ppm/cm: 9.75401

Figure S18. ¹³C NMR of Product 9.

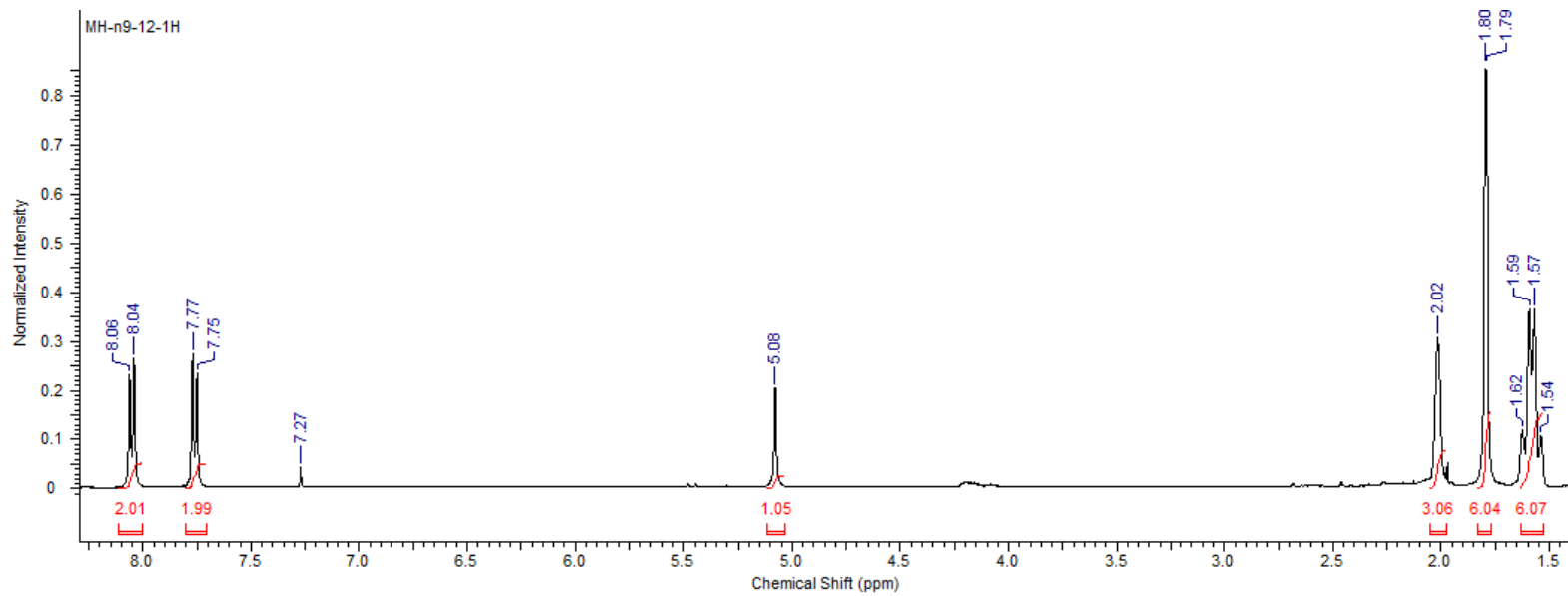
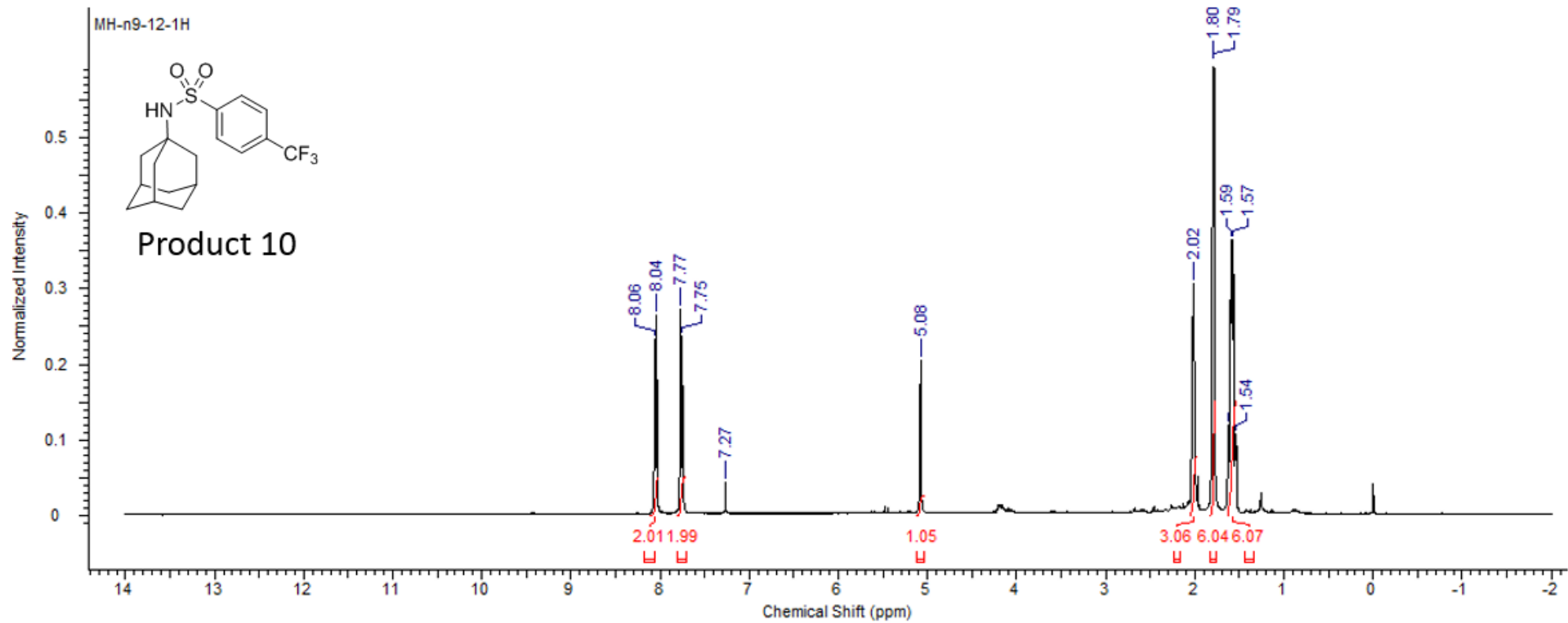
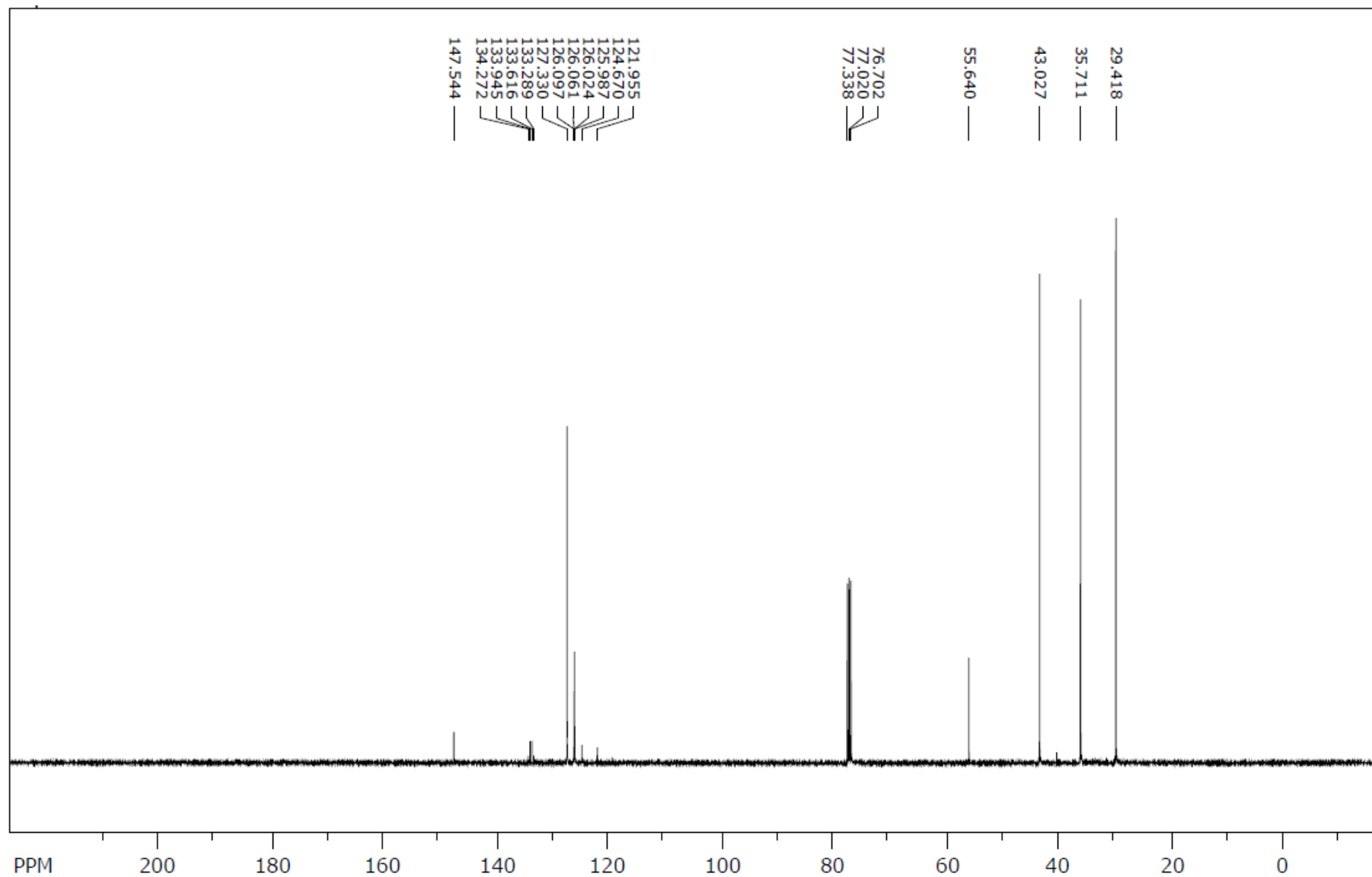


Figure S19. ^1H NMR of Product 10.



file: ...tane, 4-CF3Ph\MH-n9-12-13C.fid\fid block# 1 expt: "s2pul"
 transmitter freq.: 100.511715 MHz
 time domain size: 63750 points
 width: 24509.80 Hz = 243.8502 ppm = 0.384468 Hz/pt
 number of scans: 2496

freq. of 0 ppm: 100.501162 MHz
 processed size: 65536 complex points
 LB: 0.500 GF: 0.0000
 Hz/cm: 980.392 ppm/cm: 9.75401

Figure S20. ^{13}C NMR of Product **10**.

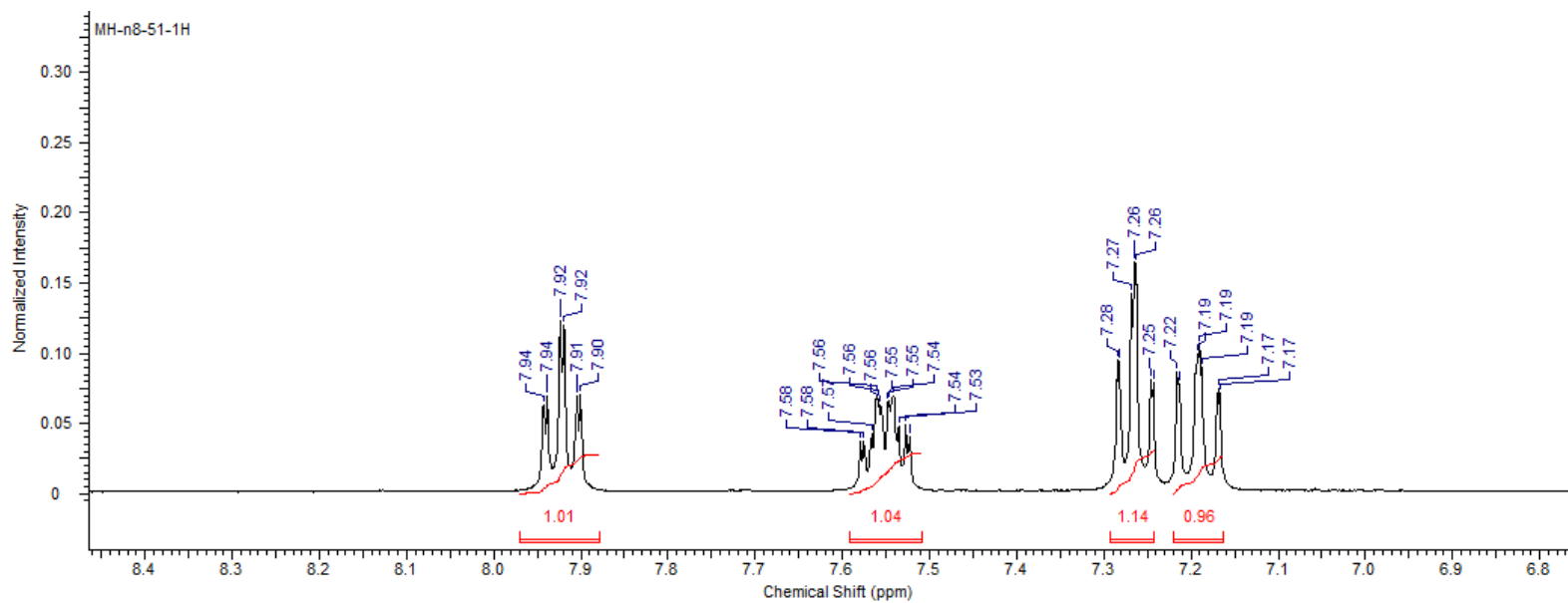
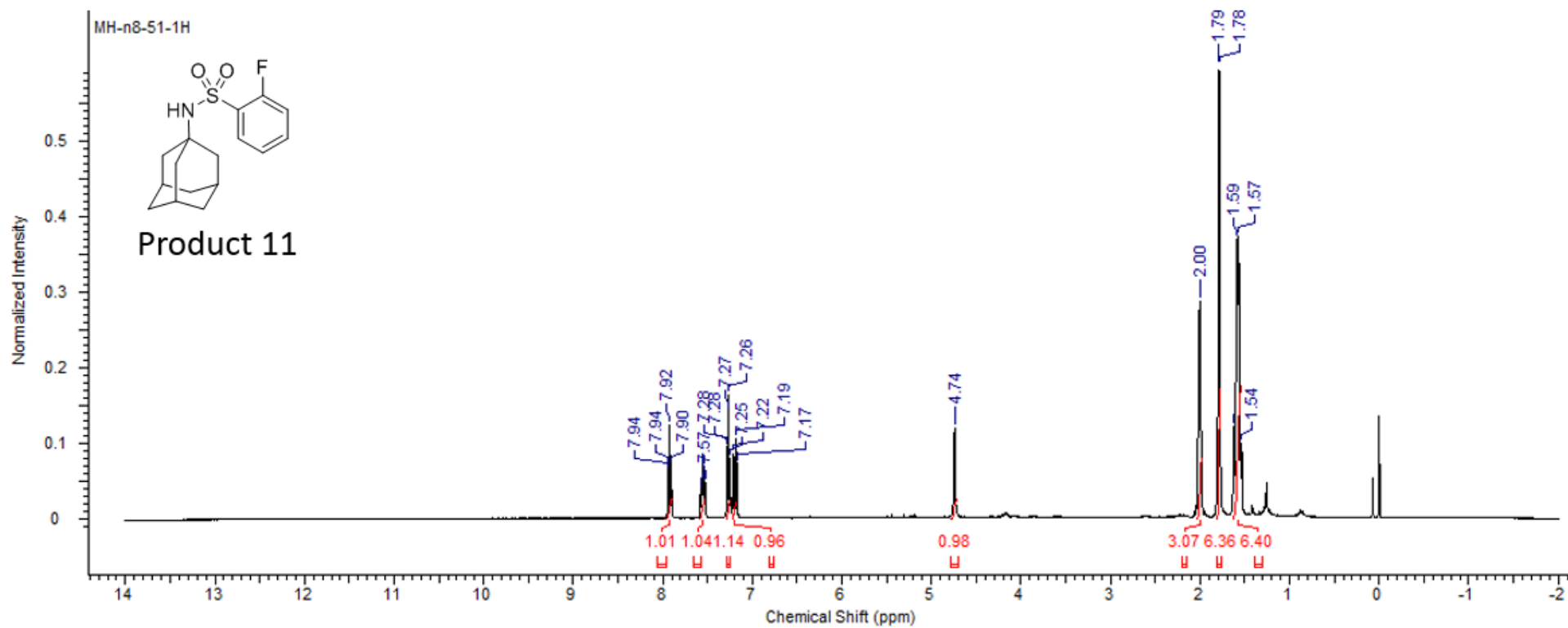
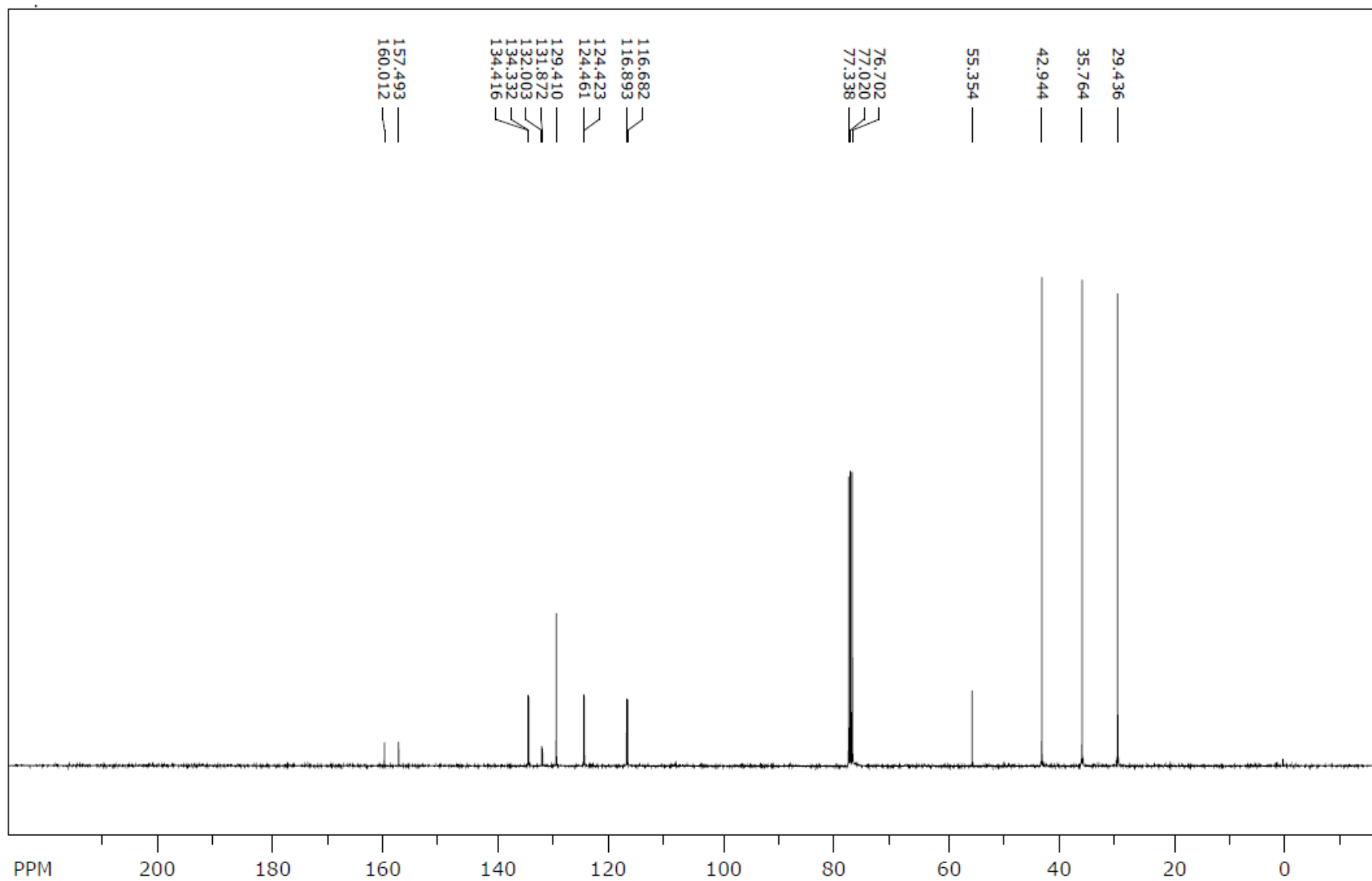


