

Supporting Information 2

Development of Clickable Photoaffinity Ligands for Metabotropic Glutamate Receptor 2 Based on Two Positive Allosteric Modulator Chemotypes

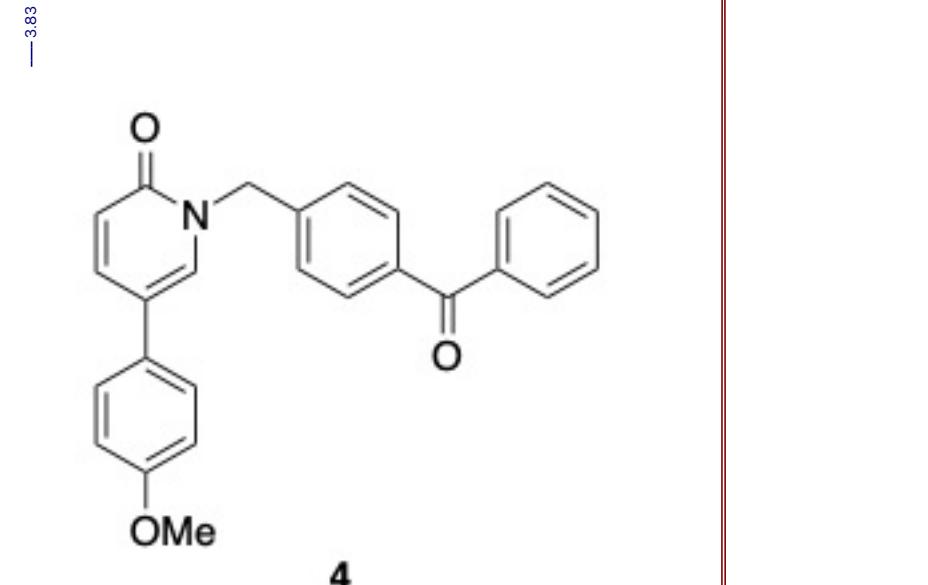
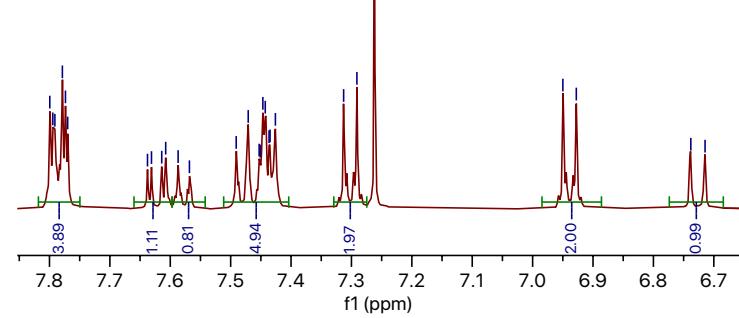
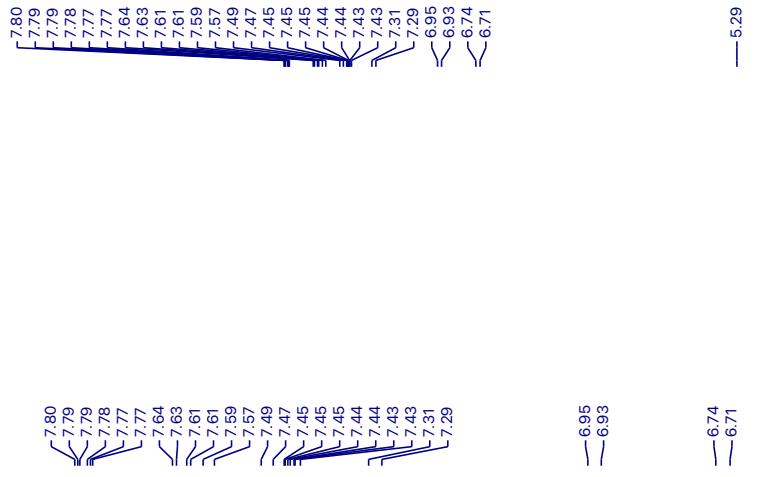
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^a Drug Discovery Biology, Monash Institute of Pharmaceutical Sciences and Department of Pharmacology, Monash University, 399 Royal Parade, Parkville, VIC, Australia

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Catalog of ¹H and ¹³C NMR spectra for all novel compounds synthesized during the course of this work



¹H NMR (400 MHz, CDCl₃ with TMS)

¹H NMR (400 MHz, Chloroform-*d*) δ 7.82 – 7.75 (m, 4H), 7.62 (dd, *J* = 9.4, 2.6 Hz, 1H), 7.57 (d, *J* = 7.5 Hz, 1H), 7.51 – 7.40 (m, 5H), 7.30 (d, *J* = 8.8 Hz, 2H), 6.94 (d, *J* = 8.9 Hz, 2H), 6.73 (d, *J* = 9.4 Hz, 1H), 5.29 (s, 2H), 3.83 (s, 3H).

— 196.1

— 161.8

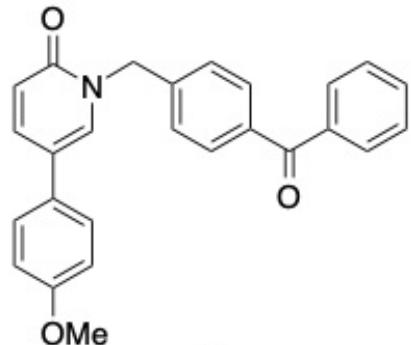
— 159.2

140.9
139.8
137.4
137.3
133.7
132.6
130.7
130.1
128.7
128.3
127.7
127.0
121.3
120.6

— 114.5

— 55.4

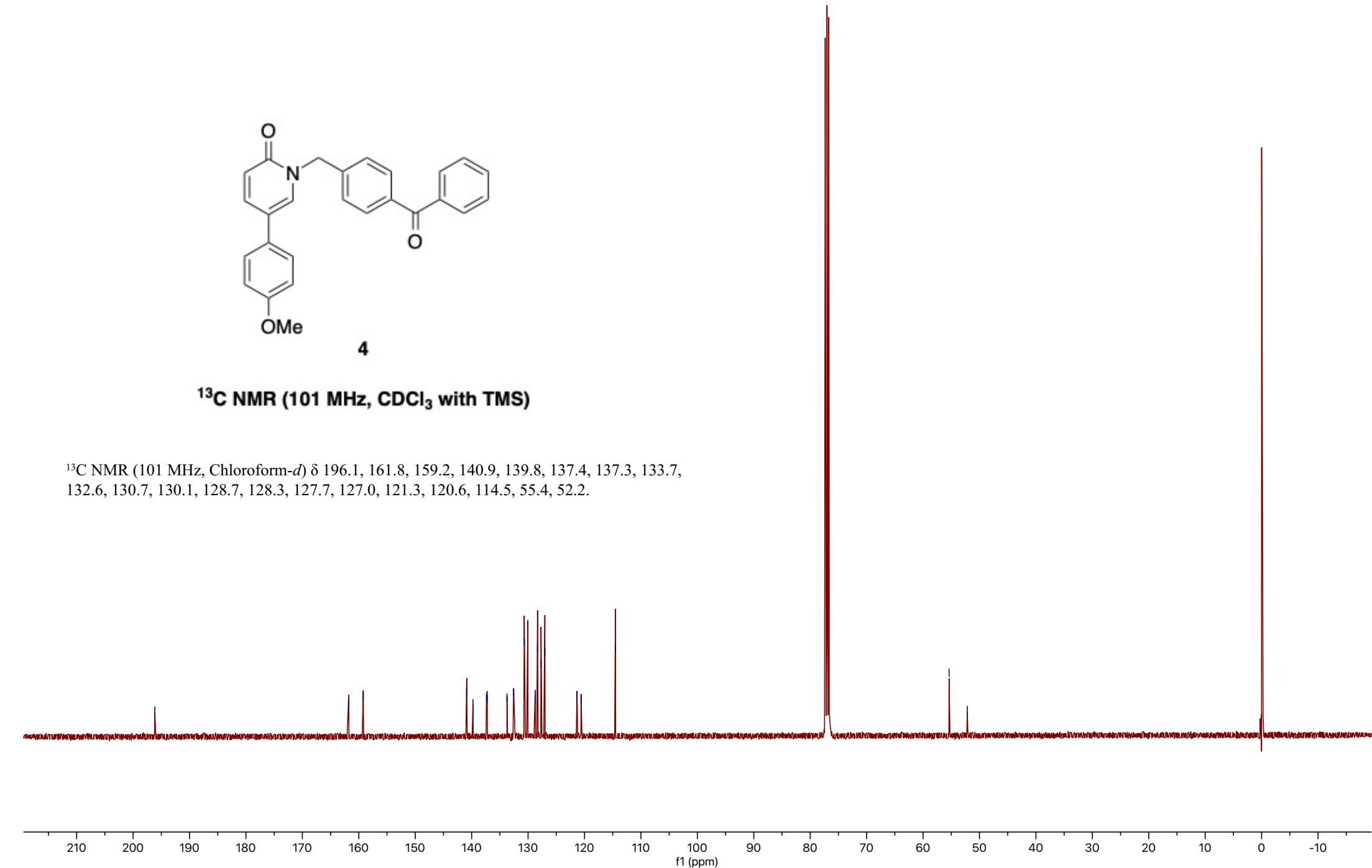
— 52.2

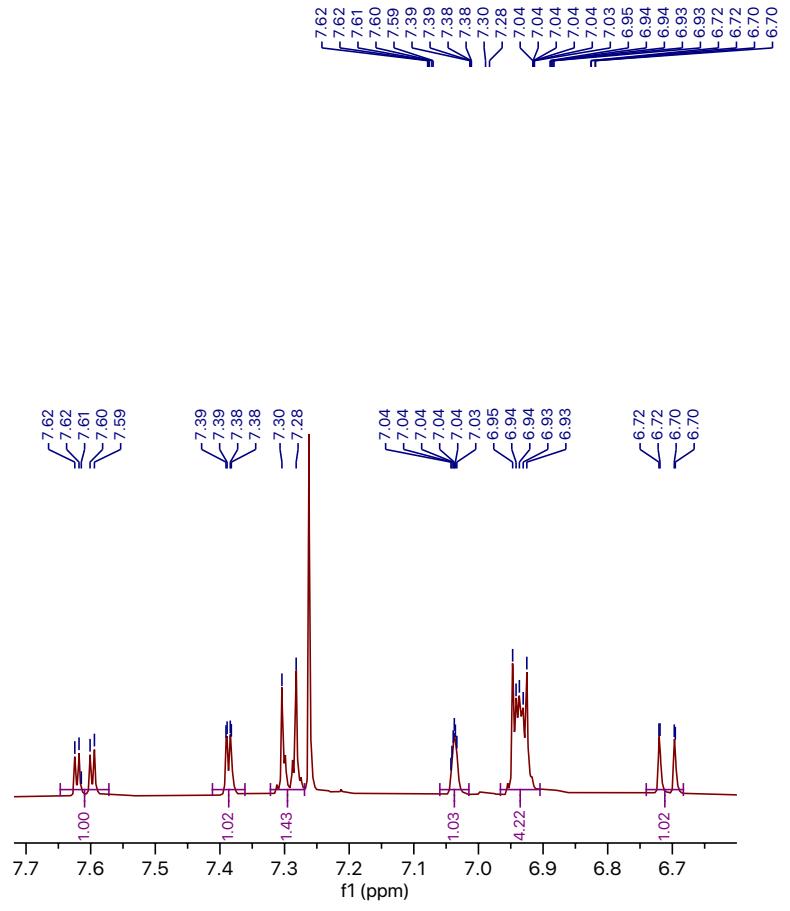


4

¹³C NMR (101 MHz, CDCl₃ with TMS)

¹³C NMR (101 MHz, Chloroform-*d*) δ 196.1, 161.8, 159.2, 140.9, 139.8, 137.4, 137.3, 133.7, 132.6, 130.7, 130.1, 128.7, 128.3, 127.7, 127.0, 121.3, 120.6, 114.5, 55.4, 52.2.

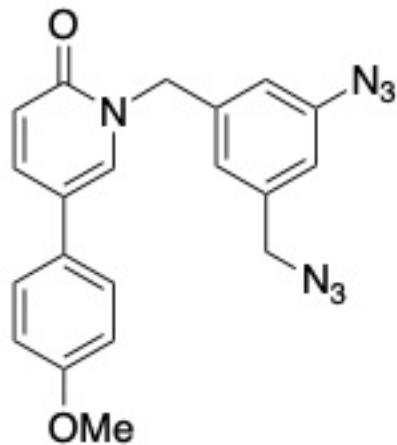




5.18

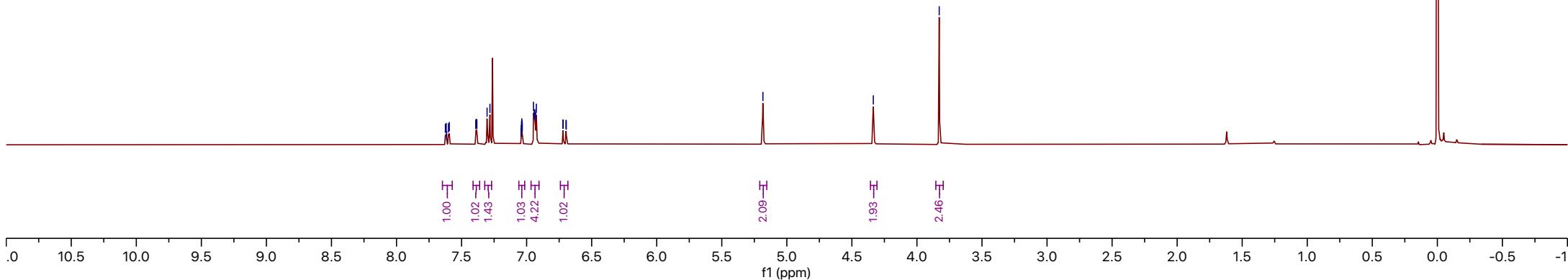
4.34

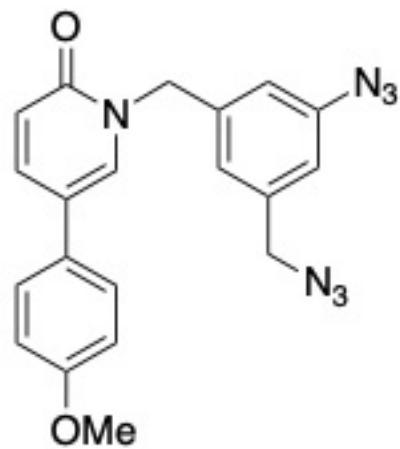
3.83



¹H NMR (400 MHz, CDCl₃ with TMS)

¹H NMR (400 MHz, Chloroform-*d*) δ 7.61 (dd, *J*=9.5, 2.7 Hz, 1H), 7.39 (dd, *J*=2.6, 0.7 Hz, 1H), 7.29 (d, *J*=8.8 Hz, 2H), 7.04 (td, *J*=1.5, 0.8 Hz, 1H), 6.97–6.90 (m, 4H), 6.71 (dd, *J*=9.5, 0.7 Hz, 1H), 5.18 (s, 2H), 4.34 (s, 2H), 3.83 (s, 3H).



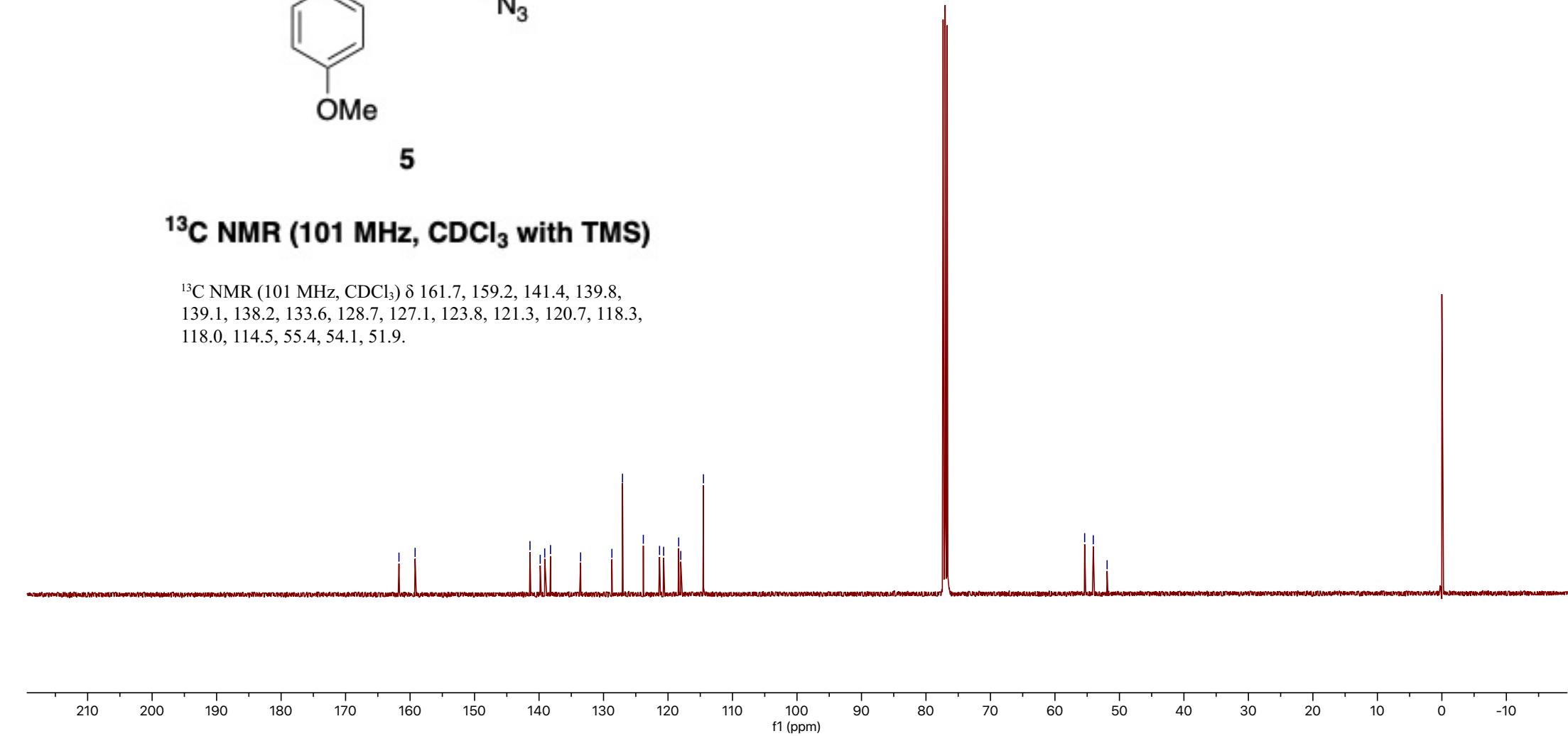


5

¹³C NMR (101 MHz, CDCl₃ with TMS)

¹³C NMR (101 MHz, CDCl₃) δ 161.7, 159.2, 141.4, 139.8, 139.1, 138.2, 133.6, 128.7, 127.1, 123.8, 121.3, 120.7, 118.3, 118.0, 114.5, 55.4, 54.1, 51.9.

161.7
159.2
141.4
139.8
139.1
138.2
133.6
128.7
127.1
123.8
121.3
120.7
118.3
118.0
114.5
55.4
54.1
51.9

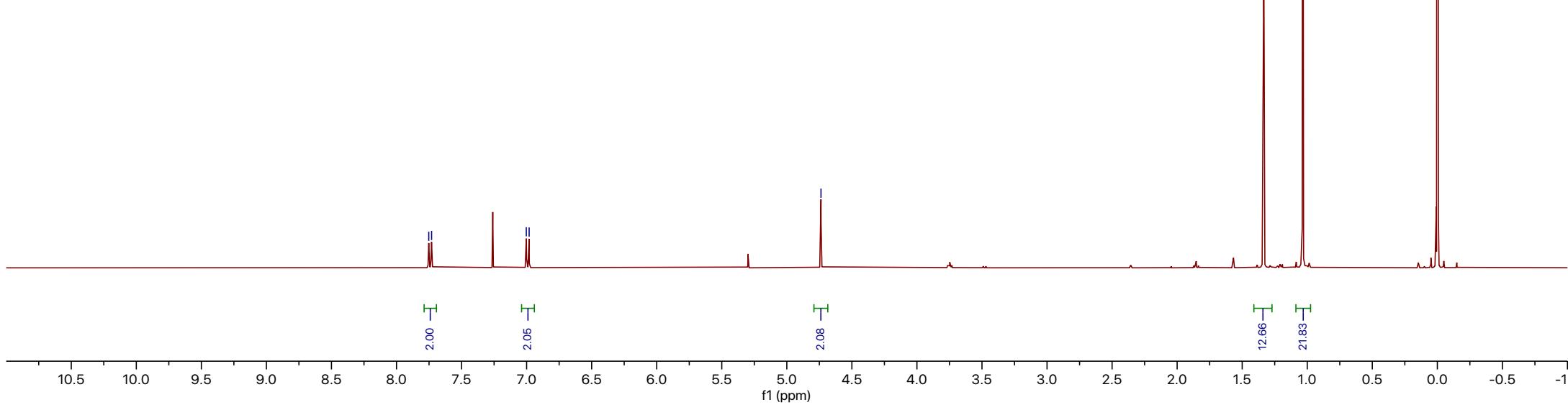


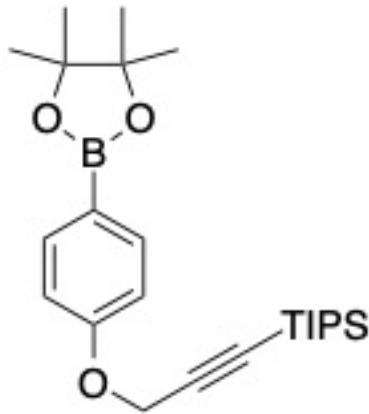


S6

¹H NMR (400 MHz, CDCl₃ with TMS)

¹H NMR (400 MHz, Chloroform-*d*) δ 7.74 (d, *J* = 8.7 Hz, 2H), 6.99 (d, *J* = 8.8 Hz, 2H), 4.74 (s, 2H), 1.34 (s, 12H), 1.03 (s, 21H).

