

Supporting Information

CONCISE PRACTICAL AVENUES TO 5,5-SPIRO- α -PROLINES

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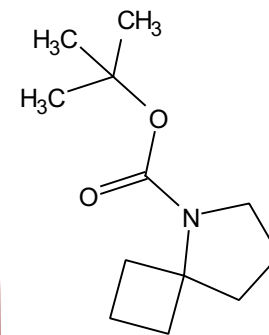
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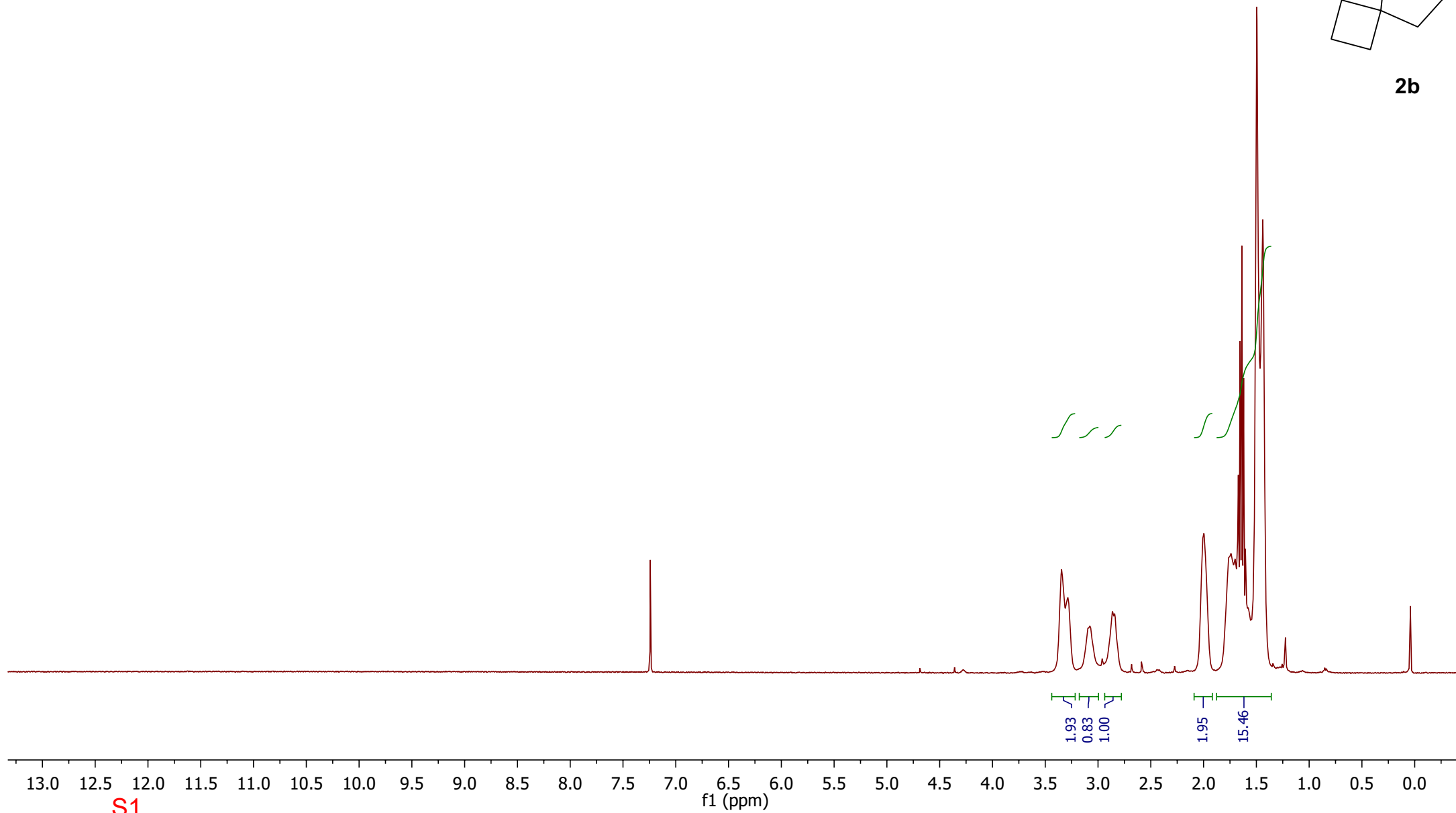
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^1H NMR (400 MHz, Chloroform-*d*) δ 3.44 – 3.22 (m, 2H), 3.18 – 3.00 (m, 1H), 2.85 (q, J = 10.1, 9.5 Hz, 1H), 2.09 – 1.92 (m, 2H), 1.88 – 1.36 (m, 15H).

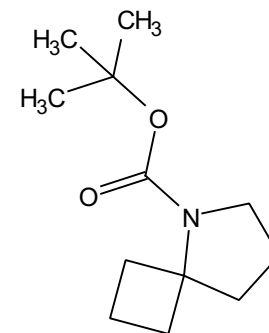


2b

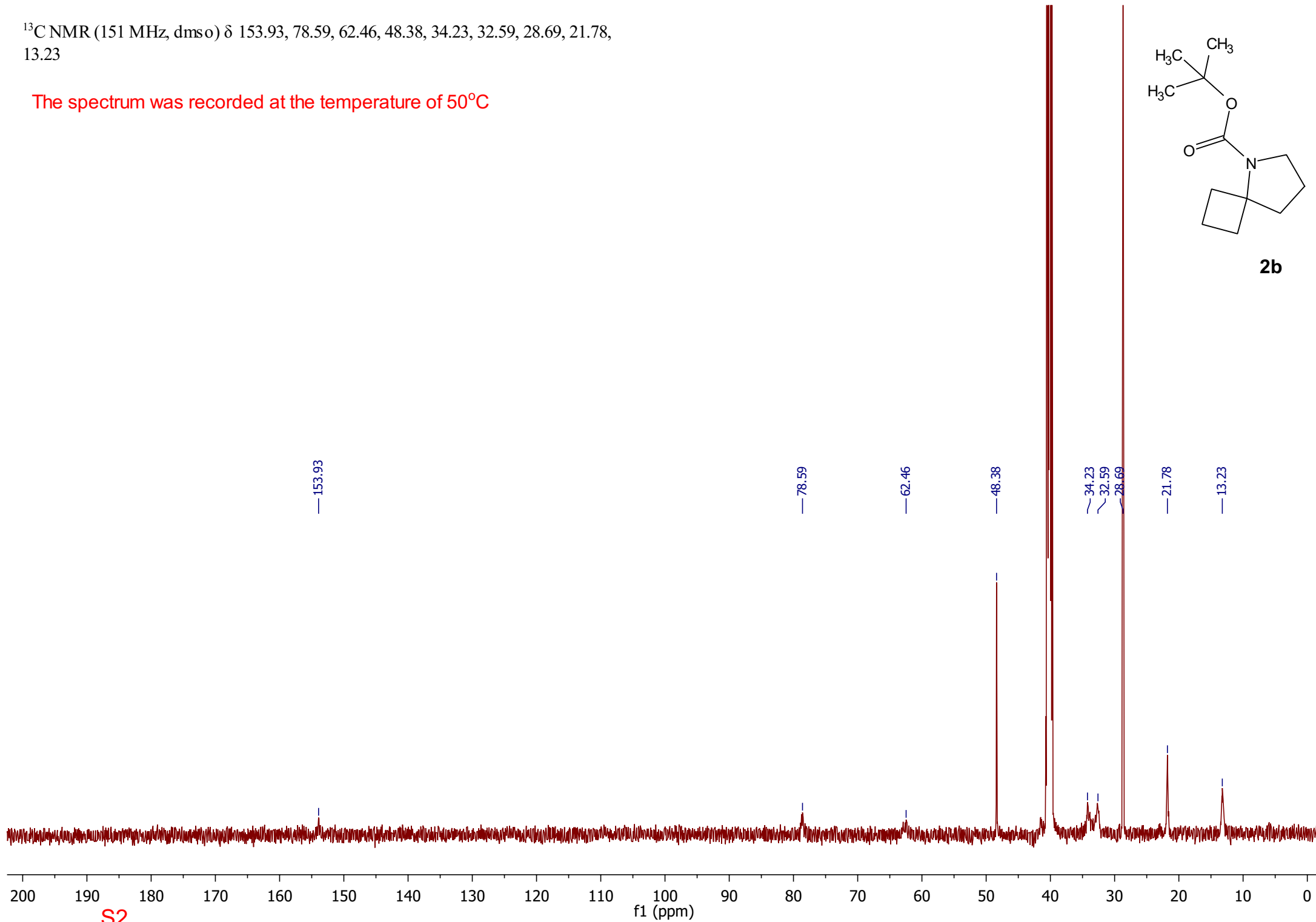


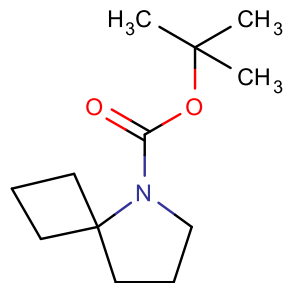
^{13}C NMR (151 MHz, dms-o) δ 153.93, 78.59, 62.46, 48.38, 34.23, 32.59, 28.69, 21.78, 13.23

The spectrum was recorded at the temperature of 50°C



2b

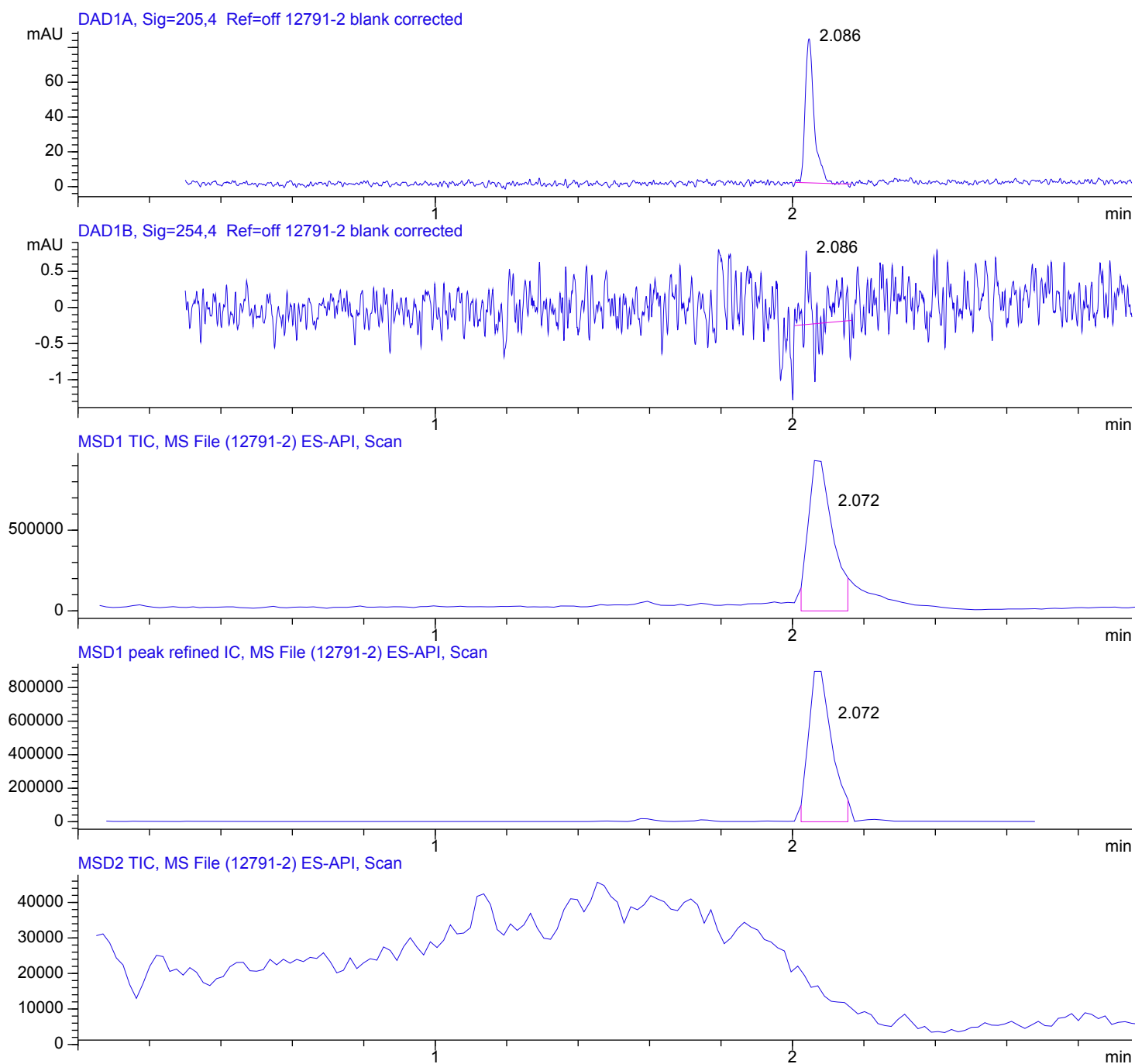


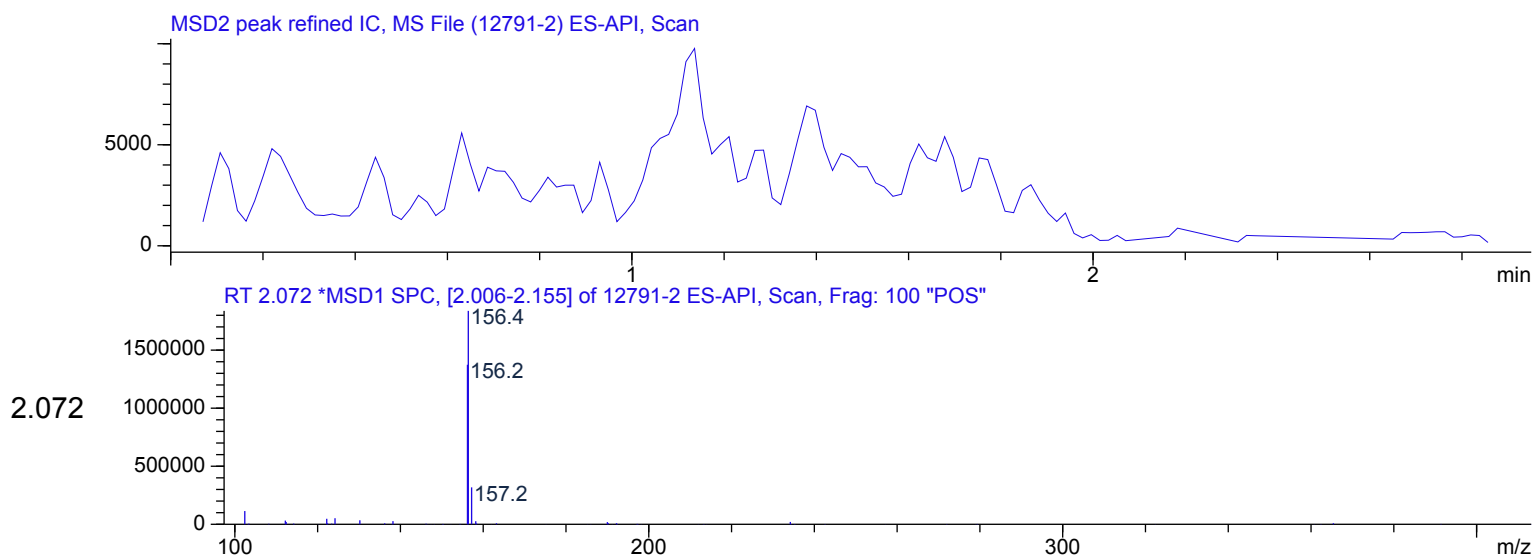


[211.16]

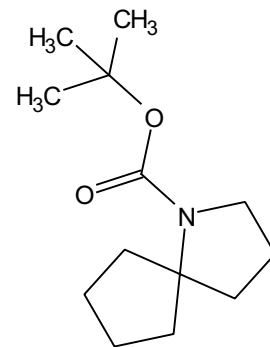
2b

#	RT	DAD1A	DAD1B	MSD1	MSD2	ELSD	MSD1 ions	MSD1 rt	MSD2 ions	MSD2 rt	Info
1	2.086	100.0%	100.0%	100.0%	---	---	156.4(100)	2.072	---	---	

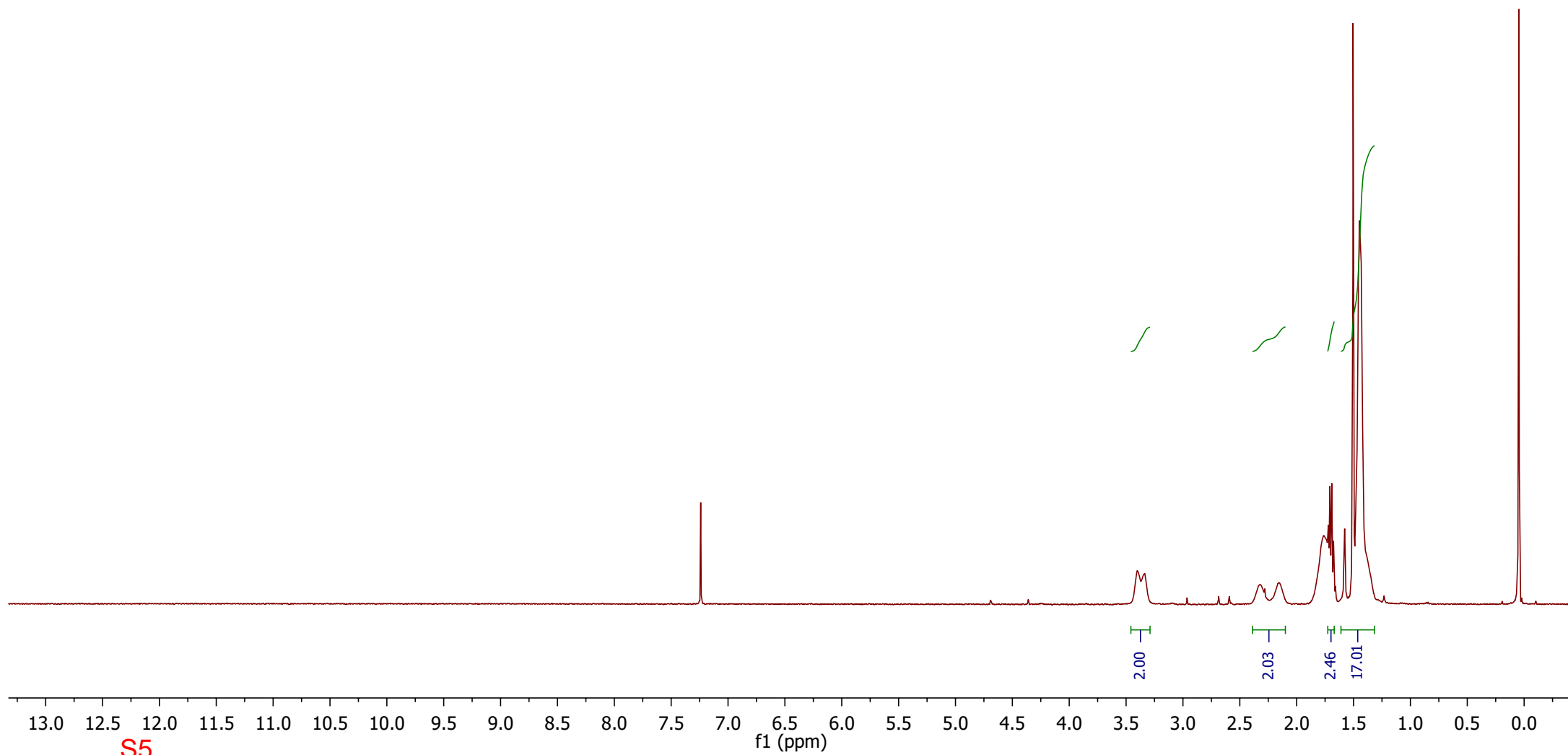




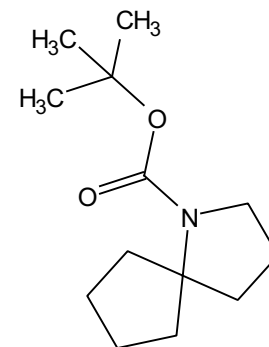
^1H NMR (400 MHz, Chloroform-*d*) δ 3.46 – 3.29 (m, 2H), 2.39 – 2.10 (m, 2H), 1.70 (q, $J = 6.1$ Hz, 2H), 1.61 – 1.32 (m, 17H).



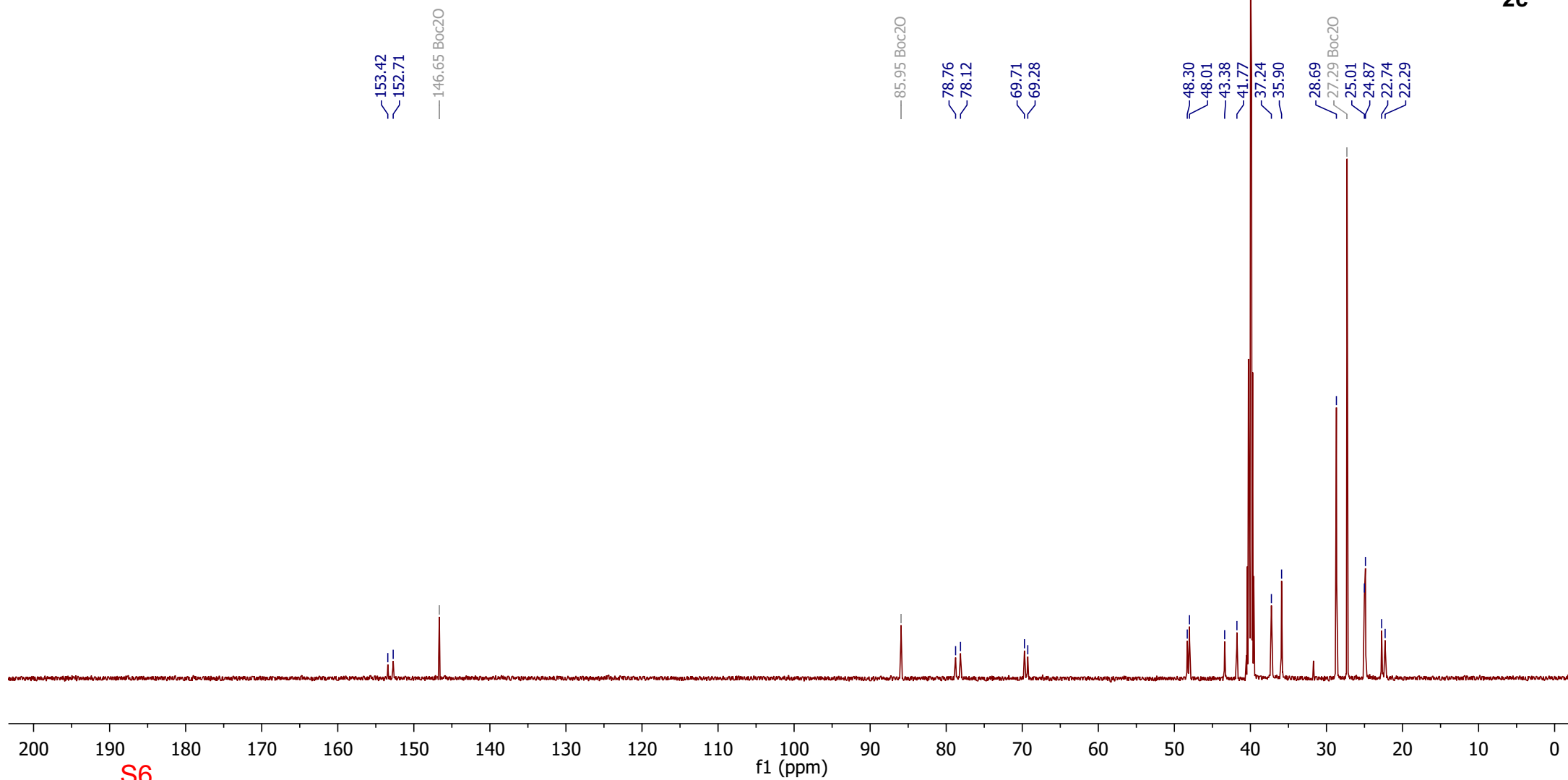
2c

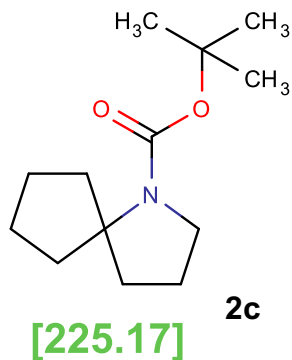


^{13}C NMR (151 MHz, dms_o) δ 153.42, 152.71, 78.76, 78.12, 69.71, 69.28, 48.30, 48.01, 43.38, 41.77, 37.24, 35.90, 28.69, 25.01, 24.87, 22.74, 22.29

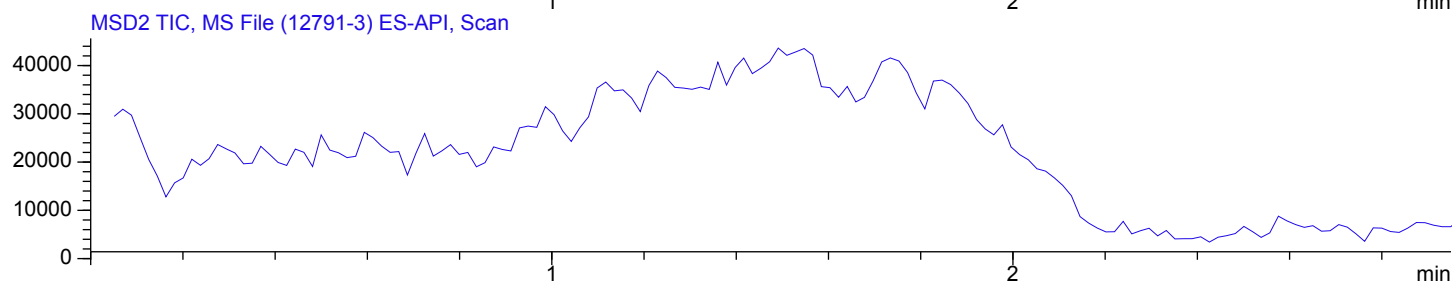
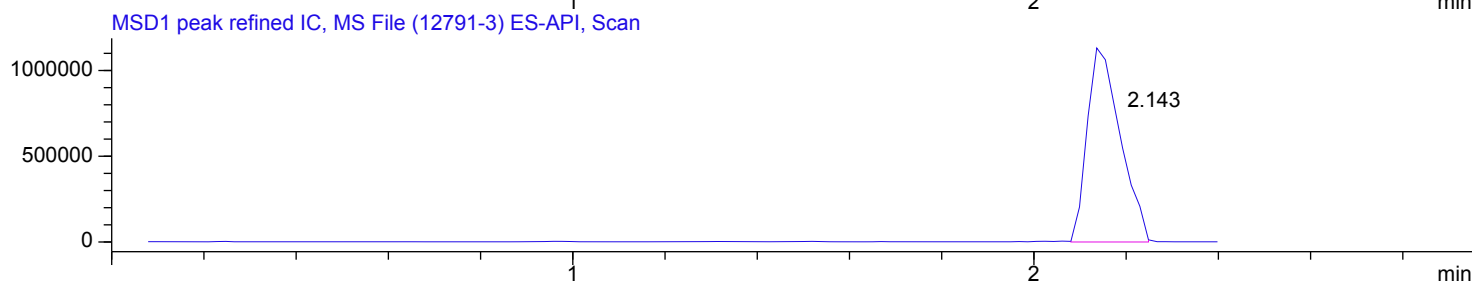
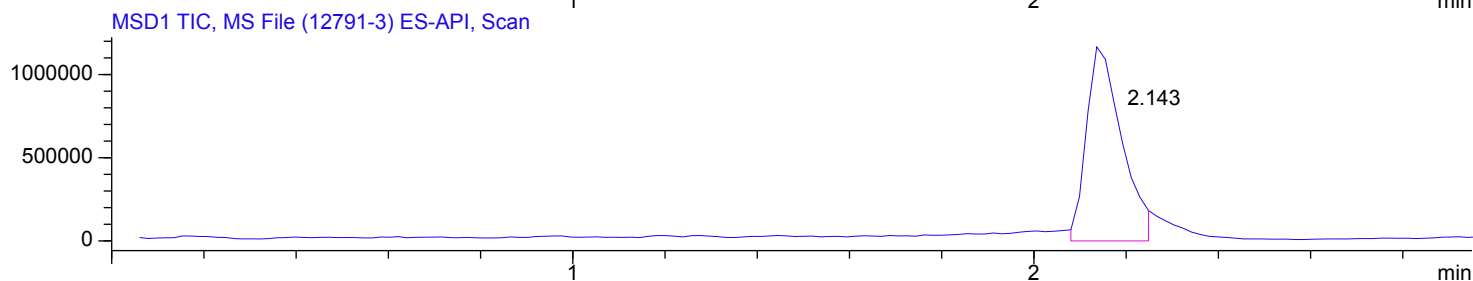
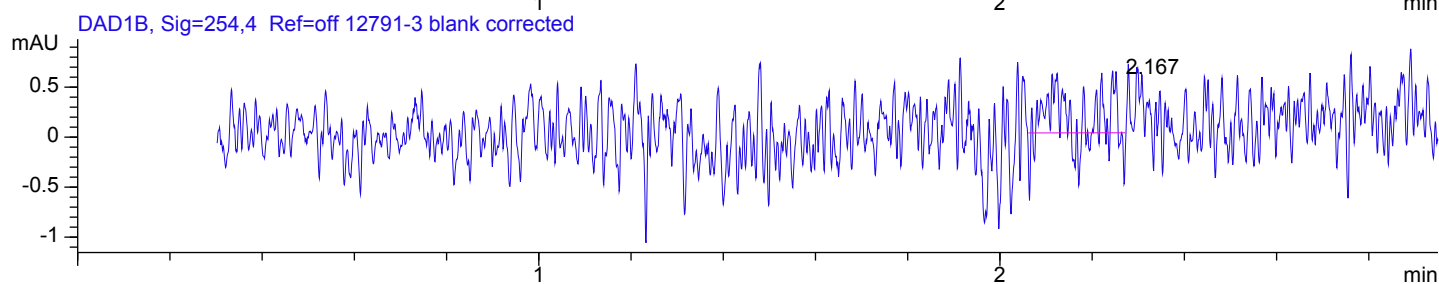
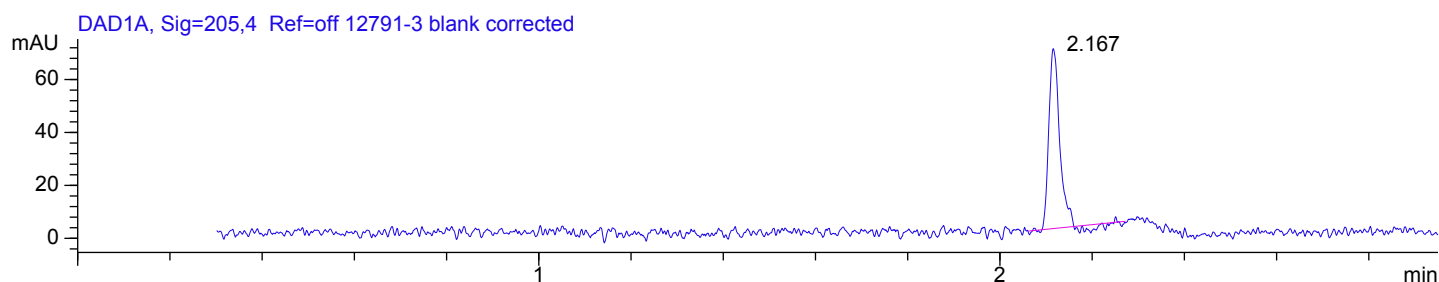


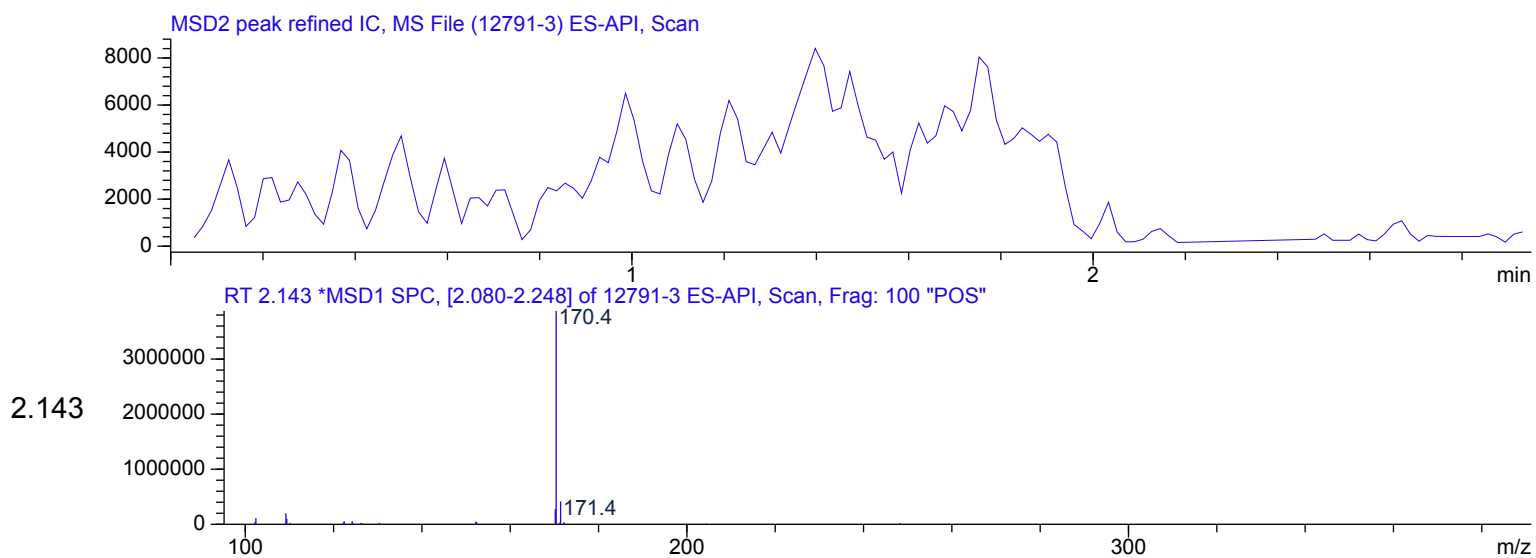
2c



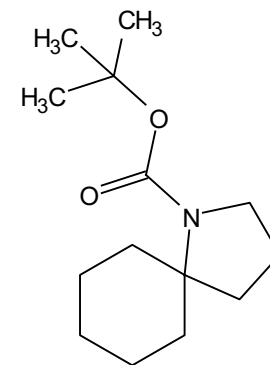


#	RT	DAD1A	DAD1B	MSD1	MSD2	ELSD	MSD1 ions	MSD1 rt	MSD2 ions	MSD2 rt	Info
1	2.167	100.0%	100.0%	100.0%	---	---	170.4(92),109.2(7),152.2(2)	2.143	---	---	

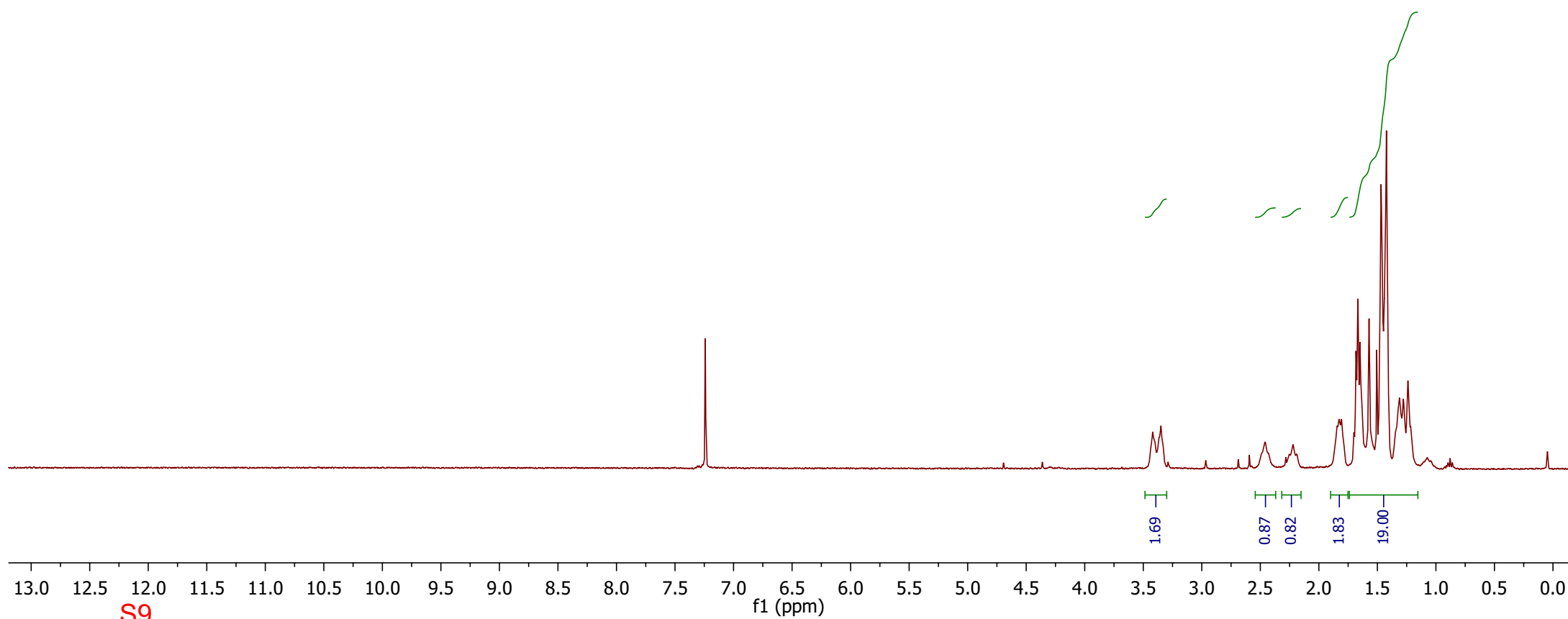




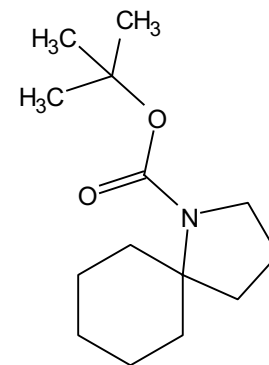
^1H NMR (400 MHz, Chloroform-*d*) δ 3.38 (dt, J = 28.4, 6.9 Hz, 2H), 2.46 (t, J = 10.3 Hz, 1H), 2.22 (t, J = 13.2 Hz, 1H), 1.90 – 1.75 (m, 2H), 1.74 – 1.15 (m, 19H).



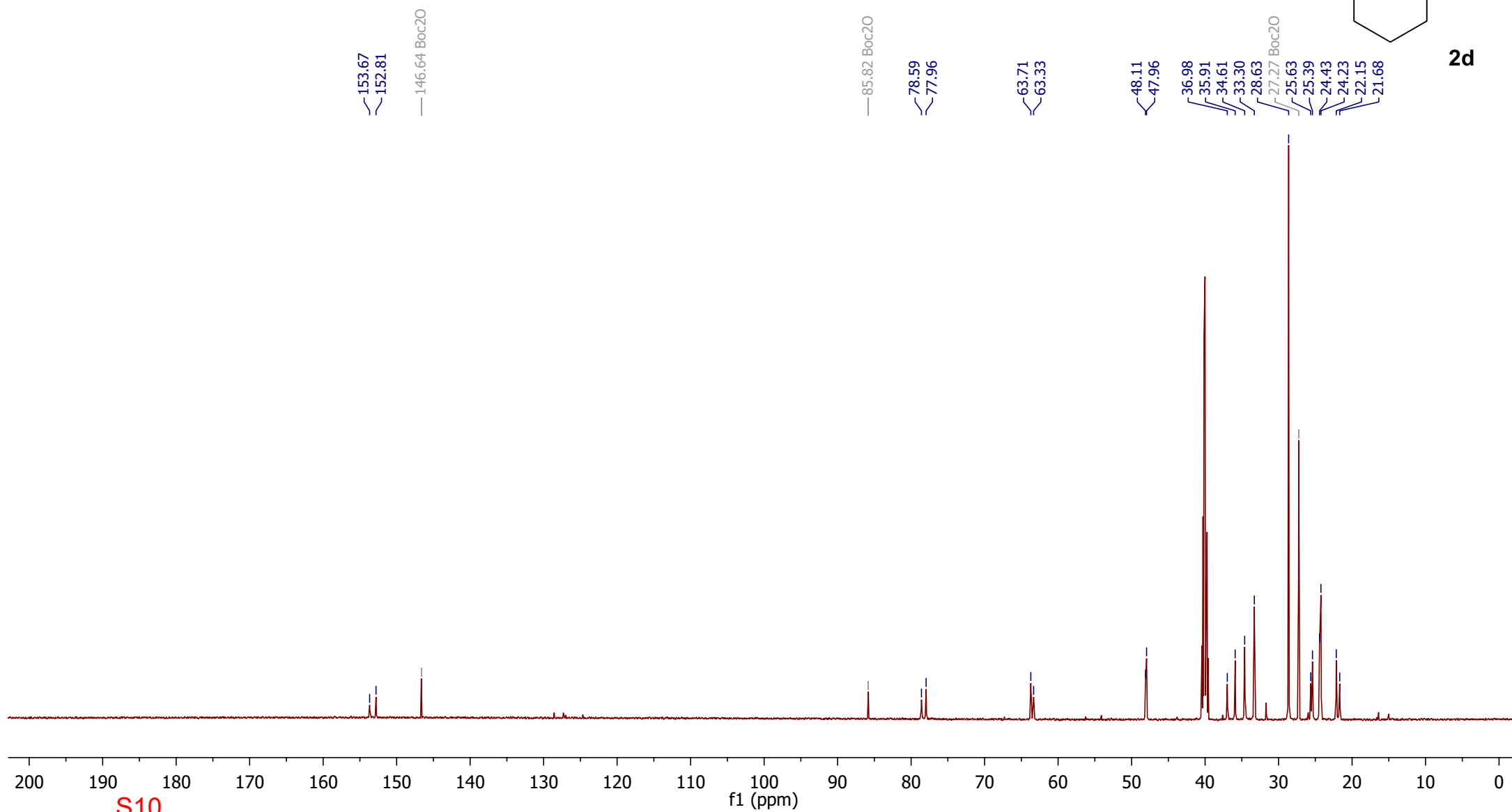
2d

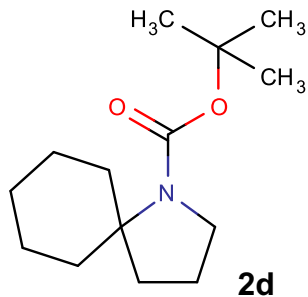


^{13}C NMR (151 MHz, $\text{dms}\text{-}d_6$) δ 153.67, 152.81, 78.59, 77.96, 63.71, 63.33, 48.11, 47.96, 36.98, 35.91, 34.61, 33.30, 28.63, 25.63, 25.39, 24.43, 24.23, 22.15, 21.68