Figure S21. ^1H NMR of Product 11.



file: ...antane, 2-FPh\MH-n8-51-13C.fid\fid_block# 1 expt: "s2pul"
 transmitter freq.: 100.511715 MHz
 time domain size: 63750 points
 width: 24509.80 Hz = 243.8502 ppm = 0.384468 Hz/pt
 number of scans: 17500

freq. of 0 ppm: 100.501162 MHz
 processed size: 65536 complex points
 LB: 0.500 GF: 0.0000
 Hz/cm: 980.392 ppm/cm: 9.75401

Figure S22. ^{13}C NMR of Product **11**.

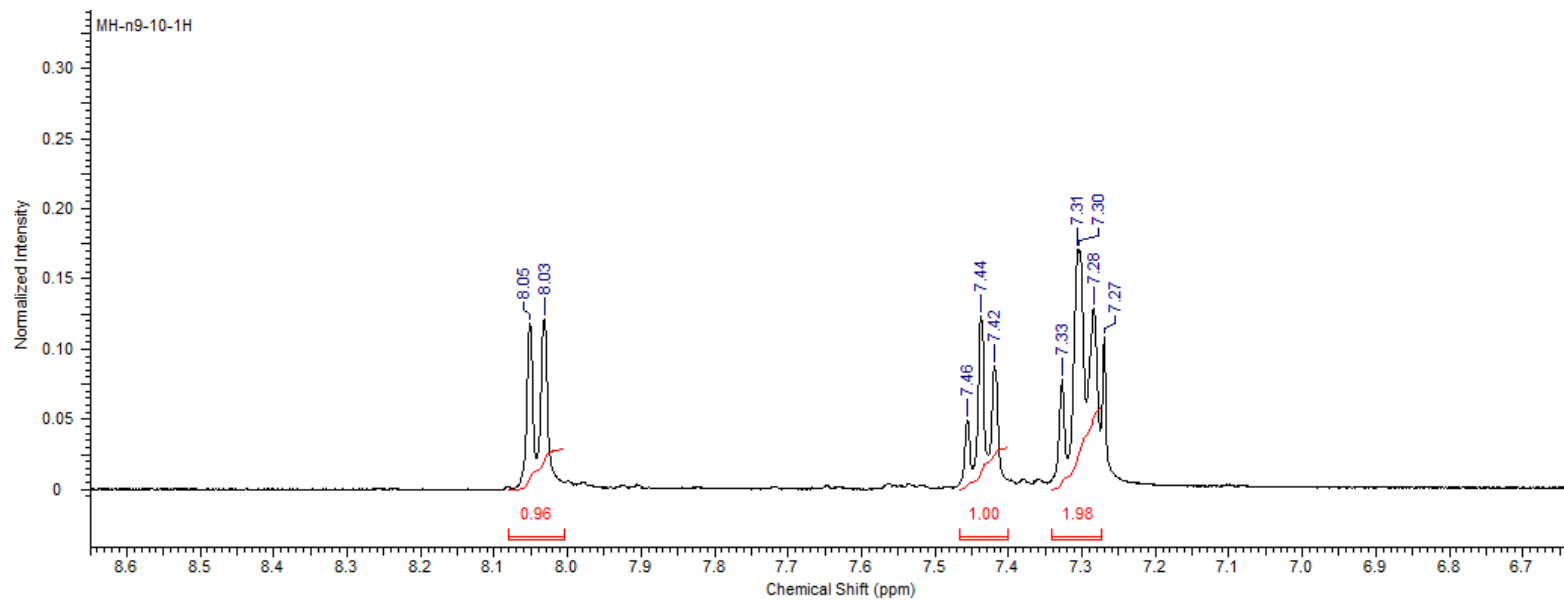
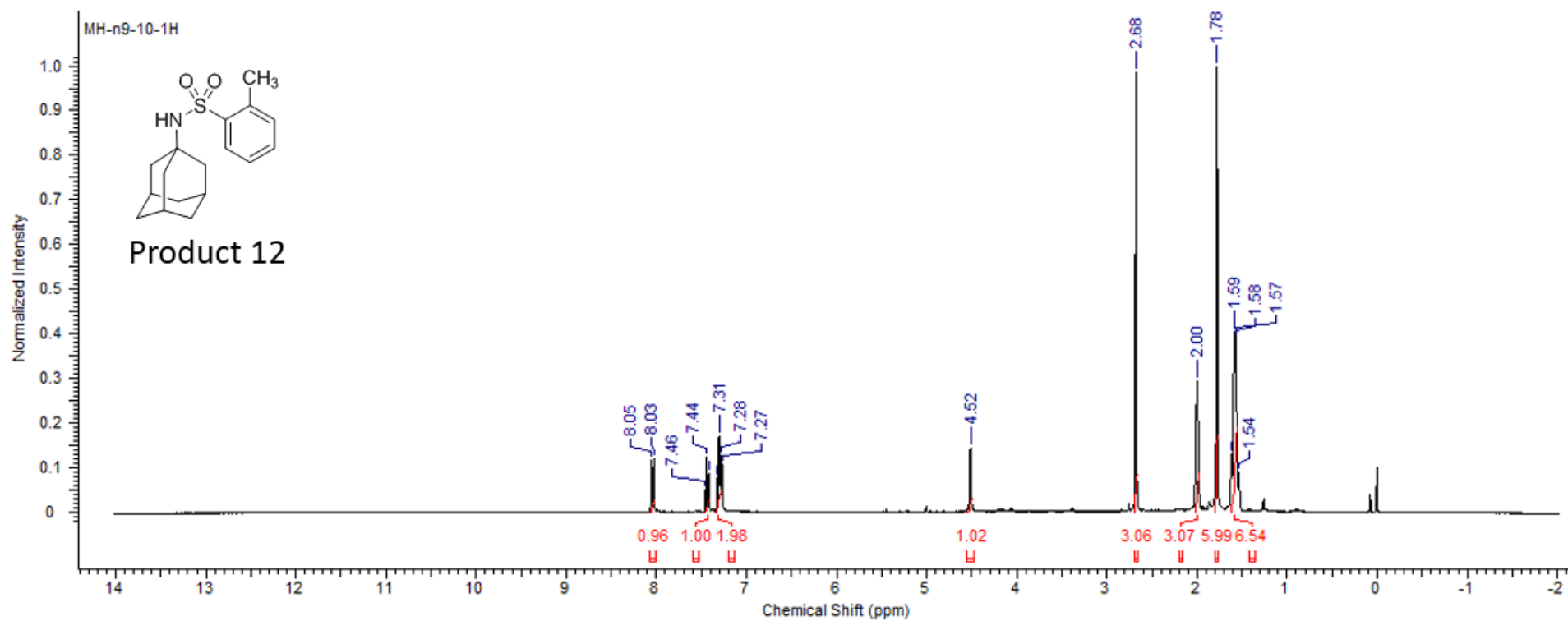
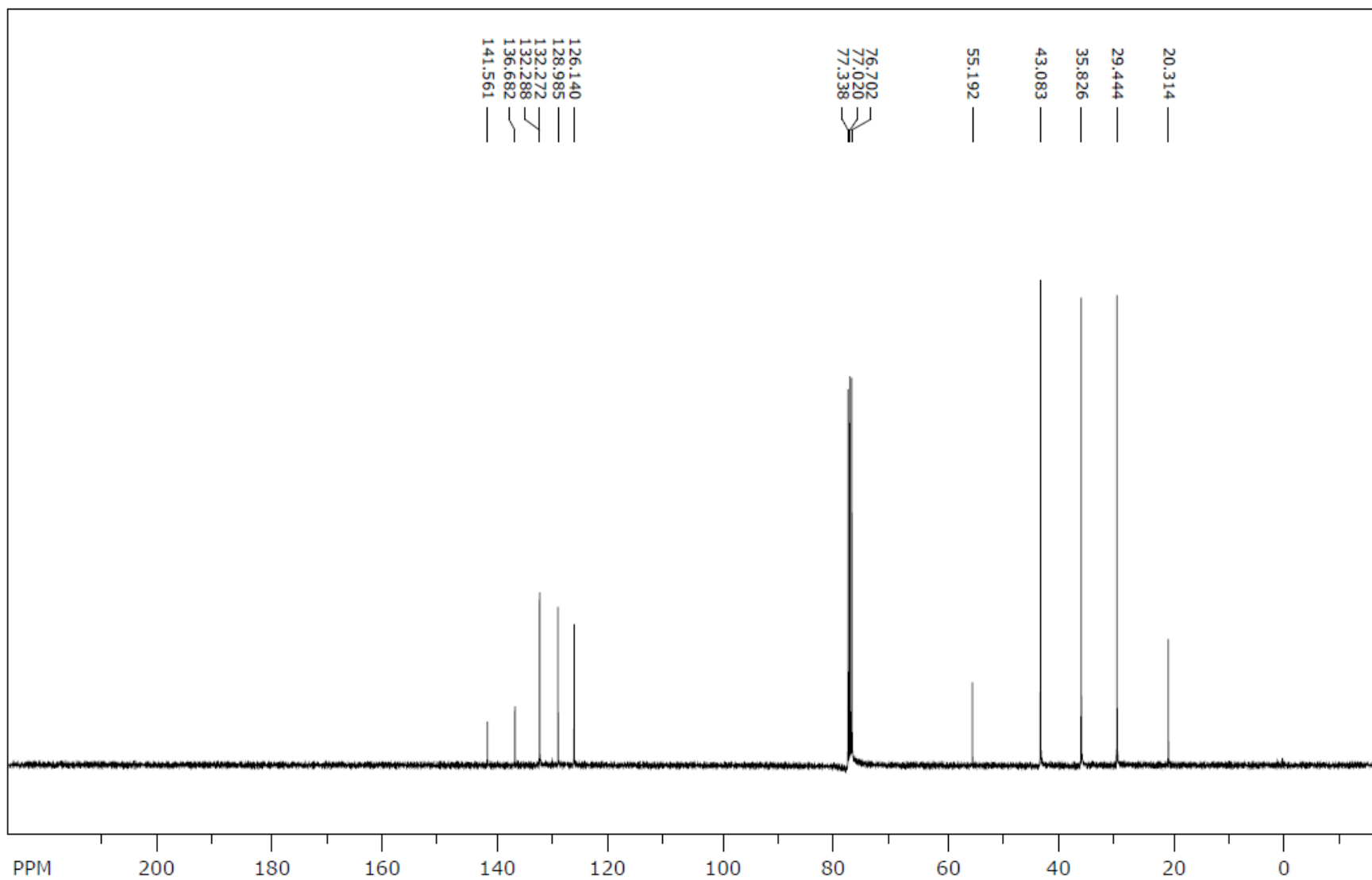


Figure S23. ^1H NMR of Product 12.



file: ...tane, 2-CH3Ph\MH-n9-10-13C.fid\fid block# 1 expt: "s2pul"
 transmitter freq.: 100.511715 MHz
 time domain size: 63750 points
 width: 24509.80 Hz = 243.8502 ppm = 0.384468 Hz/pt
 number of scans: 17000

freq. of 0 ppm: 100.501161 MHz
 processed size: 65536 complex points
 LB: 0.500 GF: 0.0000
 Hz/cm: 980.392 ppm/cm: 9.75401

Figure S24. ¹³C NMR of Product 12.

