

SUPPORTING INFORMATION

Synthesis of 6-SF₅-Indazoles and SF₅-Analog of Gamendazole

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Gainesville, FL 32611-7200 (USA)

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1. General experimental methods

Chemical precursors were purchased from Acros, Aldrich, Oakwood Chemical and Combi-Blocks and were used as received from the commercial suppliers without further purification unless mentioned otherwise. All solvents were purchased from Acros, Aldrich or Fischer Scientific and were used as received from the commercial supplier without further purification unless mentioned otherwise.

Reactions were monitored by thin layer chromatography or/and ^{19}F NMR. TLC was performed on Sorbtech silica gel UV254 polyester backed 200 μm thickness TLC plates and visualized with UV light. Chromatographic purification was done using flash chromatography technique on Sorbtech silica gel 60 \AA 40-63 μm (230-400 mesh) at positive nitrogen pressure.

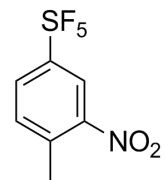
NMR spectra were recorded on Varian Mercury (300 MHz) or Varian Inova (500 MHz) instruments. All measurements were carried out at 25 $^{\circ}\text{C}$. Chemical shifts (δ) are reported in ppm and are referenced to the residual solvent signal as internal standard (CDCl_3 at 7.26 ppm for ^1H NMR, and 77.16 ppm for ^{13}C NMR; $(\text{CD}_3)_2\text{CO}$ at 2.05 ppm for ^1H NMR and 29.84 ppm for ^{13}C NMR). ^{19}F NMR spectra are referenced to CFCl_3 as internal standard. Coupling constants (J) are reported in Hz. Multiplicities are abbreviated as follows: s (singlet), d (doublet), t (triplet), q (quartet), p (pentuplet), m (multiplet or unresolved) and br (broad signal). High resolution mass spectrometric measurements were performed by the University of Florida Department of Chemistry Mass Spectrometry Services. Melting points were determined using Uni-Melt Thomas Hoover capillary melting point apparatus.

151.97
151.81
151.66
148.64

137.83
133.44
130.06
130.03
129.99

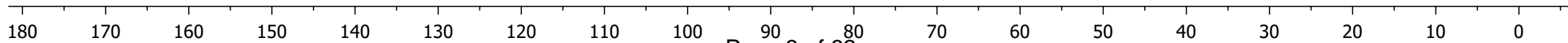
77.41
77.16
76.91

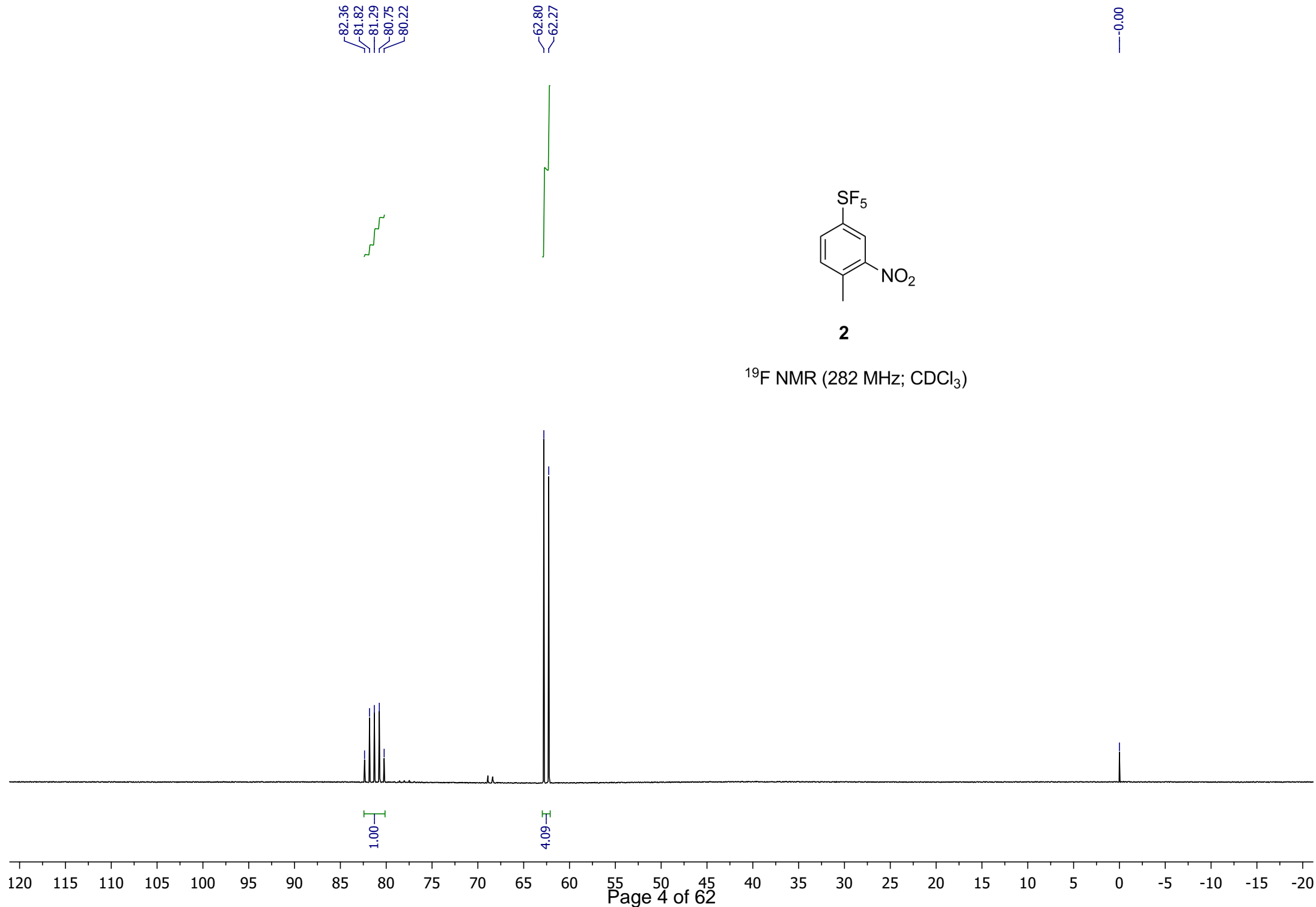
20.42



2

¹³C NMR (125 MHz; CDCl₃)

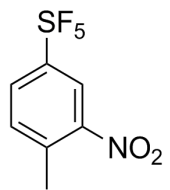




8.39
8.39
7.90
7.89
7.87
7.86
7.51
7.48
7.26

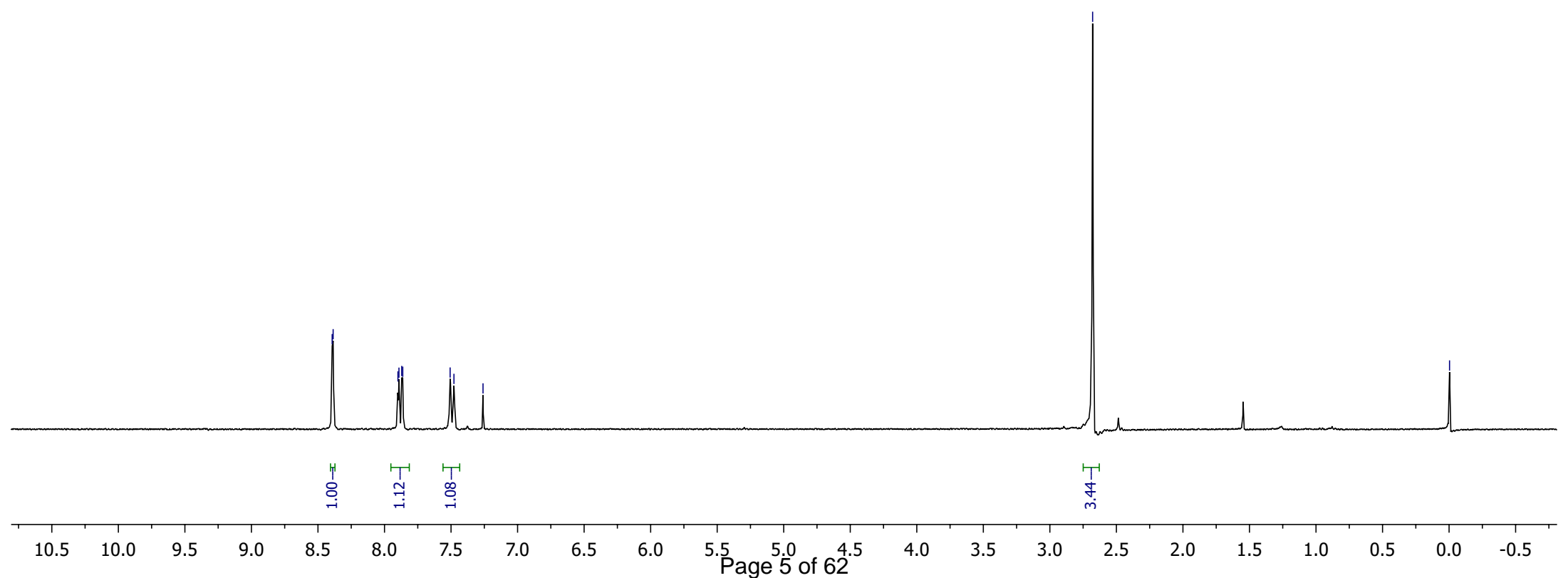
2.68

-0.00



2

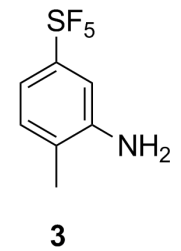
¹H NMR (300 MHz; CDCl₃)



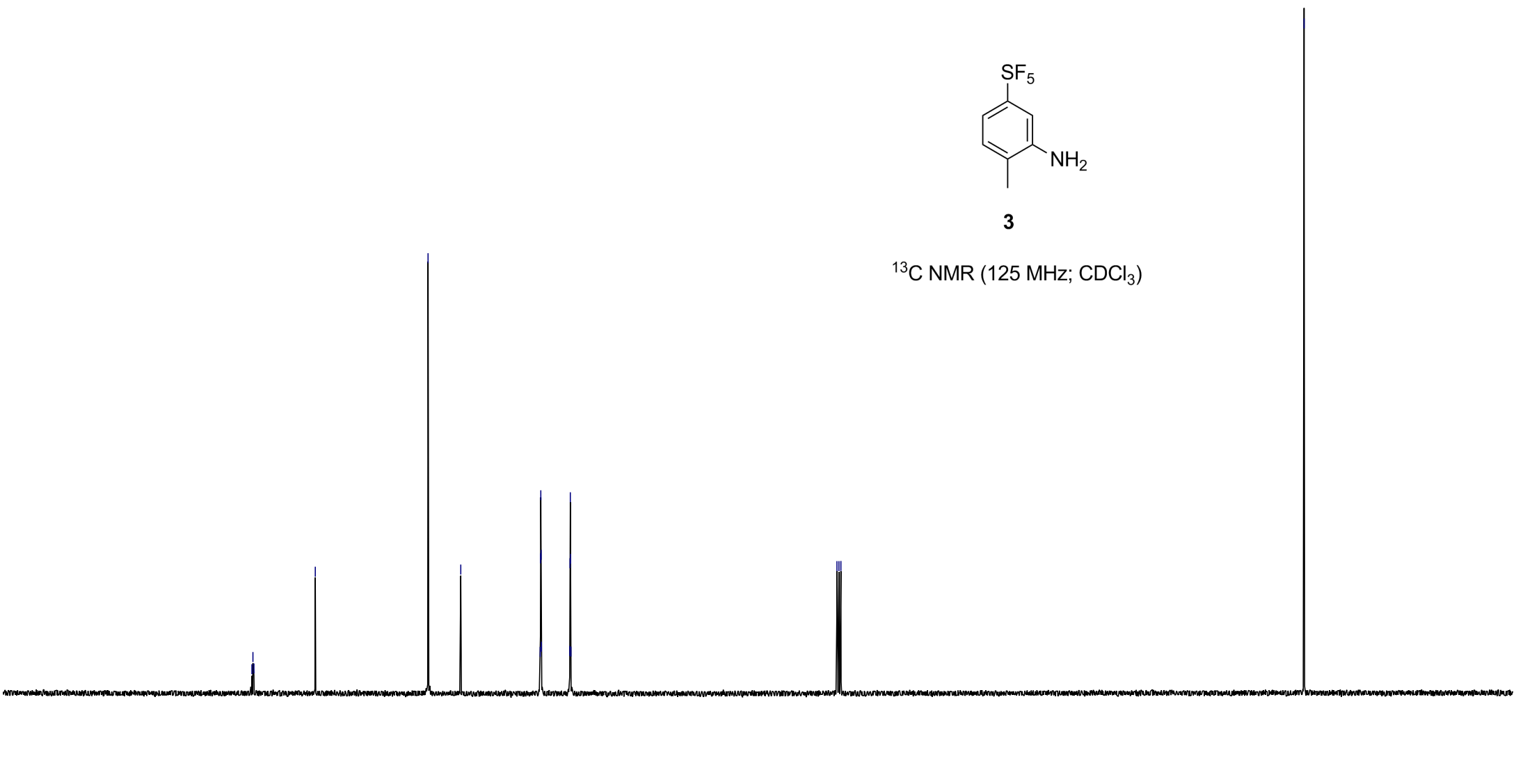
152.99
152.85
152.73
— 144.80
— 130.23
— 126.01
115.74
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111.93
111.89
111.86
111.82
111.78

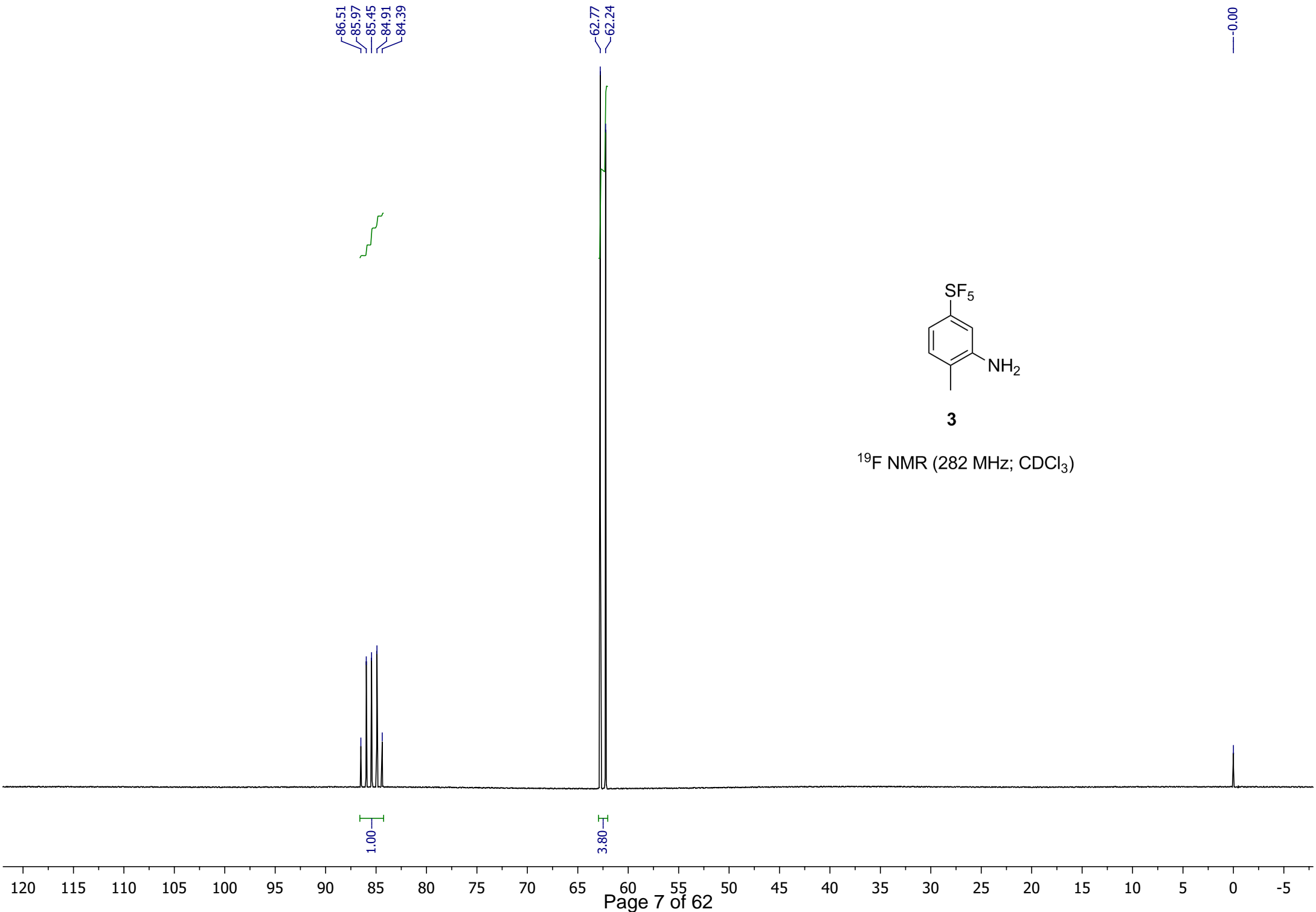
77.41
77.16
76.91

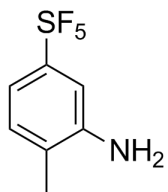
— 17.07



¹³C NMR (125 MHz; CDCl₃)

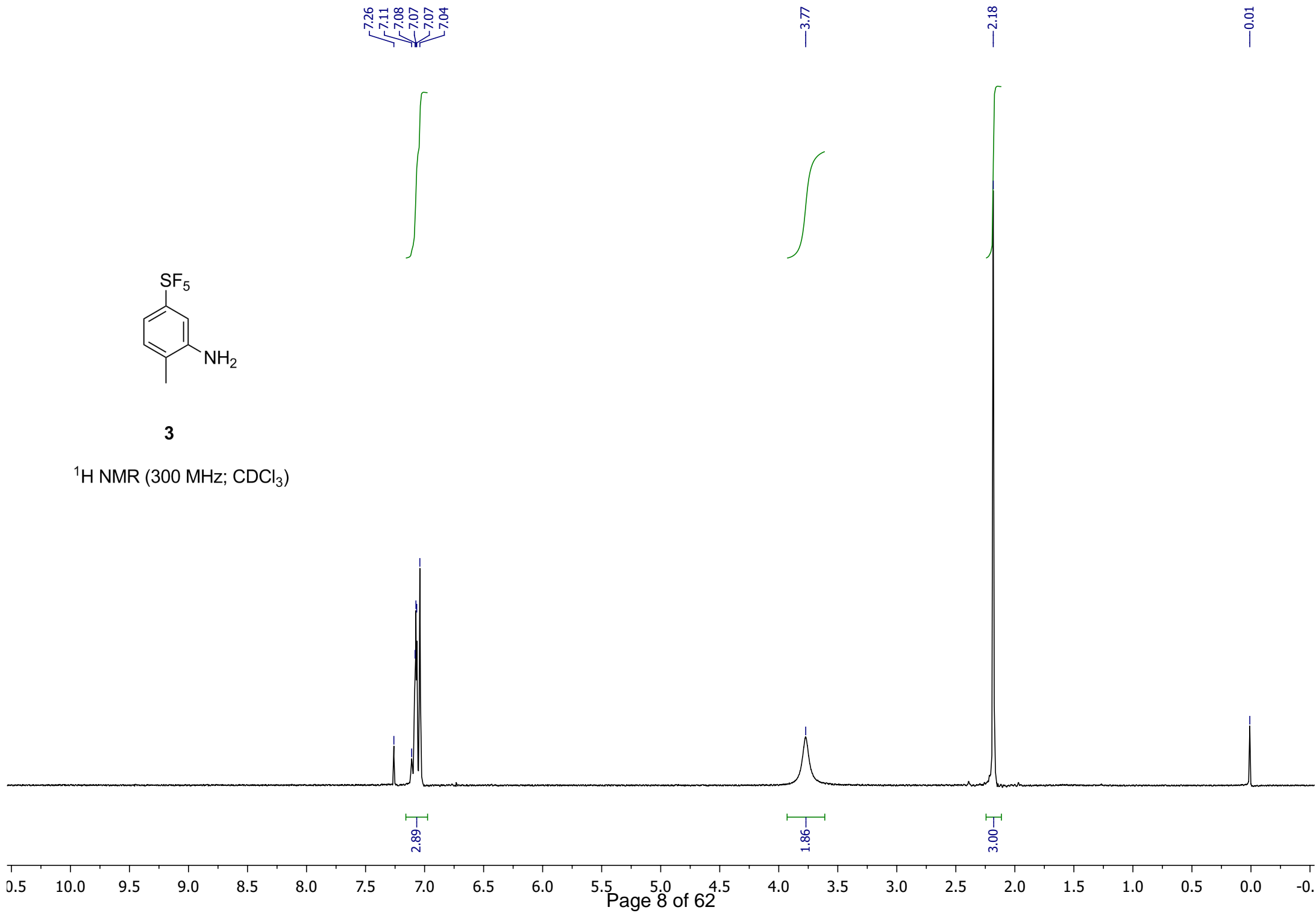






3

¹H NMR (300 MHz; CDCl₃)