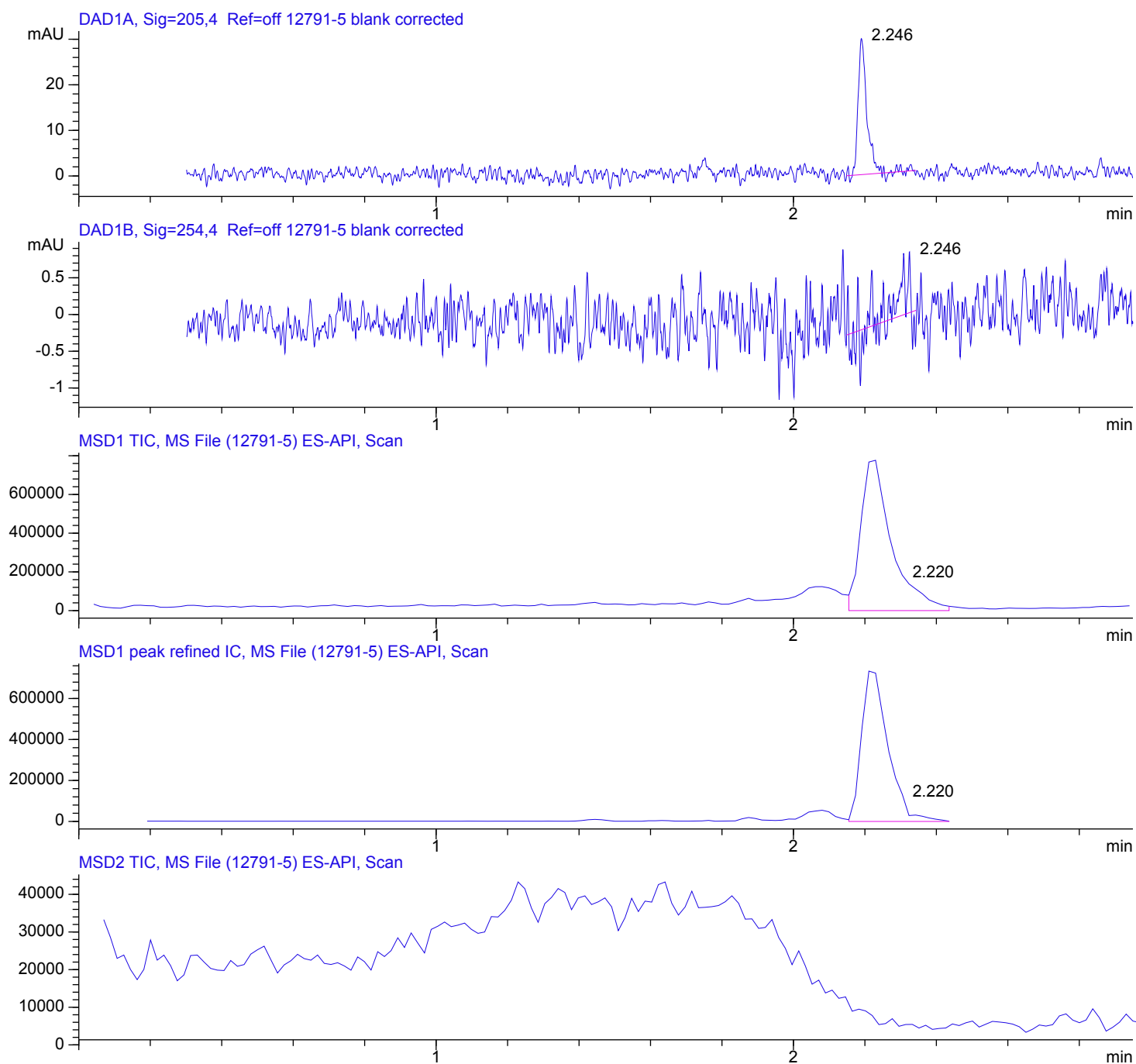
2d

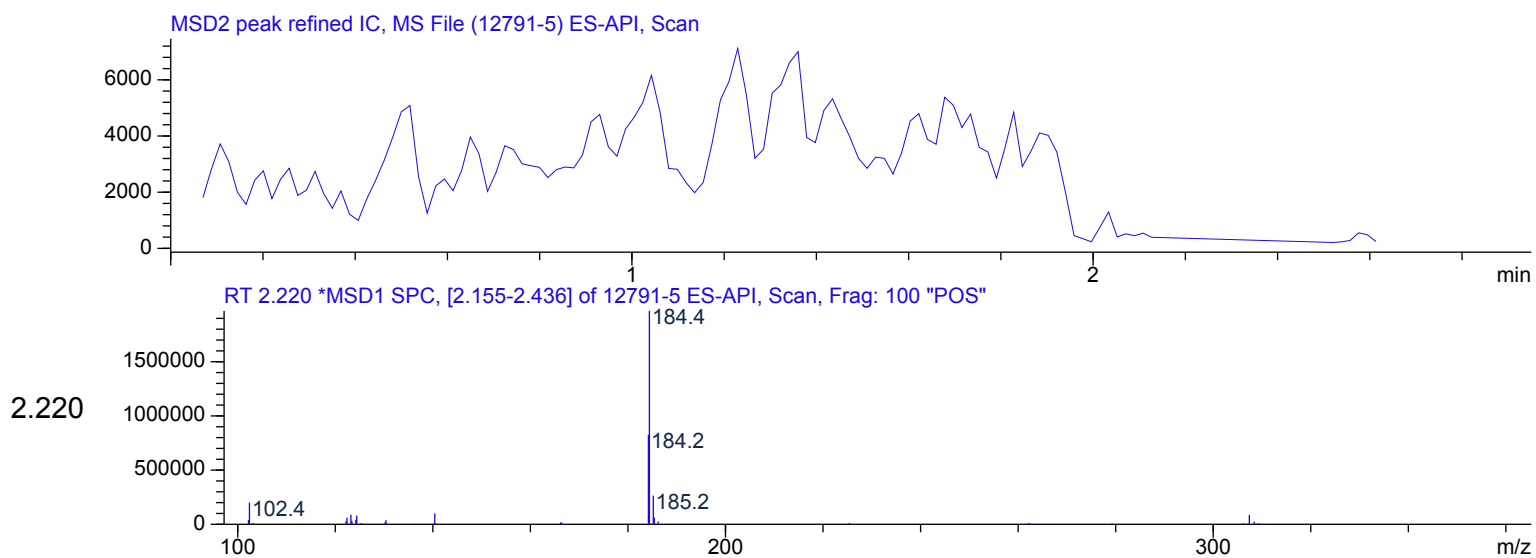




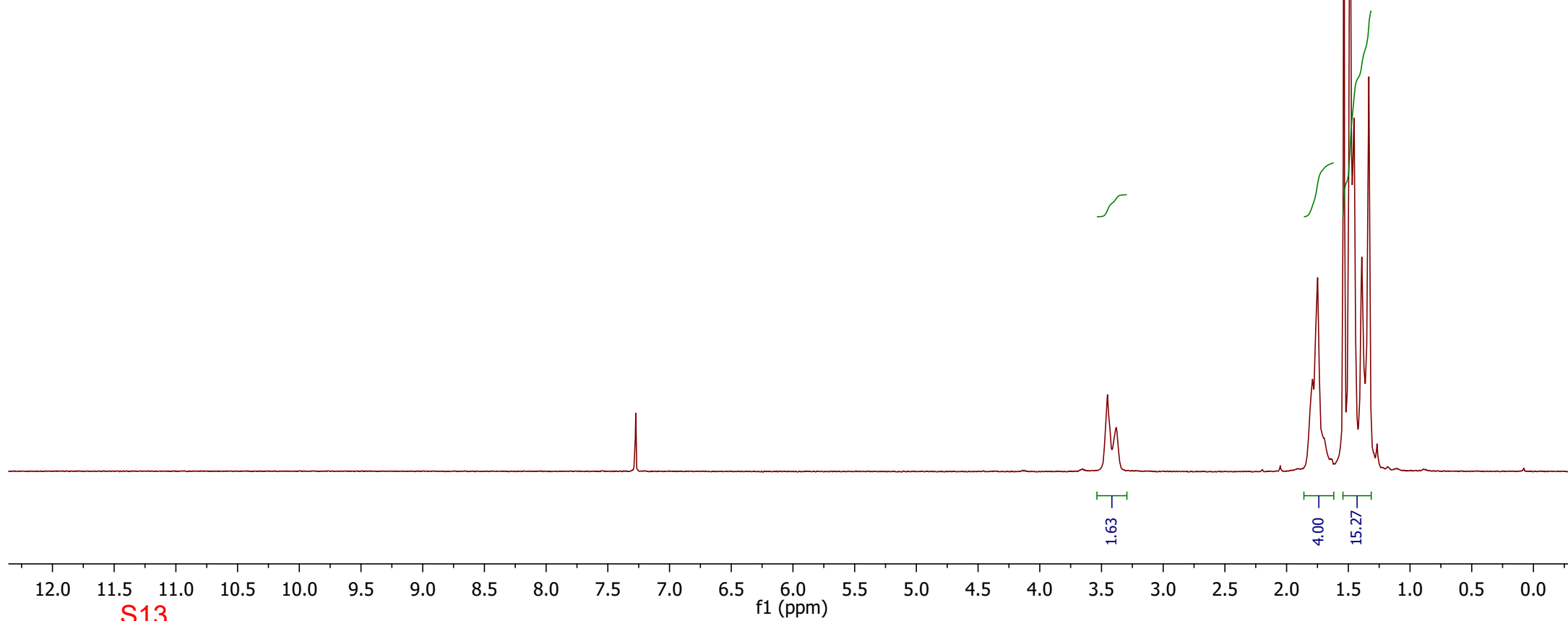
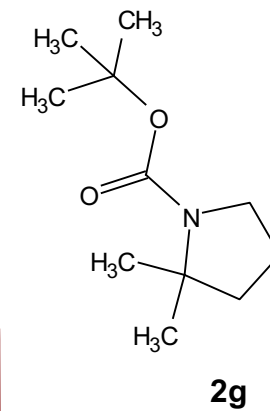
[239.19]

#	RT	DAD1A	DAD1B	MSD1	MSD2	ELSD	MSD1 ions	MSD1 rt	MSD2 ions	MSD2 rt	Info
1	2.246	100.0%	100.0%	100.0%	---	---	184.4(90),123.2(3),140.4(3)	2.220	---	---	

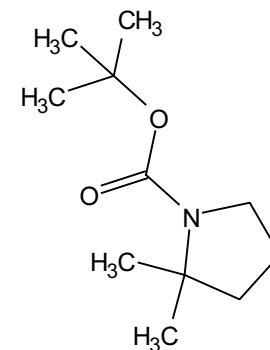




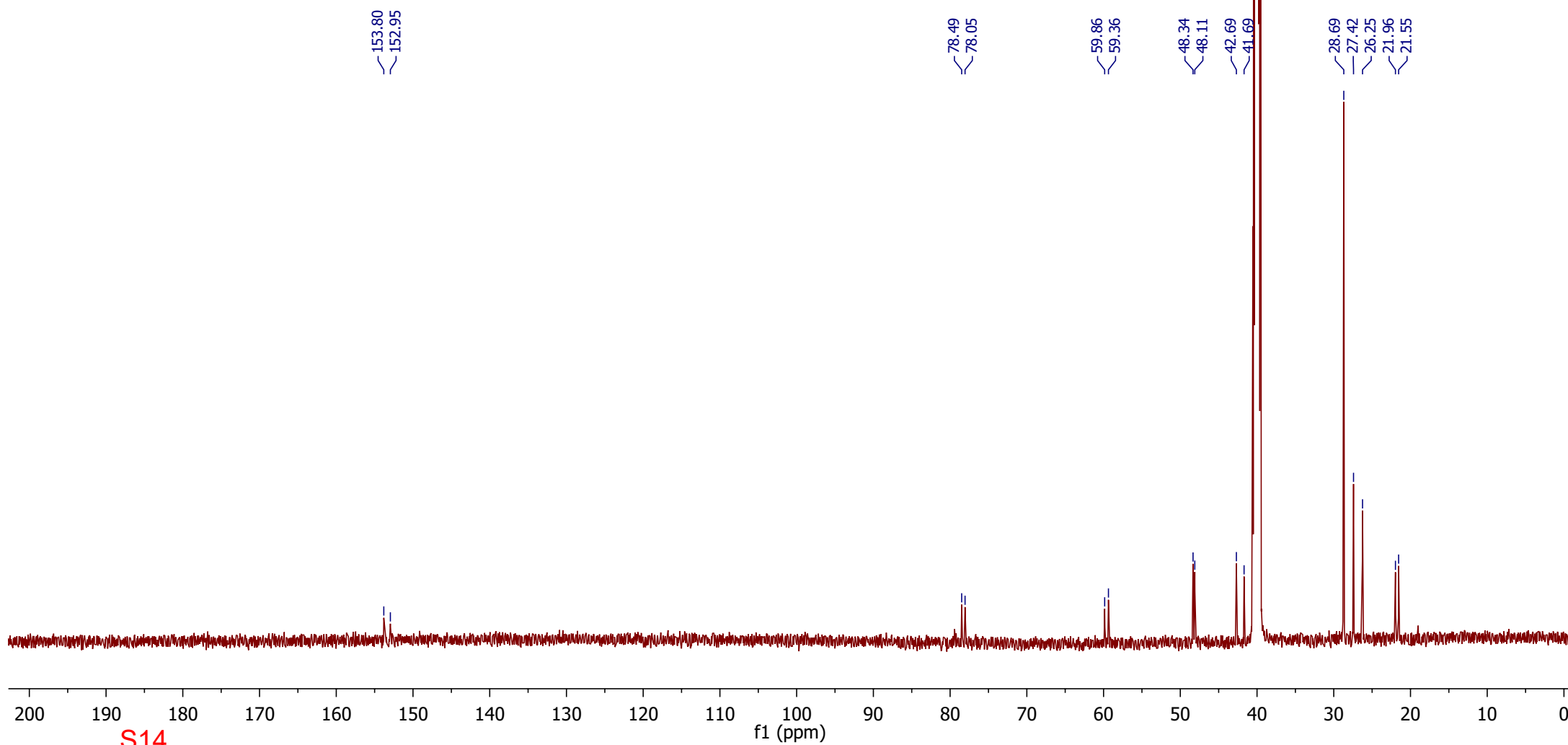
^1H NMR (400 MHz, Chloroform-*d*) δ 3.54 – 3.29 (m, 2H), 1.86 – 1.62 (m, 4H), 1.54 – 1.31 (m, 15H).

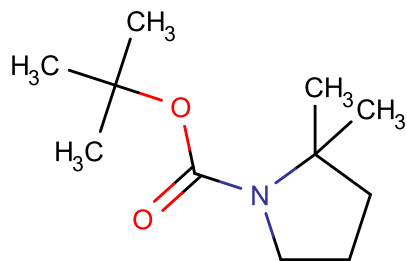


^{13}C NMR (151 MHz, dms-o) δ 153.80, 152.95, 78.49, 78.05, 59.86, 59.36, 48.34, 48.11, 42.69, 41.69, 28.69, 27.42, 26.25, 21.96, 21.55



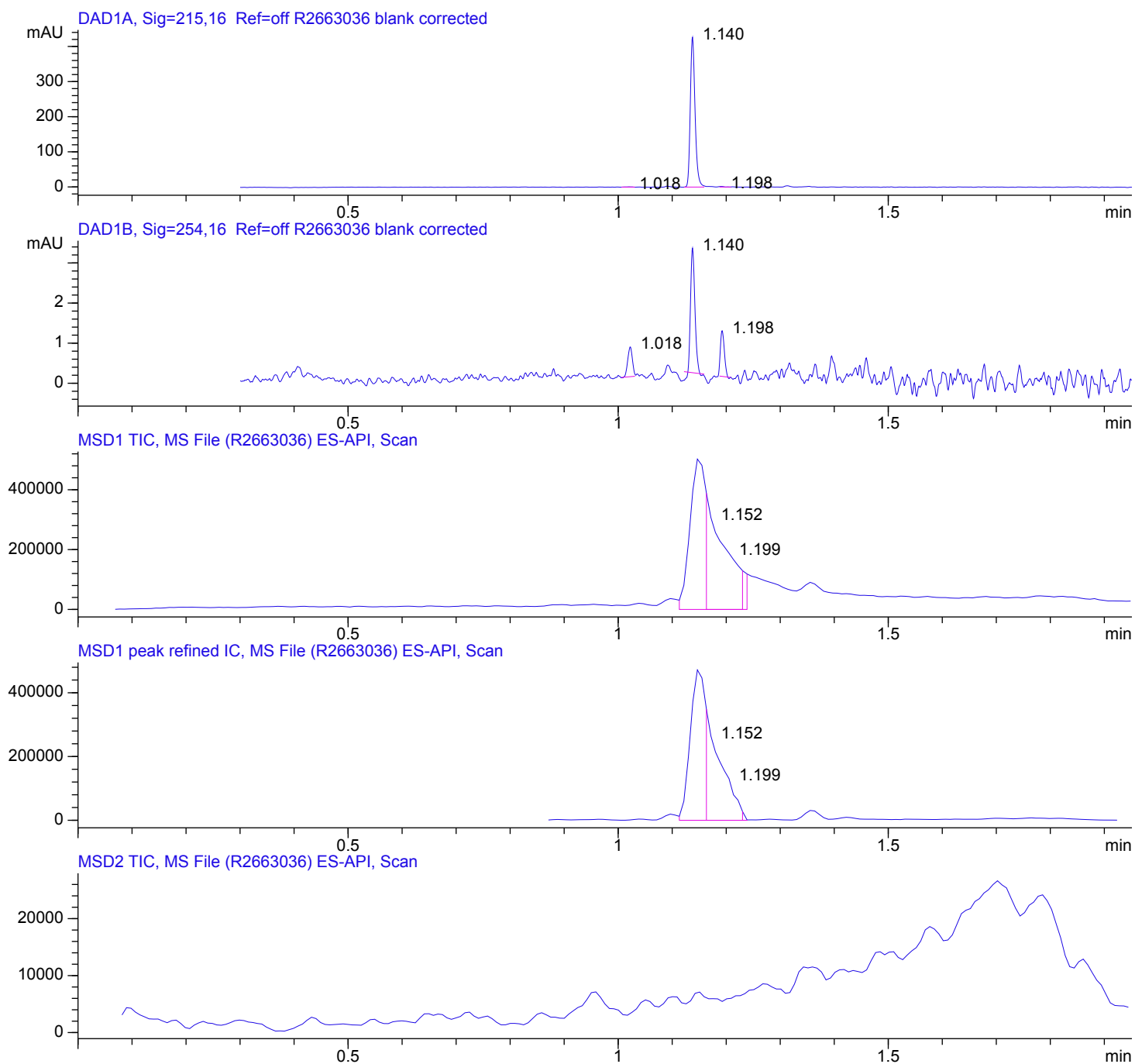
2g



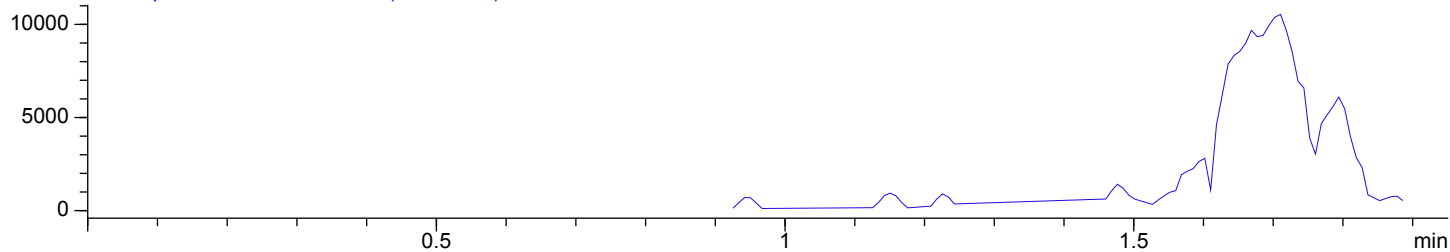


[199.16] **2g**

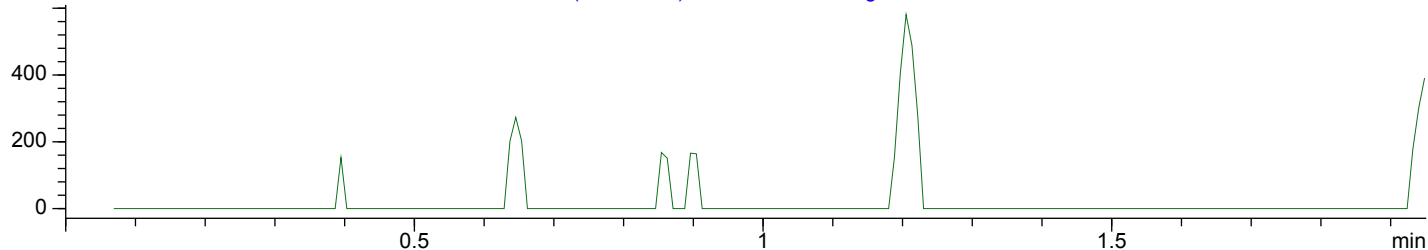
#	RT	DAD1A	DAD1B	MSD1	MSD2	ELSD	MSD1 ions	MSD1 rt	MSD2 ions	MSD2 rt	Info
1	1.018	0.1%	16.5%	---	---	110.4%	---	---	---	---	
2	1.140	99.6%	62.2%	99.3%	---	-44.2%	421.2(36),126.2(27),144.2(17)	1.152	---	---	
3	1.198	0.3%	21.3%	0.7%	---	33.7%	479.2(100)	1.199	---	---	



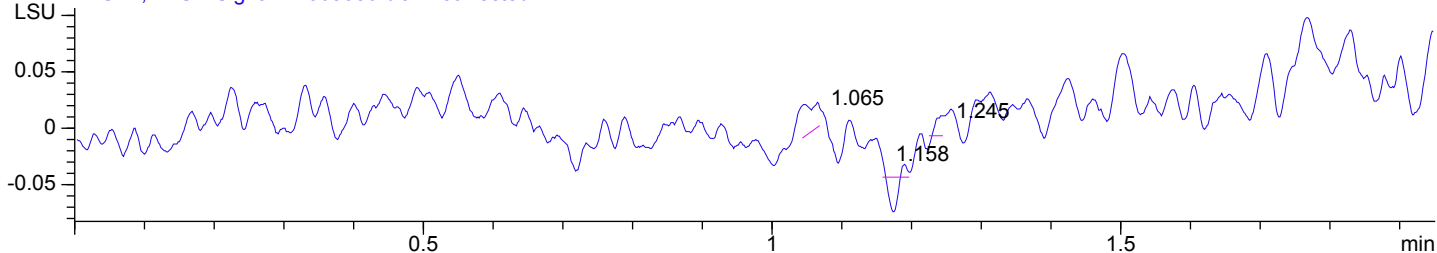
MSD2 peak refined IC, MS File (R2663036) ES-API, Scan



Ion Extract m/z 199.6-200.6 from MSD1 TIC, MS File (R2663036) ES-API, Scan, Frag: 100, "POS"

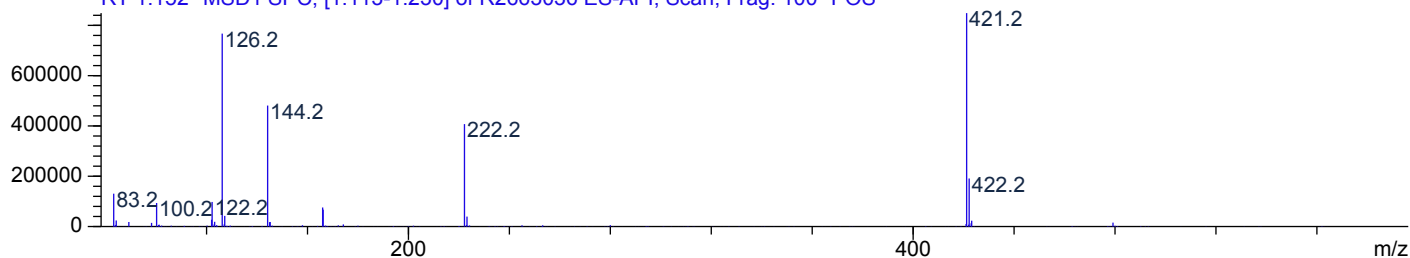


ELS1A, ELSD Signal R2663036 blank corrected



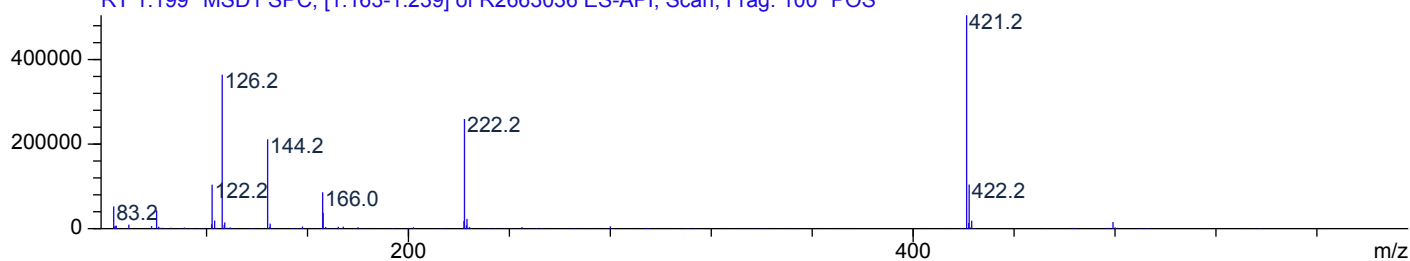
RT 1.152 *MSD1 SPC, [1.113-1.230] of R2663036 ES-API, Scan, Frag: 100 "POS"

1.152

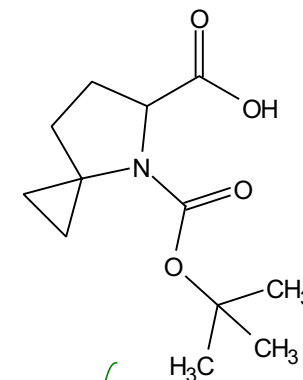


RT 1.199 *MSD1 SPC, [1.163-1.239] of R2663036 ES-API, Scan, Frag: 100 "POS"

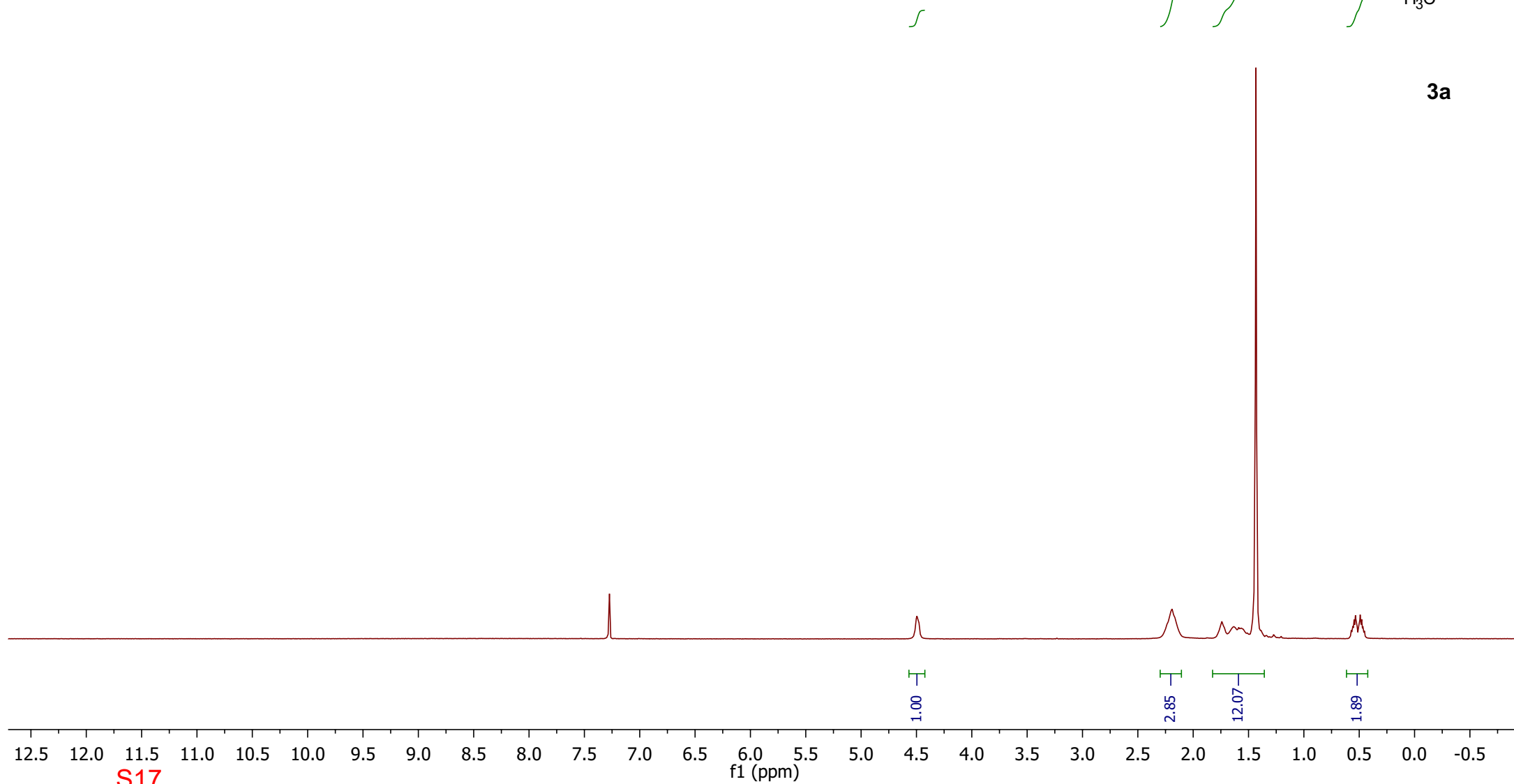
1.199



^1H NMR (400 MHz, Chloroform-*d*) δ 4.49 (s, 1H), 2.19 (s, 3H), 1.82 – 1.36 (m, 12H), 0.51 (dtd, J = 19.7, 10.2, 5.6 Hz, 2H).

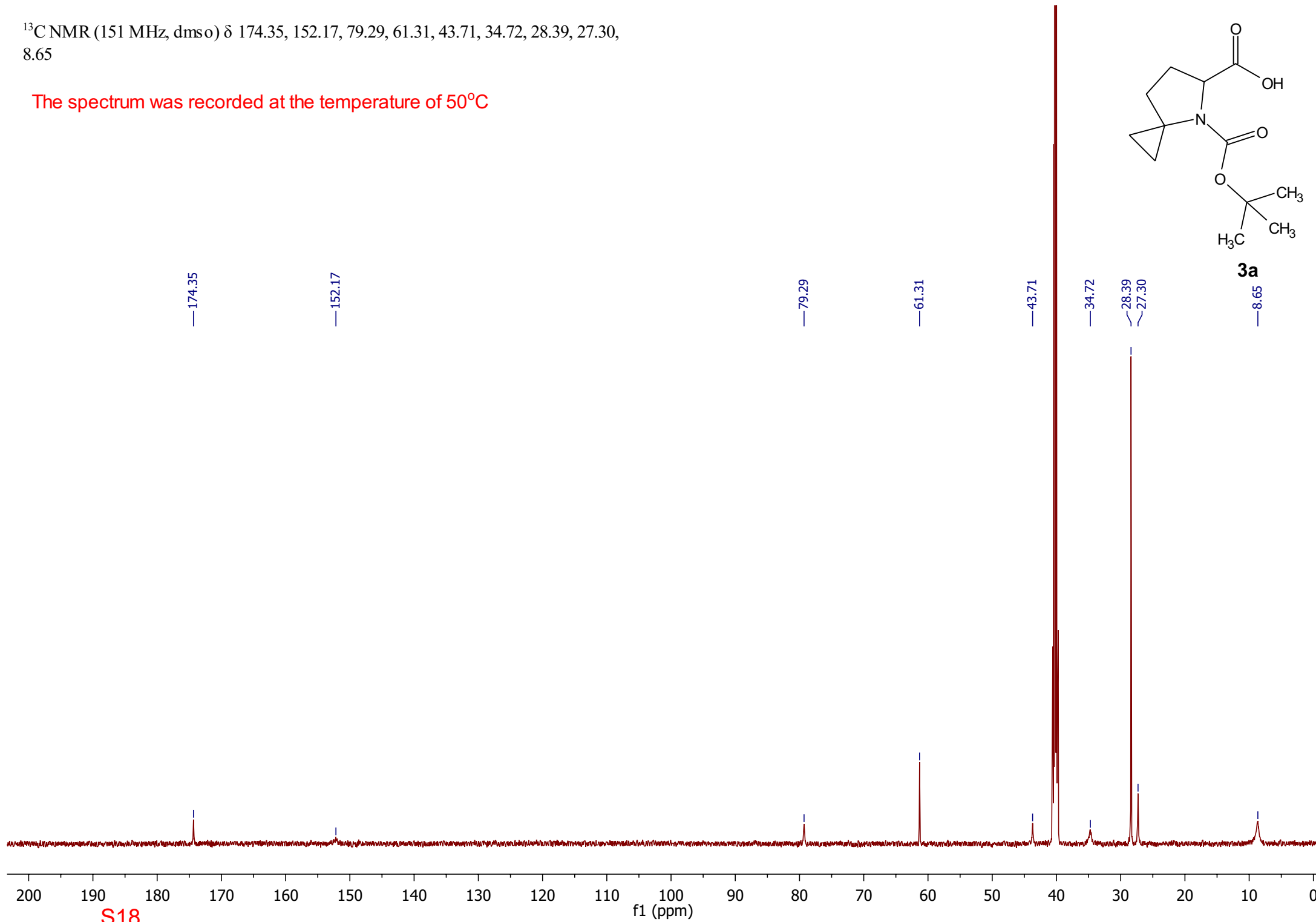
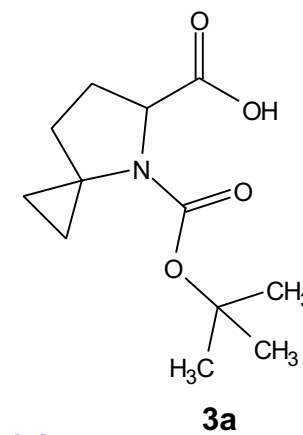


3a

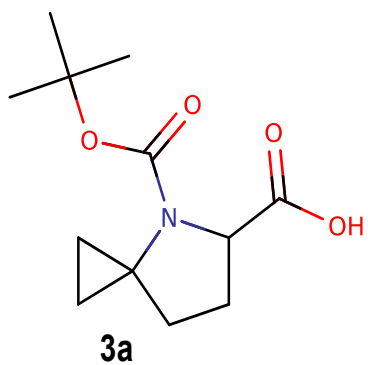


^{13}C NMR (151 MHz, dms-o) δ 174.35, 152.17, 79.29, 61.31, 43.71, 34.72, 28.39, 27.30, 8.65

The spectrum was recorded at the temperature of 50°C



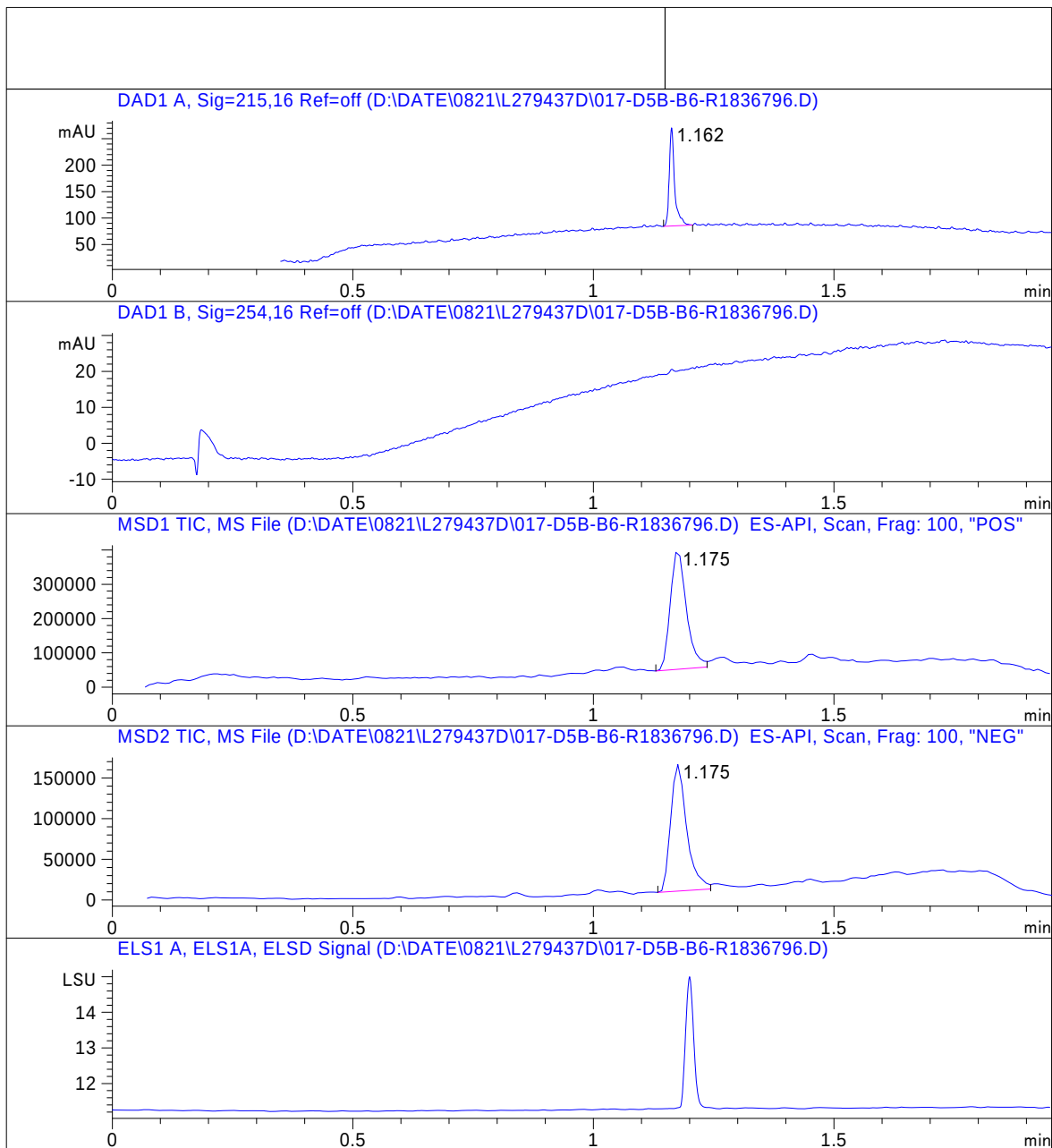
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Ret_Time: 1.162 min



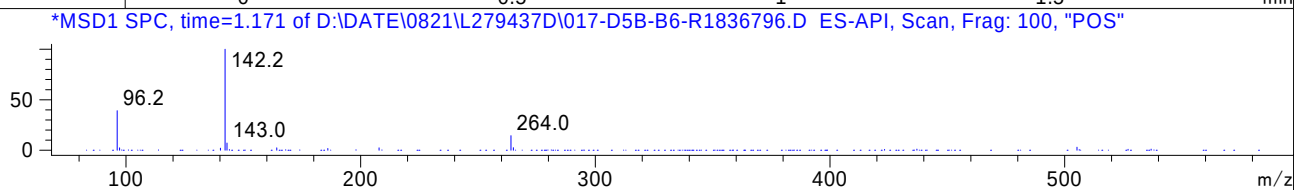
Mol Wt 241.28

Exact Mass 241.15

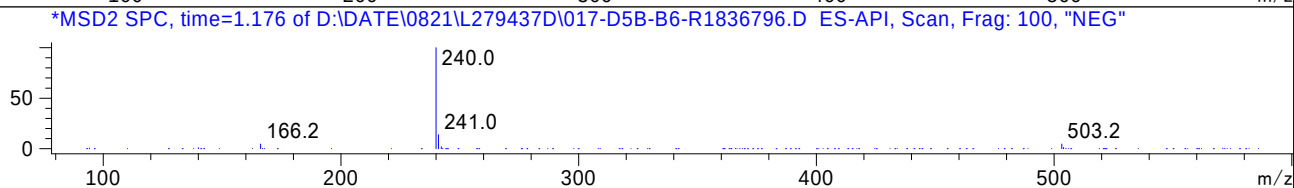
#	Time	Area%
1	1.162	100.00



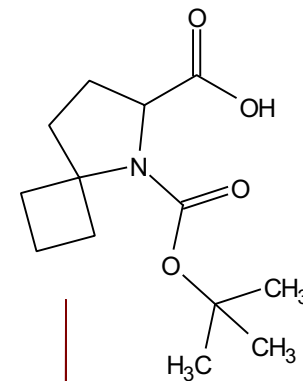
RT 1.175



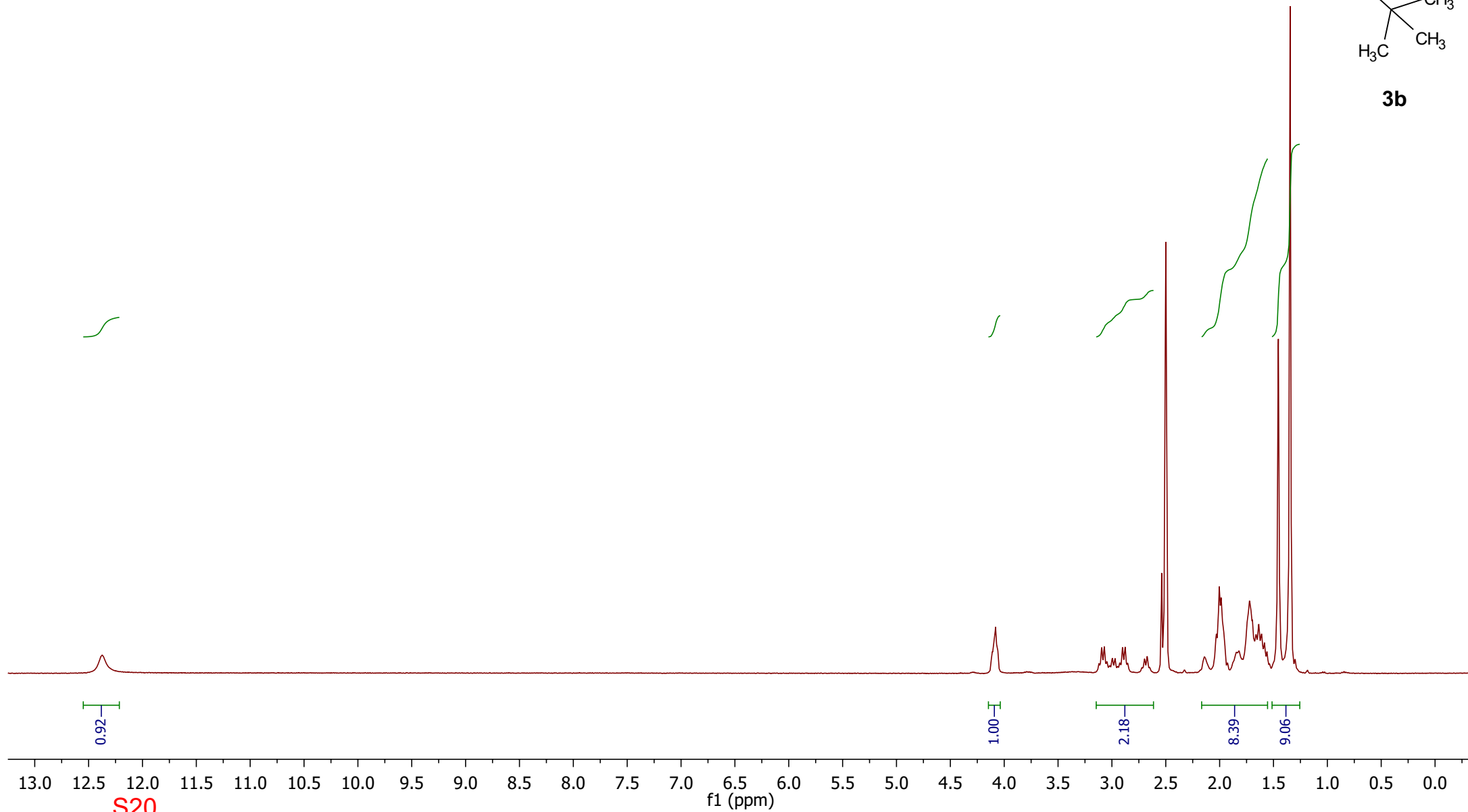
RT 1.175



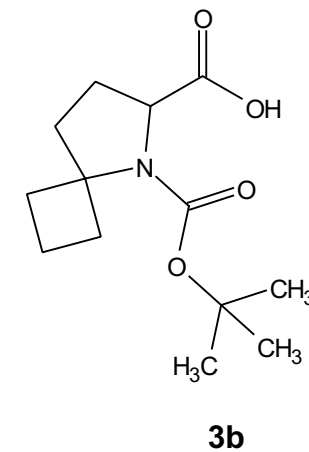
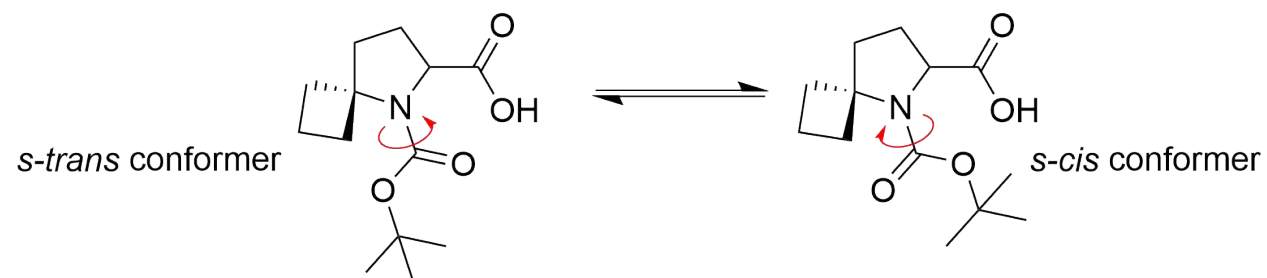
^1H NMR (400 MHz, $\text{DMSO}-d_6$) δ 12.37 (s, 1H), 4.15 – 4.04 (m, 1H), 3.15 – 2.61 (m, 2H), 2.17 – 1.56 (m, 8H), 1.51 – 1.26 (m, 9H).