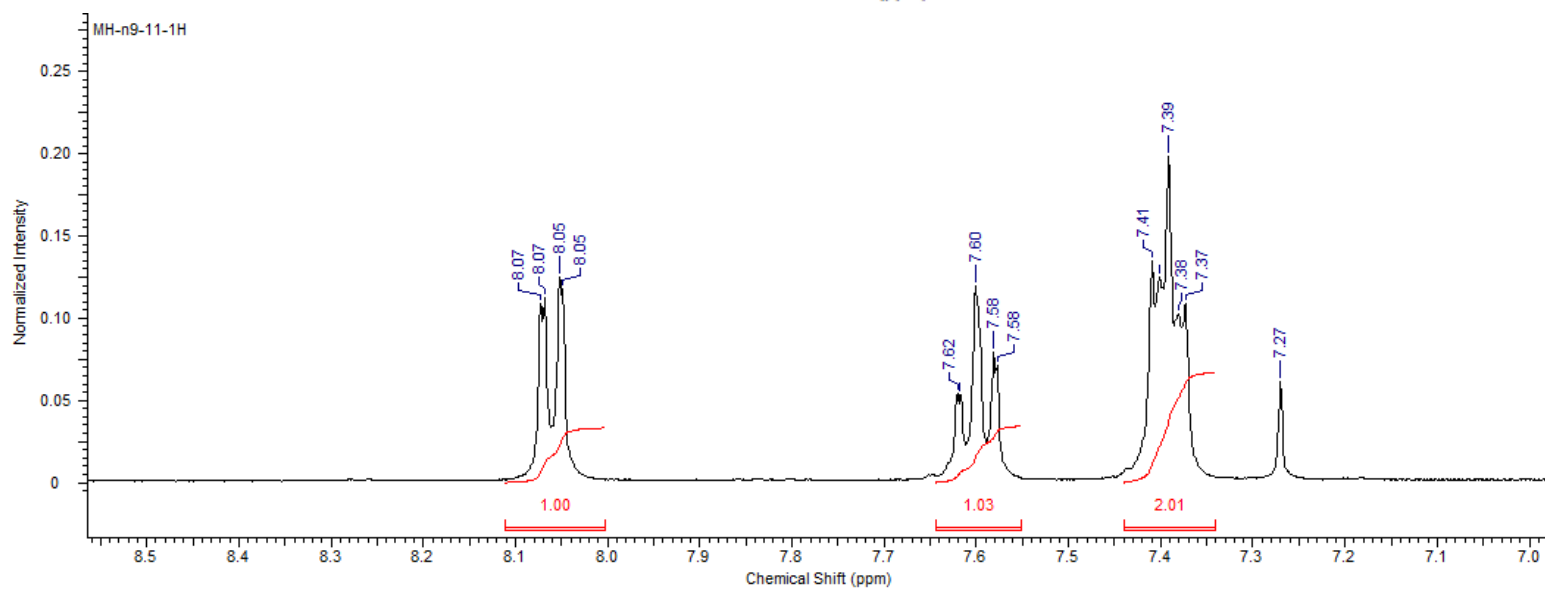
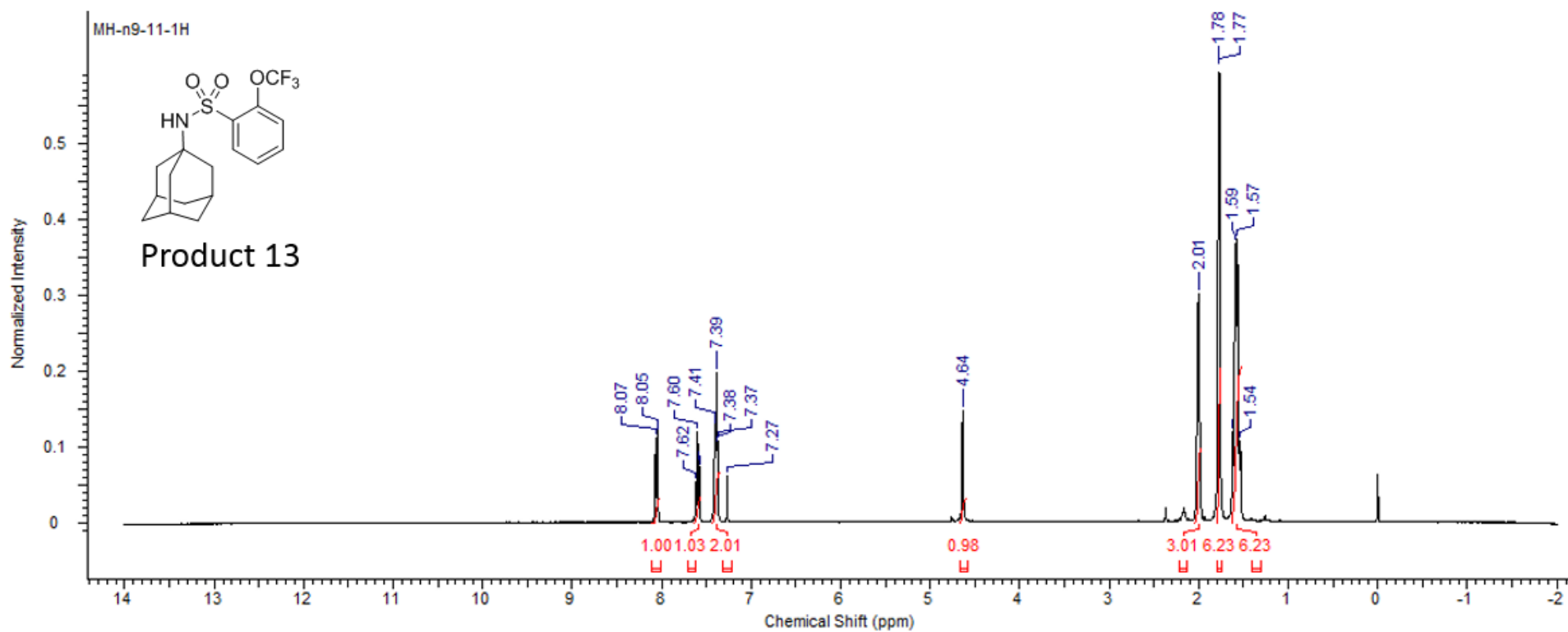
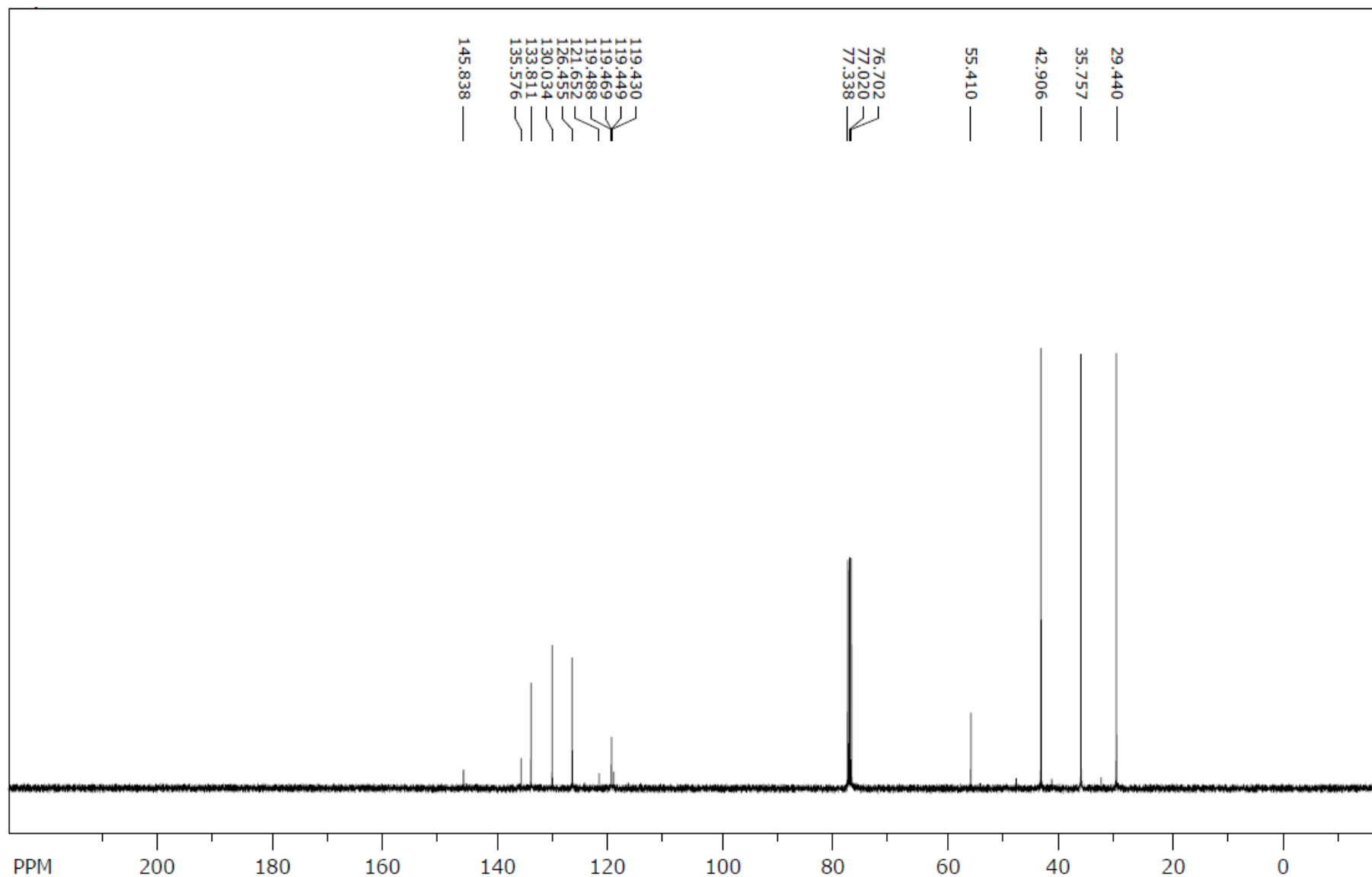


Figure S25. ^1H NMR of Product 13.



file: ...Data\NMR Fids\MH-n9-11-13C.fid\fid_block# 1 expt: "s2pul"
 transmitter freq.: 100.511715 MHz
 time domain size: 63750 points
 width: 24509.80 Hz = 243.8502 ppm = 0.384468 Hz/pt
 number of scans: 1296

freq. of 0 ppm: 100.501161 MHz
 processed size: 65536 complex points
 LB: 0.500 GF: 0.0000
 Hz/cm: 980.392 ppm/cm: 9.75401

Figure S26. ¹³C NMR of Product 13.

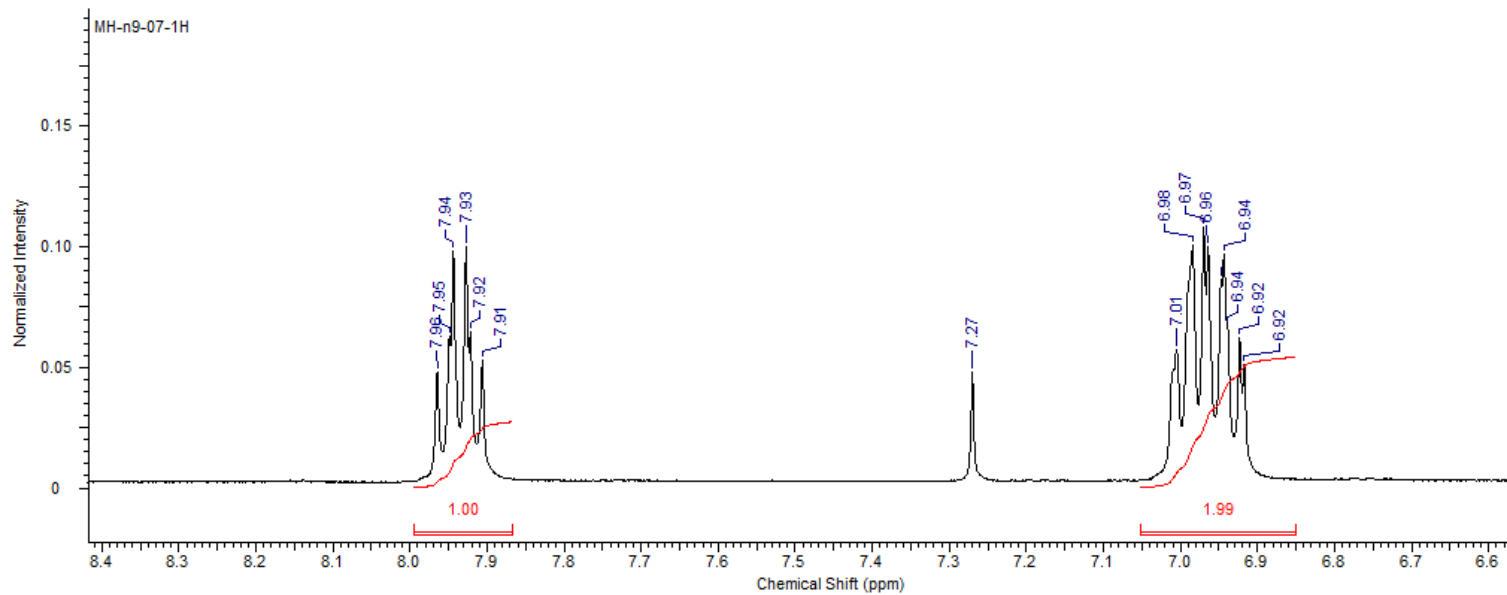
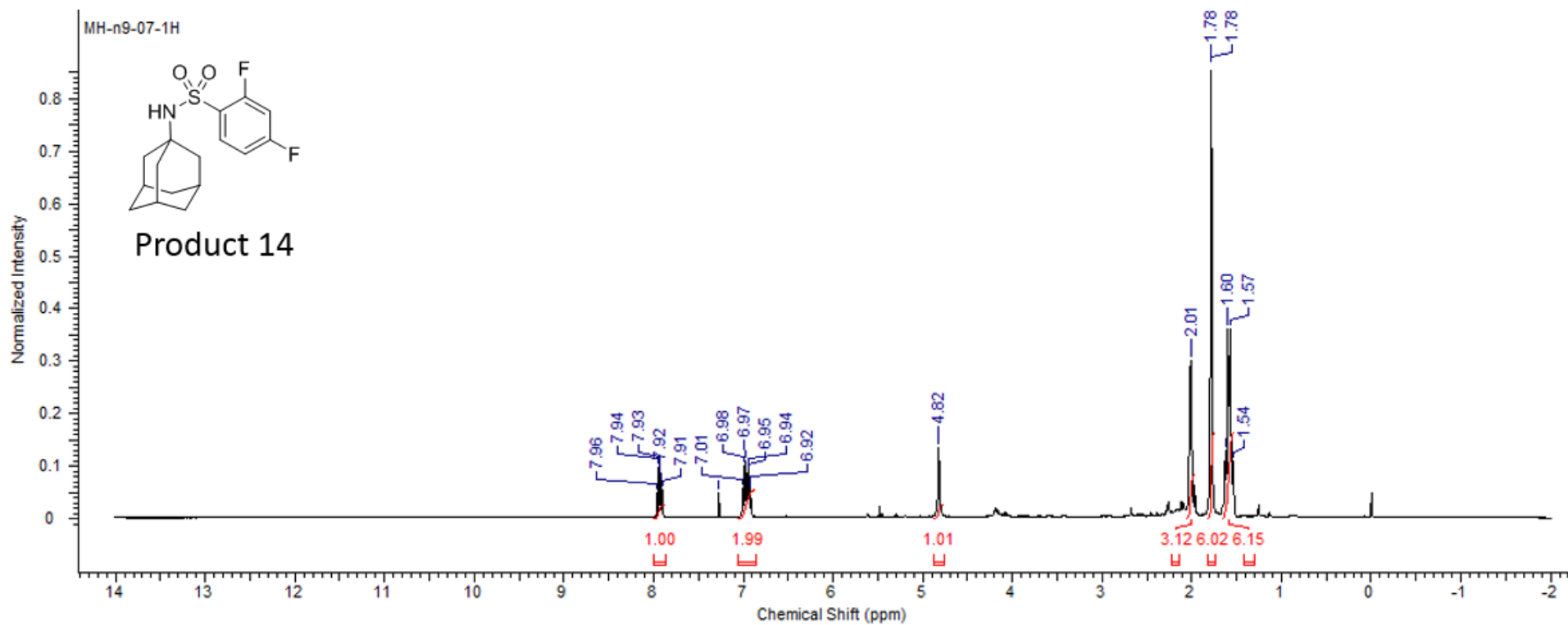
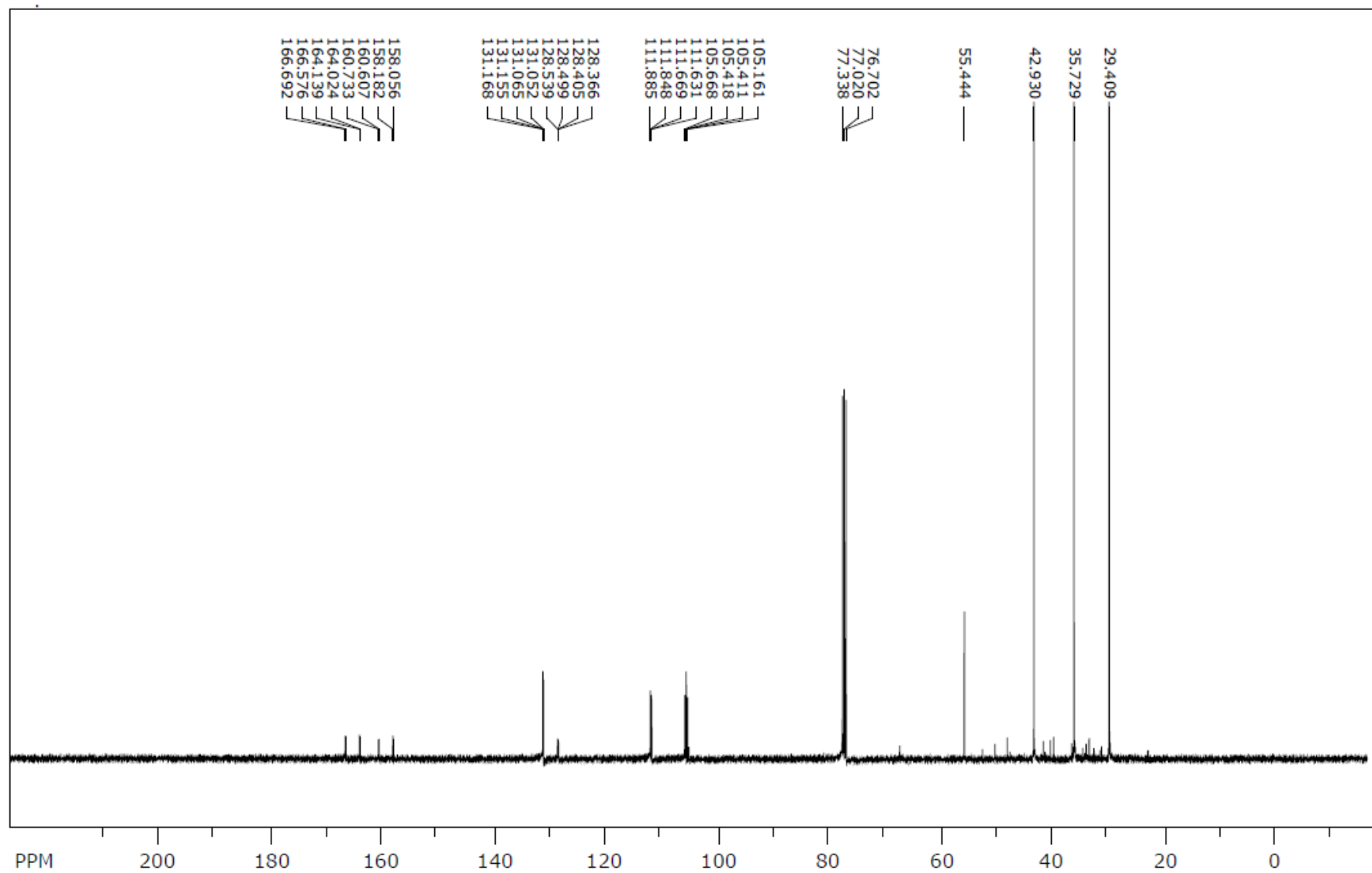


Figure S27. ^1H NMR of Product 14.



file: ...ne, 2,4-diFPh\MH-n9-07-13C.fid\fid block# 1 expt: "s2pul"
 transmitter freq.: 100.511715 MHz
 time domain size: 63750 points
 width: 24509.80 Hz = 243.8502 ppm = 0.384468 Hz/pt
 number of scans: 2448

freq. of 0 ppm: 100.501163 MHz
 processed size: 65536 complex points
 LB: 0.500 GF: 0.0000
 Hz/cm: 980.392 ppm/cm: 9.75401

Figure S28. ^{13}C NMR and ^1H NMR of Product **14**.