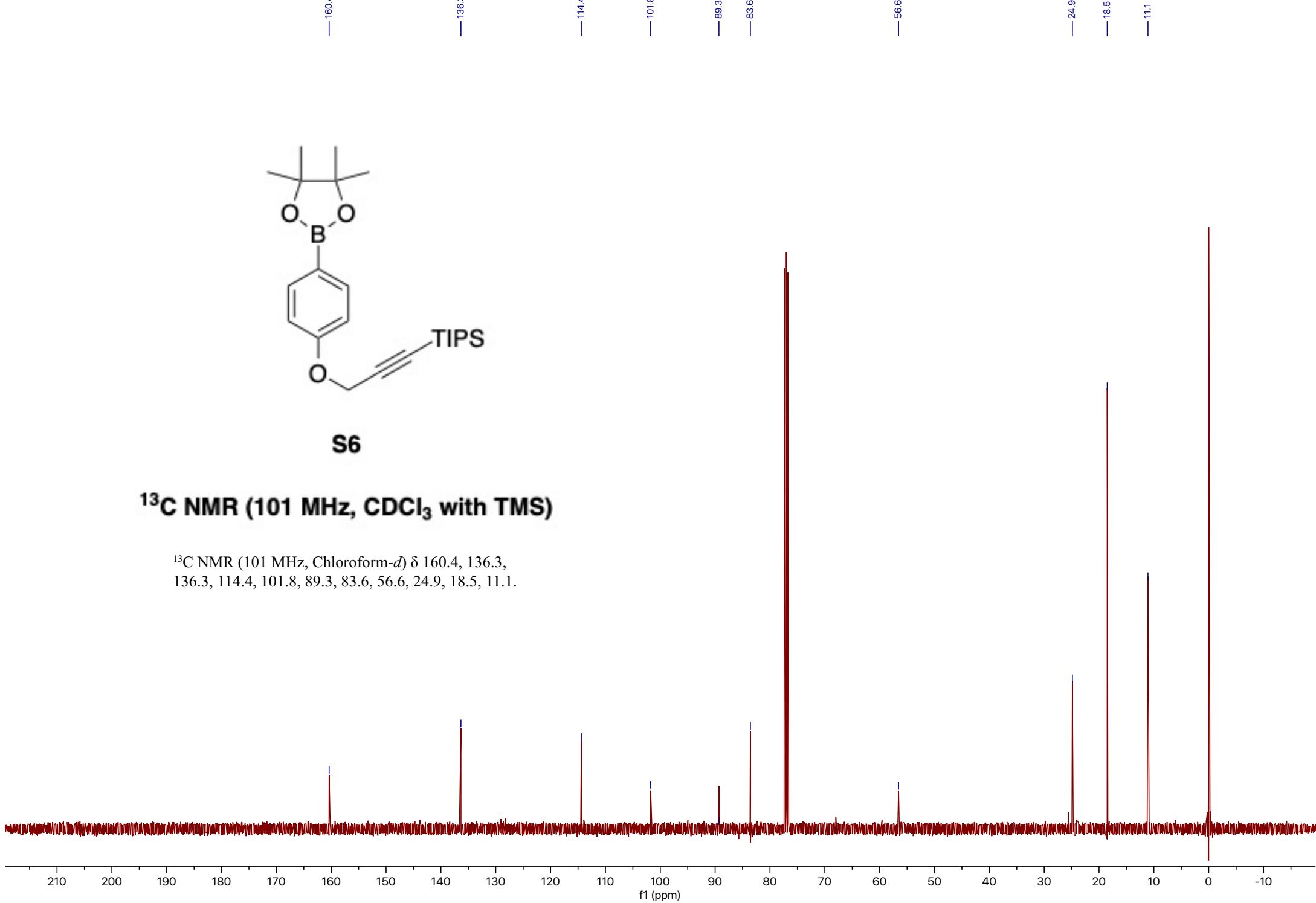


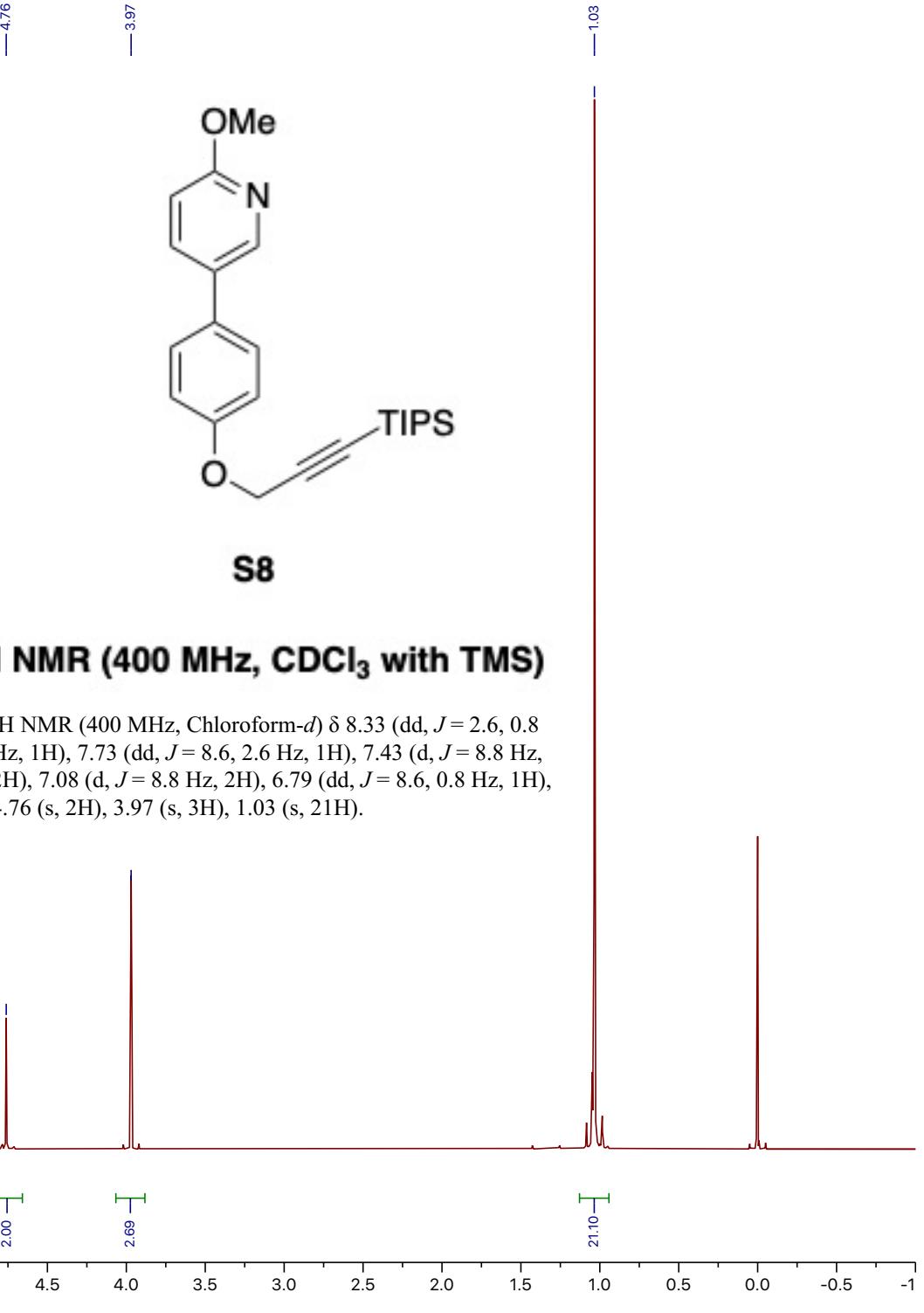
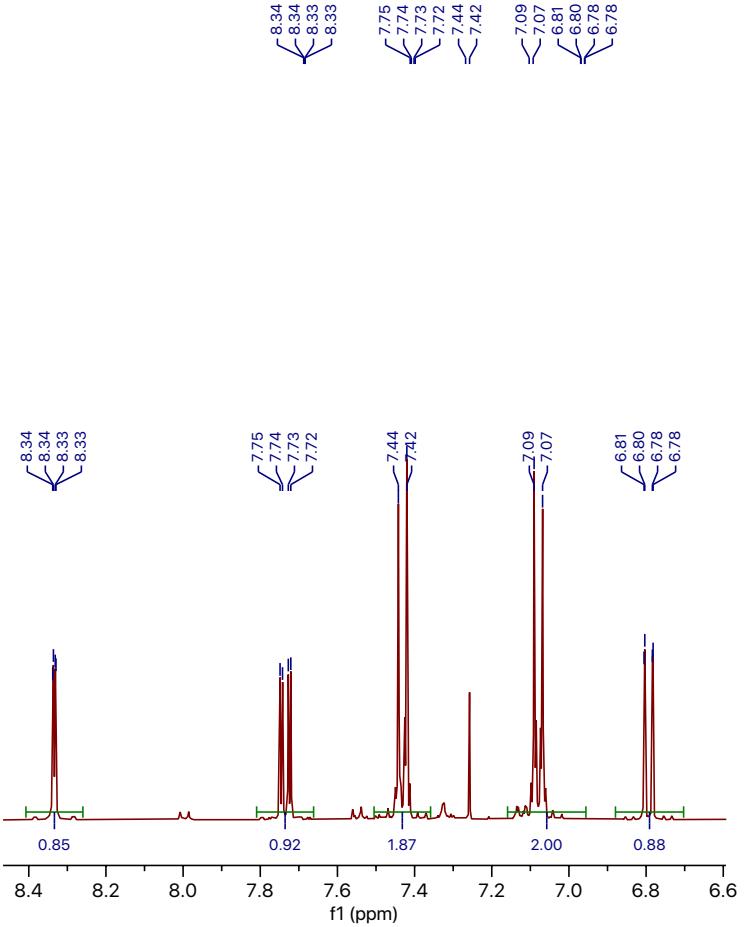


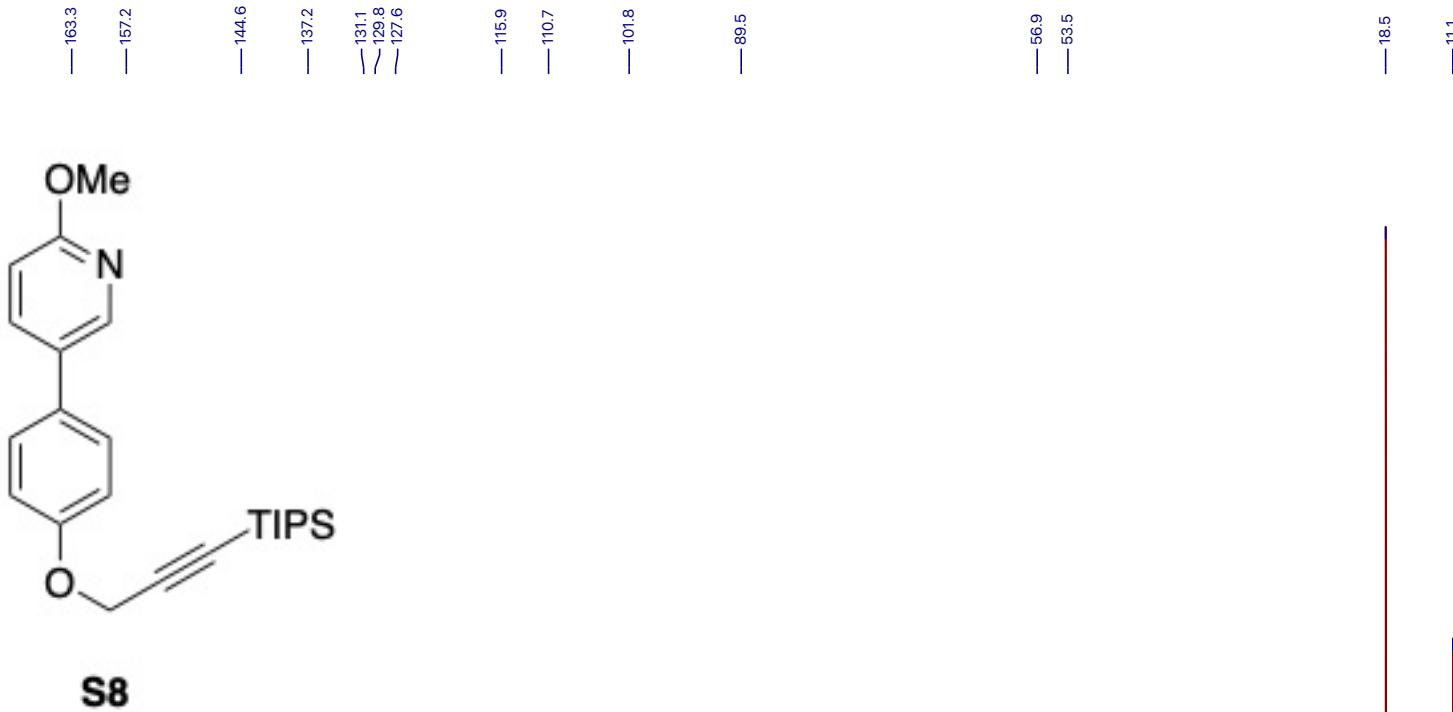
S6

^{13}C NMR (101 MHz, CDCl_3 with TMS)

^{13}C NMR (101 MHz, Chloroform-*d*) δ 160.4, 136.3, 136.3, 114.4, 101.8, 89.3, 83.6, 56.6, 24.9, 18.5, 11.1.







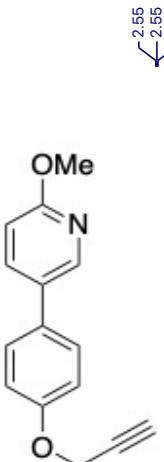
S8

¹³C NMR (101 MHz, CDCl₃ with TMS)

¹³C NMR (101 MHz, Chloroform-*d*) δ 163.2, 157.2, 144.6, 137.2, 131.1, 129.8, 127.6, 115.9, 110.7, 101.8, 89.5, 56.9, 53.5, 18.5, 11.1.

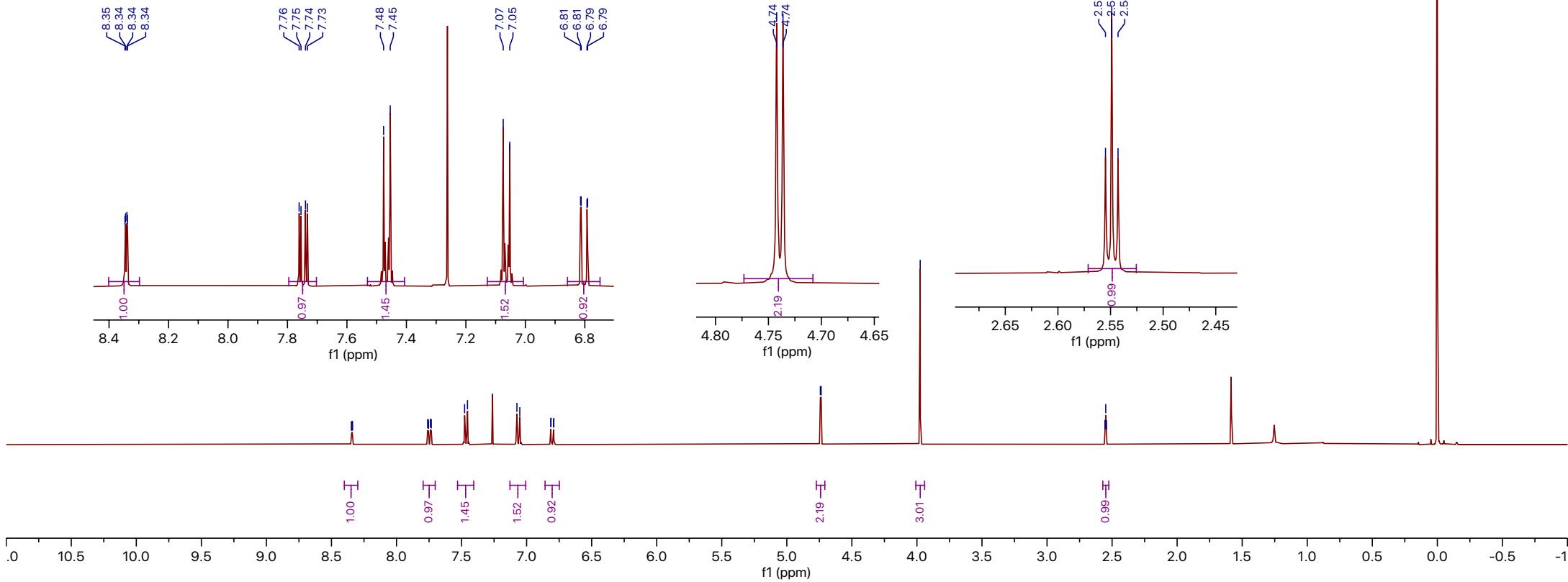


¹H NMR (400 MHz, Chloroform-*d*) δ 8.34 (dd, *J* = 2.5, 0.8 Hz, 1H), 7.75 (dd, *J* = 8.6, 2.6 Hz, 1H), 7.47 (d, *J* = 8.9 Hz, 2H), 7.06 (d, *J* = 8.8 Hz, 2H), 6.80 (dd, *J* = 8.6, 0.8 Hz, 1H), 4.74 (d, *J* = 2.4 Hz, 2H), 3.97 (s, 3H), 2.55 (t, *J* = 2.4 Hz, 1H).

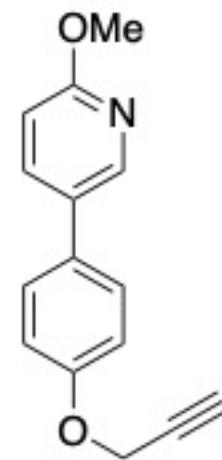


S9

¹H NMR (400 MHz, CDCl₃ with TMS)



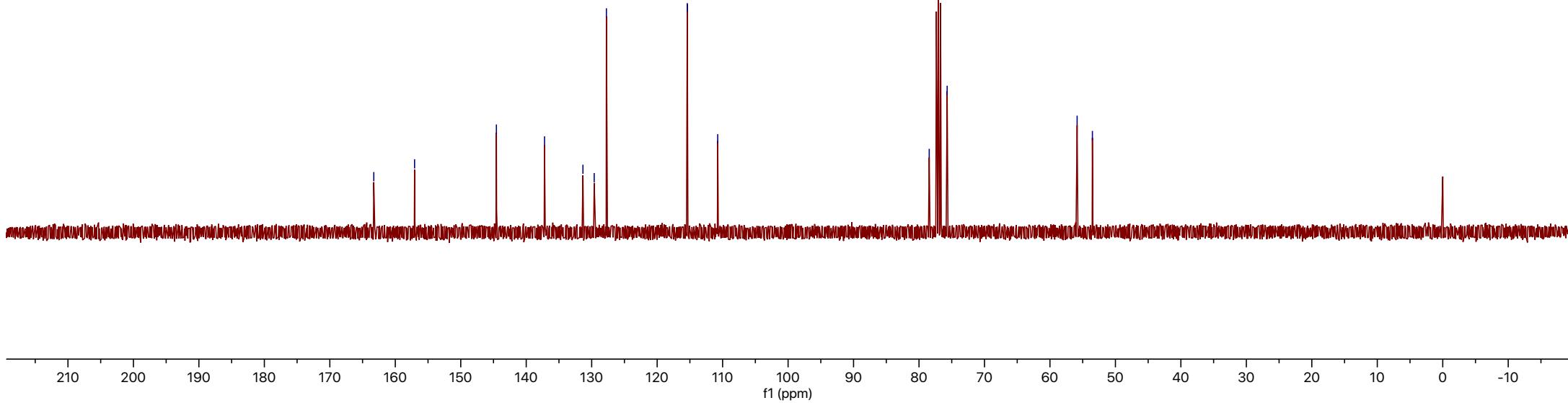
— 163.3
— 157.1
— 144.6
— 137.2
— 131.3
— 129.6
— 127.8
— 115.4
— 110.7
— 78.4
— 75.7
— 55.8
— 53.5

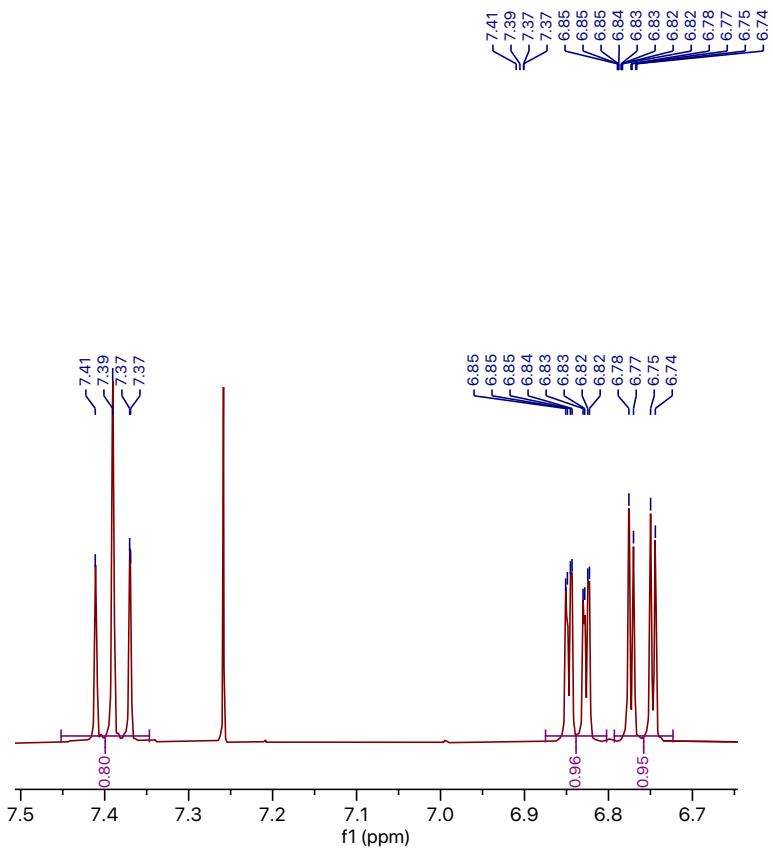


¹³C NMR (101 MHz, CDCl₃) δ 163.3, 157.1, 144.6, 137.2, 131.3, 129.6, 127.8, 115.4, 110.7, 78.4, 75.7, 55.8, 53.5.

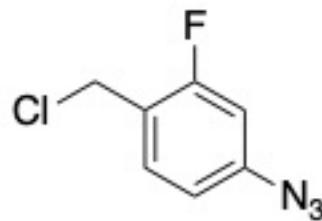
S9

¹³C NMR (101 MHz, CDCl₃ with TMS)





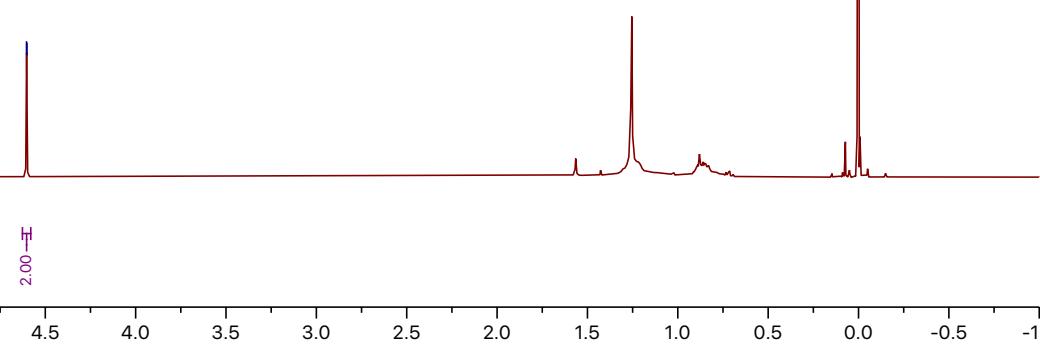
4.60



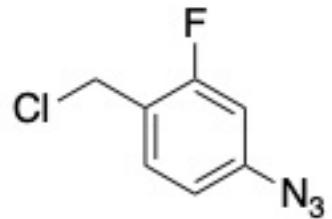
S11

¹H NMR (400 MHz, CDCl_3 with TMS)

¹H NMR (400 MHz, Chloroform-*d*) δ 7.39 (dd, *J* = 8.2, 0.3 Hz, 1H), 6.84 (ddd, *J* = 8.3, 2.3, 0.8 Hz, 1H), 6.76 (dd, *J* = 10.4, 2.2 Hz, 1H), 4.60 (d, *J* = 1.1 Hz, 2H).