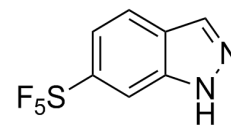
—206.57

152.93
152.80
152.67
152.54
152.41

—139.62
—134.95

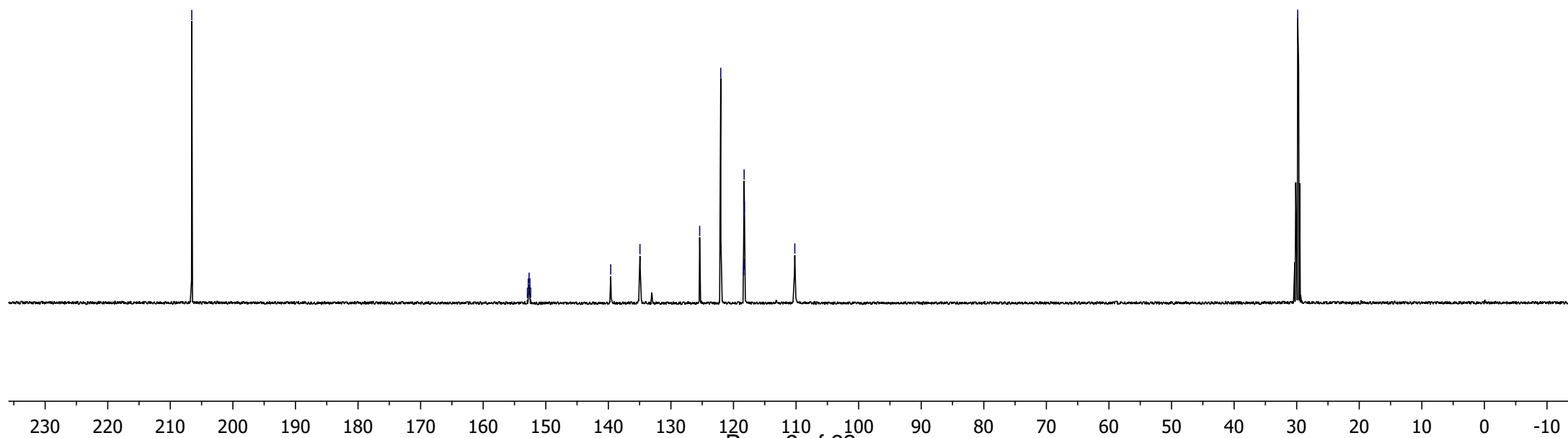
125.40
122.04
118.37
118.34
118.30
118.27
118.23
—110.20

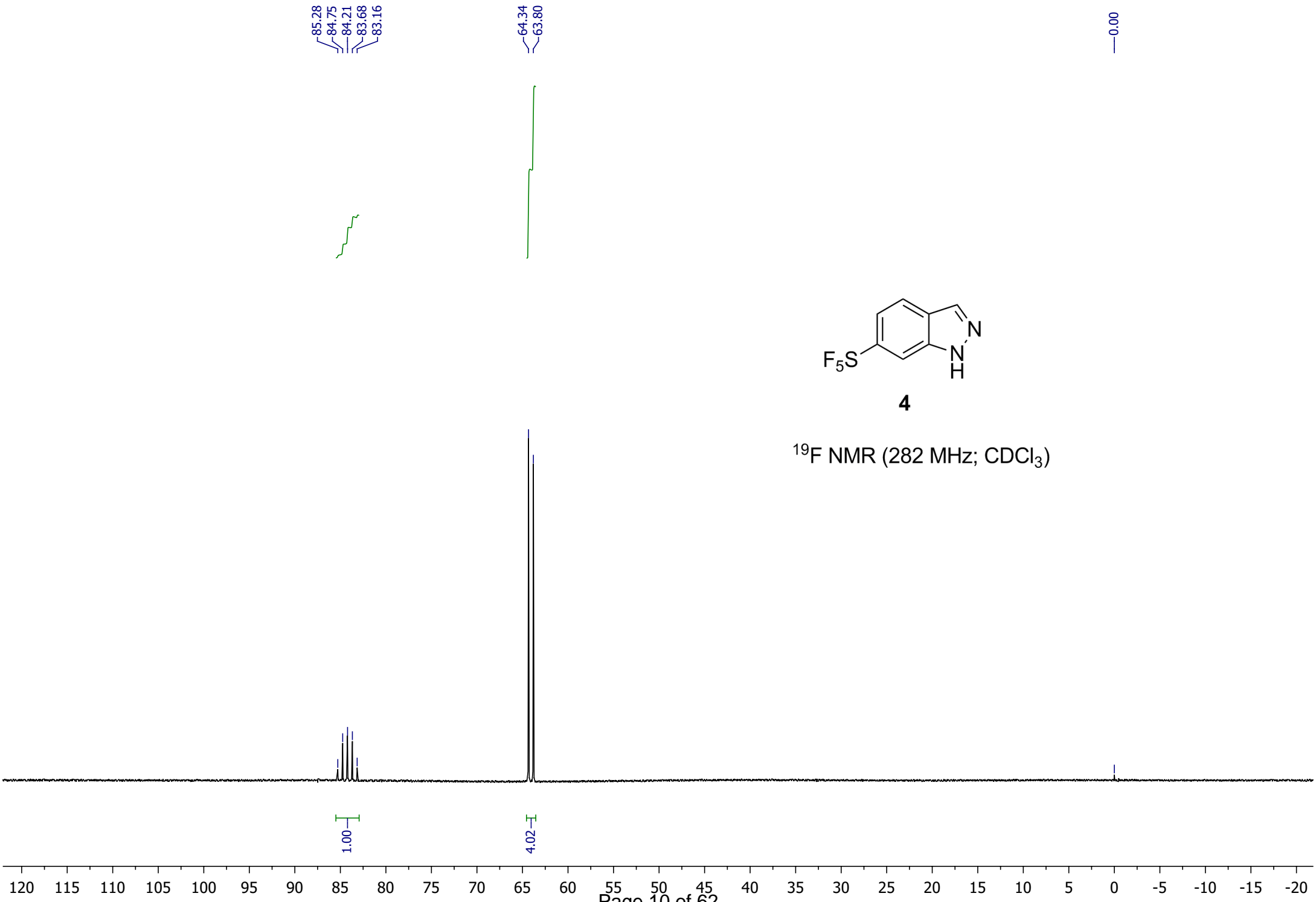
—29.84

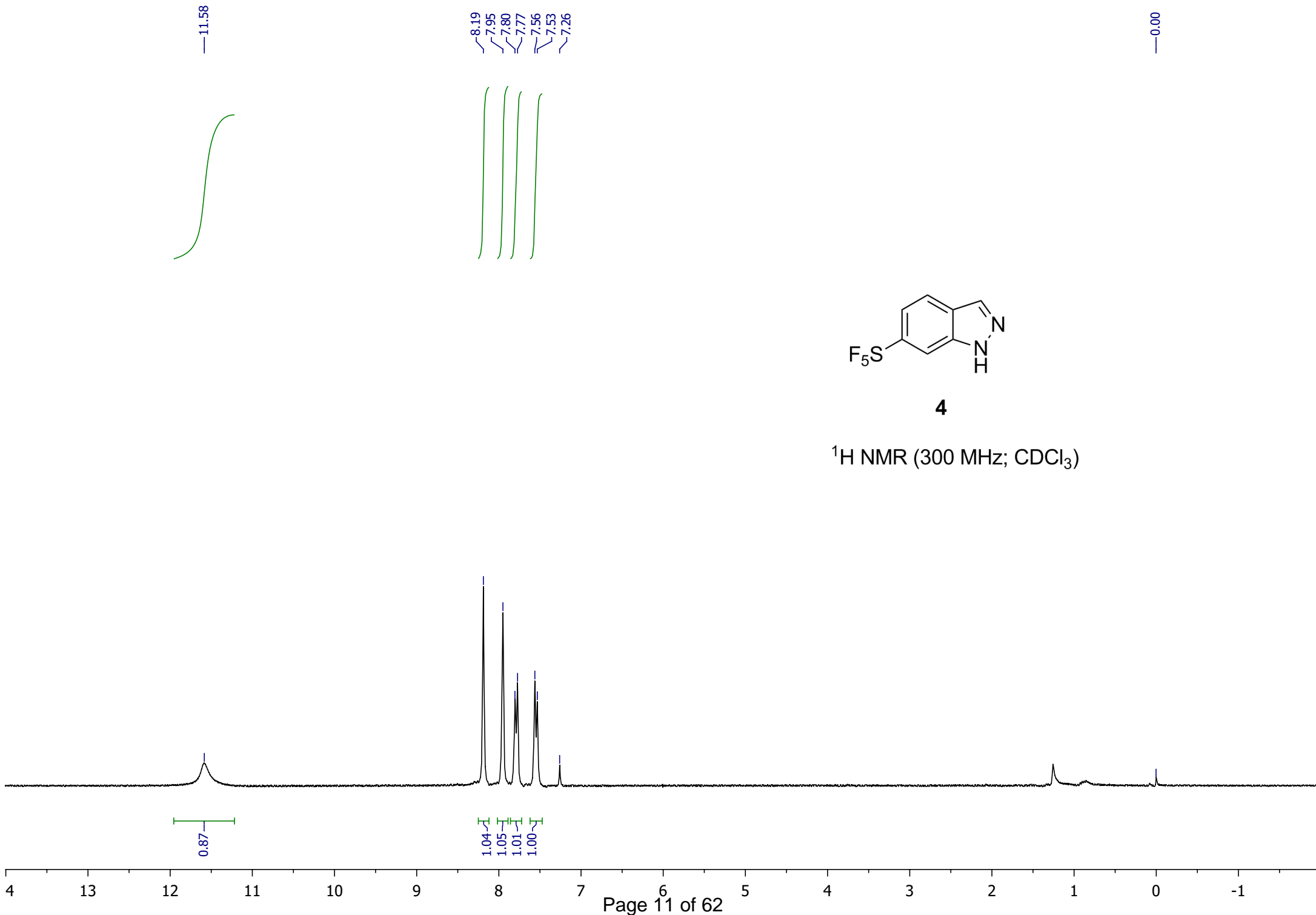


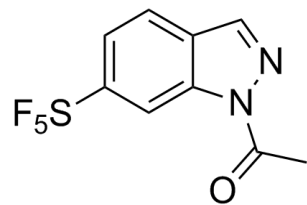
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¹³C NMR (125 MHz; (CD₃)₂CO)



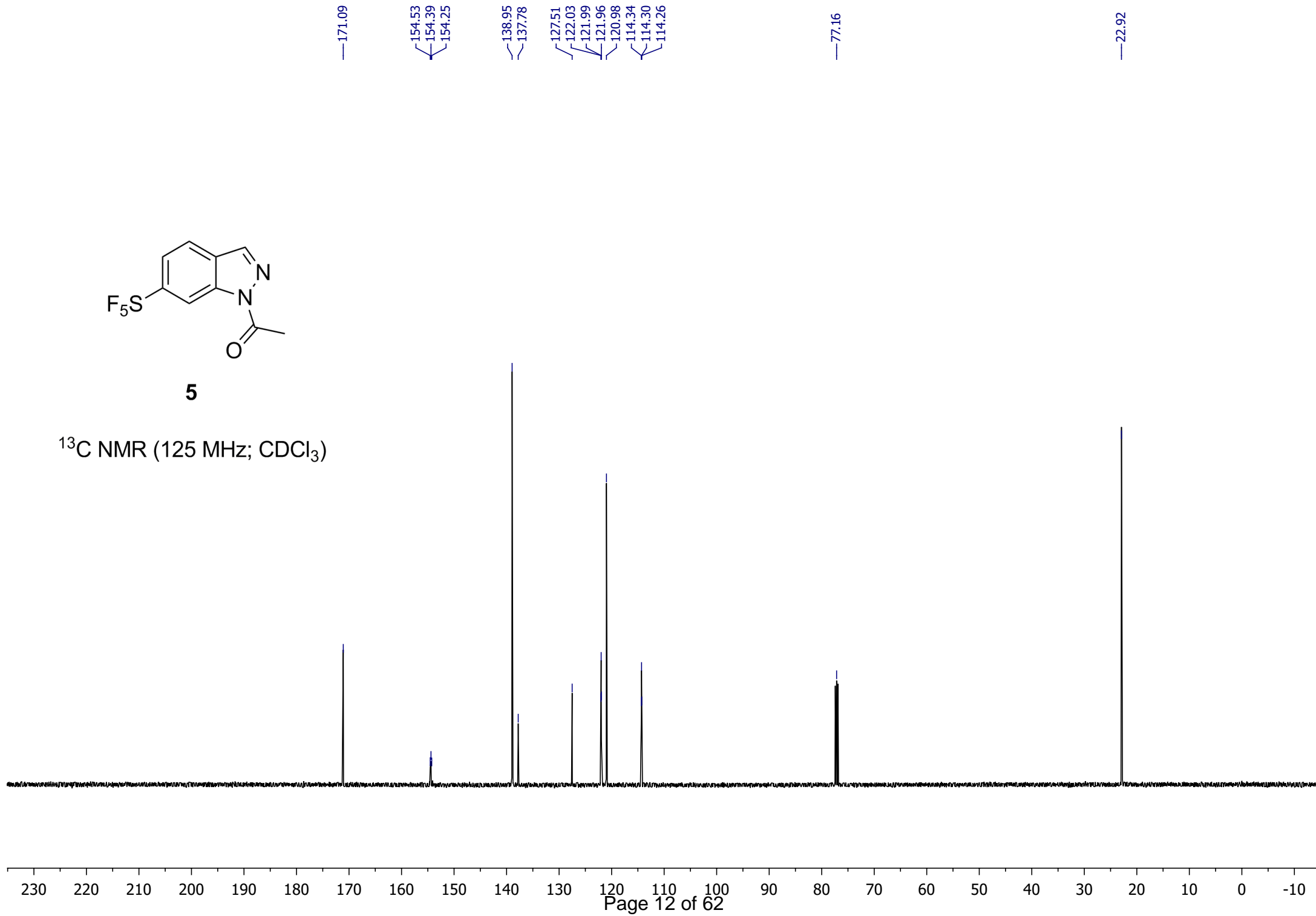


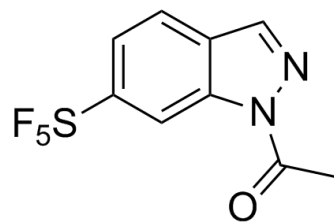




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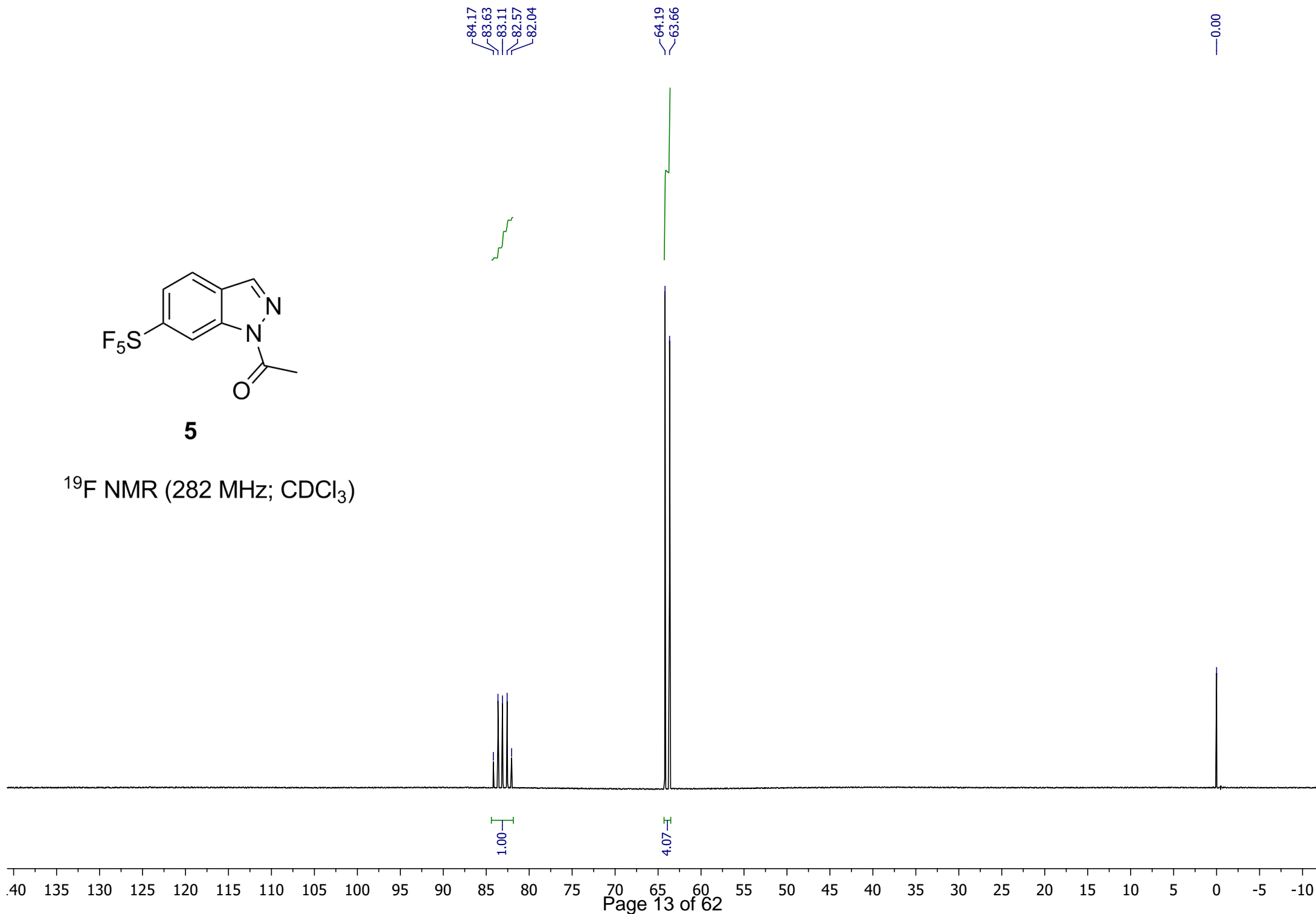
^{13}C NMR (125 MHz; CDCl_3)