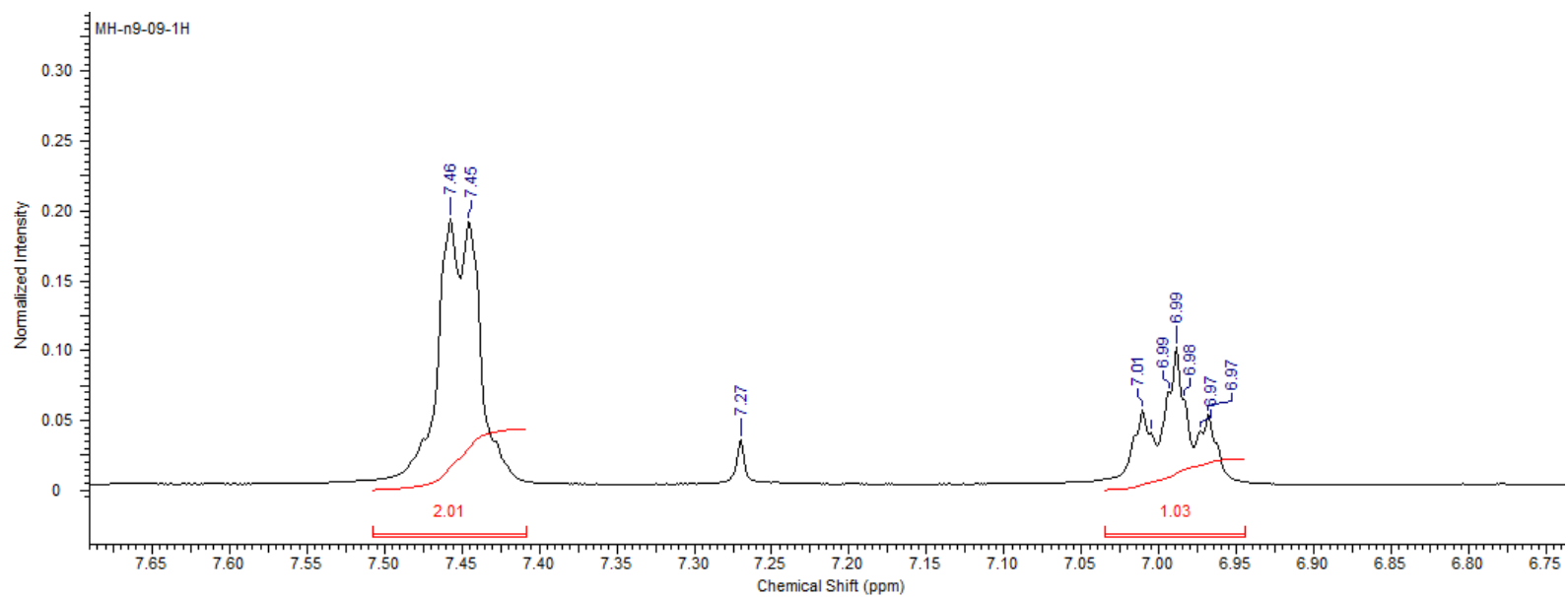
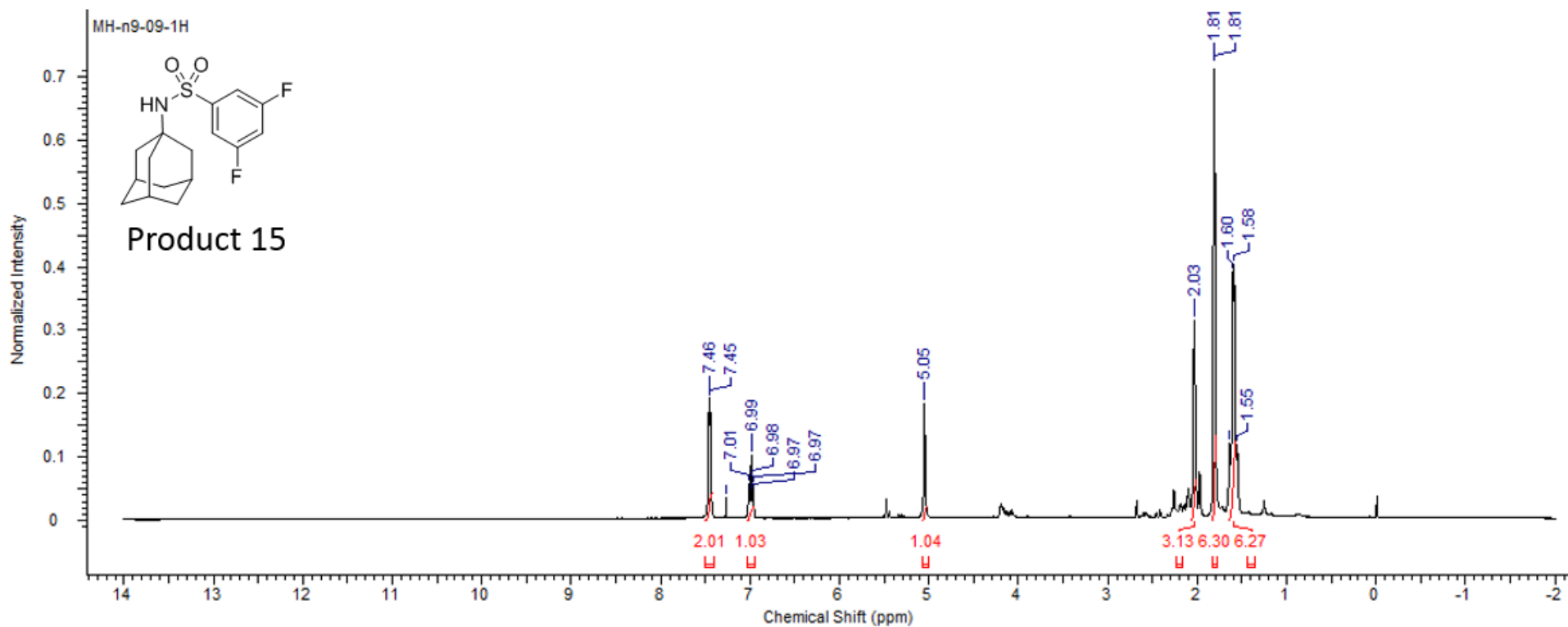
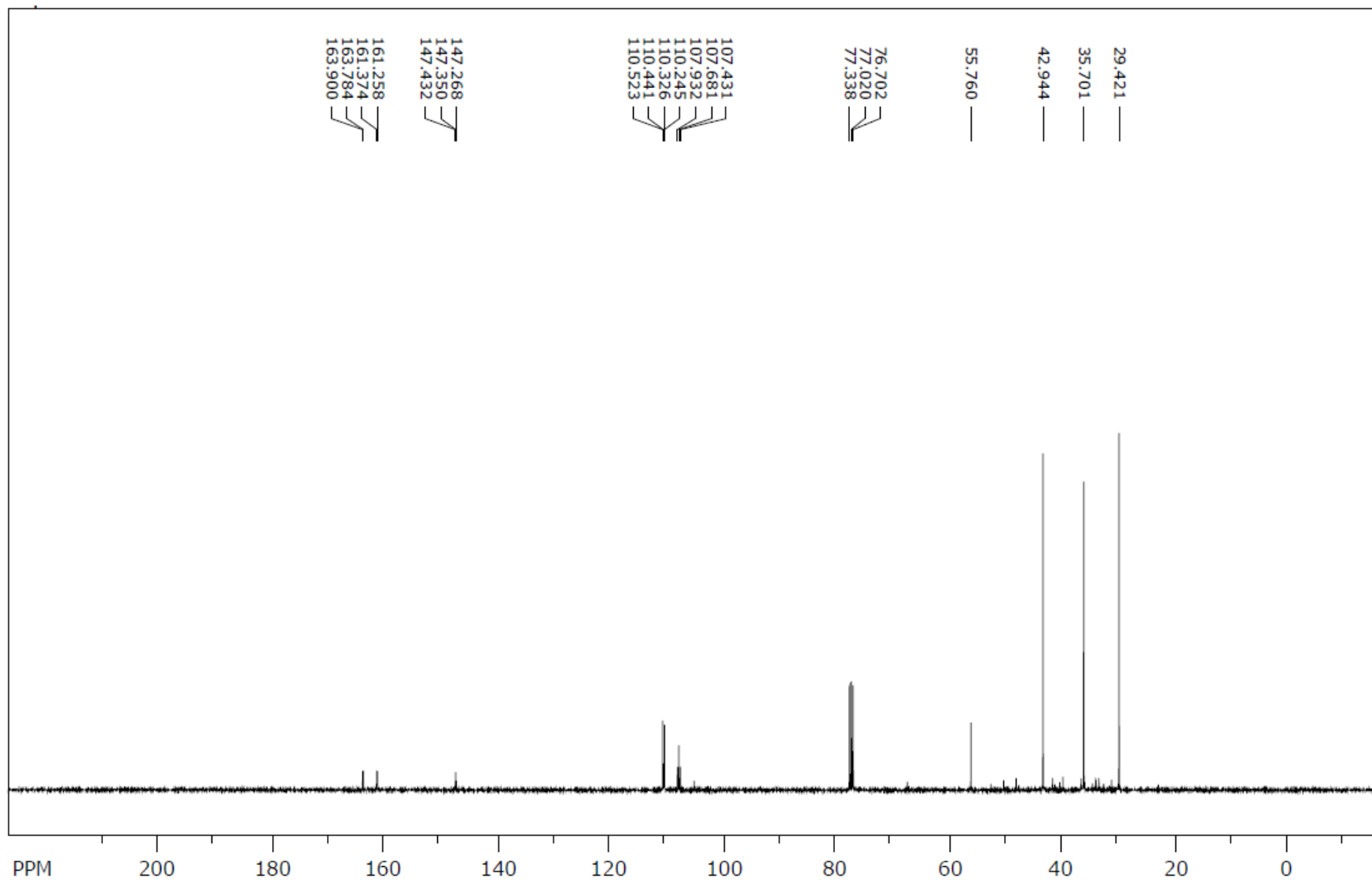


Figure S29. ¹H NMR of Product 15.



file: ...Data\NMR Fids\MH-n9-09-13C.fid\fid_block# 1 expt: "s2pul"
 transmitter freq.: 100.511715 MHz
 time domain size: 63750 points
 width: 24509.80 Hz = 243.8502 ppm = 0.384468 Hz/pt
 number of scans: 1360

freq. of 0 ppm: 100.501163 MHz
 processed size: 65536 complex points
 LB: 0.500 GF: 0.0000
 Hz/cm: 980.392 ppm/cm: 9.75401

Figure S30. ¹³C NMR of Product 15.

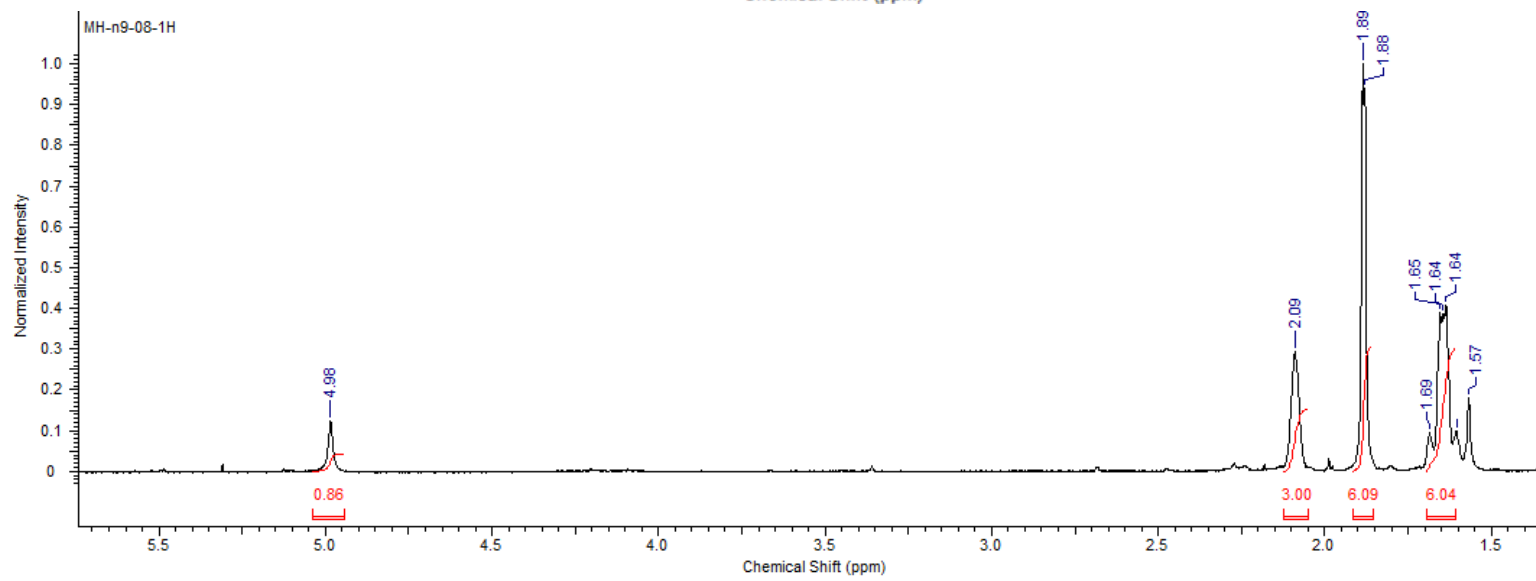
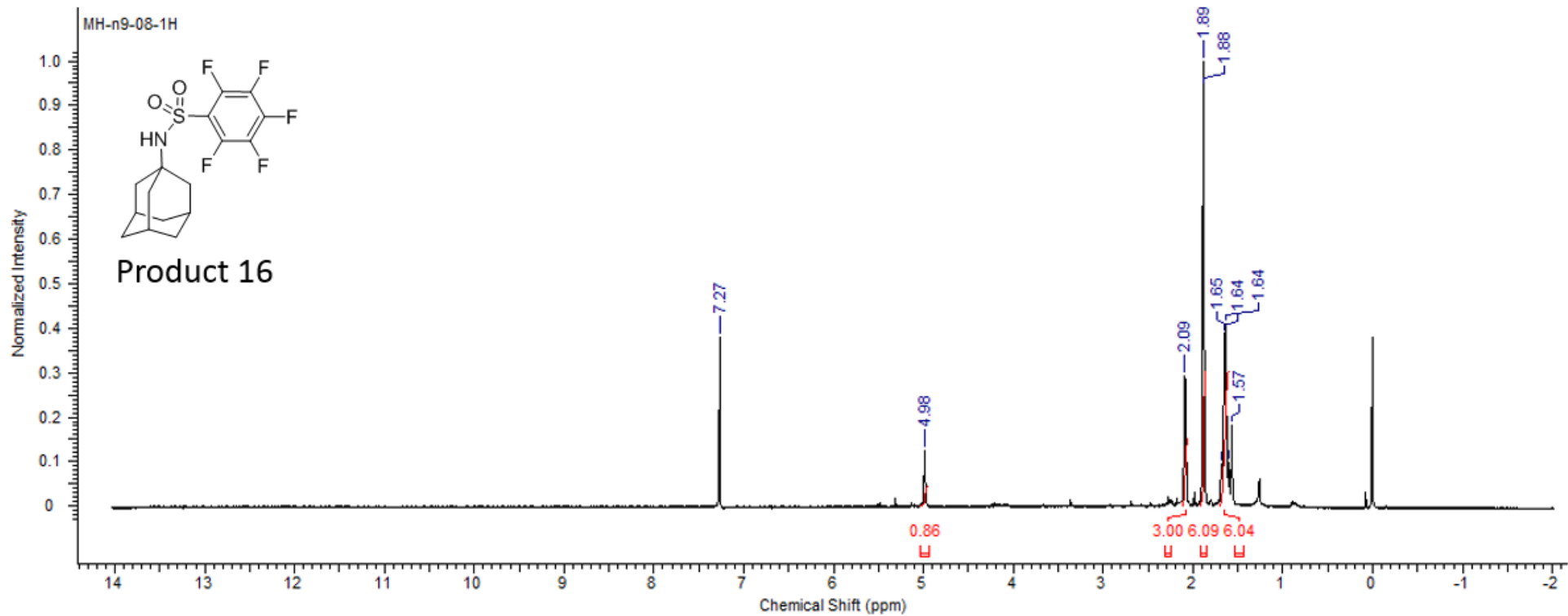
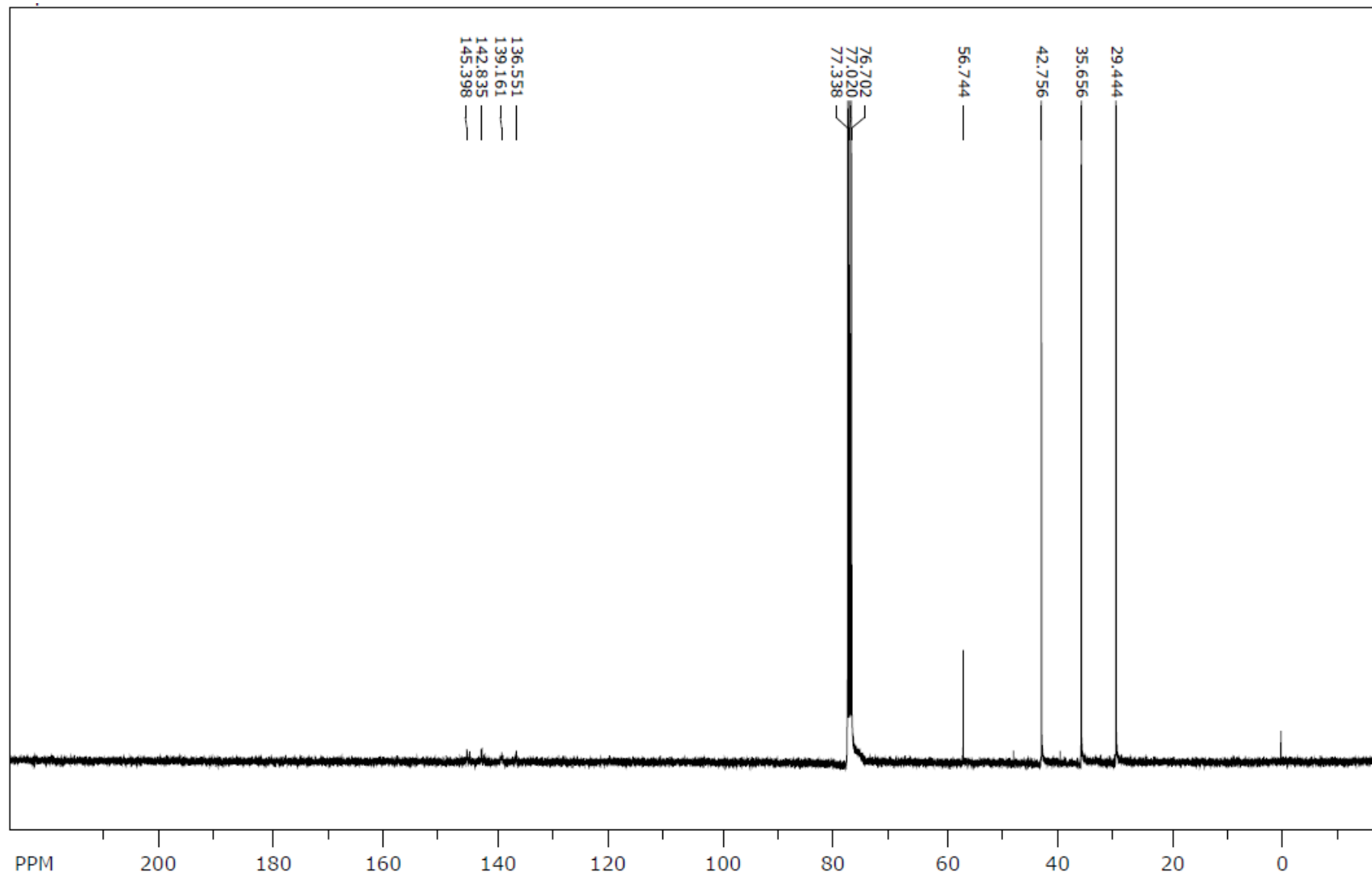


Figure S31. ^1H NMR of Product 16.



file: ...,5,6-pentaFPh\MH-n9-08-13C.fid\fid block# 1 expt: "s2pul"
transmitter freq.: 100.511715 MHz
time domain size: 63750 points
width: 24509.80 Hz = 243.8502 ppm = 0.384468 Hz/pt
number of scans: 18000

freq. of 0 ppm: 100.501160 MHz
processed size: 65536 complex points
LB: 0.500 GF: 0.0000
Hz/cm: 980.392 ppm/cm: 9.75401

Figure S32. ¹³C NMR of Product 16.