3b



^{13}C NMR (151 MHz, dms o) δ 174.69, 174.17, 154.02, 153.11, 79.49, 79.02, 63.92, 63.15, 61.20, 38.42, 34.34, 34.19, 32.88, 32.42, 28.57, 28.40, 26.61, 26.22, 13.55, 13.00



174.69
174.17

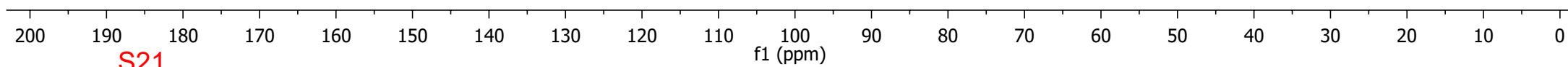
154.02
153.11

79.49
79.02

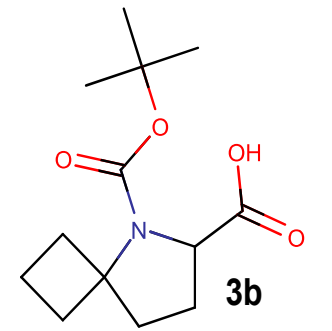
63.92
63.15
61.20

38.42
34.34
34.19
32.88
32.42
28.57
28.40
26.61
26.22

13.55
13.00

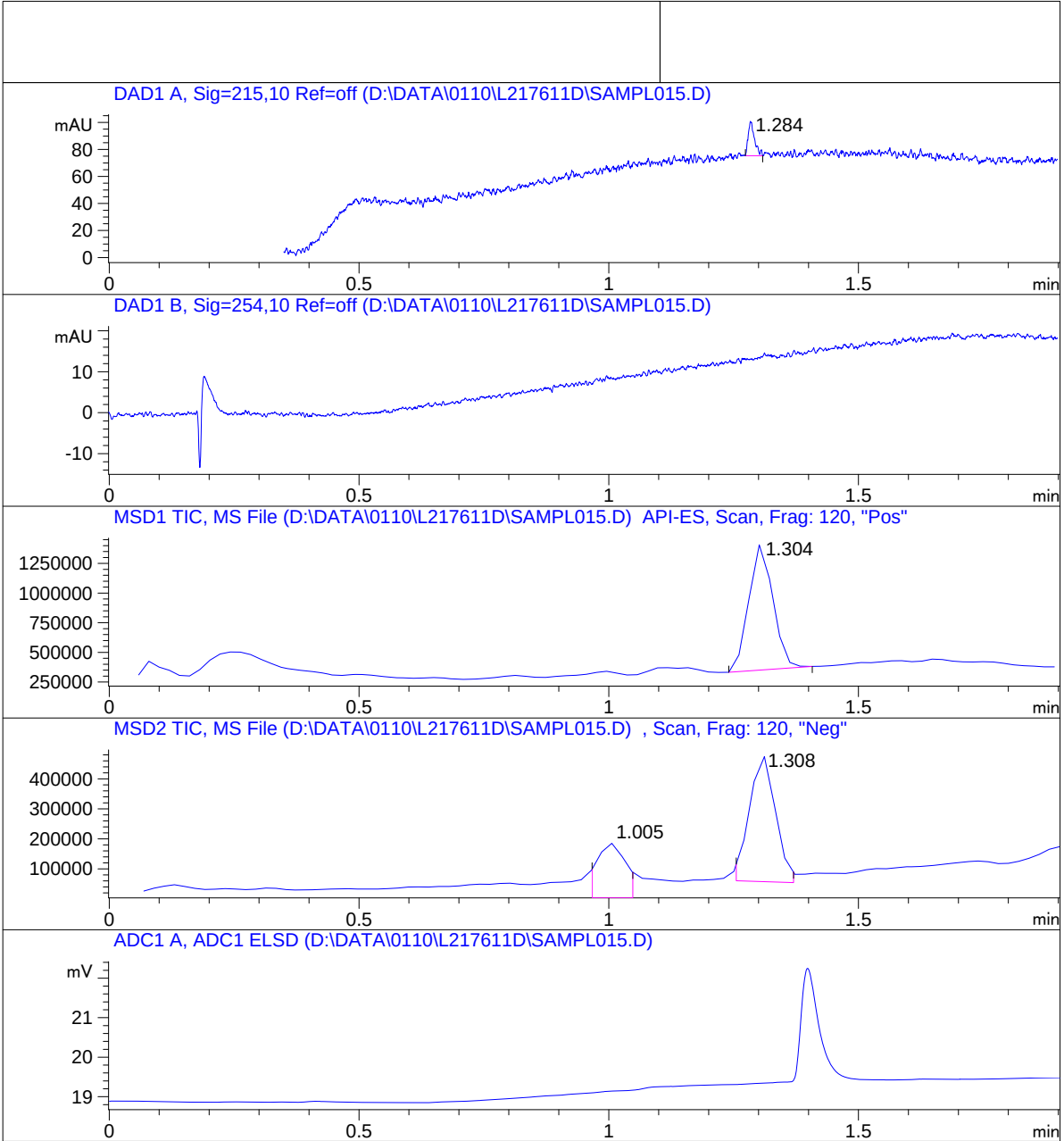


MaxPeak: 100.00%
Ret_Time: 1.284 min

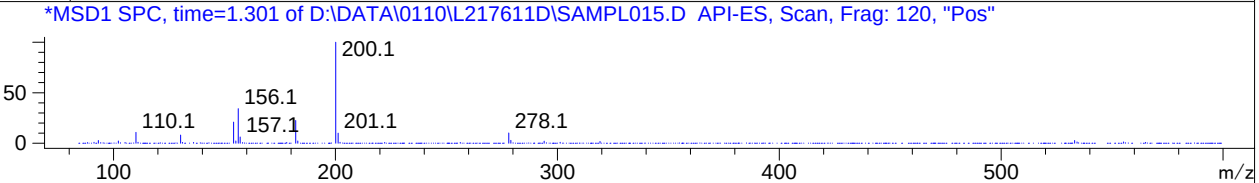


Mol Wt	255.31	
Exact Mass	255.17	
#	Time	Area%

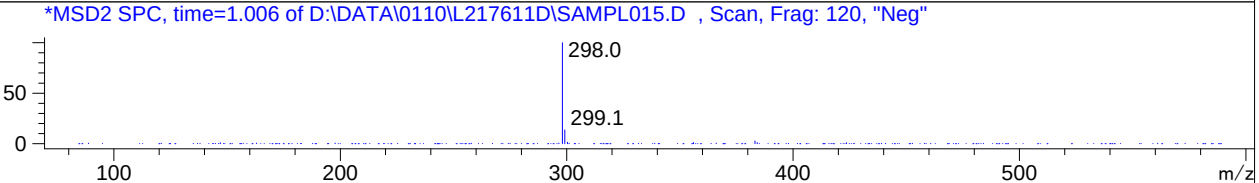
1	1.284	100.00



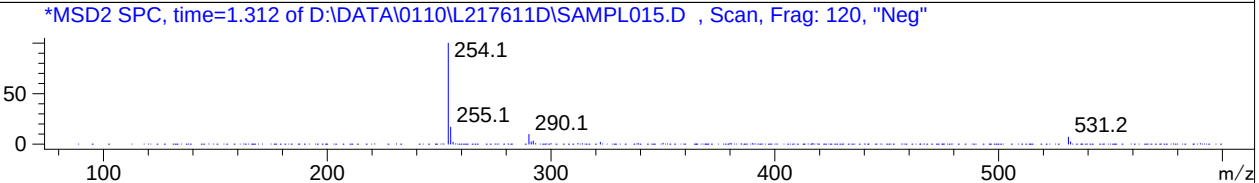
RT 1.304



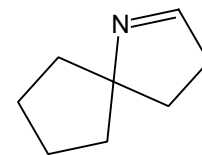
RT 1.005



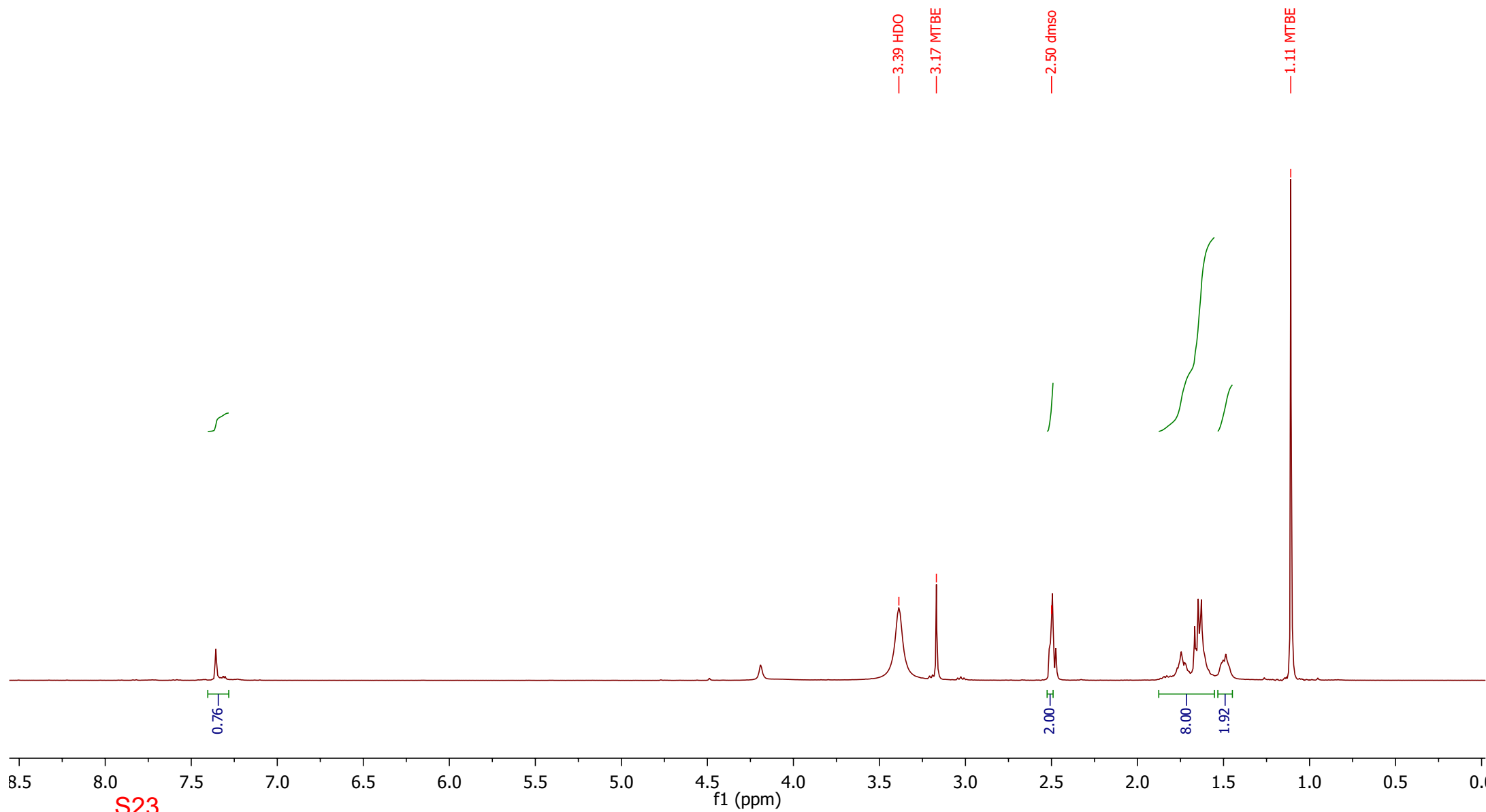
RT 1.308



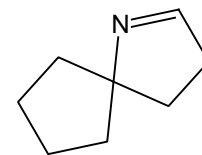
^1H NMR (400 MHz, $\text{DMSO}-d_6$) δ 7.36 (s, 1H), 2.53 – 2.49 (m, 2H), 1.88 – 1.55 (m, 8H), 1.53 – 1.45 (m, 2H).



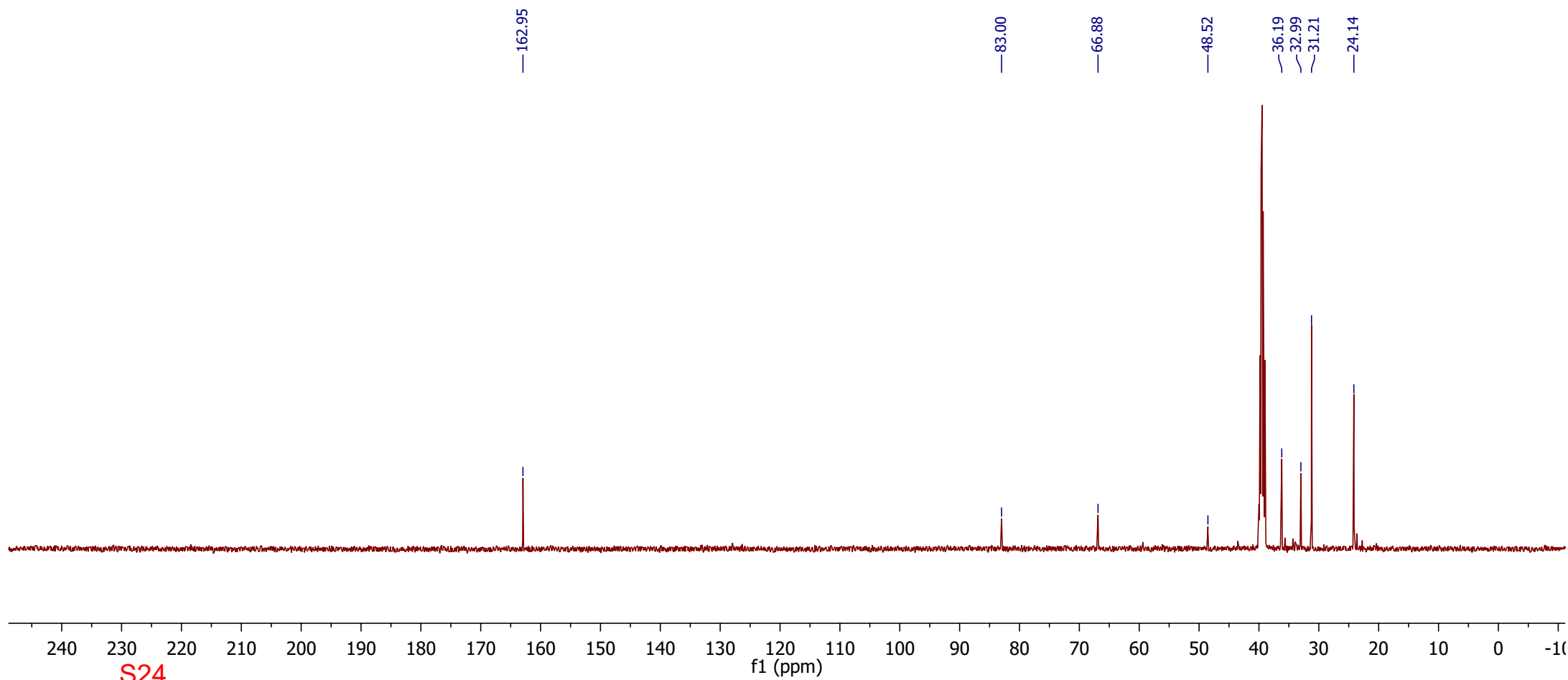
6c

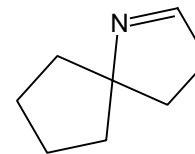


^{13}C NMR (DMSO, 126 MHz) δ 162.95, 83.00, 66.88, 48.52, 36.19, 32.99, 31.21, 24.14.



6c

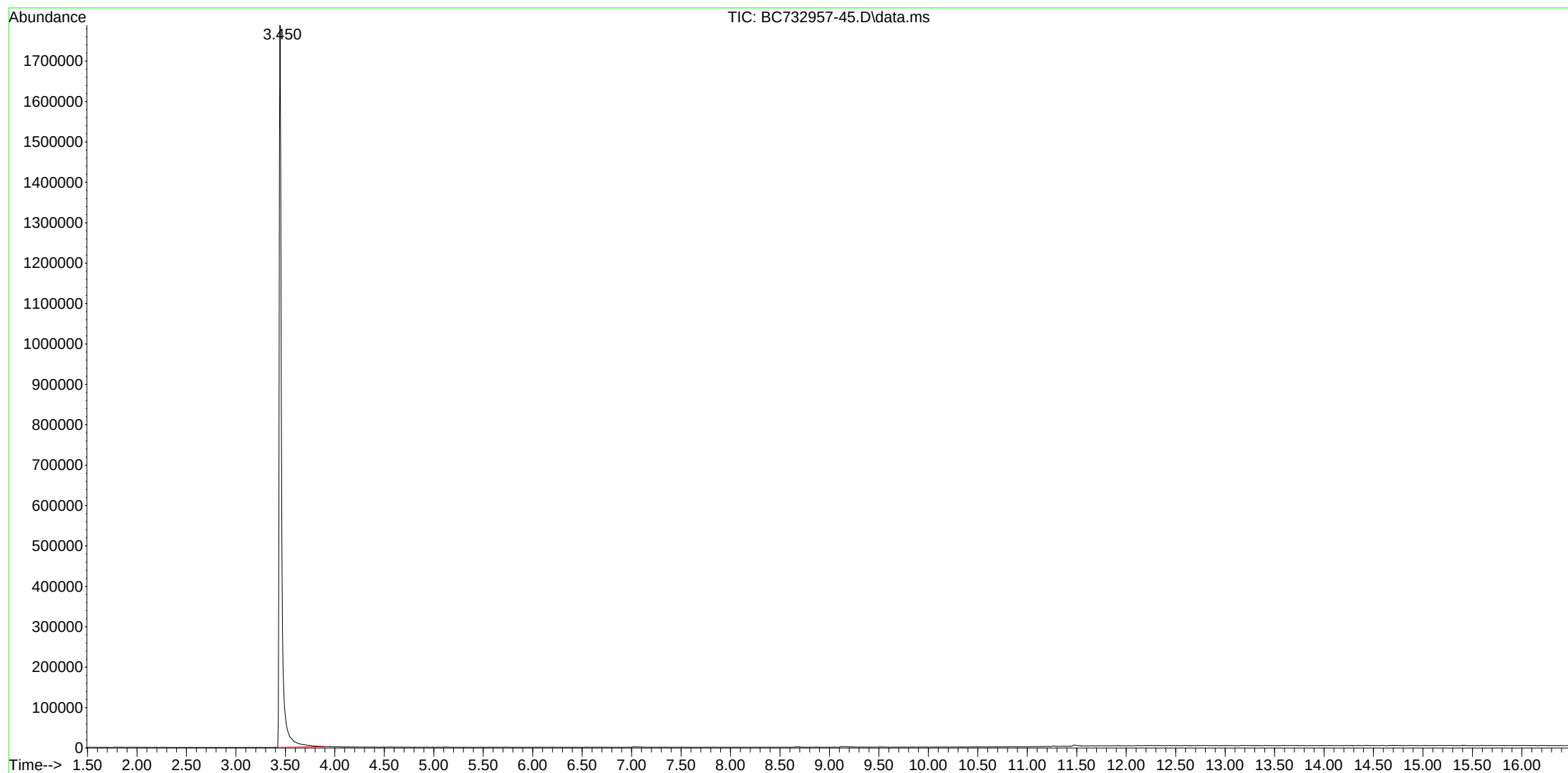


**6c**

Misc : CH3OH
ALS Vial : 8 Sample Multiplier: 1

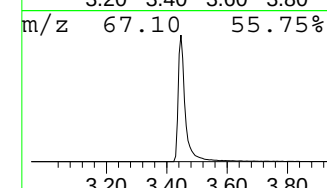
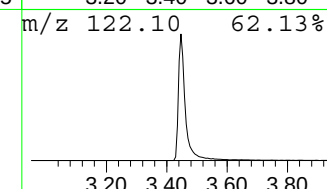
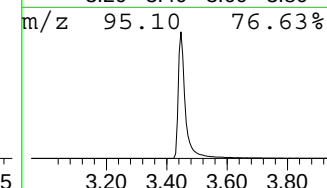
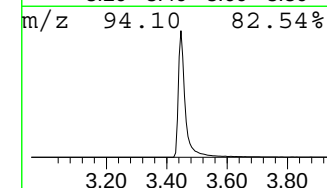
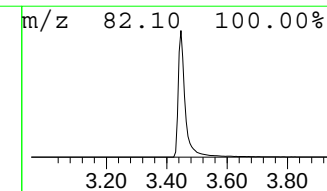
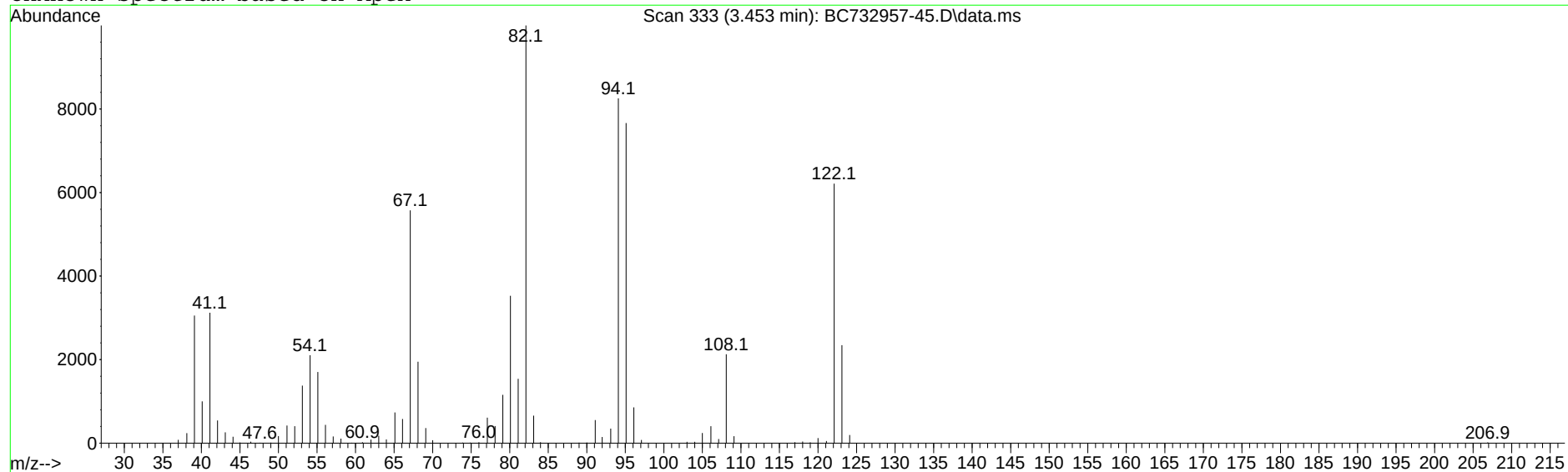
Search Libraries: C:\Database\EMPTY.L Minimum Quality: 0

Unknown Spectrum: Apex
Integration Events: ChemStation Integrator - autoint1.e



Unknown Spectrum based on Apex

Scan 333 (3.453 min): BC732957-45.D\data.ms



Peak Number: 1 at 3.450 min Area: 29507845 Area % 100.00

The 3 best hits from each library.

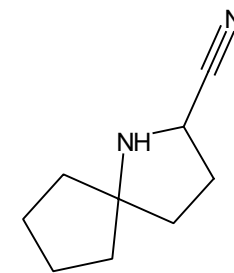
Ref\#

CAS\#

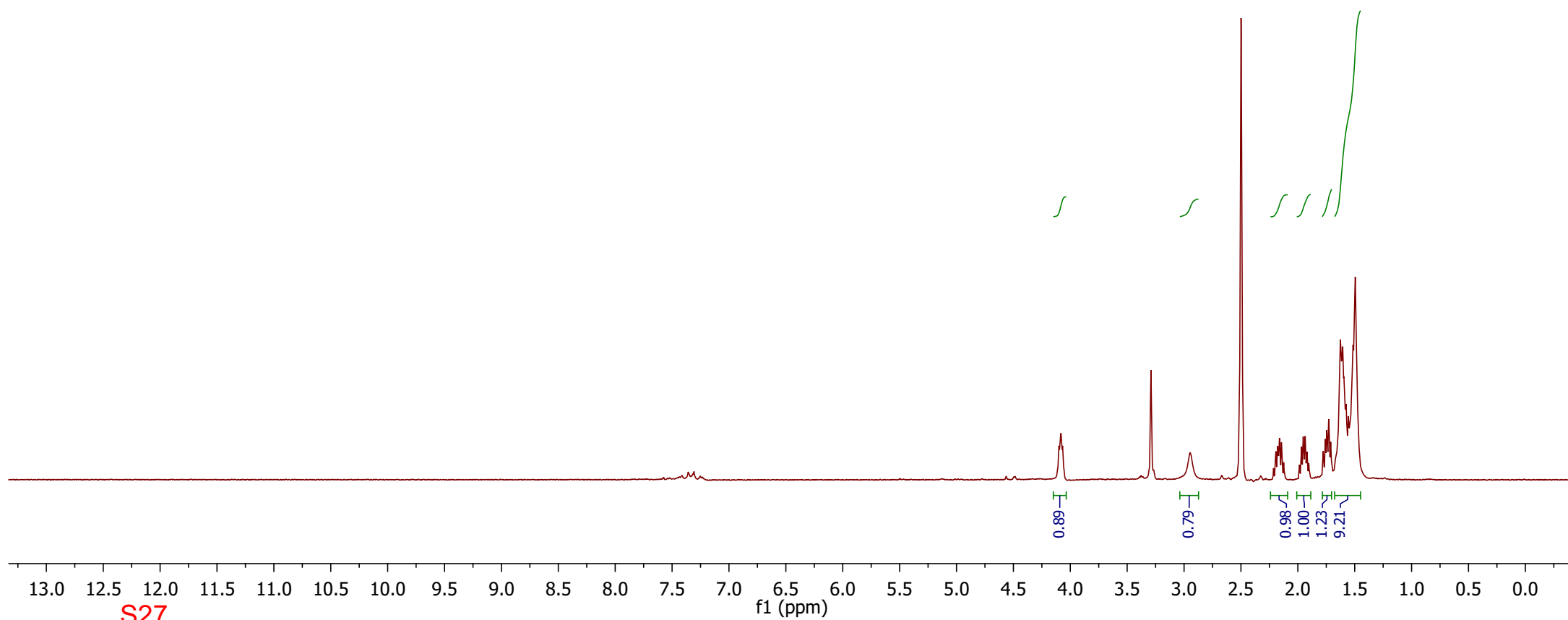
Qual

C:\Database\EMPTY.L No hits were retrieved.

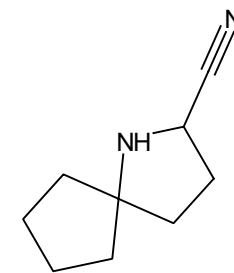
^1H NMR (400 MHz, $\text{DMSO}-d_6$) δ 4.08 (dd, $J = 8.4, 5.4$ Hz, 1H), 2.95 (s, 1H), 2.17 (dq, $J = 14.8, 7.8$ Hz, 1H), 1.94 (dq, $J = 12.8, 6.6$ Hz, 1H), 1.74 (dt, $J = 12.4, 7.1$ Hz, 1H), 1.68 – 1.45 (m, 9H).



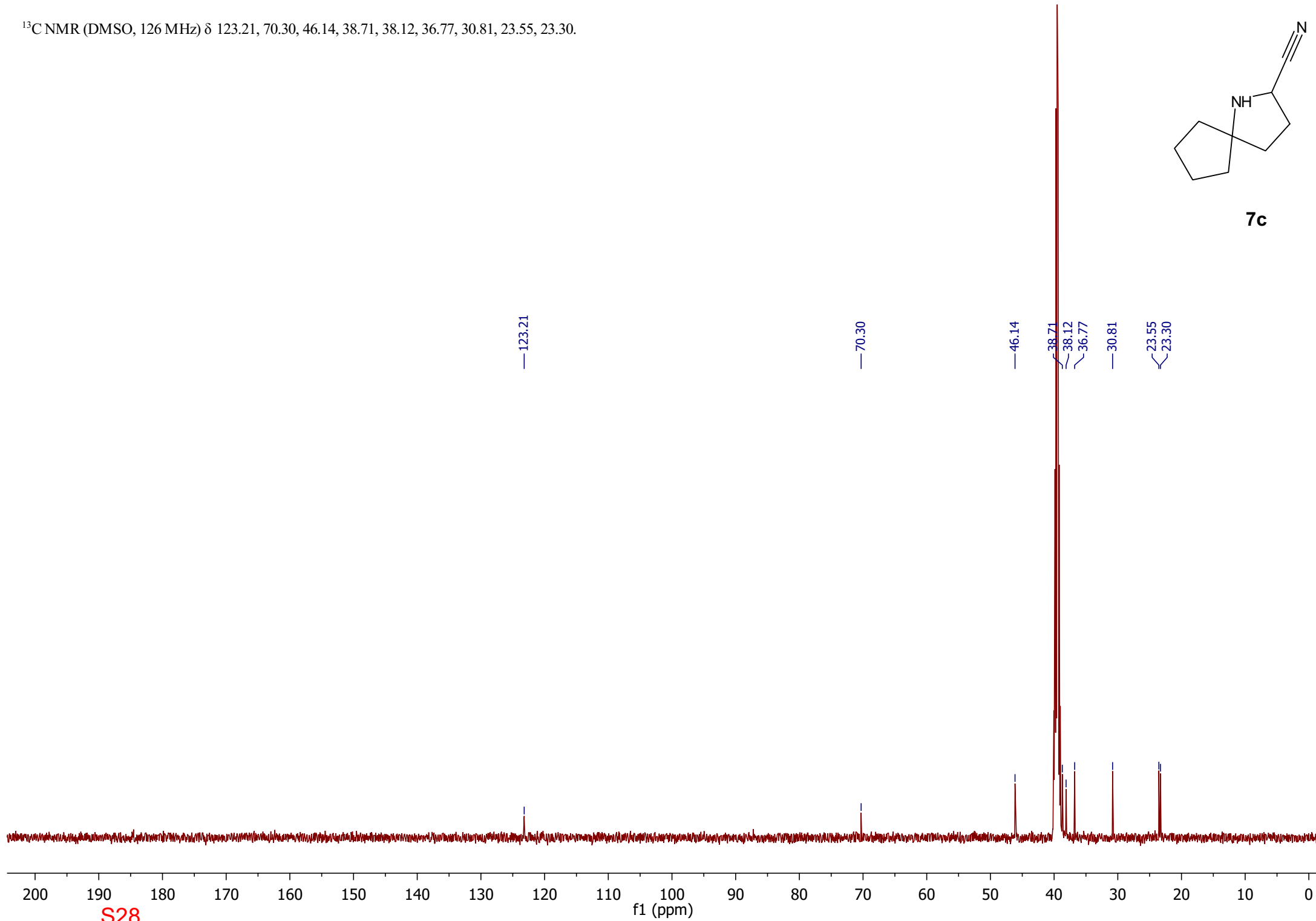
7c



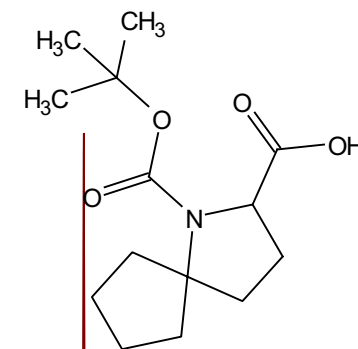
^{13}C NMR (DMSO, 126 MHz) δ 123.21, 70.30, 46.14, 38.71, 38.12, 36.77, 30.81, 23.55, 23.30.



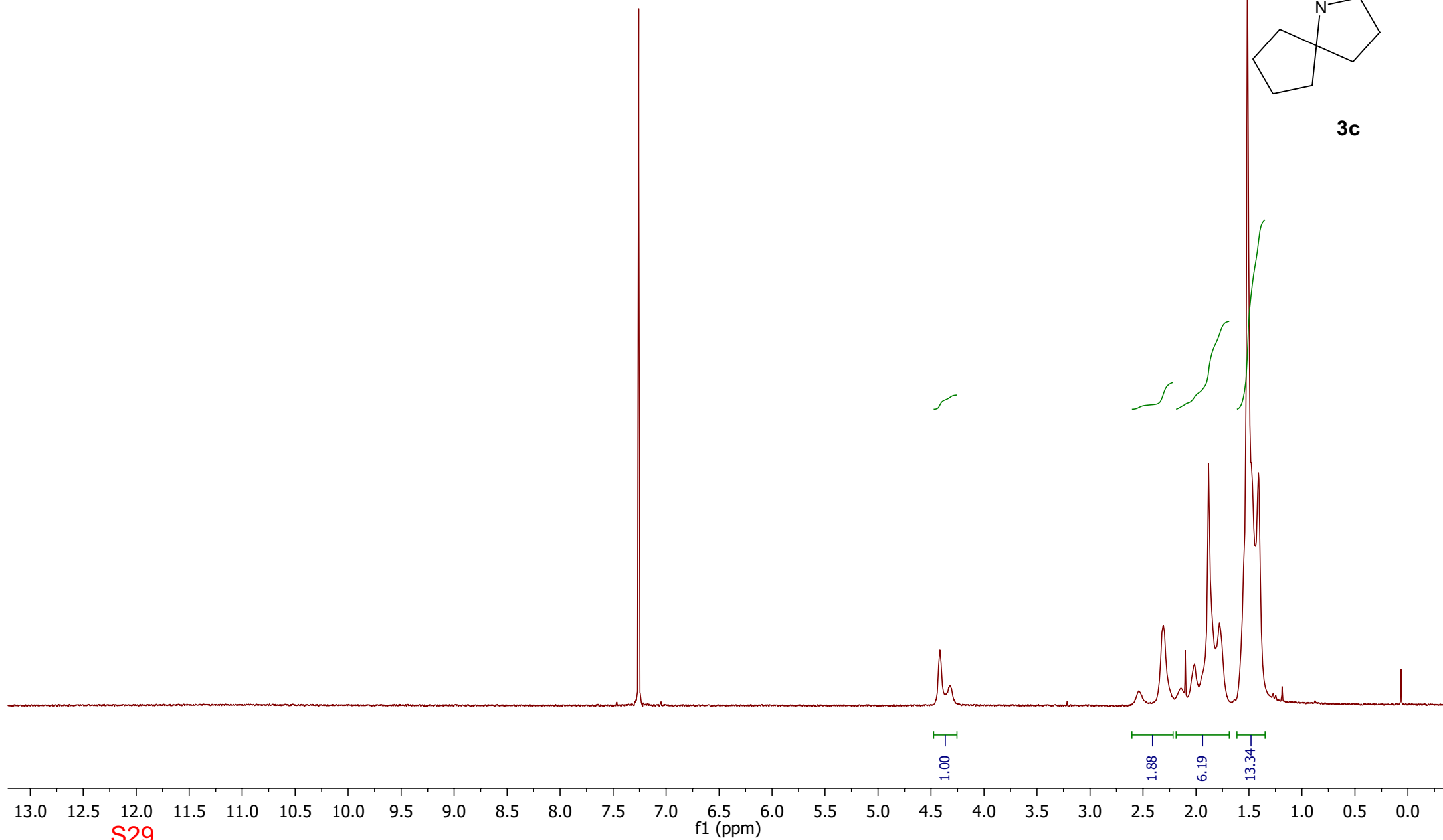
7c



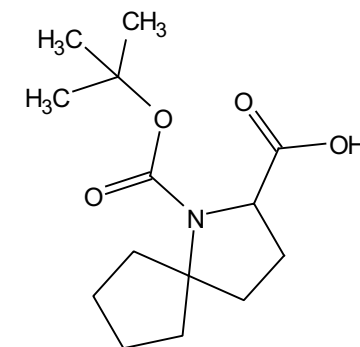
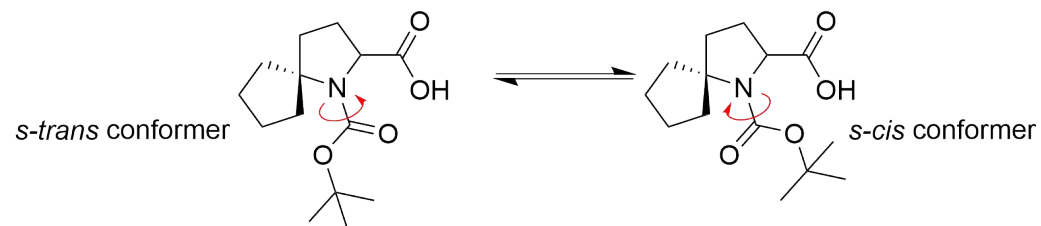
^1H NMR (500 MHz, Chloroform-*d*) δ 4.47 – 4.25 (m, 1H), 2.60 – 2.22 (m, 2H), 2.19 – 1.69 (m, 6H), 1.61 – 1.35 (m, 13H).



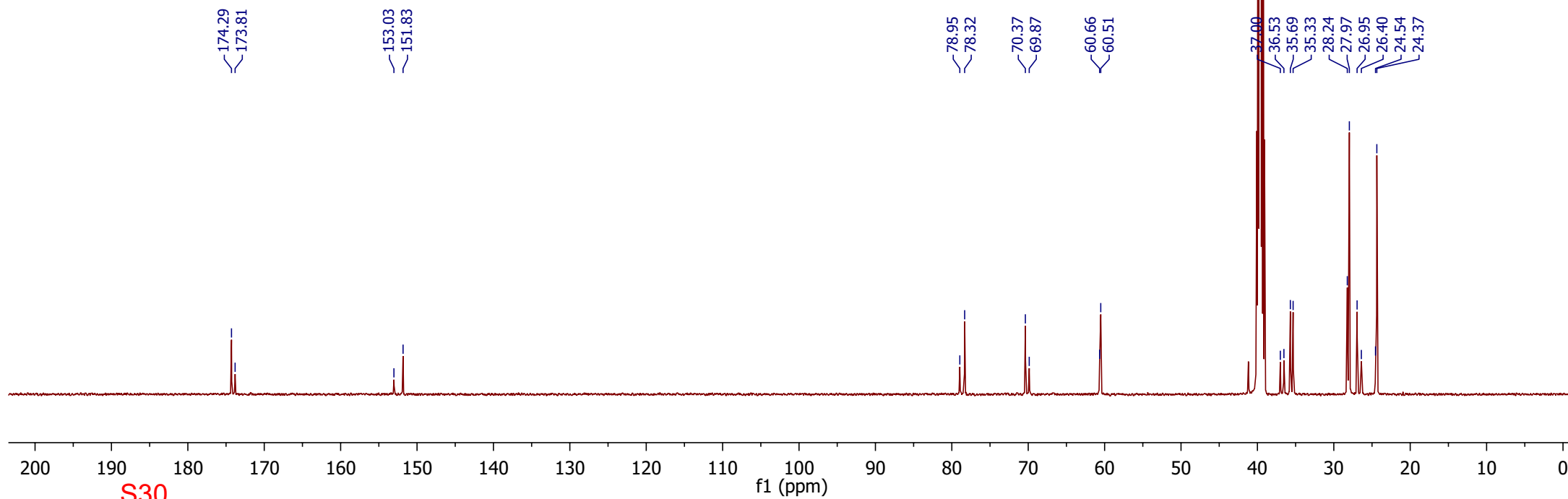
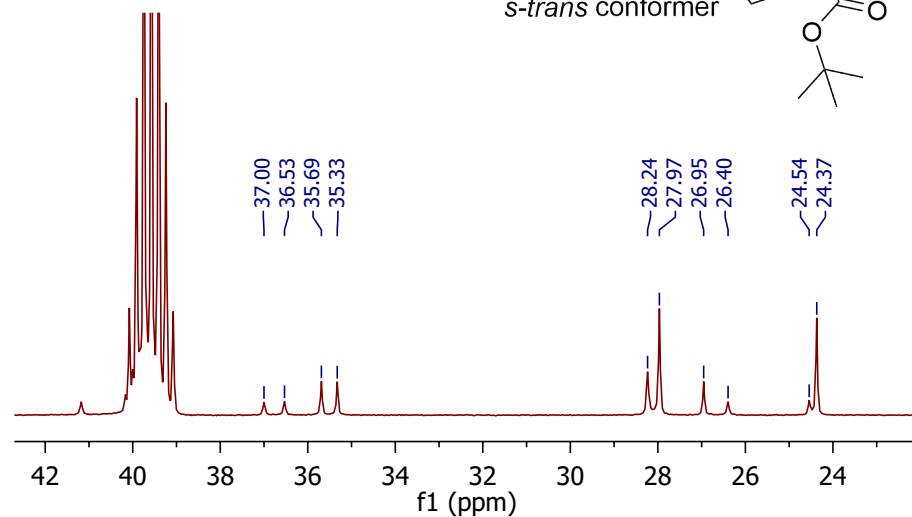
3c



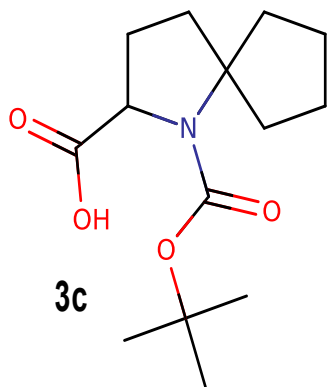
^{13}C NMR (126 MHz, DMSO) δ 174.29, 173.81, 153.03, 151.83, 78.95, 78.32, 70.37, 69.87, 60.66, 60.51, 37.00, 36.53, 35.69, 35.33, 28.24, 27.97, 26.95, 26.40, 24.54, 24.37



3c



MaxPeak: 100.00%
Ret_Time: 1.159 min



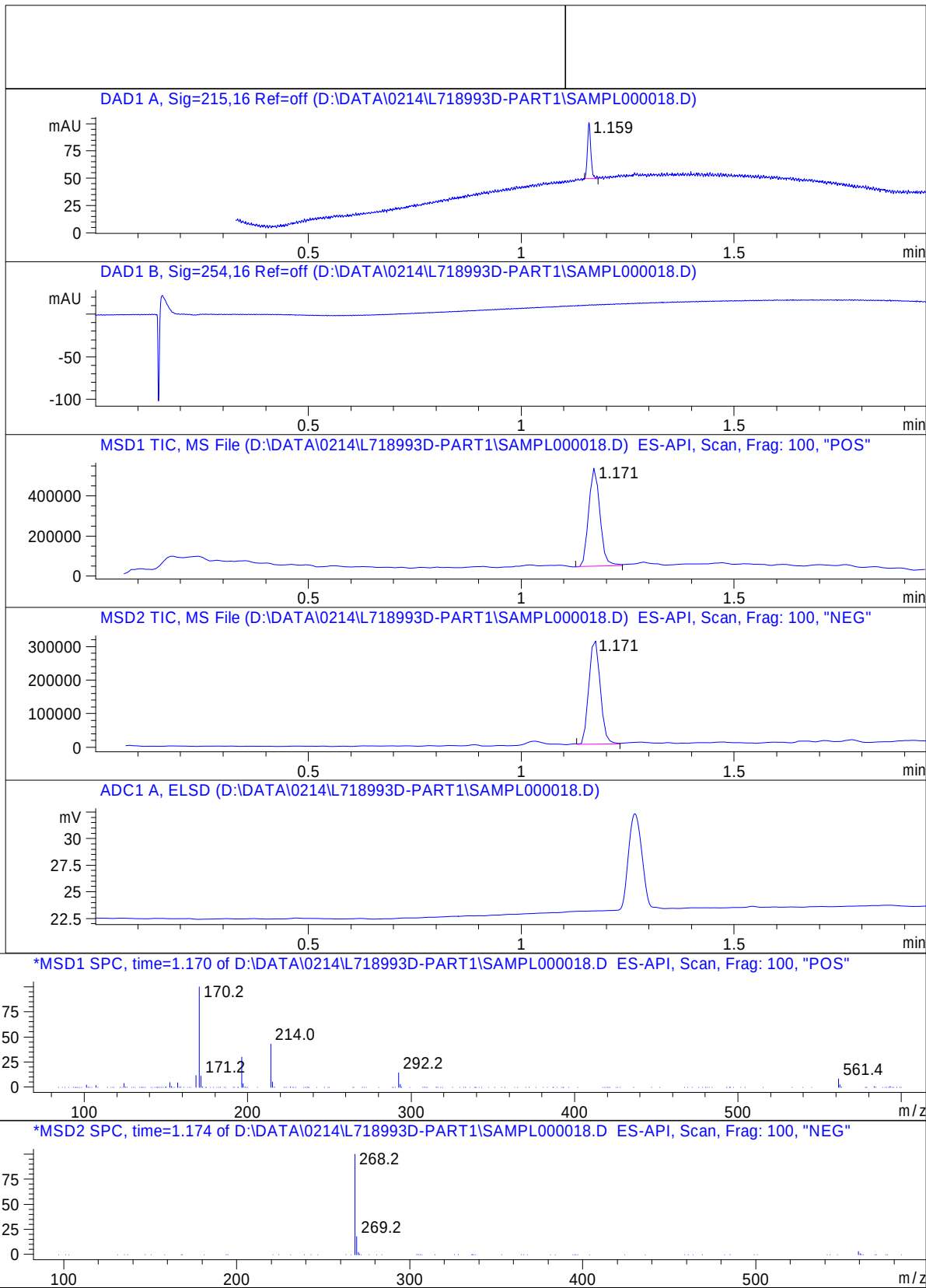
Mol Wt 269.34

Exact Mass 269.19

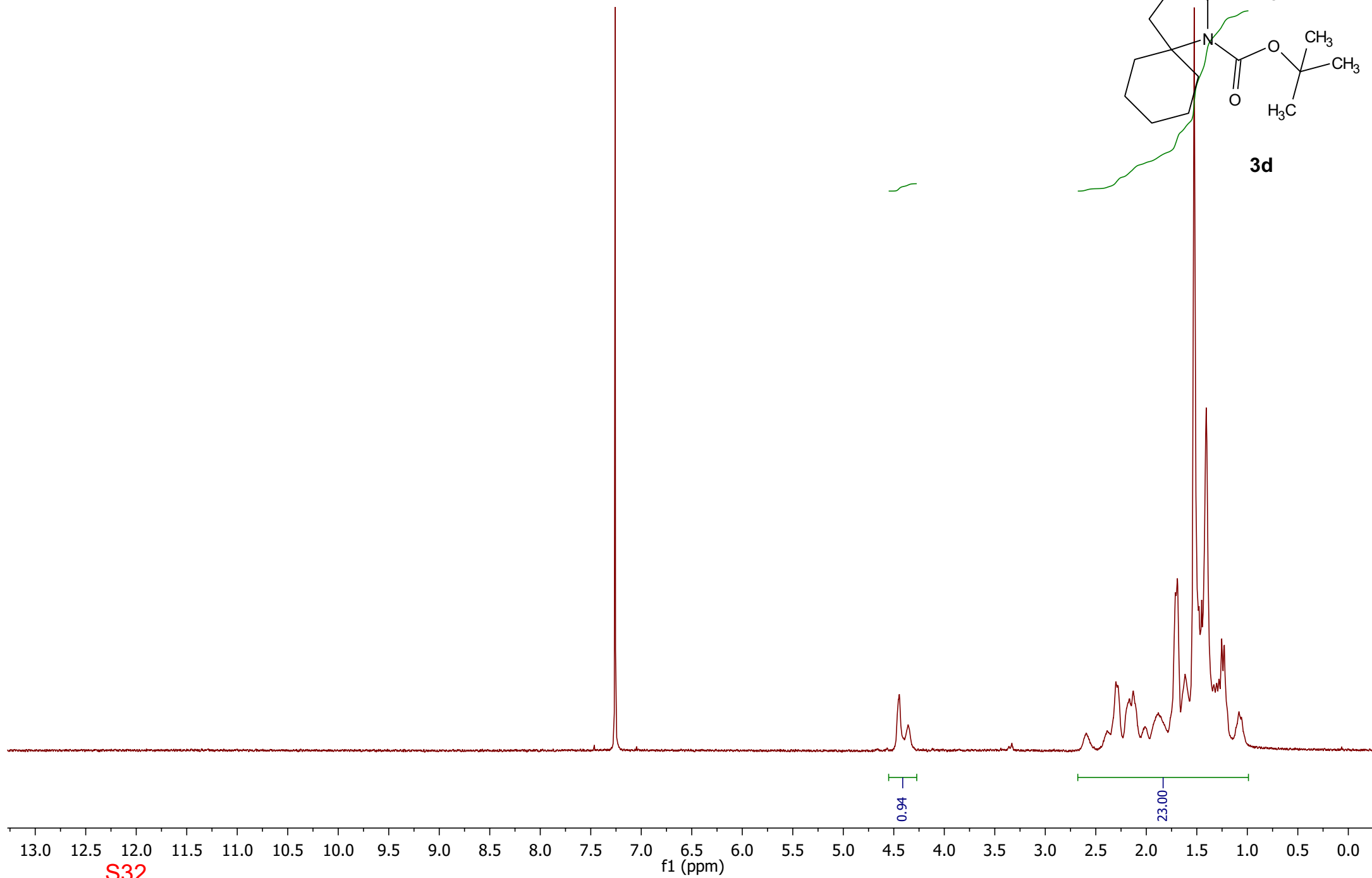
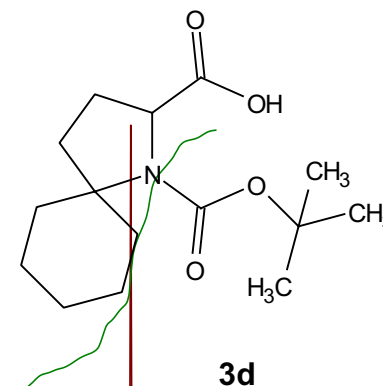
#	Time	Area%
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RT 1.171

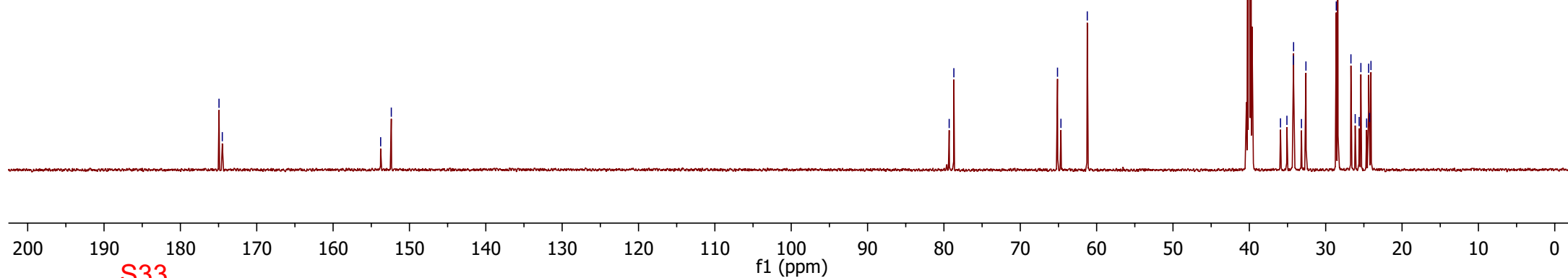
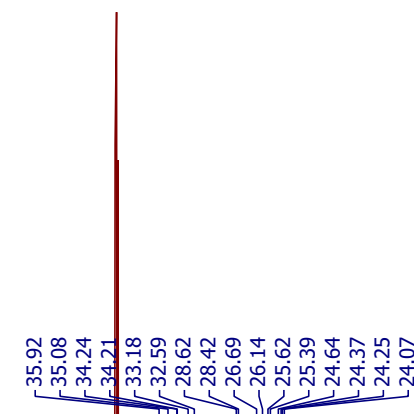
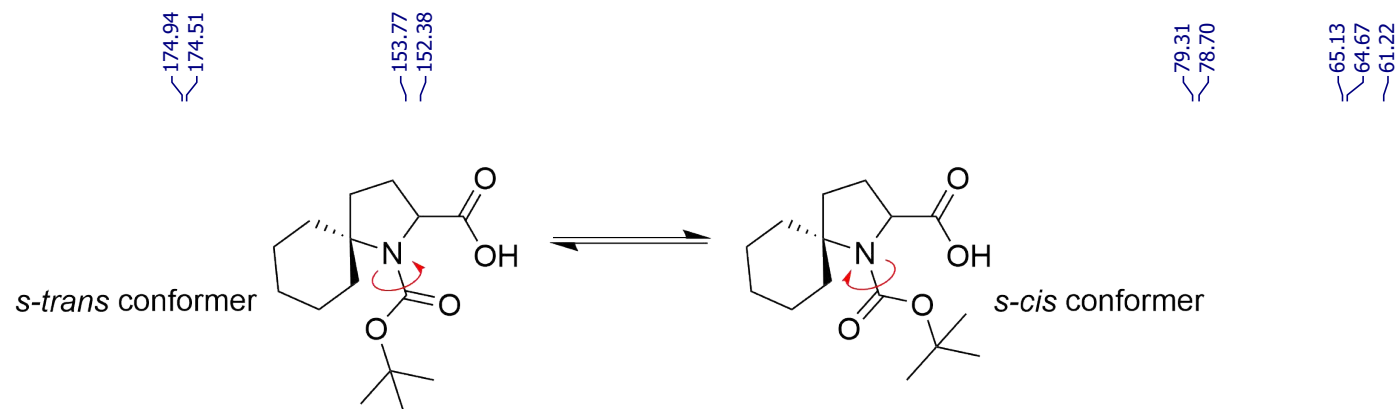
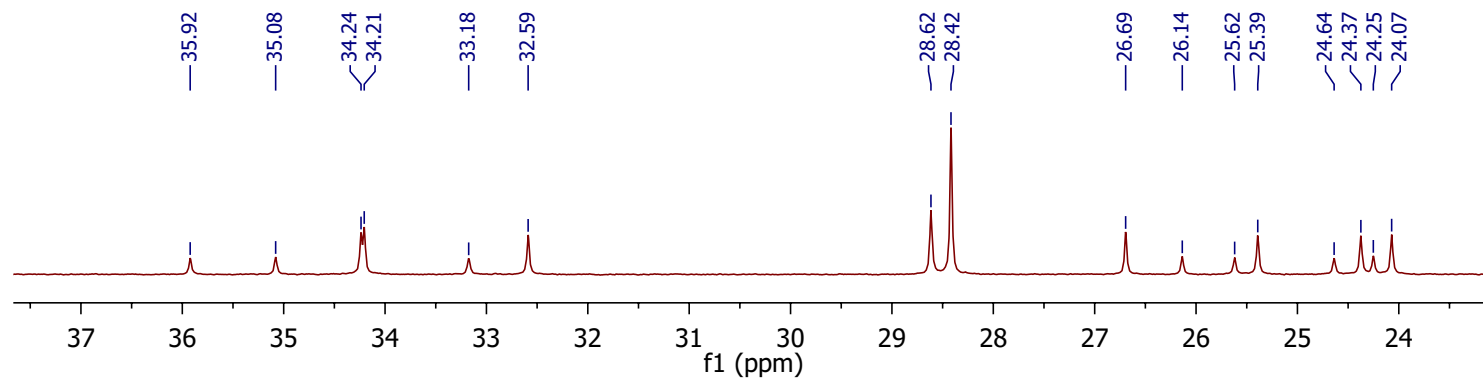
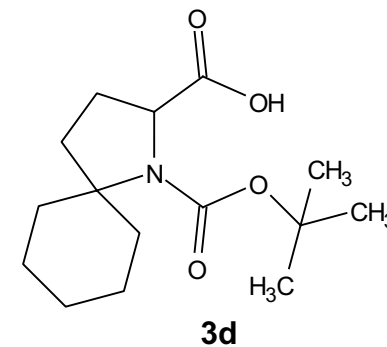
RT 1.171



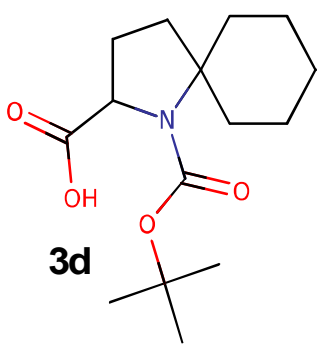
^1H NMR (500 MHz, Chloroform-*d*) δ 4.55 – 4.27 (m, 1H), 2.68 – 0.99 (m, 23H).