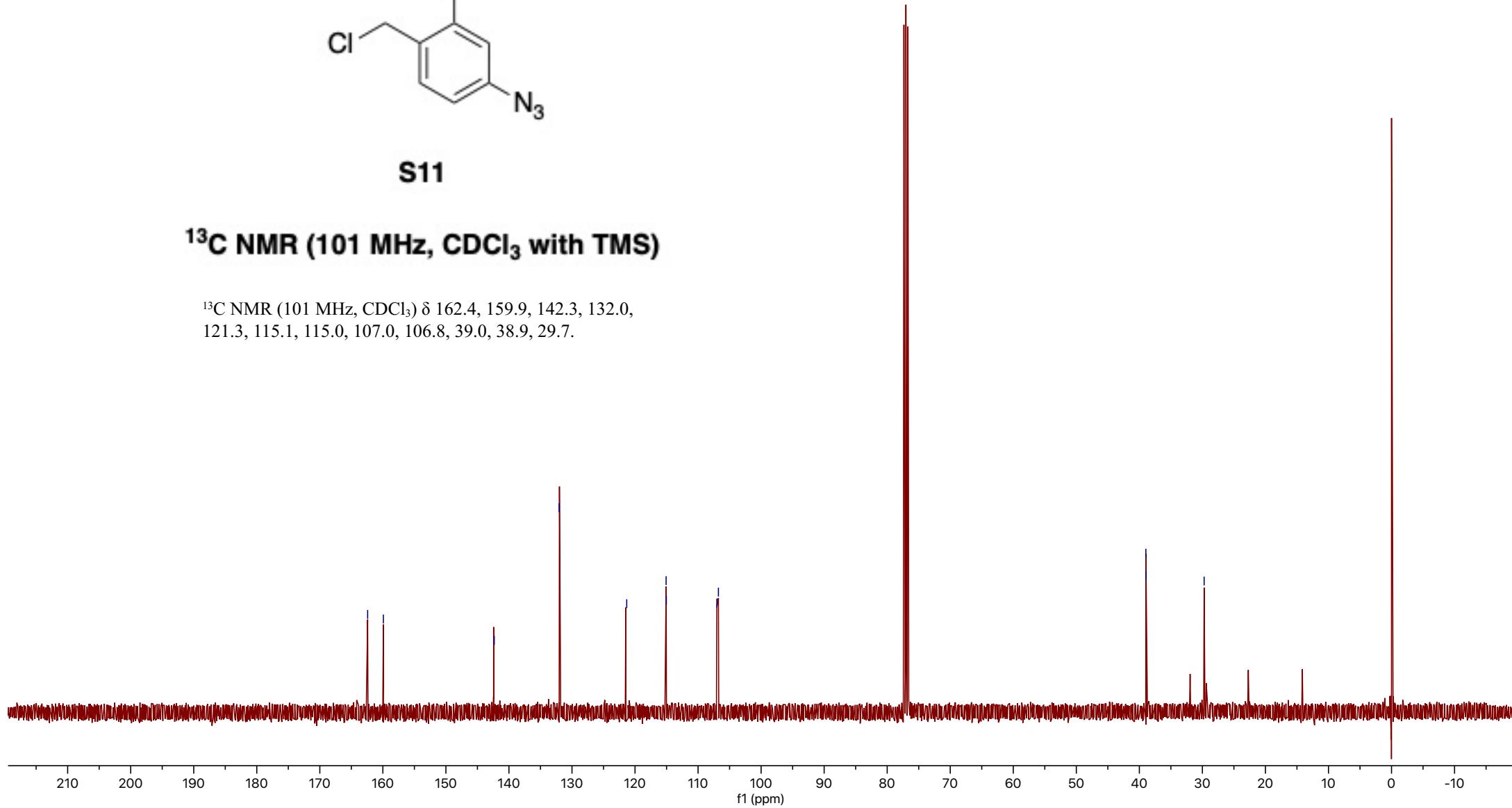
— 162.4
— 159.9
— 142.3
— 132.0
— 121.3
↙ 115.1
↙ 115.0
↙ 107.0
↙ 106.8
↙ 39.0
↙ 38.9
— 29.7

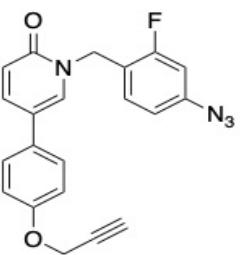


S11

^{13}C NMR (101 MHz, CDCl_3 with TMS)

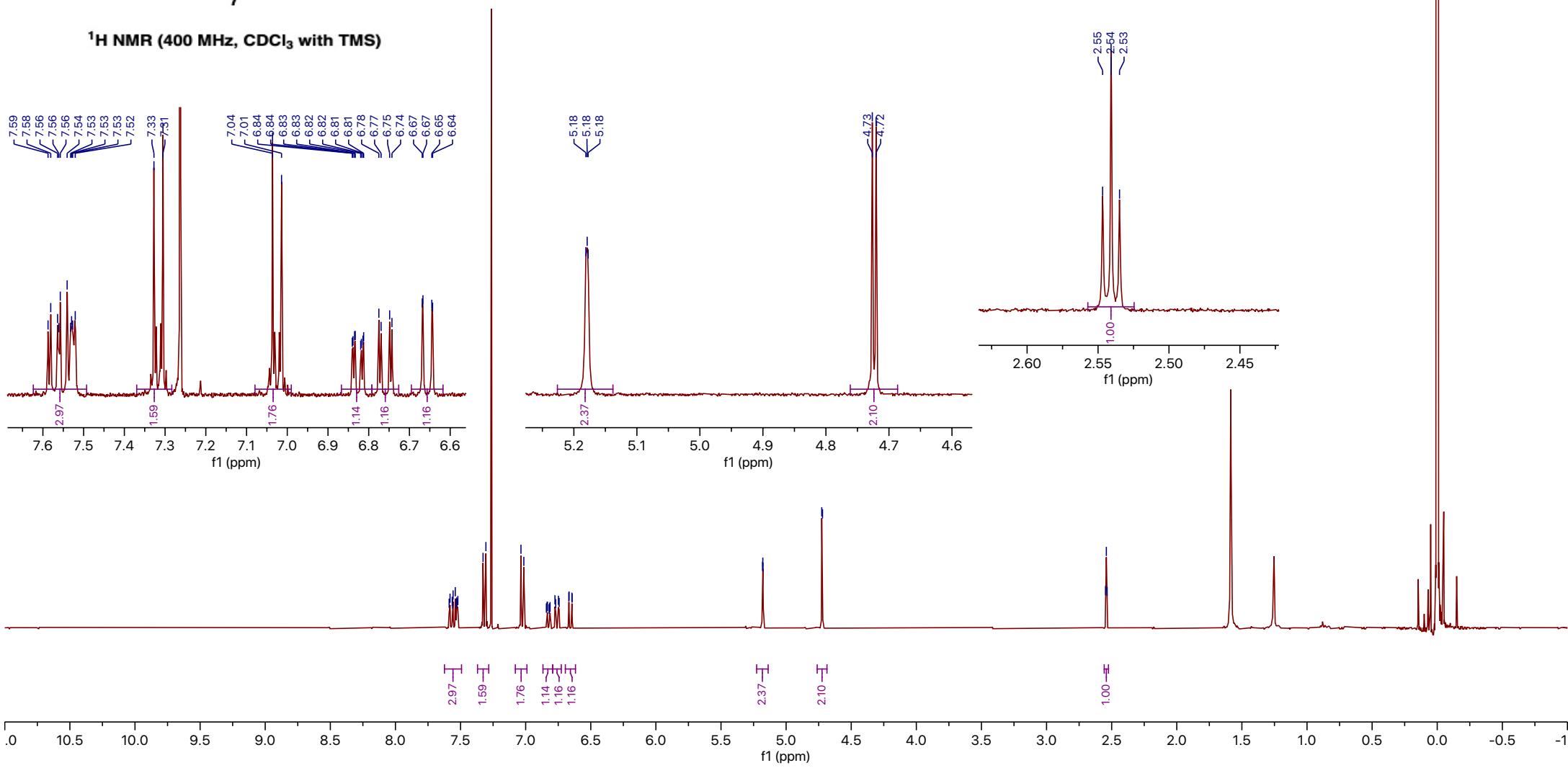
^{13}C NMR (101 MHz, CDCl_3) δ 162.4, 159.9, 142.3, 132.0, 121.3, 115.1, 115.0, 107.0, 106.8, 39.0, 38.9, 29.7.





7

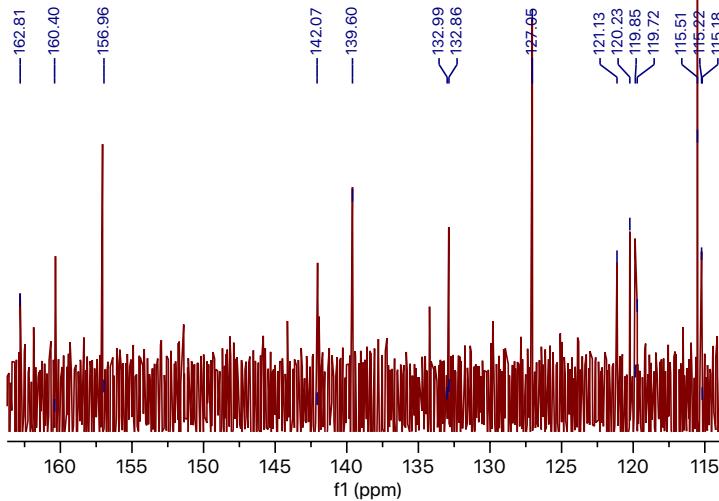
¹H NMR (400 MHz, CDCl₃ with TMS)



¹H NMR (400 MHz, Chloroform-*d*) δ 7.62 – 7.49 (m, 3H), 7.32 (d, *J* = 8.8 Hz, 2H), 7.03 (d, *J* = 8.8 Hz, 2H), 6.83 (ddd, *J* = 8.2, 2.2, 0.7 Hz, 1H), 6.76 (dd, *J* = 10.6, 2.2 Hz, 1H), 6.66 (dd, *J* = 9.4, 0.7 Hz, 1H), 5.23 – 5.14 (m, 2H), 4.72 (d, *J* = 2.4 Hz, 2H), 2.54 (t, *J* = 2.4 Hz, 1H).

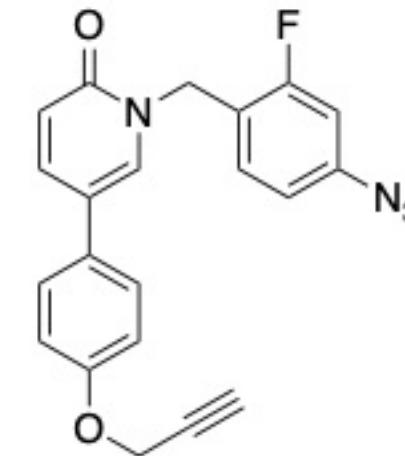
— 162.81
— 160.40
— 156.96

— 142.07
— 139.60
— 132.99
— 132.86
— 127.05
— 127.06



— 78.33
— 75.77

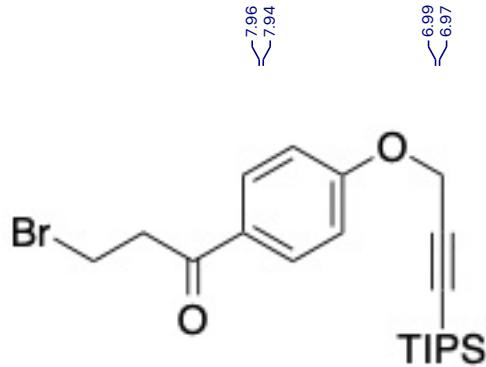
— 55.88



7

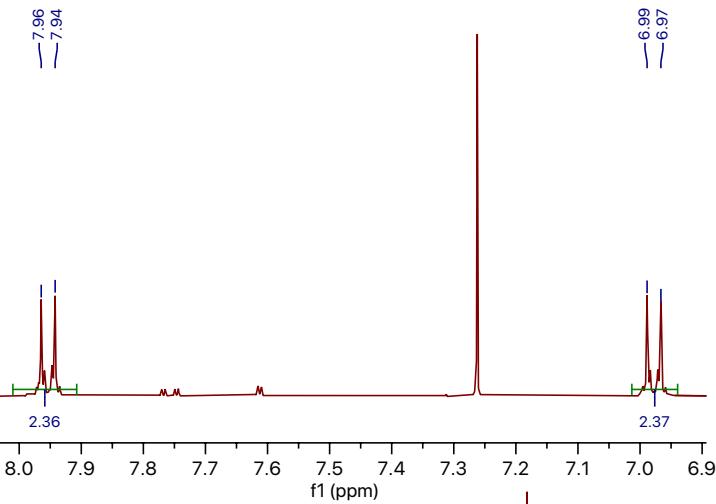
13C NMR (101 MHz, CDCl₃ with TMS)

¹³C NMR (101 MHz, CDCl₃) δ 162.8, 160.4, 157.0, 142.1, 139.6, 133.0, 132.9, 127.0, 121.1, 120.2, 119.9, 119.7, 115.5, 115.2, 115.2, 78.3, 75.8, 55.9, 55.9.

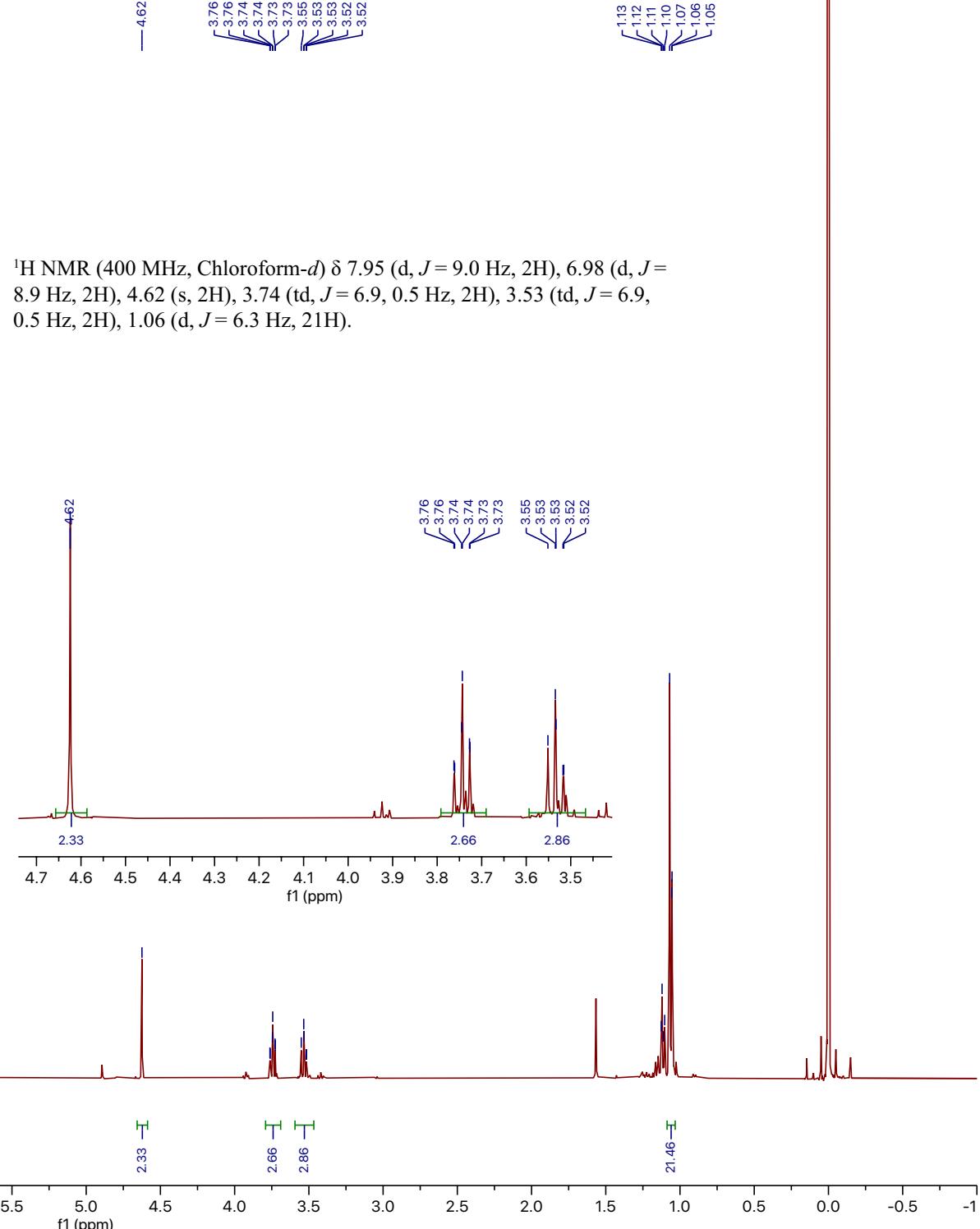


S14

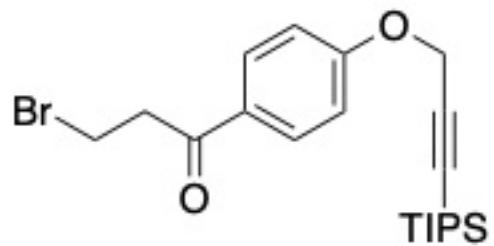
^1H NMR (400 MHz, CDCl_3 with TMS)



^1H NMR (400 MHz, Chloroform-*d*) δ 7.95 (d, $J = 9.0$ Hz, 2H), 6.98 (d, $J = 8.9$ Hz, 2H), 4.62 (s, 2H), 3.74 (td, $J = 6.9, 0.5$ Hz, 2H), 3.53 (td, $J = 6.9, 0.5$ Hz, 2H), 1.06 (d, $J = 6.3$ Hz, 21H).



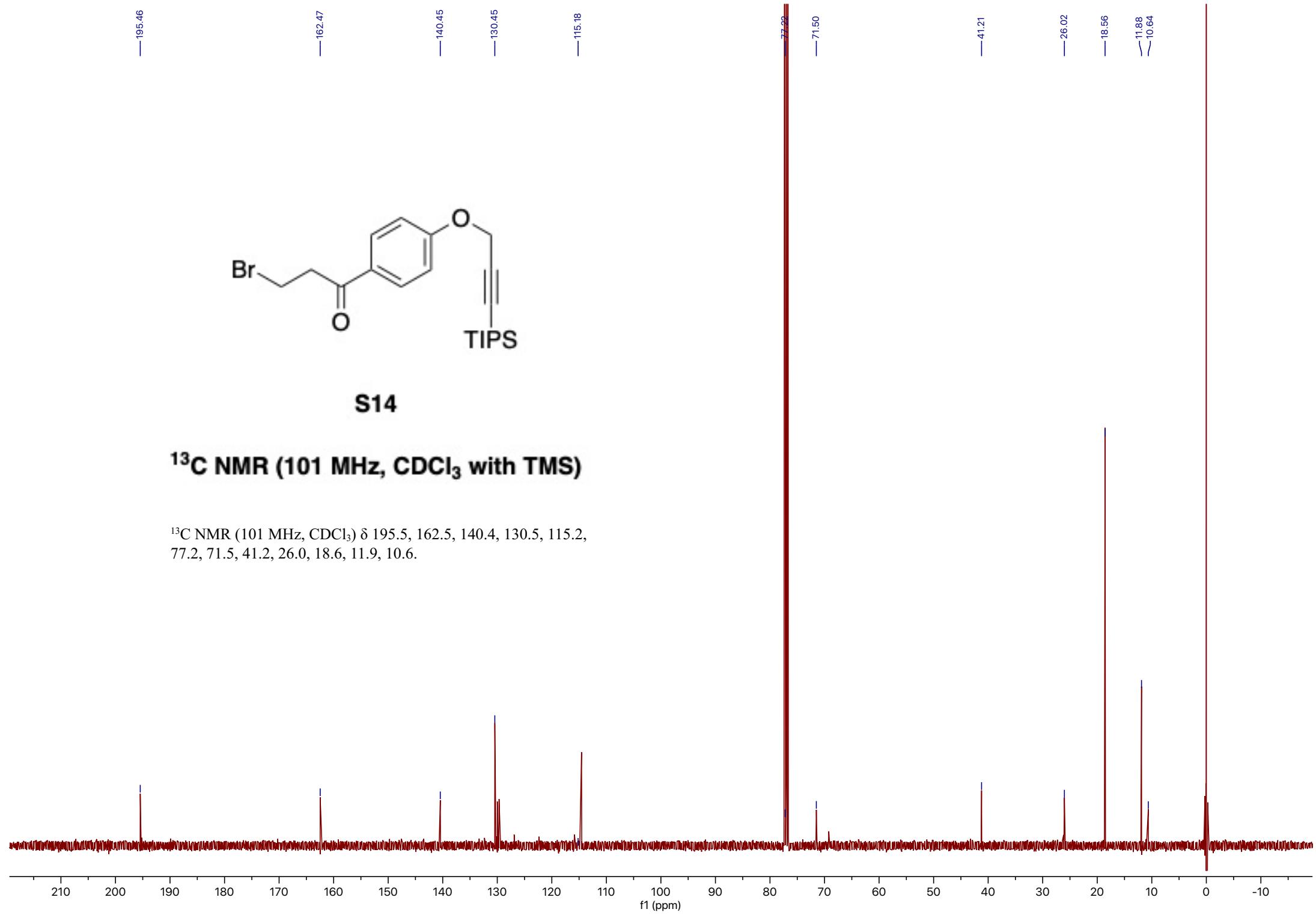
— 195.46
— 162.47
— 140.45
— 130.45
— 115.18
— 77.22
— 71.50
— 41.21
— 26.02
— 18.56
— 11.88
— 10.64

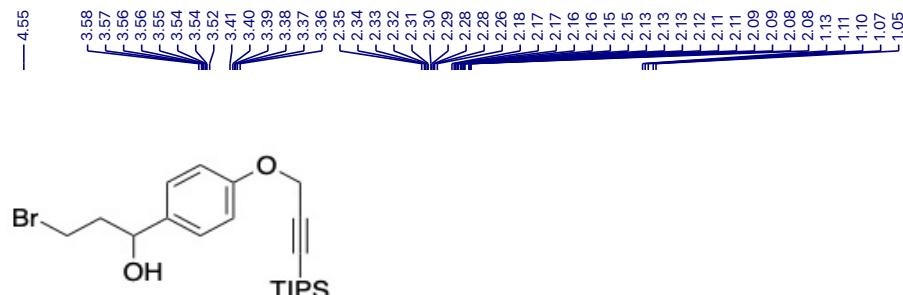
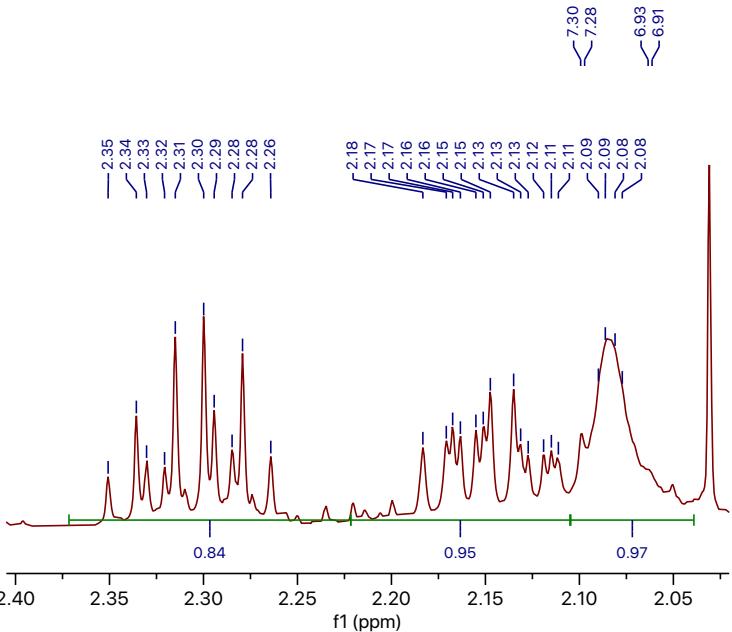


S14

¹³C NMR (101 MHz, CDCl₃ with TMS)

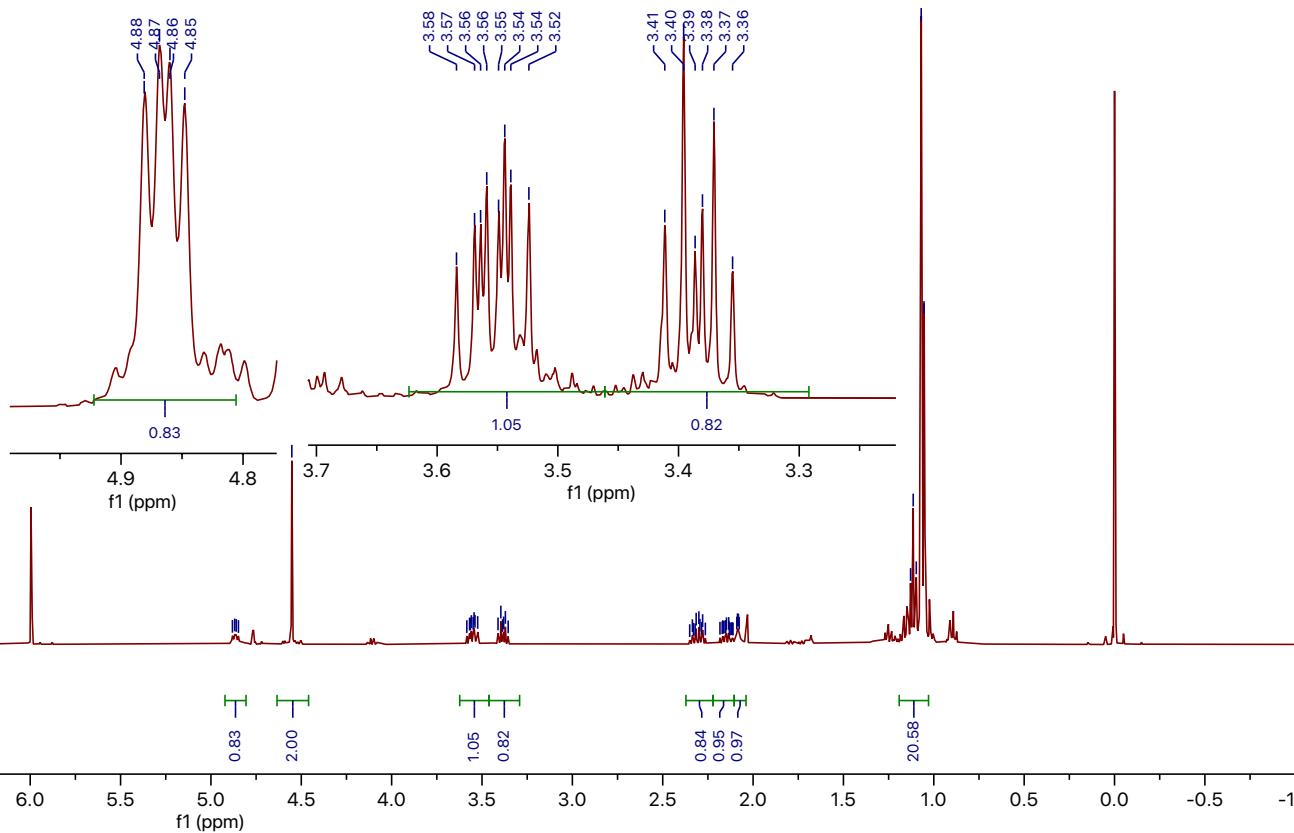
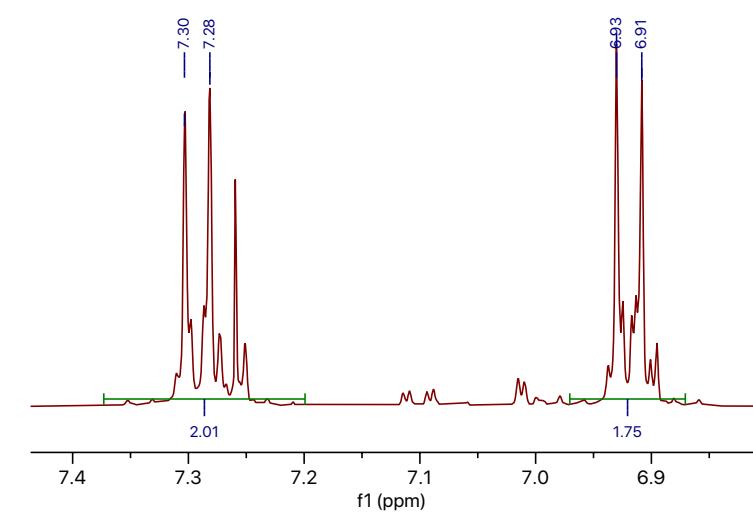
¹³C NMR (101 MHz, CDCl₃) δ 195.5, 162.5, 140.4, 130.5, 115.2, 77.2, 71.5, 41.2, 26.0, 18.6, 11.9, 10.6.