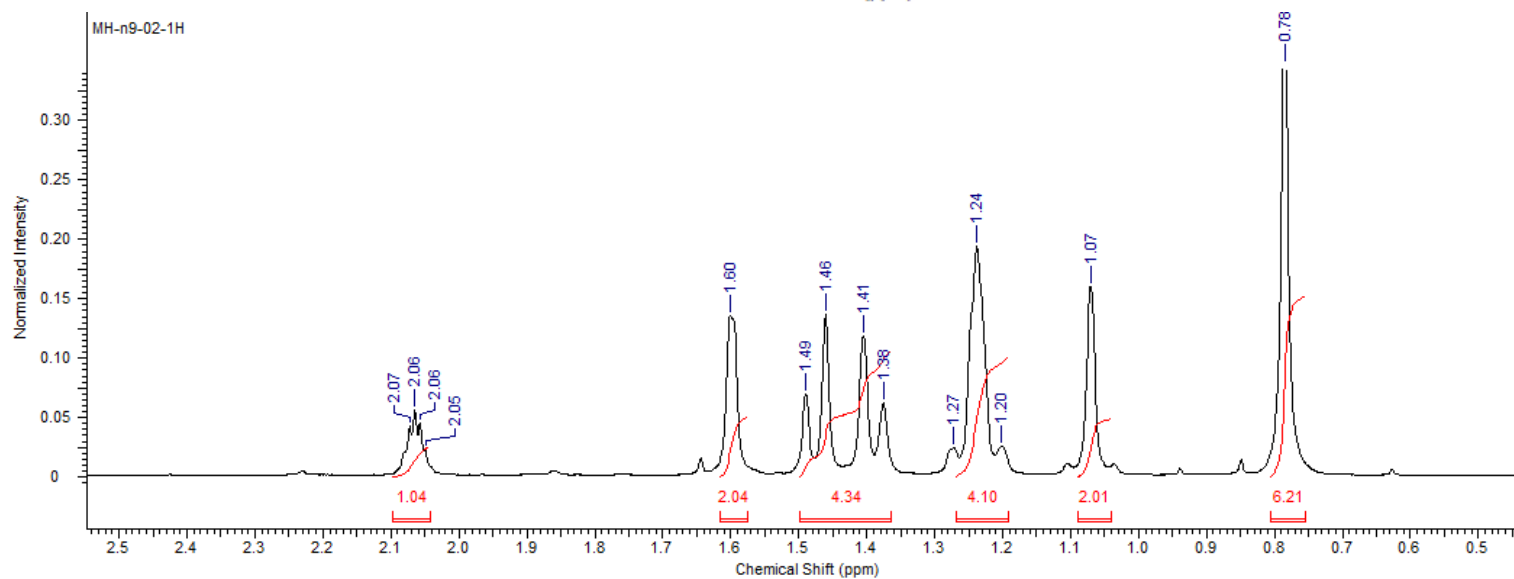
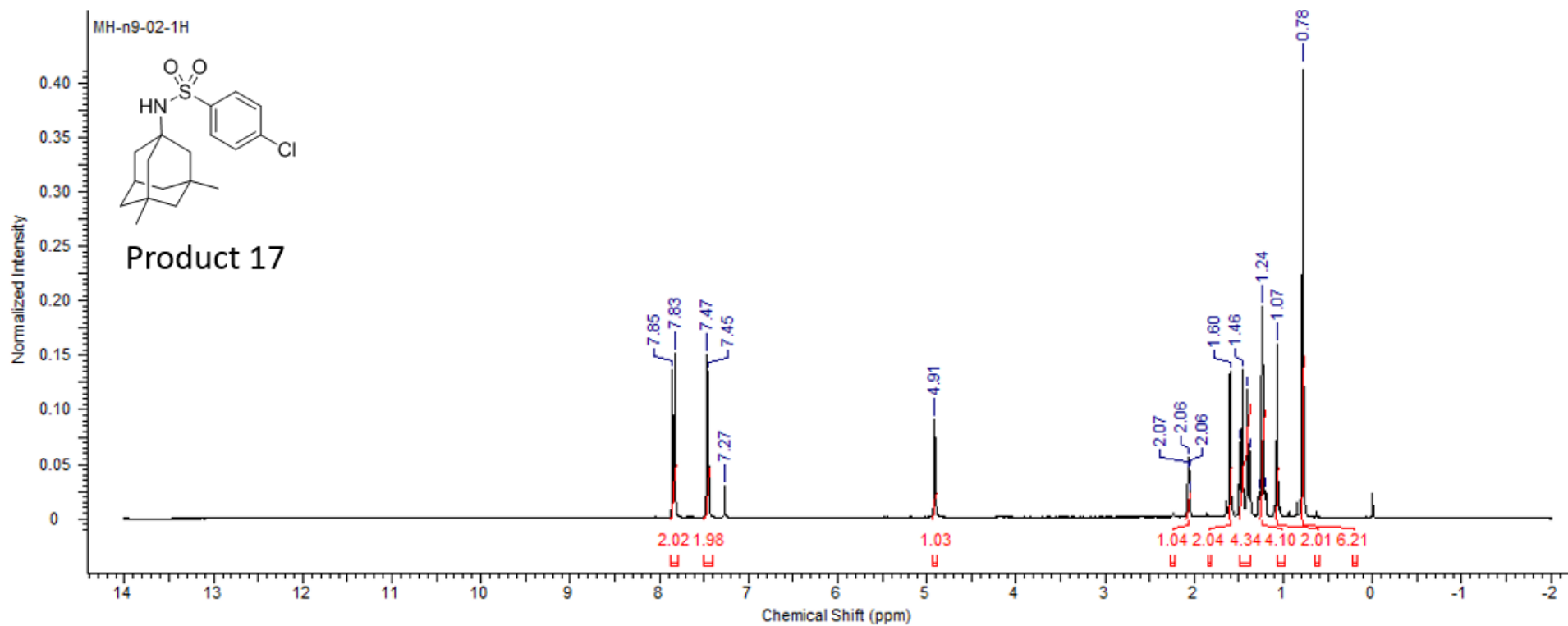
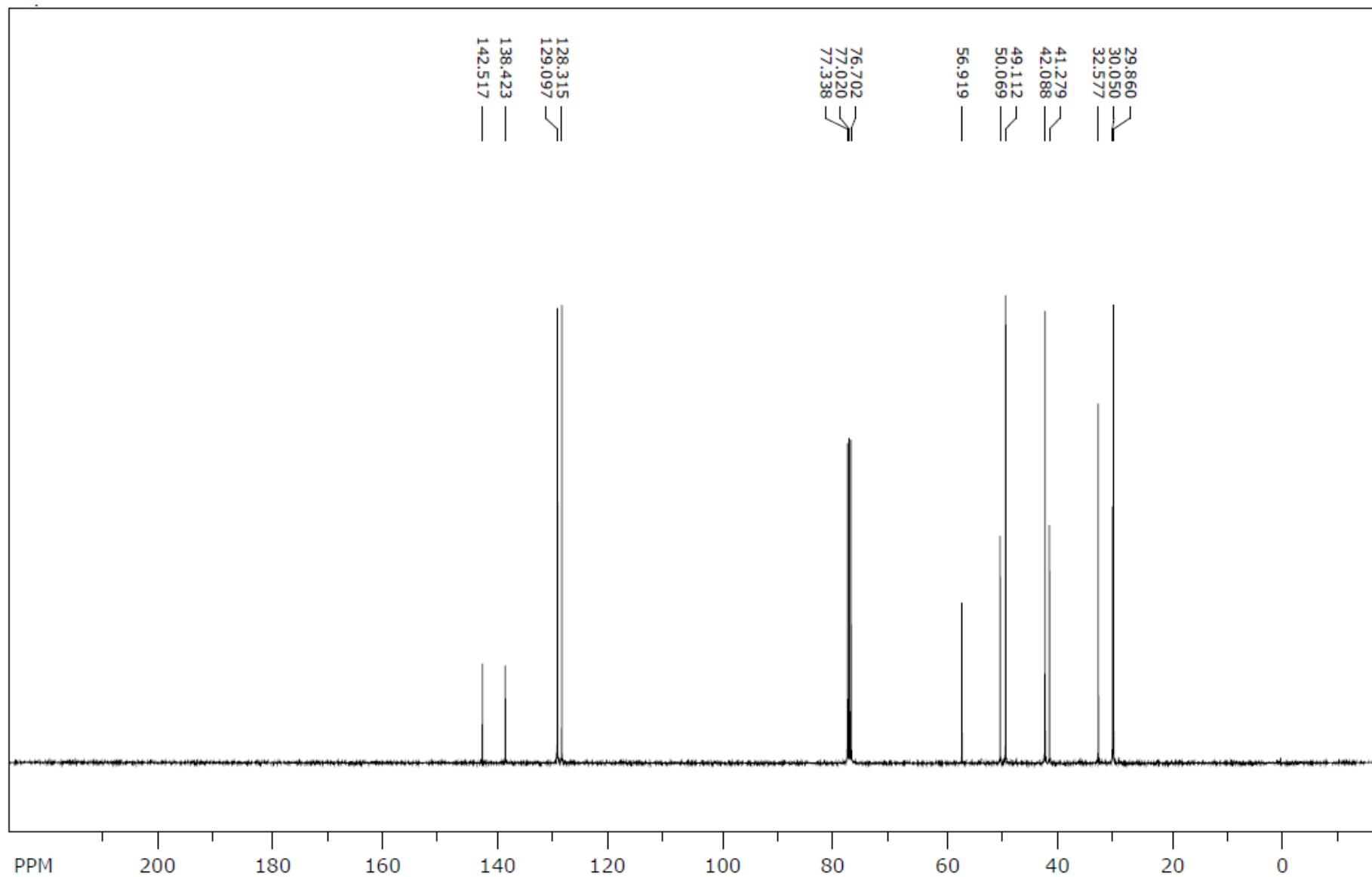


Figure S33. ^1H NMR of Product 17.



file: ...ntane, 4-ClPh\MH-n9-02-13C.fid\fid_block# 1_expt: "s2pul"
 transmitter freq.: 100.511715 MHz
 time domain size: 63750 points
 width: 24509.80 Hz = 243.8502 ppm = 0.384468 Hz/pt
 number of scans: 15000

freq. of 0 ppm: 100.501162 MHz
 processed size: 65536 complex points
 LB: 0.500 GF: 0.0000
 Hz/cm: 980.392 ppm/cm: 9.75401

Figure S34. ¹³C NMR of Product 17.

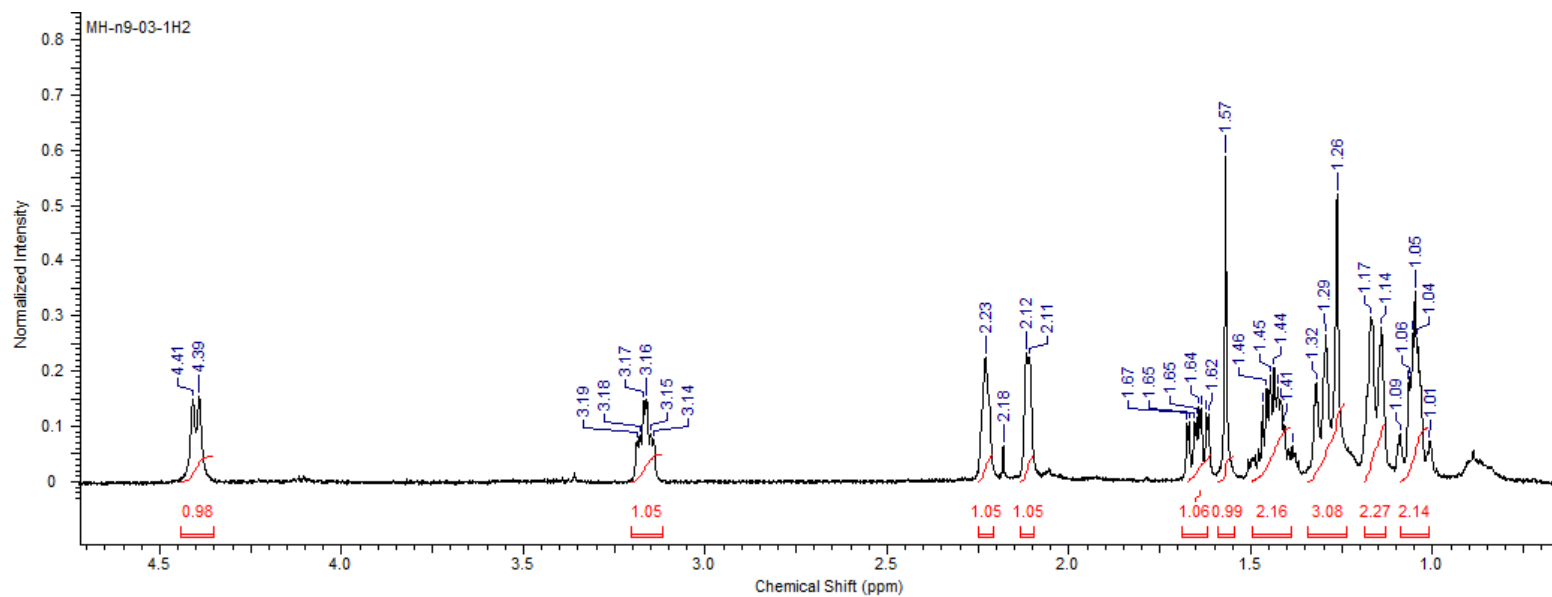
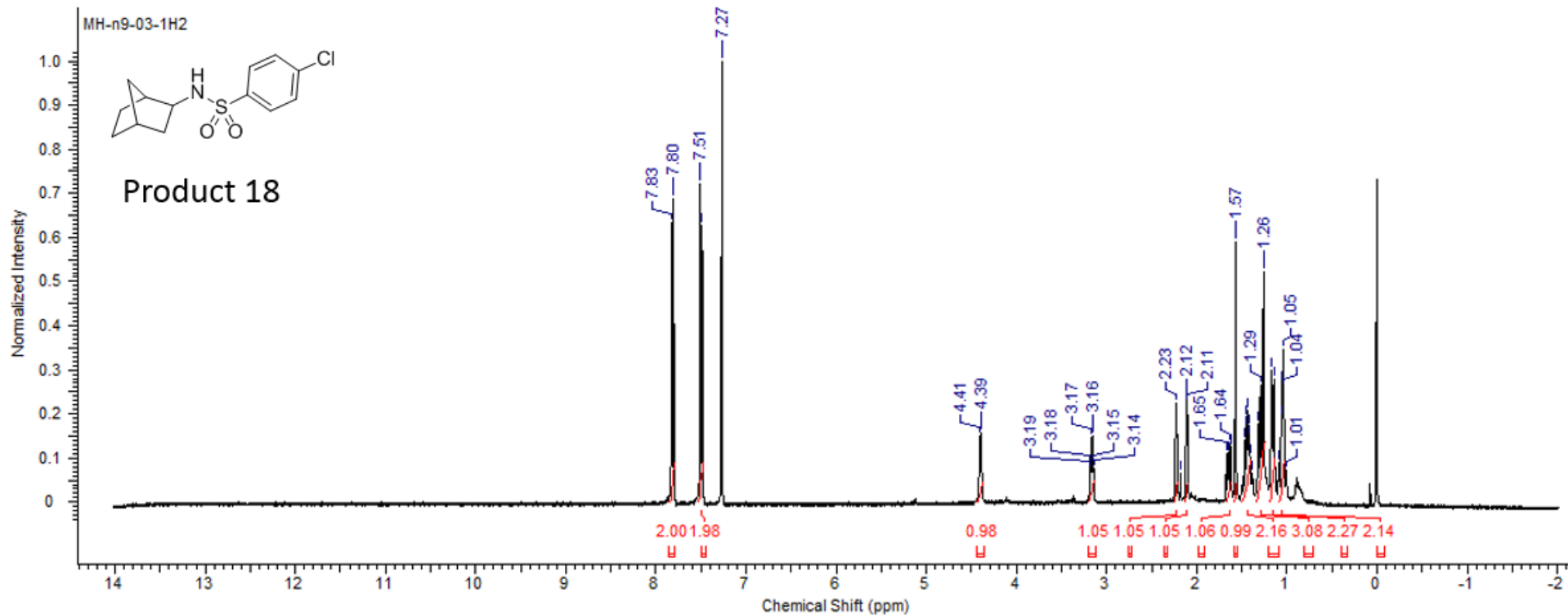
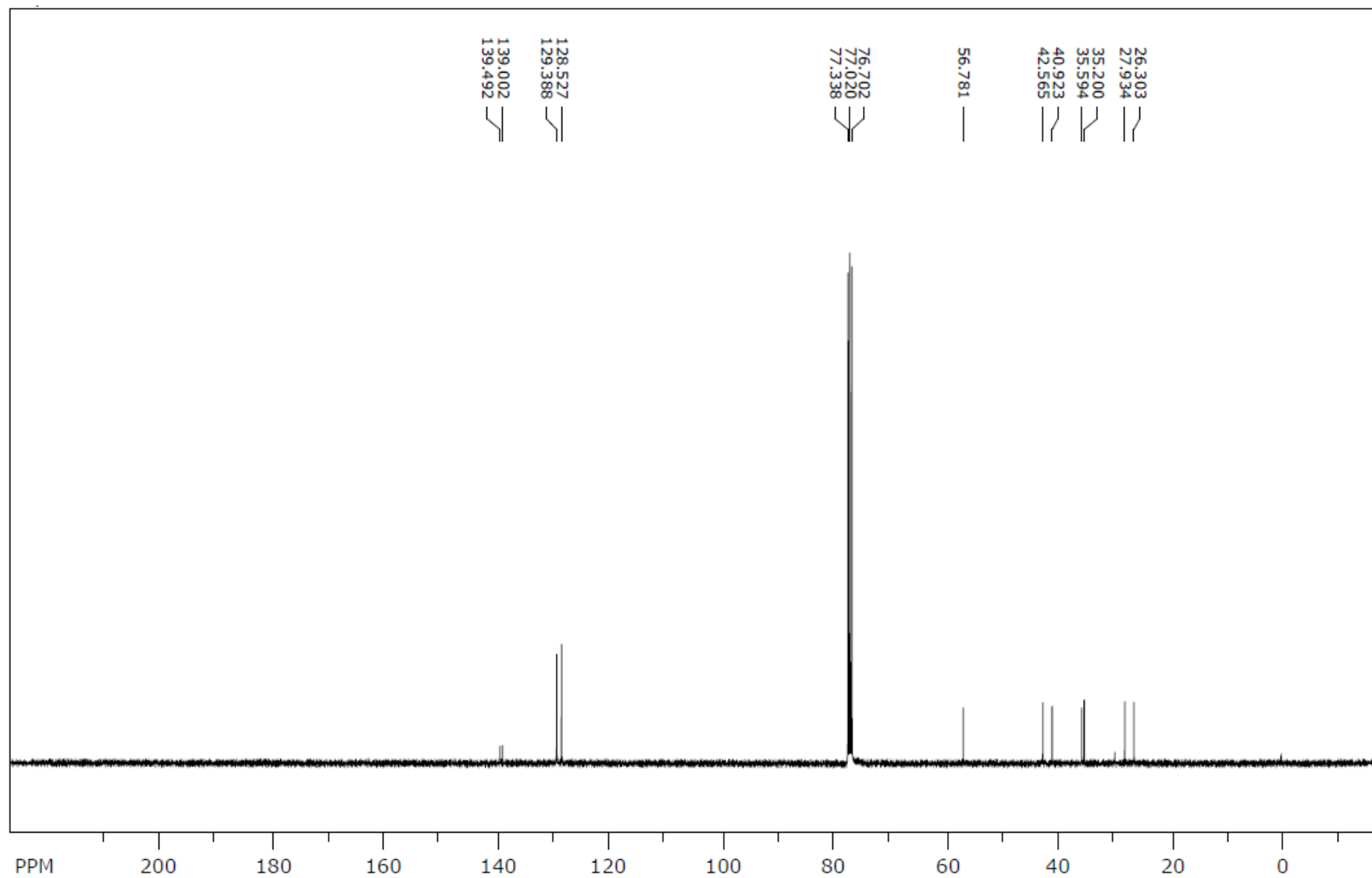


Figure S35. ^1H NMR of Product 18.



file: ...nane, 4-ClPh\MH-n9-03-13C2.fid\fid_block# 1 expt: "s2pul"
 transmitter freq.: 100.511715 MHz
 time domain size: 63750 points
 width: 24509.80 Hz = 243.8502 ppm = 0.384468 Hz/pt
 number of scans: 18000

freq. of 0 ppm: 100.501160 MHz
 processed size: 65536 complex points
 LB: 0.500 GF: 0.0000
 Hz/cm: 980.392 ppm/cm: 9.75401

Figure S36. ¹³C NMR of Product 18.