^{13}C NMR (151 MHz, $\text{dms-}d_6$) δ 174.94, 174.51, 153.77, 152.38, 79.31, 78.70, 65.13, 64.67, 61.22, 35.92, 35.08, 34.24, 34.21, 33.18, 32.59, 28.62, 28.42, 26.69, 26.14, 25.62, 25.39, 24.64, 24.37, 24.25, 24.07

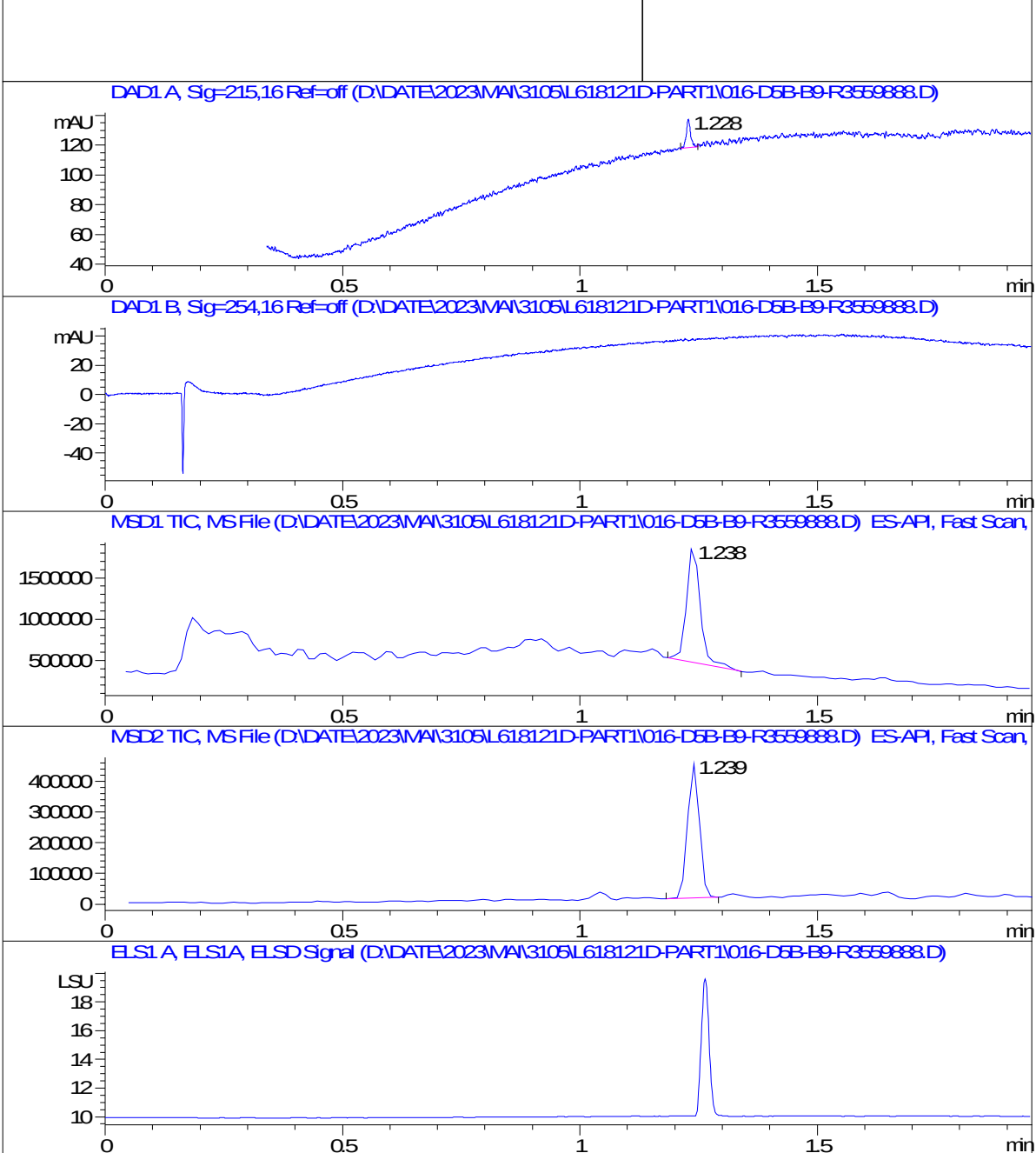


MaxPeak: 100.00%
Ret_Time: 1.228 min

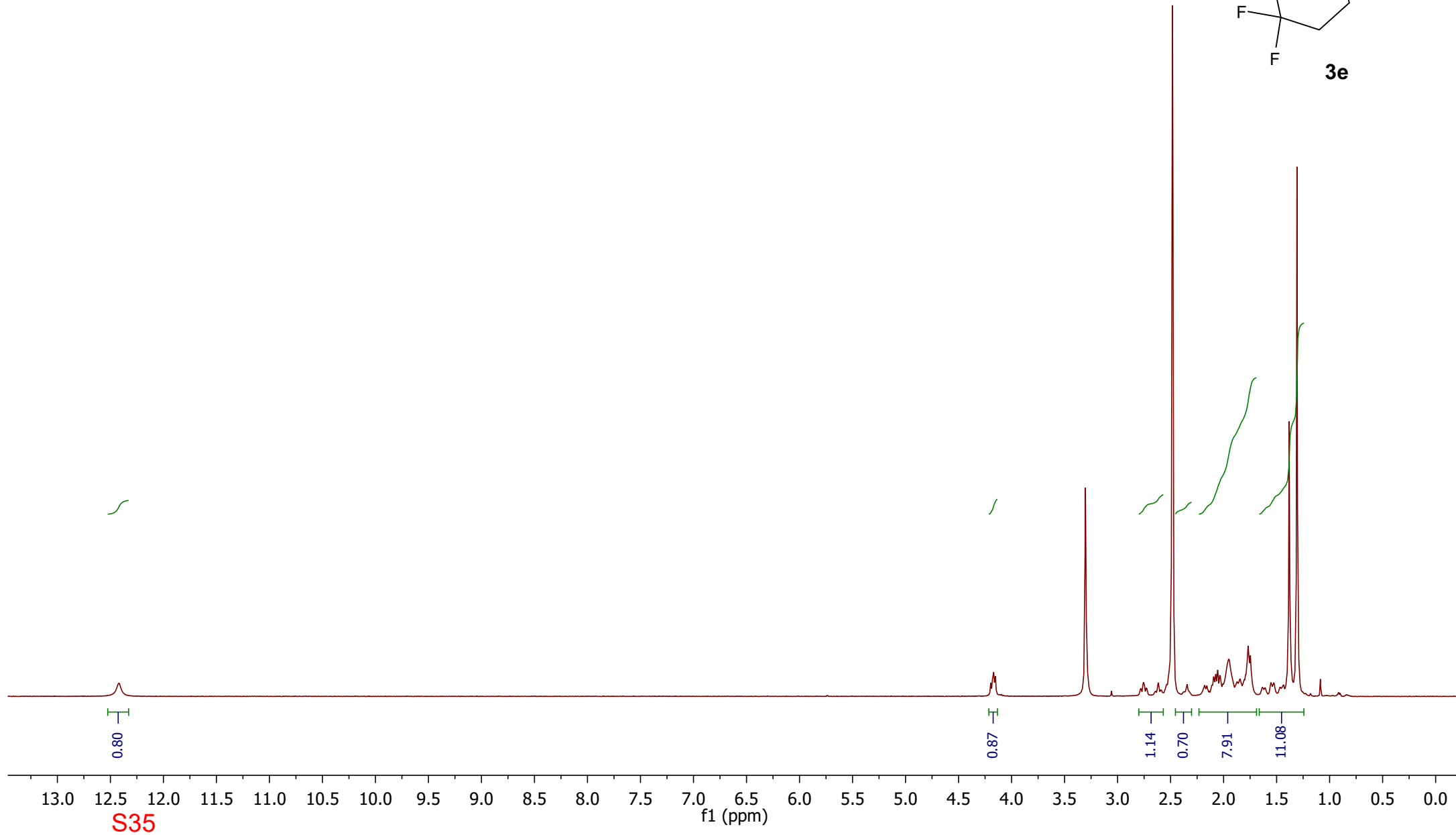
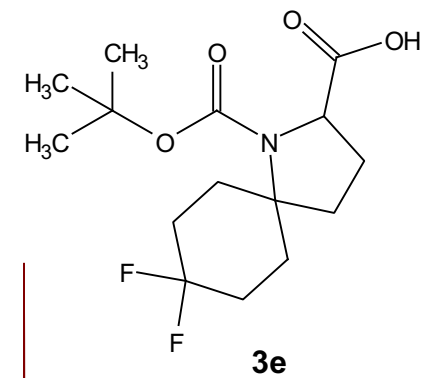


Mol Wt 283.36
Exact Mass 283.21

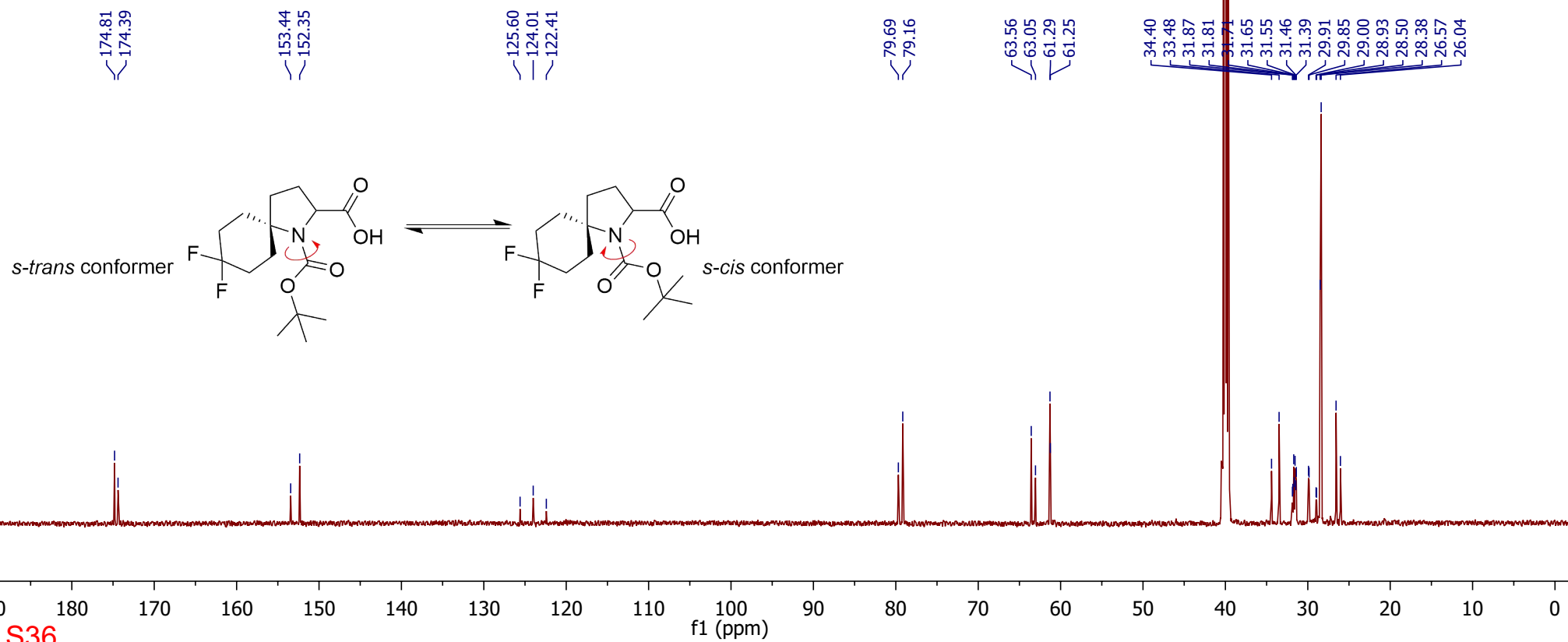
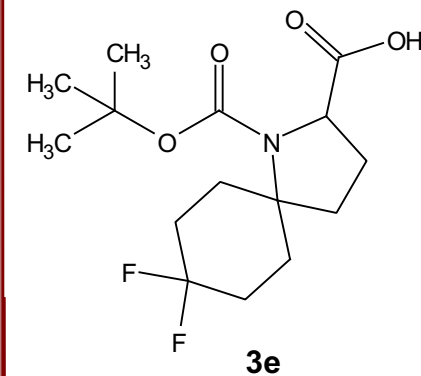
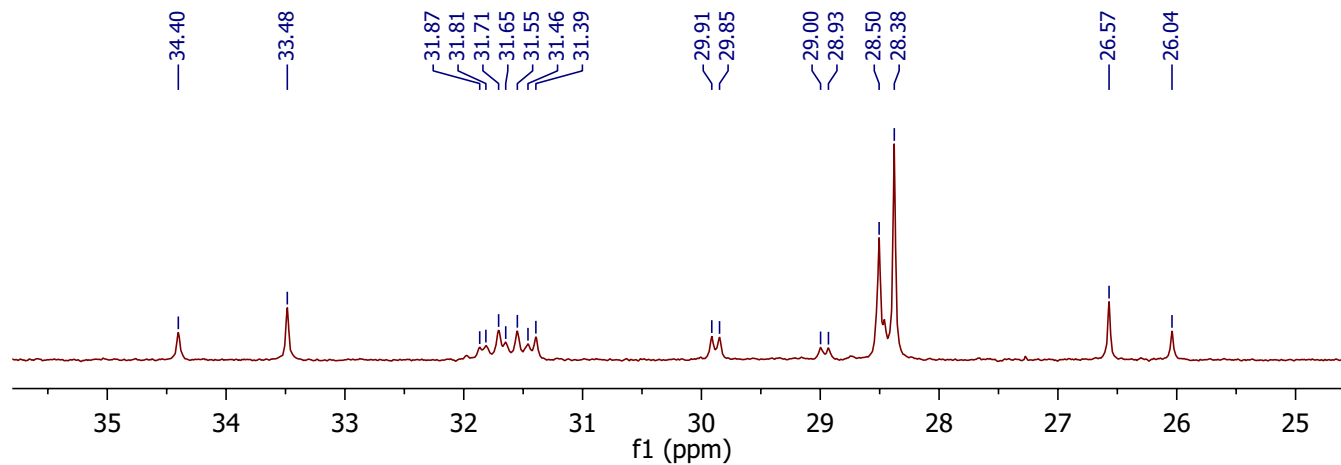
#	Time	Area%
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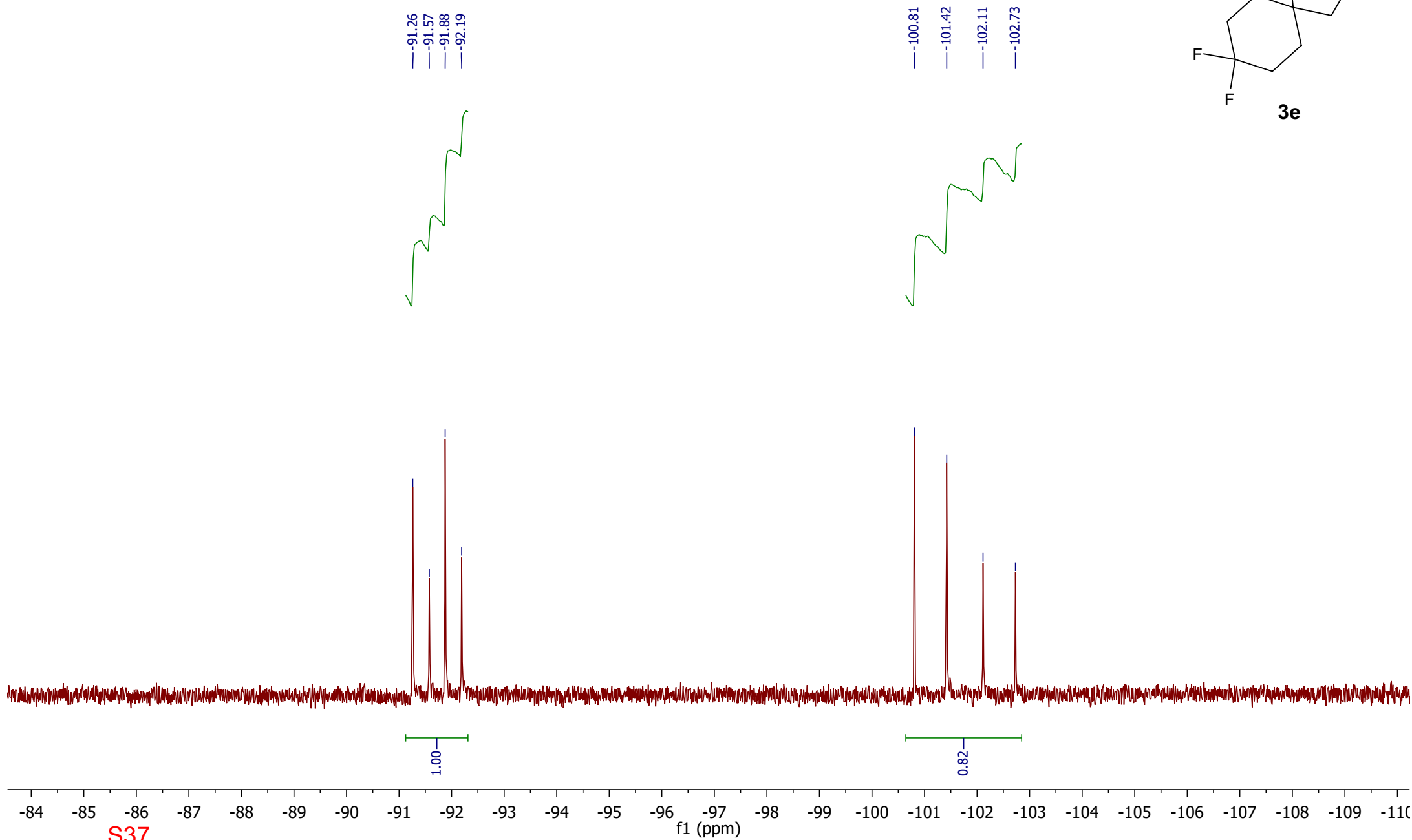
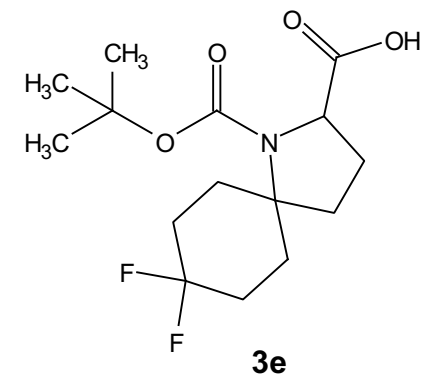
¹H NMR (500 MHz, DMSO-*d*₆) δ 12.42 (s, 1H), 4.21–4.13 (m, 1H), 2.80–2.57 (m, 1H), 2.45–2.30 (m, 1H), 2.23–1.69 (m, 8H), 1.66–1.24 (m, 11H).

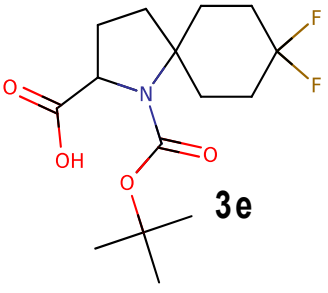


^{13}C NMR (151 MHz, dms o) δ 174.81, 174.39, 153.44, 152.35, 124.01 (t, $^1J_{\text{CF}} = 241.6$ Hz), 79.69, 79.16, 63.56, 63.05, 61.29, 61.25, 34.40, 33.48, 31.87, 31.81, 31.71, 31.65, 31.55, 31.46, 31.39, 29.91, 29.85, 29.00, 28.93, 28.50, 28.38, 26.57, 26.04

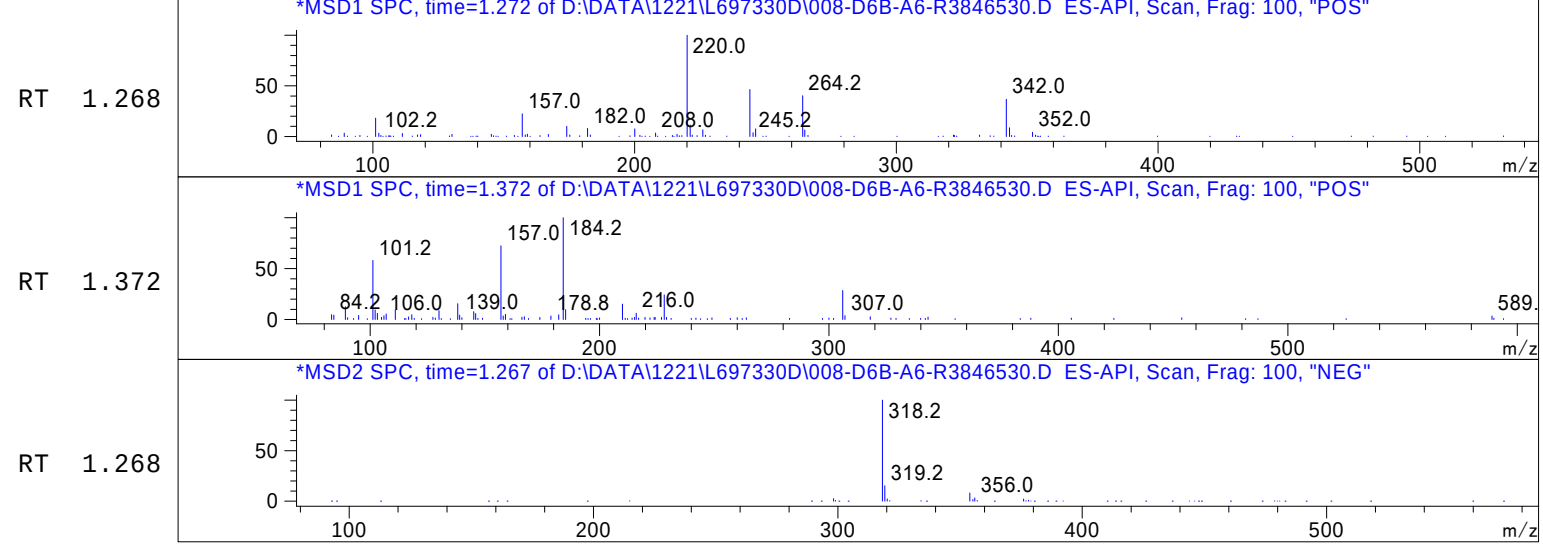
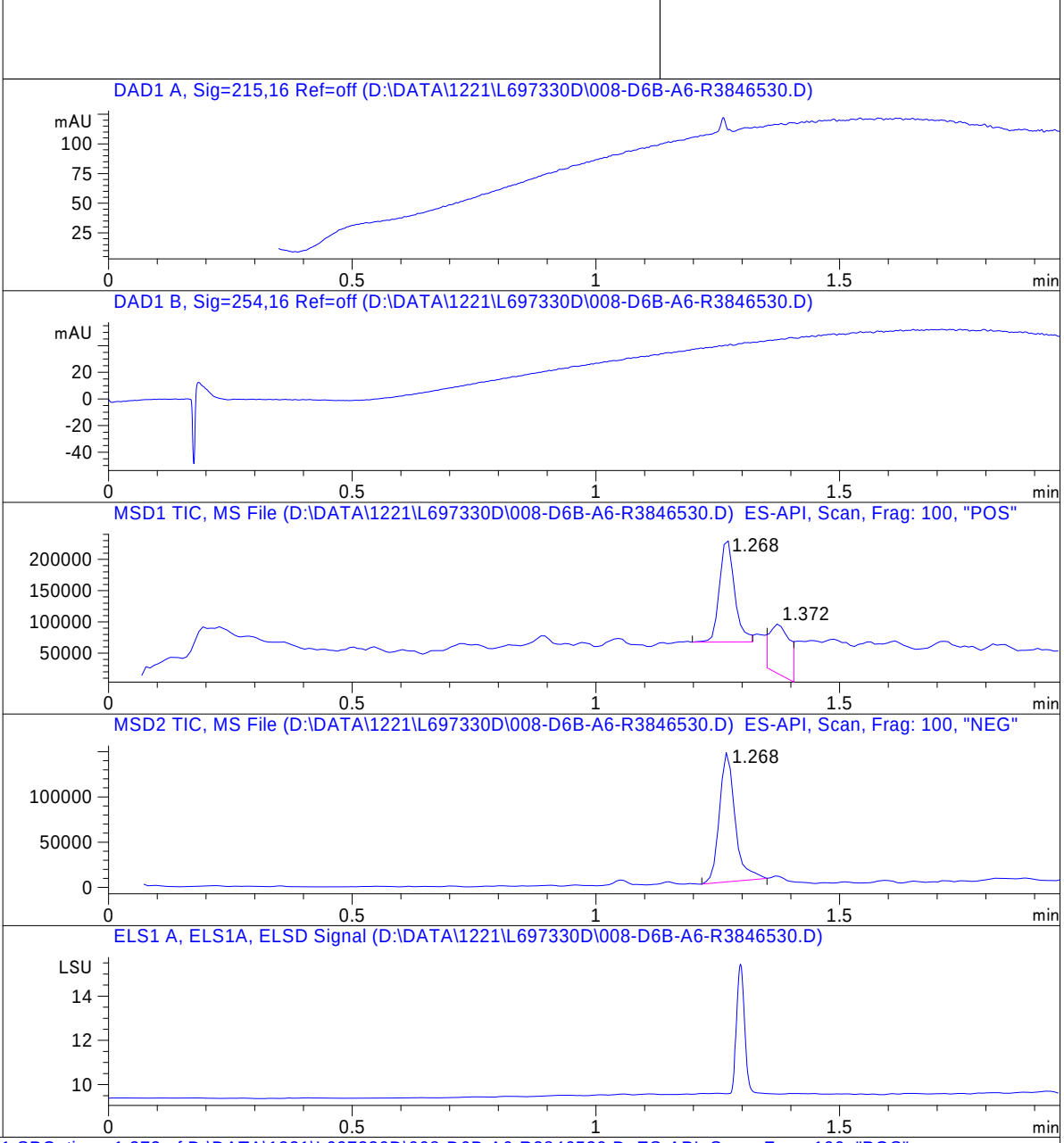


^{19}F NMR (376 MHz, $\text{DMSO-}d_6$) δ -91.57 (d, $J = 233$ Hz), -91.88 (d, $J = 233$ Hz), -101.11 (d, $J = 232$ Hz), -102.42 (d, $J = 232$ Hz)

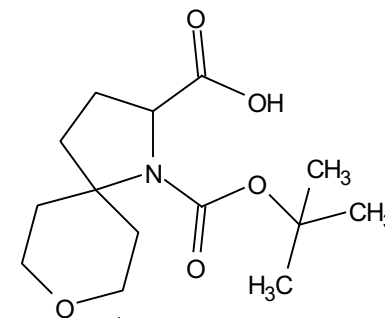




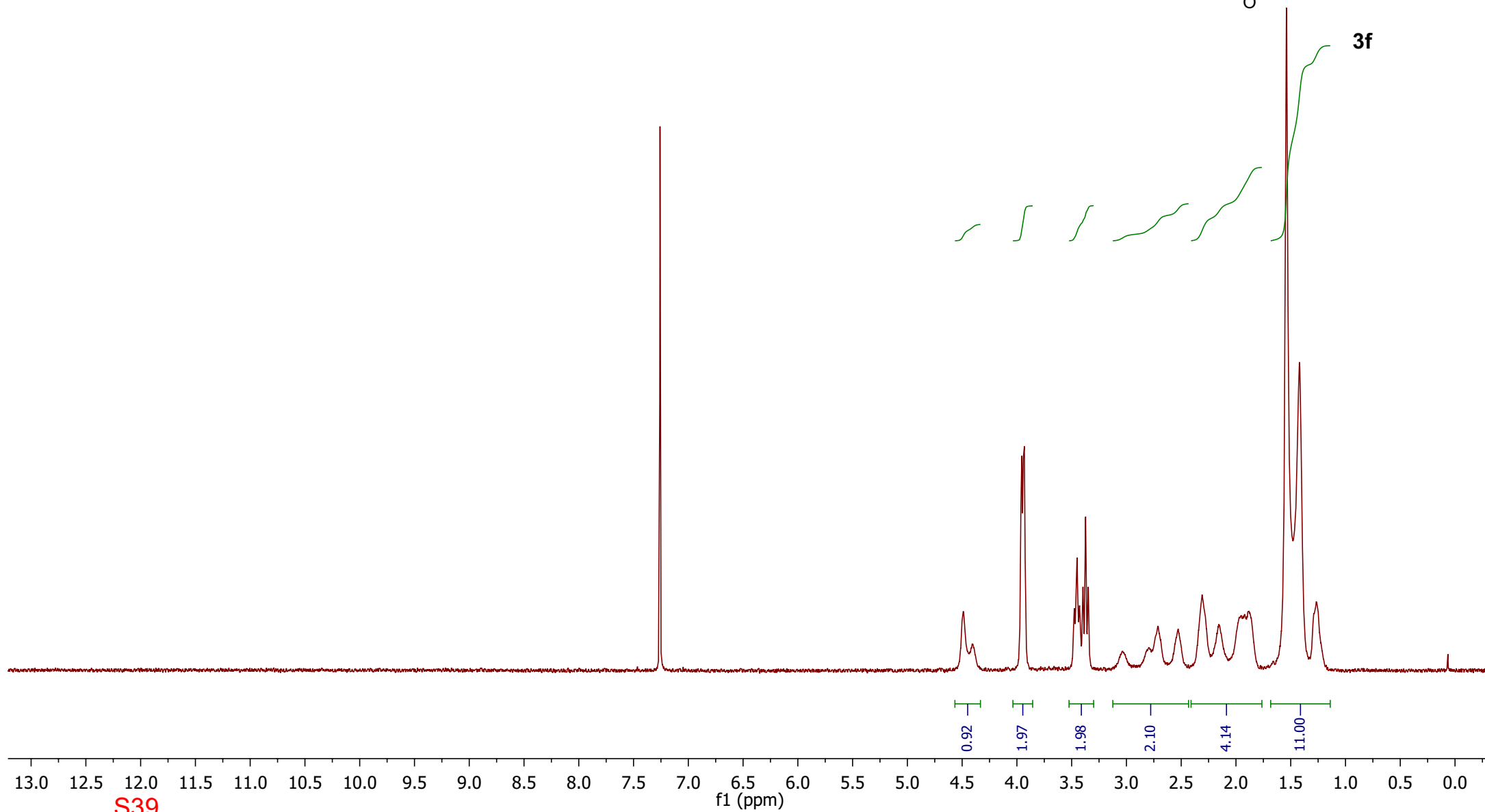
Mol Wt 319.34
Exact Mass 319.19



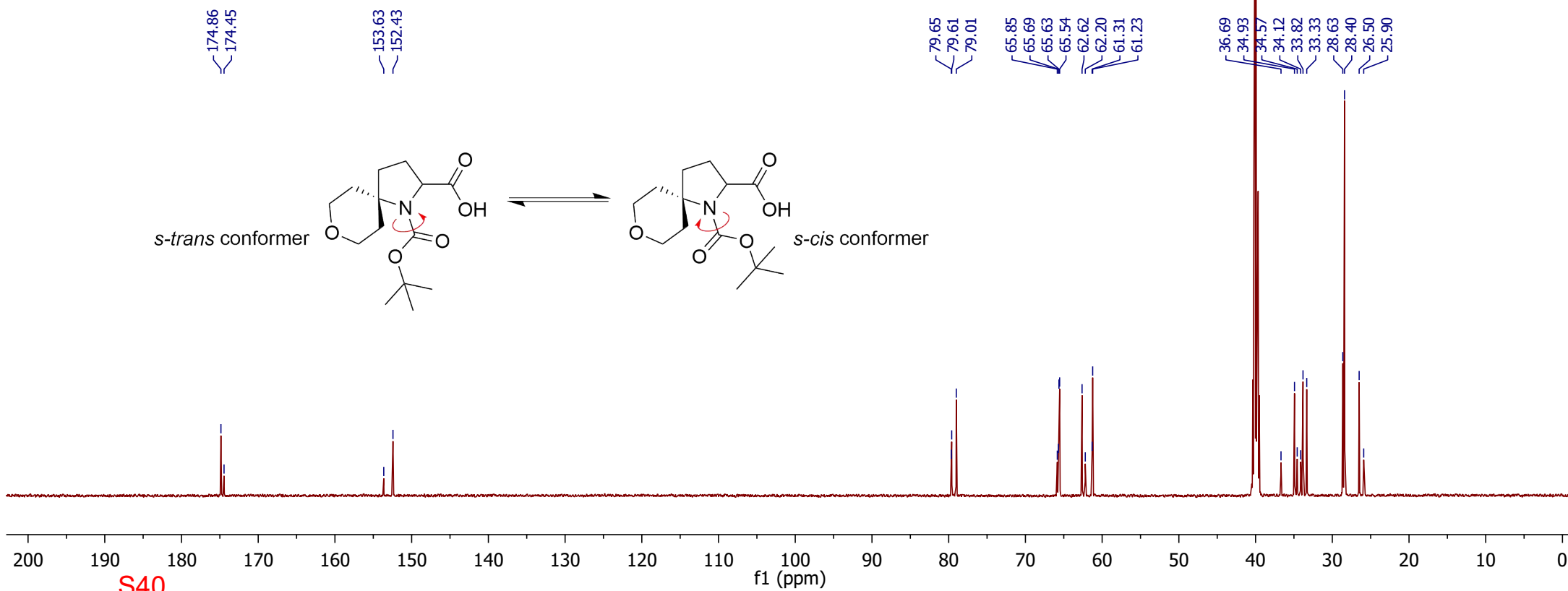
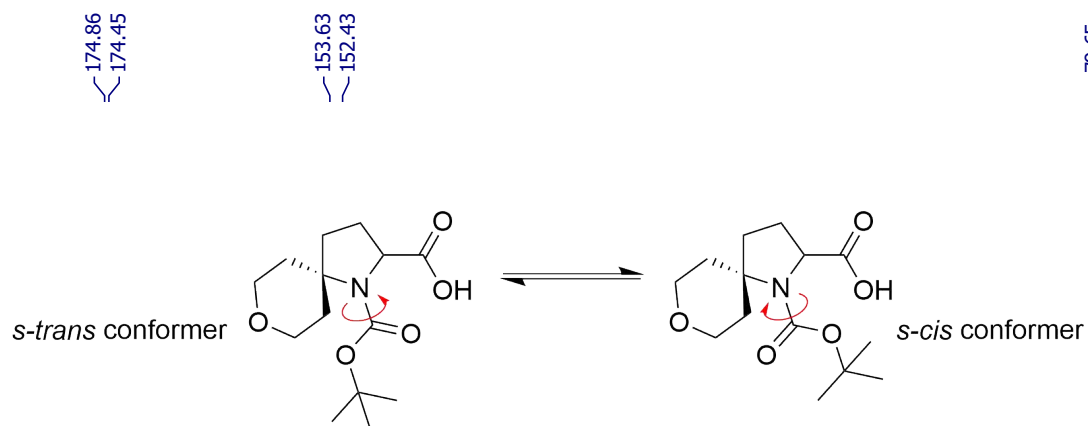
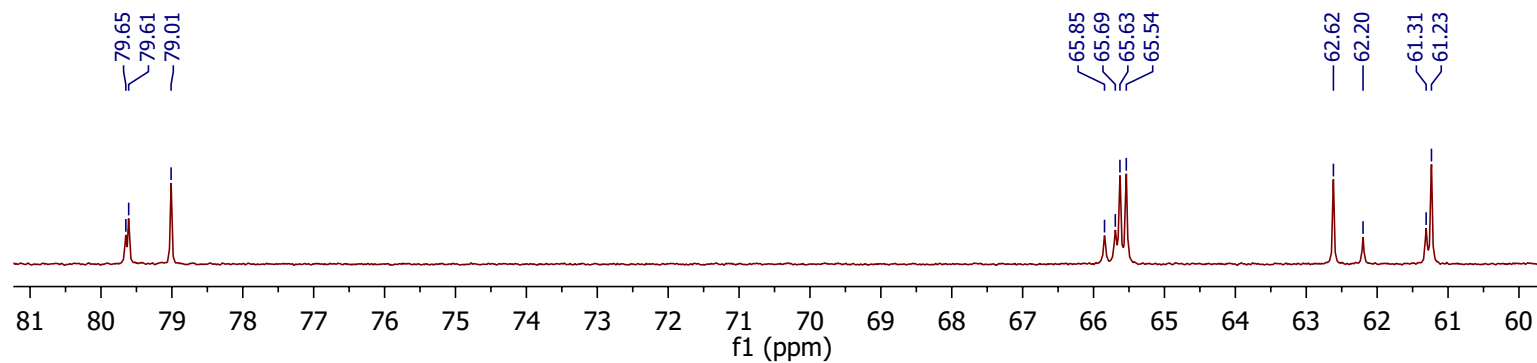
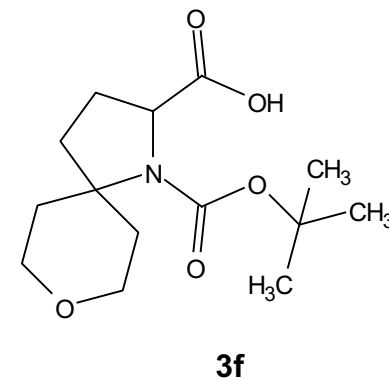
^1H NMR (500 MHz, Chloroform-*d*) δ 4.57 – 4.33 (m, 1H), 3.95 (dd, J = 11.9, 5.2 Hz, 2H), 3.41 (dt, J = 39.5, 12.3 Hz, 2H), 3.12 – 2.43 (m, 2H), 2.41 – 1.76 (m, 4H), 1.68 – 1.14 (m, 11H).



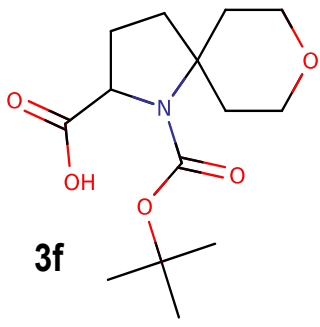
3f



^{13}C NMR (151 MHz, dms o) δ 174.86, 174.45, 153.63, 152.43, 79.65, 79.61, 79.01, 65.85, 65.69, 65.63, 65.54, 62.62, 62.20, 61.31, 61.23, 36.69, 34.93, 34.57, 34.12, 33.82, 33.33, 28.63, 28.40, 26.50, 25.90

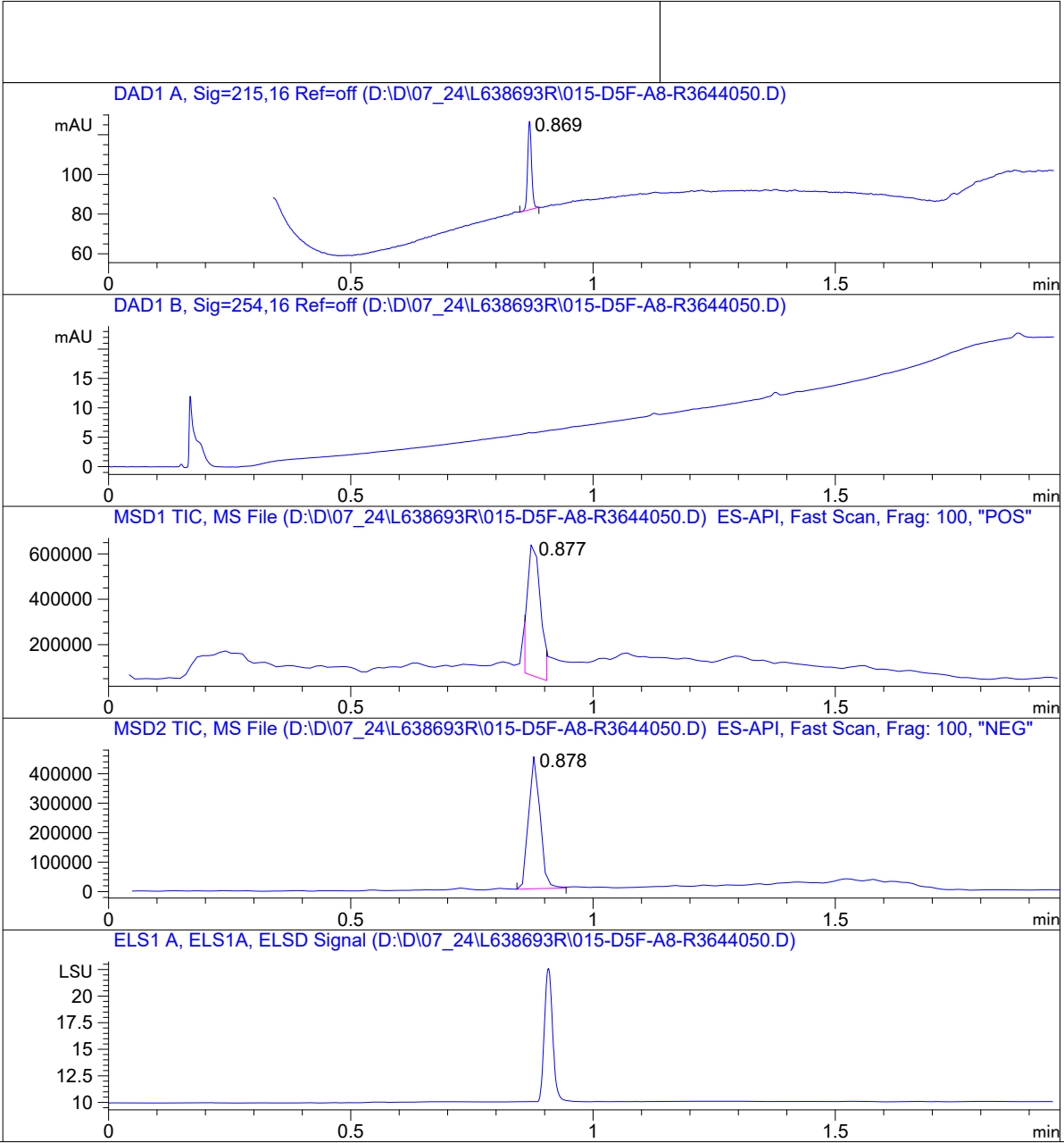


MaxPeak: 100.00%
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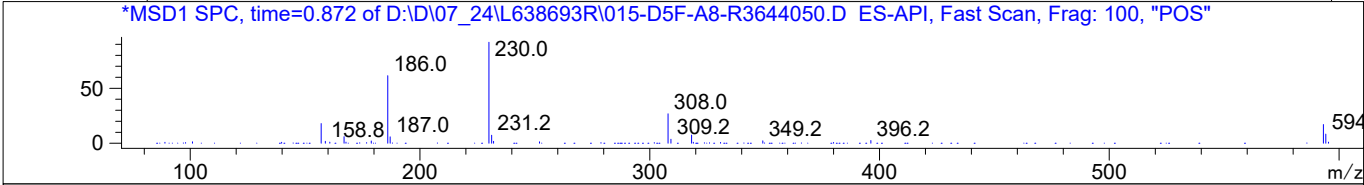