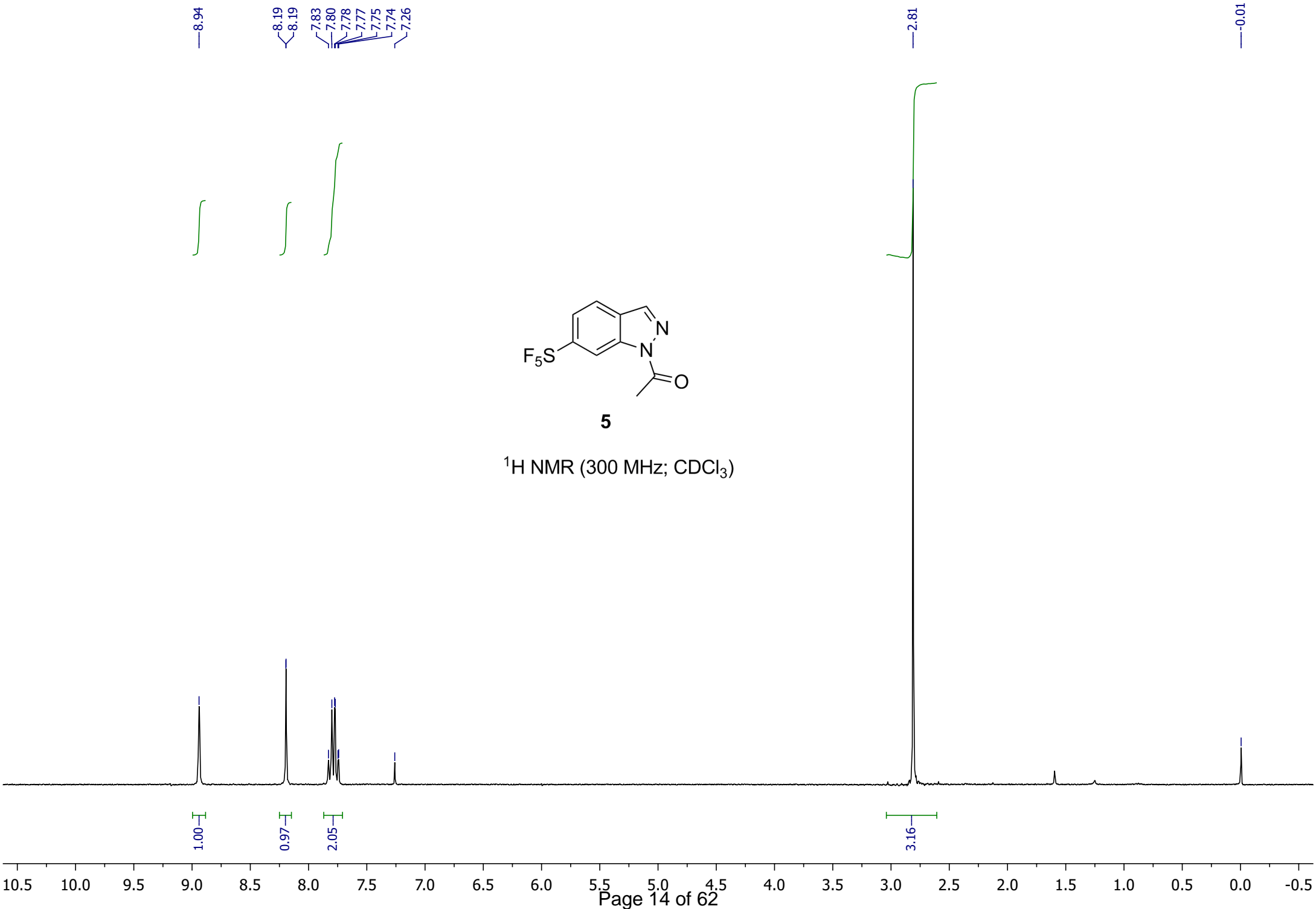




5

^{19}F NMR (282 MHz; CDCl_3)





—207.16

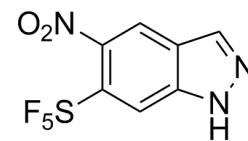
142.91
142.75
142.59
141.92
139.02
136.33

—123.98

—119.21

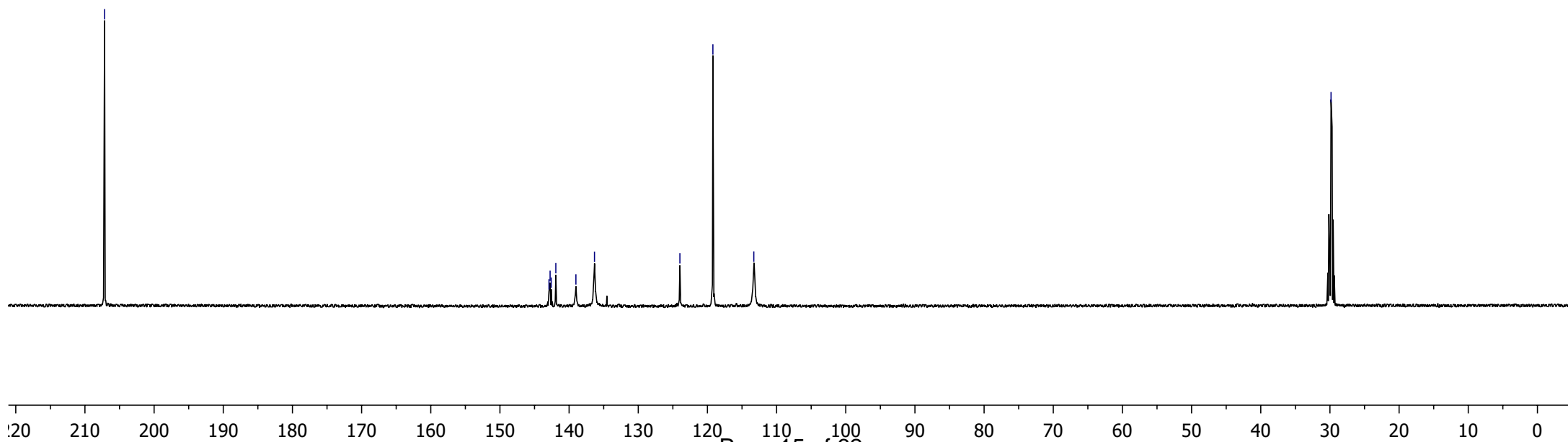
—113.30

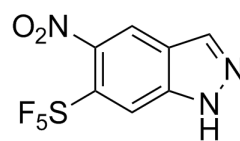
—29.84



6

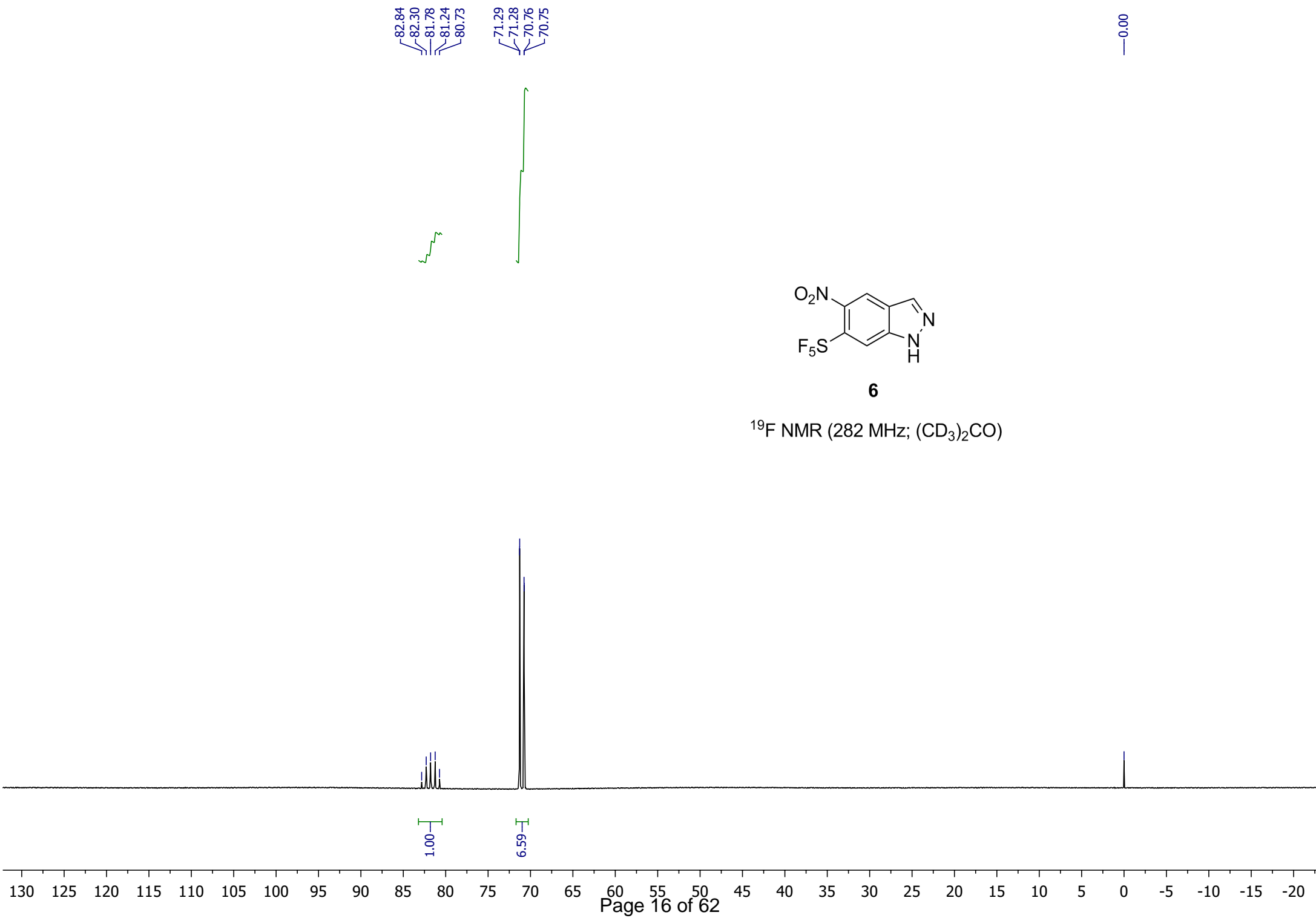
^{13}C NMR (125 MHz; $(\text{CD}_3)_2\text{CO}$)

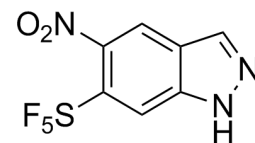




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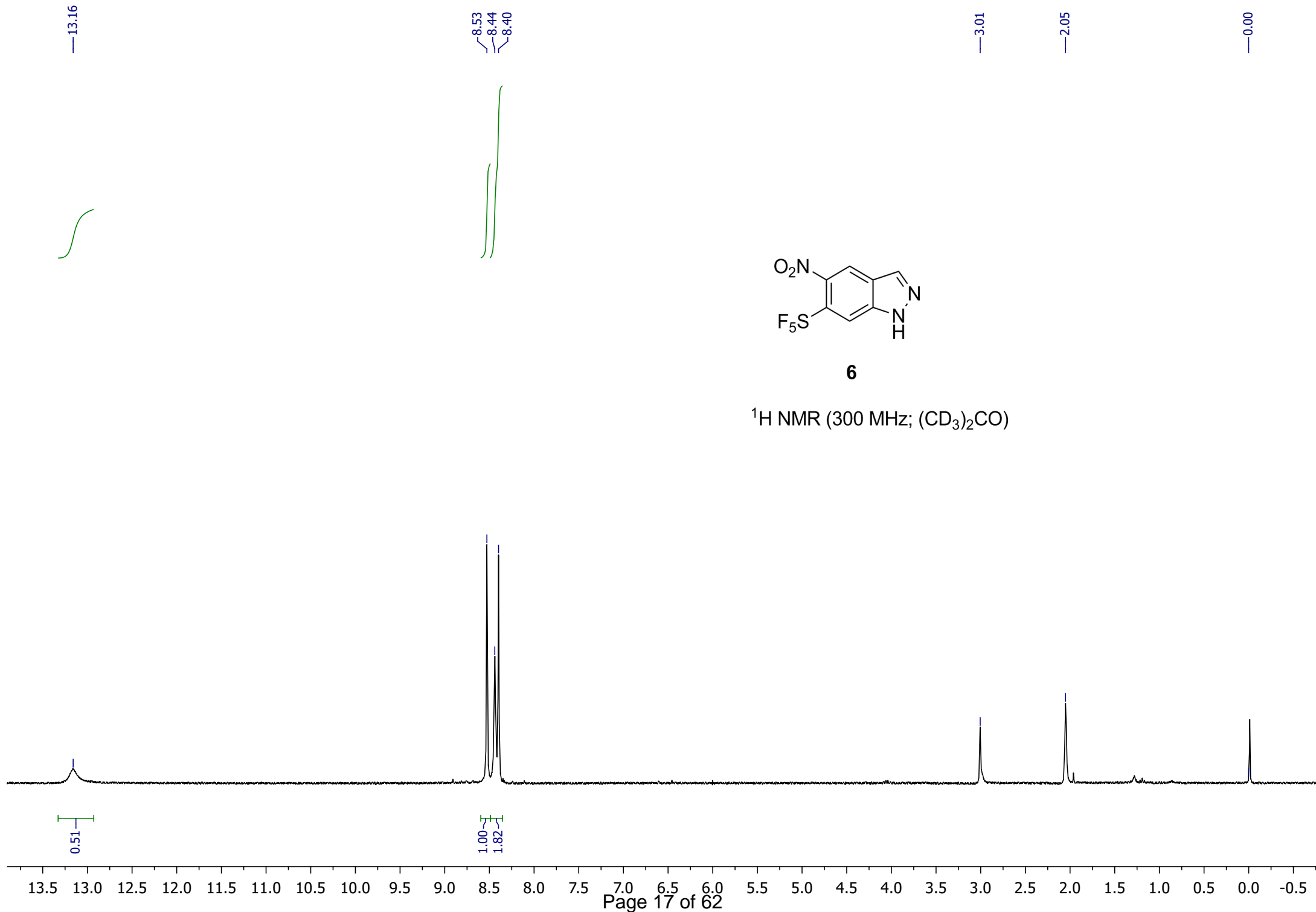
^{19}F NMR (282 MHz; $(\text{CD}_3)_2\text{CO}$)





6

^1H NMR (300 MHz; $(\text{CD}_3)_2\text{CO}$)



—206.60

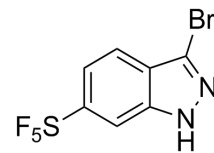
153.75
153.61
153.48

—140.57

124.79
122.12
121.04
119.28

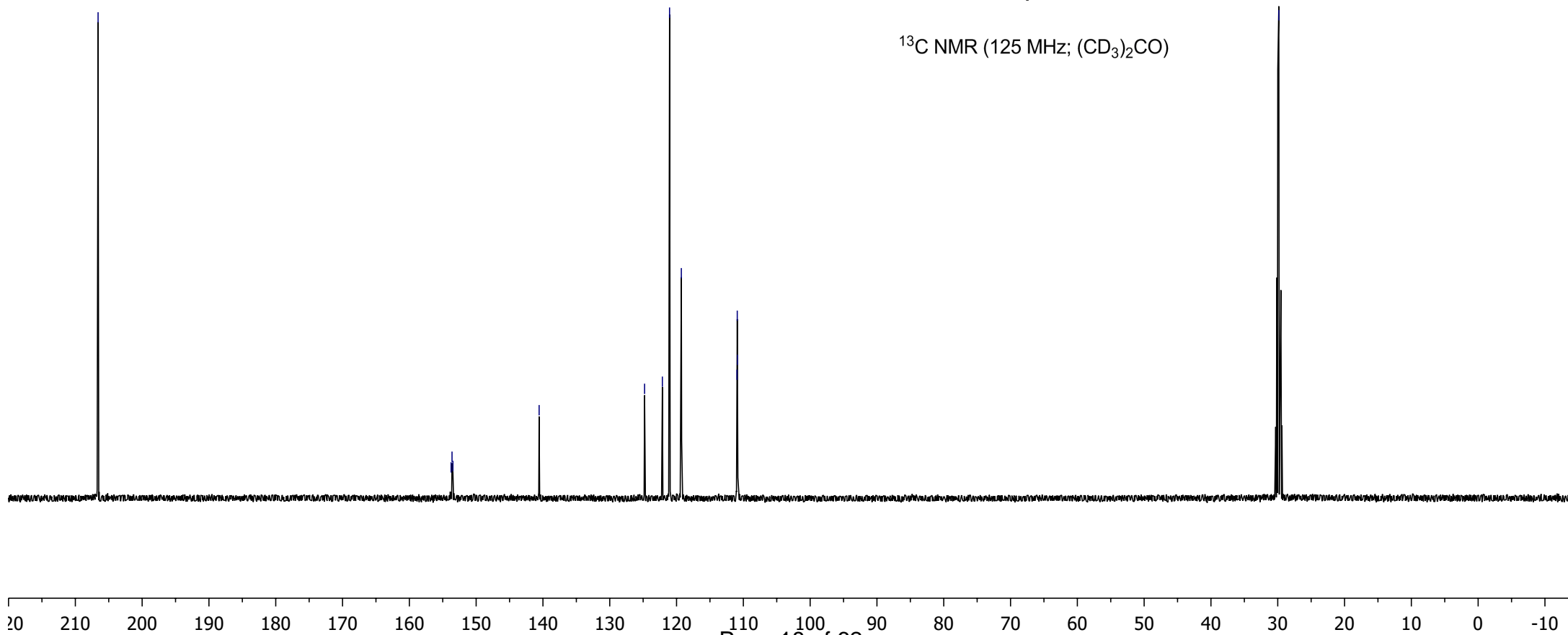
110.95
110.91
110.87

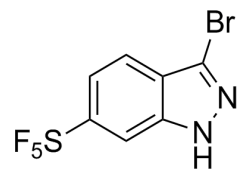
—29.84



7

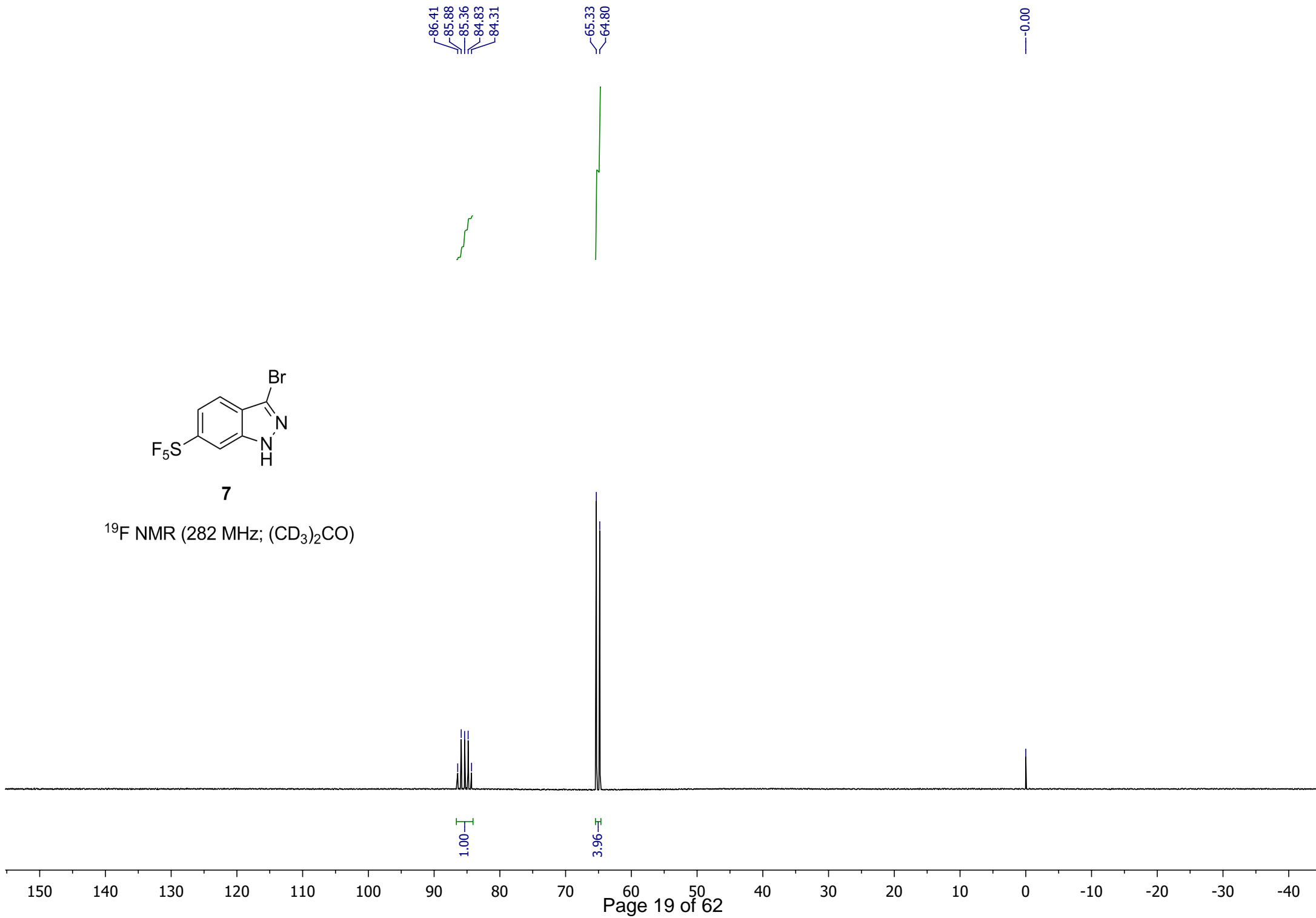
¹³C NMR (125 MHz; (CD₃)₂CO)