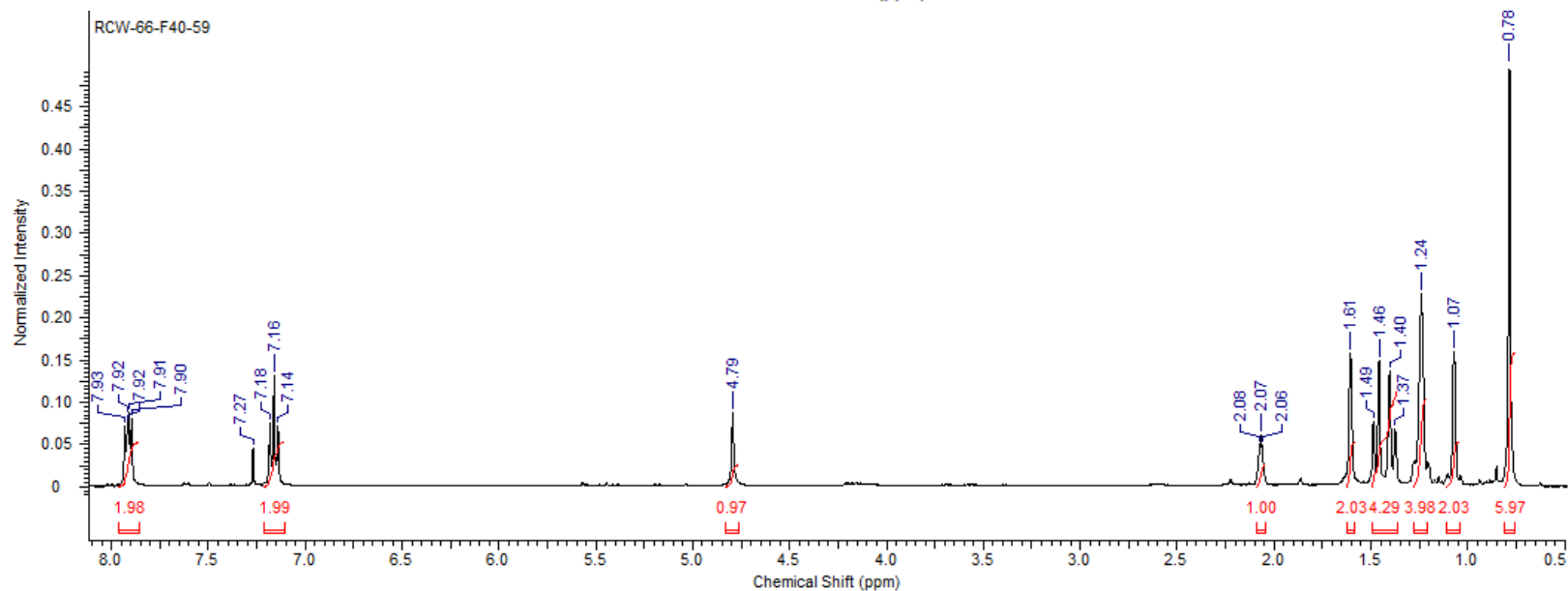
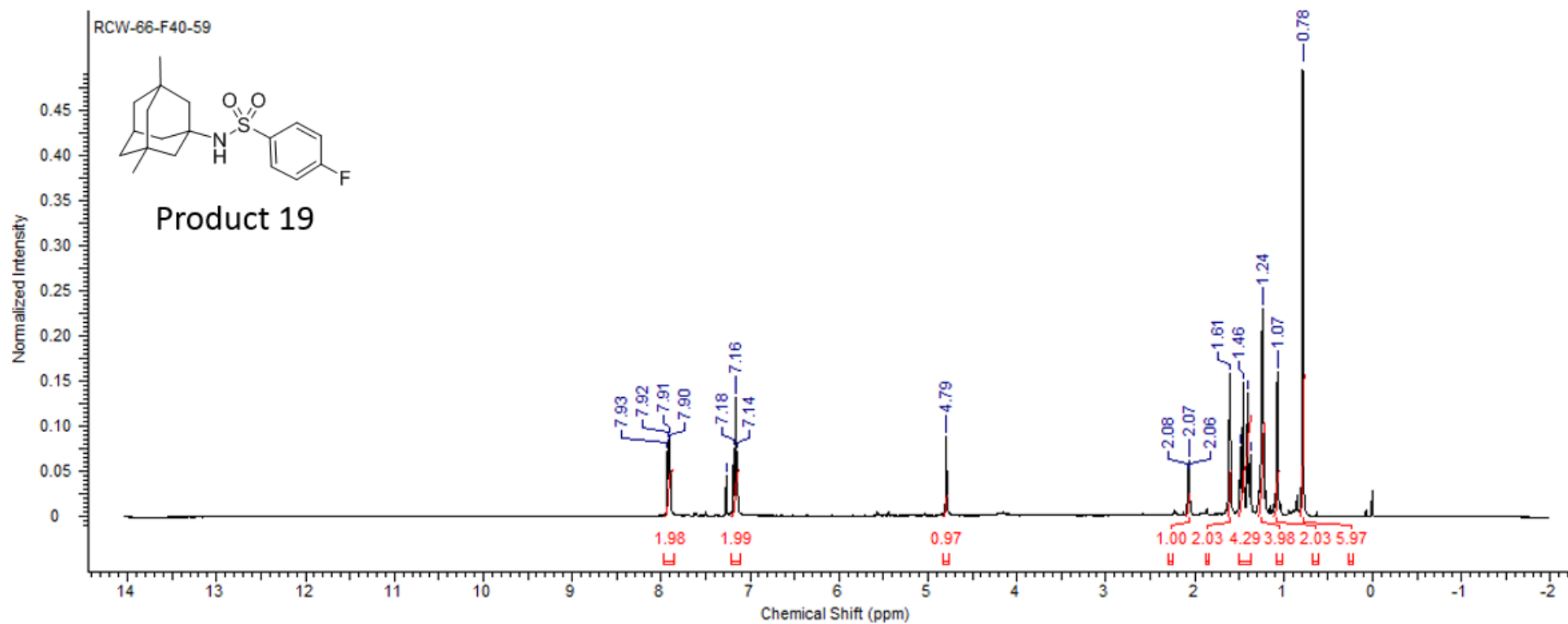
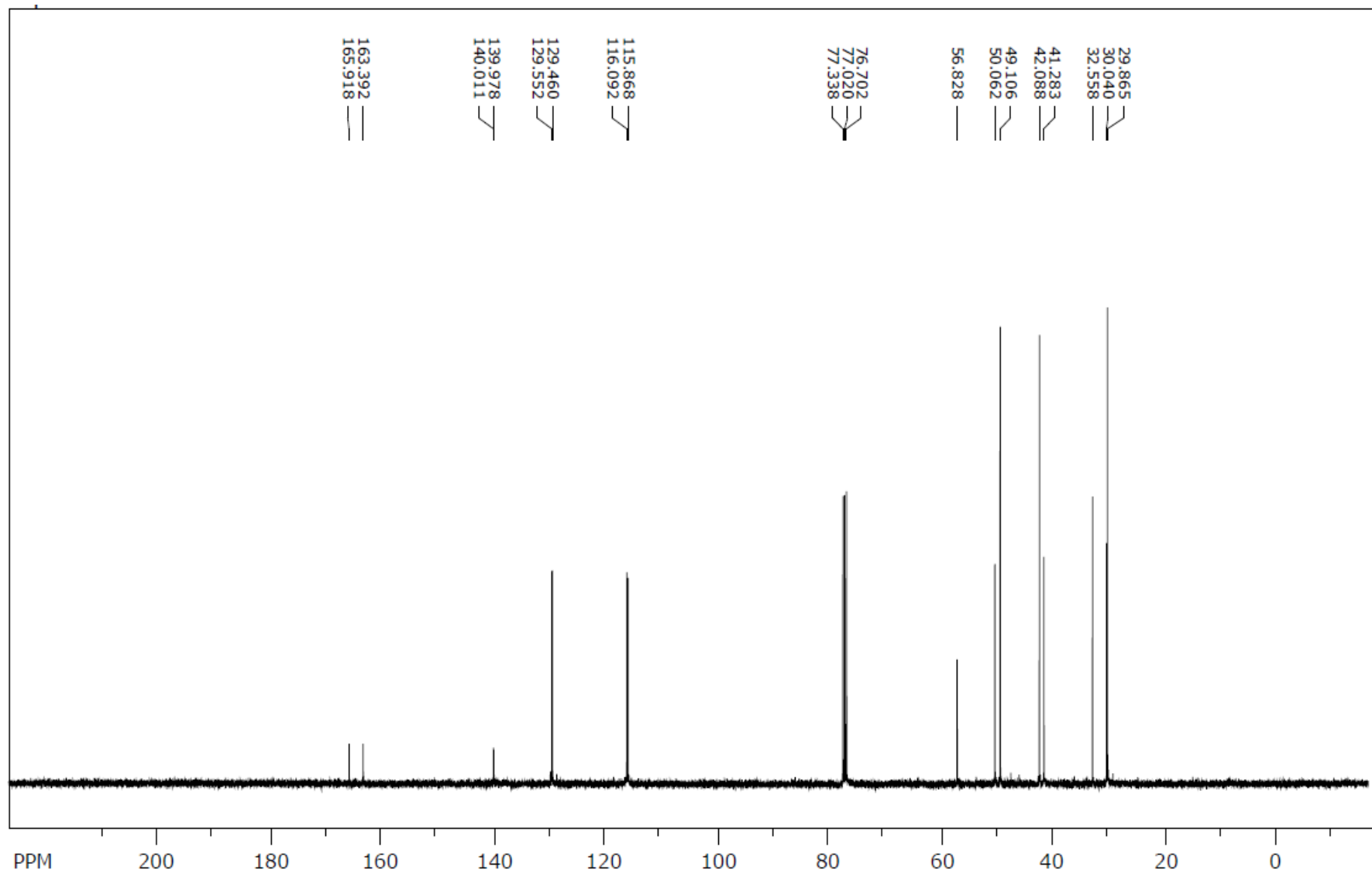


Figure S37. ^1H NMR of Product 19.



file: ...R Data\NMR Fids\RCW-66-13C.fid\fid_block# 1_expt: "s2pul"
 transmitter freq.: 100.508439 MHz
 time domain size: 63750 points
 width: 24509.80 Hz = 243.8582 ppm = 0.384468 Hz/pt
 number of scans: 750

freq. of 0 ppm: 100.497882 MHz
 processed size: 65536 complex points
 LB: 0.500 GF: 0.0000
 Hz/cm: 980.392 ppm/cm: 9.75433

Figure S38. ^{13}C NMR of Product 19.

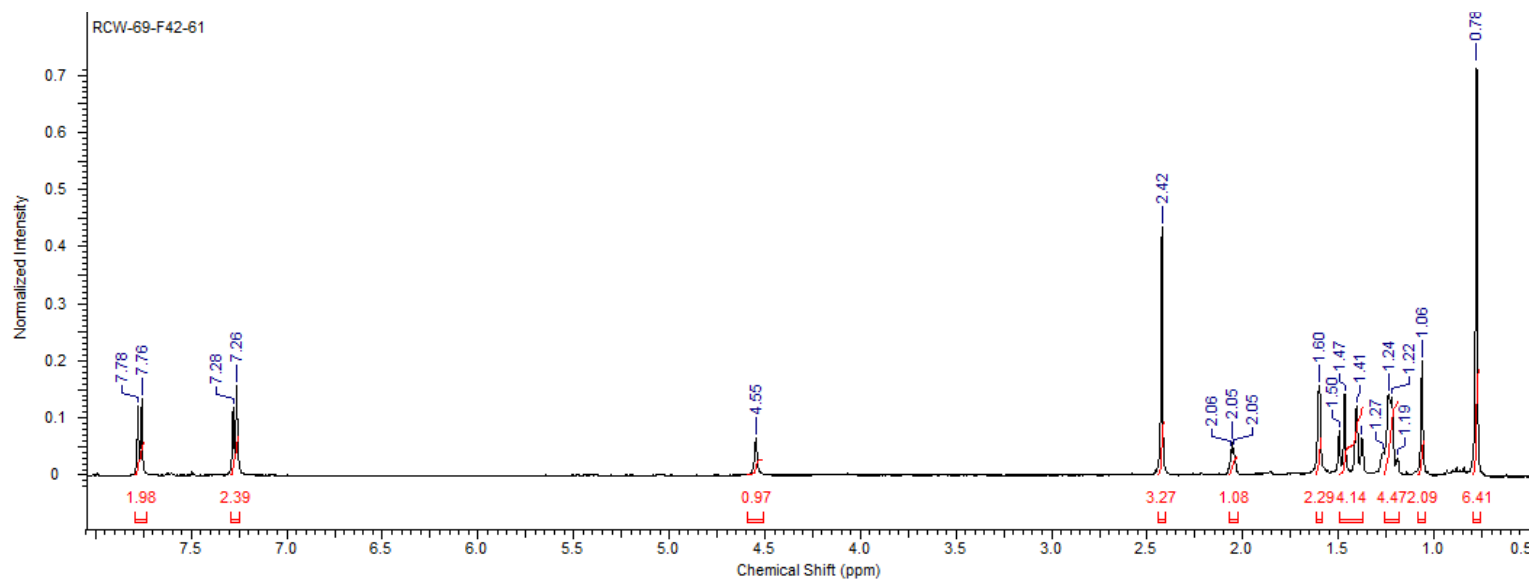
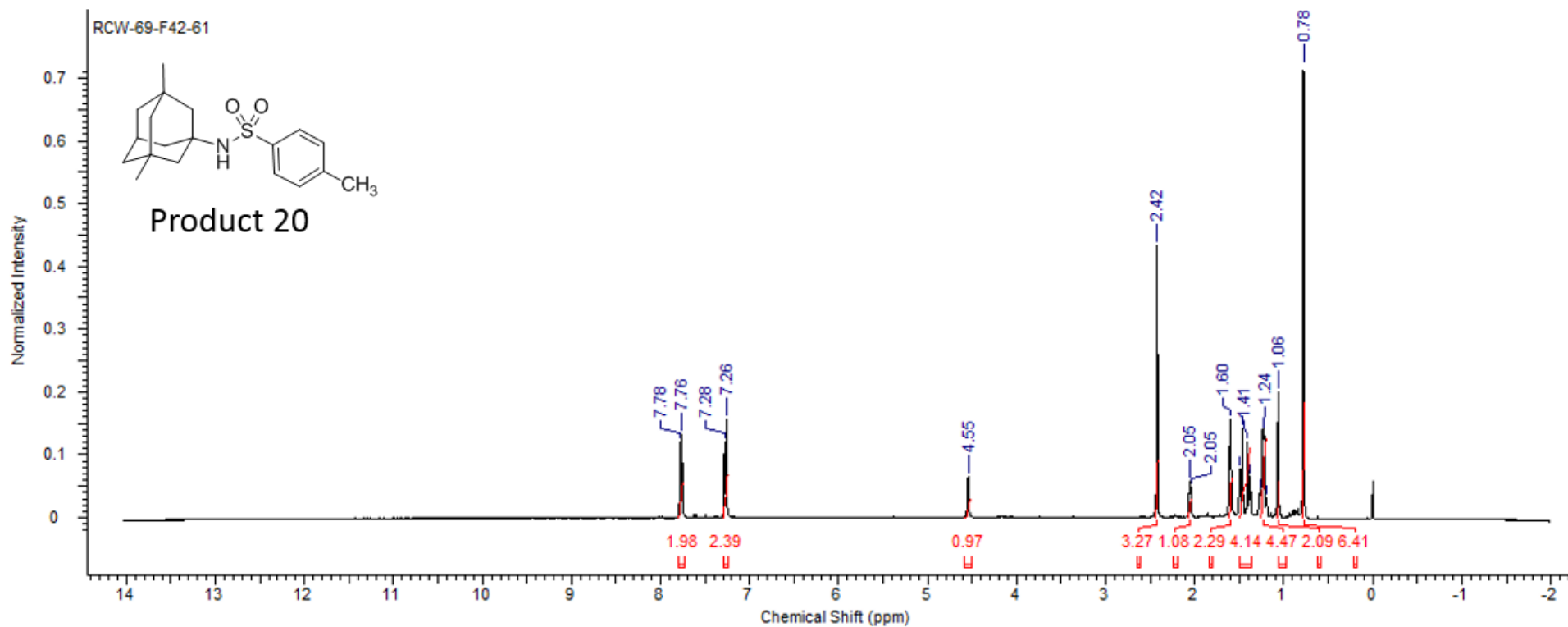
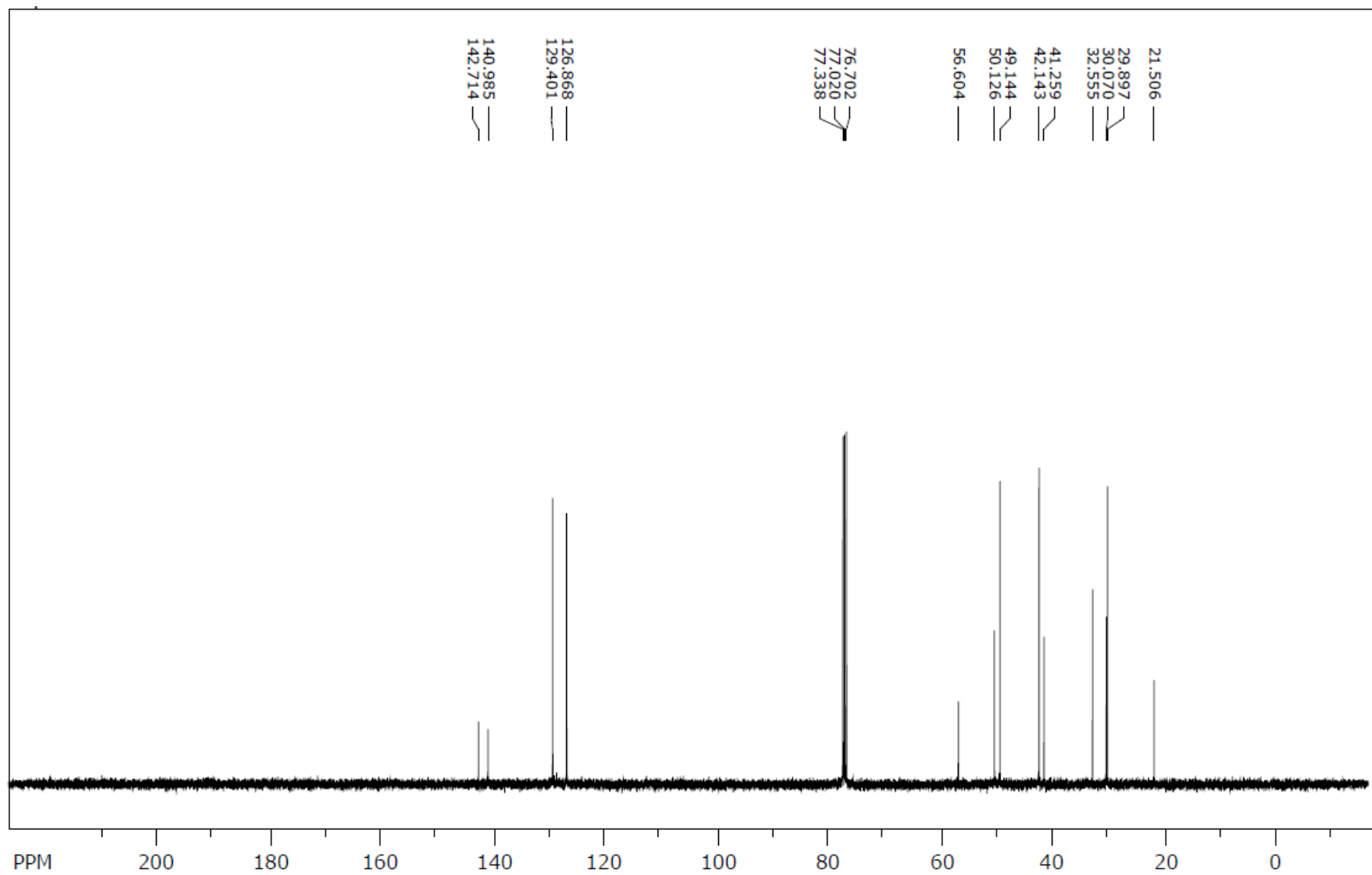