Mol Wt	285.34	
Exact Mass	285.18	
#	Time	Area%

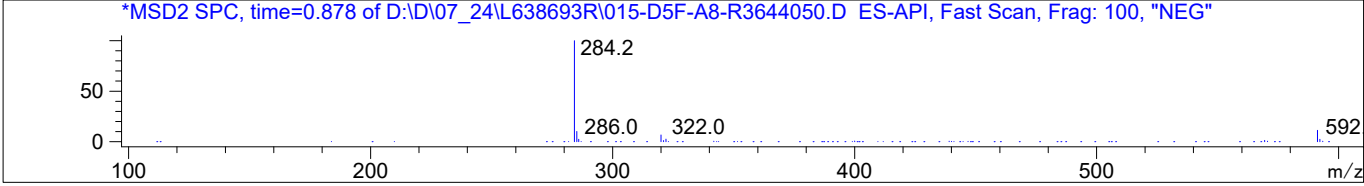
1	0.869	100.00



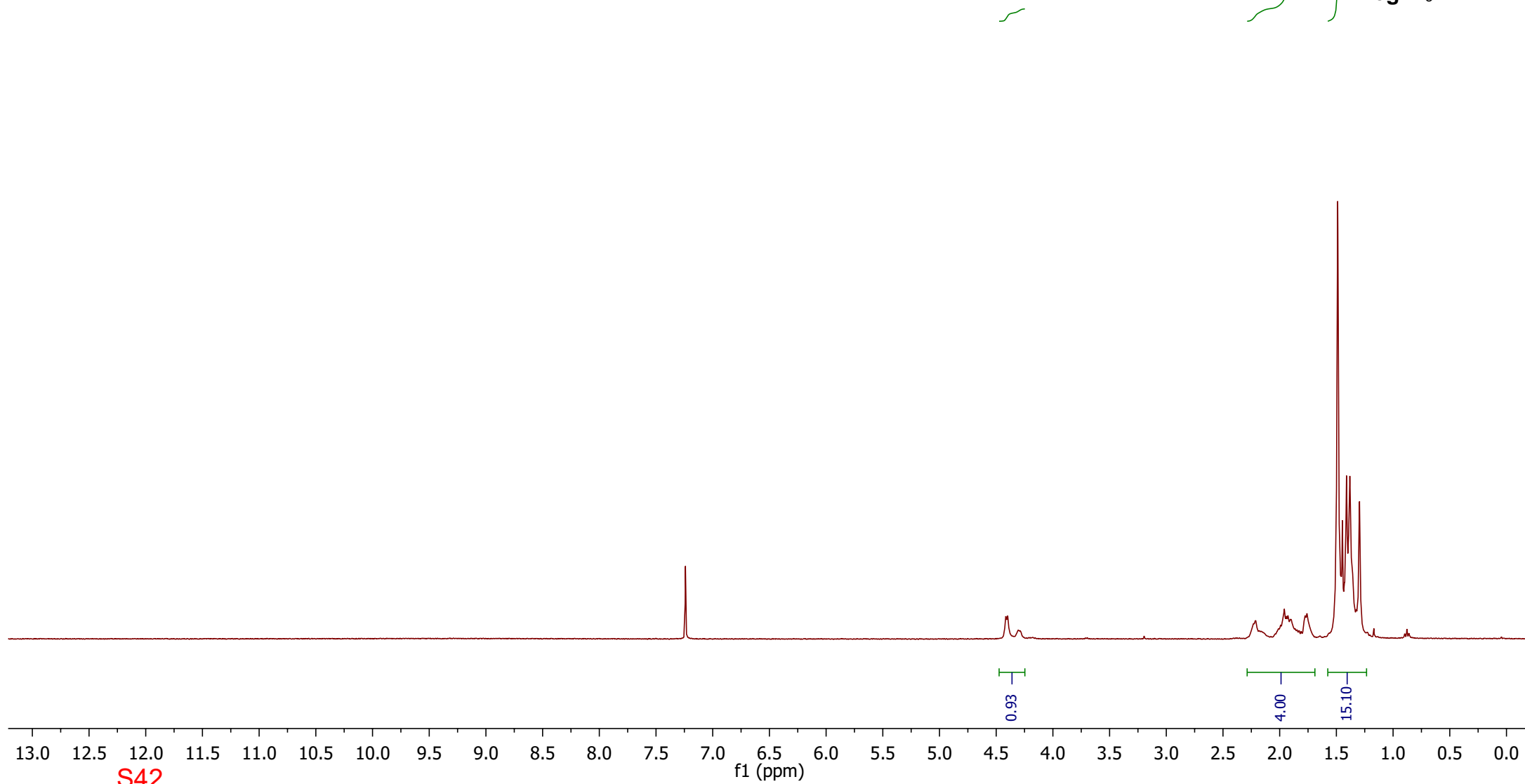
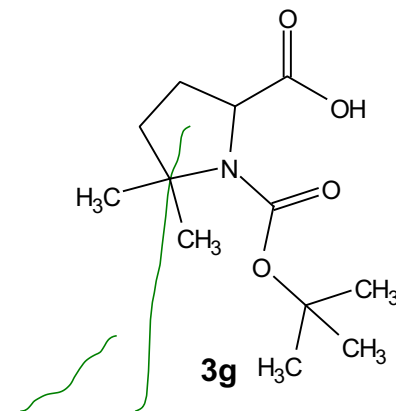
RT 0.877



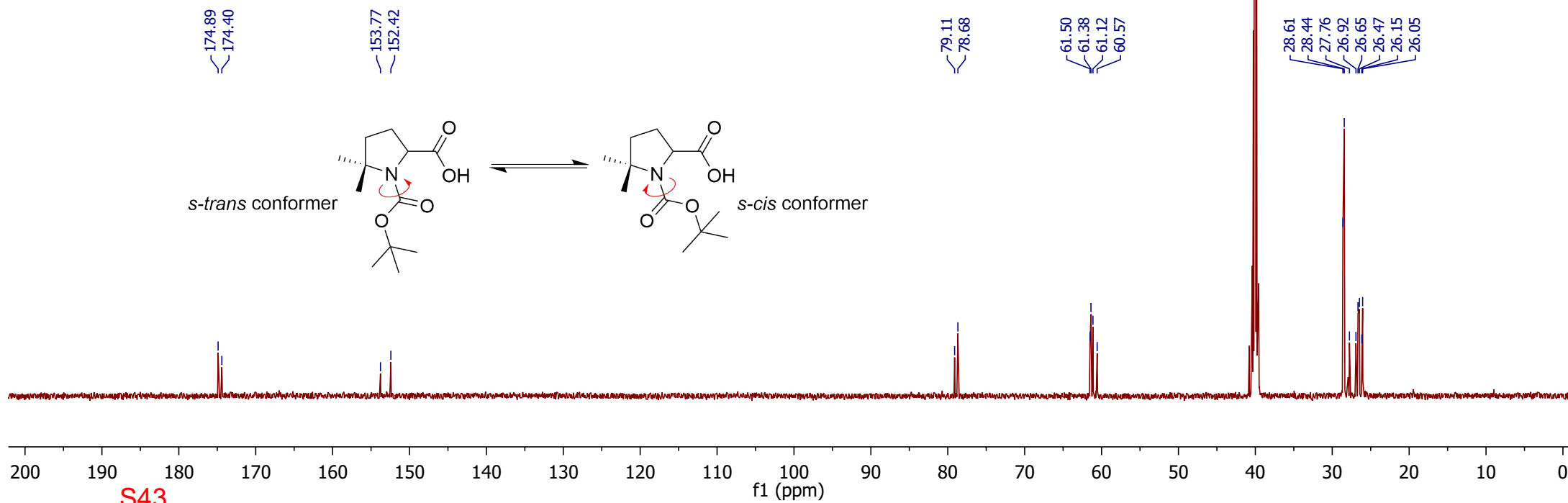
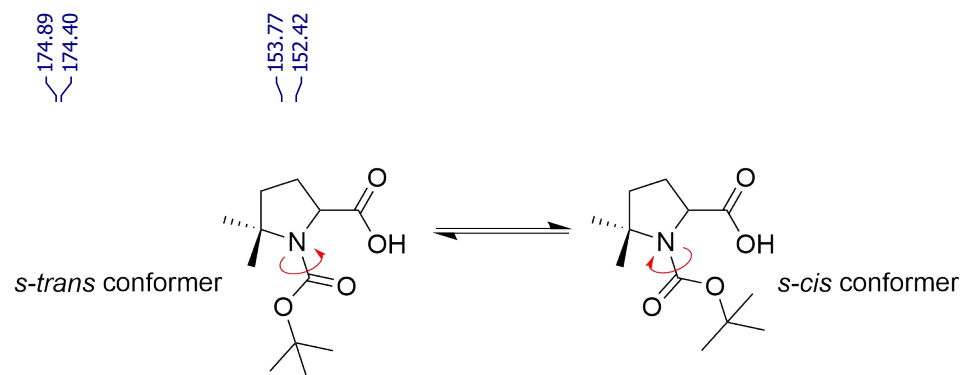
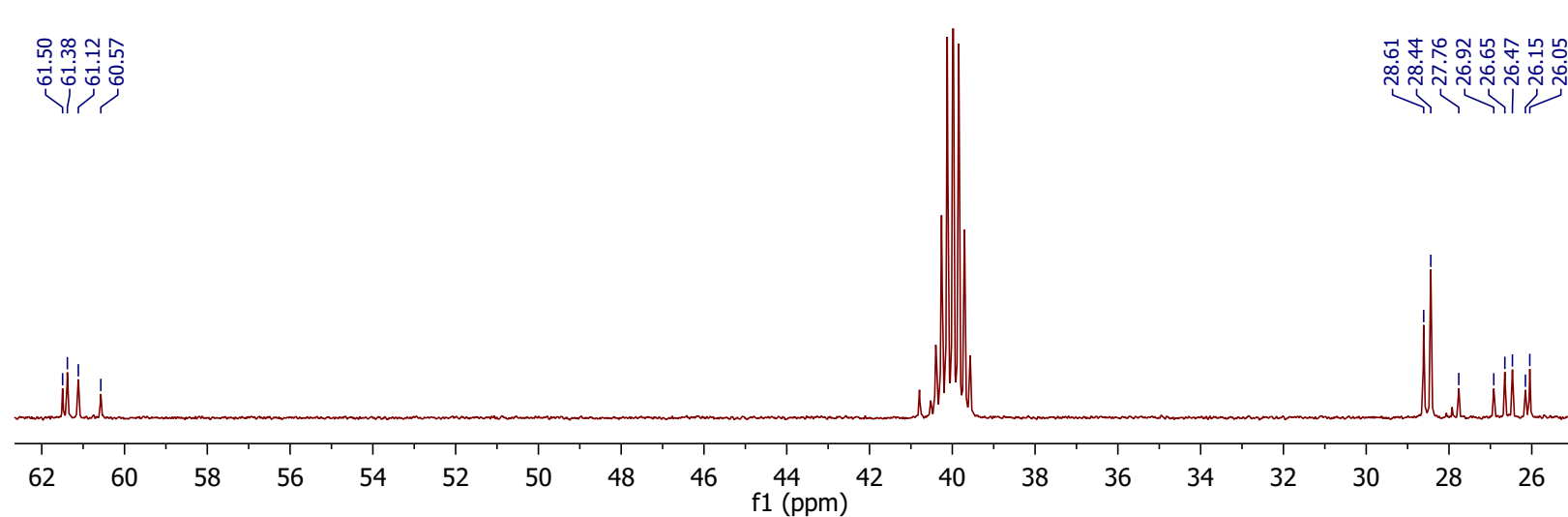
RT 0.878



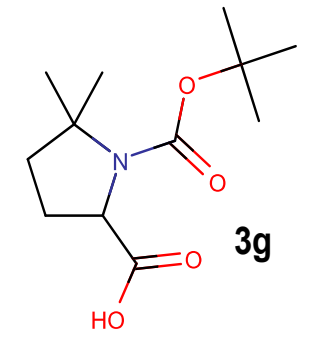
^1H NMR (400 MHz, Chloroform-*d*) δ 4.47 – 4.25 (m, 1H), 2.29 – 1.69 (m, 4H), 1.58 – 1.23 (m, 15H).



^{13}C NMR (151 MHz, dms o) δ 174.89, 174.40, 153.77, 152.42, 79.11, 78.68, 61.50, 61.38, 61.12, 60.57, 28.61, 28.44, 27.76, 26.92, 26.65, 26.47, 26.15, 26.05

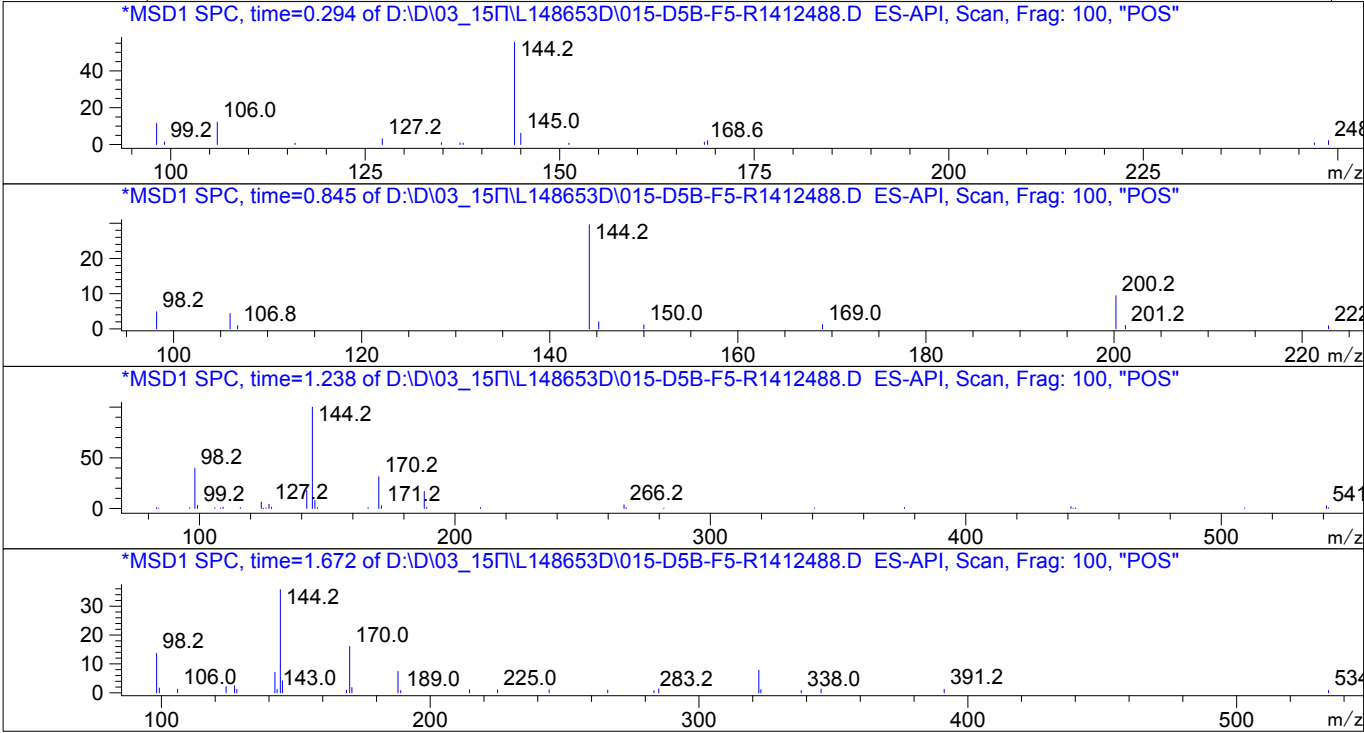
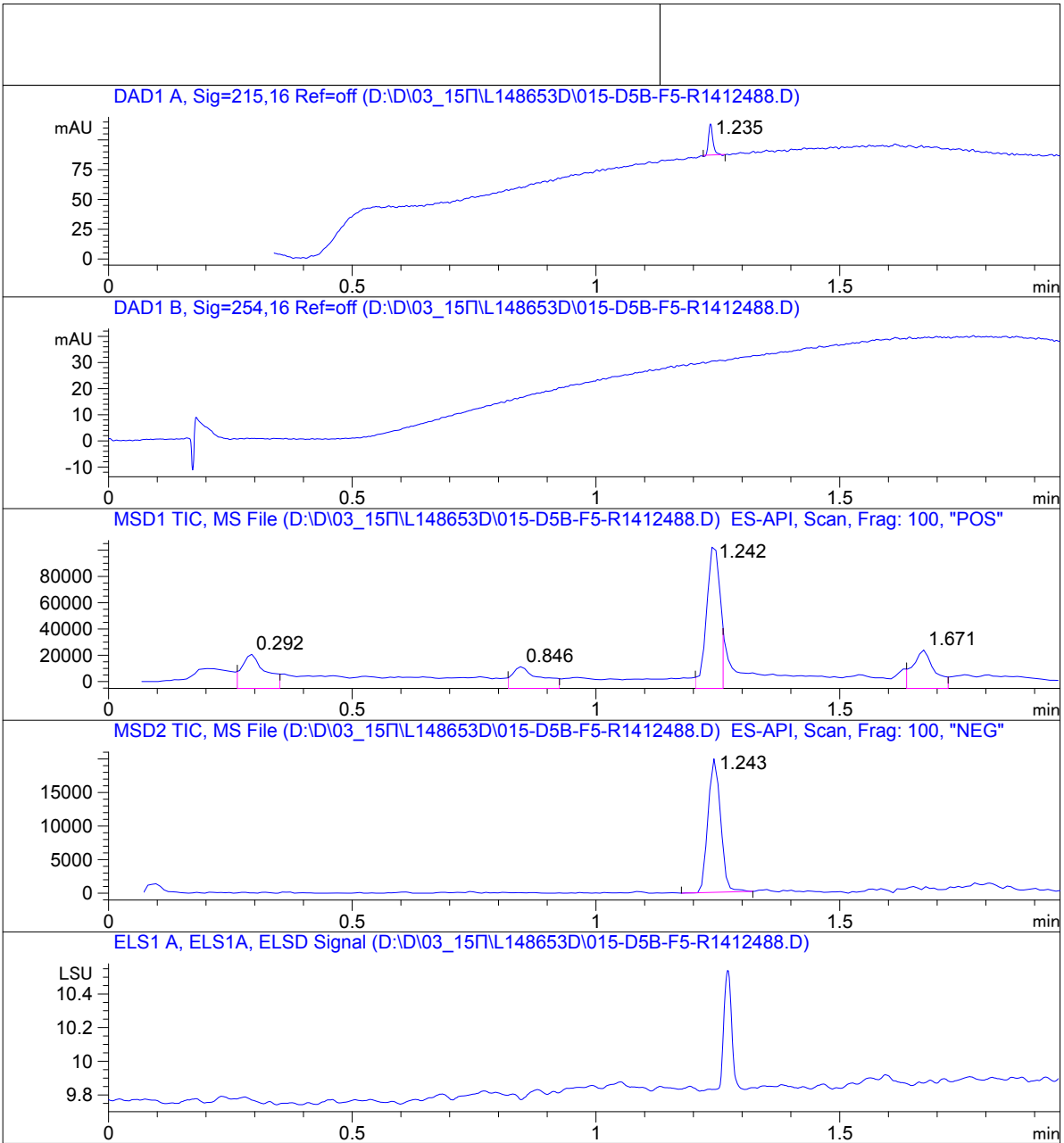


MaxPeak: 100.00%
Ret_Time: 1.235 min

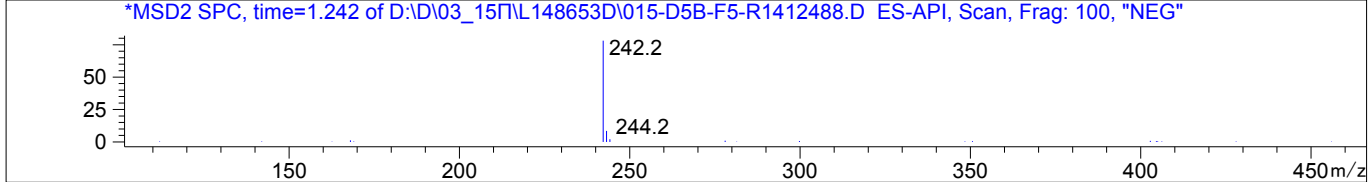


Mol Wt	243.3	
Exact Mass	243.17	
#	Time	Area%

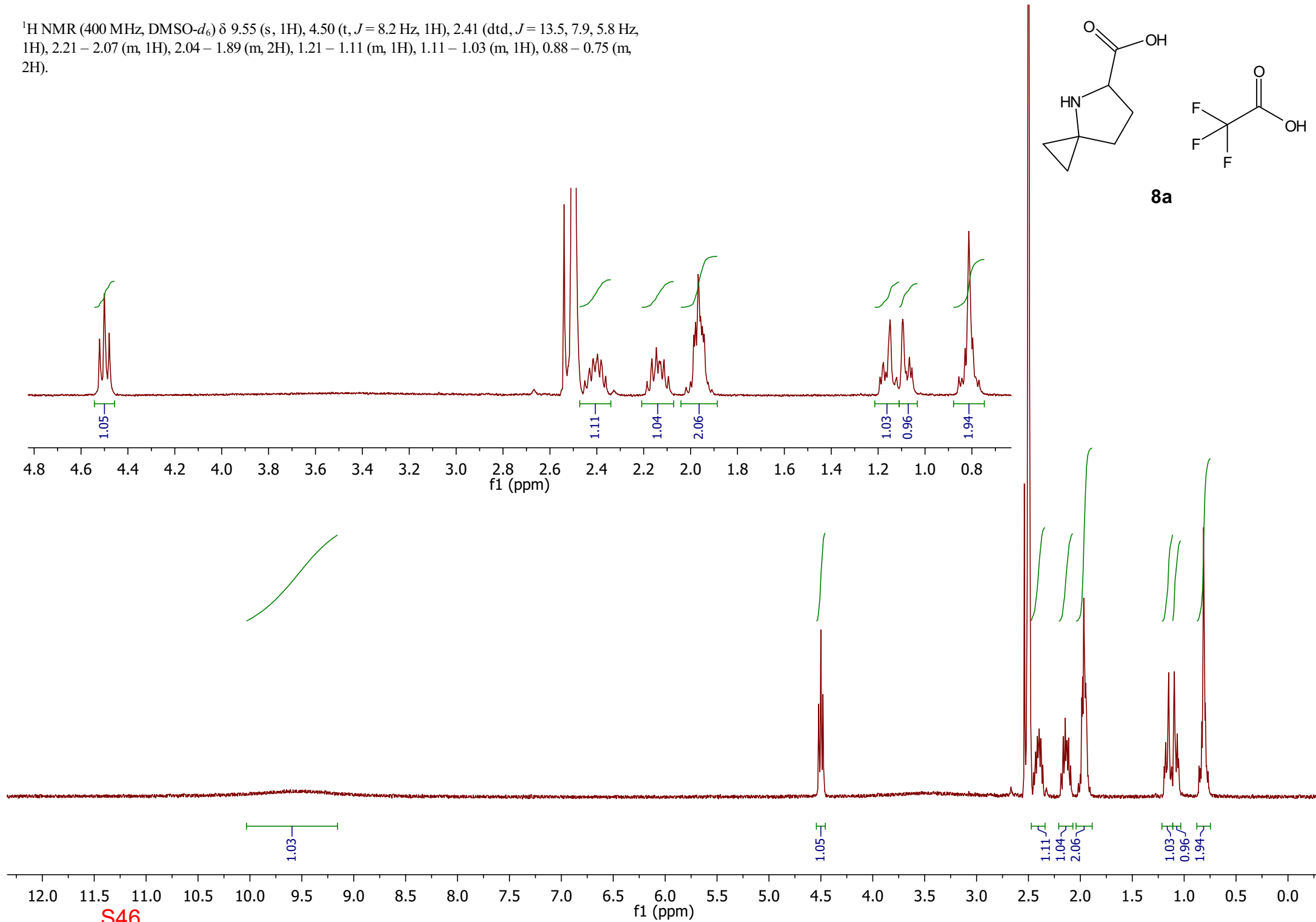
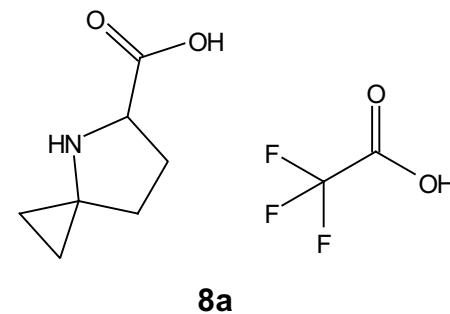
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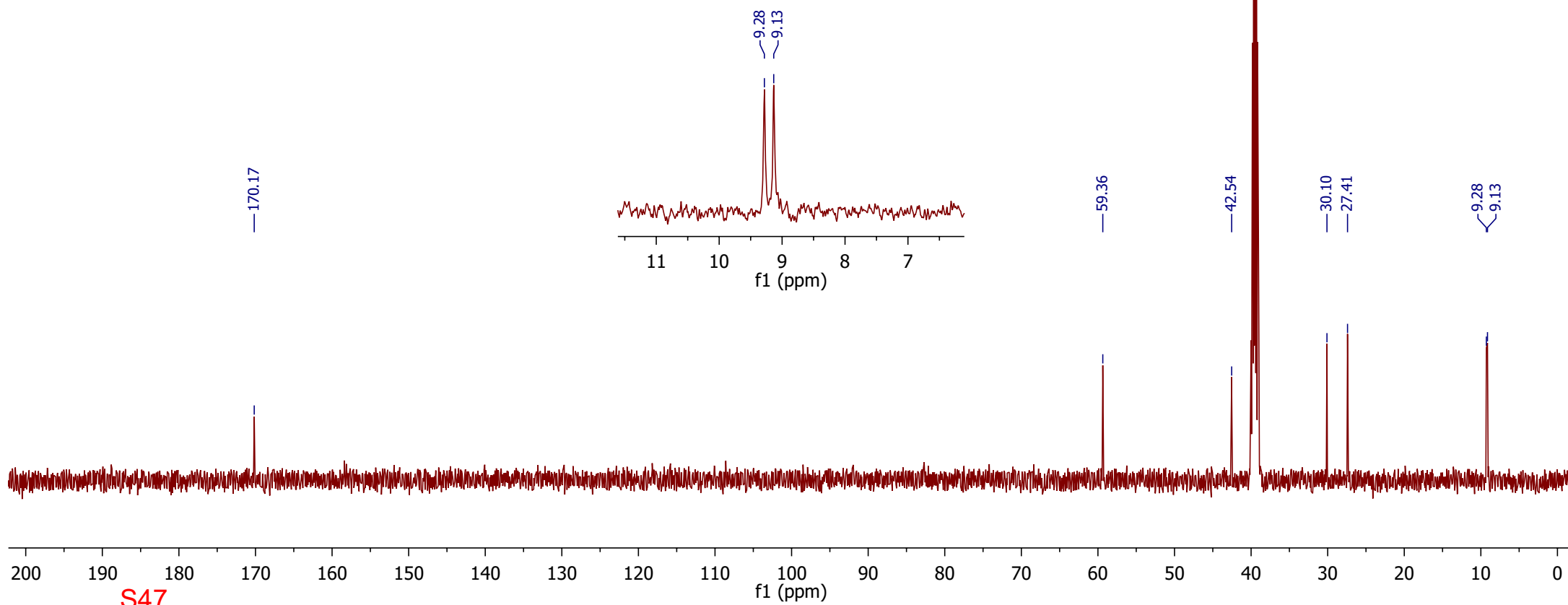
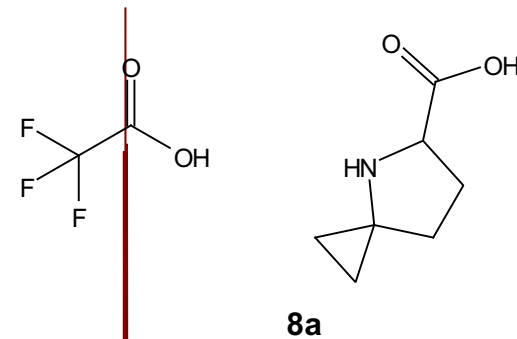
RT 1.243

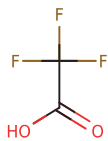
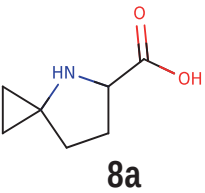


^1H NMR (400 MHz, $\text{DMSO}-d_6$) δ 9.55 (s, 1H), 4.50 (t, $J = 8.2$ Hz, 1H), 2.41 (dtd, $J = 13.5, 7.9, 5.8$ Hz, 1H), 2.21 – 2.07 (m, 1H), 2.04 – 1.89 (m, 2H), 1.21 – 1.11 (m, 1H), 1.11 – 1.03 (m, 1H), 0.88 – 0.75 (m, 2H).

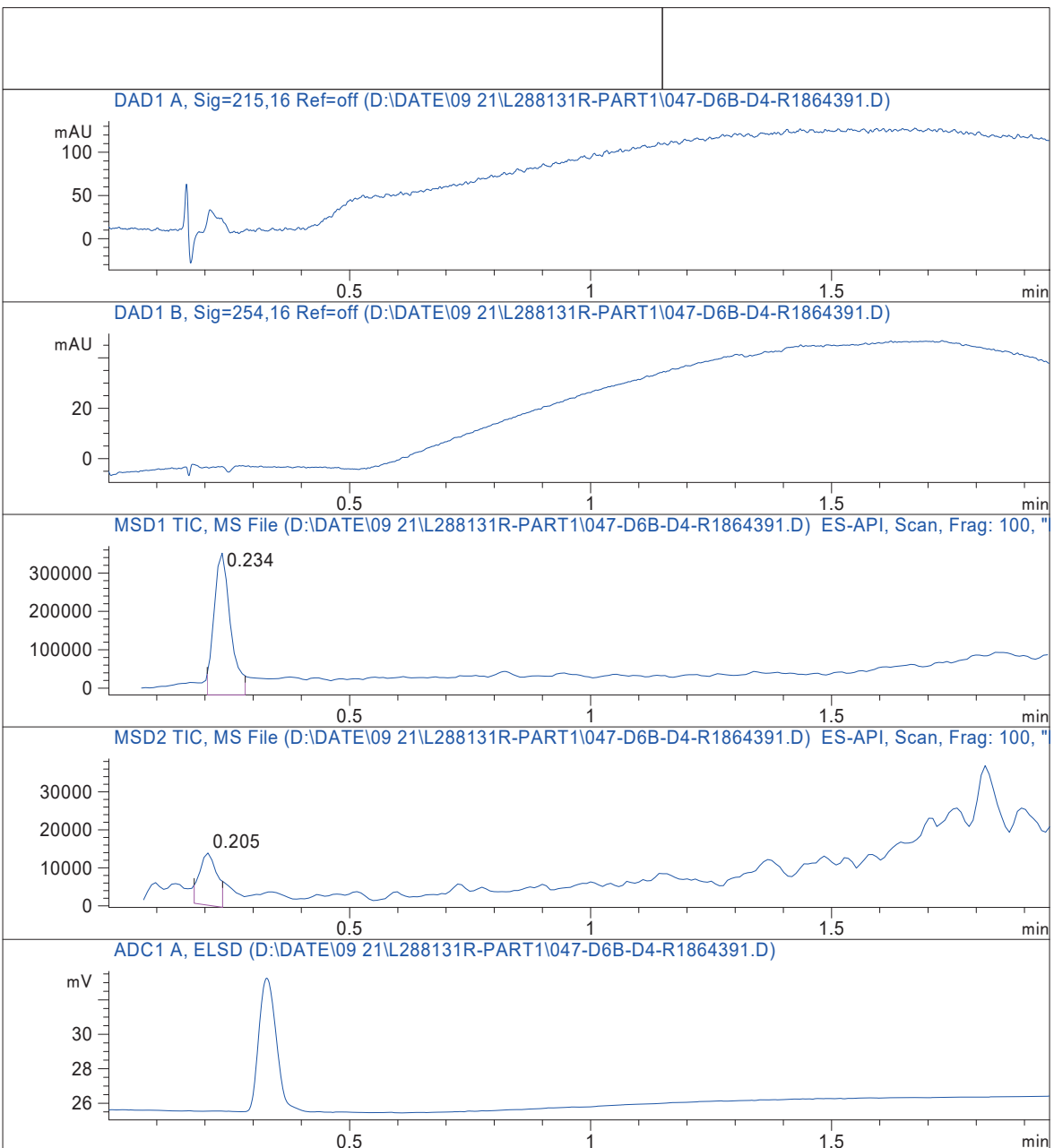


^{13}C NMR (DMSO, 126 MHz) δ 170.17, 59.36, 42.54, 30.10, 27.41, 9.28, 9.13.

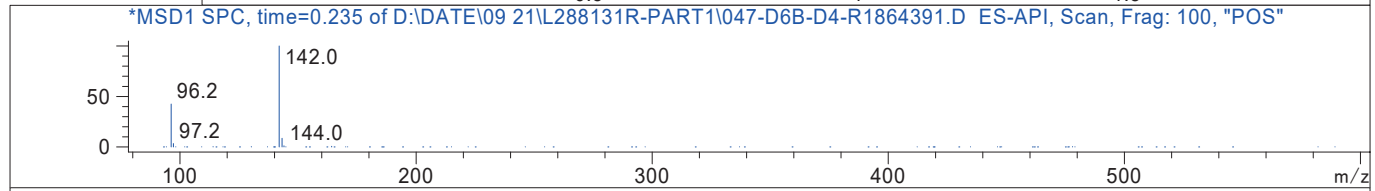




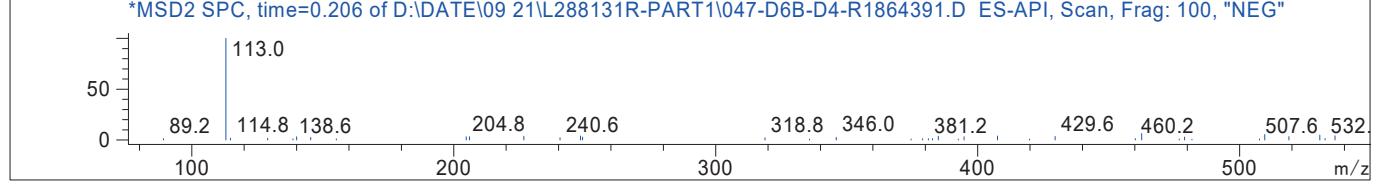
Mol Wt 255.19
Exact Mass 141.09



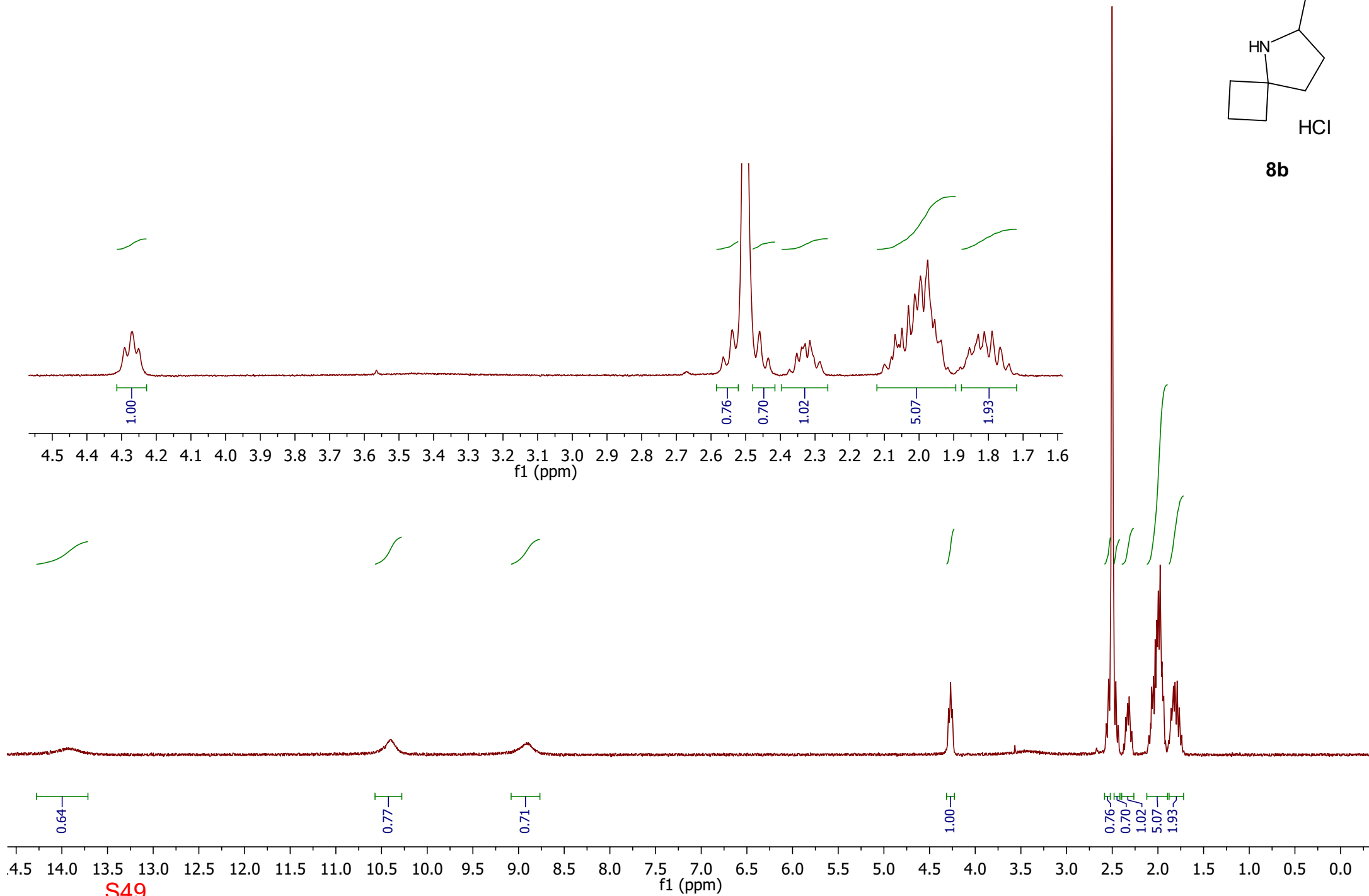
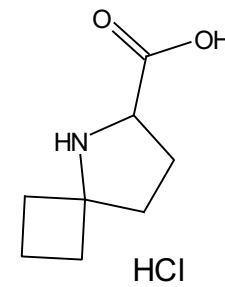
RT 0.234



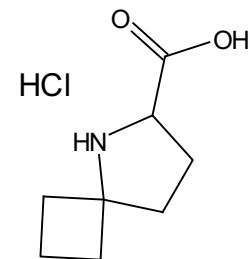
RT 0.205



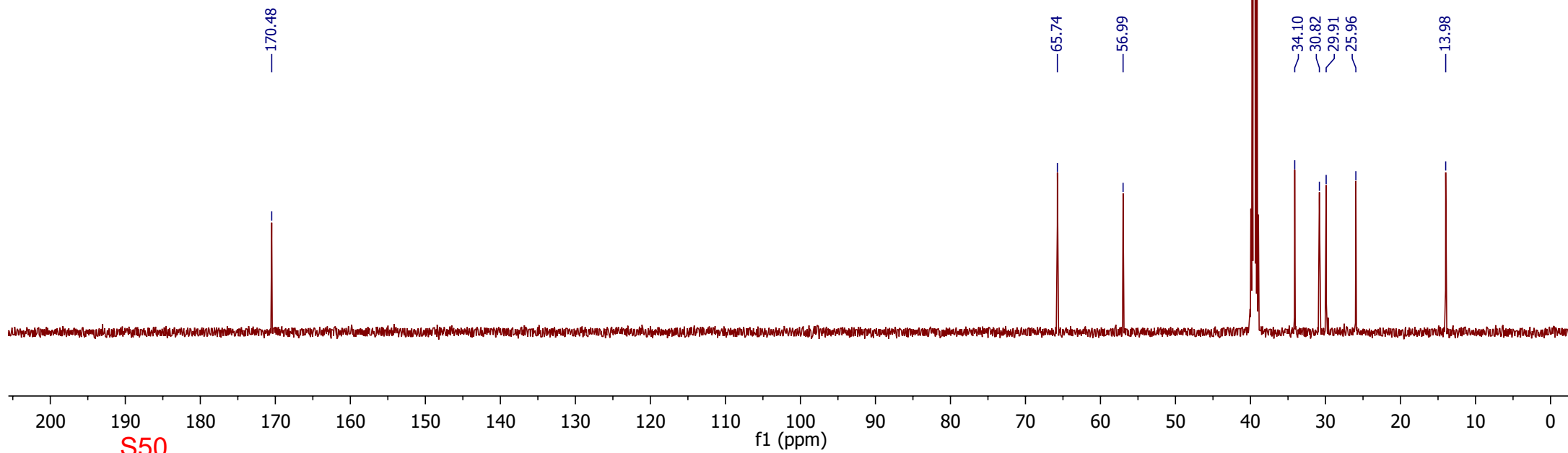
^1H NMR (400 MHz, $\text{DMSO}-d_6$) δ 13.92 (s, 1H), 10.40 (s, 1H), 8.91 (s, 1H), 4.27 (t, $J = 8.2$ Hz, 1H), 2.58 – 2.52 (m, 1H), 2.48 – 2.42 (m, 1H), 2.40 – 2.26 (m, 1H), 2.12 – 1.89 (m, 5H), 1.88 – 1.72 (m, 2H).



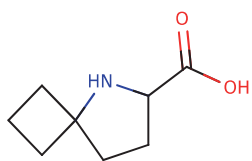
^{13}C NMR (DMSO, 126 MHz) δ 170.48, 65.74, 56.99, 34.10, 30.82, 29.91, 25.96, 13.98.



8b



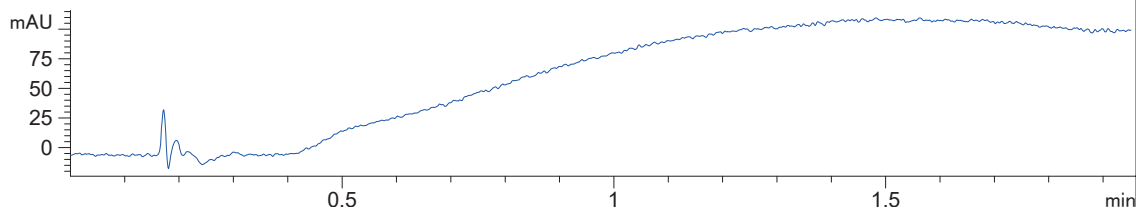
HCl



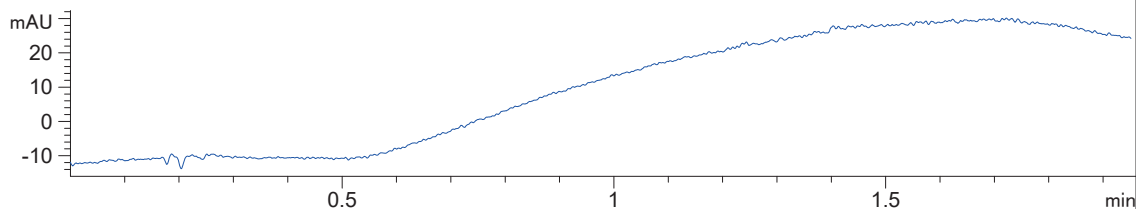
8b

Mol Wt 191.66
Exact Mass 155.11

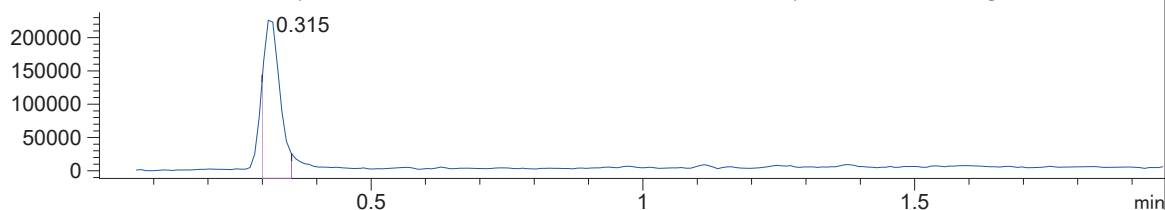
DAD1 A, Sig=215,16 Ref=off (D:\DATA\0221\L227867D\019-D7B-B3-R1675867.D)



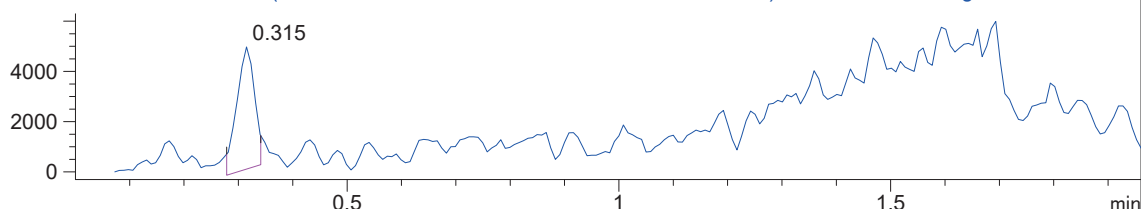
DAD1 B, Sig=254,16 Ref=off (D:\DATA\0221\L227867D\019-D7B-B3-R1675867.D)



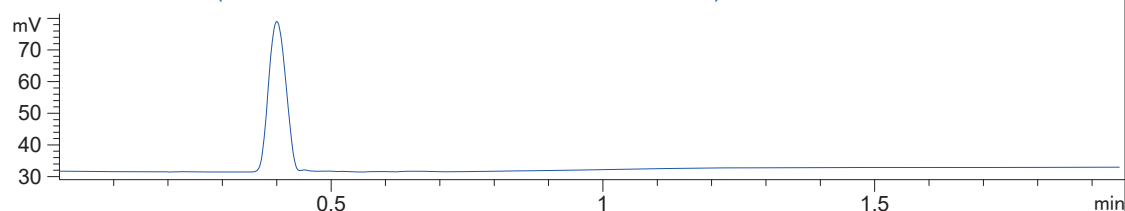
MSD1 TIC, MS File (D:\DATA\0221\L227867D\019-D7B-B3-R1675867.D) ES-API, Scan, Frag: 100, "POS"



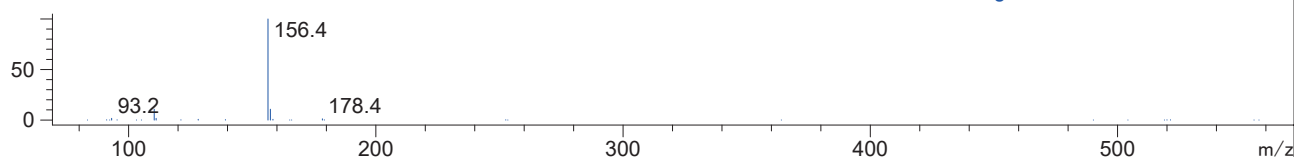
MSD2 TIC, MS File (D:\DATA\0221\L227867D\019-D7B-B3-R1675867.D) ES-API, Scan, Frag: 100, "NEG"



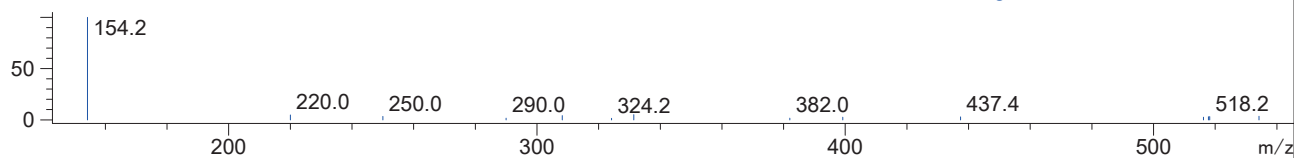
ADC1 A, ELSD (D:\DATA\0221\L227867D\019-D7B-B3-R1675867.D)



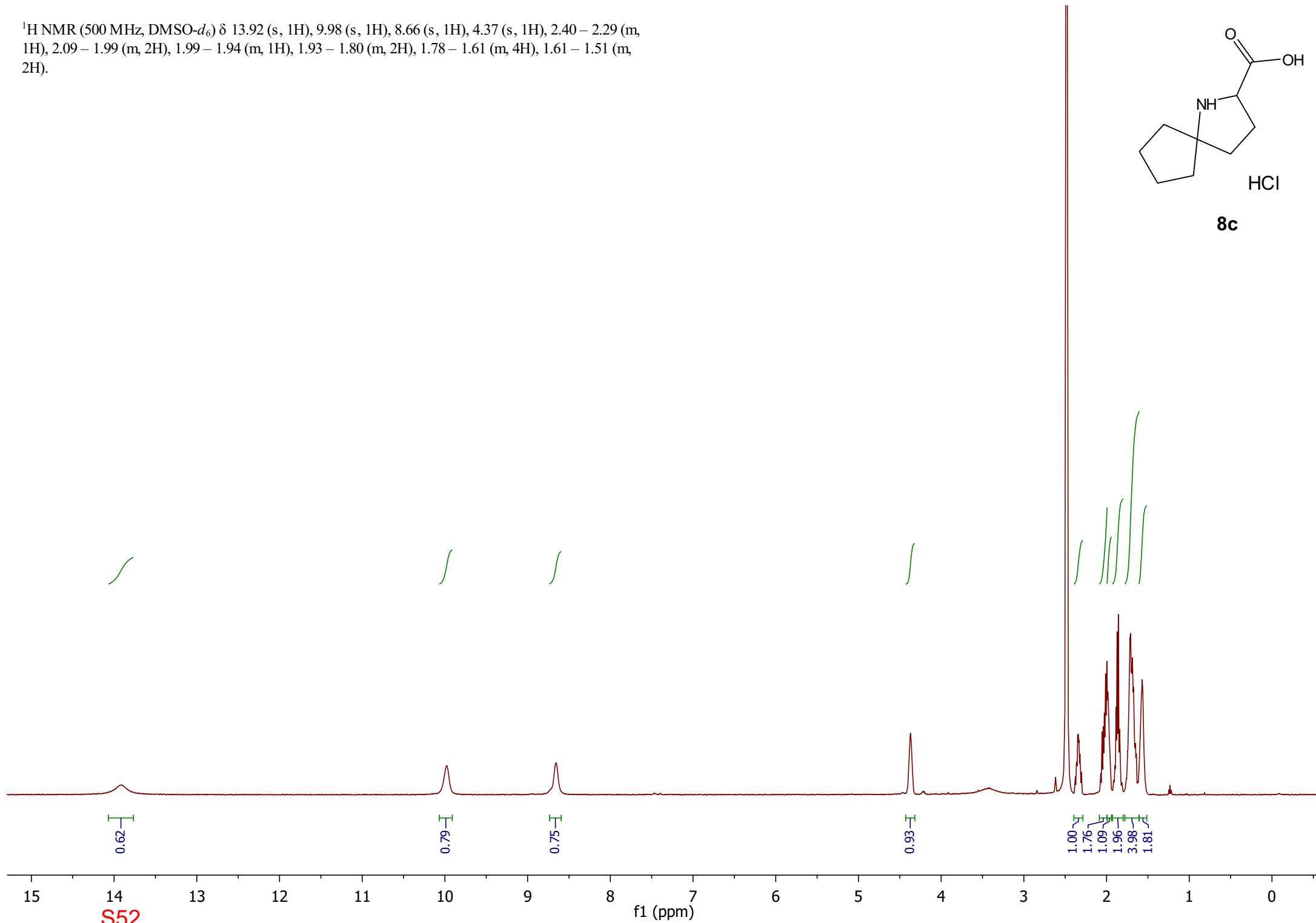
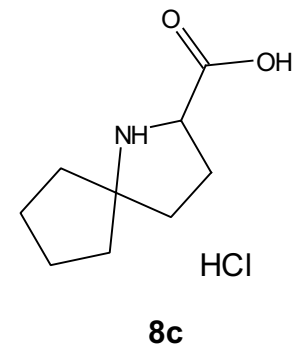
*MSD1 SPC, time=0.311 of D:\DATA\0221\L227867D\019-D7B-B3-R1675867.D ES-API, Scan, Frag: 100, "POS"



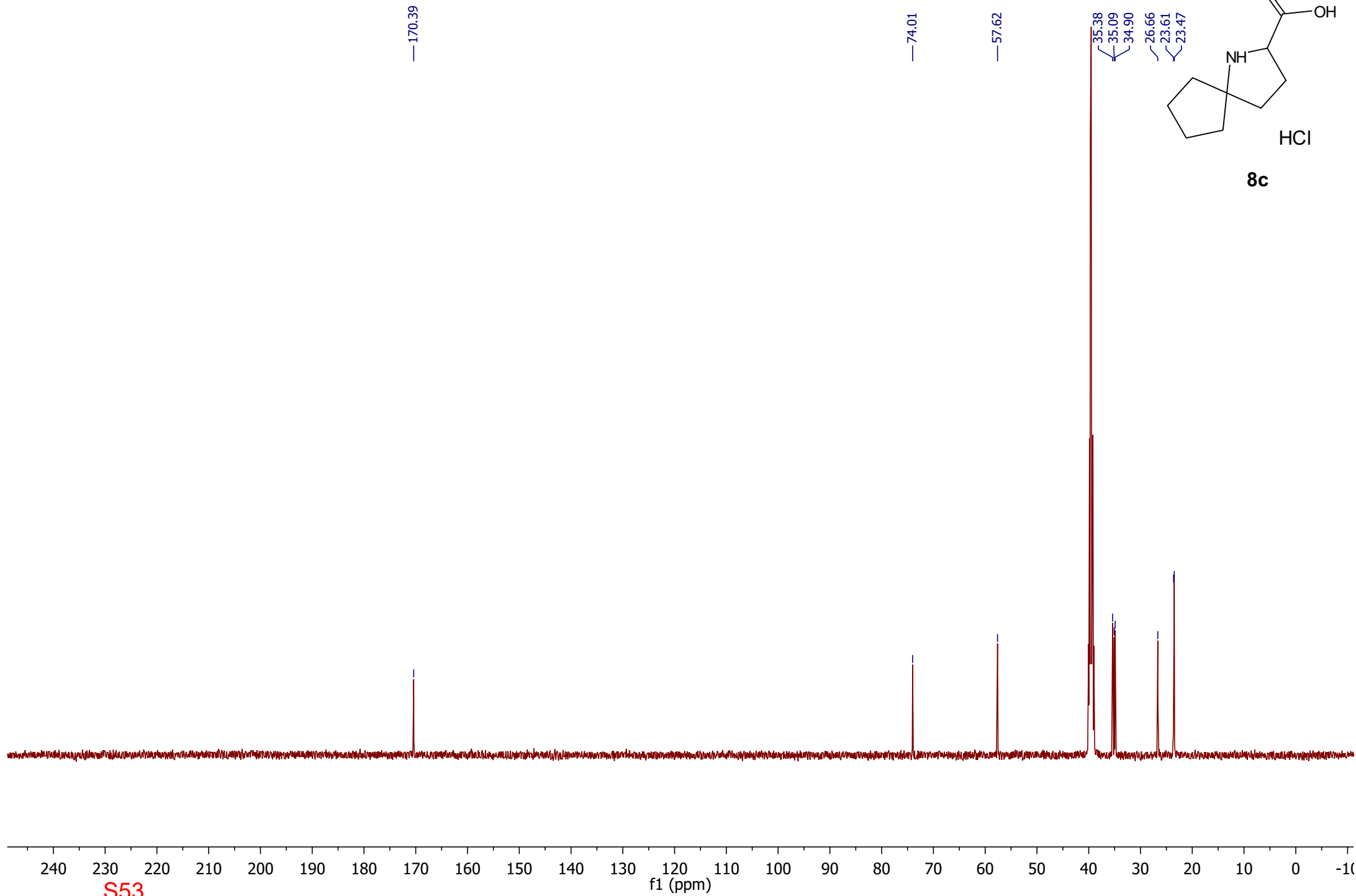
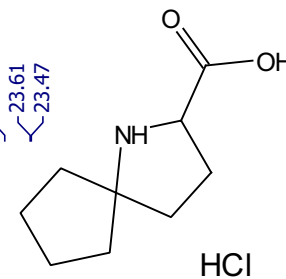
*MSD2 SPC, time=0.315 of D:\DATA\0221\L227867D\019-D7B-B3-R1675867.D ES-API, Scan, Frag: 100, "NEG"



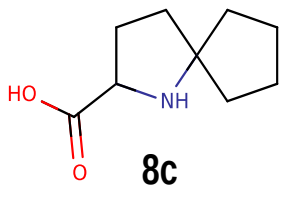
^1H NMR (500 MHz, $\text{DMSO}-d_6$) δ 13.92 (s, 1H), 9.98 (s, 1H), 8.66 (s, 1H), 4.37 (s, 1H), 2.40 – 2.29 (m, 1H), 2.09 – 1.99 (m, 2H), 1.99 – 1.94 (m, 1H), 1.93 – 1.80 (m, 2H), 1.78 – 1.61 (m, 4H), 1.61 – 1.51 (m, 2H).