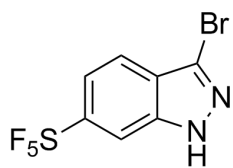




7

^{19}F NMR (282 MHz; $(\text{CD}_3)_2\text{CO}$)





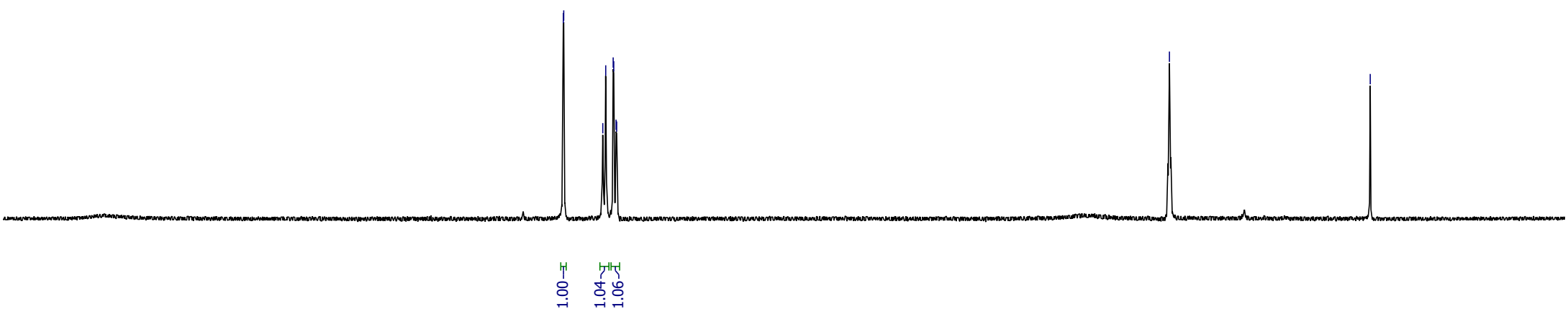
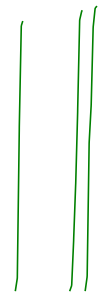
7

^1H NMR (300 MHz; $(\text{CD}_3)_2\text{CO}$)

8.26
8.26
7.86
7.83
7.75
7.74
7.72
7.71

2.05

0.01



1.00
1.04
1.06

151.63
151.47
151.30
150.52

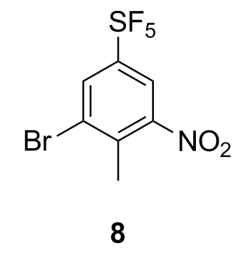
136.84
133.55
133.51
133.47

127.41

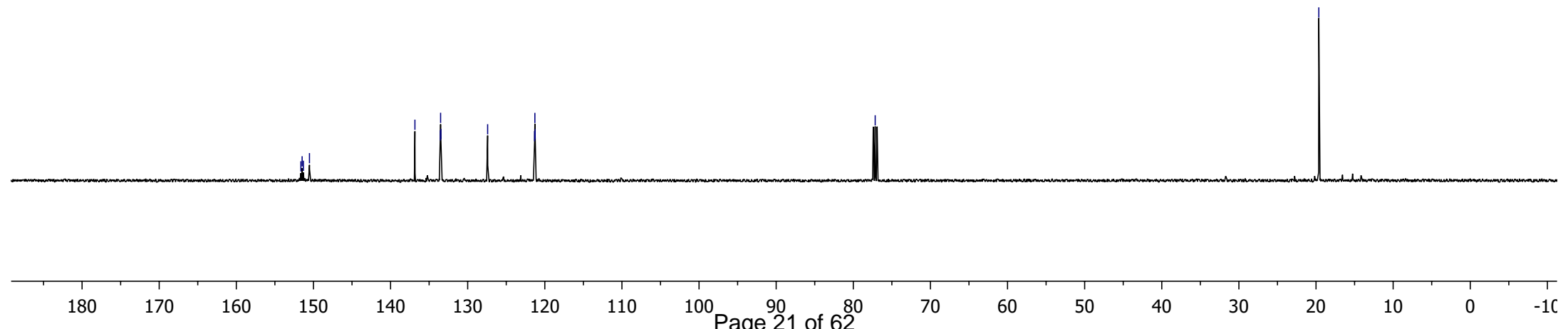
121.33
121.29
121.25

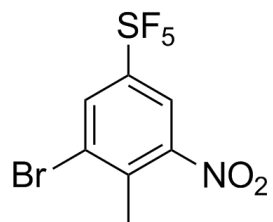
77.16

19.64



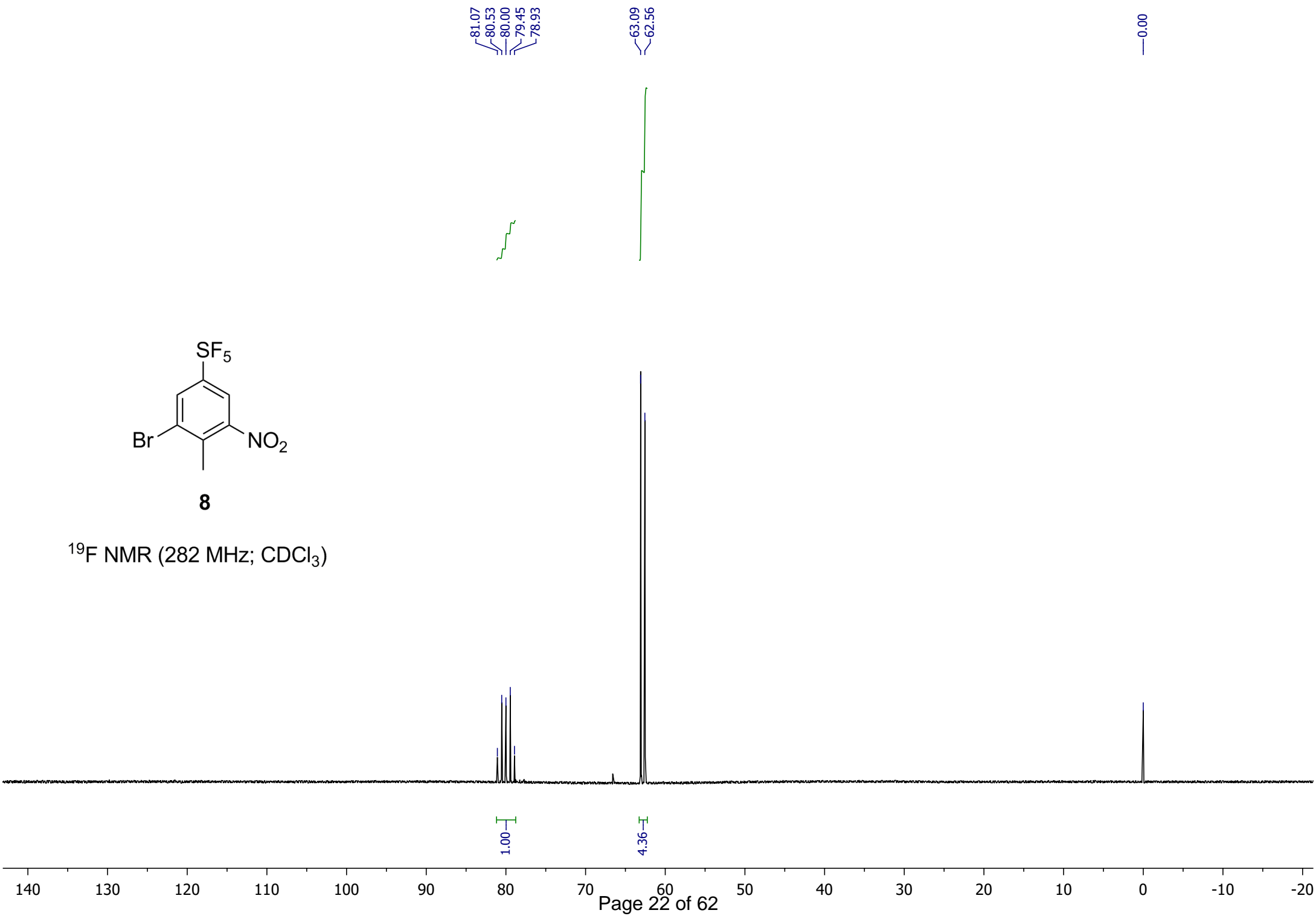
¹³C NMR (125 MHz; CDCl₃)





8

¹⁹F NMR (282 MHz; CDCl₃)

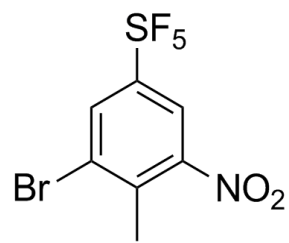


8.18
8.17
8.14
8.14

7.26

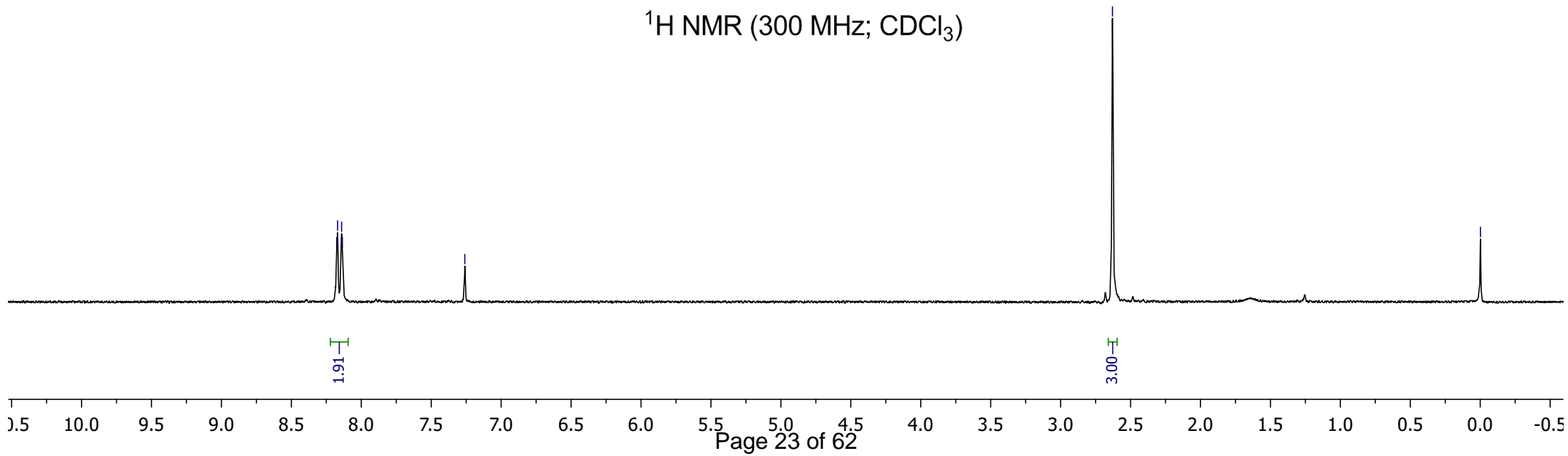
2.63

0.00



8

¹H NMR (300 MHz; CDCl₃)



152.56
152.42
152.28

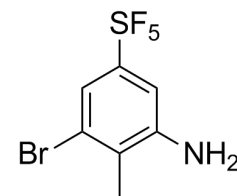
145.65

125.50
125.05
119.65
119.61
119.57

111.13
111.10
111.06

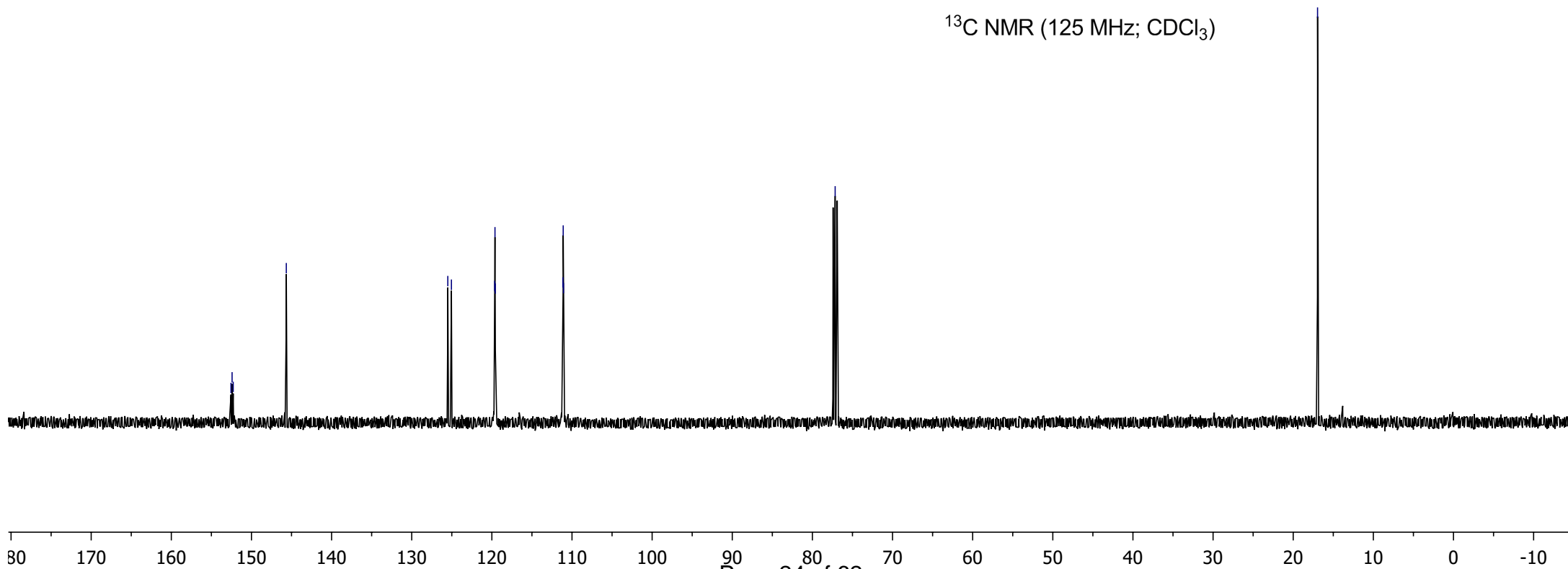
77.16

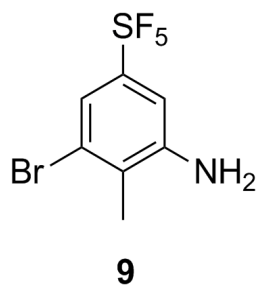
16.96



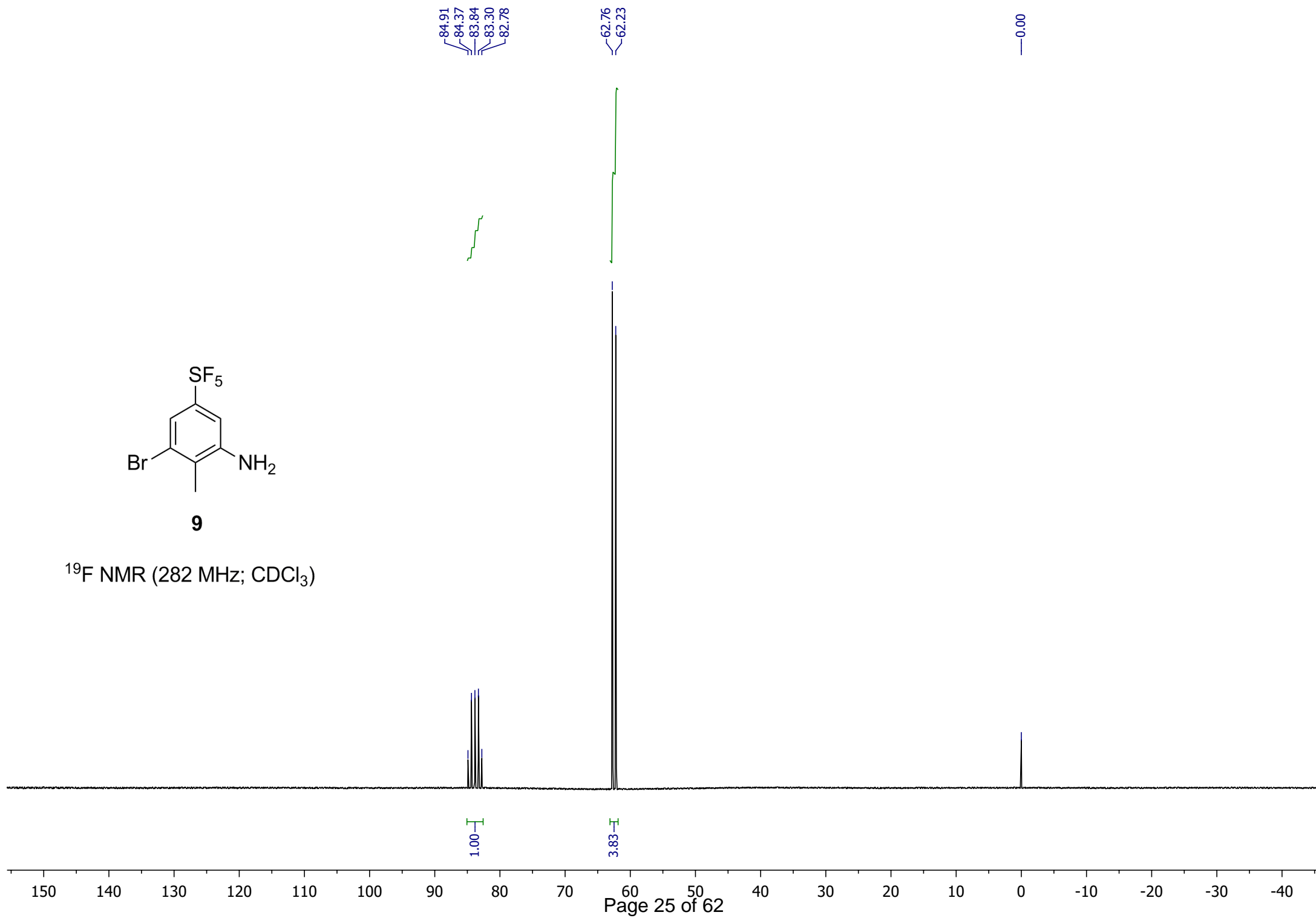
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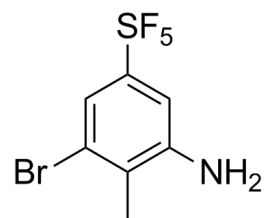
^{13}C NMR (125 MHz; CDCl_3)