Figure S39. ^1H NMR of Product 20.



file: ...R Data\NMR Fids\RCW-69-13C.fid\fid block# 1 expt: "s2pul"
 transmitter freq.: 100.508439 MHz
 time domain size: 63750 points
 width: 24509.80 Hz = 243.8582 ppm = 0.384468 Hz/pt
 number of scans: 496

freq. of 0 ppm: 100.497882 MHz
 processed size: 65536 complex points
 LB: 0.500 GF: 0.0000
 Hz/cm: 980.392 ppm/cm: 9.75433

Figure S40. ¹³C NMR of Product 20.

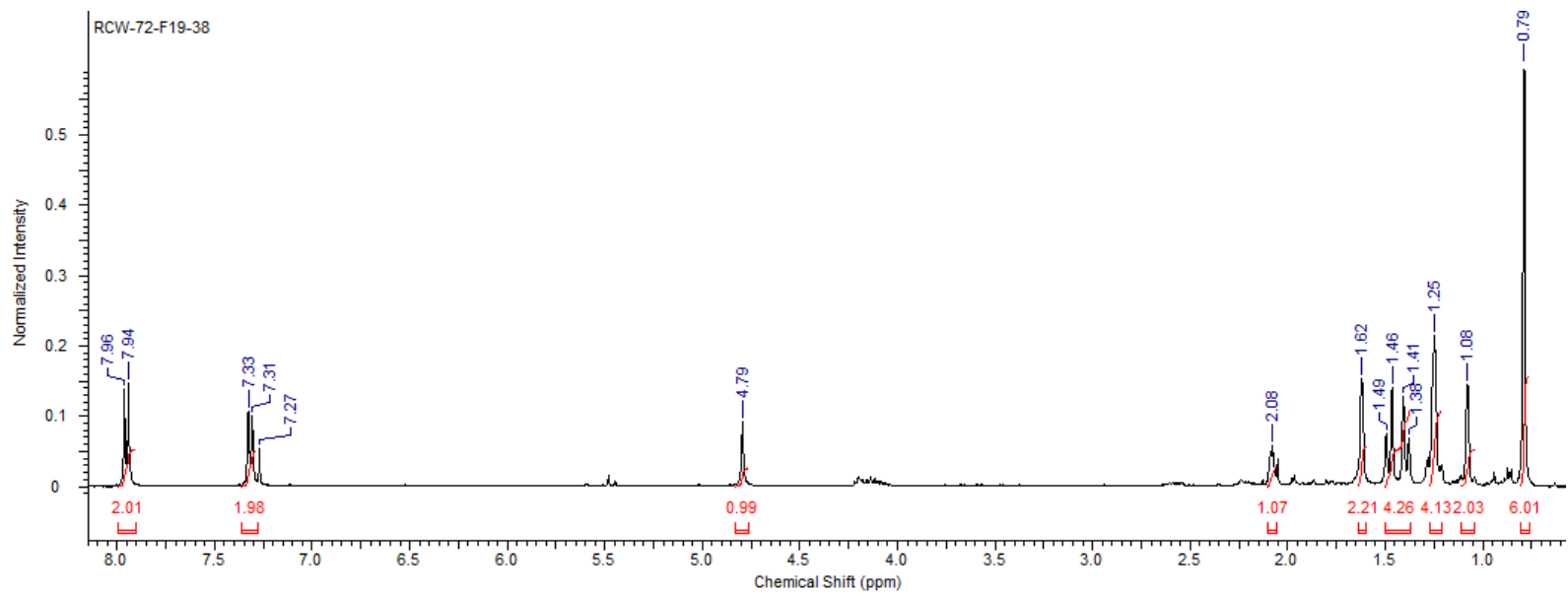
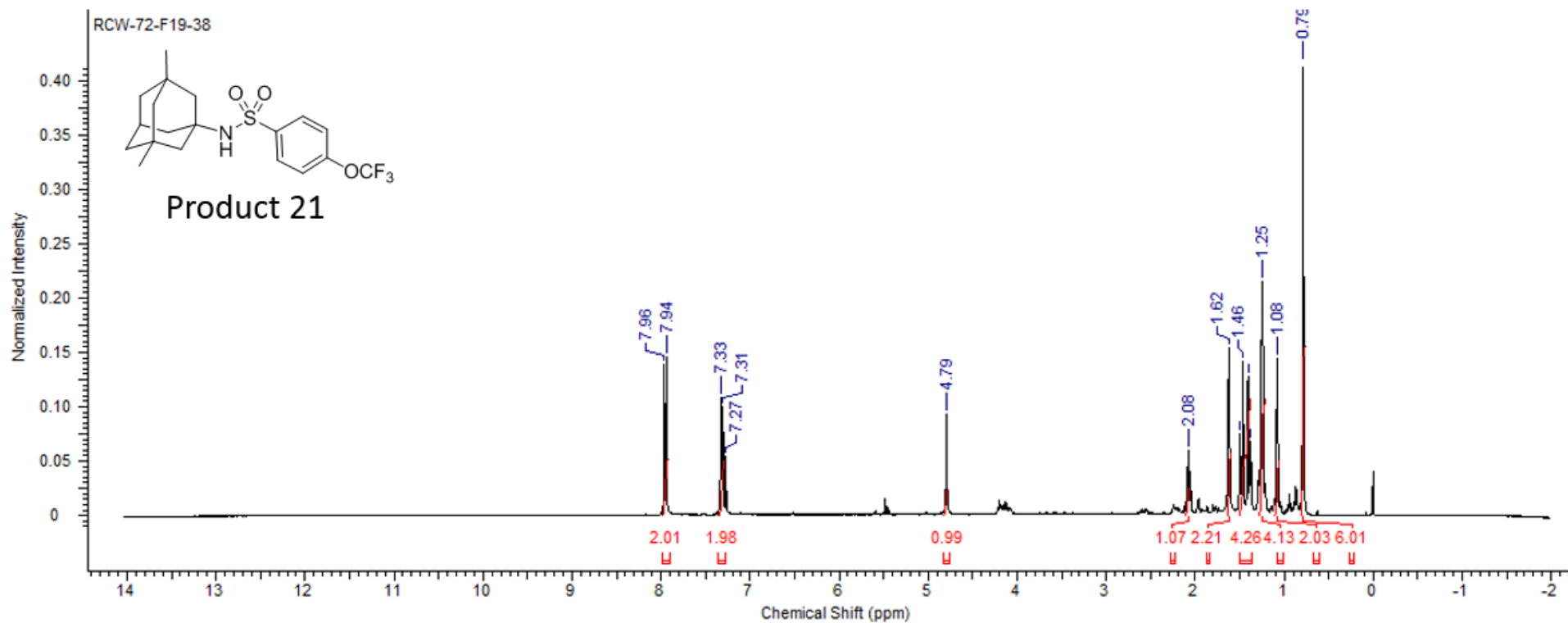
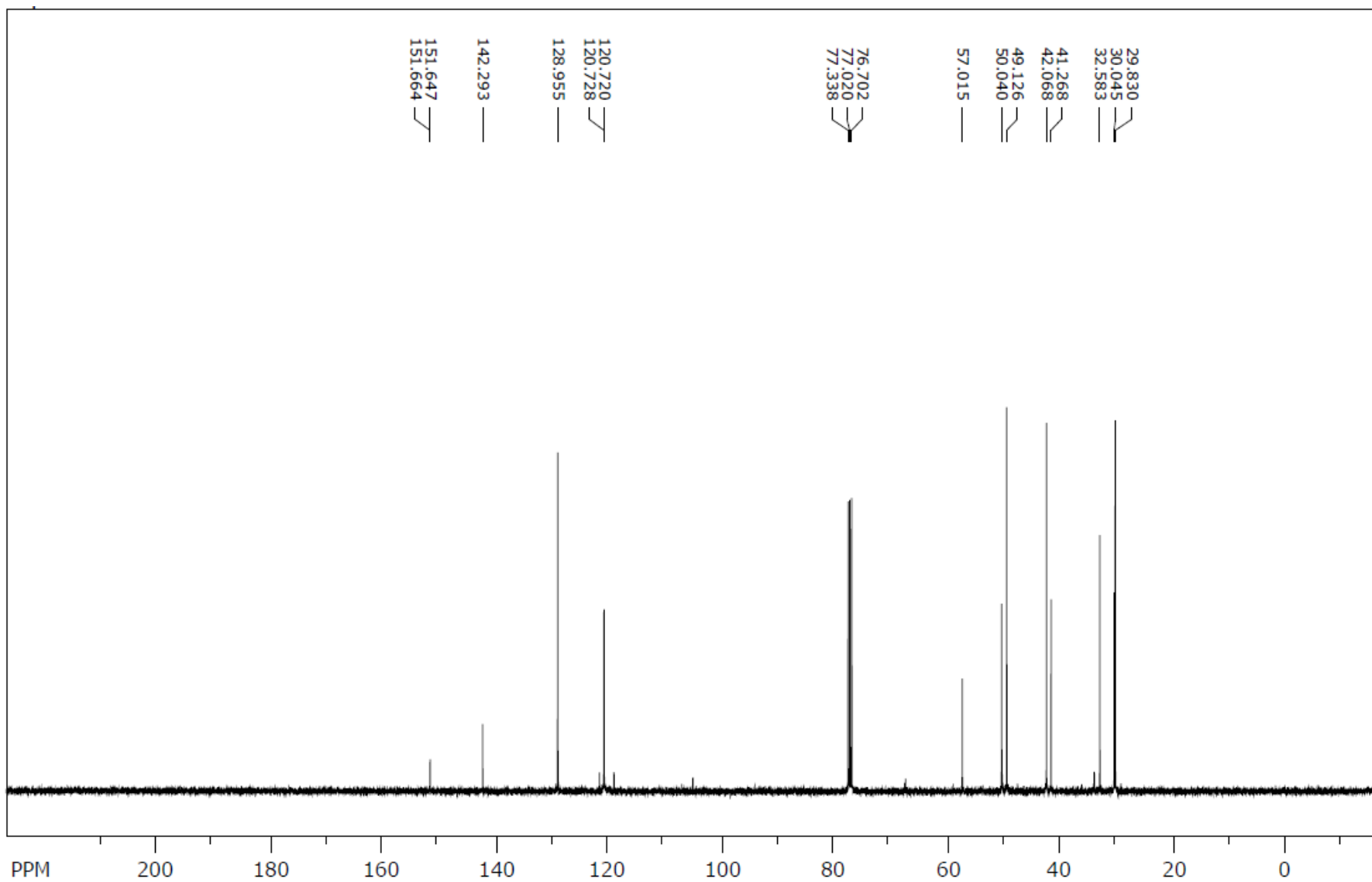


Figure S41. ^1H NMR of Product 21.



file: ...R Data\NMR Fids\RCW-72-13C.fid\fid_block# 1 expt: "s2pul"
 transmitter freq.: 100.508439 MHz
 time domain size: 63750 points
 width: 24509.80 Hz = 243.8582 ppm = 0.384468 Hz/pt
 number of scans: 750

freq. of 0 ppm: 100.497881 MHz
 processed size: 65536 complex points
 LB: 0.500 GF: 0.0000
 Hz/cm: 980.392 ppm/cm: 9.75433

Figure S42. ^{13}C NMR of Product **21**.