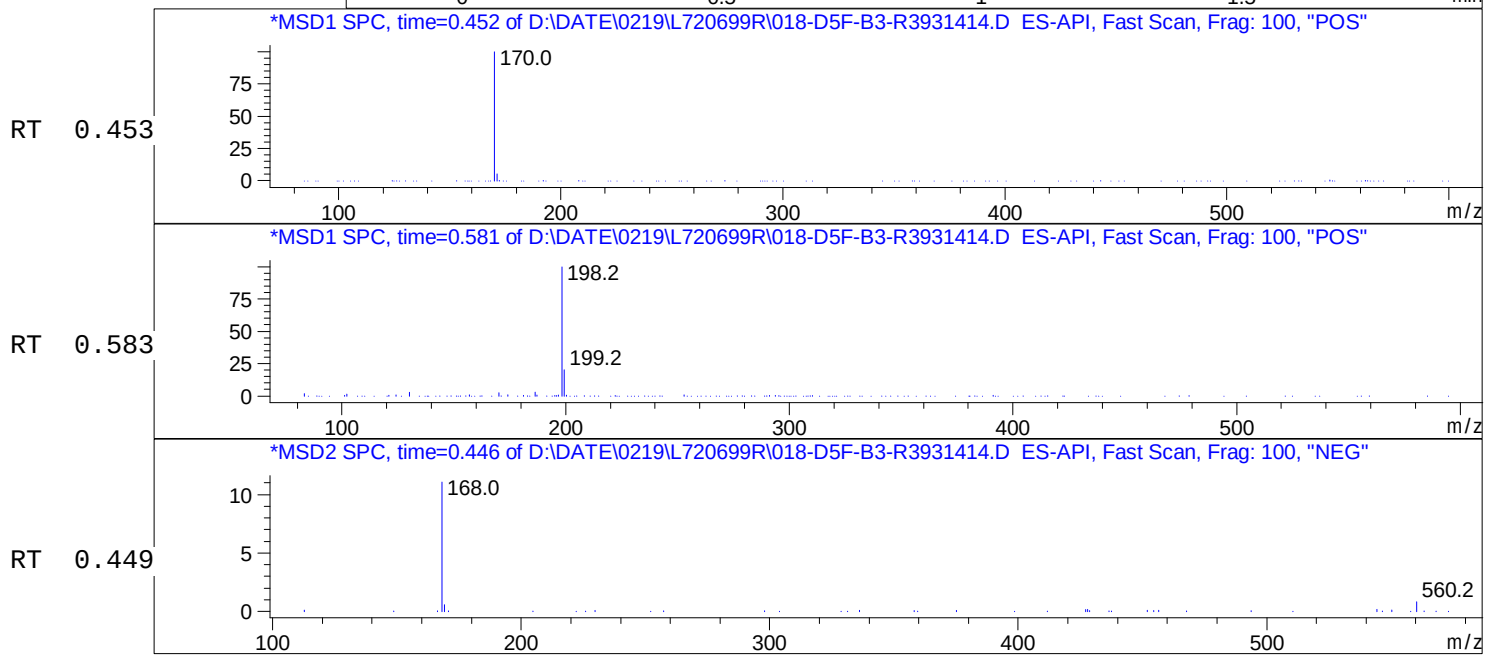
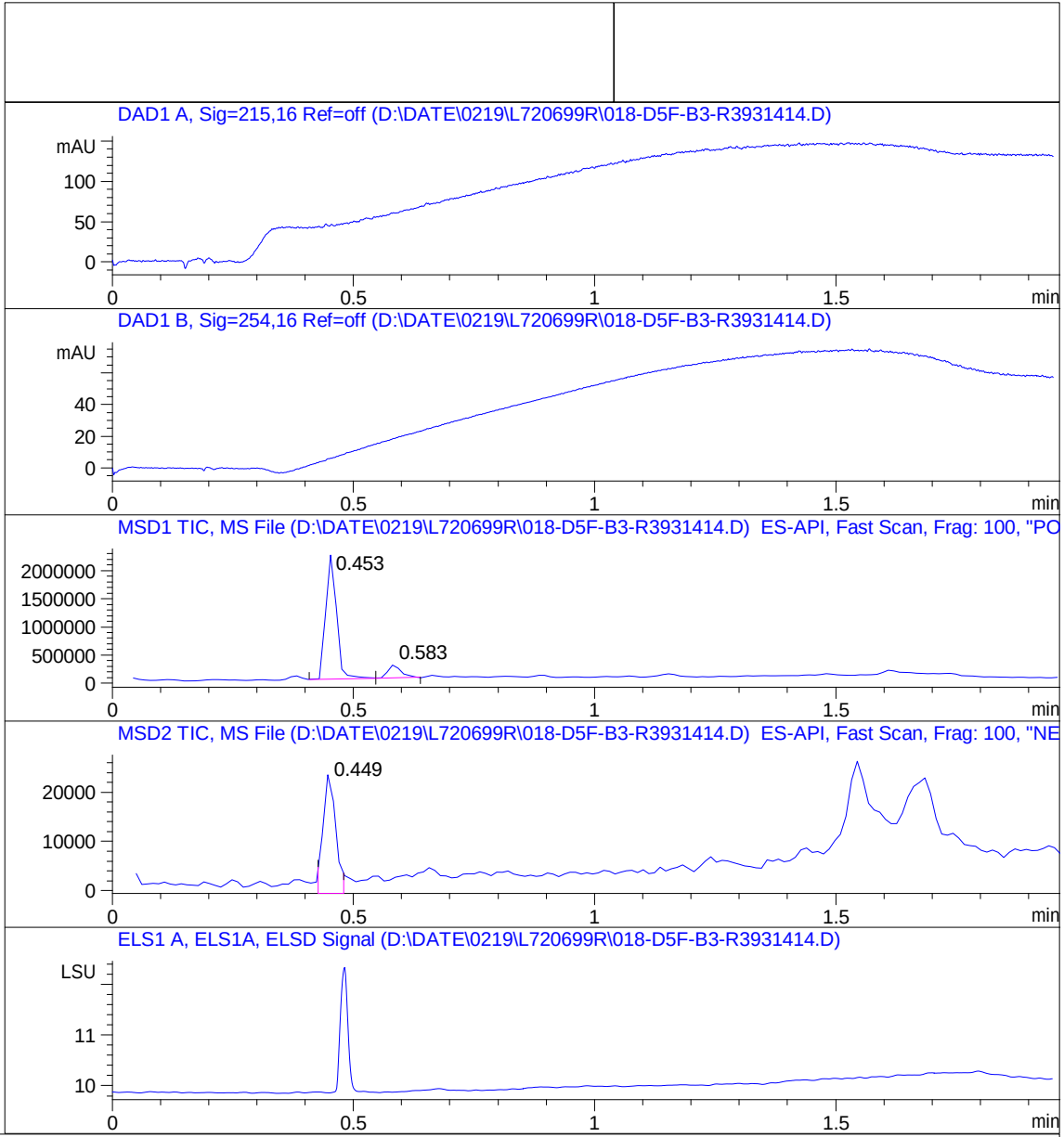
^{13}C NMR (126 MHz, DMSO) δ 170.4, 74.0, 57.6, 35.4, 35.1, 34.9, 26.7, 23.6, 23.5.



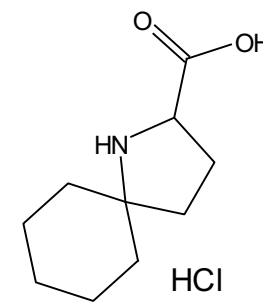
HCl



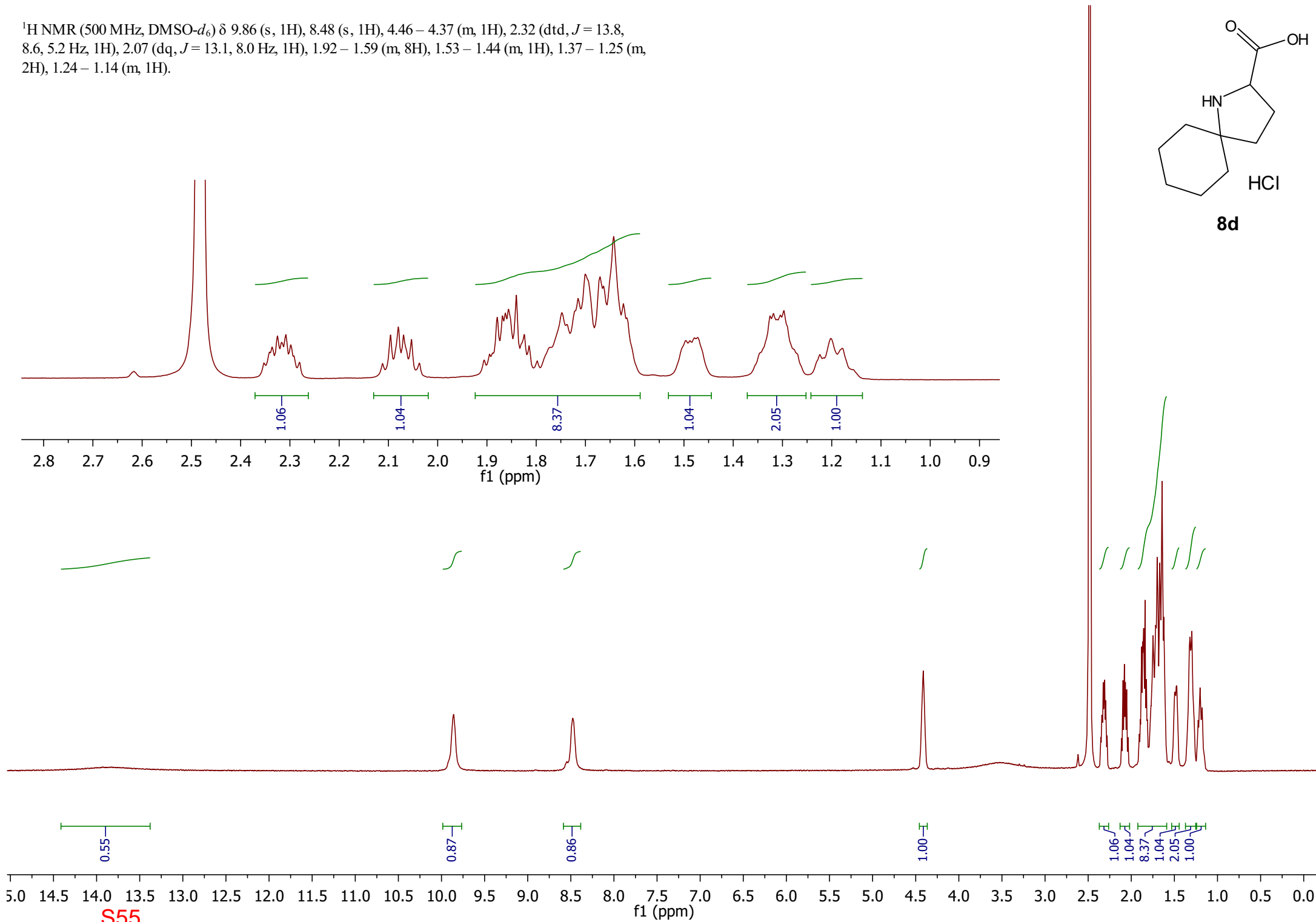
Mol Wt 205.68
Exact Mass 169.13



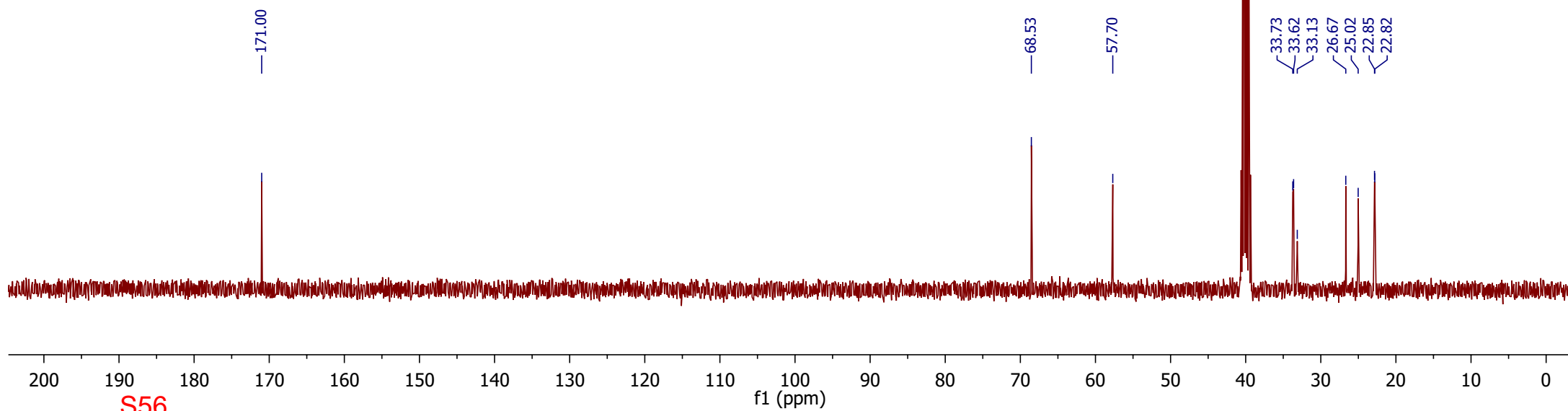
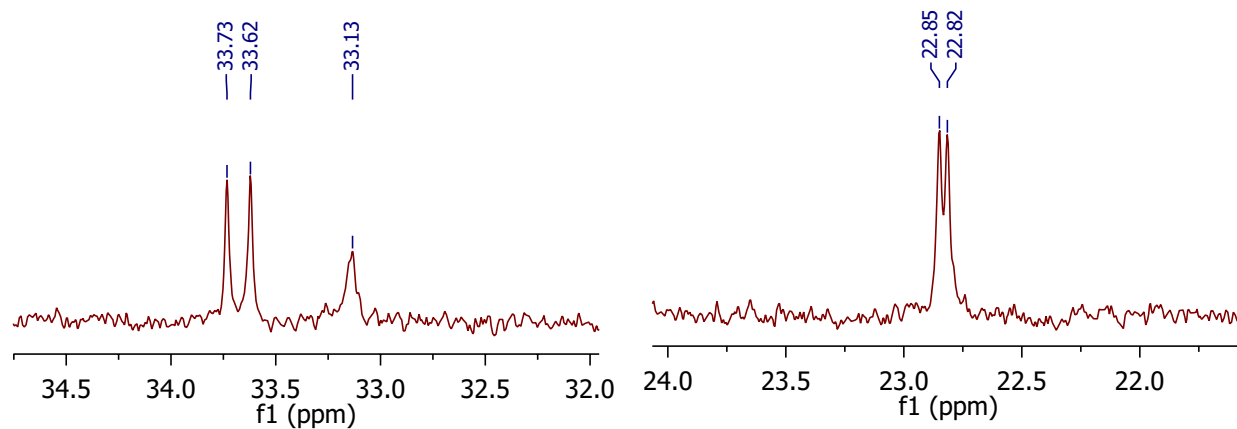
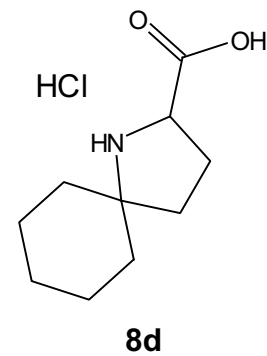
^1H NMR (500 MHz, $\text{DMSO}-d_6$) δ 9.86 (s, 1H), 8.48 (s, 1H), 4.46 – 4.37 (m, 1H), 2.32 (dtd, $J = 13.8$, 8.6, 5.2 Hz, 1H), 2.07 (dq, $J = 13.1$, 8.0 Hz, 1H), 1.92 – 1.59 (m, 8H), 1.53 – 1.44 (m, 1H), 1.37 – 1.25 (m, 2H), 1.24 – 1.14 (m, 1H).



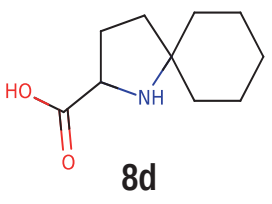
8d



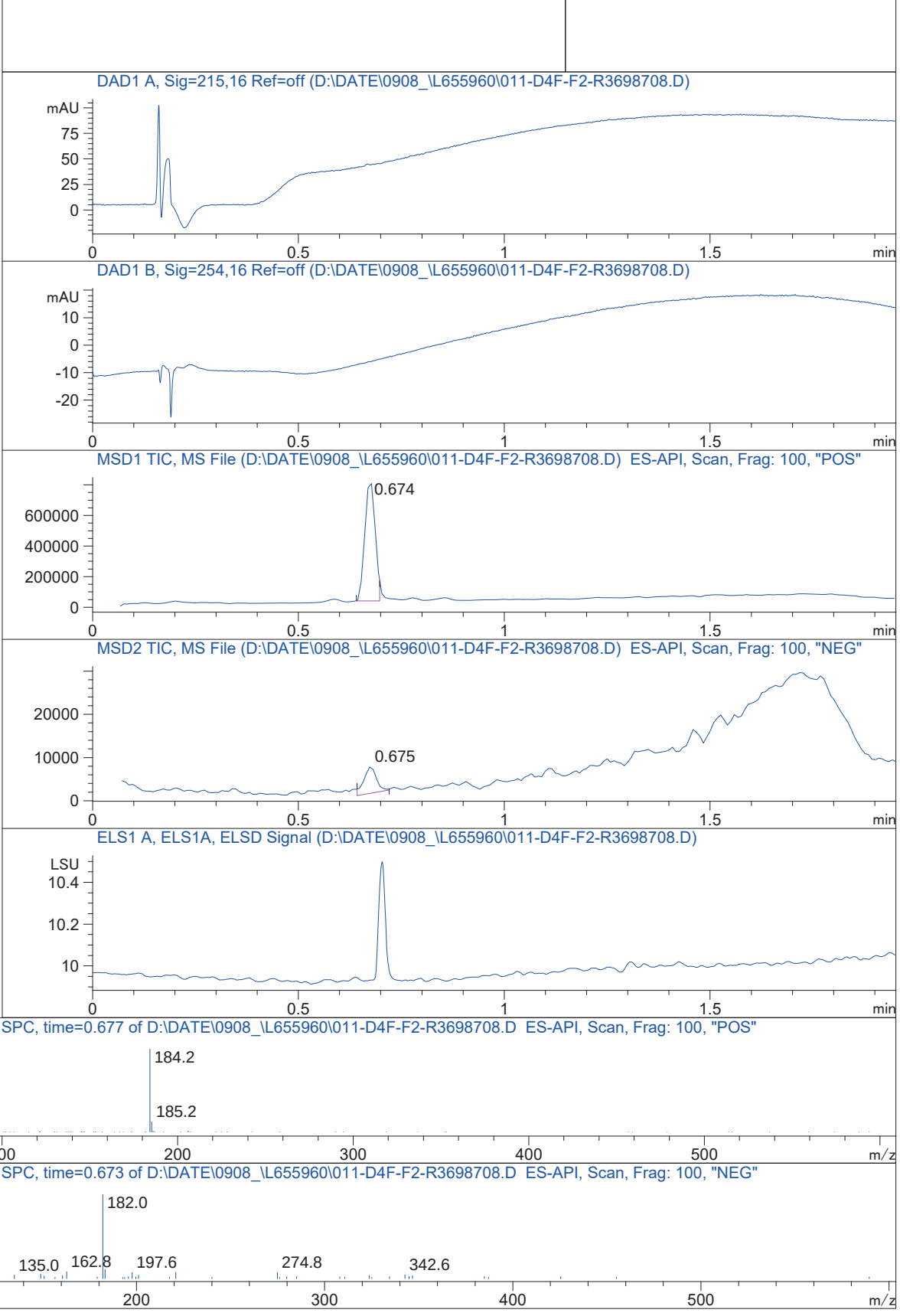
^{13}C NMR (DMSO, 101 MHz) δ 171.00, 68.53, 57.70, 33.73, 33.62, 33.13, 26.67, 25.02, 22.85, 22.82.



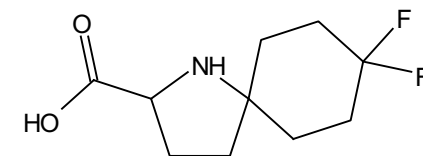
HCl



Mol Wt 219.71
Exact Mass 183.15

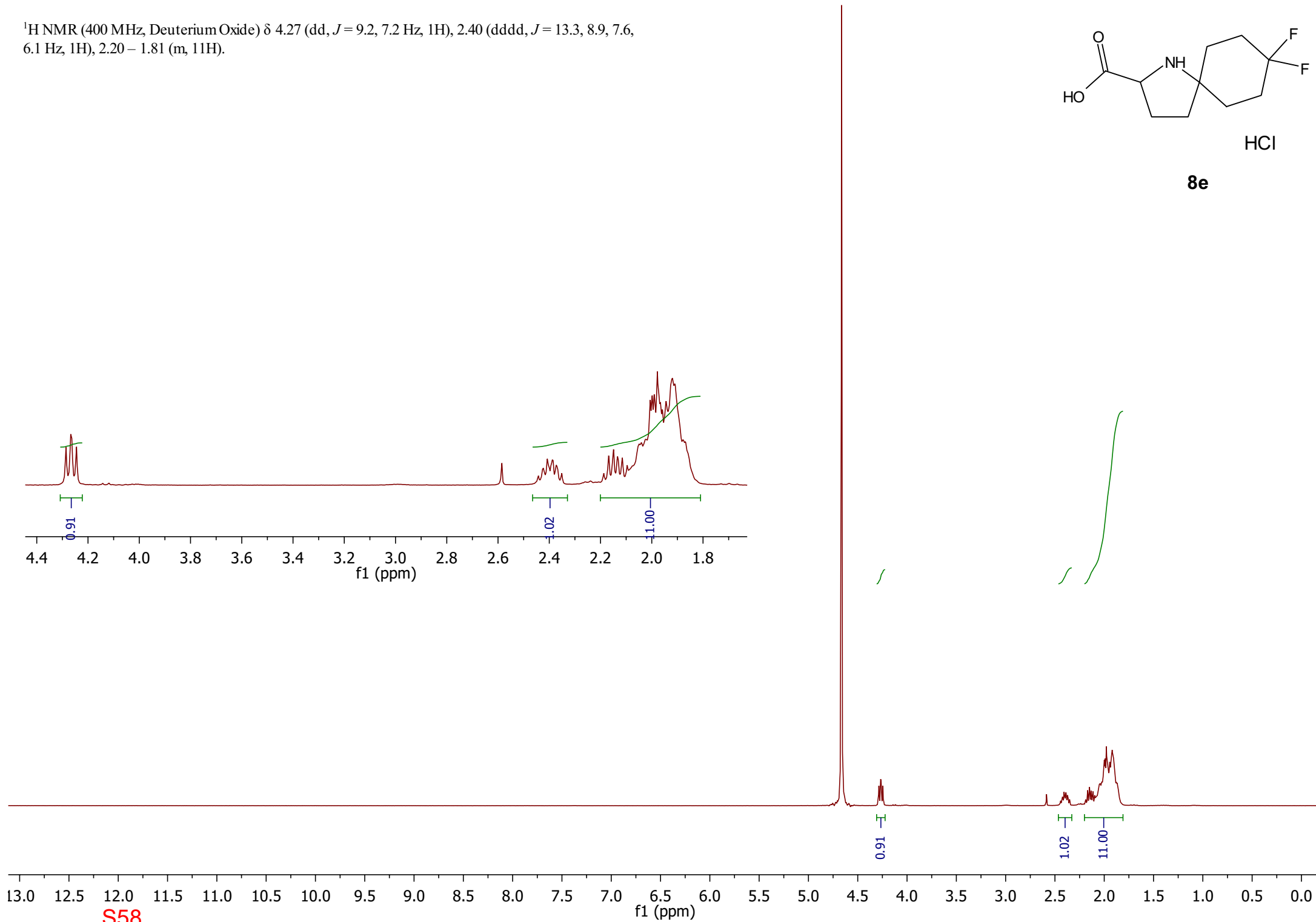


^1H NMR (400 MHz, Deuterium Oxide) δ 4.27 (dd, $J = 9.2, 7.2$ Hz, 1H), 2.40 (dddd, $J = 13.3, 8.9, 7.6, 6.1$ Hz, 1H), 2.20 – 1.81 (m, 11H).

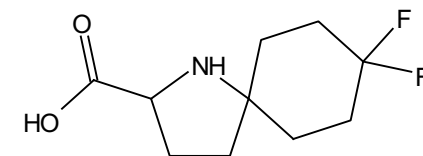


HCl

8e

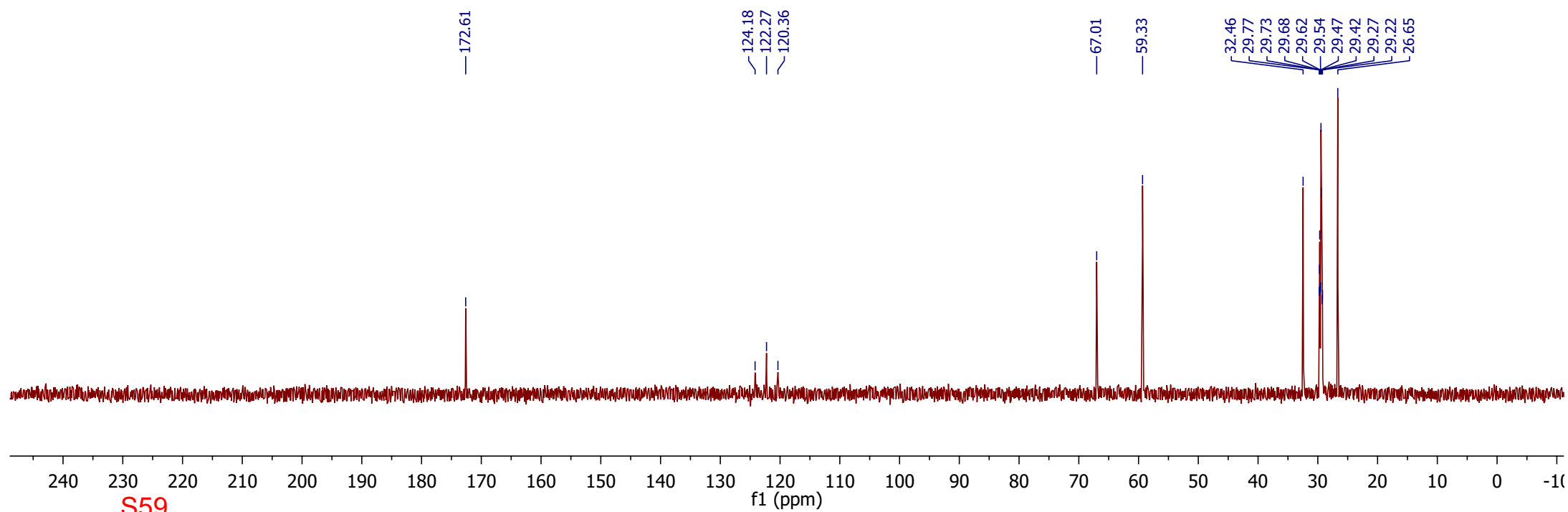
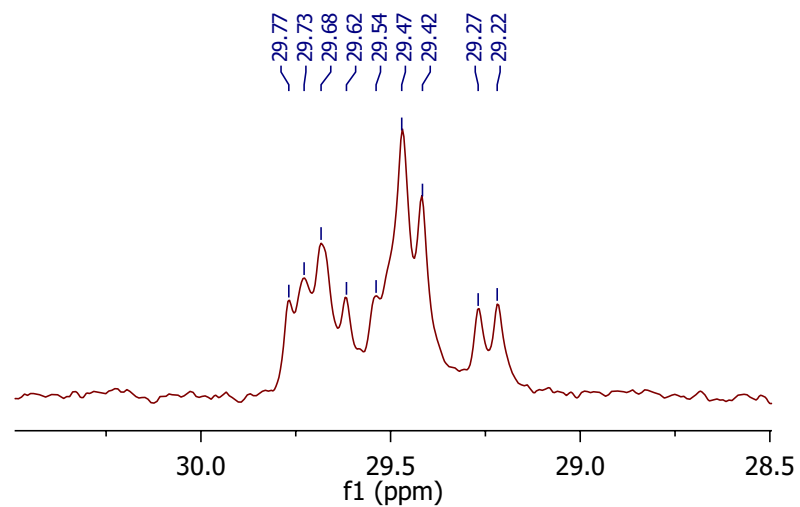


^{13}C NMR (126 MHz, D_2O) δ 172.61, 122.27 (t, $^1J_{\text{CF}} = 240$ Hz), 67.01, 59.33, 32.46, 29.86 – 29.09 (m), 26.65

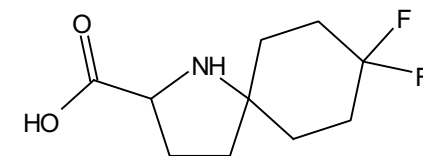


HCl

8e



^{19}F NMR (376 MHz, $\text{DMSO-}d_6$) δ -97.84 (d, $J = 235.7$ Hz), -100.85 (d, $J = 235.7$ Hz).

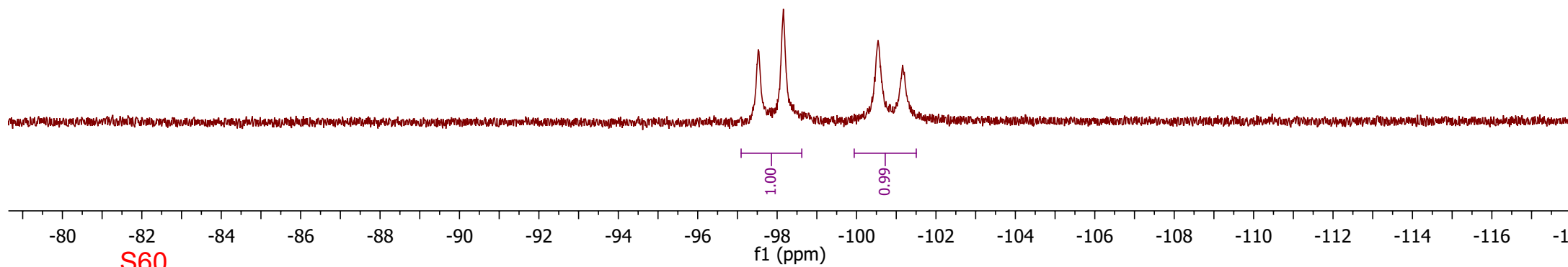


HCl

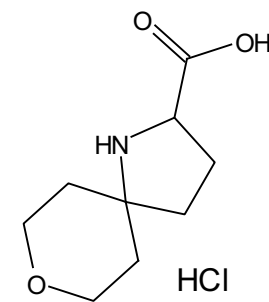
8e

A (d)
-97.84

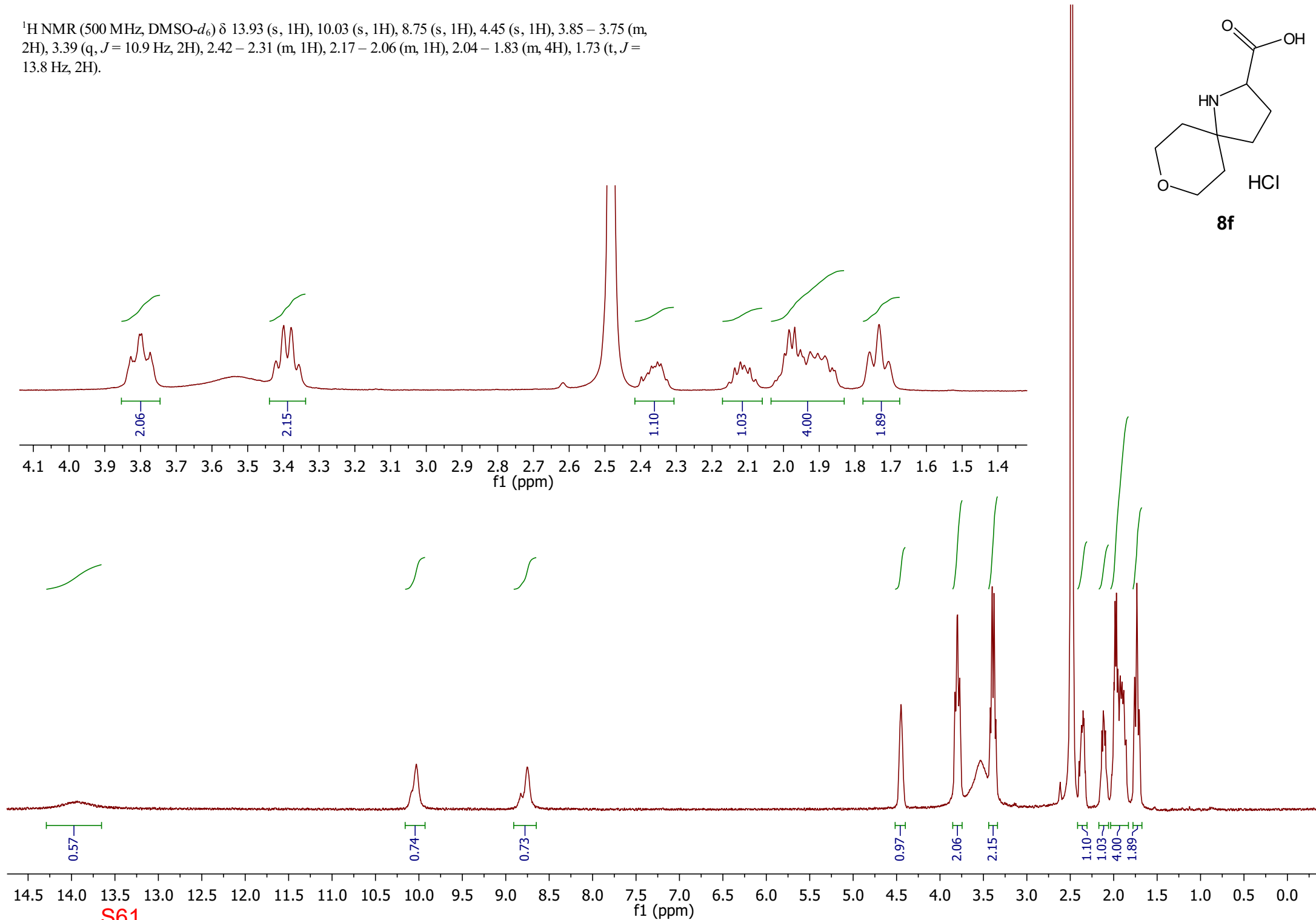
B (d)
-100.85



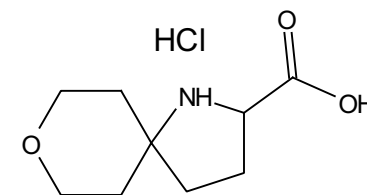
^1H NMR (500 MHz, $\text{DMSO-}d_6$) δ 13.93 (s, 1H), 10.03 (s, 1H), 8.75 (s, 1H), 4.45 (s, 1H), 3.85 – 3.75 (m, 2H), 3.39 (q, J = 10.9 Hz, 2H), 2.42 – 2.31 (m, 1H), 2.17 – 2.06 (m, 1H), 2.04 – 1.83 (m, 4H), 1.73 (t, J = 13.8 Hz, 2H).



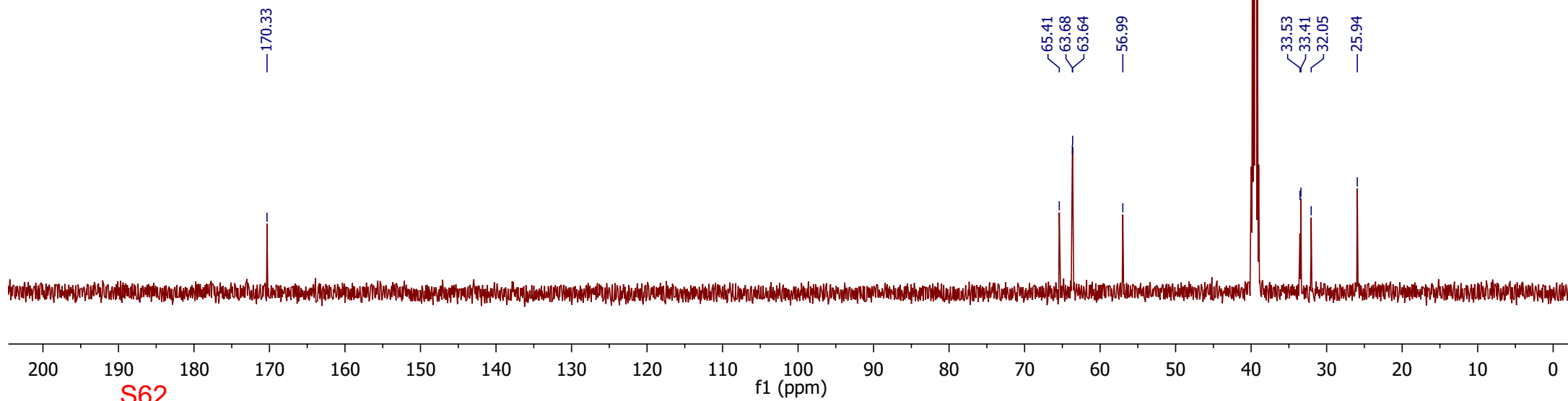
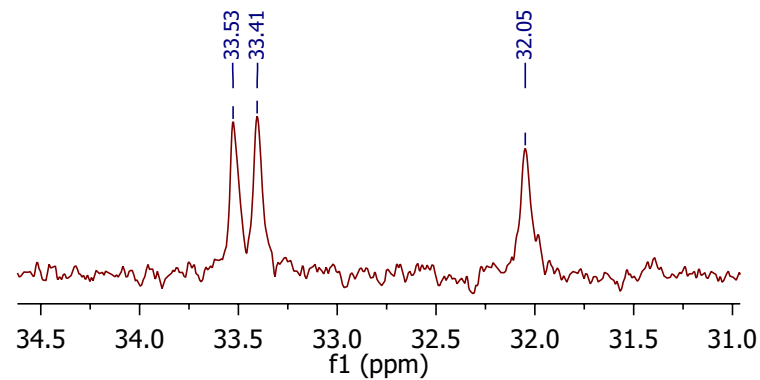
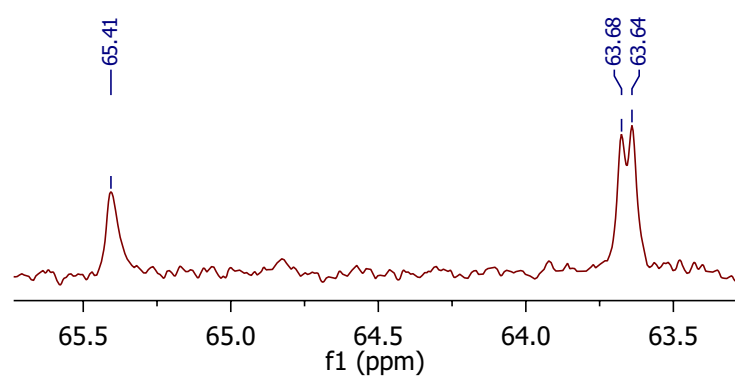
8f



^{13}C NMR (DMSO, 126 MHz) δ 170.33, 65.41, 63.68, 63.64, 56.99, 33.53, 33.41, 32.05, 25.94.