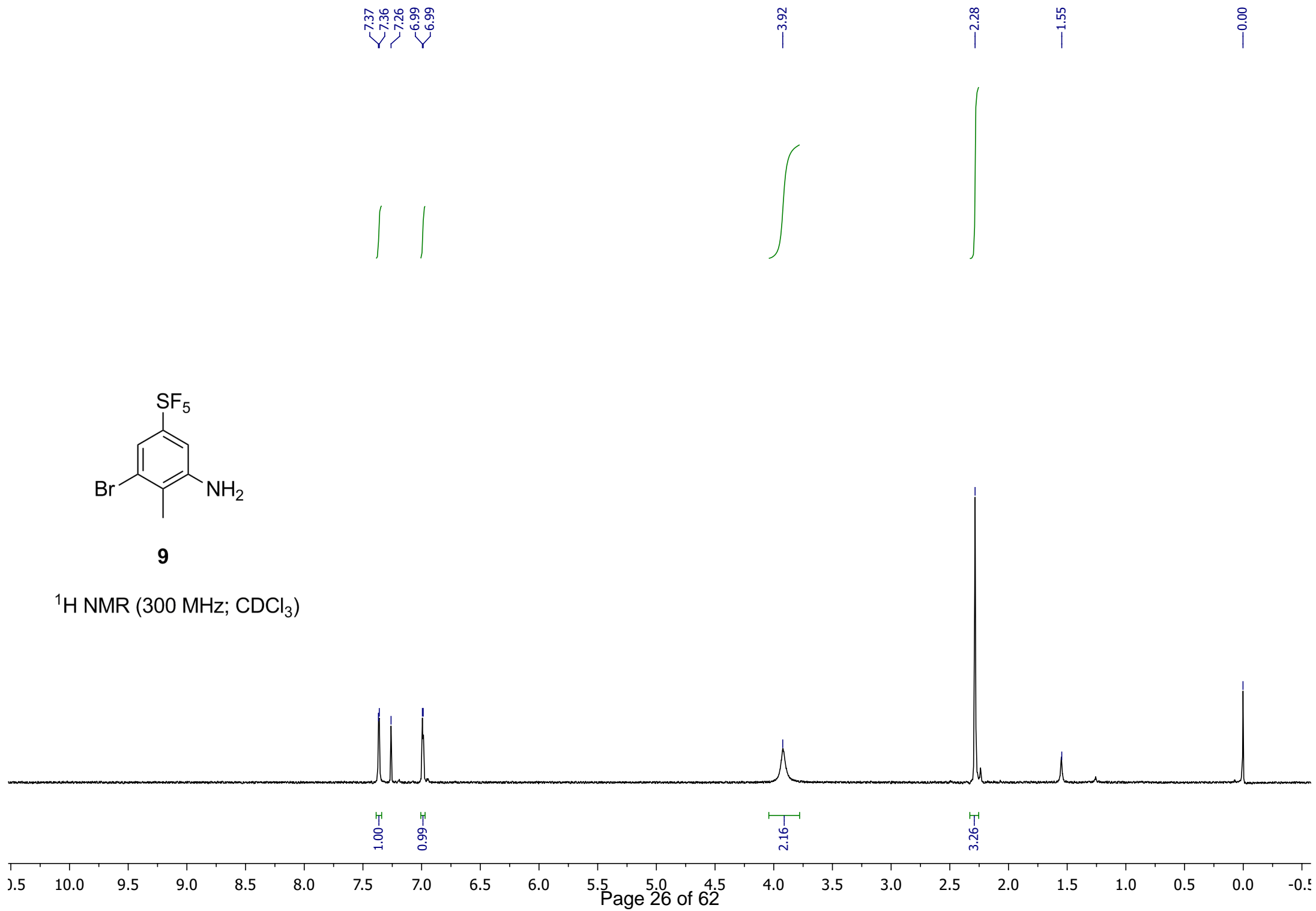
^{19}F NMR (282 MHz; CDCl_3)





9

^1H NMR (300 MHz; CDCl_3)



—206.41

152.65
152.51
152.37

—139.79

—134.69

126.35

120.86

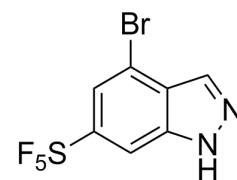
120.82

120.79

—114.40

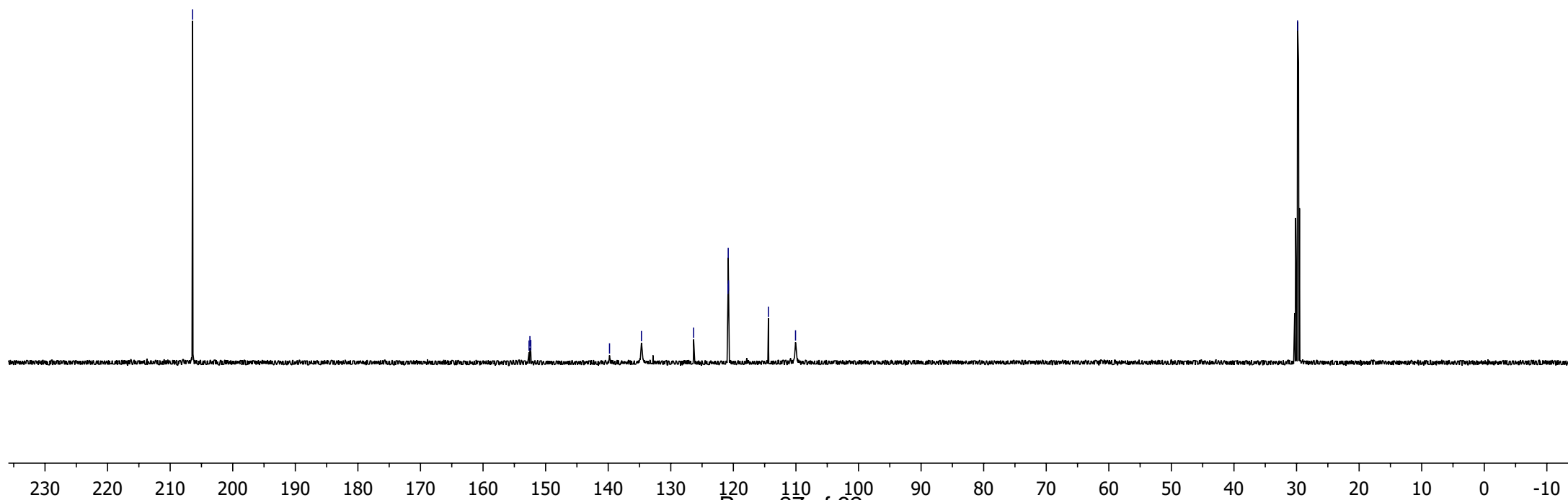
—110.06

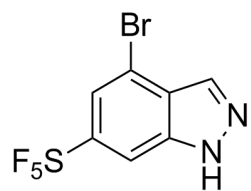
—29.84



10

^{13}C NMR (125 MHz; $(\text{CD}_3)_2\text{CO}$)





10

^{19}F NMR (282 MHz; $(\text{CD}_3)_2\text{CO}$)

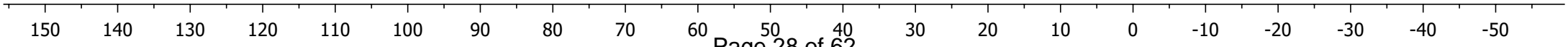
85.82
85.28
84.76
84.23
83.71

65.79
65.26

-0.00

1.00

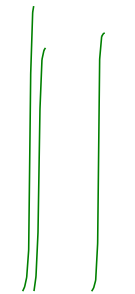
3.97



—13.05



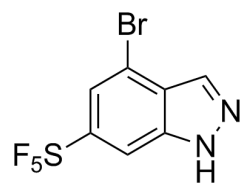
8.27
8.21
7.81
7.81



—2.87

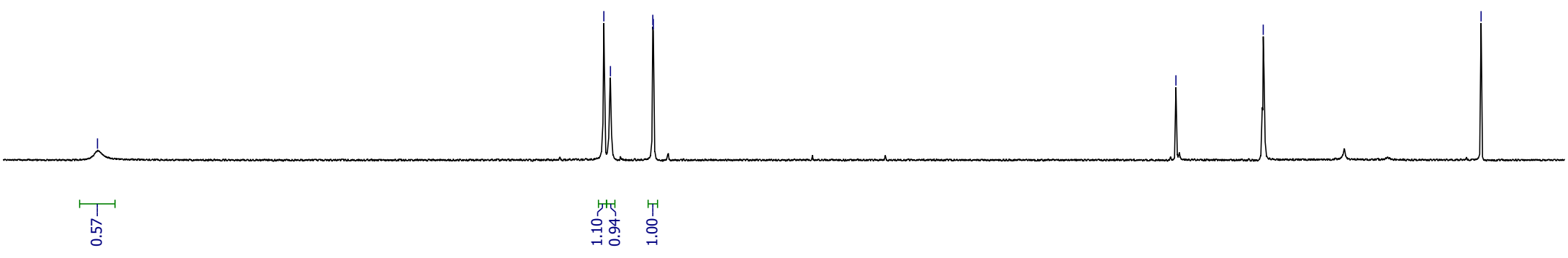
—2.05

—0.01



10

¹H NMR (300 MHz; (CD₃)₂CO)

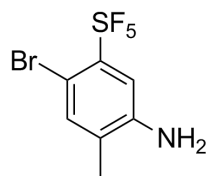


0.57

1.10

0.94

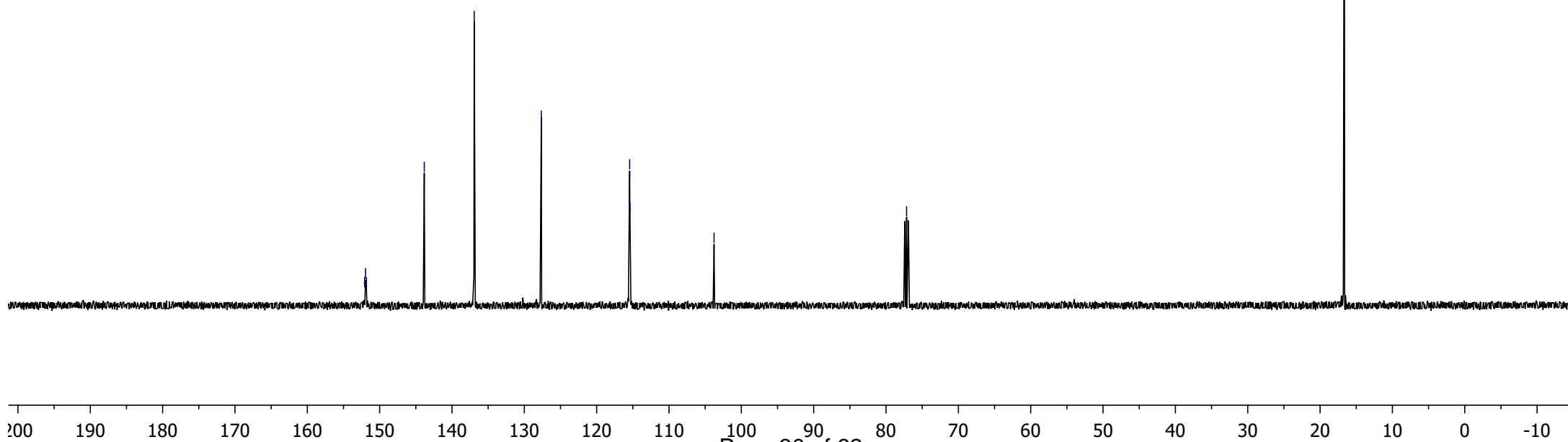
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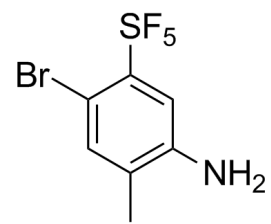


11

^{13}C NMR (125 MHz; CDCl_3)

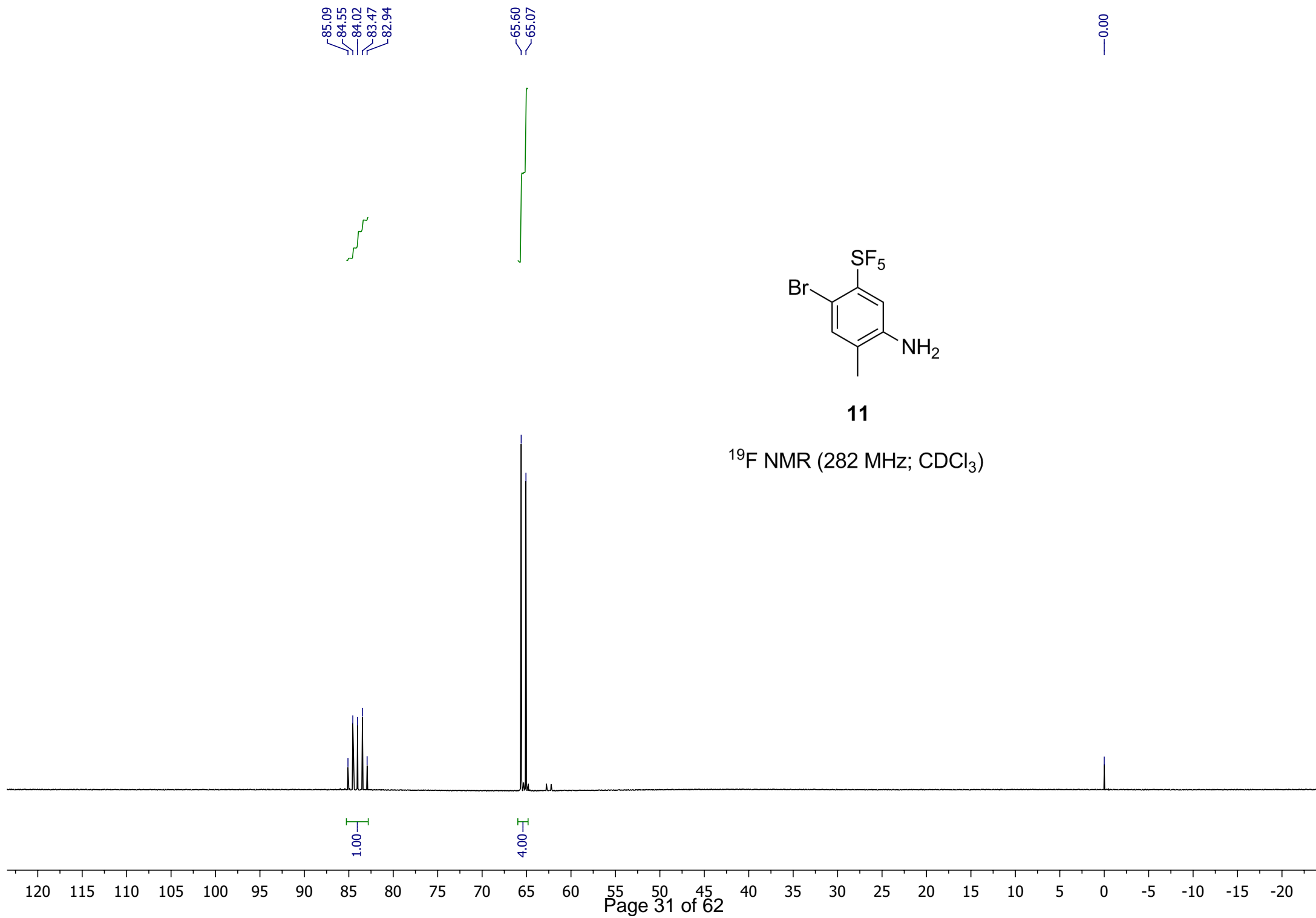
152.08
151.95
151.83
143.83
136.93
127.65
115.48
115.44
115.40
103.77
77.16
16.64

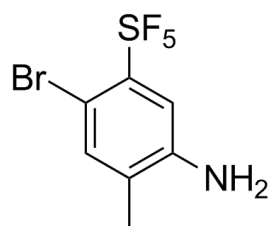




11

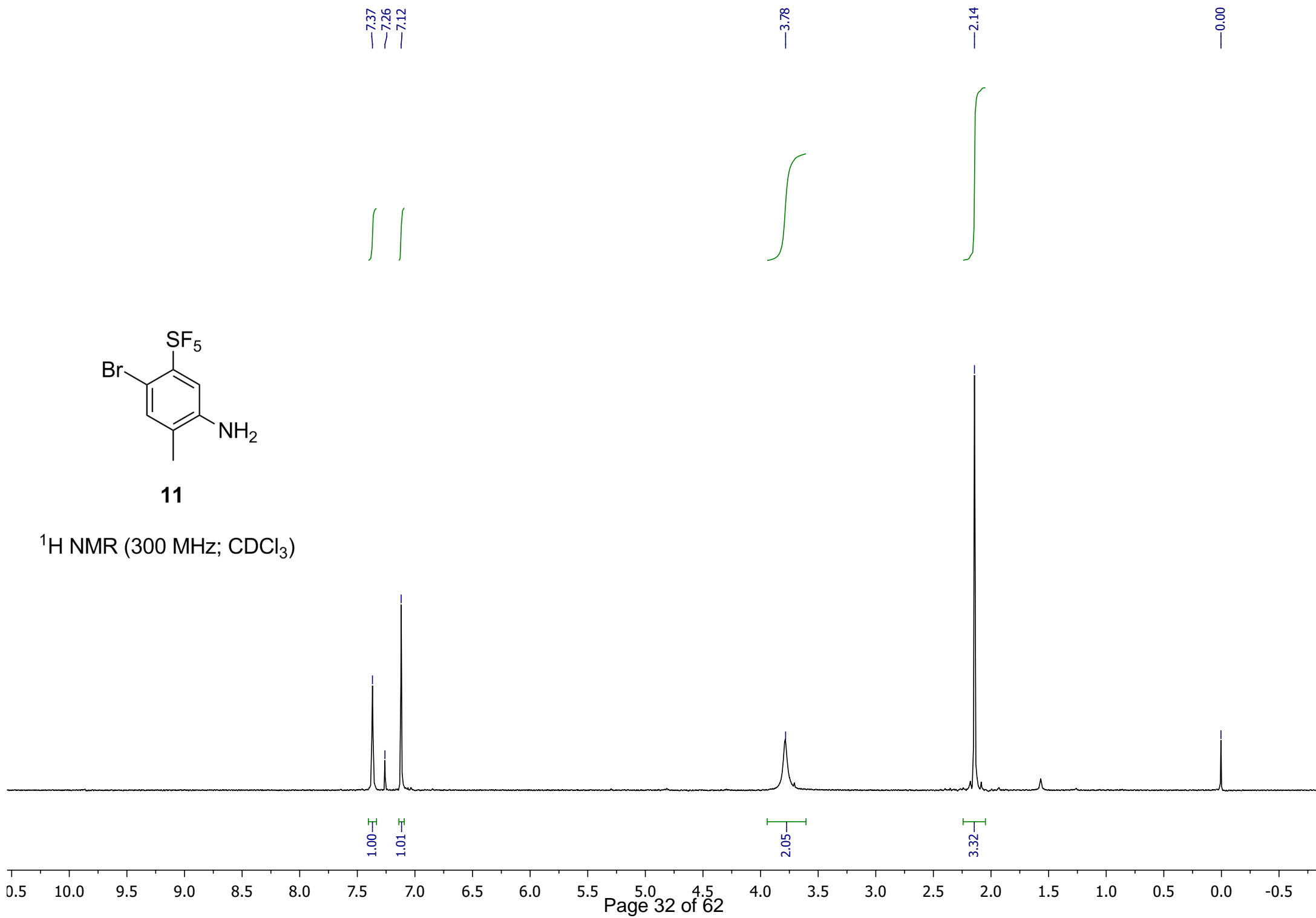
¹⁹F NMR (282 MHz; CDCl₃)