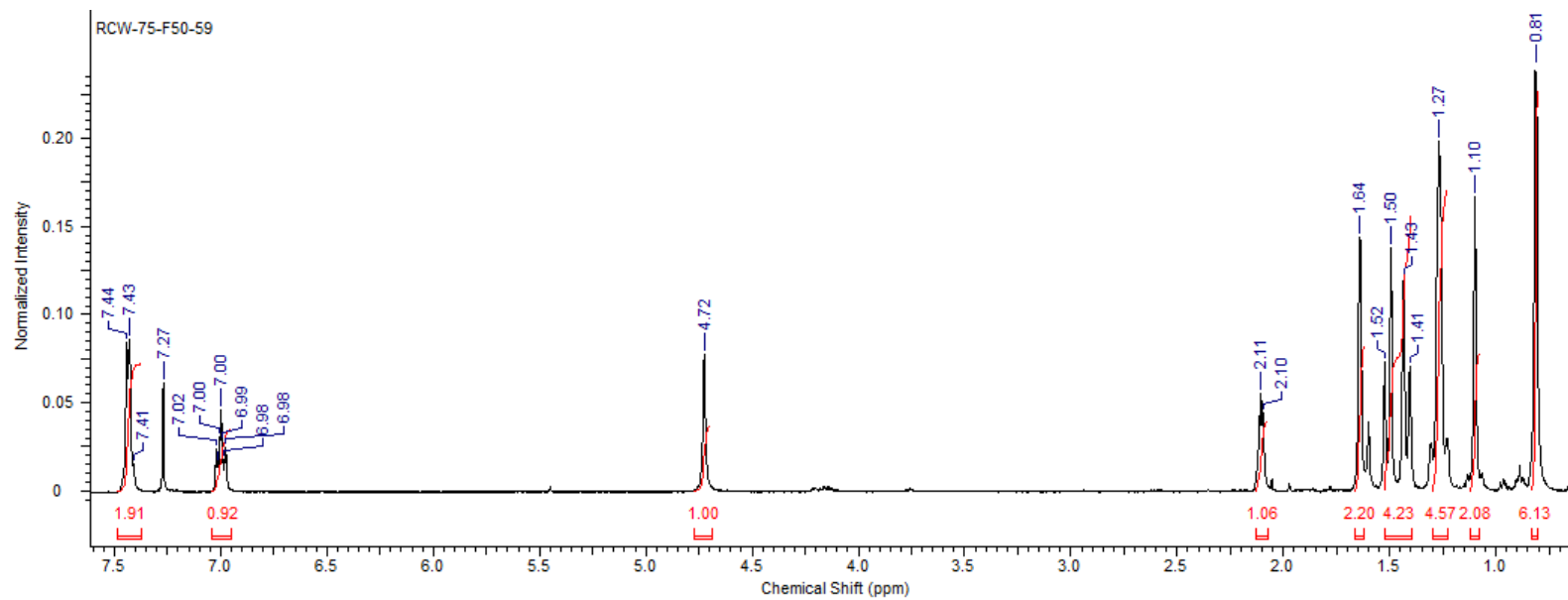
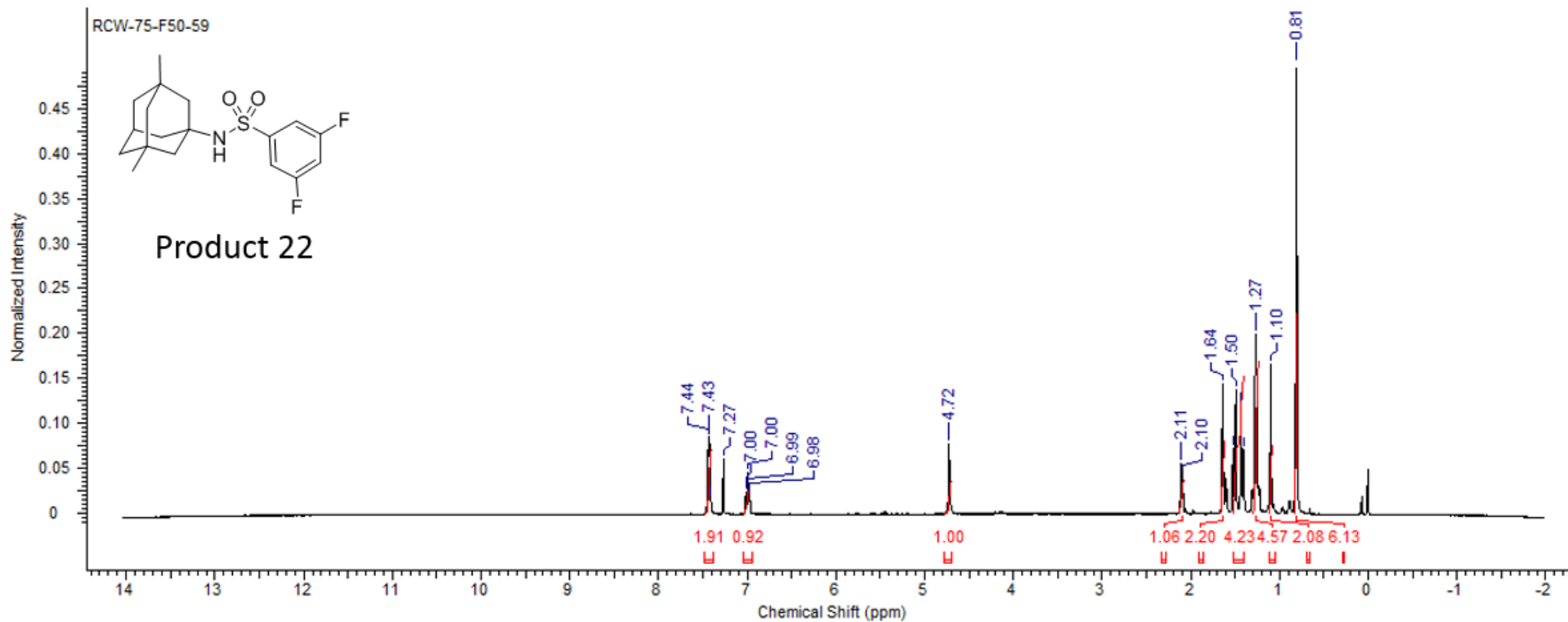
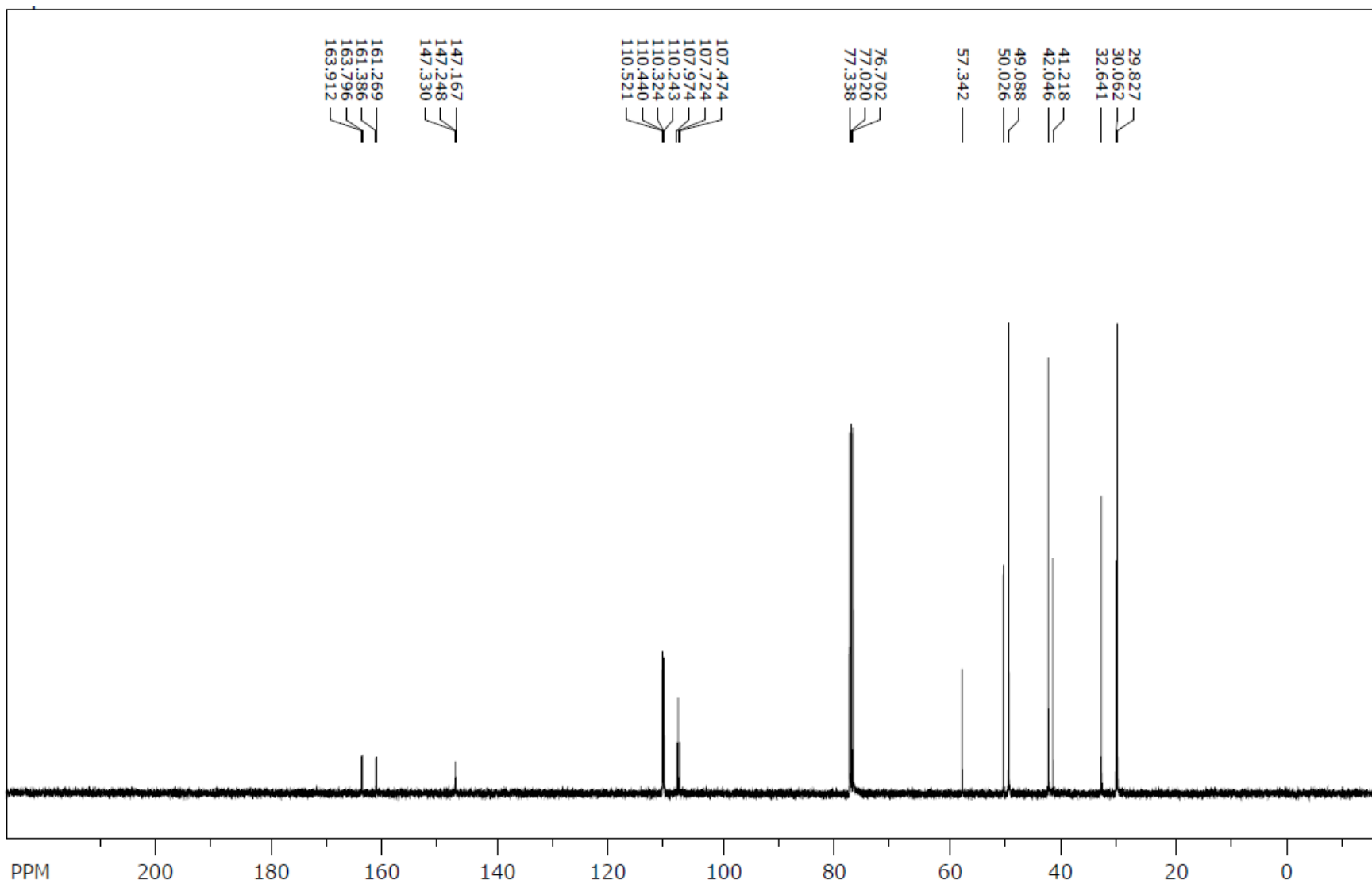


Figure S43. ^1H NMR of Product 22.



file: ...R Data\NMR Fids\RCW-75-13C.fid\fid_block# 1_expt: "s2pul"
 transmitter freq.: 100.508439 MHz
 time domain size: 63750 points
 width: 24509.80 Hz = 243.8582 ppm = 0.384468 Hz/pt
 number of scans: 750

freq. of 0 ppm: 100.497881 MHz
 processed size: 65536 complex points
 LB: 0.500 GF: 0.0000
 Hz/cm: 980.392 ppm/cm: 9.75433

Figure S44. ¹³C NMR of Product 22.

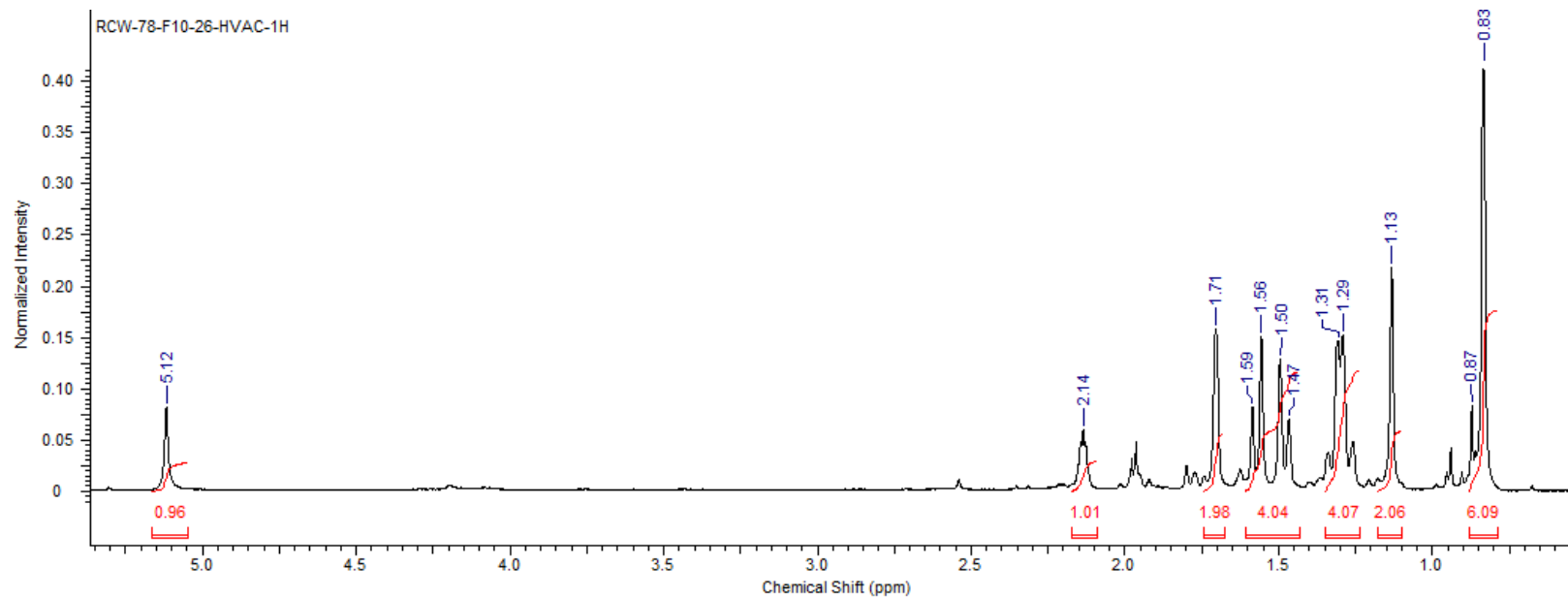
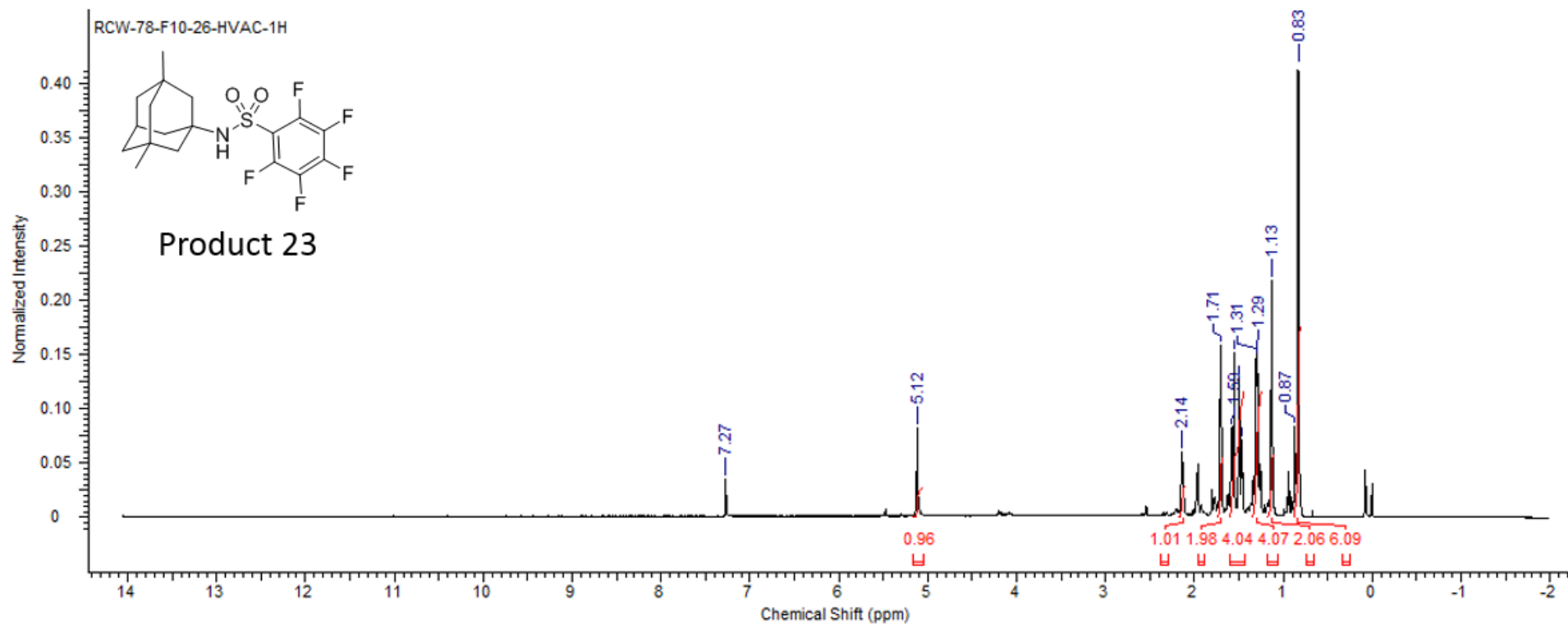
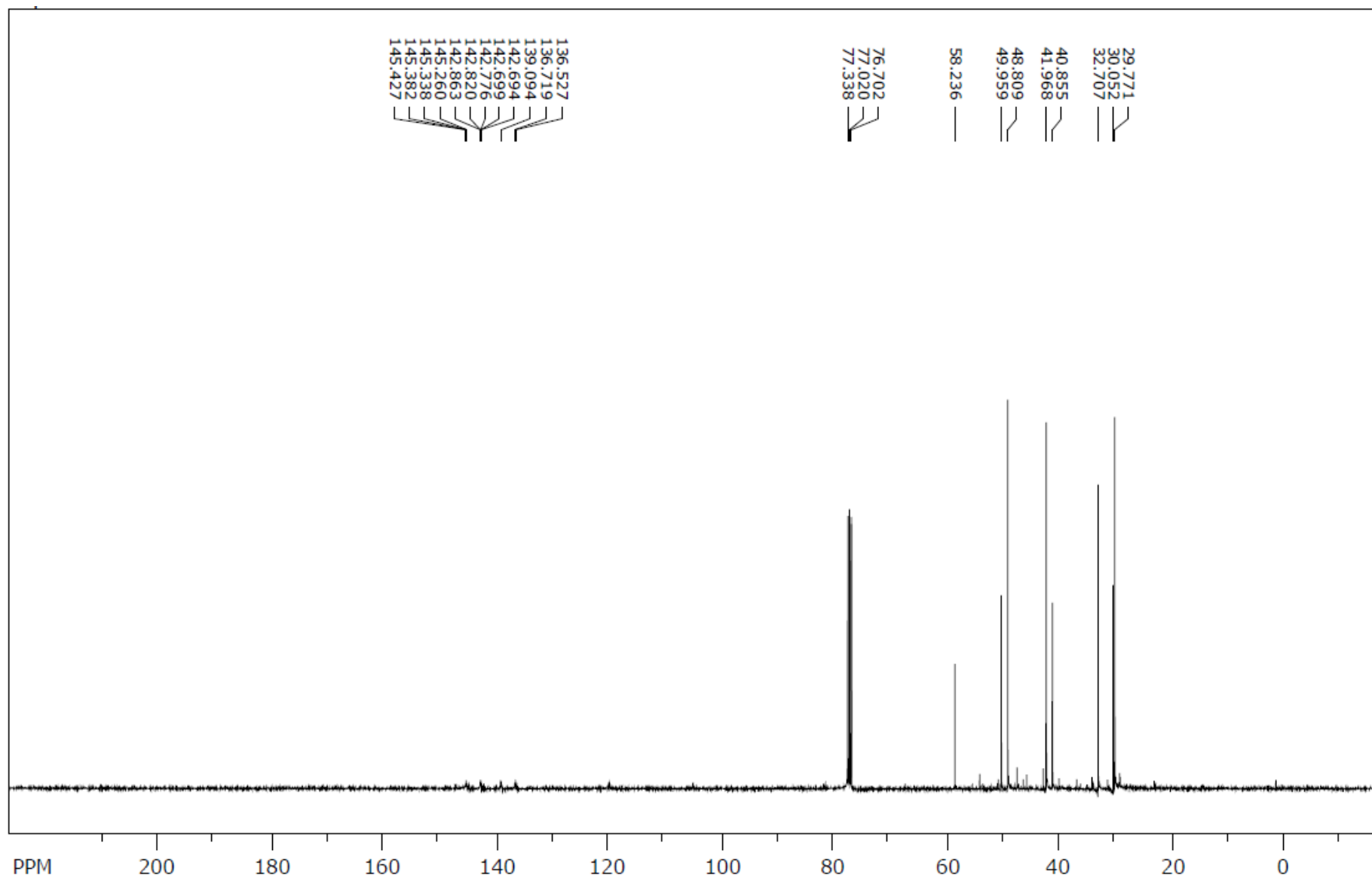


Figure S45. ^1H NMR of Product 23.



file: ...Data\NMR Fids\RCW-78-13C-B.fid\fid_block# 1 expt: "s2pul"
 transmitter freq.: 100.508439 MHz
 time domain size: 63750 points
 width: 24509.80 Hz = 243.8582 ppm = 0.384468 Hz/pt
 number of scans: 11000

freq. of 0 ppm: 100.497882 MHz
 processed size: 65536 complex points
 LB: 0.500 GF: 0.0000
 Hz/cm: 980.392 ppm/cm: 9.75433

Figure S46. ^{13}C NMR of Product 23.

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