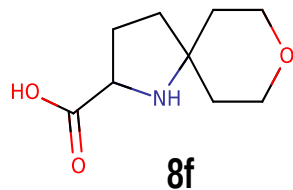


8f



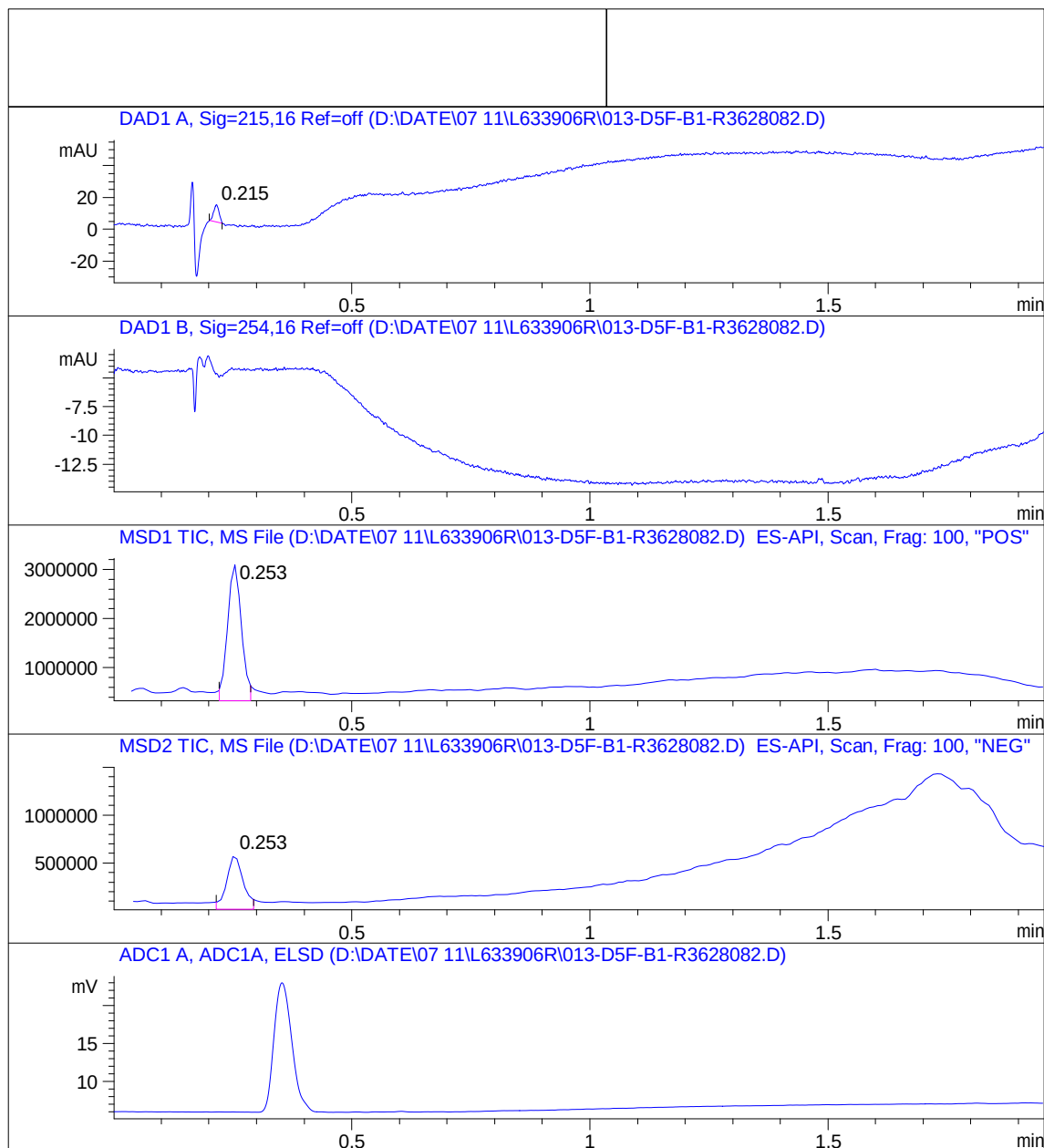
MaxPeak: 100.00%
Ret_Time: 0.215 min

HCl

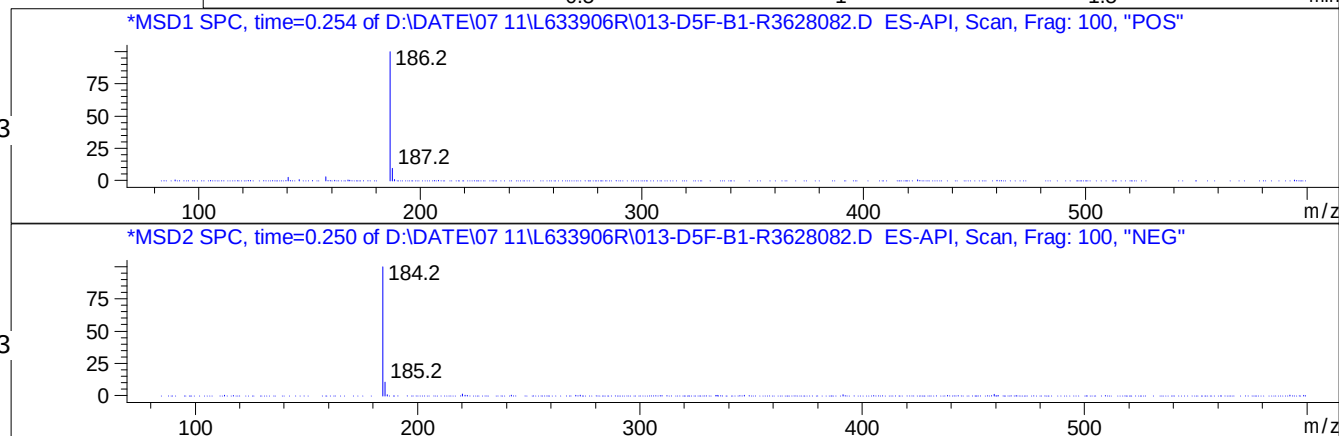


Mol Wt 221.68
Exact Mass 185.12

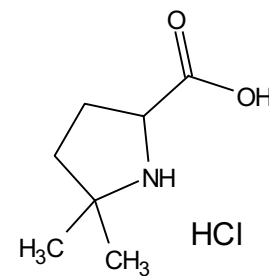
#	Time	Area%
1	0.215	100.00



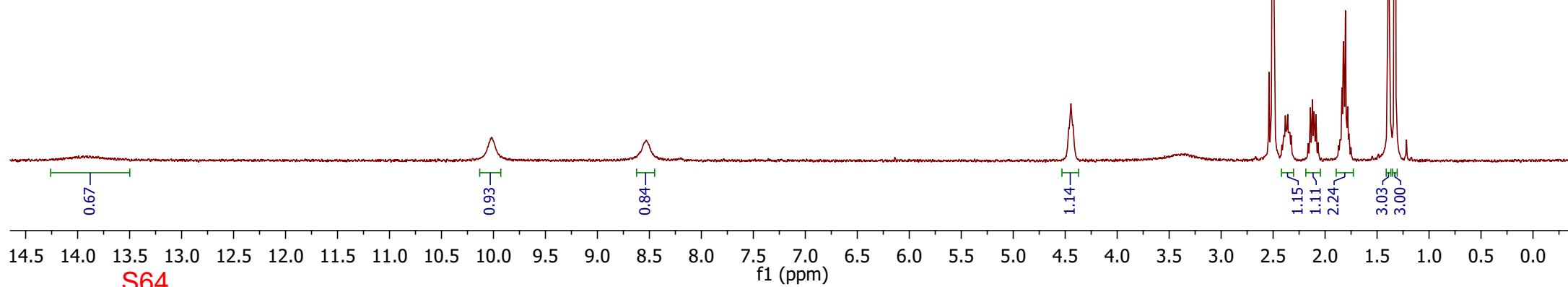
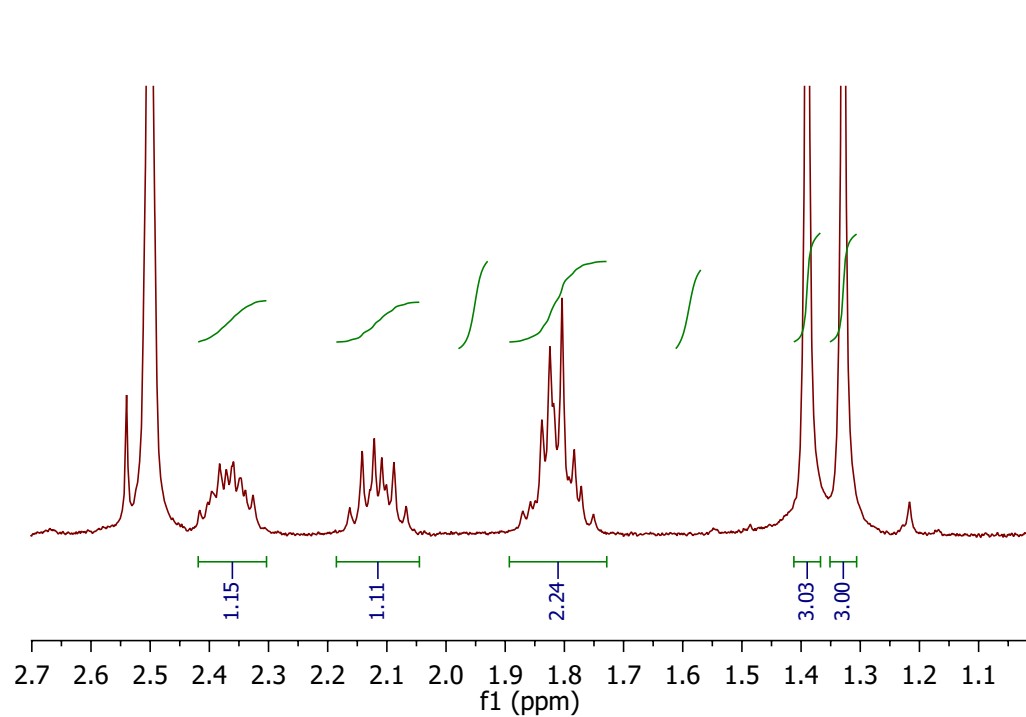
RT 0.253



^1H NMR (400 MHz, $\text{DMSO}-d_6$) δ 13.99 (s, 1H), 10.02 (s, 1H), 8.53 (s, 1H), 4.45 (t, $J = 8.1$ Hz, 1H), 2.44 – 2.30 (m, 1H), 2.12 (dq, $J = 13.3, 8.3$ Hz, 1H), 1.89 – 1.73 (m, 2H), 1.39 (s, 3H), 1.33 (s, 3H).

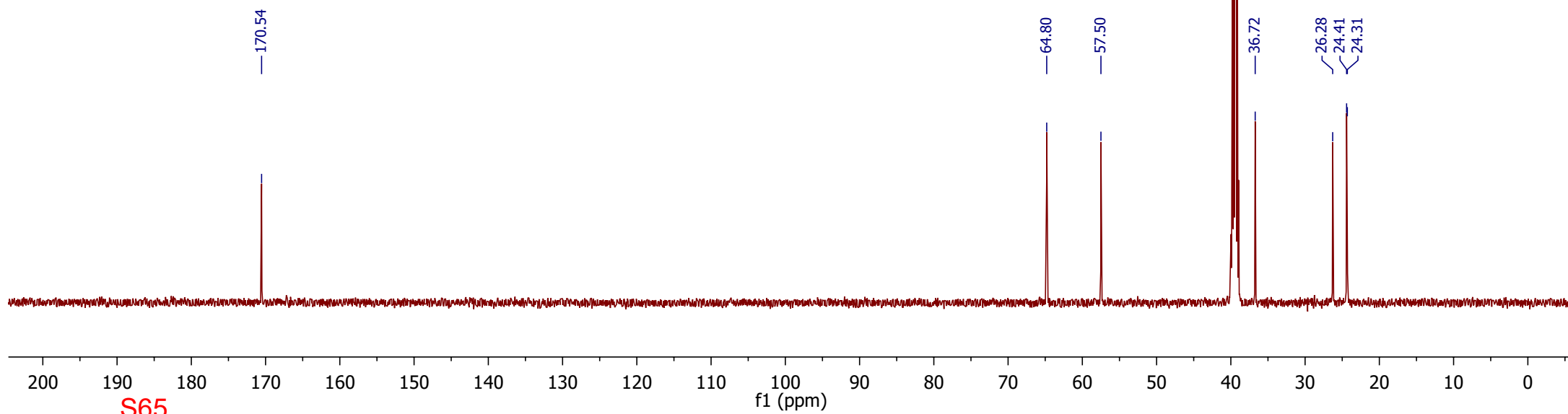
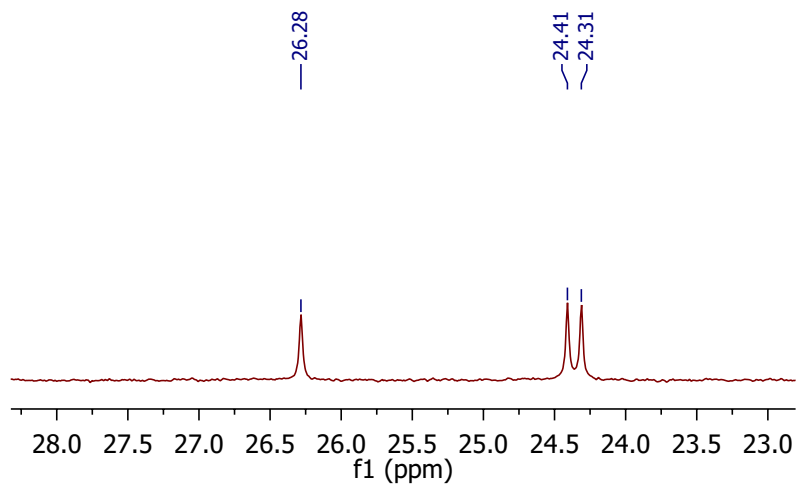
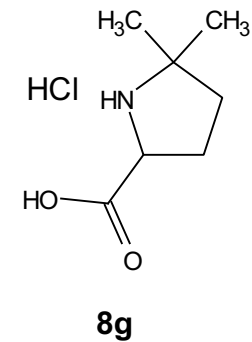


8g

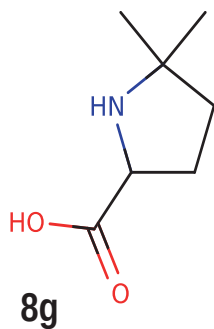


S64

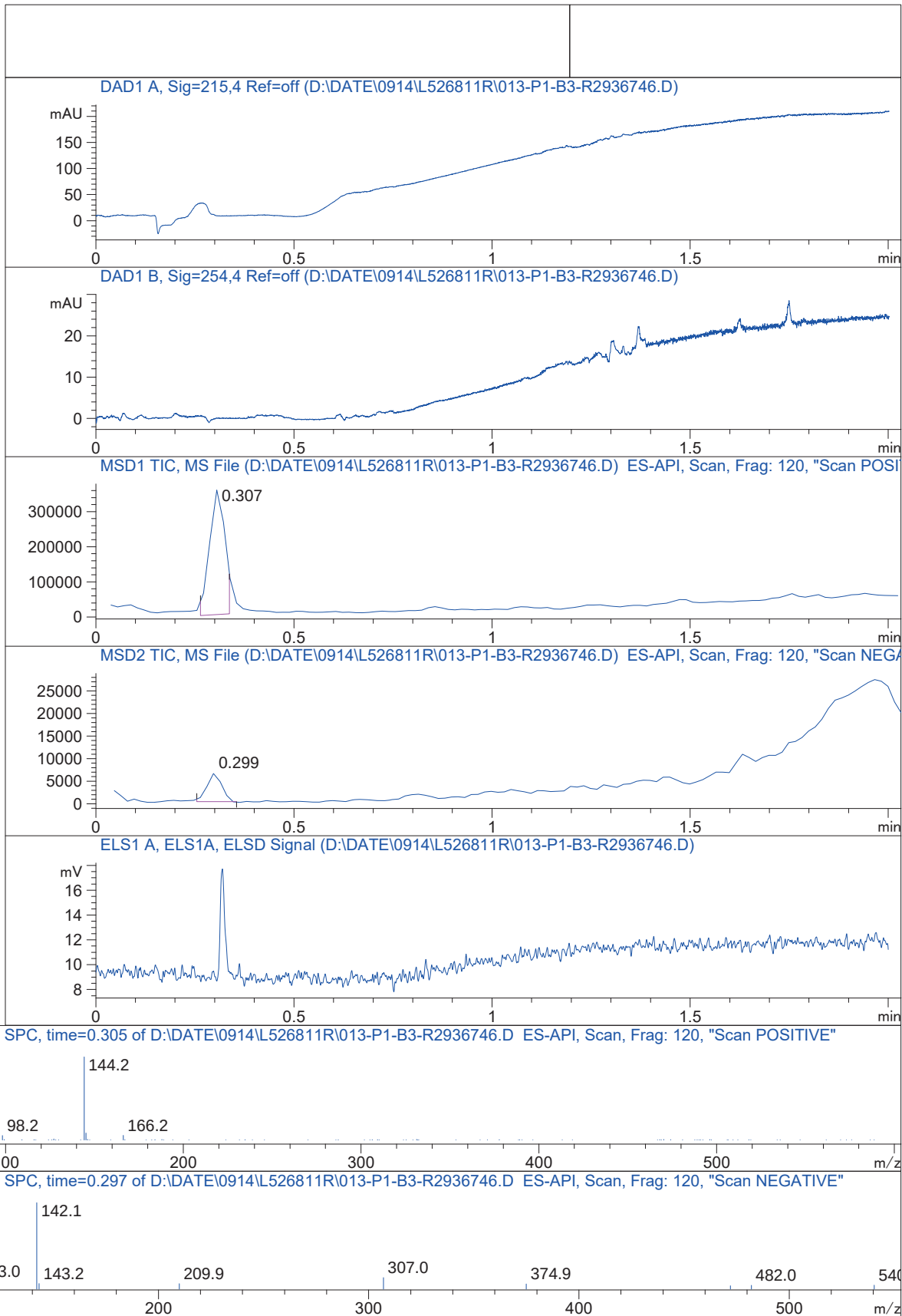
^{13}C NMR (DMSO, 126 MHz) δ 170.54, 64.80, 57.50, 36.72, 26.28, 24.41, 24.31.



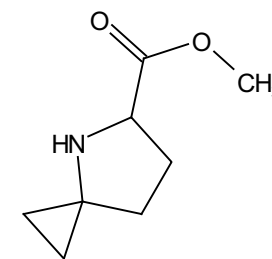
HCl



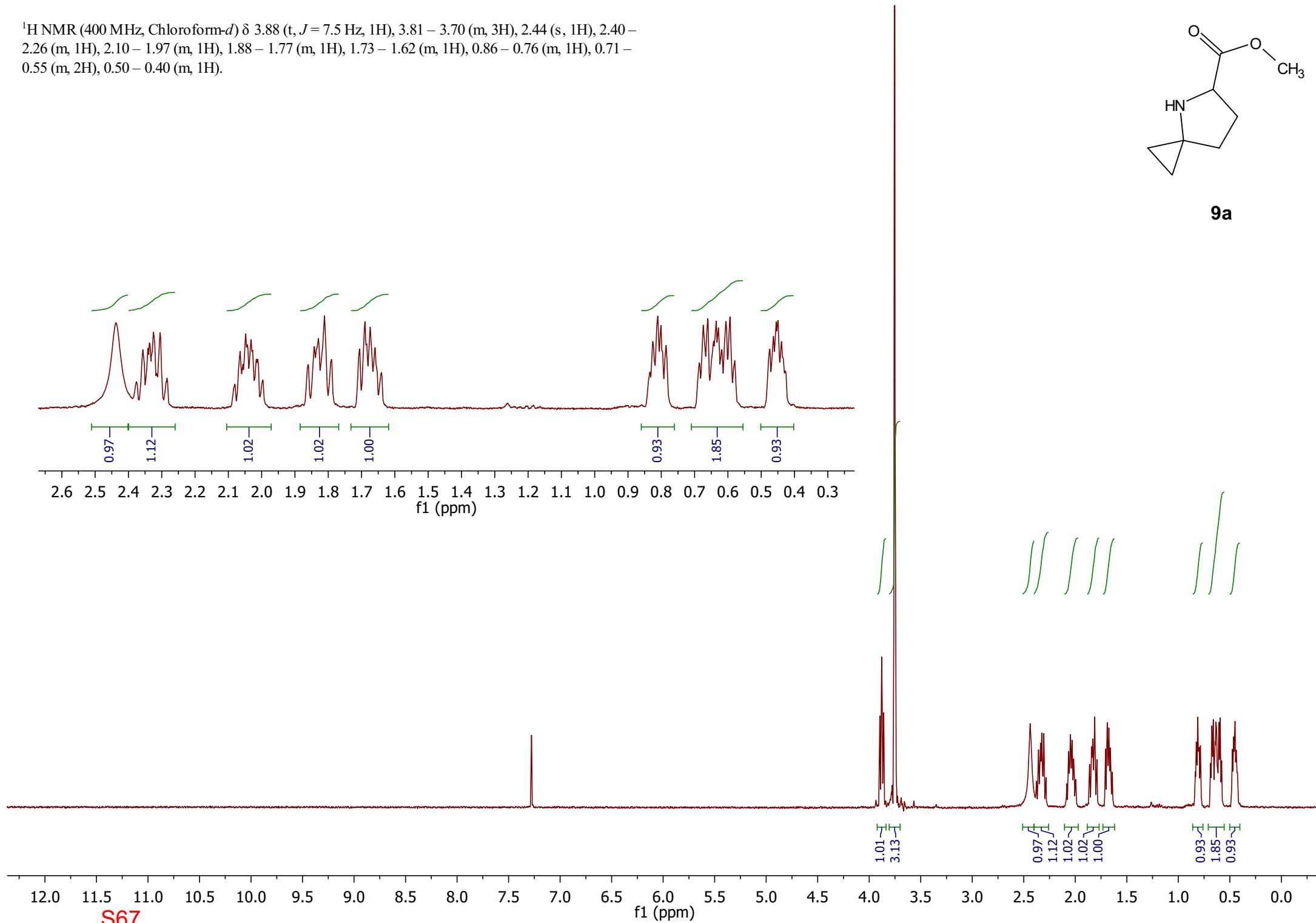
Mol Wt 179.64
Exact Mass 143.11



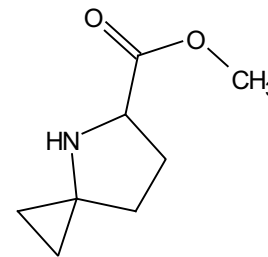
^1H NMR (400 MHz, Chloroform-*d*) δ 3.88 (t, $J = 7.5$ Hz, 1H), 3.81 – 3.70 (m, 3H), 2.44 (s, 1H), 2.40 – 2.26 (m, 1H), 2.10 – 1.97 (m, 1H), 1.88 – 1.77 (m, 1H), 1.73 – 1.62 (m, 1H), 0.86 – 0.76 (m, 1H), 0.71 – 0.55 (m, 2H), 0.50 – 0.40 (m, 1H).



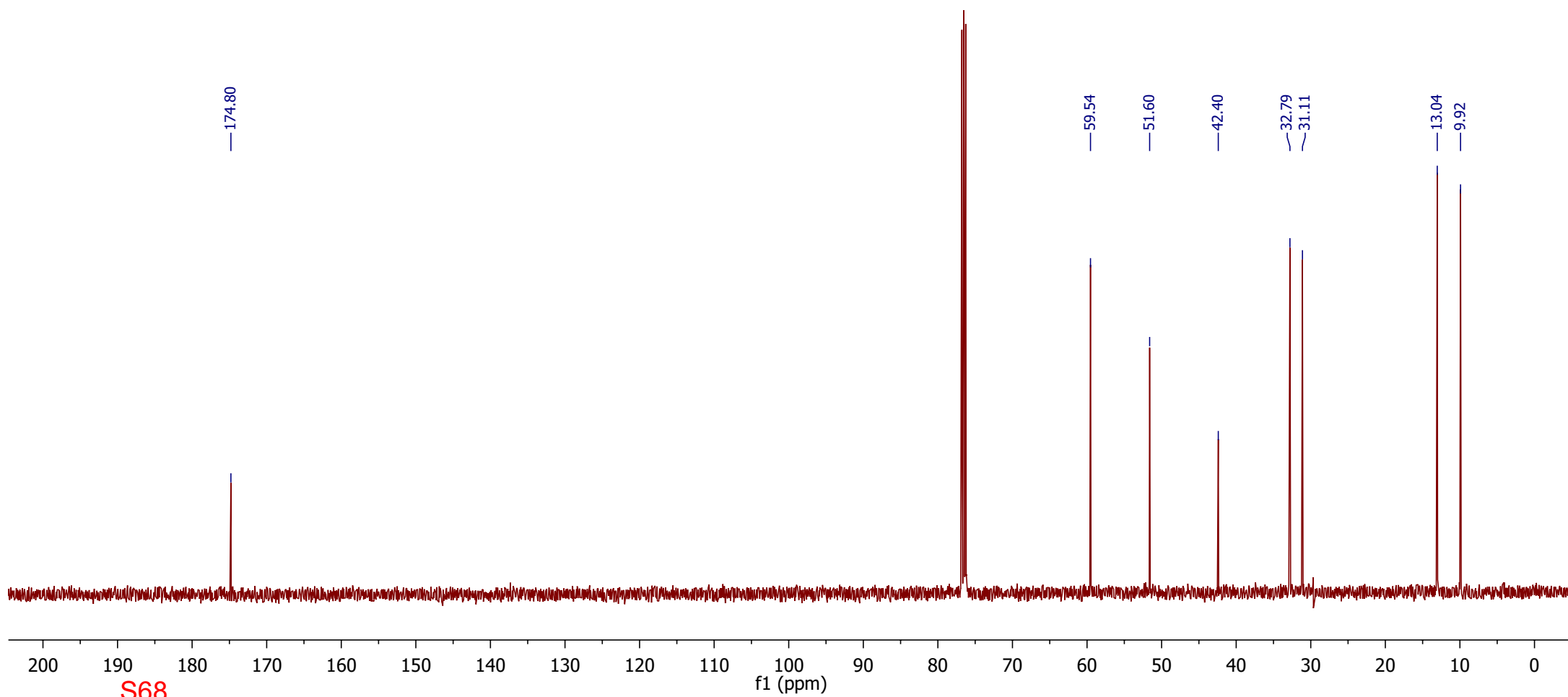
9a

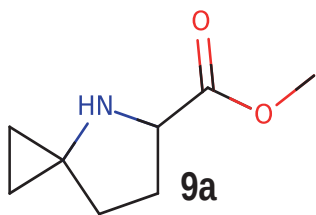


^{13}C NMR (CDCl_3 , 126 MHz) δ 174.80, 59.54, 51.60, 42.40, 32.79, 31.11, 13.04, 9.92.



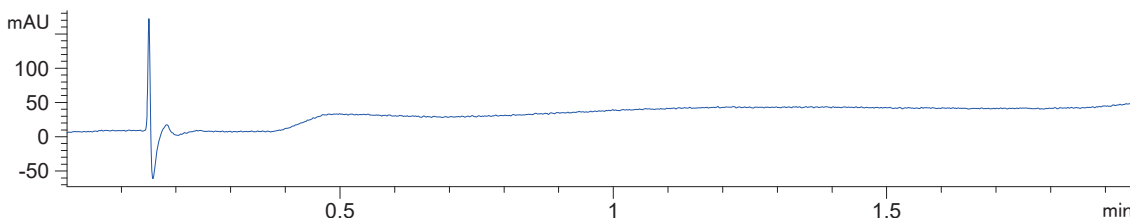
9a



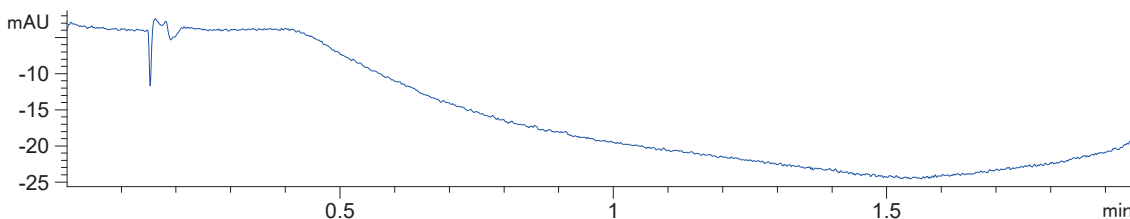


Mol Wt **155.19**
Exact Mass **155.11**

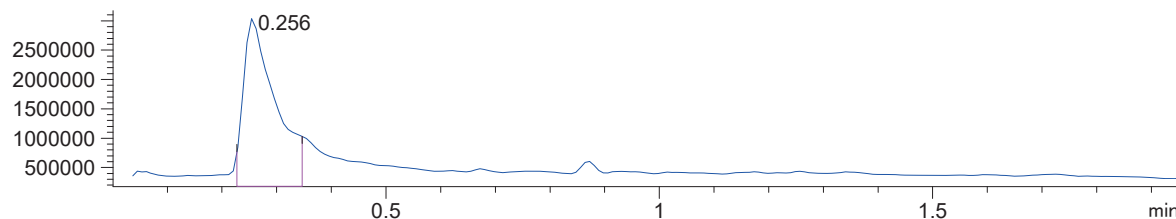
DAD1 A, Sig=215,16 Ref=off (D:\DATE\0913\L657471R-PART2\009-D5F-A7-R2399405.D)



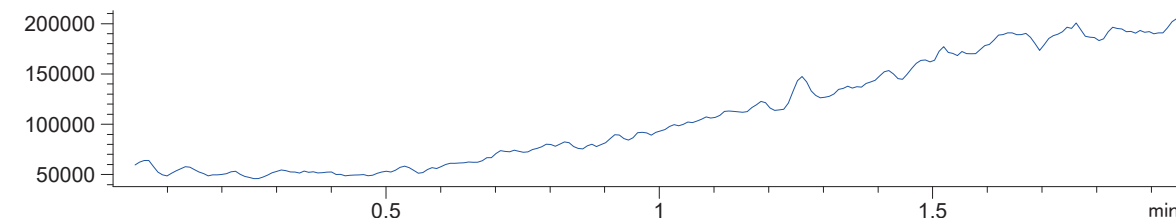
DAD1 B, Sig=254,16 Ref=off (D:\DATE\0913\L657471R-PART2\009-D5F-A7-R2399405.D)



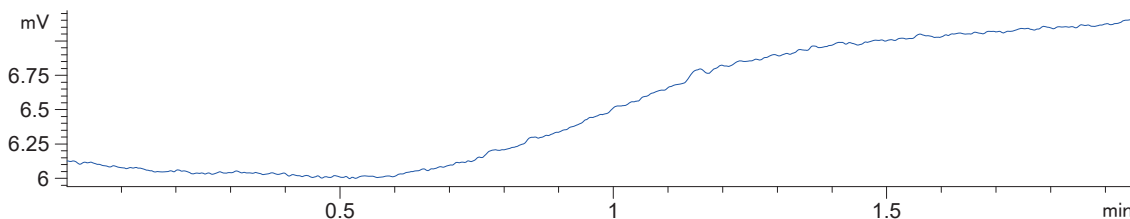
MSD1 TIC, MS File (D:\DATE\0913\L657471R-PART2\009-D5F-A7-R2399405.D) ES-API, Scan, Frag: 100, "POS"



MSD2 TIC, MS File (D:\DATE\0913\L657471R-PART2\009-D5F-A7-R2399405.D) ES-API, Scan, Frag: 100, "NEG"

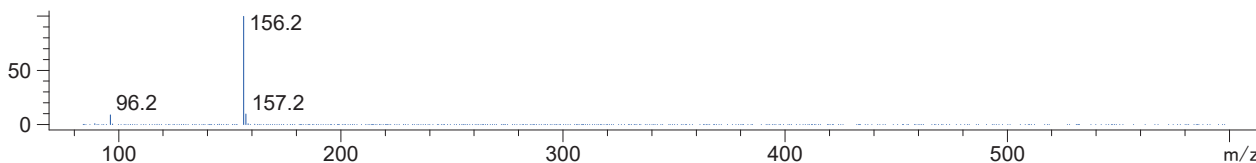


ADC1 A, ADC1A, ELSD (D:\DATE\0913\L657471R-PART2\009-D5F-A7-R2399405.D)

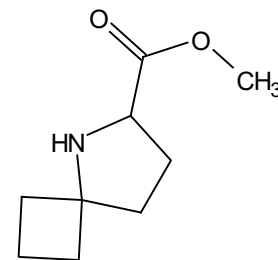


RT 0.256

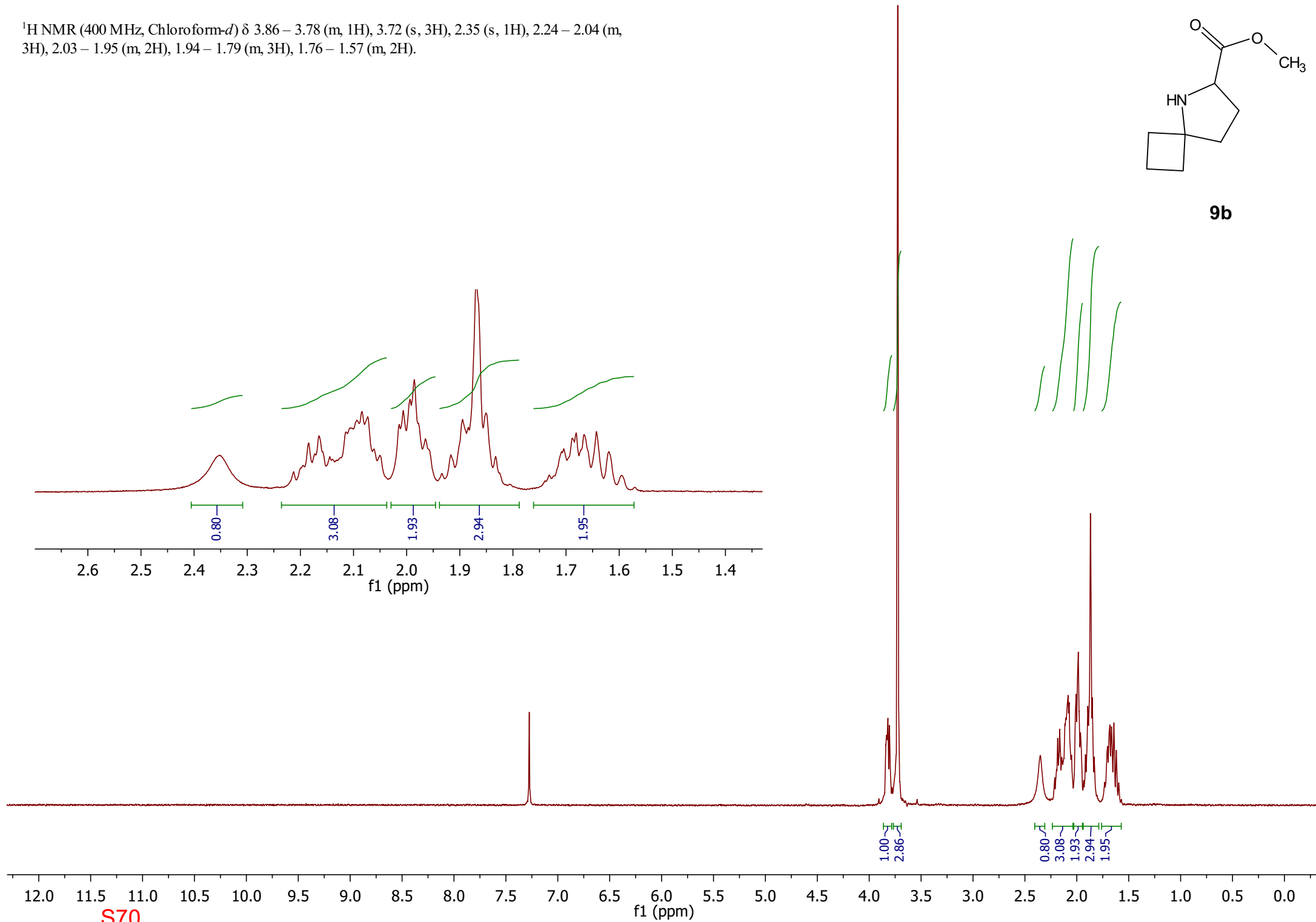
*MSD1 SPC, time=0.254 of D:\DATE\0913\L657471R-PART2\009-D5F-A7-R2399405.D ES-API, Scan, Frag: 100, "POS"



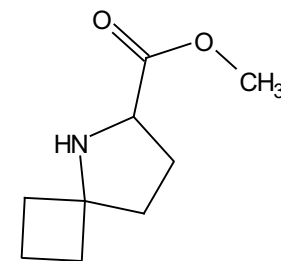
^1H NMR (400 MHz, Chloroform- d) δ 3.86 – 3.78 (m, 1H), 3.72 (s, 3H), 2.35 (s, 1H), 2.24 – 2.04 (m, 3H), 2.03 – 1.95 (m, 2H), 1.94 – 1.79 (m, 3H), 1.76 – 1.57 (m, 2H).



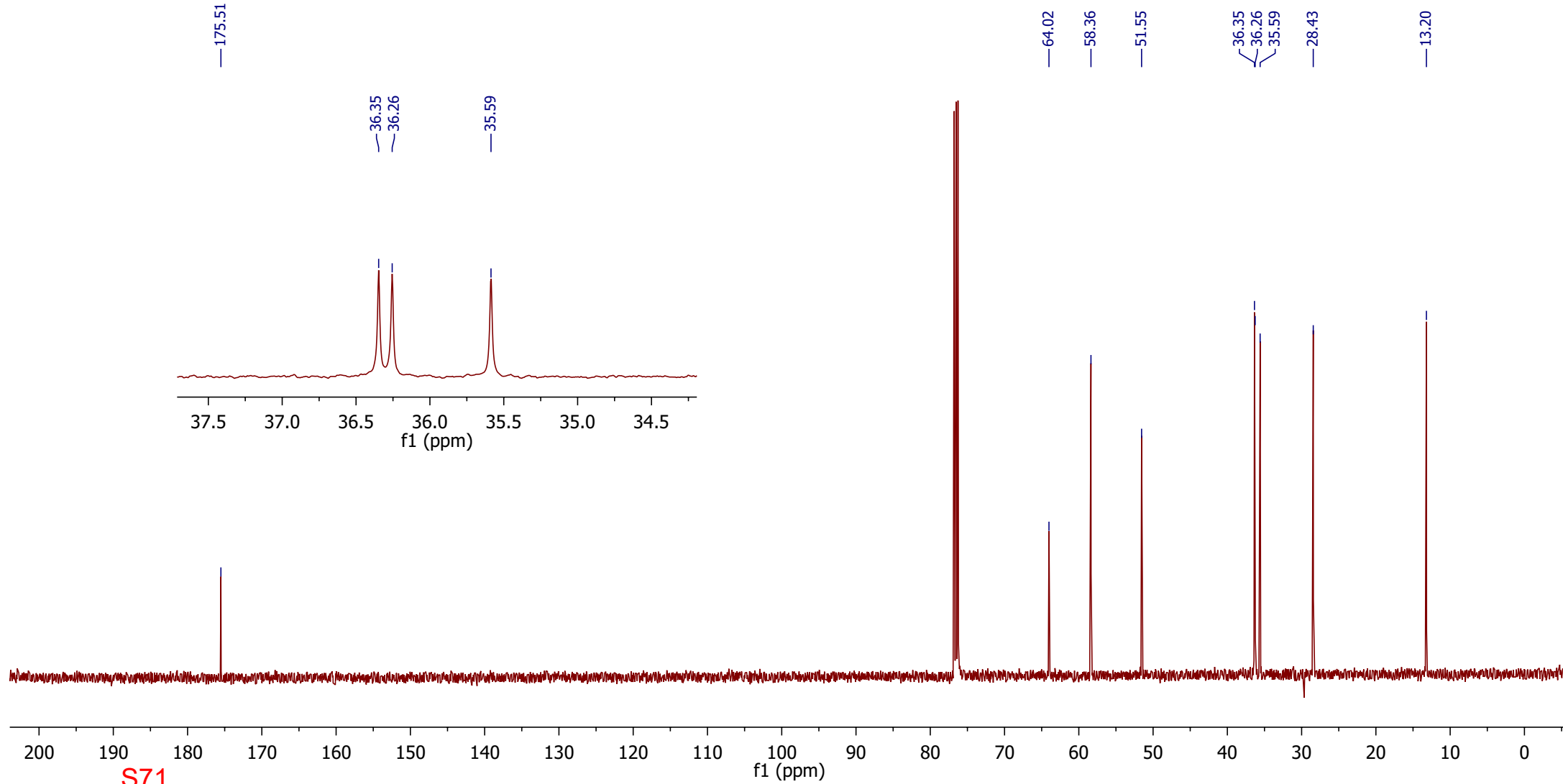
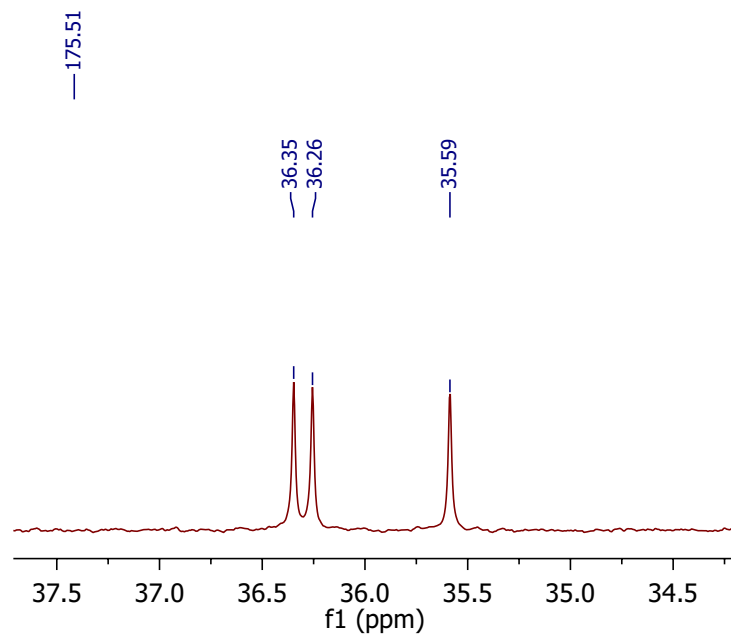
9b



^{13}C NMR (CDCl_3 , 126 MHz) δ 175.51, 64.02, 58.36, 51.55, 36.35, 36.26, 35.59, 28.43, 13.20.



9b

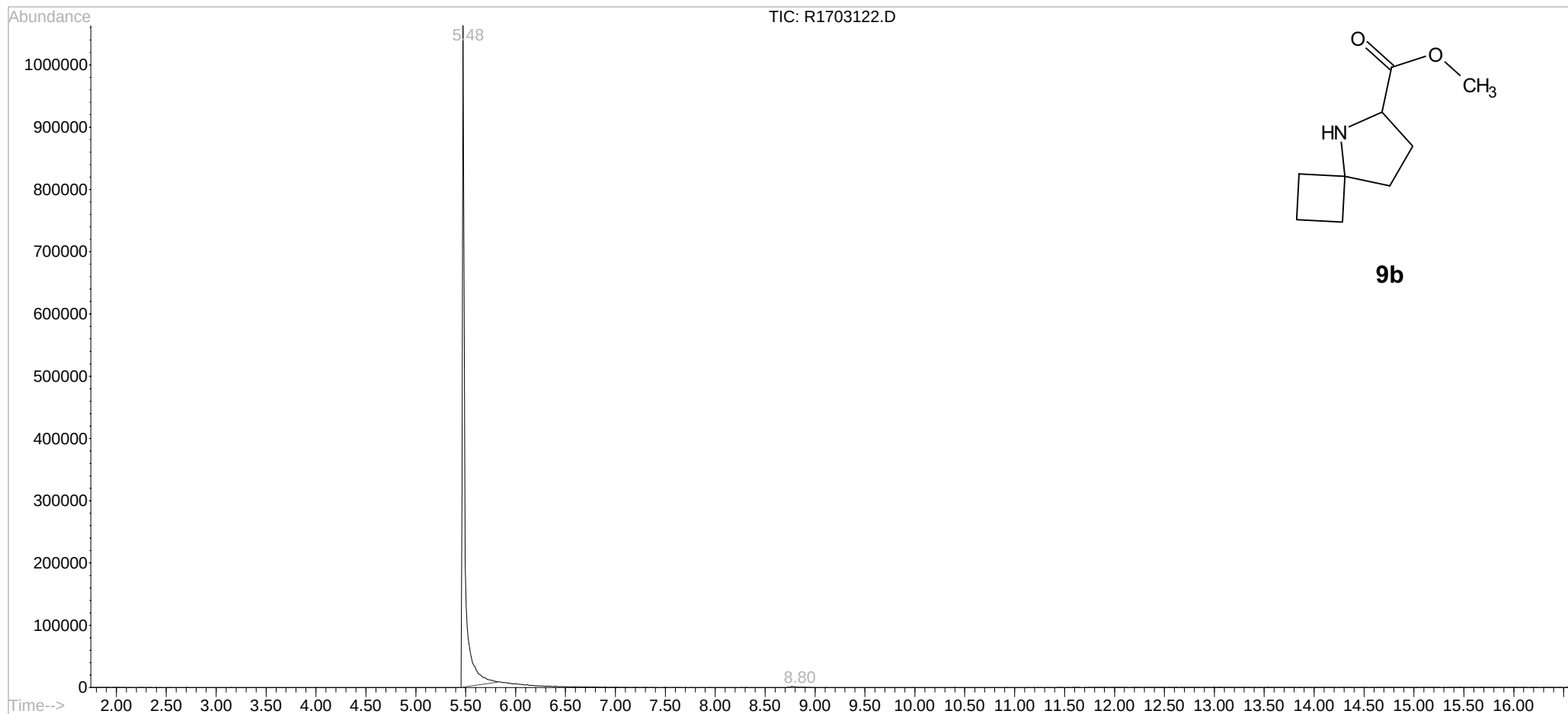


Library Search Report

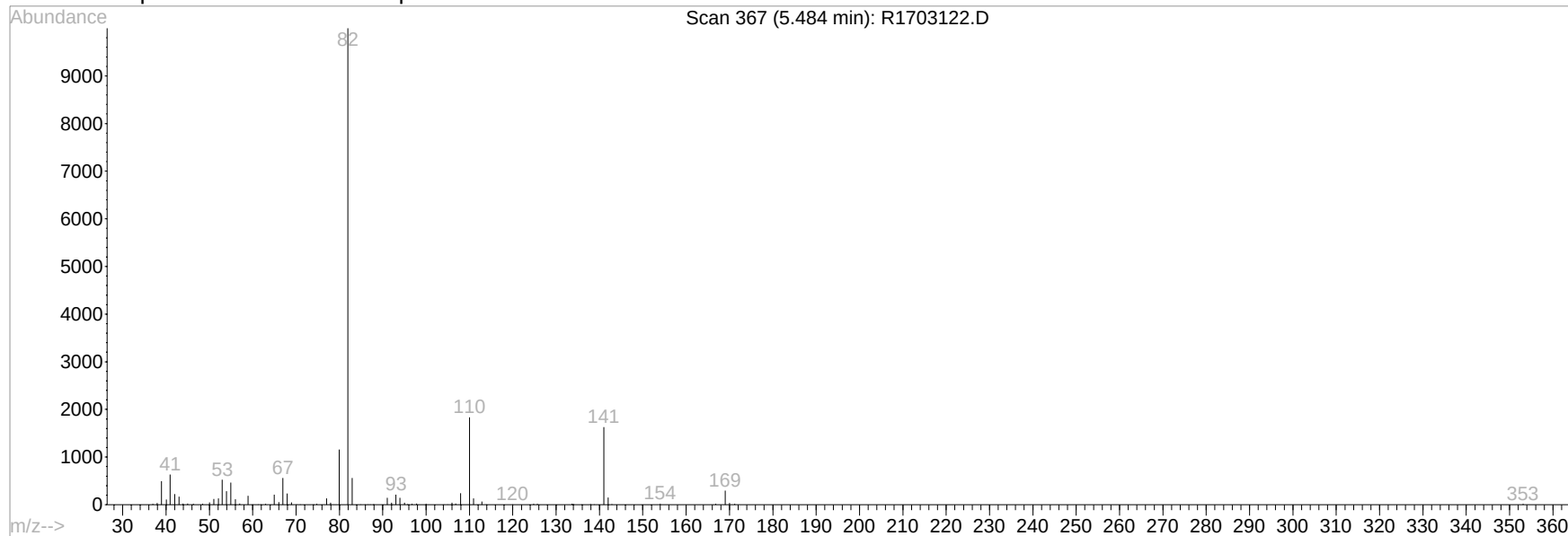
Data File :
Acq On
Sample : R1703122
Misc : CH3CN

Vial: 15
Operator:
Inst : GC/MS Ins
Multiplr: 1.00
Sample Amount: 0.00

MS Integration Params: autoint1.e
Method
Title :



Unknown Spectrum based on Apex

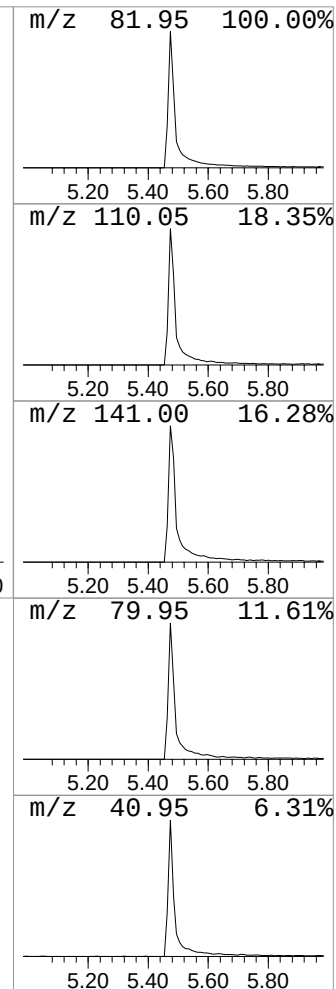


Peak Number: 1 at 5.48 min Area: 18123240 Area % 99.68

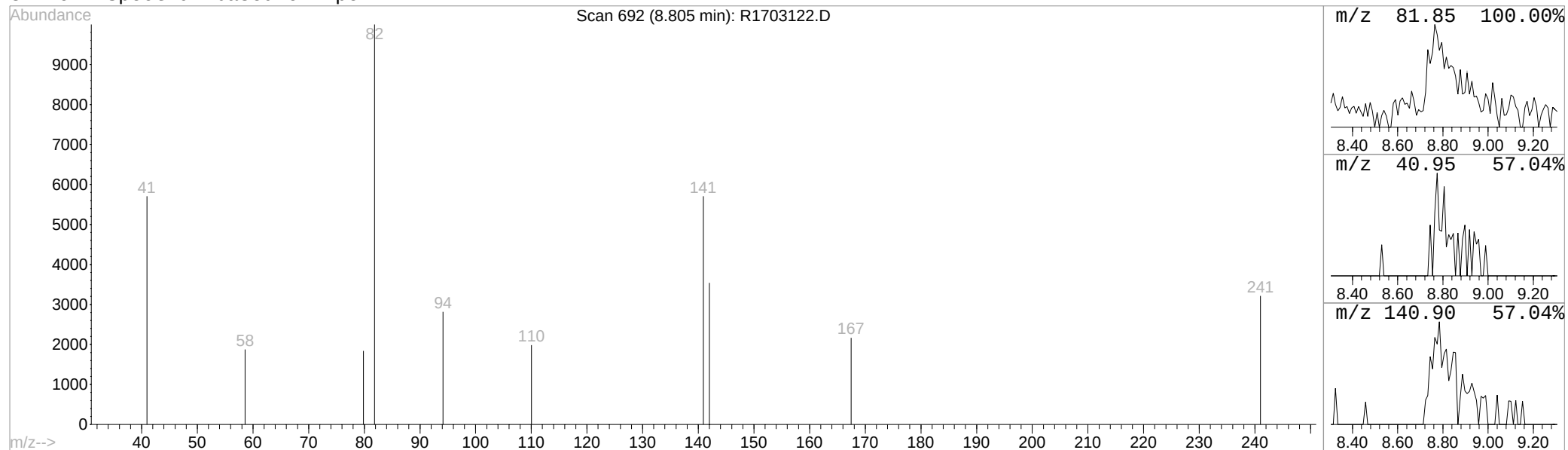
The 3 best hits from each library.