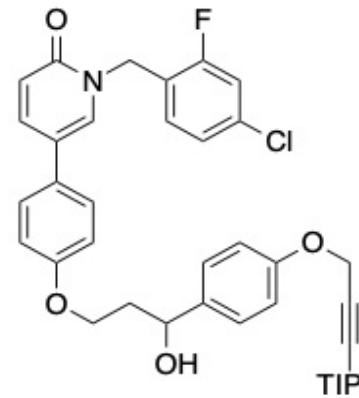
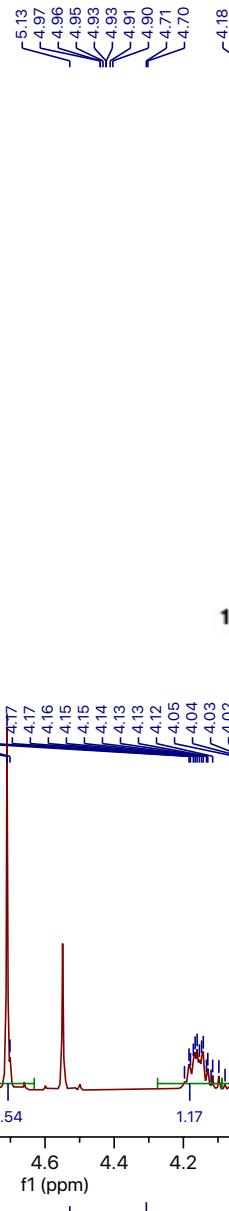
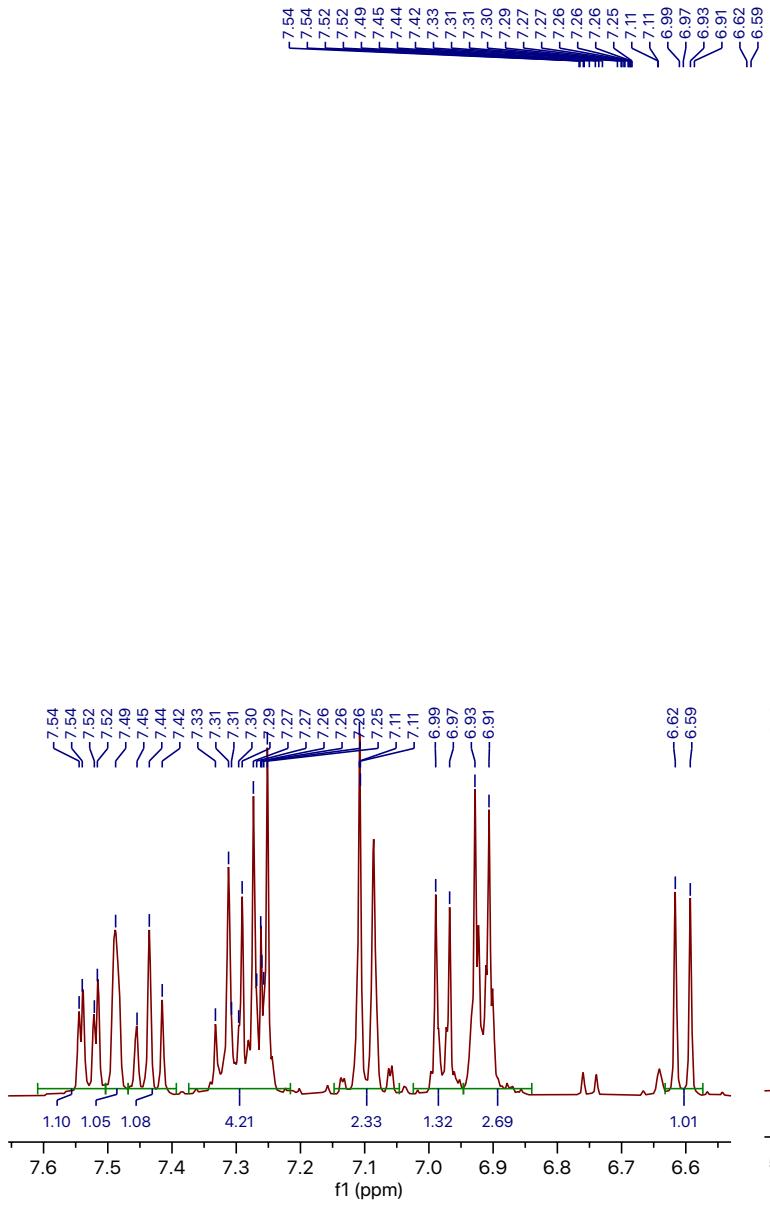




(±)-S15

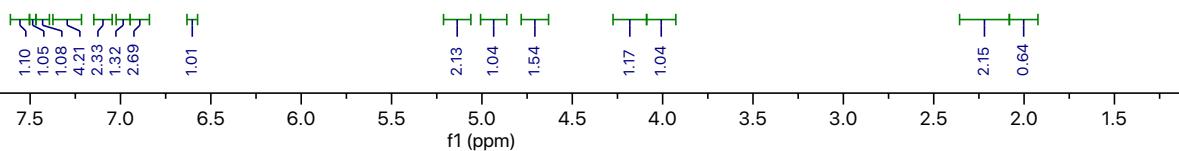
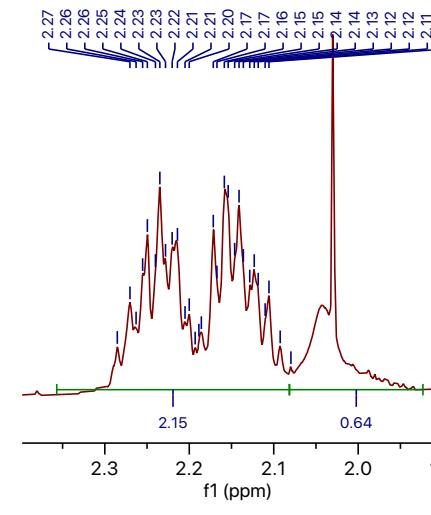
¹H NMR (400 MHz, CDCl_3 with TMS)

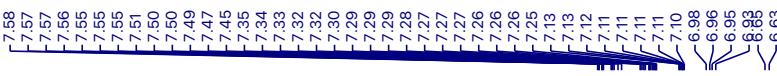




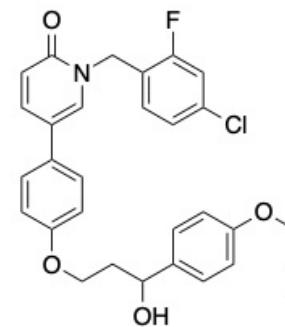
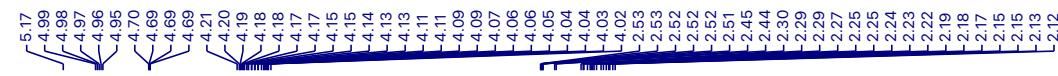
(\pm)-S16

^1H NMR (400 MHz, CDCl_3 with TMS)



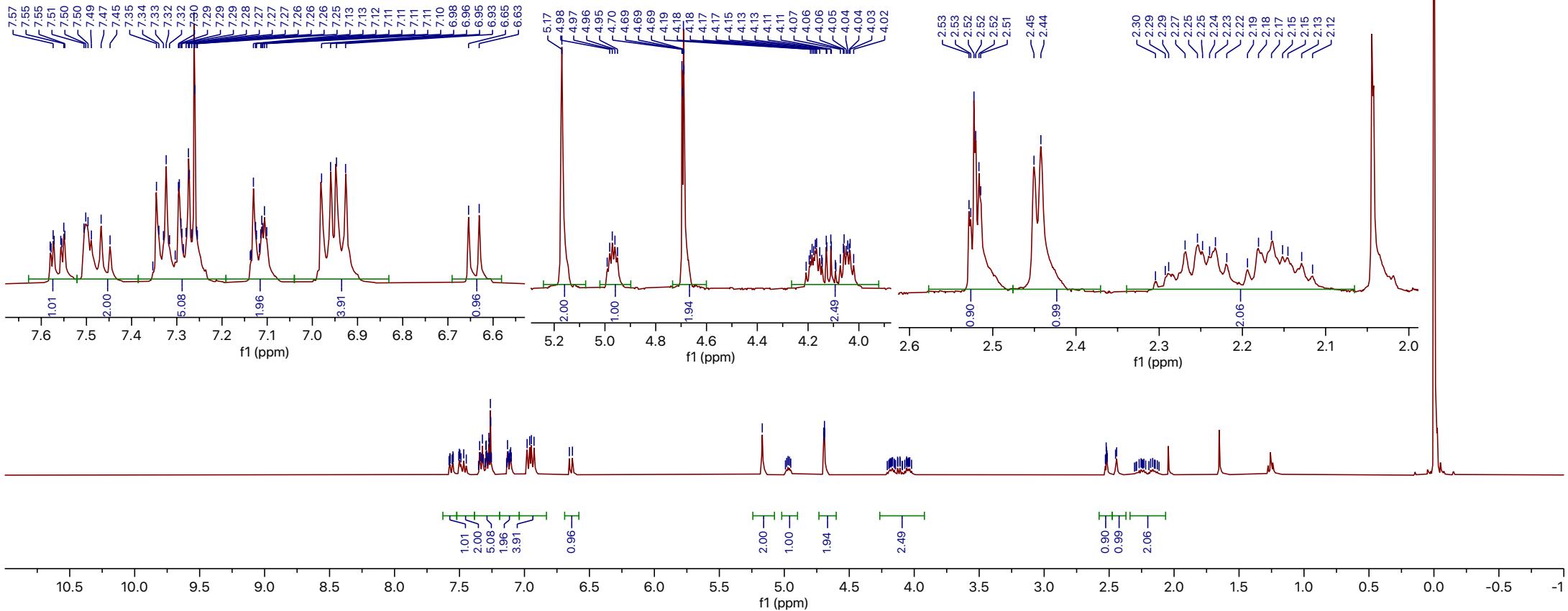


¹H NMR (400 MHz, Chloroform-*d*) δ 7.56 (ddd, *J* = 9.5, 2.7, 0.9 Hz, 1H), 7.52 – 7.43 (m, 2H), 7.37 – 7.24 (m, 4H), 7.15 – 7.08 (m, 2H), 6.95 (dd, *J* = 13.2, 8.0 Hz, 4H), 6.64 (d, *J* = 9.4 Hz, 1H), 5.17 (s, 2H), 4.97 (dt, *J* = 8.3, 4.0 Hz, 1H), 4.69 (dd, *J* = 2.4, 0.9 Hz, 2H), 4.23 – 4.00 (m, 2H), 2.52 (td, *J* = 2.4, 0.9 Hz, 1H), 2.45 (d, *J* = 3.3 Hz, 1H), 2.34 – 2.09 (m, 2H).

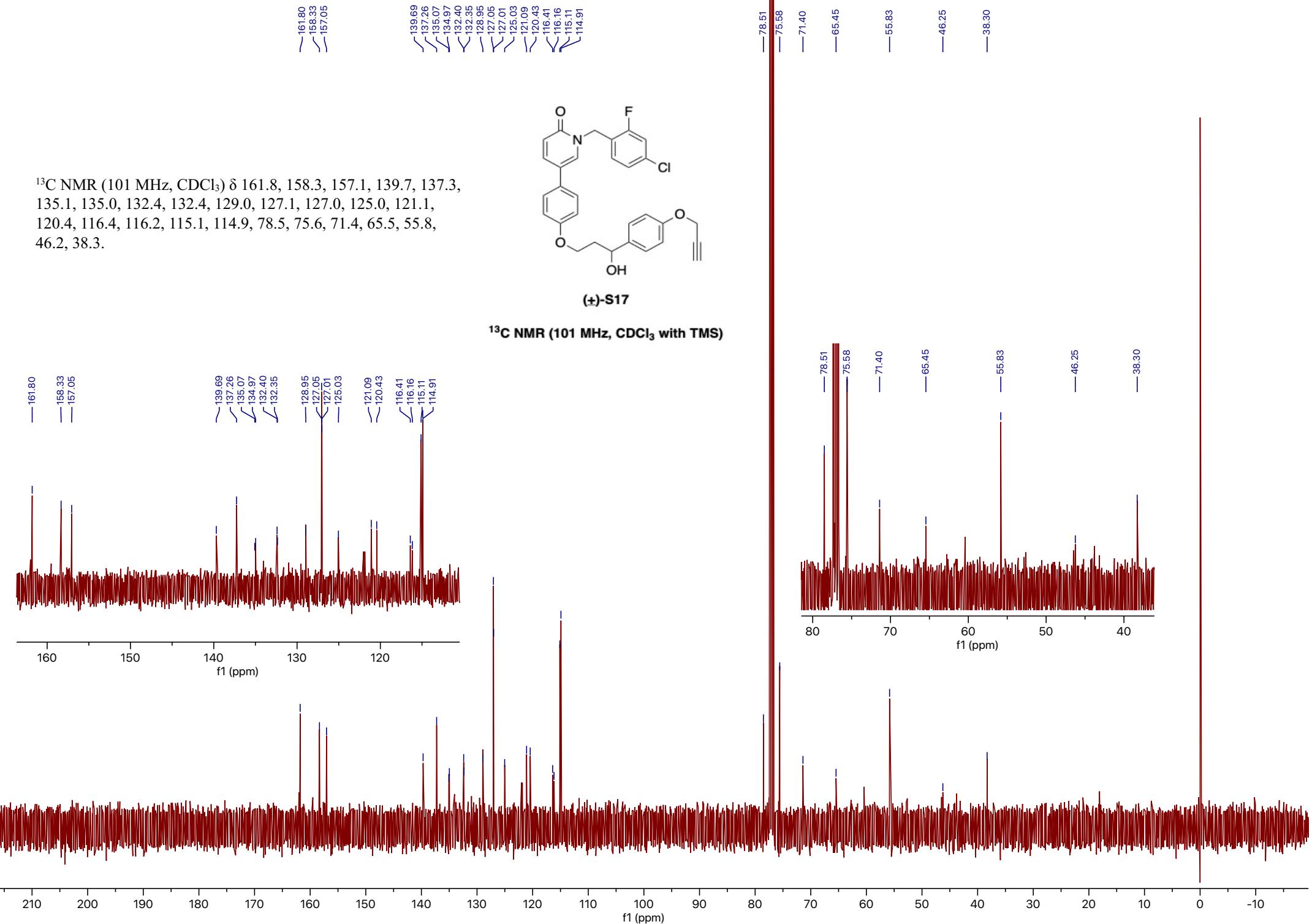


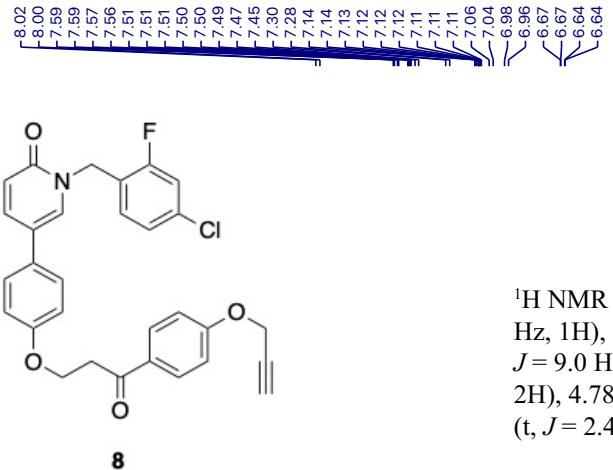
(+)-S17

¹H NMR (400 MHz, CDCl₃ with TMS)



^{13}C NMR (101 MHz, CDCl_3) δ 161.8, 158.3, 157.1, 139.7, 137.3, 135.1, 135.0, 132.4, 132.4, 129.0, 127.1, 127.0, 125.0, 121.1, 120.4, 116.4, 116.2, 115.1, 114.9, 78.5, 75.6, 71.4, 65.5, 55.8, 46.2, 38.3.