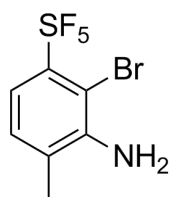




11

^1H NMR (300 MHz; CDCl_3)





12

^{13}C NMR (125 MHz; CDCl_3)

152.99
152.87
152.74

144.38

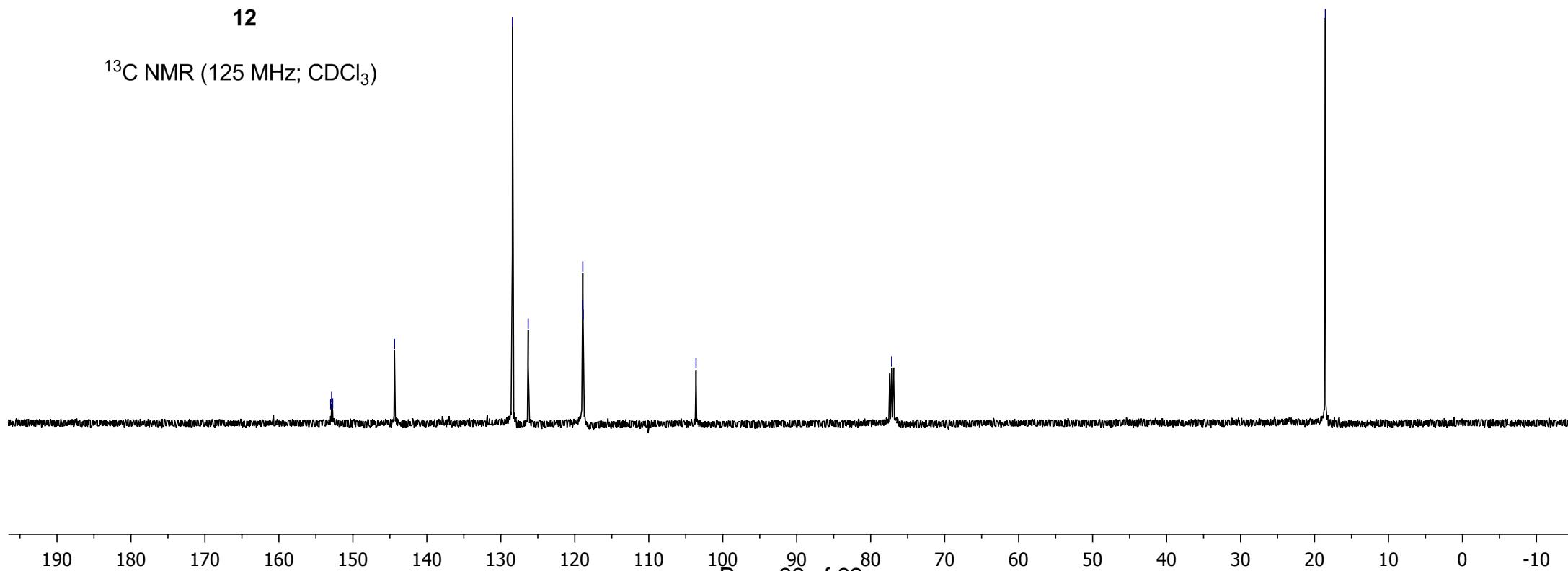
128.43
126.30

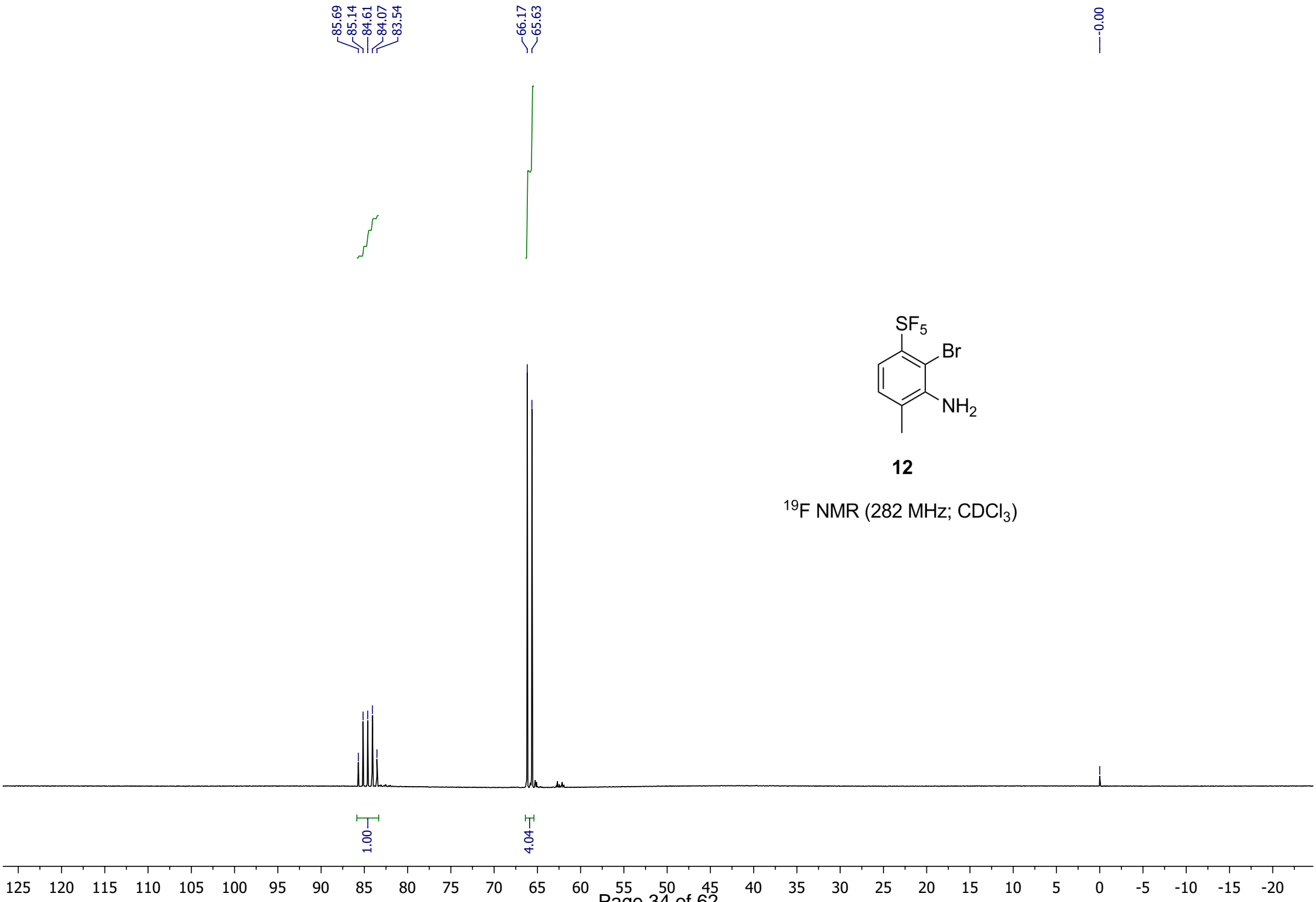
118.96
118.92
118.88

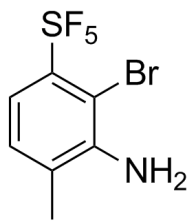
103.61

77.16

18.53

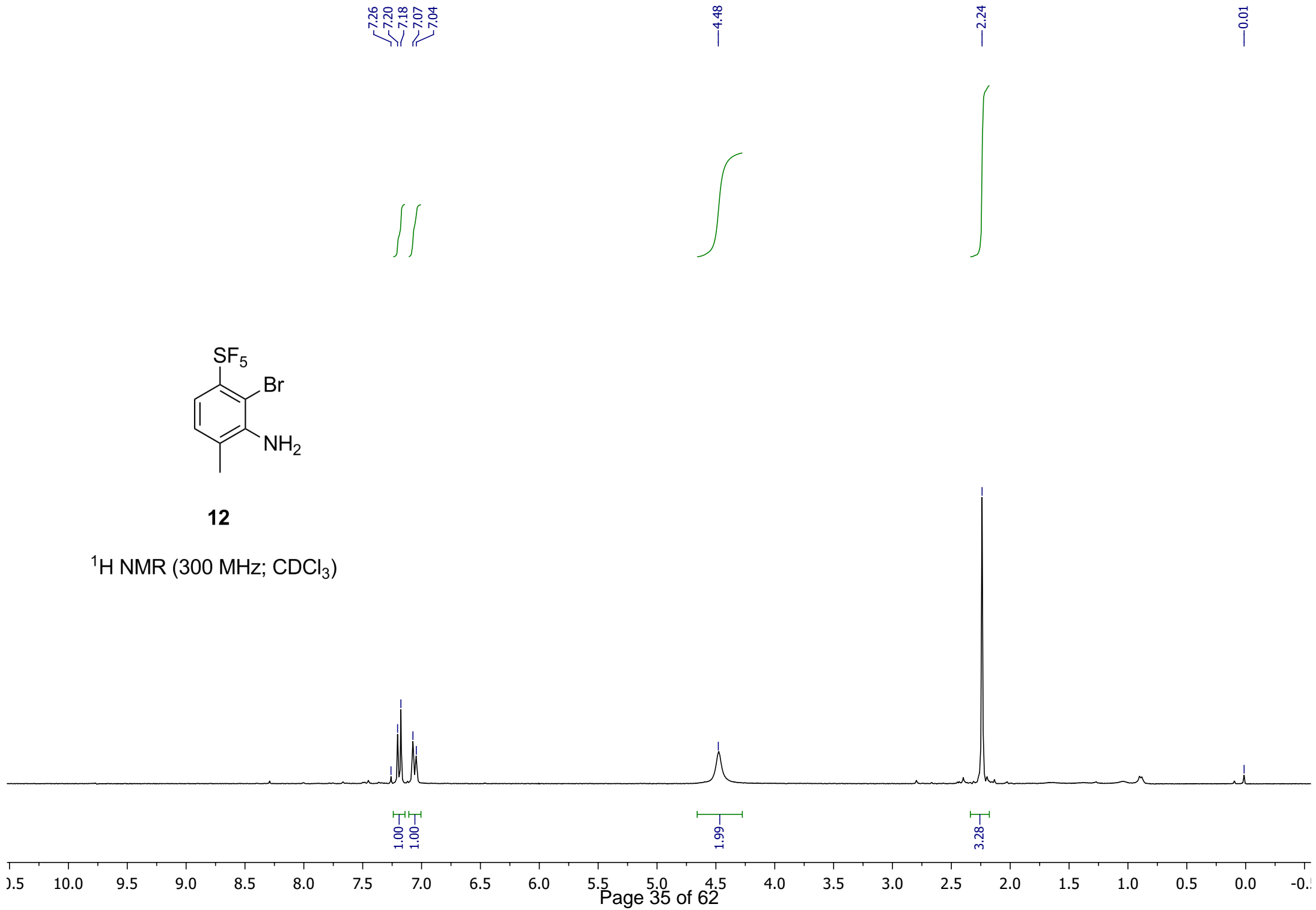






12

^1H NMR (300 MHz; CDCl_3)



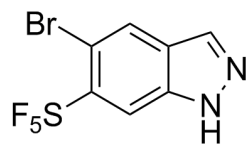
—206.40

151.68
151.56
151.42

138.17
134.17
128.86
126.54

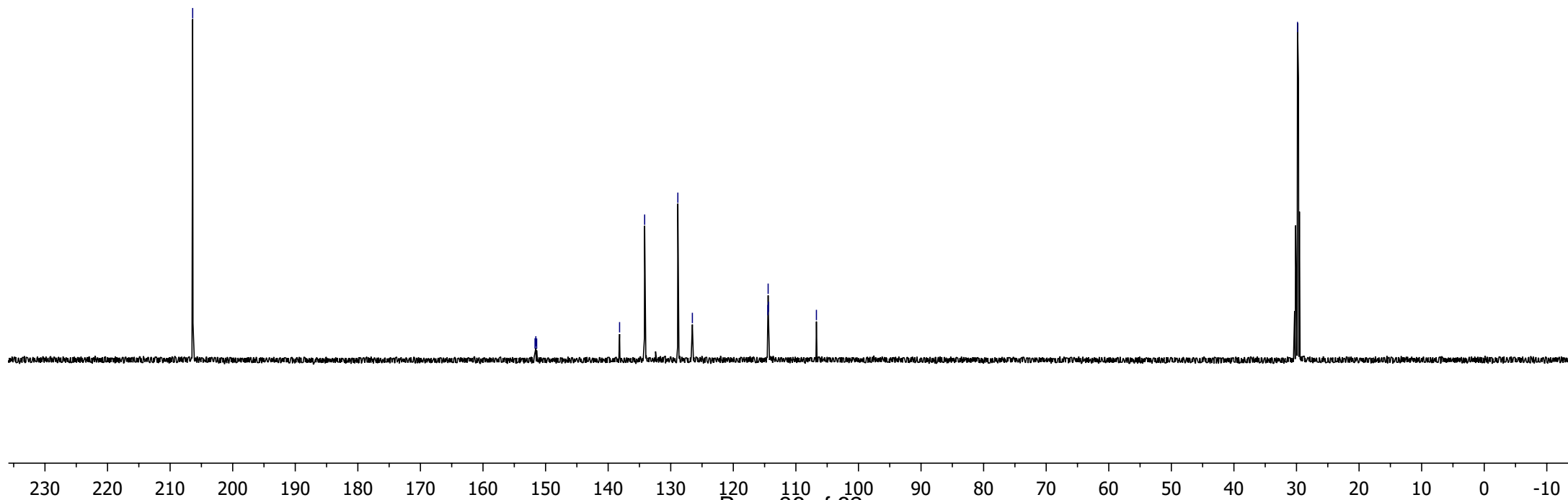
114.48
114.43
114.39
—106.72

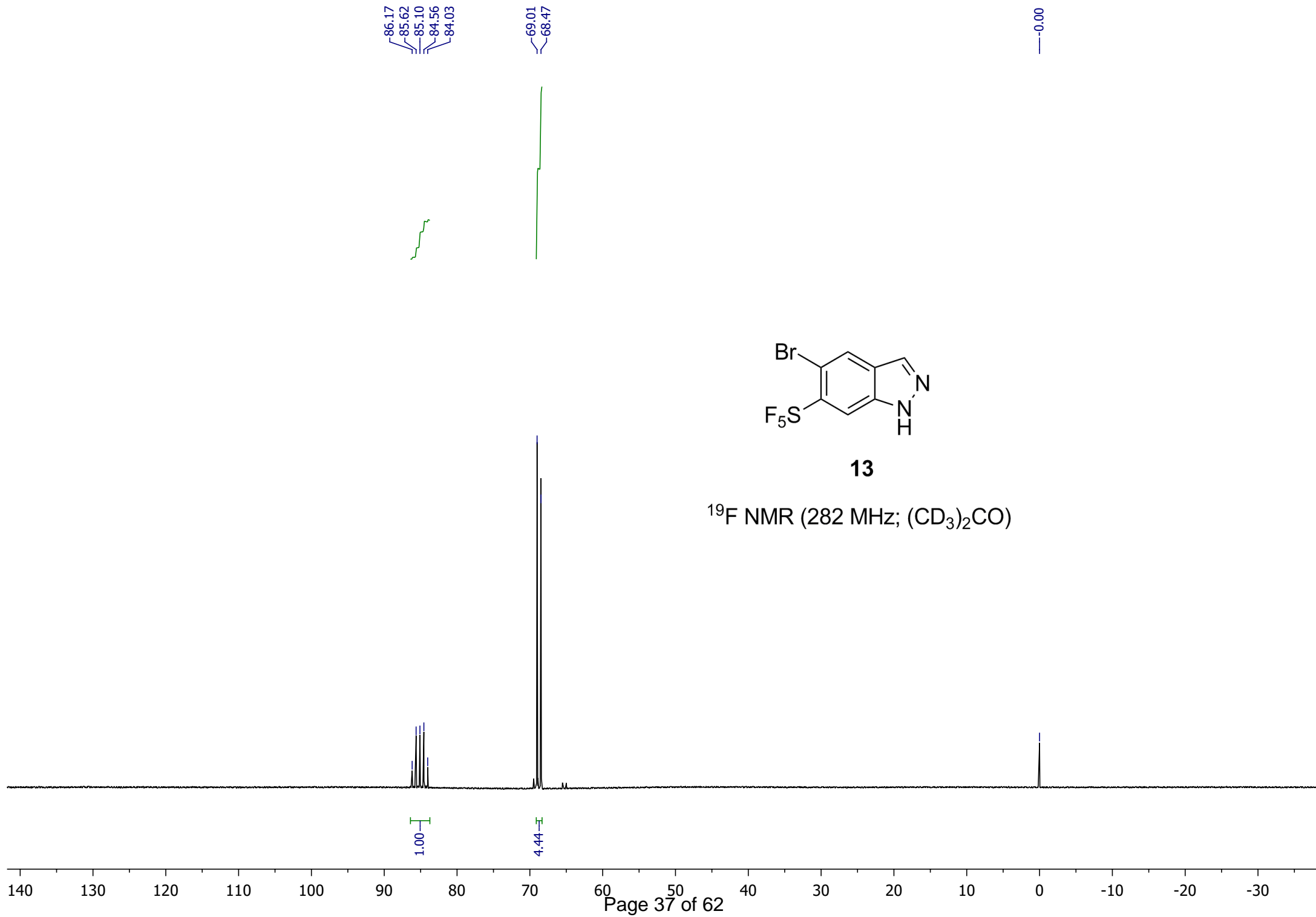
—29.84

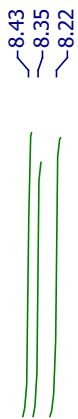


13

¹³C NMR (125 MHz; (CD₃)₂CO)

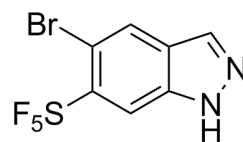






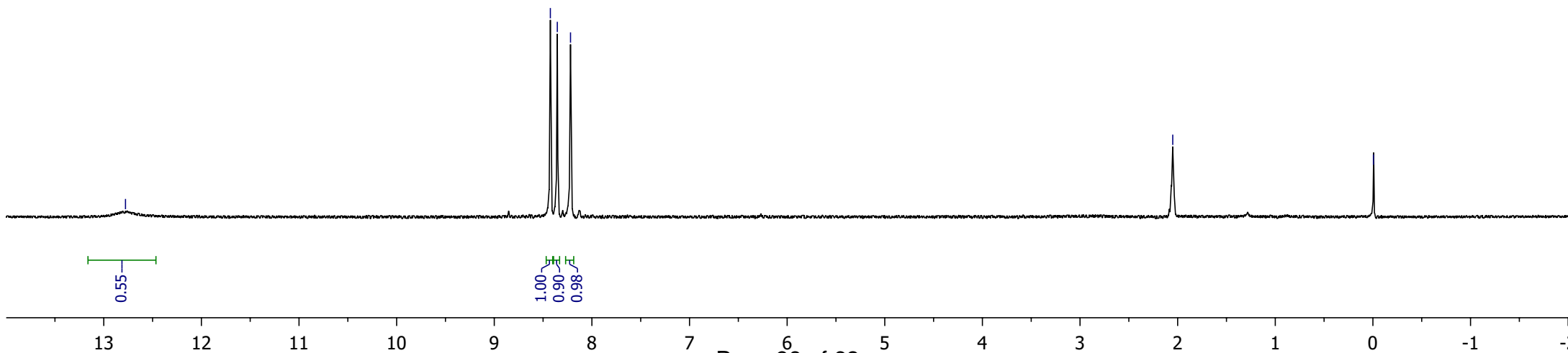
—2.05

—0.01



13

¹H NMR (300 MHz; (CD₃)₂CO)