Ref# CAS# Qual

No hits were retrieved.



Unknown Spectrum based on Apex

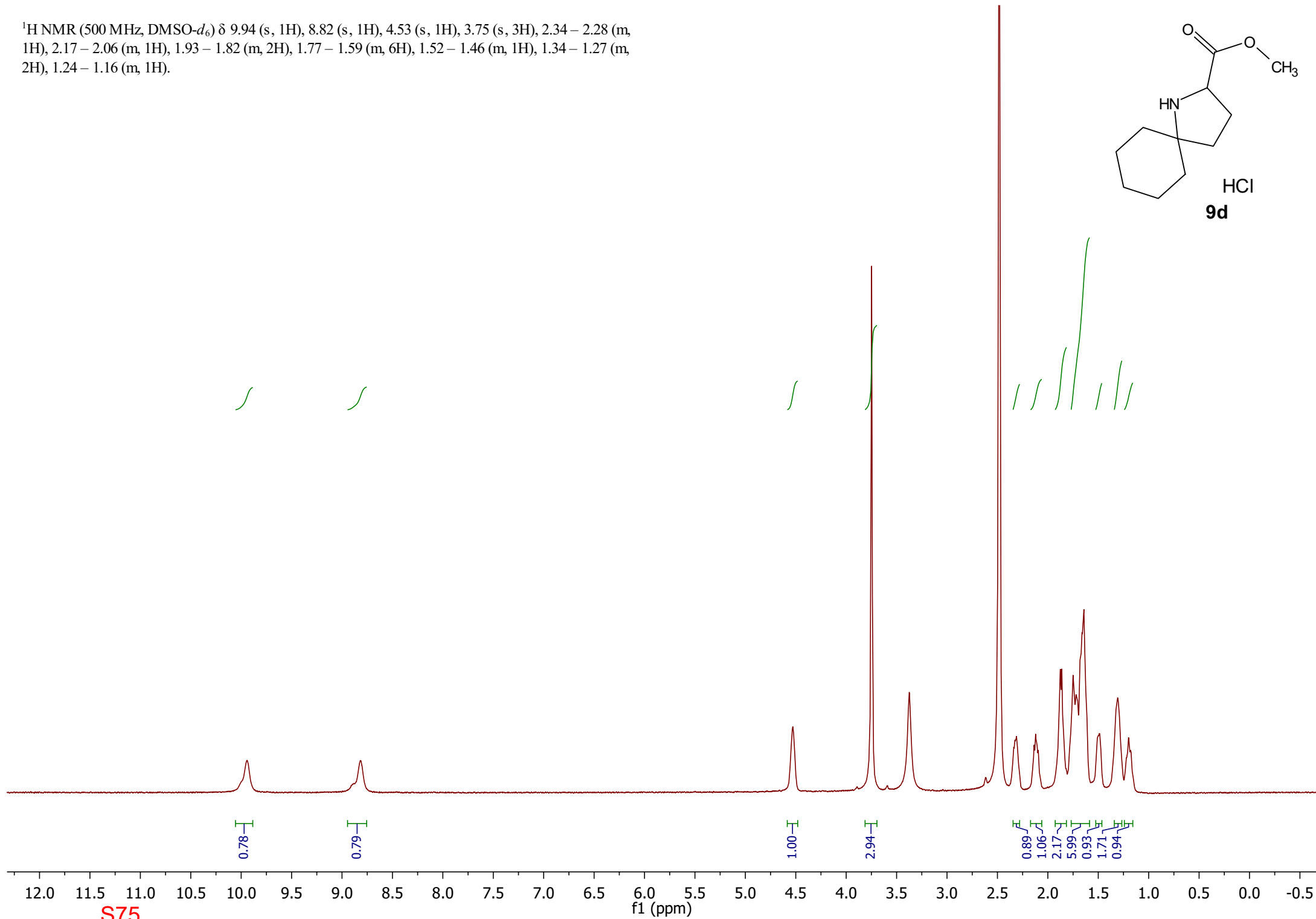
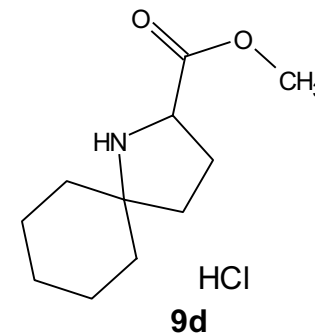


Peak Number: 2 at 8.81 min Area: 58057 Area % 0.32

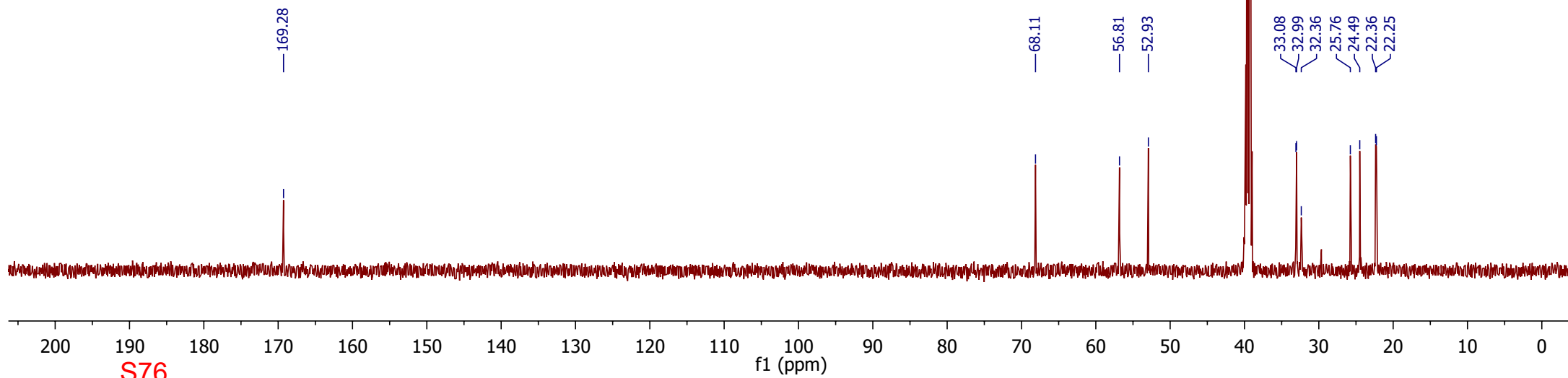
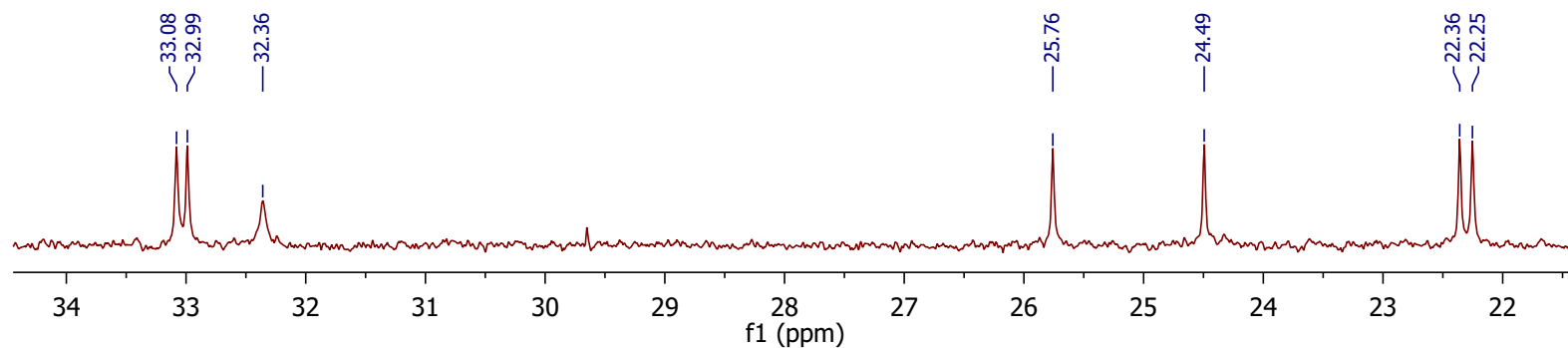
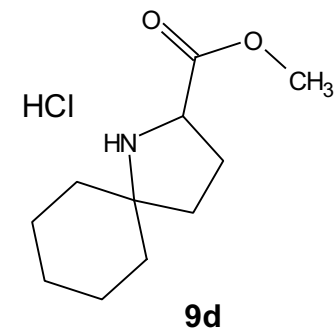
The 3 best hits from each library. Ref# CAS# Qual

No hits were retrieved.

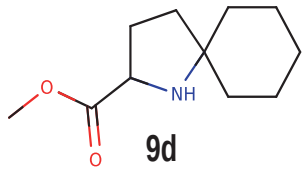
^1H NMR (500 MHz, $\text{DMSO}-d_6$) δ 9.94 (s, 1H), 8.82 (s, 1H), 4.53 (s, 1H), 3.75 (s, 3H), 2.34 – 2.28 (m, 1H), 2.17 – 2.06 (m, 1H), 1.93 – 1.82 (m, 2H), 1.77 – 1.59 (m, 6H), 1.52 – 1.46 (m, 1H), 1.34 – 1.27 (m, 2H), 1.24 – 1.16 (m, 1H).



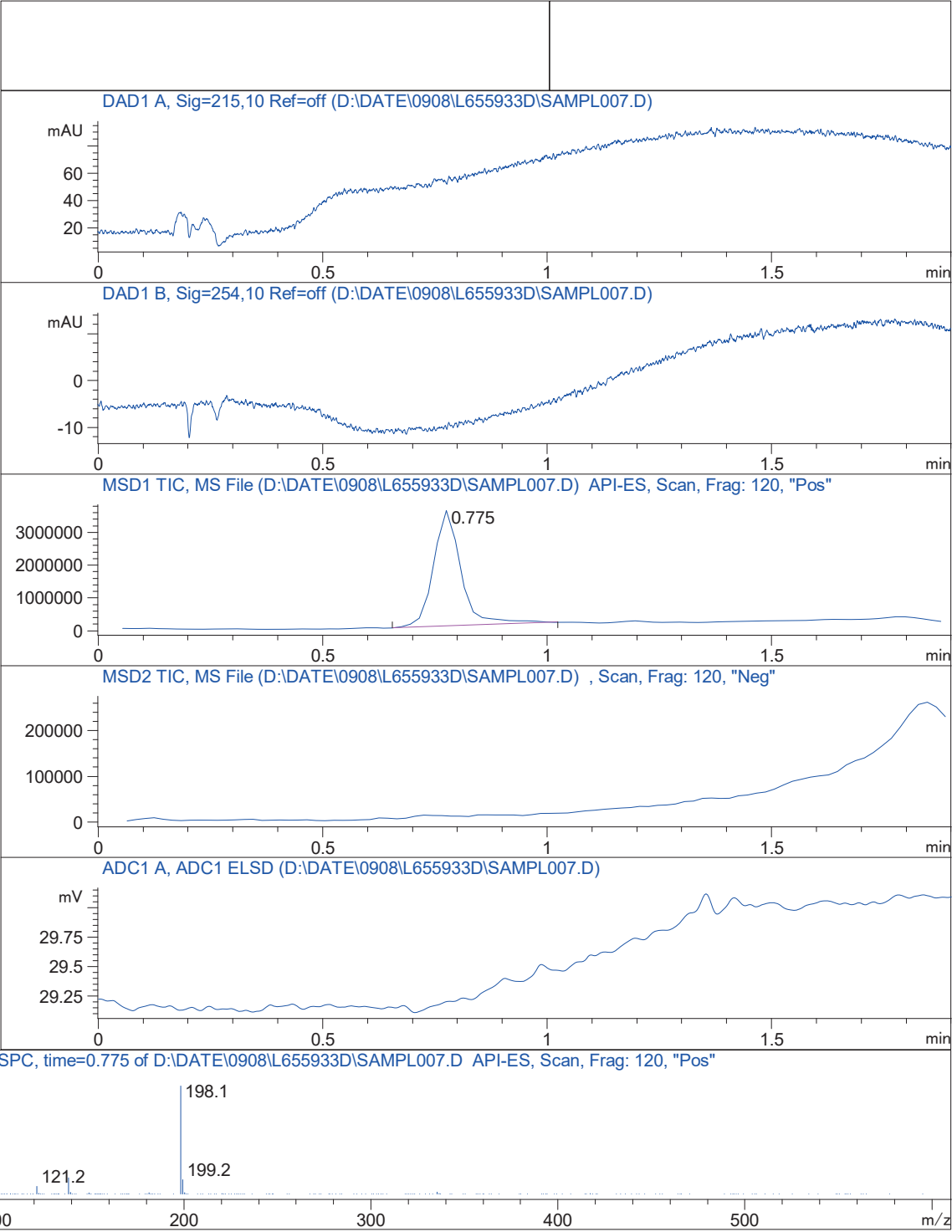
^{13}C NMR (DMSO, 126 MHz) δ 169.28, 68.11, 56.81, 52.93, 33.08, 32.99, 32.36, 25.76, 24.49, 22.36, 22.25.



HCl

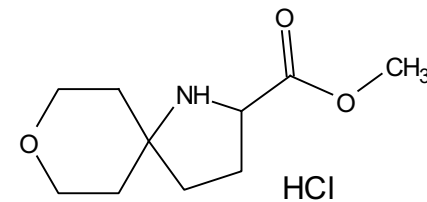


Mol Wt 233.74
Exact Mass 197.17

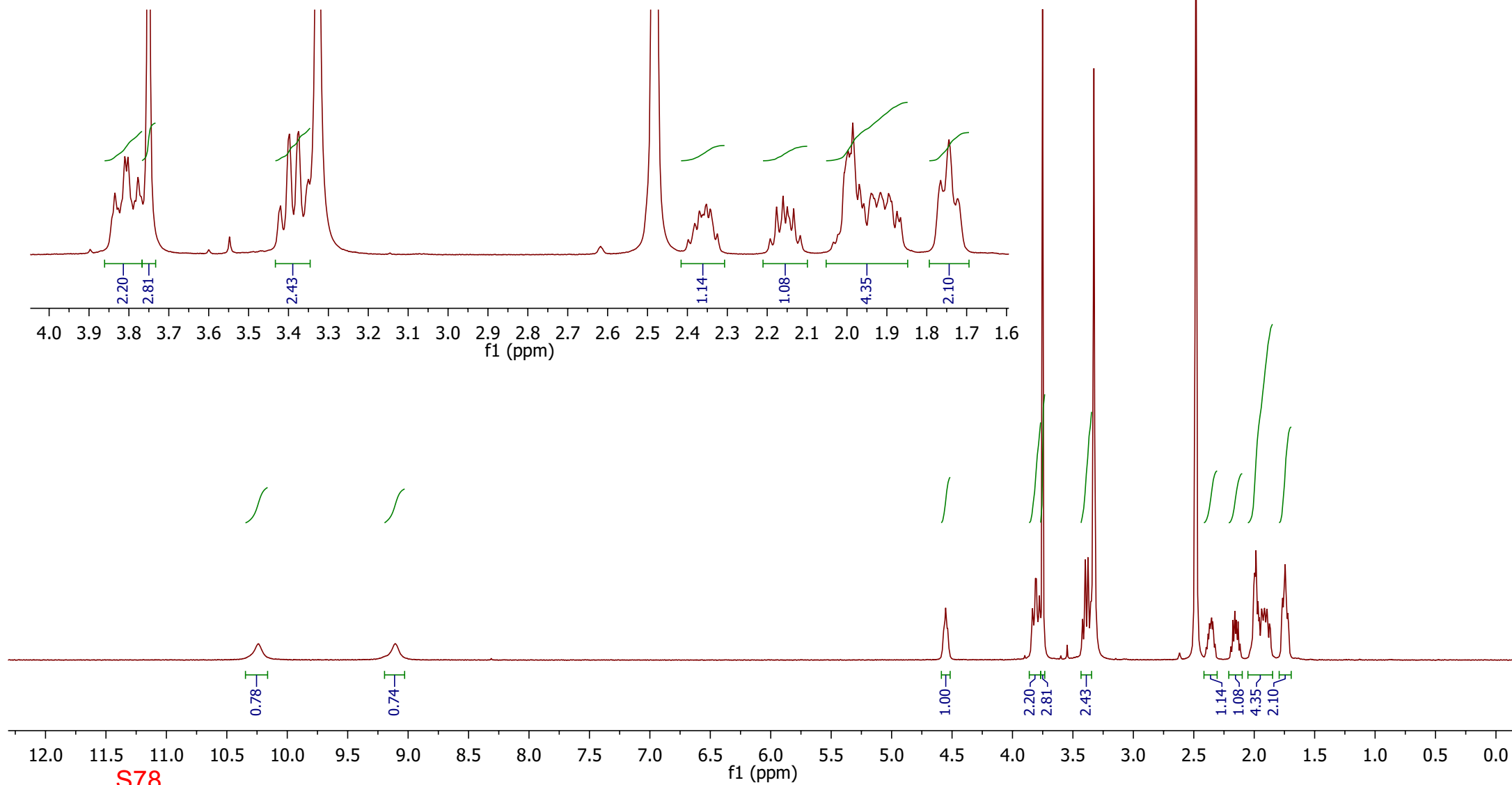


RT 0.775

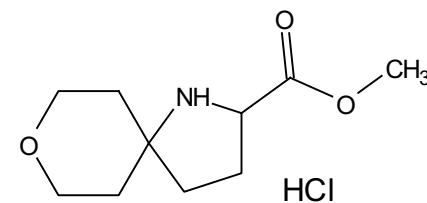
^1H NMR (500 MHz, $\text{DMSO}-d_6$) δ 10.24 (s, 1H), 9.11 (s, 1H), 4.55 (t, $J = 7.3$ Hz, 1H), 3.86 – 3.77 (m, 2H), 3.75 (s, 3H), 3.39 (q, $J = 12.4, 11.7$ Hz, 2H), 2.42 – 2.31 (m, 1H), 2.15 (dq, $J = 13.4, 8.1$ Hz, 1H), 2.05 – 1.85 (m, 4H), 1.79 – 1.69 (m, 2H).



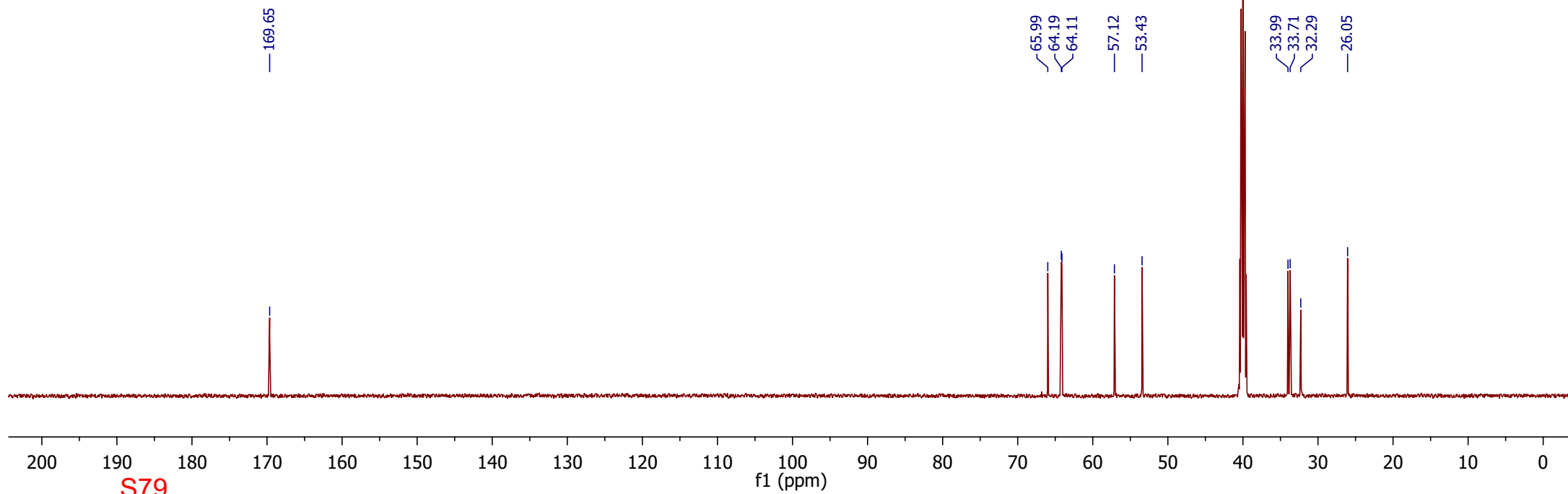
9f



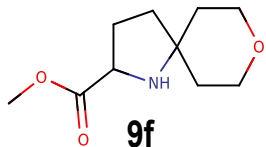
^{13}C NMR (dms o , 151 MHz) δ 169.65, 65.99, 64.19, 64.11, 57.12, 53.43, 33.99, 33.71, 32.29, 26.05.



9f

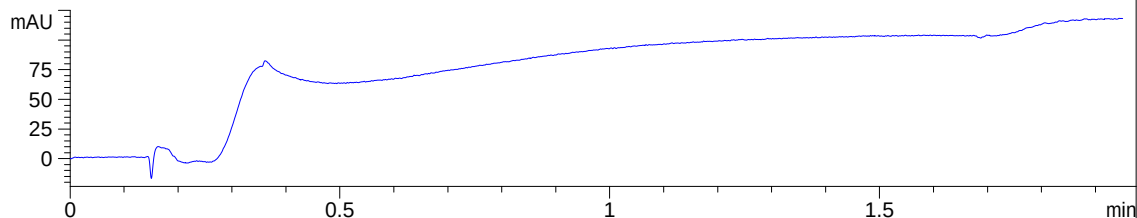


HCl

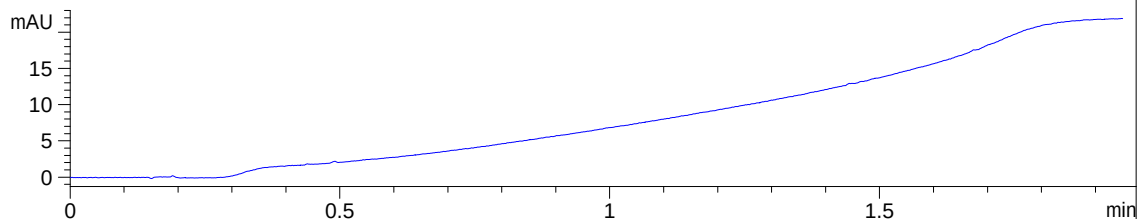


Mol Wt 235.71
Exact Mass 199.14

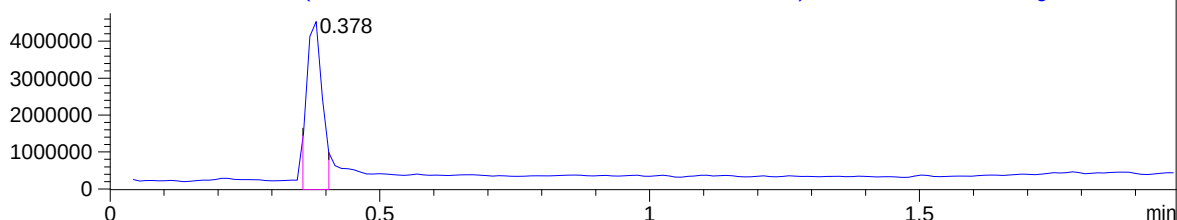
DAD1 A, Sig=215,16 Ref=off (D:\DATE\0710\L633283R\014-D5F-B8-R3625826.D)



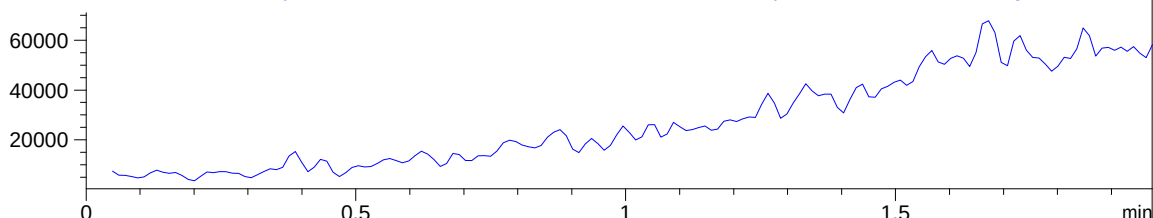
DAD1 B, Sig=254,16 Ref=off (D:\DATE\0710\L633283R\014-D5F-B8-R3625826.D)



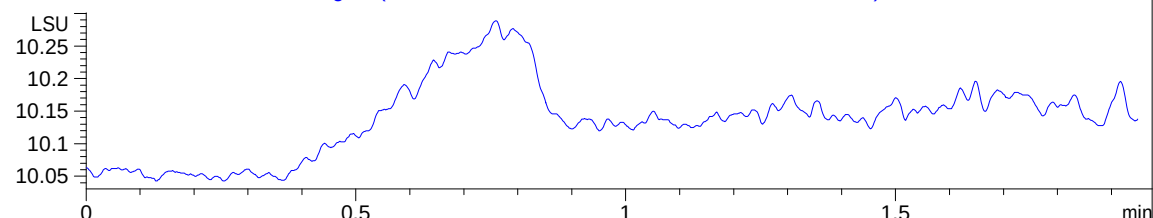
MSD1 TIC, MS File (D:\DATE\0710\L633283R\014-D5F-B8-R3625826.D) ES-API, Fast Scan, Frag: 100, "POS"



MSD2 TIC, MS File (D:\DATE\0710\L633283R\014-D5F-B8-R3625826.D) ES-API, Fast Scan, Frag: 100, "NEG"

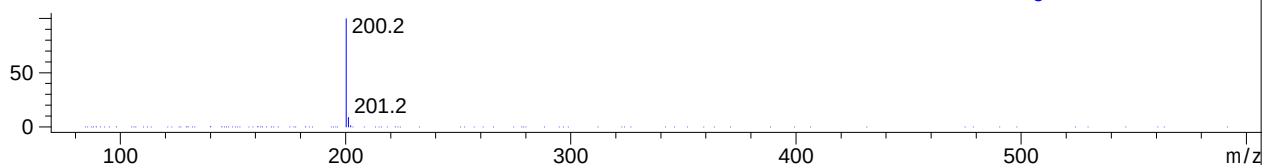


ELS1 A, ELS1A, ELSD Signal (D:\DATE\0710\L633283R\014-D5F-B8-R3625826.D)

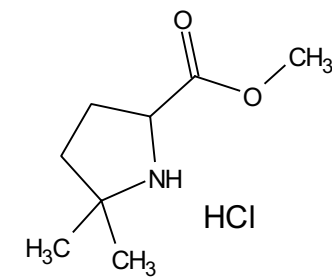


*MSD1 SPC, time=0.382 of D:\DATE\0710\L633283R\014-D5F-B8-R3625826.D ES-API, Fast Scan, Frag: 100, "POS"

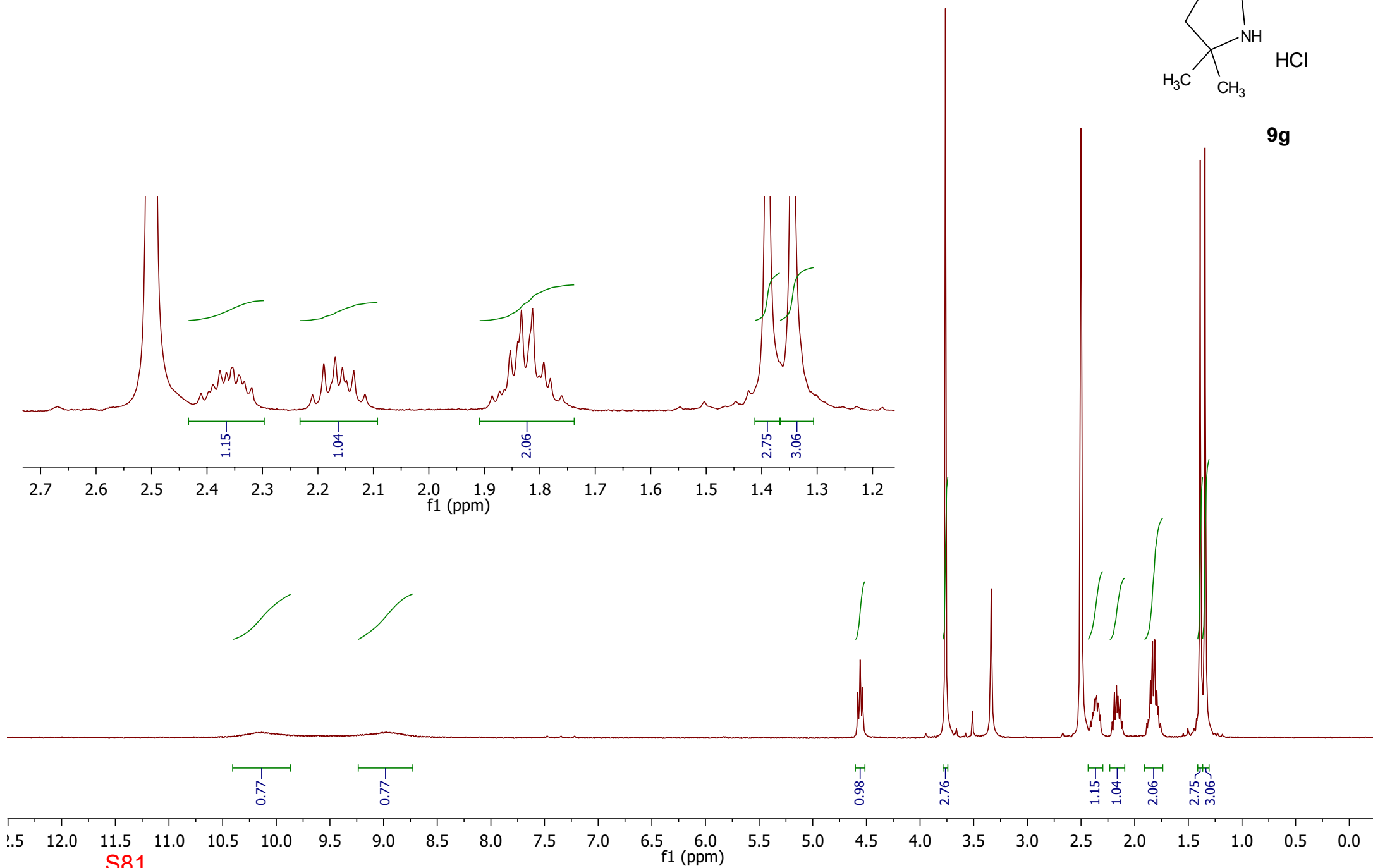
RT 0.378



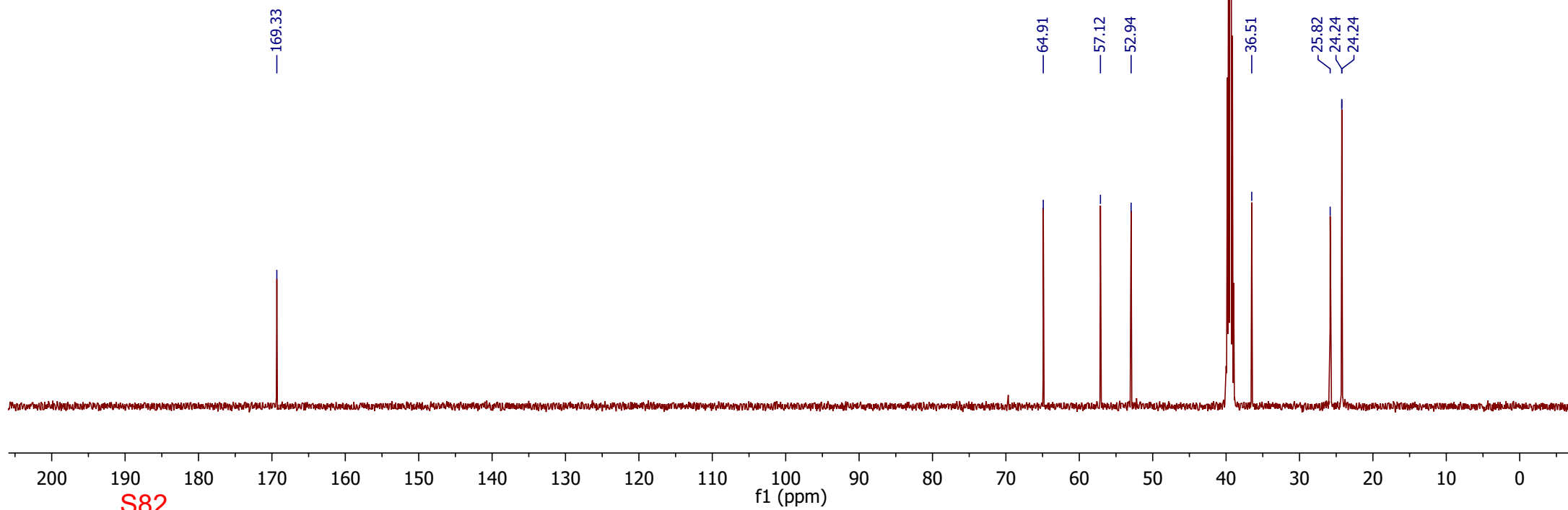
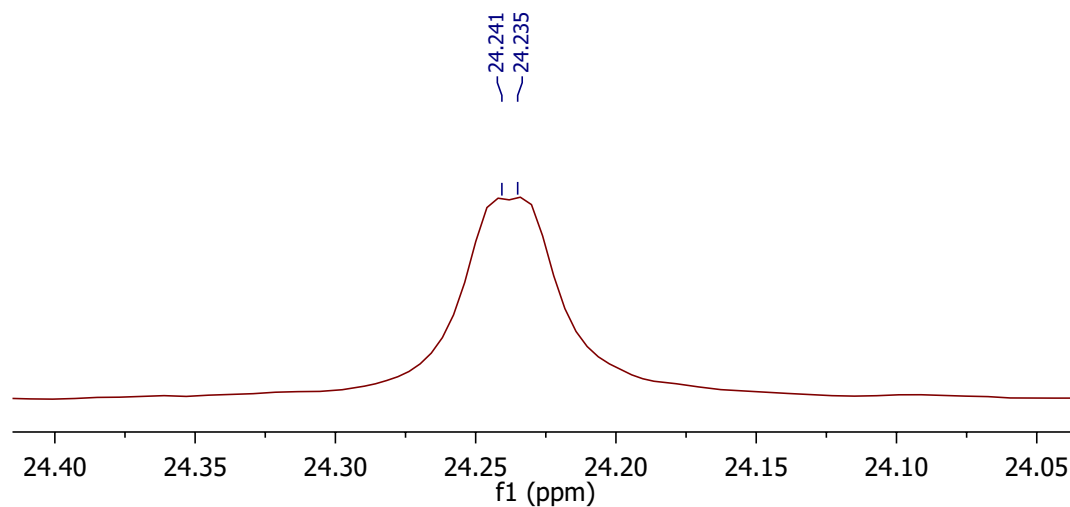
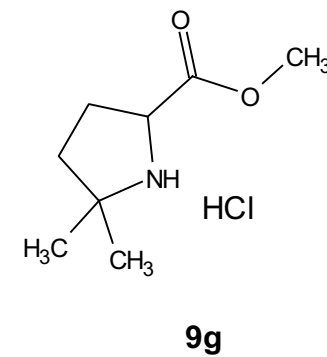
^1H NMR (400 MHz, $\text{DMSO}-d_6$) δ 10.14 (s, 1H), 8.97 (s, 1H), 4.56 (t, $J = 8.8$ Hz, 1H), 3.76 (s, 3H), 2.43 – 2.30 (m, 1H), 2.16 (dq, $J = 13.2, 8.2$ Hz, 1H), 1.91 – 1.74 (m, 2H), 1.39 (s, 3H), 1.34 (s, 3H).

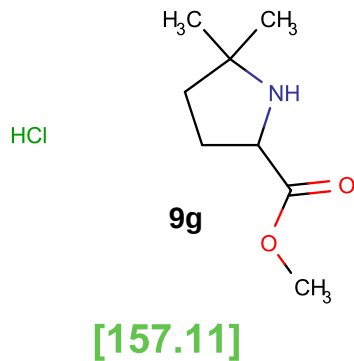


9g

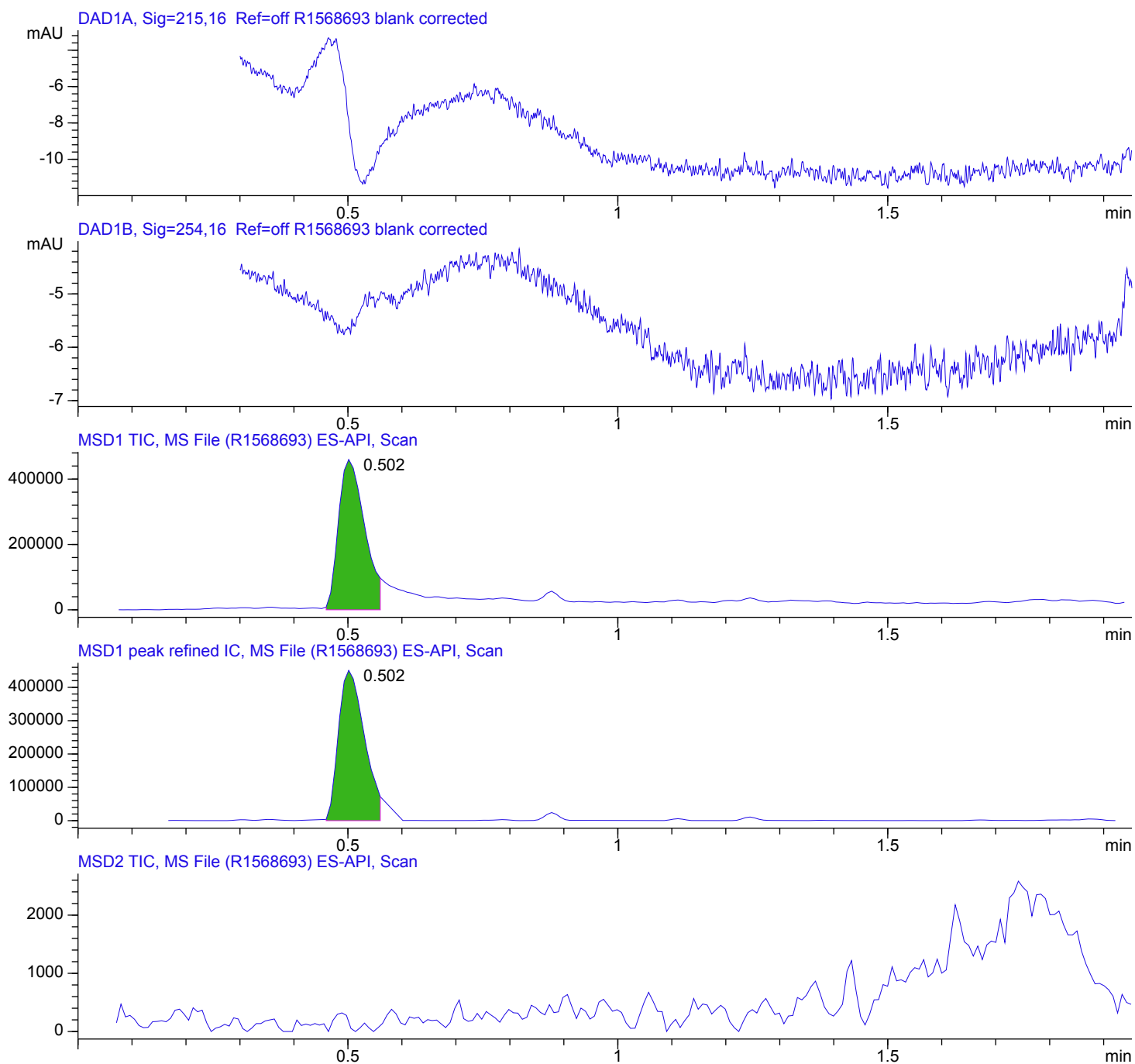


^{13}C NMR (DMSO, 126 MHz) δ 169.33, 64.91, 57.12, 52.94, 36.51, 25.82, 24.24, 24.24.

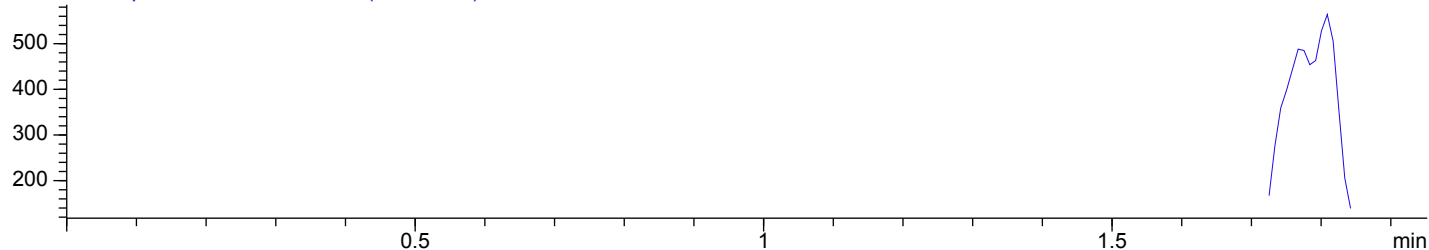




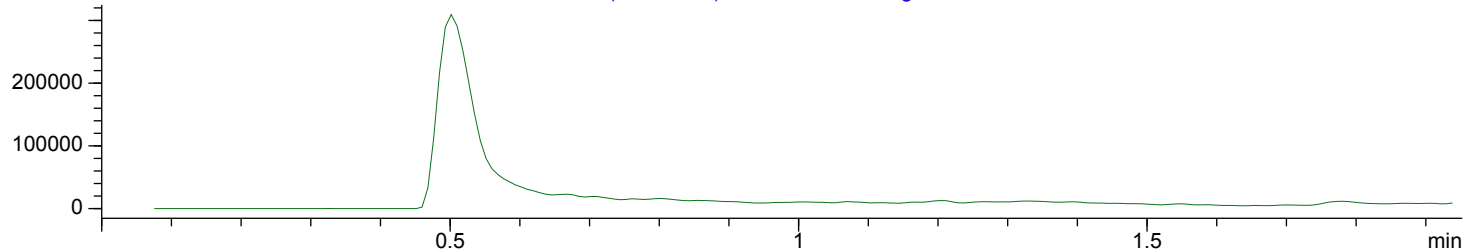
#	RT	DAD1A	DAD1B	MSD1	MSD2	ELSD	MSD1 ions	MSD1 rt	MSD2 ions	MSD2 rt	Info
1	0.492	---	---	100.0%	---	---	158.2(78),98.2(16),141.0(4)	0.502	---	---	P +H+



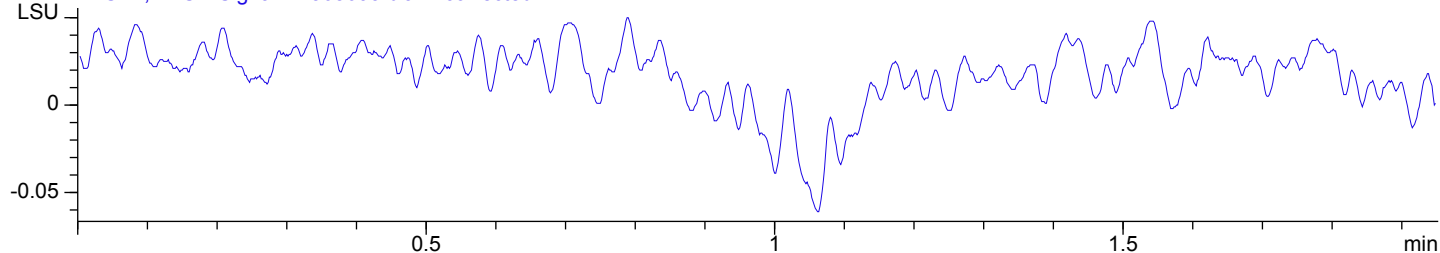
MSD2 peak refined IC, MS File (R1568693) ES-API, Scan



Ion Extract m/z 157.6-158.6 from MSD1 TIC, MS File (R1568693) ES-API, Scan, Frag: 100, "POS"



ELS1A, ELSD Signal R1568693 blank corrected



RT 0.502 *MSD1 SPC, [0.460-0.560] of R1568693 ES-API, Scan, Frag: 100 "POS"