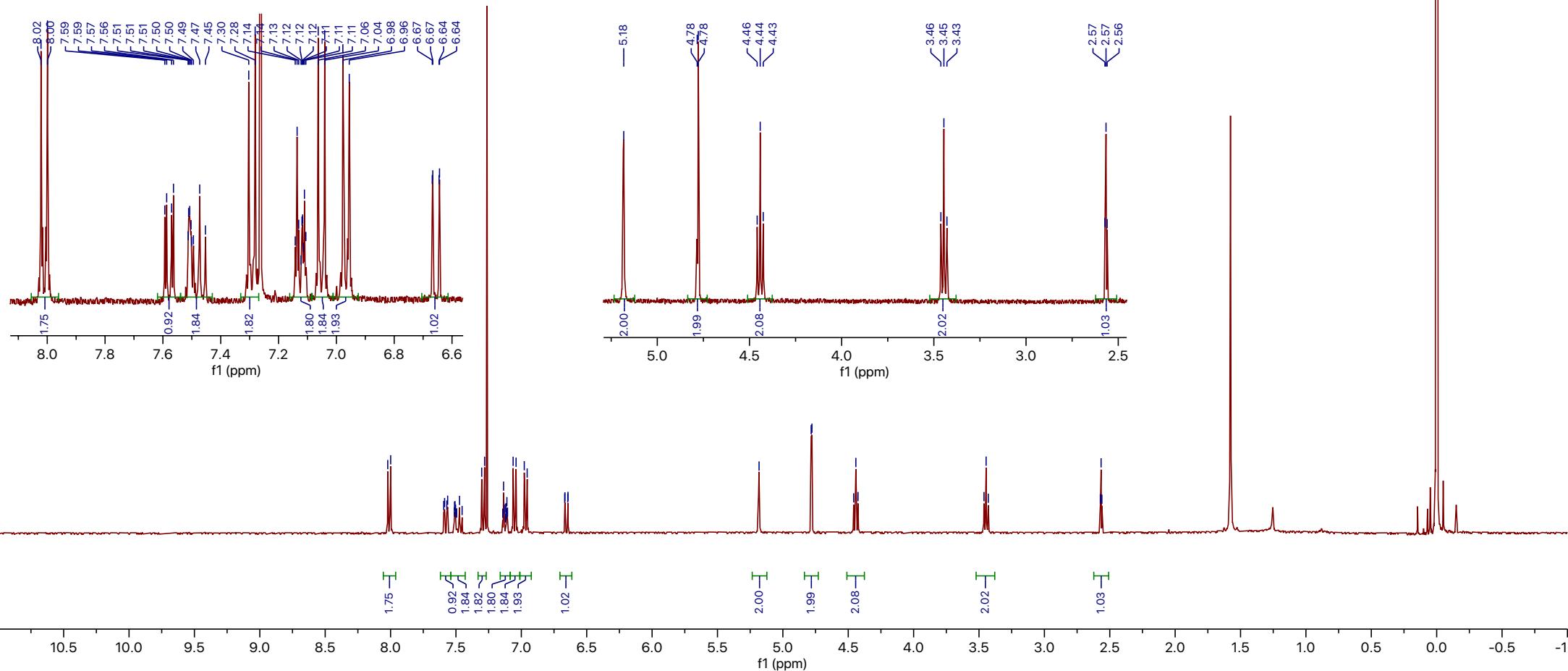


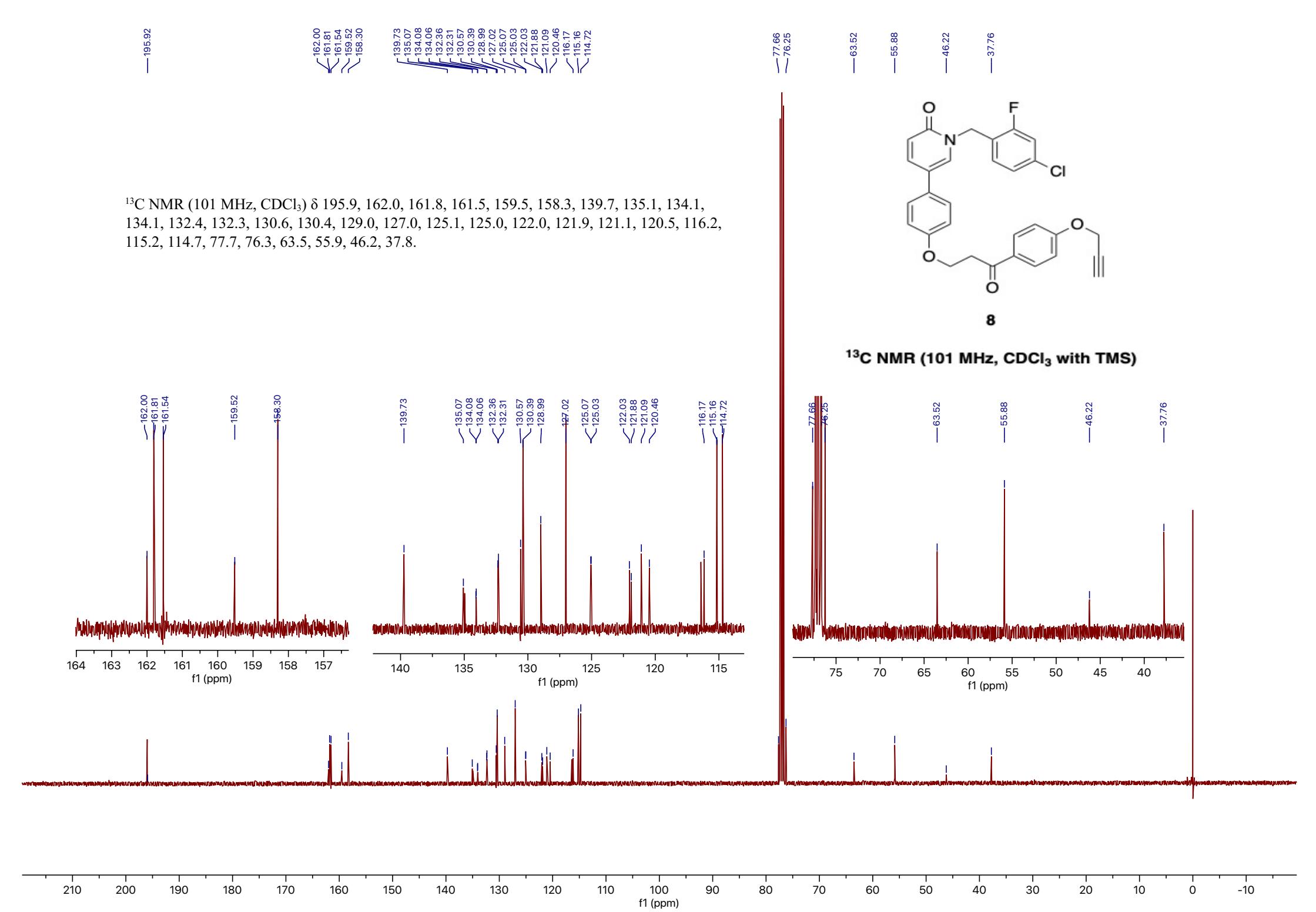


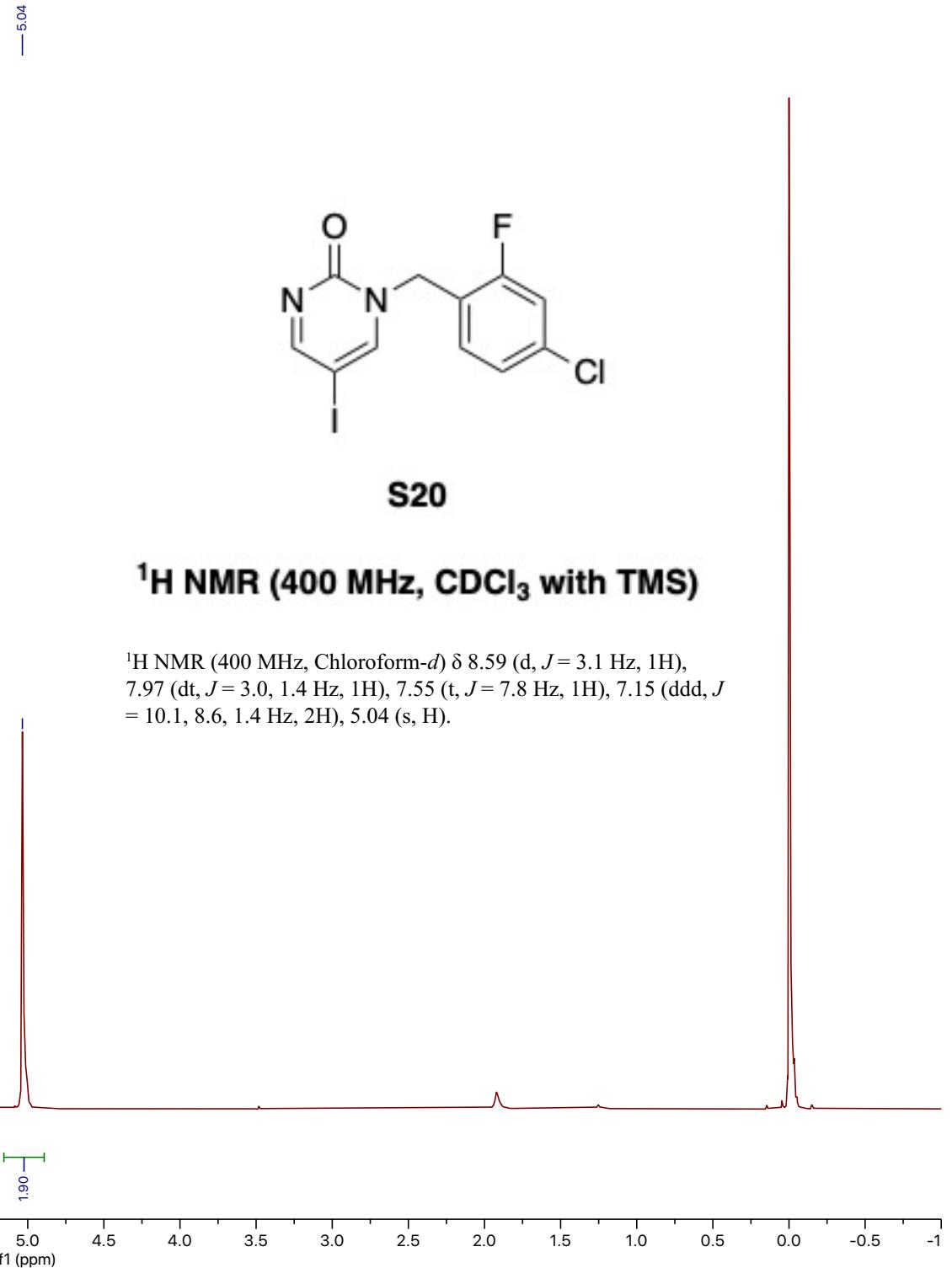
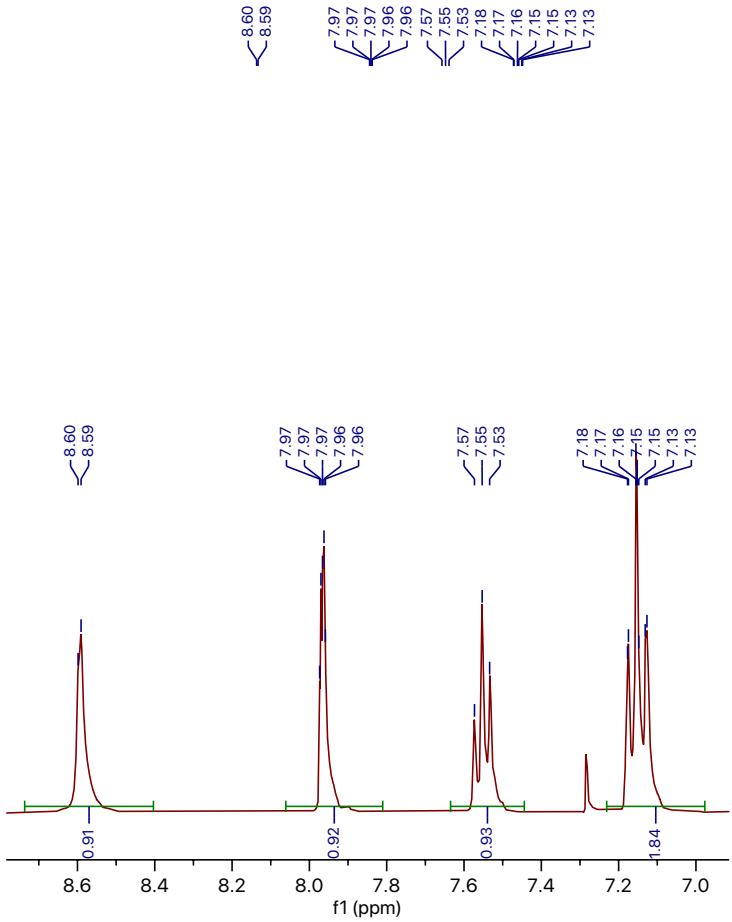
8

¹H NMR (400 MHz, Chloroform-*d*) δ 8.01 (d, *J* = 9.0 Hz, 2H), 7.58 (dd, *J* = 9.4, 2.6 Hz, 1H), 7.54 – 7.43 (m, 2H), 7.29 (d, *J* = 8.9 Hz, 2H), 7.15 – 7.09 (m, 2H), 7.05 (d, *J* = 9.0 Hz, 2H), 6.97 (d, *J* = 8.9 Hz, 2H), 6.66 (dd, *J* = 9.4, 0.6 Hz, 1H), 5.18 (s, 2H), 4.78 (d, *J* = 2.4 Hz, 2H), 4.44 (t, *J* = 6.6 Hz, 2H), 3.45 (t, *J* = 6.6 Hz, 2H), 2.57 (t, *J* = 2.4 Hz, 1H).

¹H NMR (400 MHz, CDCl₃ with TMS)







— 170.70

— 162.15

— 159.67

— 154.03

— 151.79

— 151.76

— 136.14

— 133.16

— 133.12

— 125.29

— 125.26

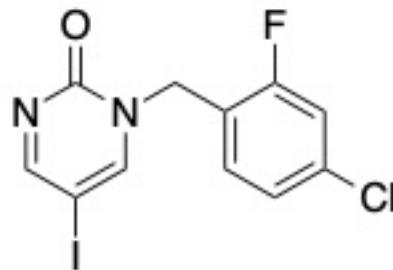
— 119.79

— 116.62

— 116.37

— 48.55

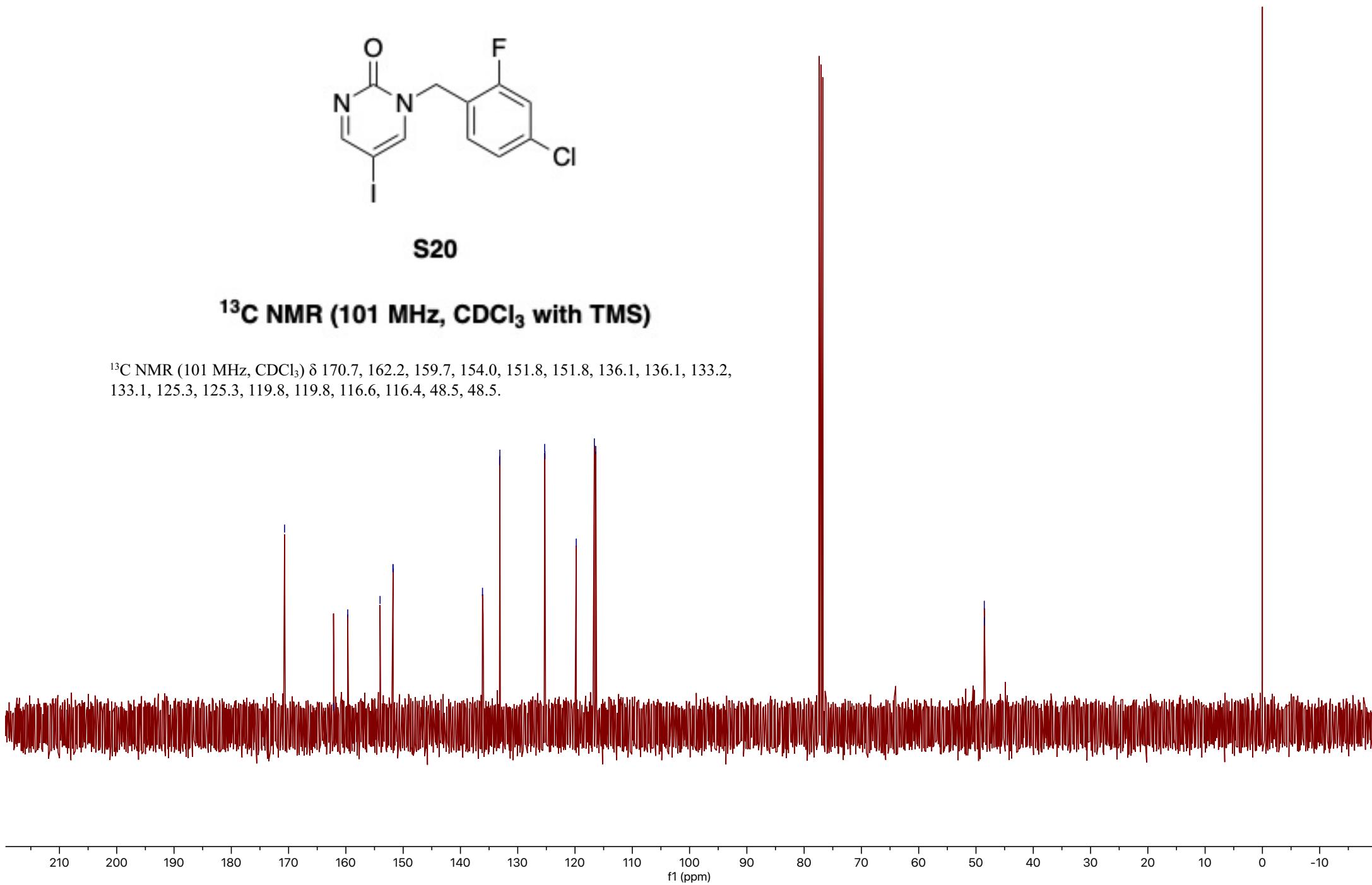
— 48.52



S20

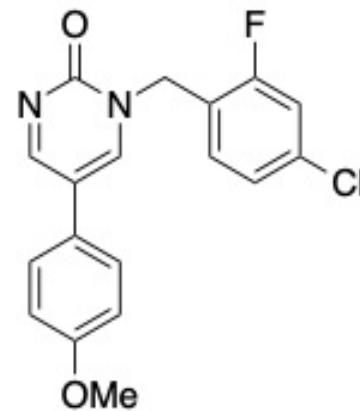
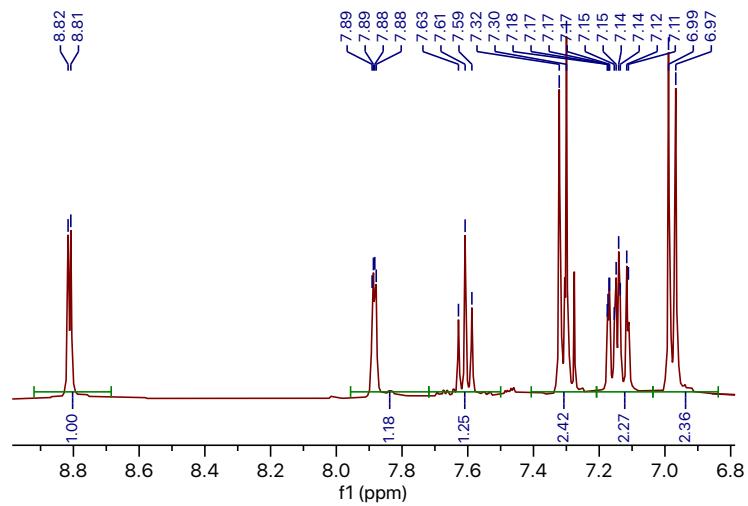
¹³C NMR (101 MHz, CDCl₃ with TMS)

¹³C NMR (101 MHz, CDCl₃) δ 170.7, 162.2, 159.7, 154.0, 151.8, 151.8, 136.1, 136.1, 133.2, 133.1, 125.3, 125.3, 119.8, 119.8, 116.6, 116.4, 48.5, 48.5.



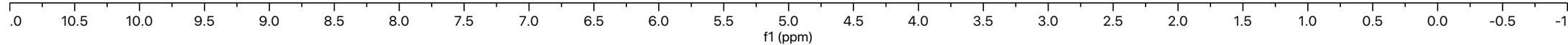


¹H NMR (400 MHz, Chloroform-*d*) δ 8.81 (d, *J* = 3.3 Hz, 1H), 7.88 (dd, *J* = 3.3, 1.7 Hz, 1H), 7.61 (t, *J* = 8.2 Hz, 1H), 7.31 (d, *J* = 8.8 Hz, 2H), 7.20 – 7.08 (m, 2H), 6.98 (d, *J* = 8.8 Hz, 2H), 5.13 (d, *J* = 1.4 Hz, 2H), 3.84 (s, 3H).



9

¹H NMR (400 MHz, CDCl₃ with TMS)



— 165.62
— 162.21
— 159.72
— 155.59

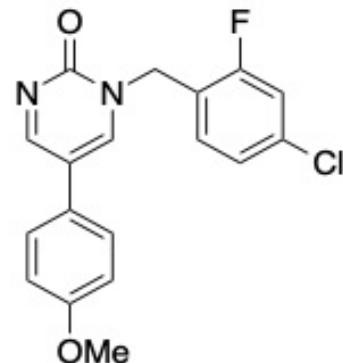
— 143.90
— 143.87

— 135.84
— 135.74
— 133.19
— 133.15
— 127.13
— 125.32
— 125.25
— 125.21
— 120.56
— 120.41
— 118.69
— 116.52
— 116.28
— 114.82

— 55.42

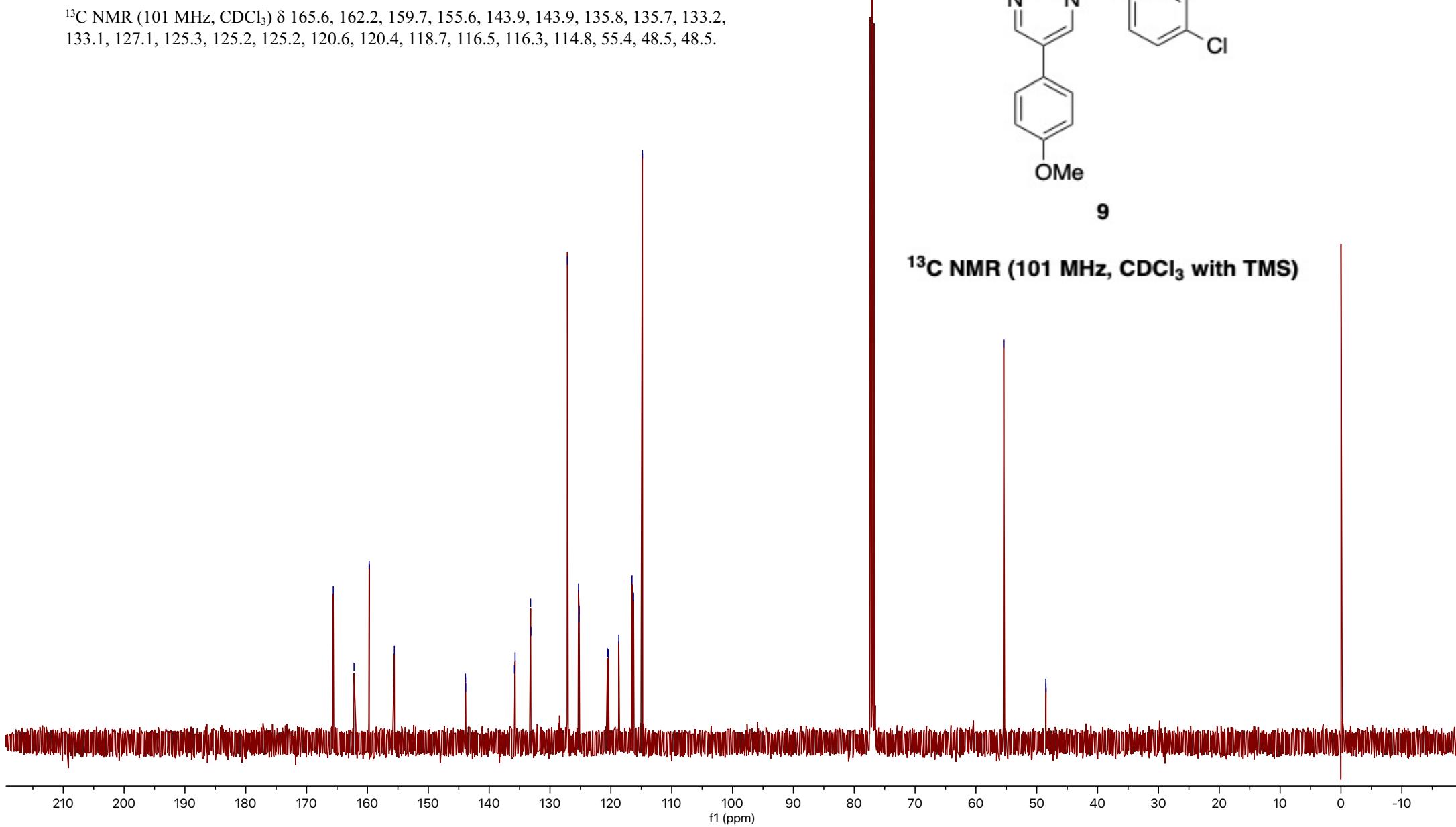
— 48.52
— 48.49

¹³C NMR (101 MHz, CDCl₃) δ 165.6, 162.2, 159.7, 155.6, 143.9, 143.9, 135.8, 135.7, 133.2, 133.1, 127.1, 125.3, 125.2, 125.2, 120.6, 120.4, 118.7, 116.5, 116.3, 114.8, 55.4, 48.5, 48.5.



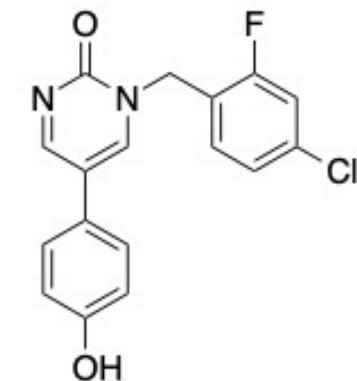
9

¹³C NMR (101 MHz, CDCl₃ with TMS)



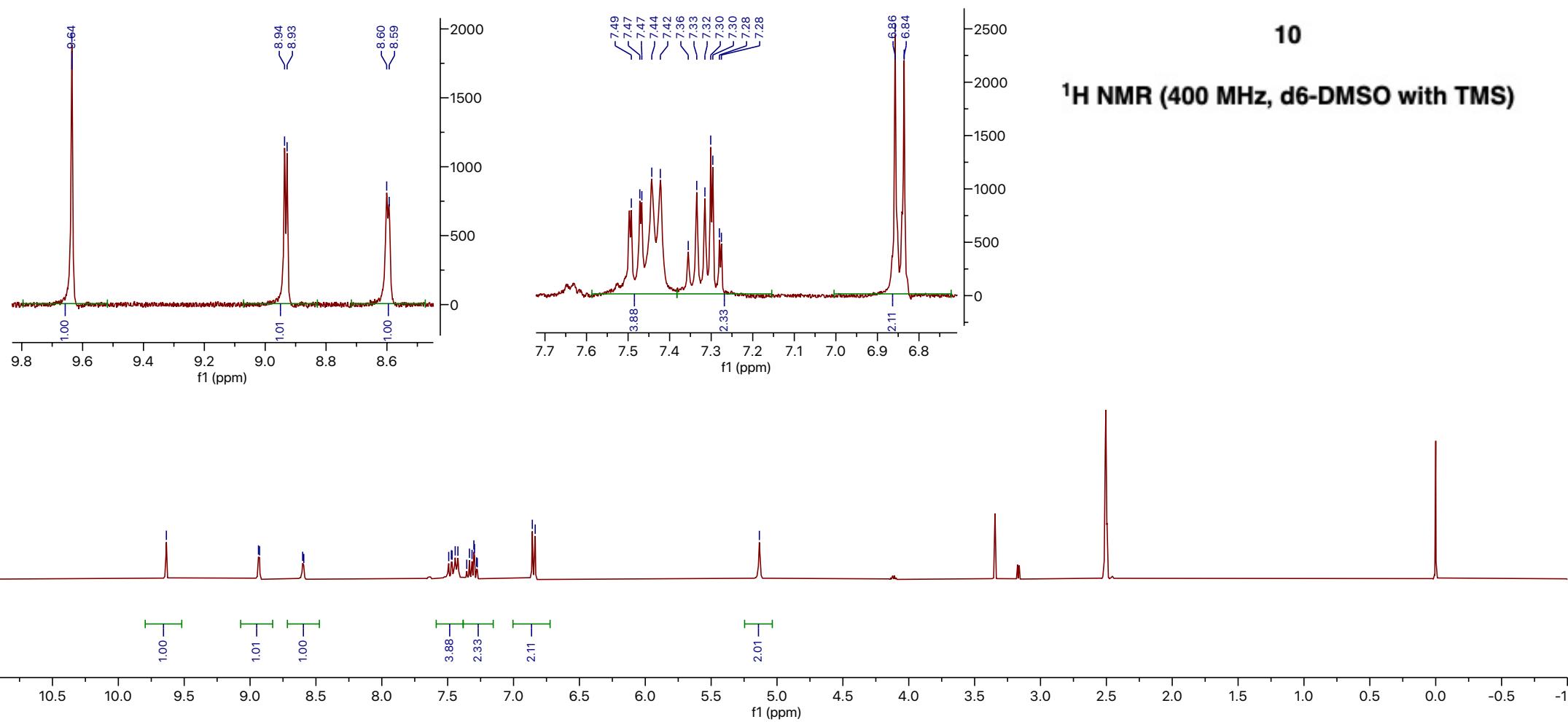


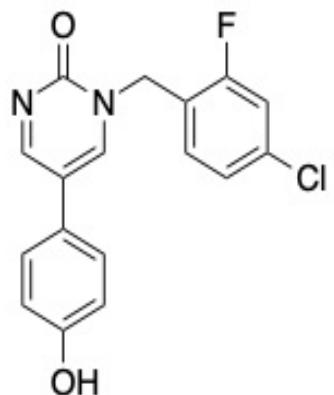
^1H NMR (400 MHz, DMSO-*d*₆) δ 9.64 (s, 1H), 8.93 (d, *J* = 3.3 Hz, 1H), 8.60 (d, *J* = 3.3 Hz, 1H), 7.59 – 7.38 (m, 3H), 7.38 – 7.15 (m, 2H), 6.85 (d, *J* = 8.7 Hz, 2H), 5.13 (s, 2H).