—206.47

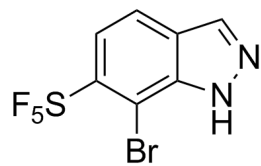
152.01
151.89
151.76

—141.35
—136.21

125.38
122.12
122.08
122.04
121.03

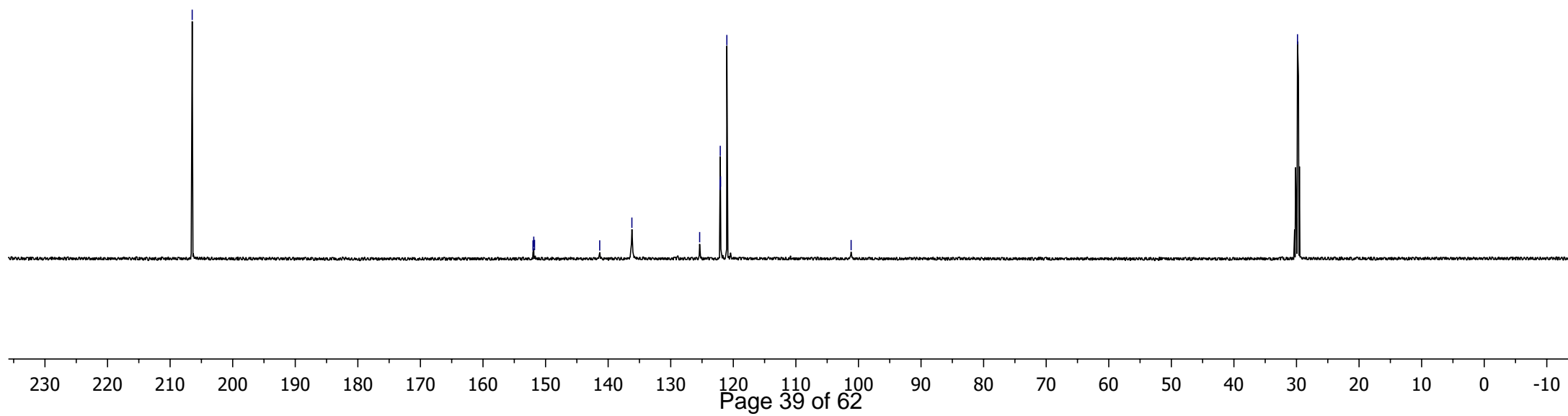
—101.17

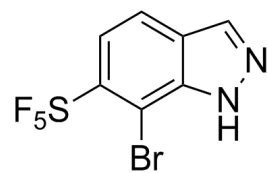
—29.84



14

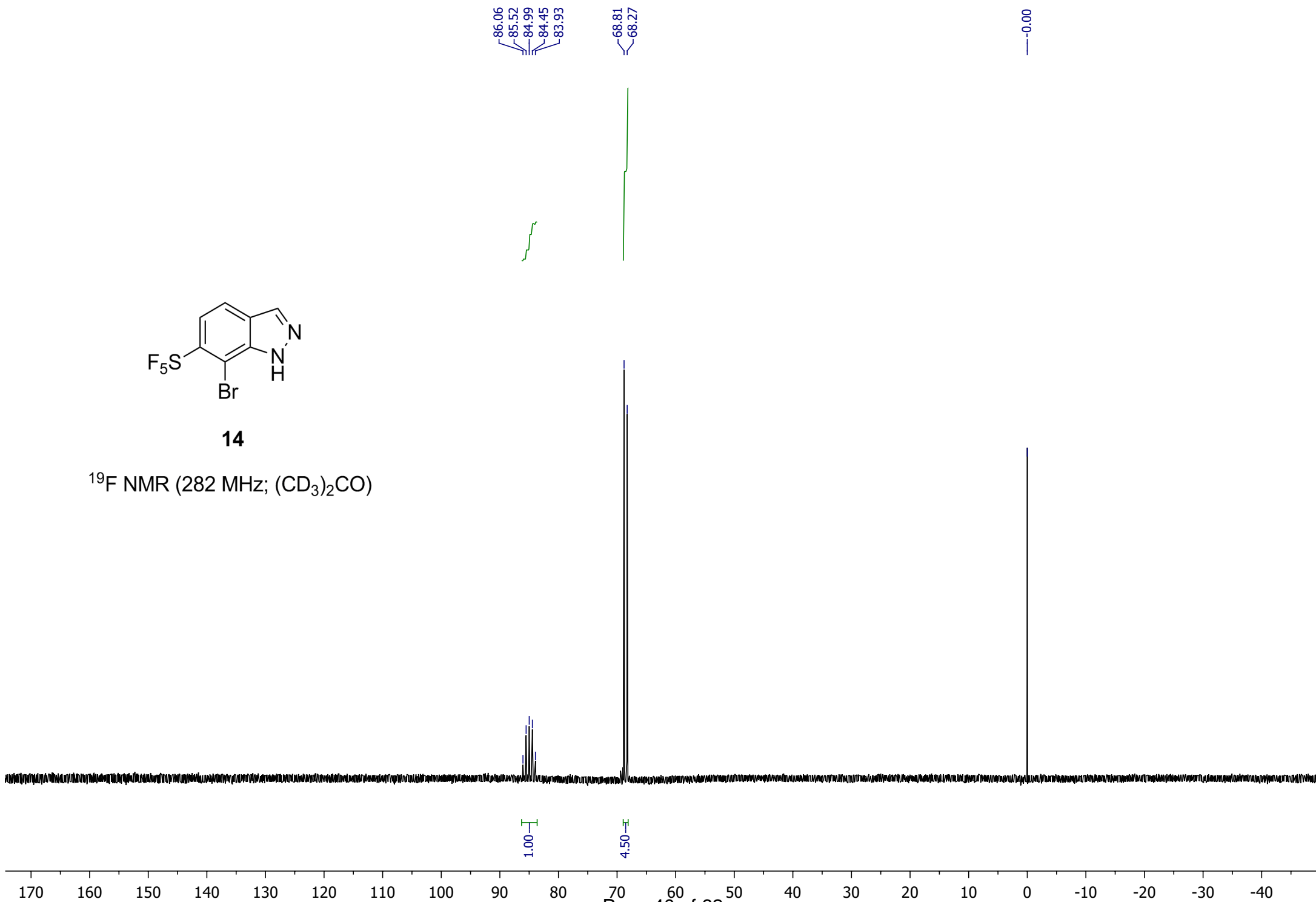
¹³C NMR (125 MHz; (CD₃)₂CO)





14

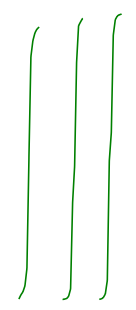
^{19}F NMR (282 MHz; $(\text{CD}_3)_2\text{CO}$)



—13.11



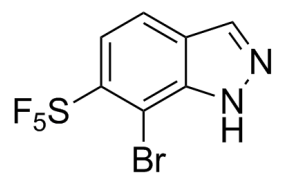
8.36
8.04
8.01
7.77
7.74



—2.83

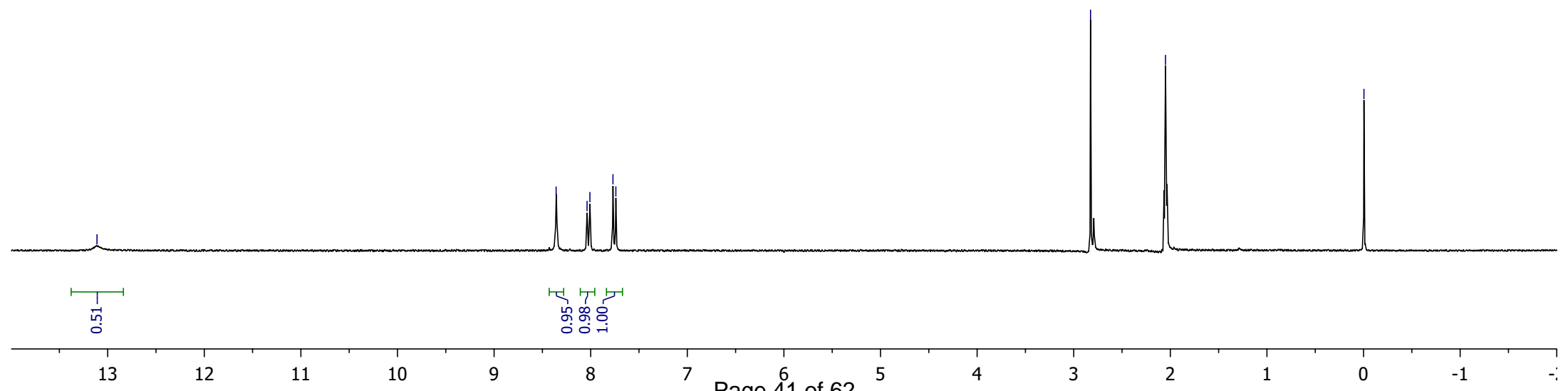
—2.05

—0.00



14

¹H NMR (300 MHz; (CD₃)₂CO)



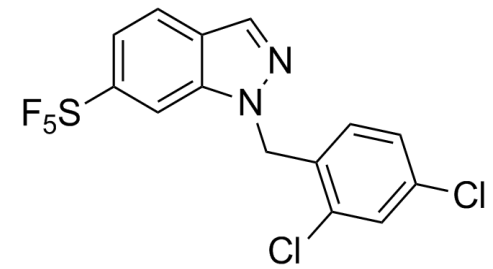
152.77
152.63
152.50

138.26
134.77
134.17
133.53
132.33
130.06
129.61
127.66
125.24
121.43
118.42
118.39
118.36

108.22
108.18
108.14

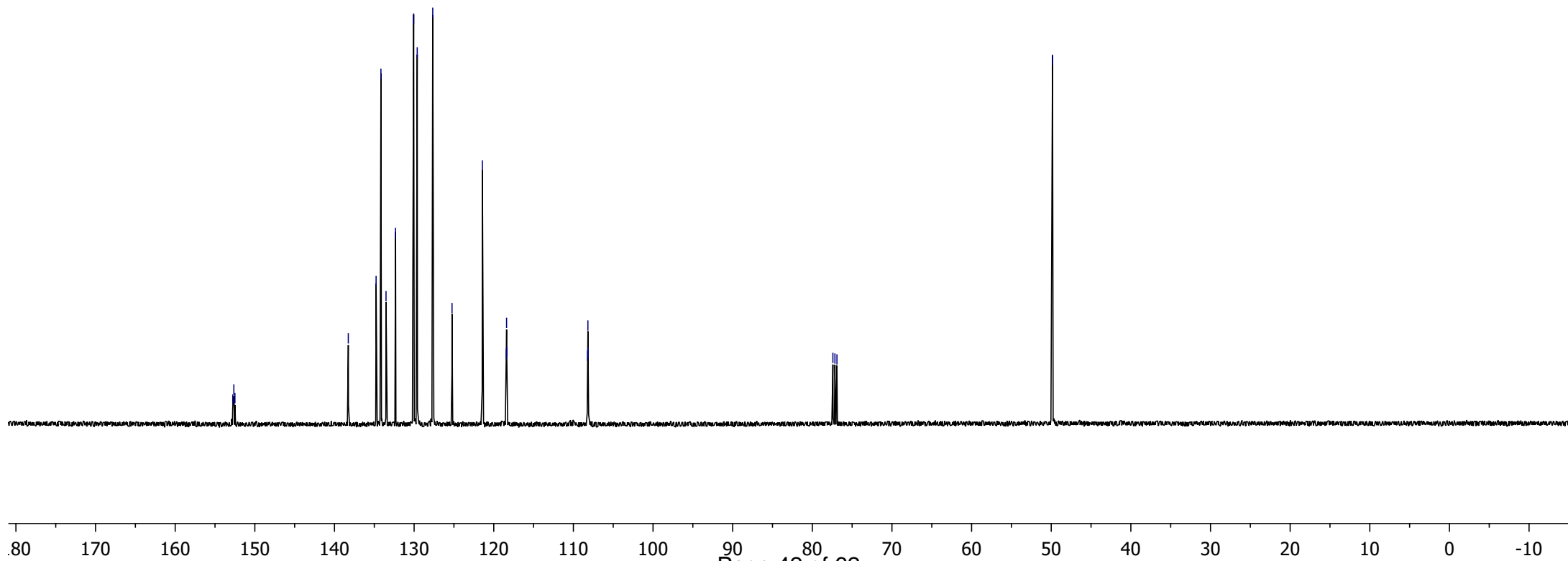
77.42
77.16
76.91

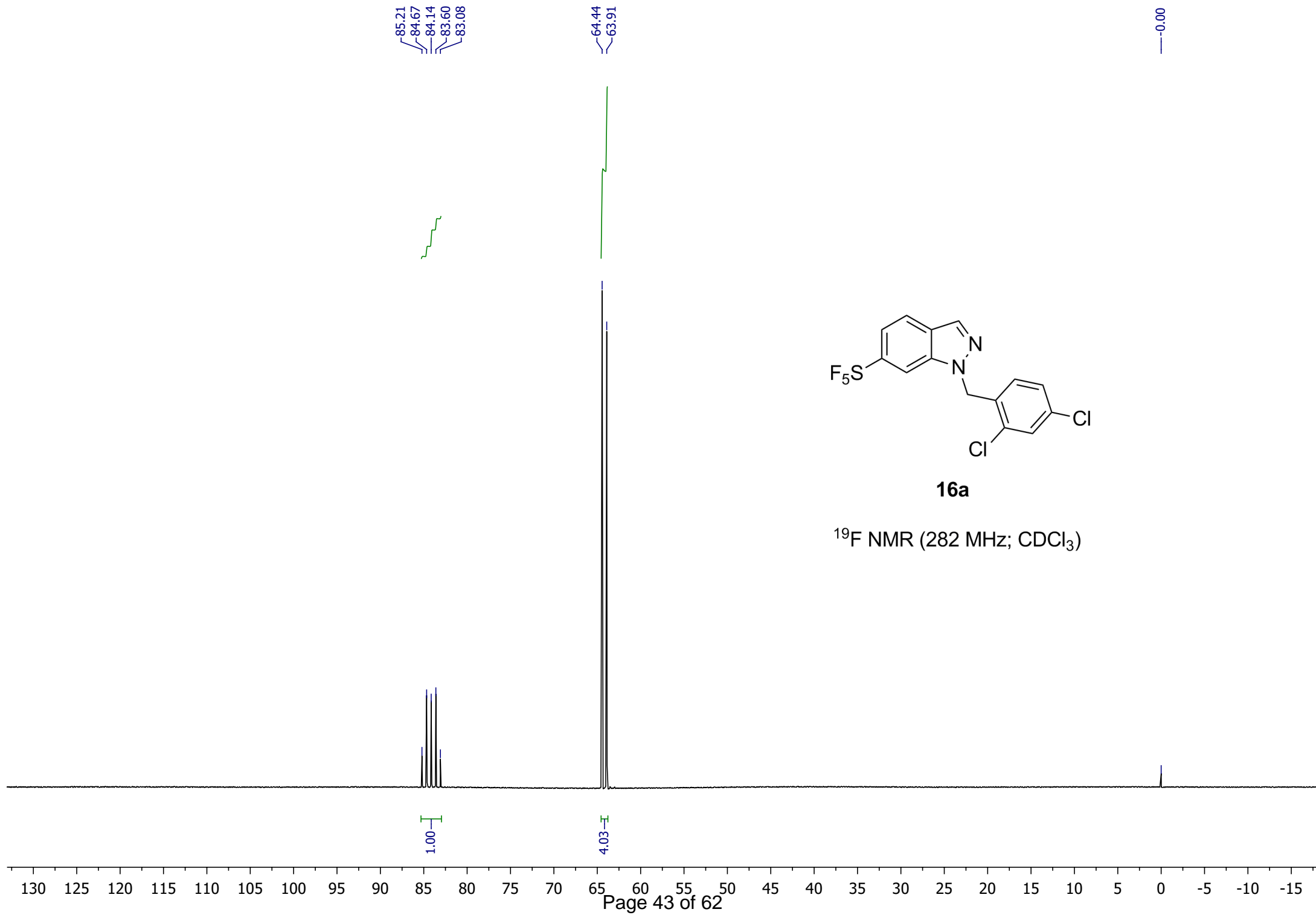
49.84

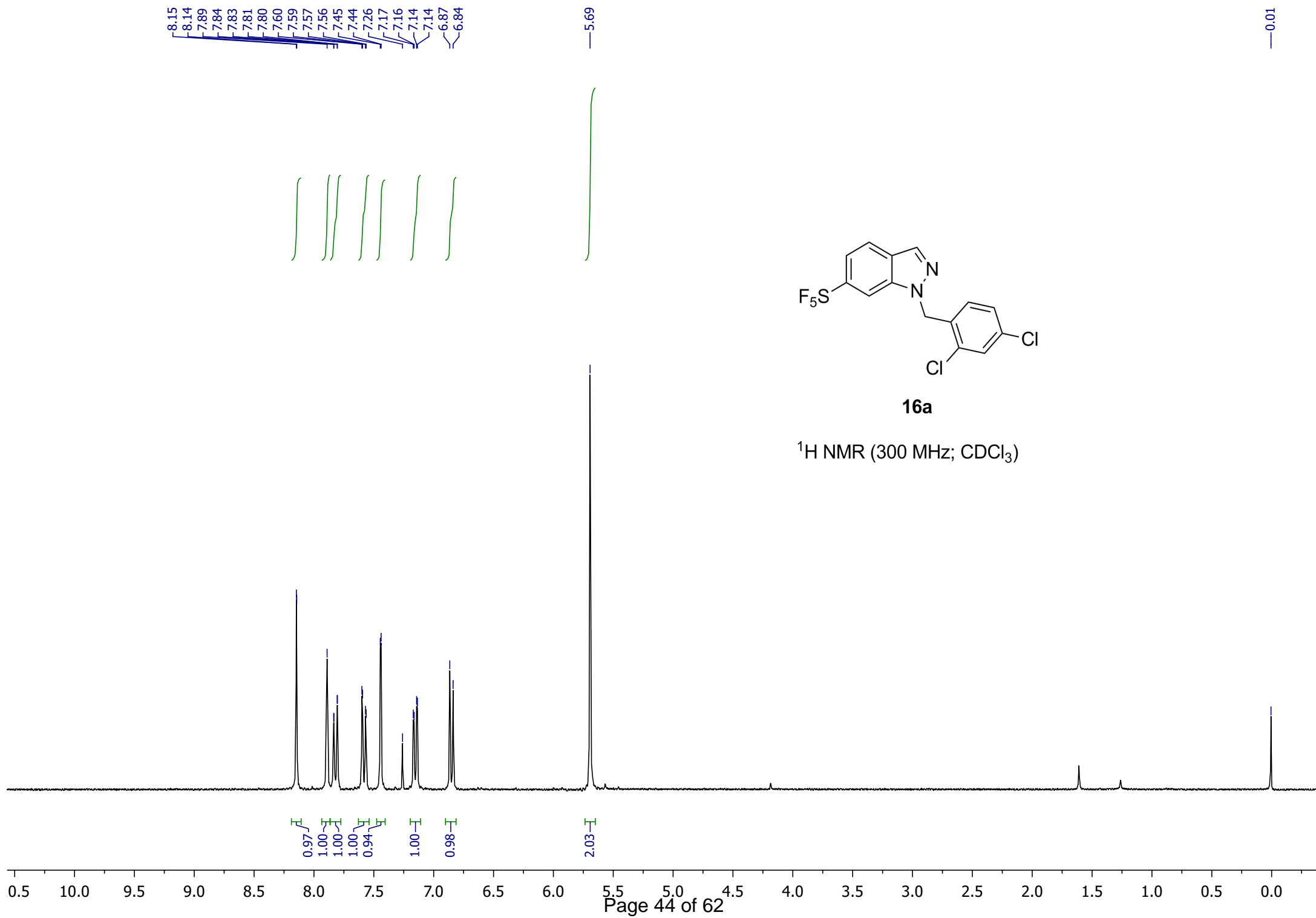


16a

^{13}C NMR (125 MHz; CDCl_3)