10

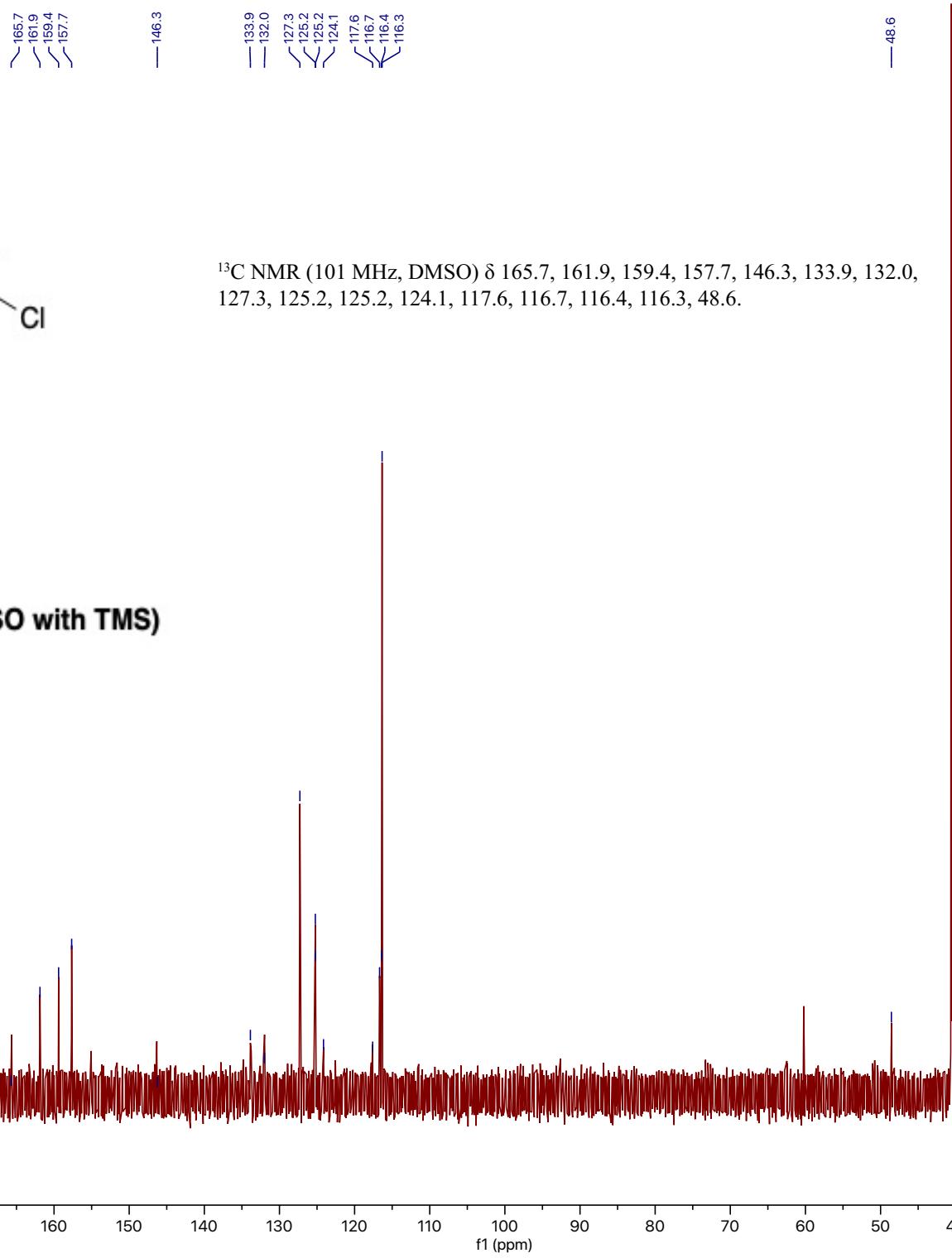
^1H NMR (400 MHz, d₆-DMSO with TMS)

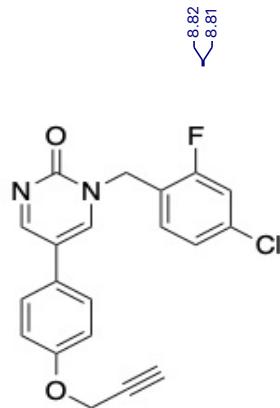




10

¹³C NMR (101 MHz, d₆-DMSO with TMS)

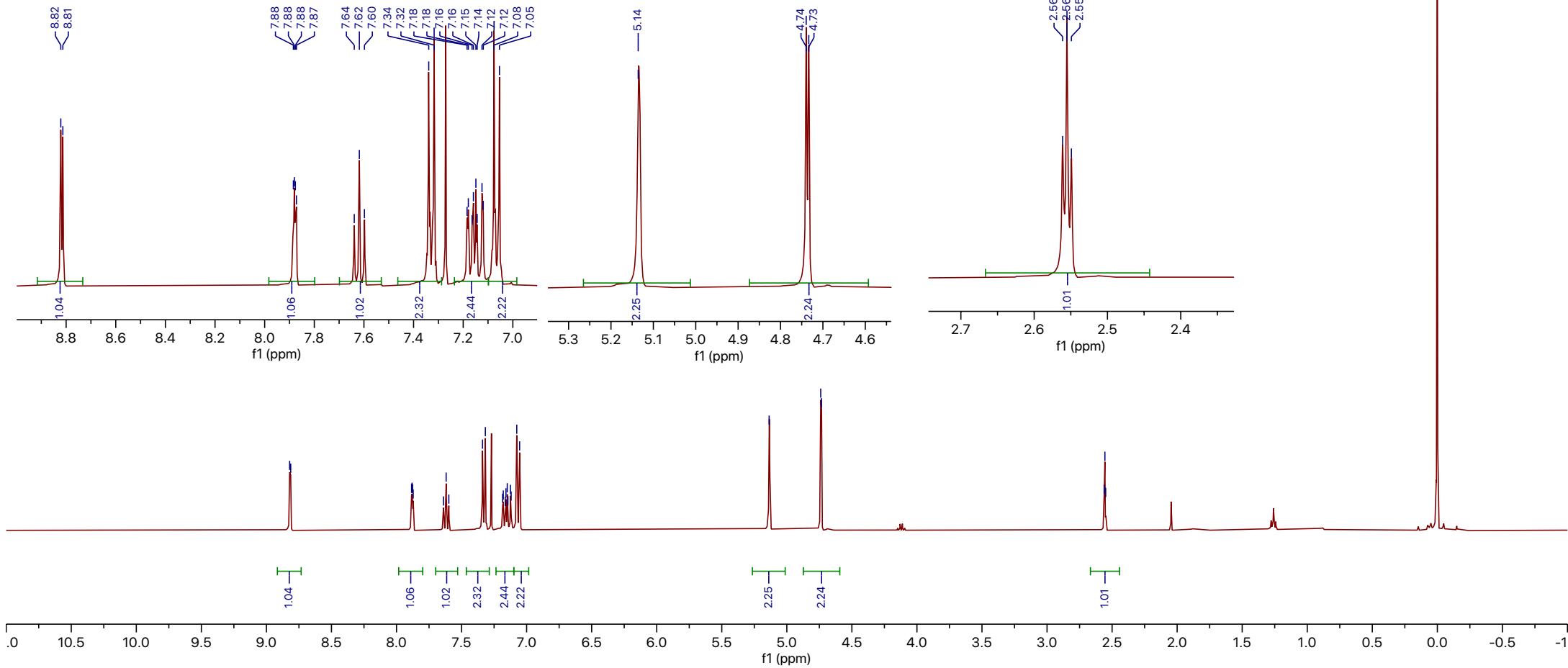


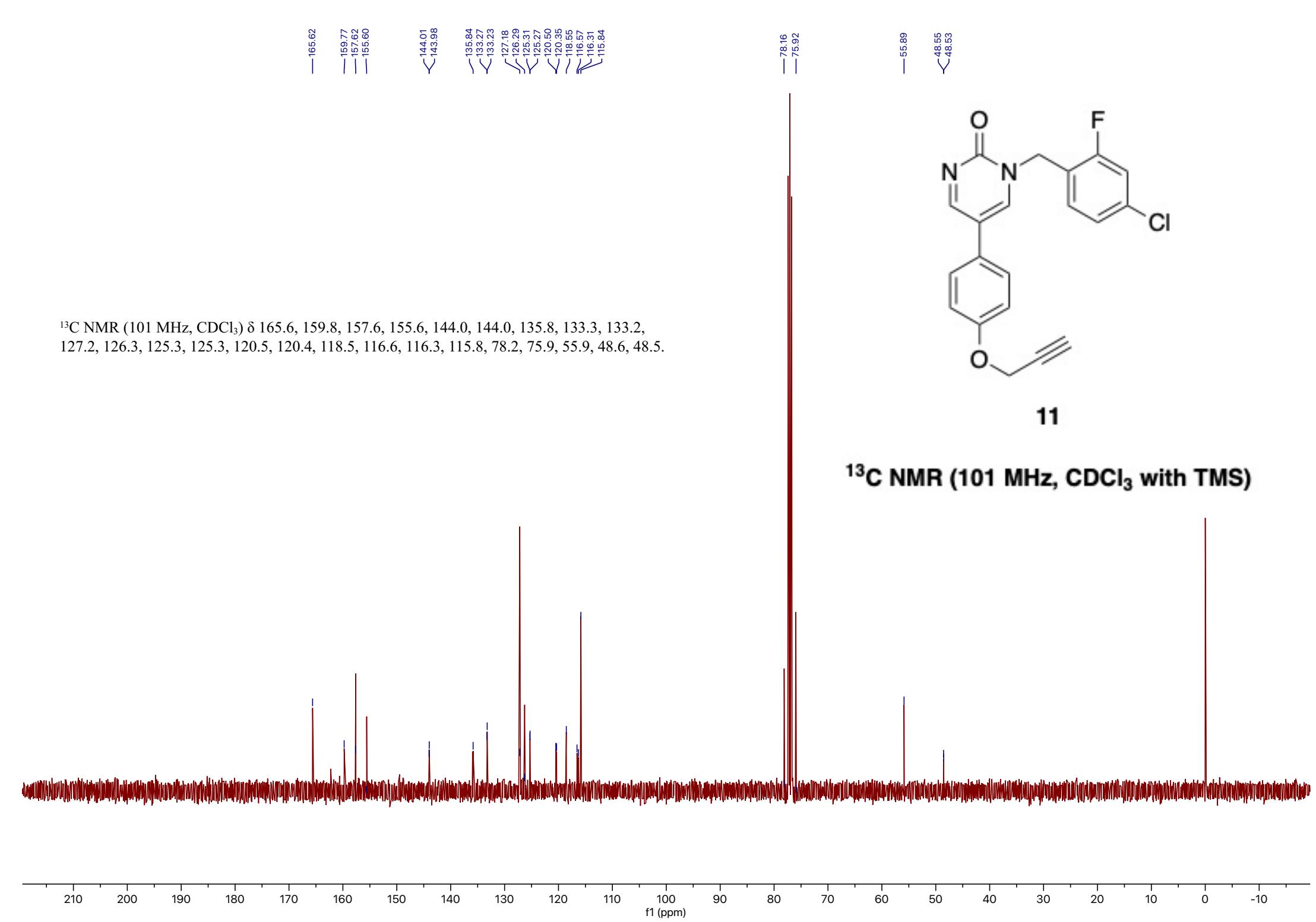


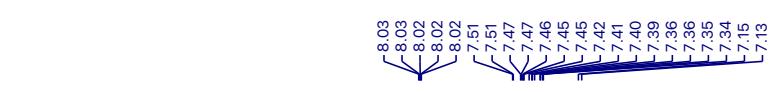
11

¹H NMR (400 MHz, Chloroform-*d*) δ 8.82 (d, *J* = 3.3 Hz, 1H), 7.88 (dd, *J* = 3.3, 1.6 Hz, 1H), 7.62 (t, *J* = 8.2 Hz, 1H), 7.33 (d, *J* = 8.7 Hz, 2H), 7.22 – 7.10 (m, 2H), 7.06 (d, *J* = 8.7 Hz, 2H), 5.14 (s, 2H), 4.74 (d, *J* = 2.4 Hz, 2H), 2.56 (t, *J* = 2.4 Hz, 1H).

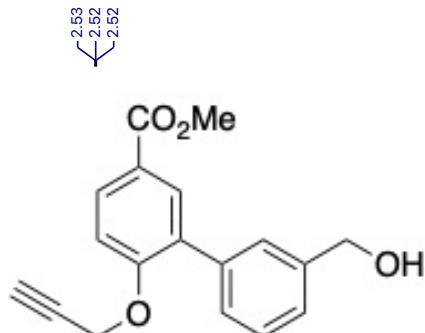
¹H NMR (400 MHz, CDCl₃ with TMS)



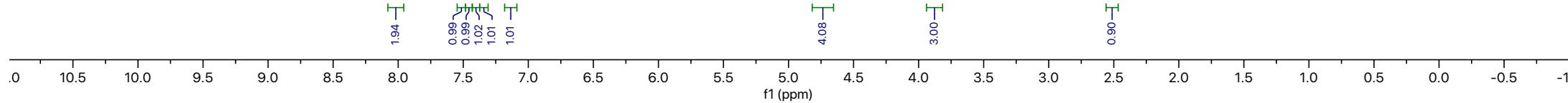
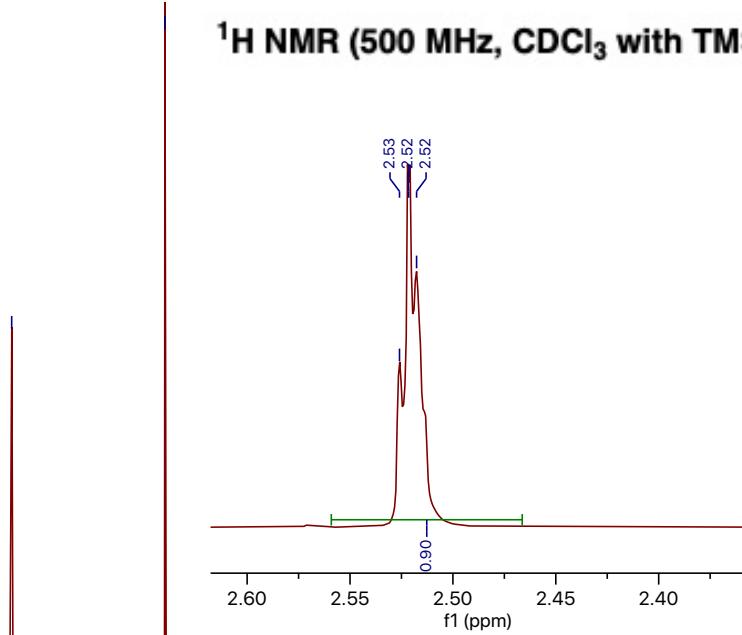
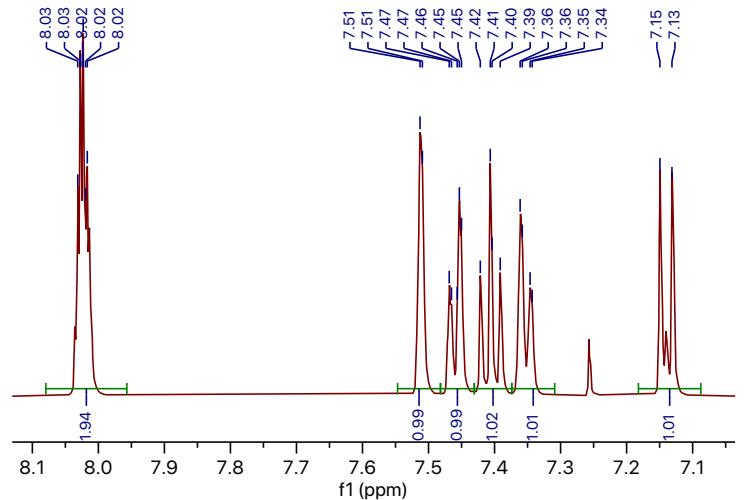


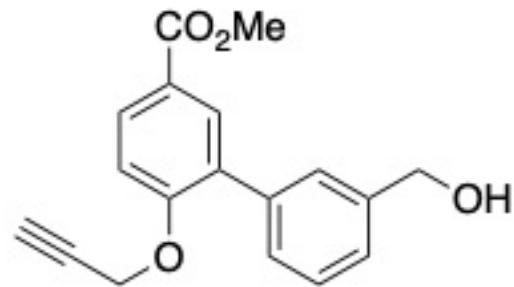


^1H NMR (500 MHz, Chloroform-d) δ 8.02 (dt, J = 3.1, 1.7 Hz, 2H), 7.51 (d, J = 1.8 Hz, 1H), 7.46 (dd, J = 7.6, 1.7 Hz, 1H), 7.41 (t, J = 7.5 Hz, 1H), 7.35 (dd, J = 7.5, 1.6 Hz, 1H), 7.14 (d, J = 9.1 Hz, 1H), 4.74 (s, 4H), 3.89 (s, 3H), 2.52 (t, J = 2.1 Hz, 1H).



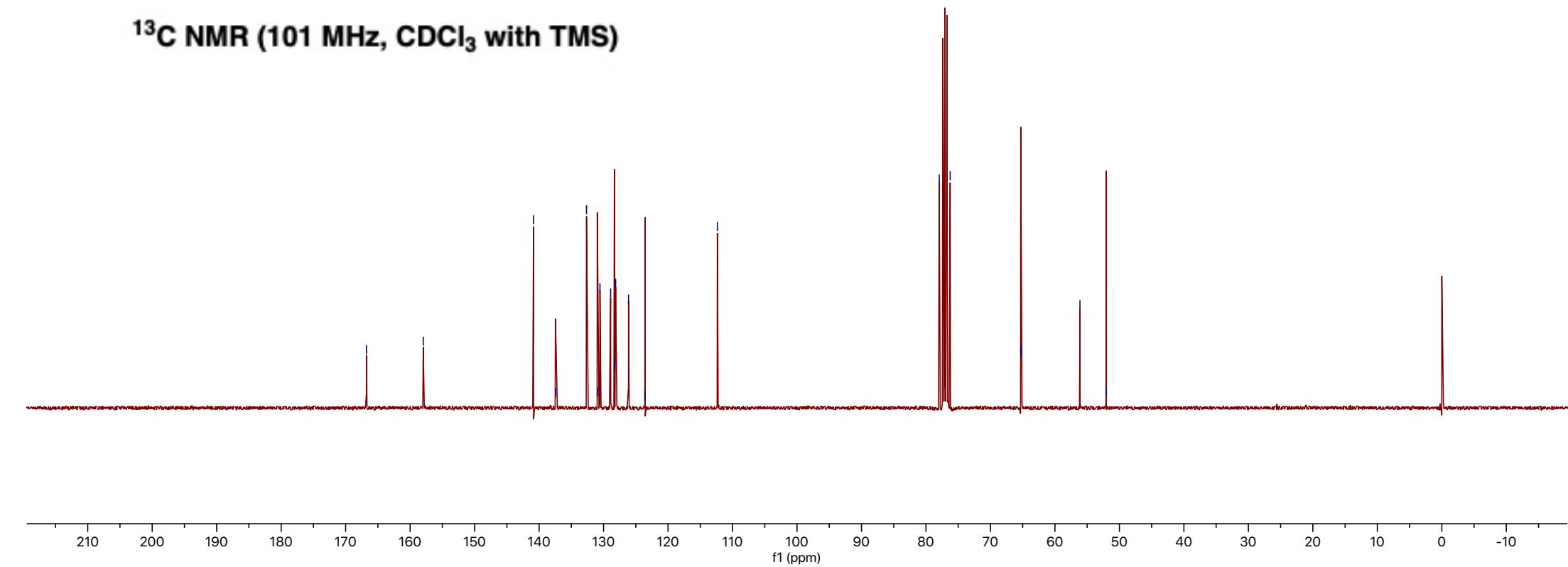
^1H NMR (500 MHz, CDCl_3 with TMS)



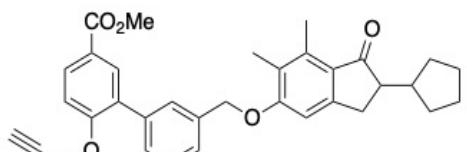
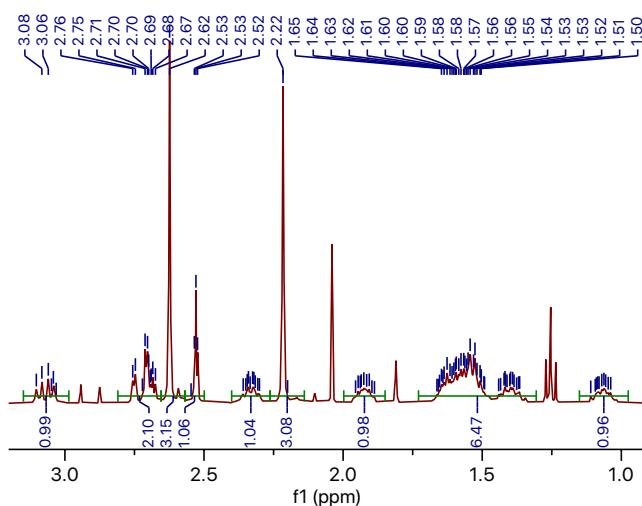
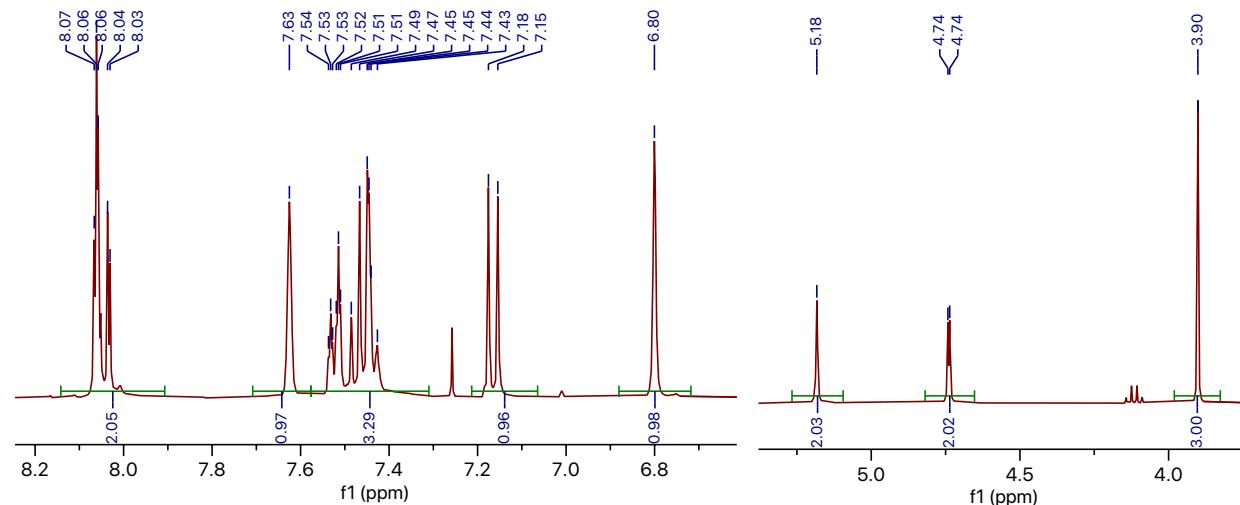


S25

^{13}C NMR (101 MHz, CDCl_3 with TMS)

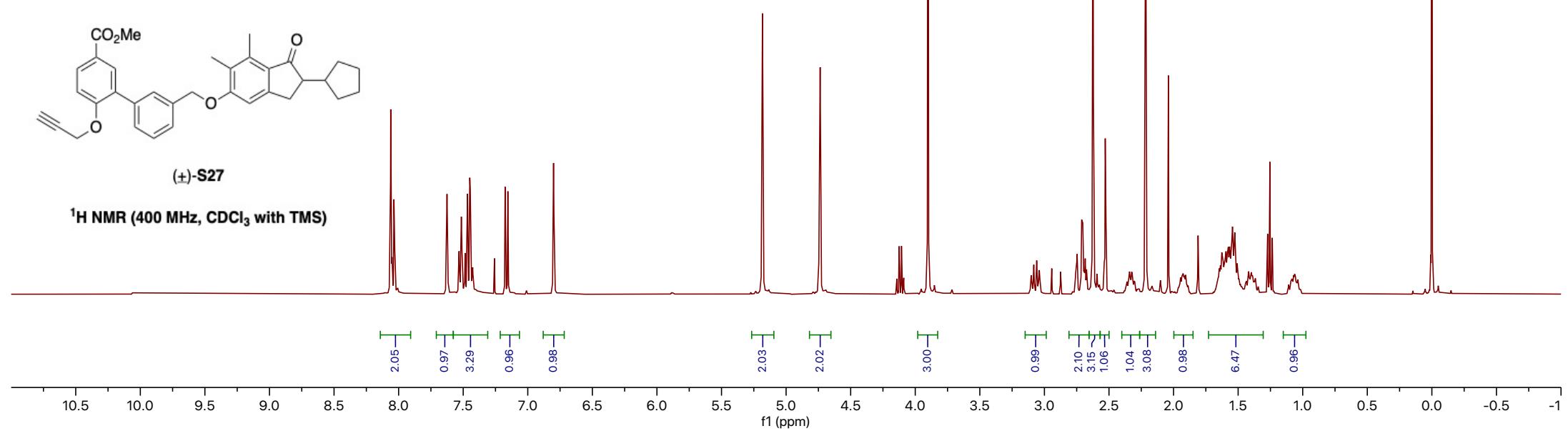


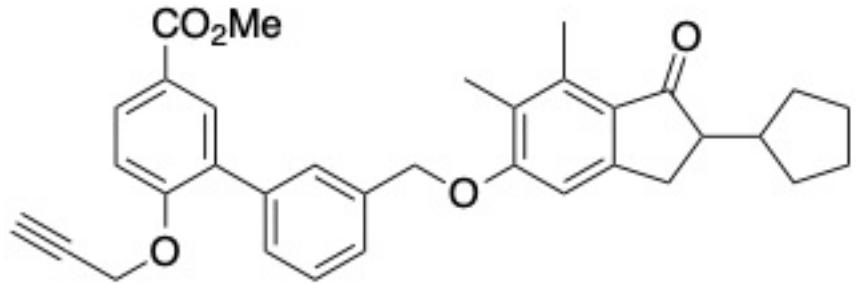
¹H NMR (400 MHz, Chloroform-*d*) δ 8.15 – 7.92 (m, 2H), 7.63 (s, 1H), 7.58 – 7.31 (m, 3H), 7.16 (d, *J* = 8.5 Hz, 1H), 6.80 (s, 1H), 5.18 (s, 2H), 4.74 (d, *J* = 2.4 Hz, 2H), 3.90 (s, 3H), 3.12 – 3.02 (m, 1H), 2.78 – 2.65 (m, 2H), 2.62 (s, 3H), 2.53 (t, *J* = 2.3 Hz, 1H), 2.33 (dtt, *J* = 8.0, 6.0, 3.0 Hz, 1H), 2.22 (s, 3H), 1.92 (ddt, *J* = 11.2, 7.4, 3.7 Hz, 1H), 1.74 – 1.32 (m, 6H), 1.13 – 0.99 (m, 1H).



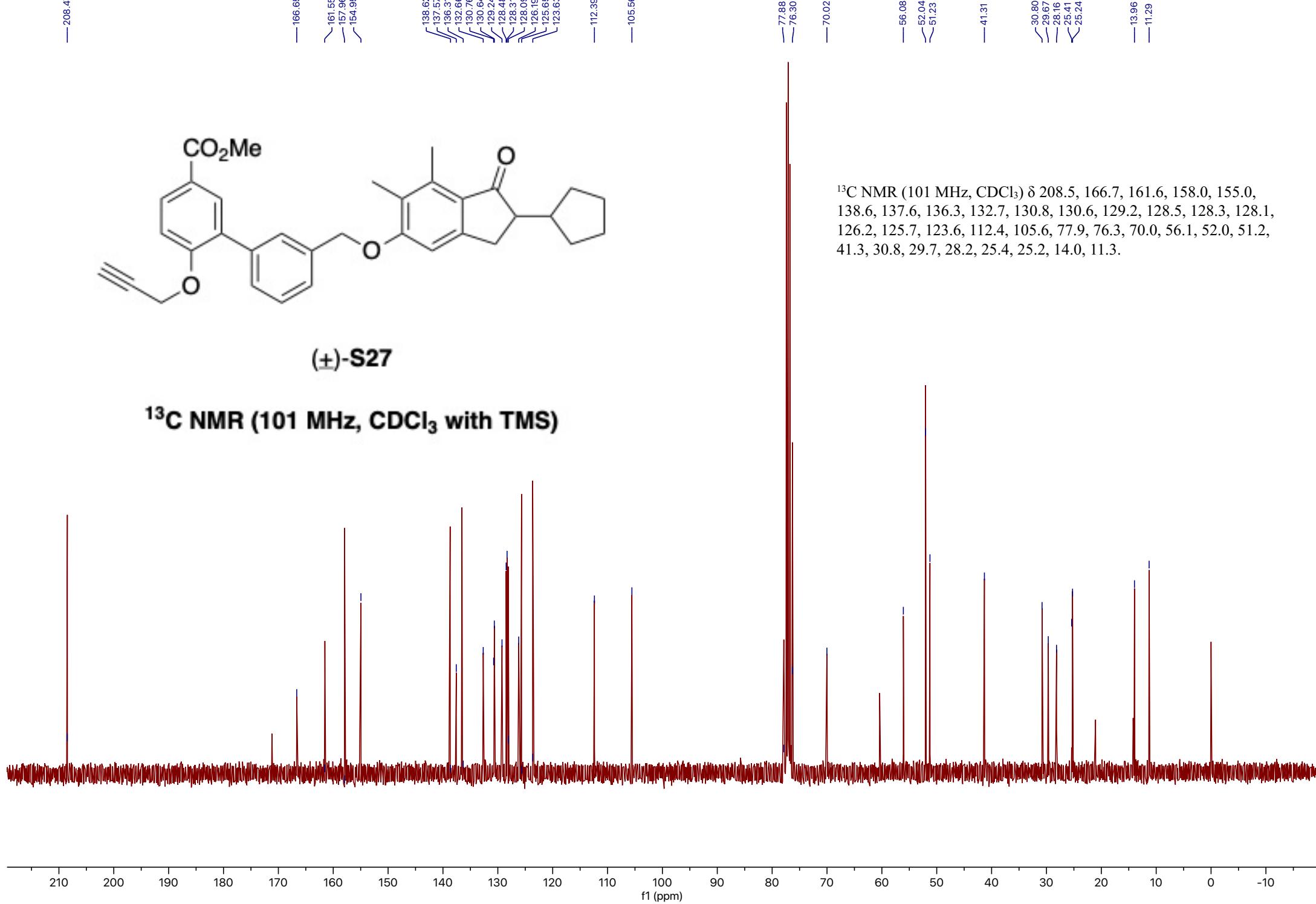
(±)-S27

¹H NMR (400 MHz, CDCl₃ with TMS)

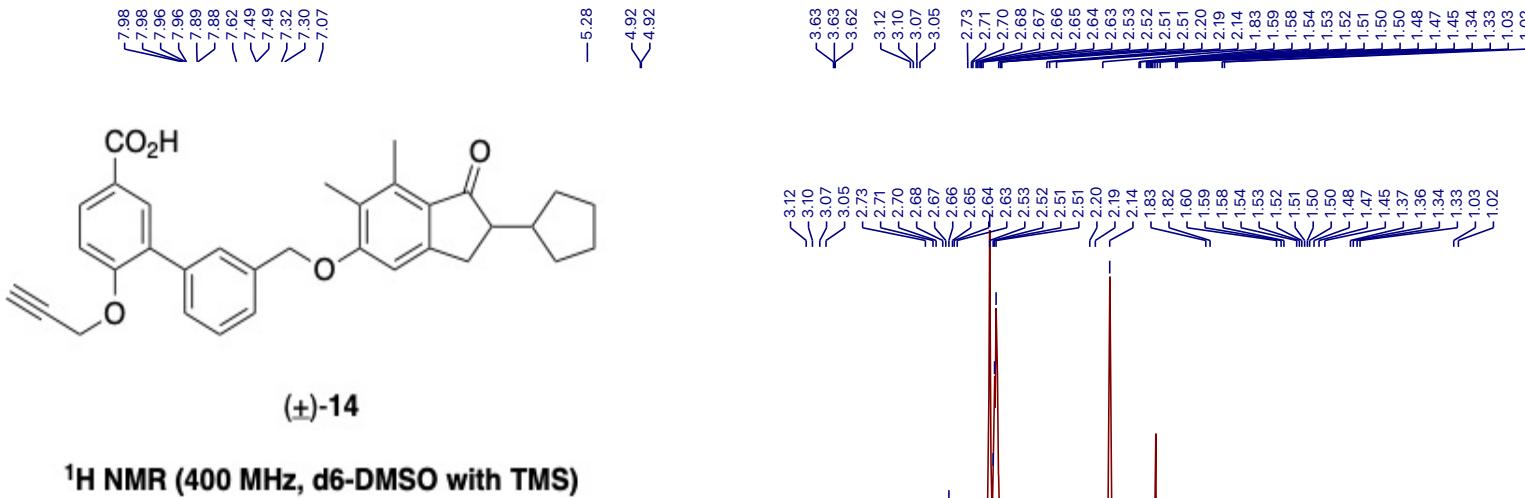




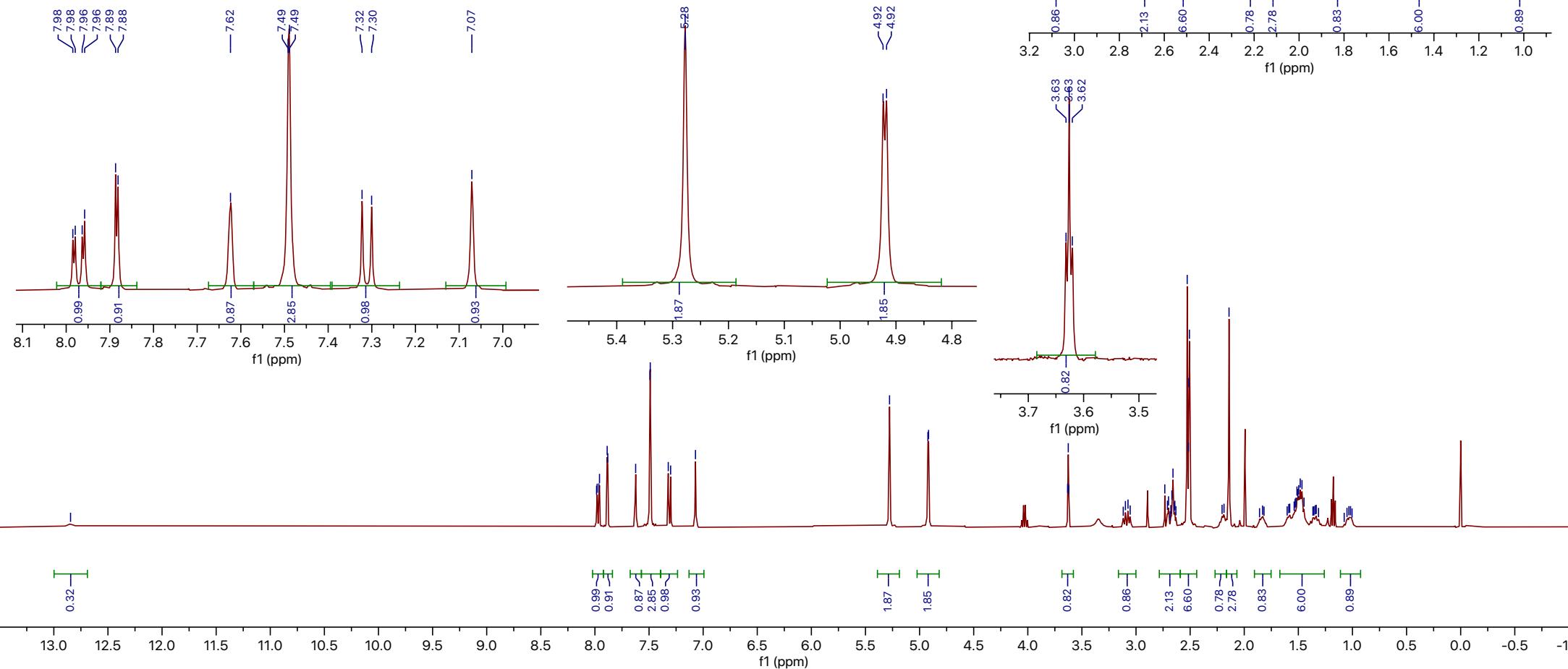
^{13}C NMR (101 MHz, CDCl_3 with TMS)



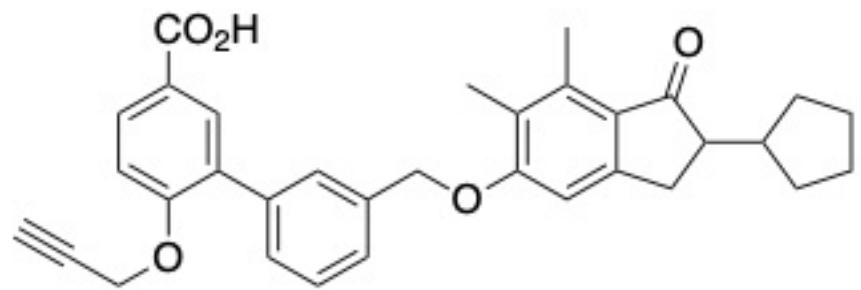
¹H NMR (400 MHz, DMSO-*d*₆) δ 12.85 (s, 1H), 7.97 (dd, *J* = 8.6, 2.2 Hz, 1H), 7.88 (d, *J* = 2.2 Hz, 1H), 7.62 (s, 1H), 7.49 (d, *J* = 1.2 Hz, 3H), 7.31 (d, *J* = 8.8 Hz, 1H), 7.07 (s, 1H), 5.28 (s, 2H), 4.92 (d, *J* = 2.5 Hz, 2H), 3.63 (t, *J* = 2.3 Hz, 1H), 3.16 – 3.01 (m, 1H), 2.76 – 2.61 (m, 2H), 2.55 – 2.50 (s, 3H), 2.20 (m, 1H), 2.14 (s, 3H), 1.84 (m, 1H), 1.68 – 1.28 (m, 6H), 1.10 – 0.96 (m, 1H).



¹H NMR (400 MHz, d6-DMSO with TMS)



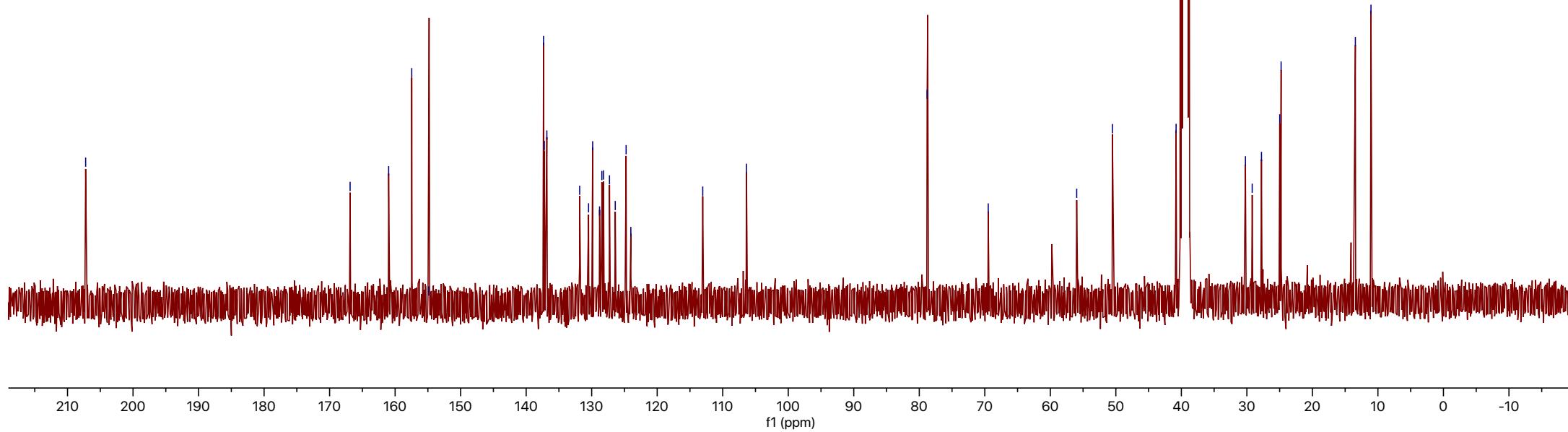
— 207.23



(\pm)-14

^{13}C NMR (101 MHz, d6-DMSO)

^{13}C NMR (101 MHz, DMSO) δ 207.2, 166.9, 161.0, 157.5, 154.9, 137.3, 137.2, 136.8, 131.8, 130.5, 129.8, 128.8, 128.4, 128.2, 127.3, 126.4, 124.7, 124.0, 113.0, 106.4, 78.8, 78.7, 69.5, 56.0, 50.5, 40.8, 30.2, 29.2, 27.8, 25.0, 24.8, 13.4, 11.1.



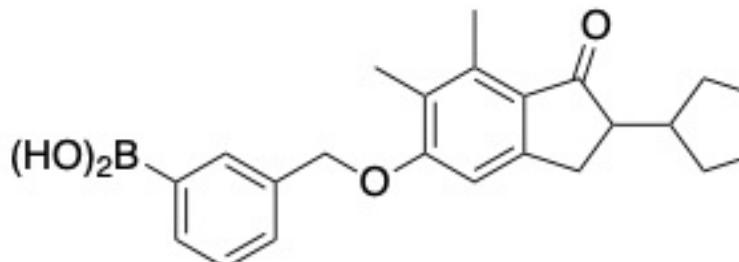
¹H NMR (400 MHz, Chloroform-*d*) δ 8.27 (d, *J* = 6.3 Hz, 1H), 8.15 (d, *J* = 7.2 Hz, 1H), 7.62 (d, *J* = 7.6 Hz, 1H), 7.52 (t, *J* = 7.3 Hz, 1H), 6.77 (s, 1H), 5.11 (s, 2H), 3.18 – 2.94 (m, 1H), 2.83 – 2.46 (m, 5H), 2.43 – 2.09 (m, 4H), 2.01 – 1.80 (m, 1H), 1.78 – 1.45 (m, 5H), 1.38 (td, *J* = 8.6, 7.8, 4.2 Hz, 1H), 1.05 (d, *J* = 10.0 Hz, 1H).

8.29
8.28
8.26
8.16
8.14
7.63
7.61
7.54
7.52
7.50

6.77

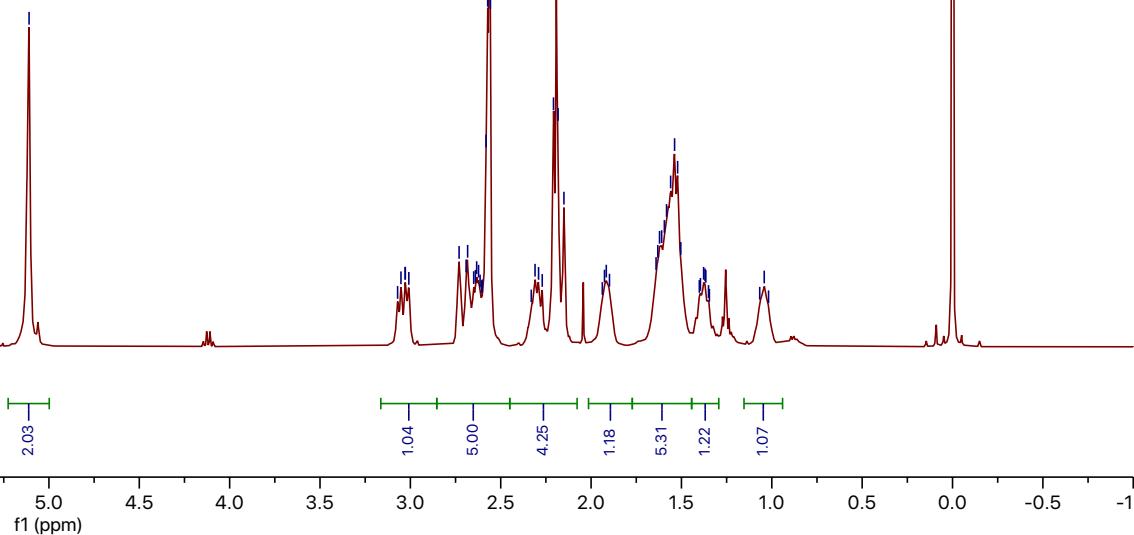
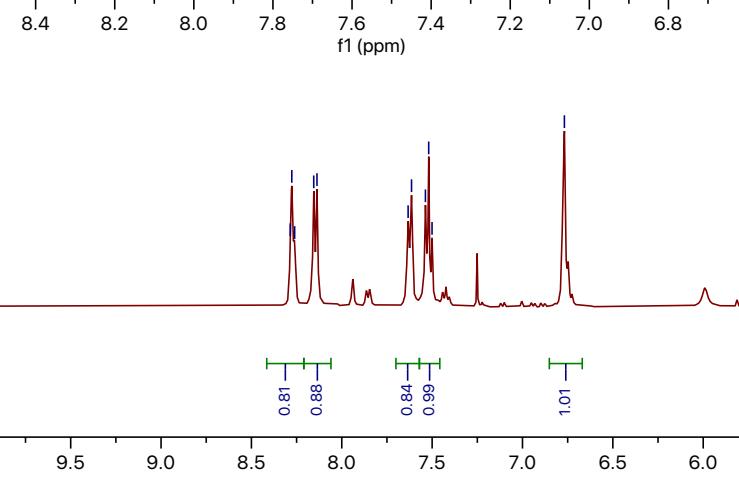
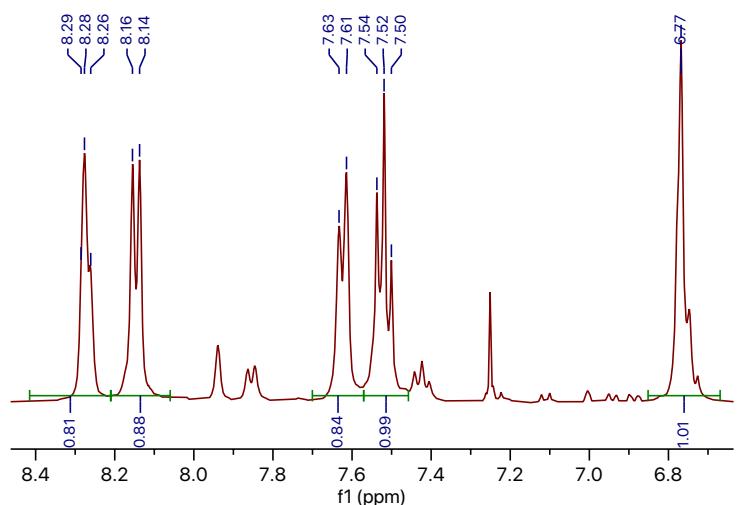
5.11

3.07
3.05
3.03
3.03
3.01
2.73
2.69
2.68
2.66
2.64
2.63
2.62
2.61
2.61
2.60
2.58
2.57
2.56
2.33
2.31
2.29
2.27
2.21
2.19
2.18
2.15
1.94
1.93
1.92
1.90
1.64
1.63
1.62
1.61
1.59
1.58
1.56
1.54
1.52
1.52
1.50
1.40
1.39
1.38
1.37
1.37
1.35
1.35
1.07
1.04
1.02



(±)-S29

¹H NMR (400 MHz, CDCl₃ with TMS)



— 208.65

— 161.43

— 155.13

— 138.78
— 136.29
— 135.20
— 134.05
— 131.51
— 128.37
— 128.13
— 125.53

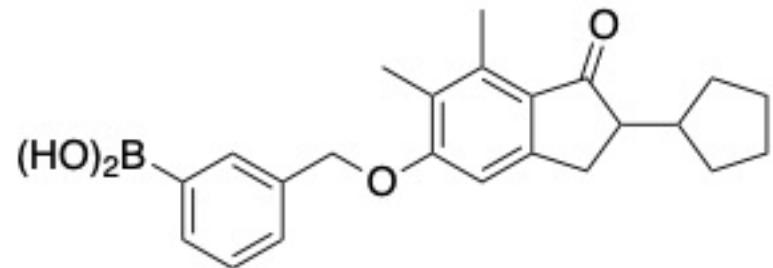
— 105.37

— 69.77

— 51.23

— 30.81
— 29.71
— 28.20
— 25.39
— 25.22

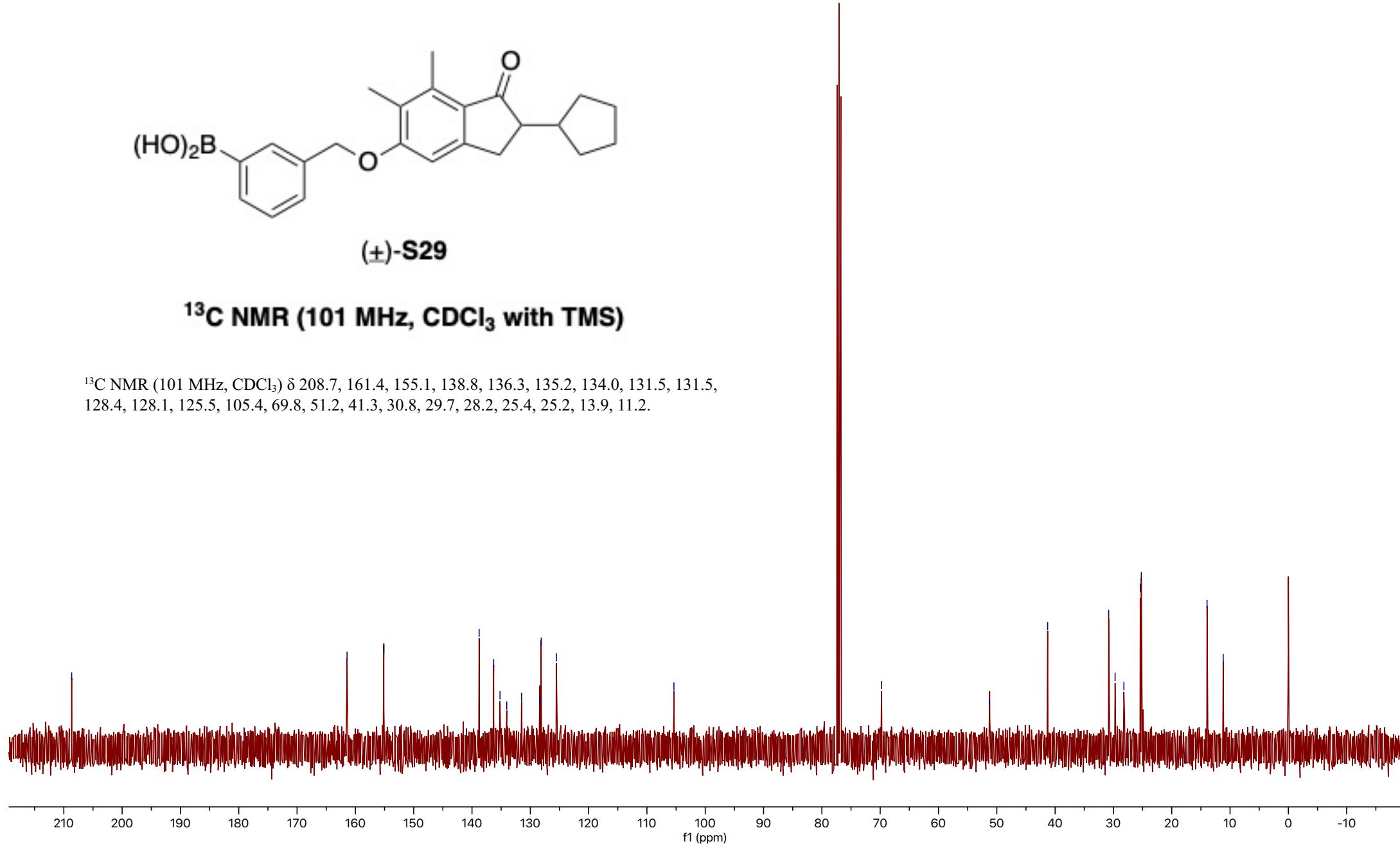
— 13.95
— 11.18

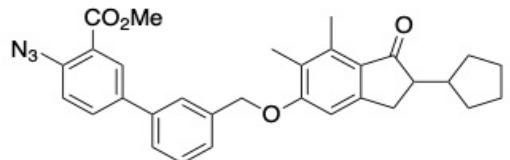


(\pm)-**S29**

^{13}C NMR (101 MHz, CDCl_3 with TMS)

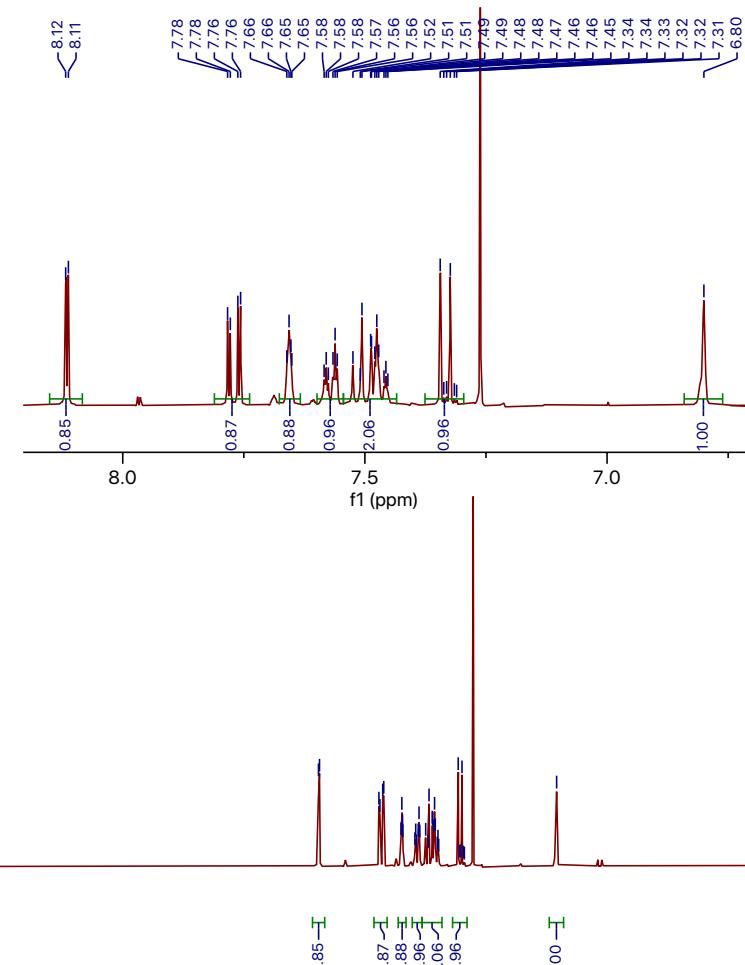
^{13}C NMR (101 MHz, CDCl_3) δ 208.7, 161.4, 155.1, 138.8, 136.3, 135.2, 134.0, 131.5, 131.5, 128.4, 128.1, 125.5, 105.4, 69.8, 51.2, 41.3, 30.8, 29.7, 28.2, 25.4, 25.2, 13.9, 11.2.





(±)-S31

^1H NMR (400 MHz, CDCl_3 with TMS)



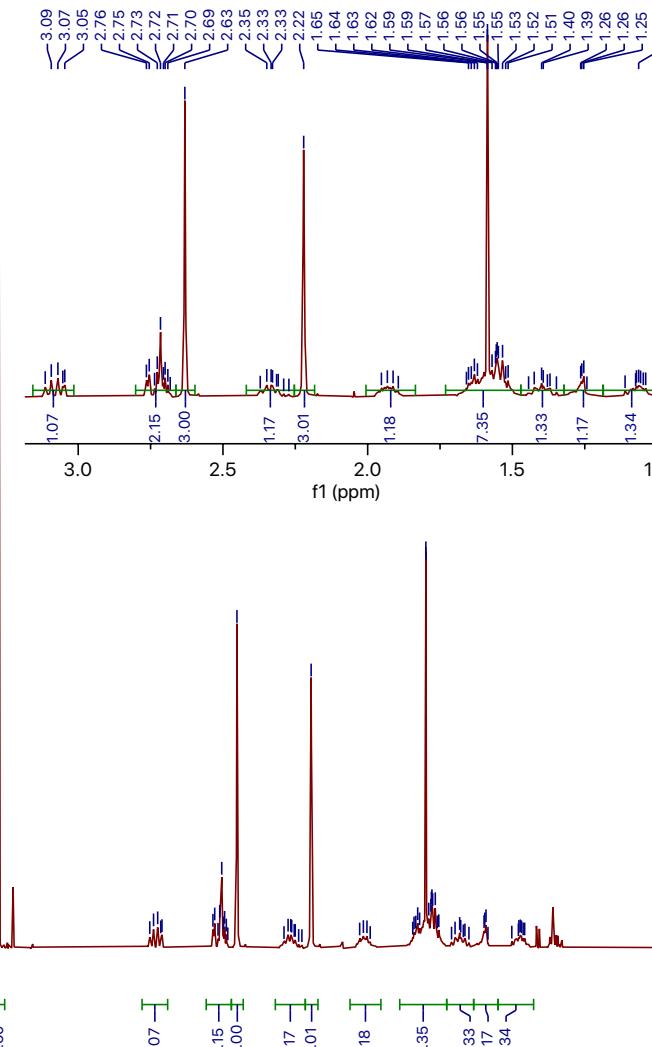
— 5.20 —

— 3.95 —

— 3.96 —

— 3.95 —

^1H NMR (400 MHz, Chloroform-*d*) δ 8.11 (d, $J = 2.3$ Hz, 1H), 7.77 (dd, $J = 8.5, 2.3$ Hz, 1H), 7.66 (q, $J = 1.3, 0.8$ Hz, 1H), 7.57 (dt, $J = 7.4, 1.8$ Hz, 1H), 7.54 – 7.43 (m, 2H), 7.33 (d, $J = 8.3$ Hz, 1H), 6.80 (s, 1H), 5.20 (s, 2H), 3.95 (s, 3H), 3.14 – 3.02 (m, 1H), 2.79 – 2.67 (m, 2H), 2.63 (s, 3H), 2.37 – 2.27 (m, 1H), 2.22 (s, 3H), 1.92 (q, $J = 7.7, 7.2$ Hz, 1H), 1.73 – 1.49 (m, 4H), 1.47 – 1.33 (m, 1H), 1.28 – 1.23 (m, 1H), 1.13 – 1.02 (m, 1H).



0.0 10.5 20.0 30.5 40.0 50.5 60.0 70.5 80.0 90.5 100.0 110.5 120.0 130.5 140.0 150.5 160.0 170.5 180.0 190.5 200.0 210.5 220.0 230.5 240.0 250.5 260.0 270.5 280.0 290.5 300.0 310.5 320.0 330.5 340.0 350.5 360.0 370.5 380.0 390.5 400.0 f1 (ppm)

— 2.60 —

— 1.07 —

— 2.15 —

— 3.00 —

— 1.17 —

— 3.01 —

— 1.18 —

— 7.35 —

— 1.33 —

— 1.17 —

— 1.34 —

— 1.06 —

— 1.05 —

—208.49

—165.76

—161.41

—154.95

—139.33
—139.30
—138.93
—137.60
—137.27
—131.60
—130.44
—129.39
—128.26
—126.61
—126.58
—125.64
—122.87
—120.49

—105.54

—69.96

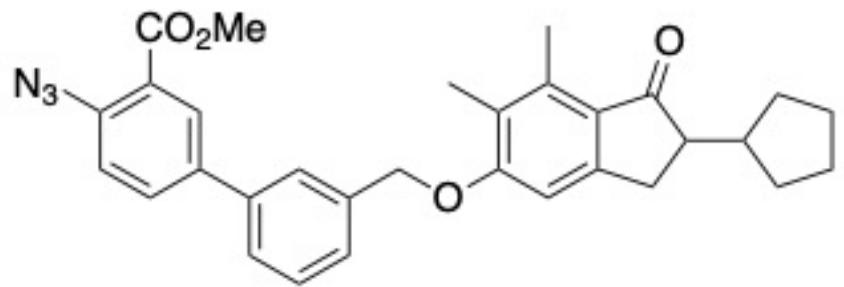
—52.60
—51.25

—41.33

—30.82
—29.68
—28.15
—25.46
—25.16

—13.89

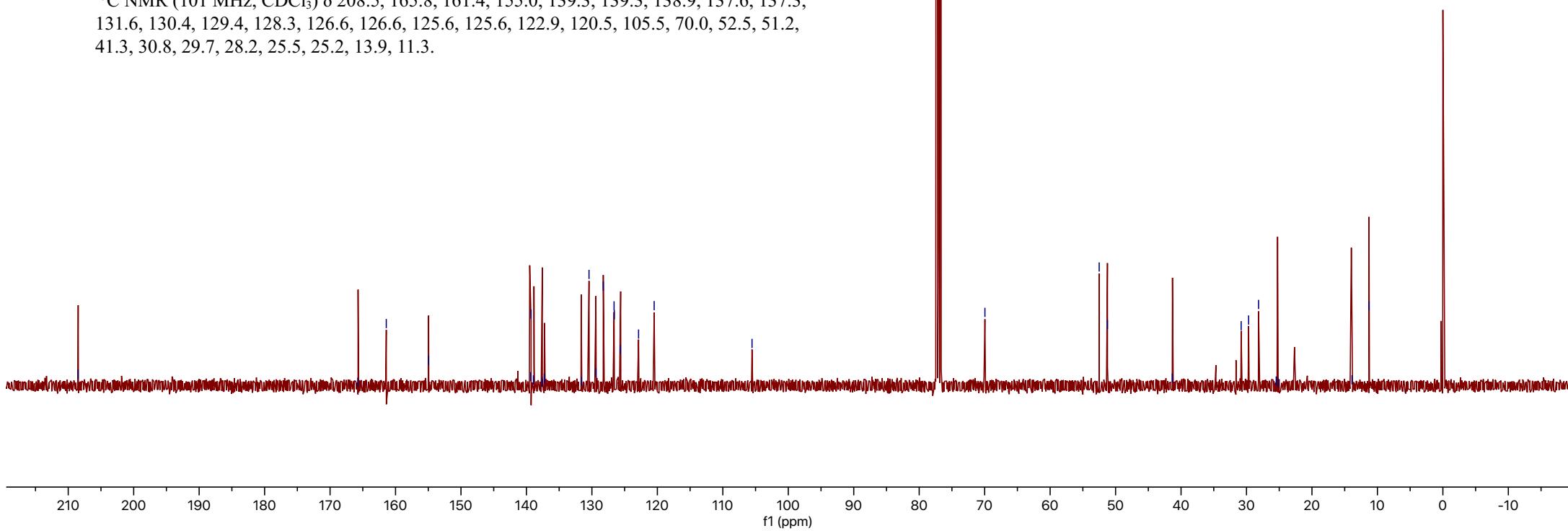
—11.28



(±)-S31

¹³C NMR (101 MHz, CDCl₃ with TMS)

¹³C NMR (101 MHz, CDCl₃) δ 208.5, 165.8, 161.4, 155.0, 139.3, 139.3, 138.9, 137.6, 137.3, 131.6, 130.4, 129.4, 128.3, 126.6, 126.6, 125.6, 125.6, 122.9, 120.5, 105.5, 70.0, 52.5, 51.2, 41.3, 30.8, 29.7, 28.2, 25.5, 25.2, 13.9, 11.3.

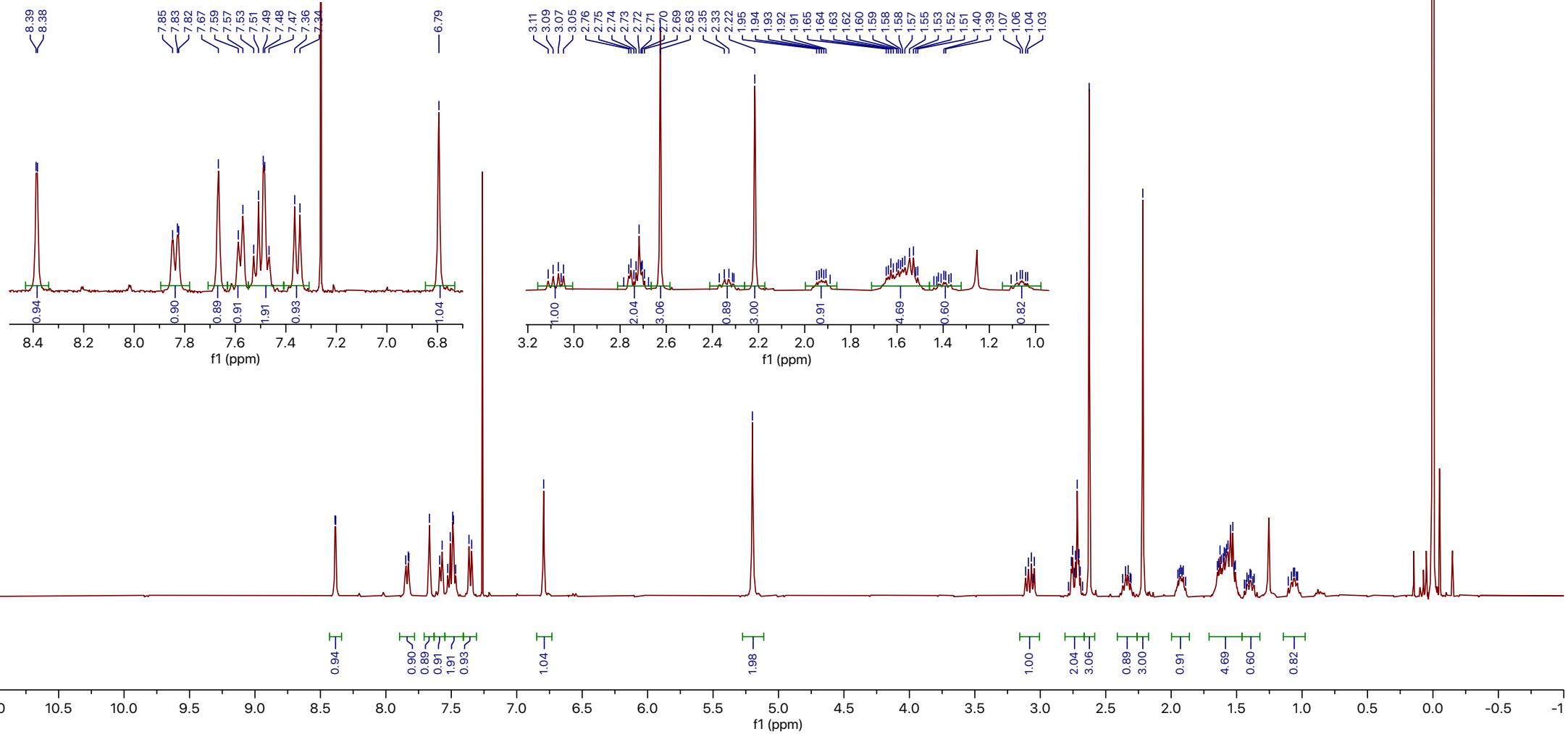




¹H NMR (400 MHz, Chloroform-*d*) δ 8.39 (d, *J* = 2.2 Hz, 1H), 7.90 – 7.78 (m, 1H), 7.67 (s, 1H), 7.58 (d, *J* = 7.3 Hz, 1H), 7.55 – 7.44 (m, 2H), 7.35 (d, *J* = 8.3 Hz, 1H), 6.79 (s, 1H), 5.20 (s, 2H), 3.08 (dd, *J* = 17.8, 8.8 Hz, 1H), 2.78 – 2.67 (m, 2H), 2.63 (s, 3H), 2.40 – 2.28 (m, 1H), 2.22 (s, 3H), 2.00 – 1.86 (m, 1H), 1.71 – 1.46 (m, 5H), 1.40 (qd, *J* = 9.4, 8.6, 4.5 Hz, 1H), 1.13 – 1.01 (m, 1H).

(±)-15

¹H NMR (400 MHz, CDCl₃ with TMS)



—208.57

—161.45

—155.02

—138.87
—137.88
—132.69
—131.87
—129.46
—128.25
—126.79
—126.58
—125.66
—125.59
—119.94

—105.55

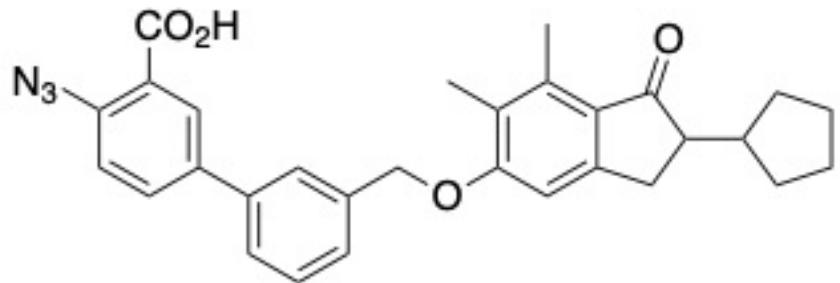
—69.91

—51.30

—41.31

—30.82
—29.67
—28.14
—25.44
—25.26

—14.00
—11.32



(\pm)-15

^{13}C NMR (101 MHz, CDCl_3 with TMS)

^{13}C NMR (101 MHz, CDCl_3) δ 208.6, 161.5, 155.0, 139.1, 138.9, 138.9, 137.9, 137.7, 137.7, 132.7, 131.9, 129.5, 129.5, 128.2, 126.8, 126.6, 125.7, 125.6, 119.9, 105.5, 69.9, 51.3, 41.3, 30.8, 29.7, 28.1, 25.4, 25.3, 14.0, 11.3.