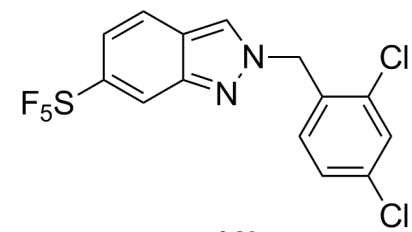




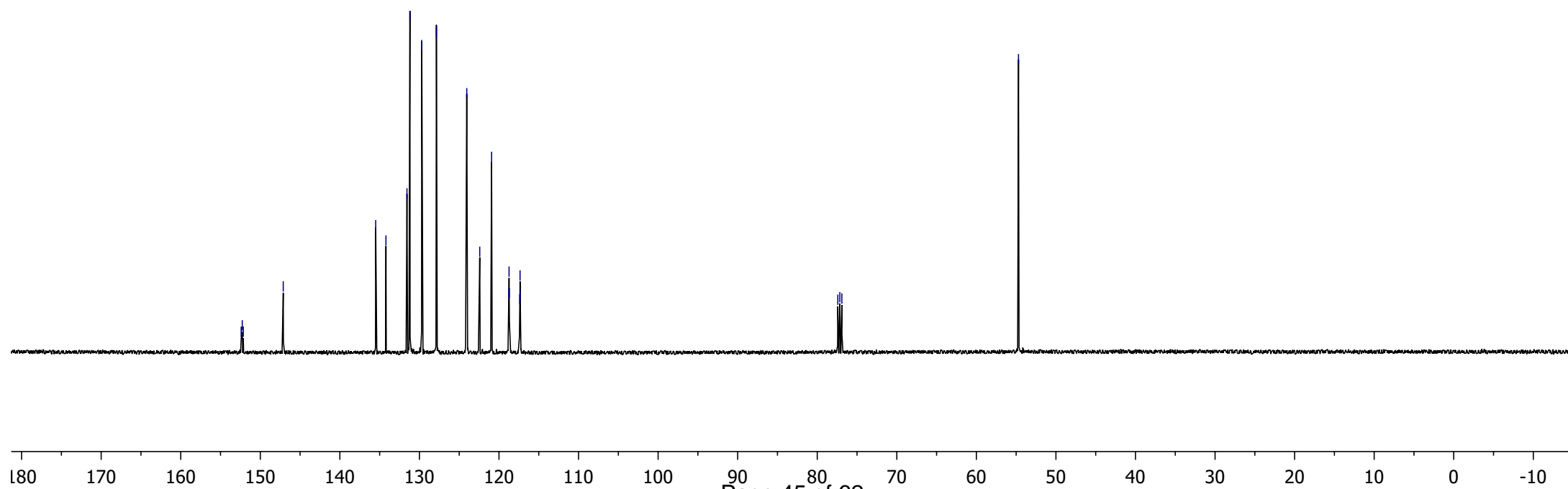
152.40
152.27
152.14
147.12
135.50
134.21
131.56
131.19
129.71
127.87
124.05
122.42
120.93
118.77
118.73
118.70
117.39
117.35
117.31

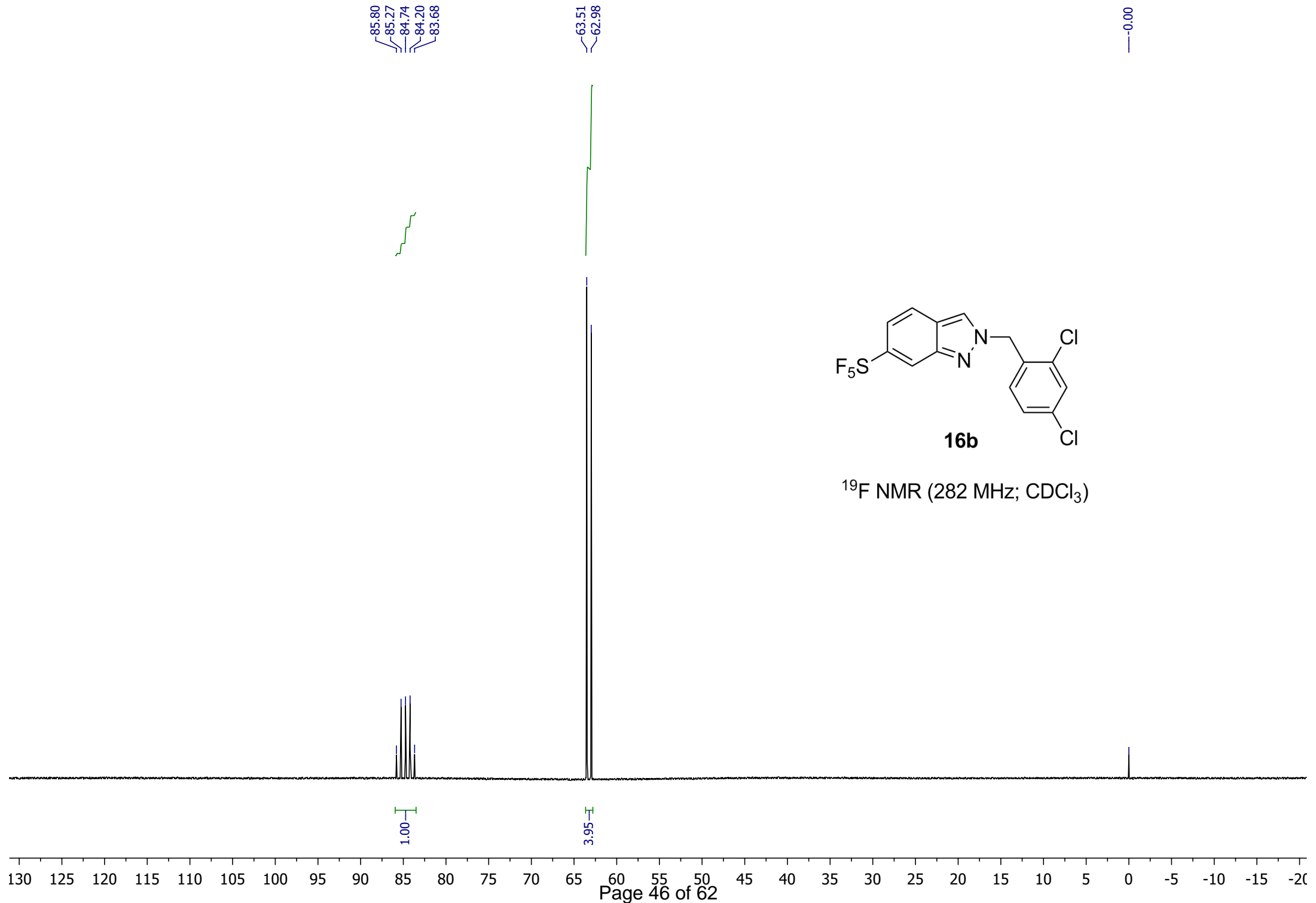
77.41
77.16
76.91

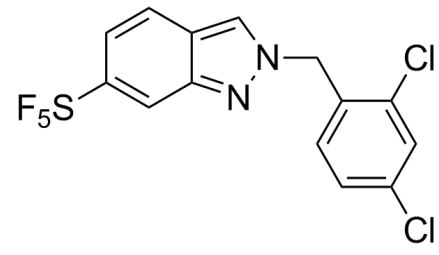
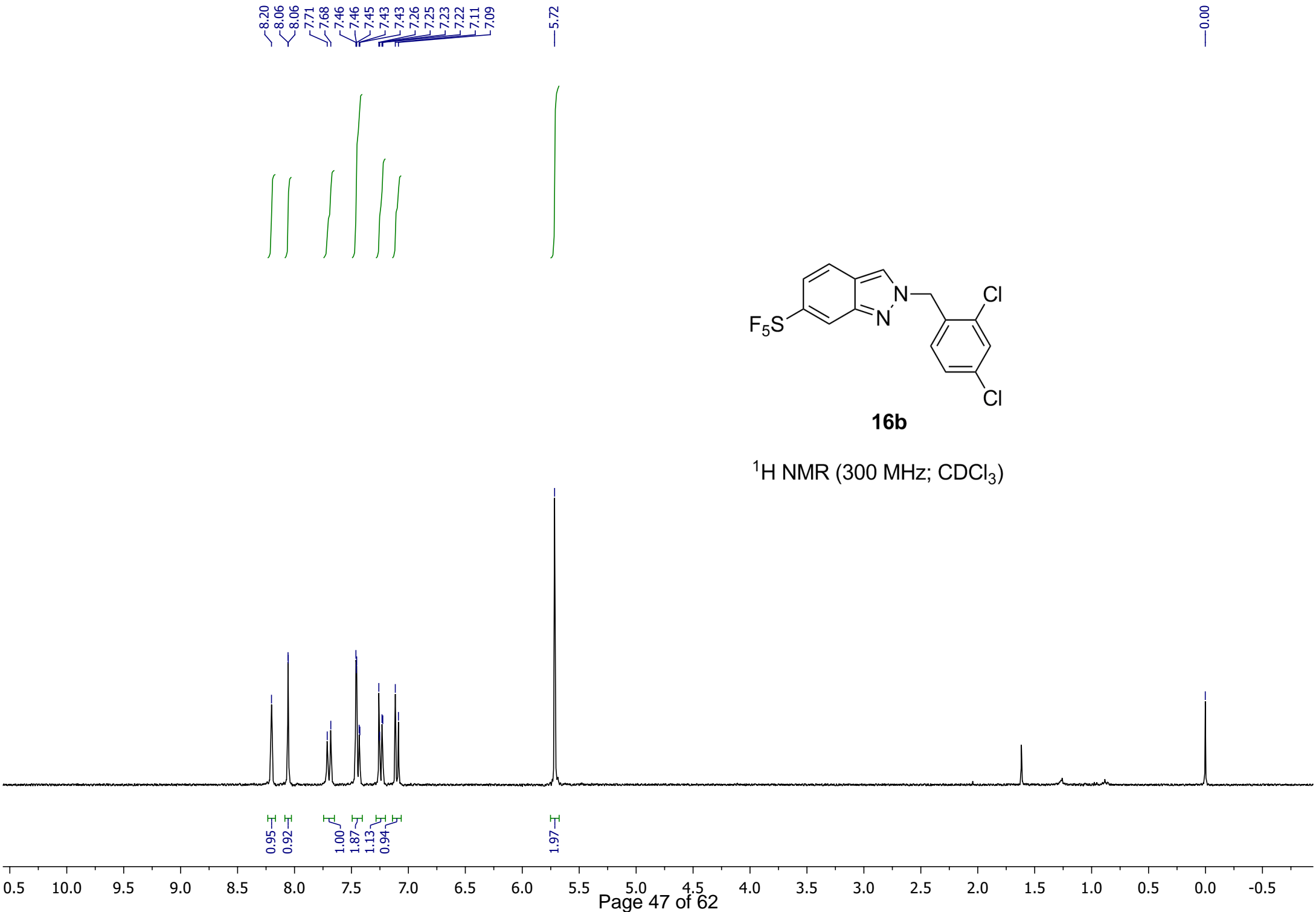
54.70



¹³C NMR (125 MHz; CDCl₃)

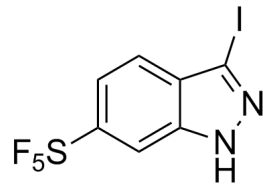






16b

$^1\text{H NMR}$ (300 MHz; CDCl_3)



17

^{13}C NMR (125 MHz; $(\text{CD}_3)_2\text{CO}$)

—206.37

153.72
153.58
153.45

—140.00

—129.40

122.27

119.21

119.18

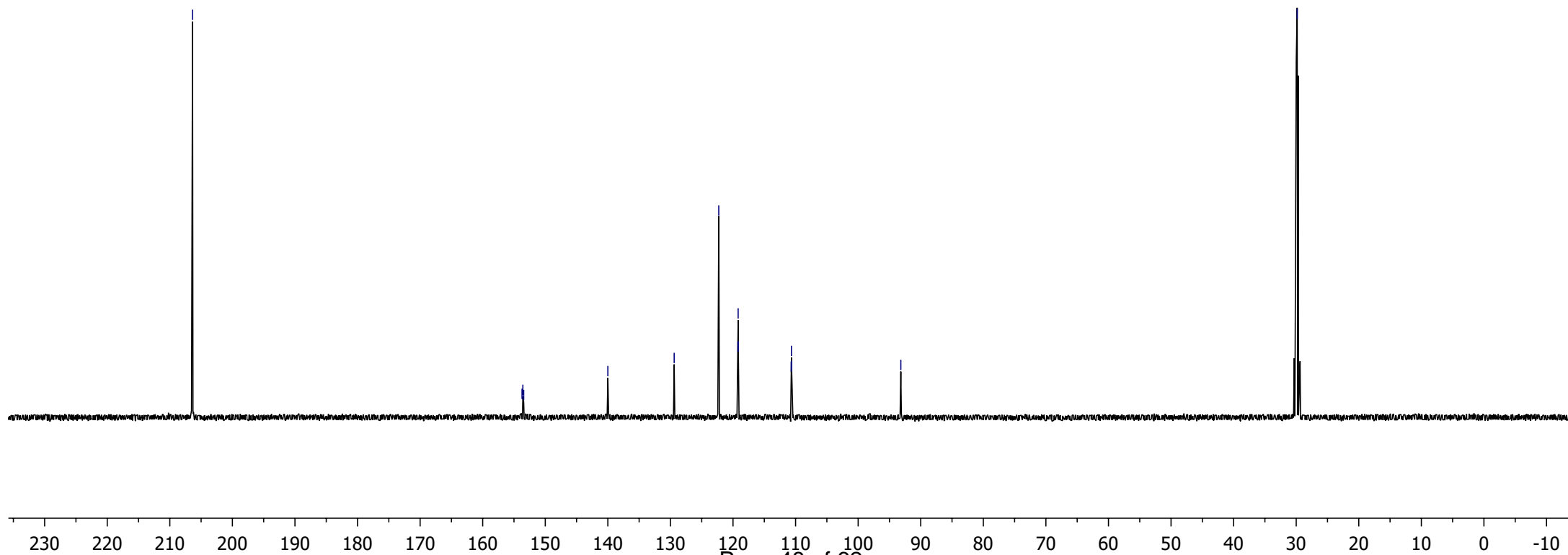
119.14

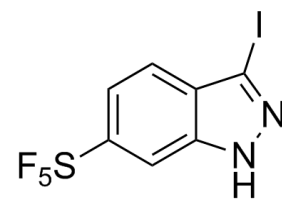
110.70

110.66

—93.18

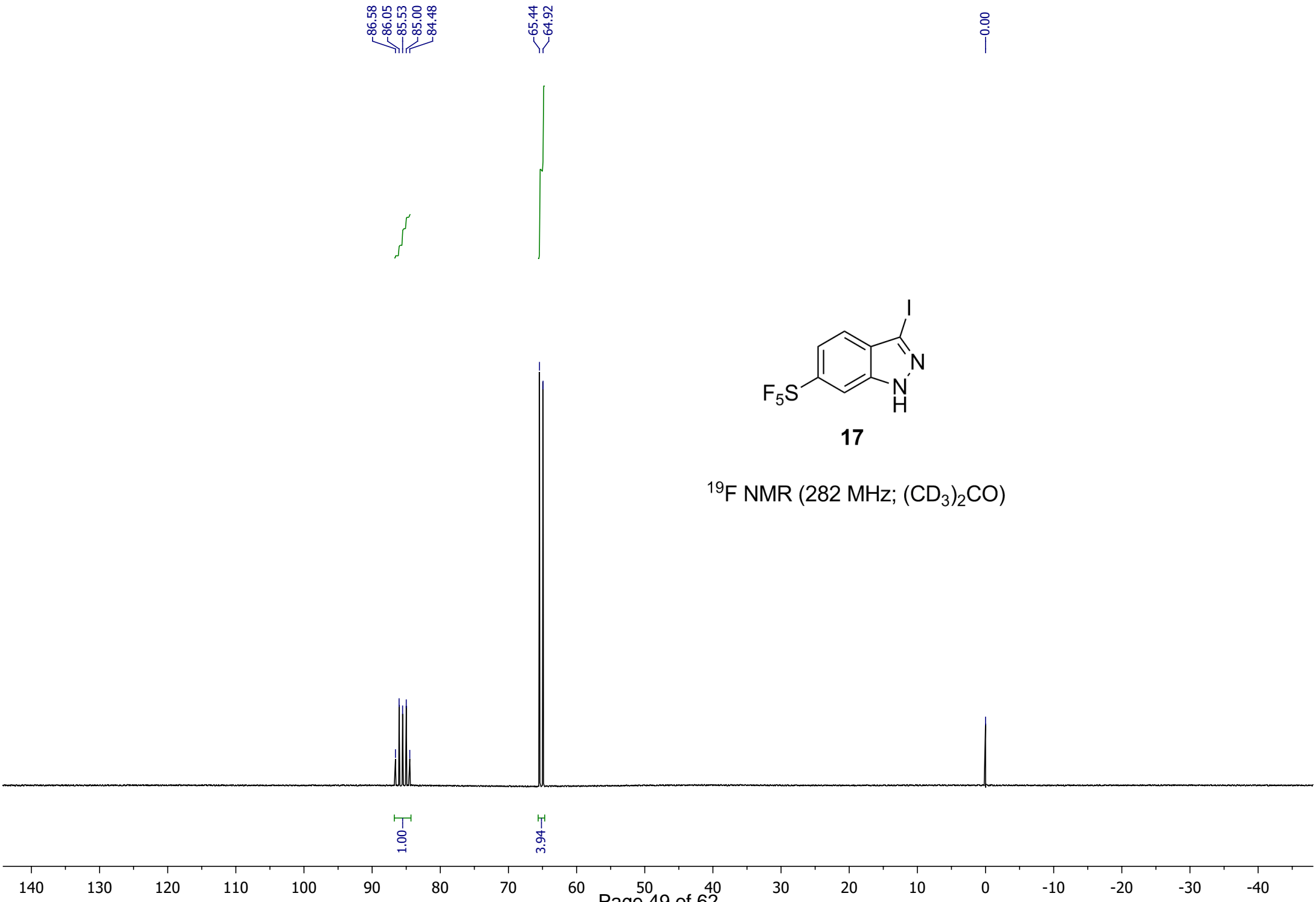
—29.84

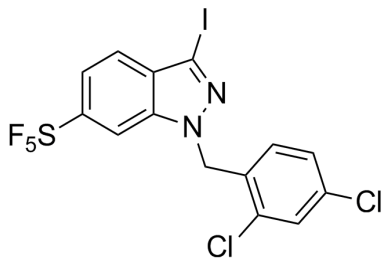




17

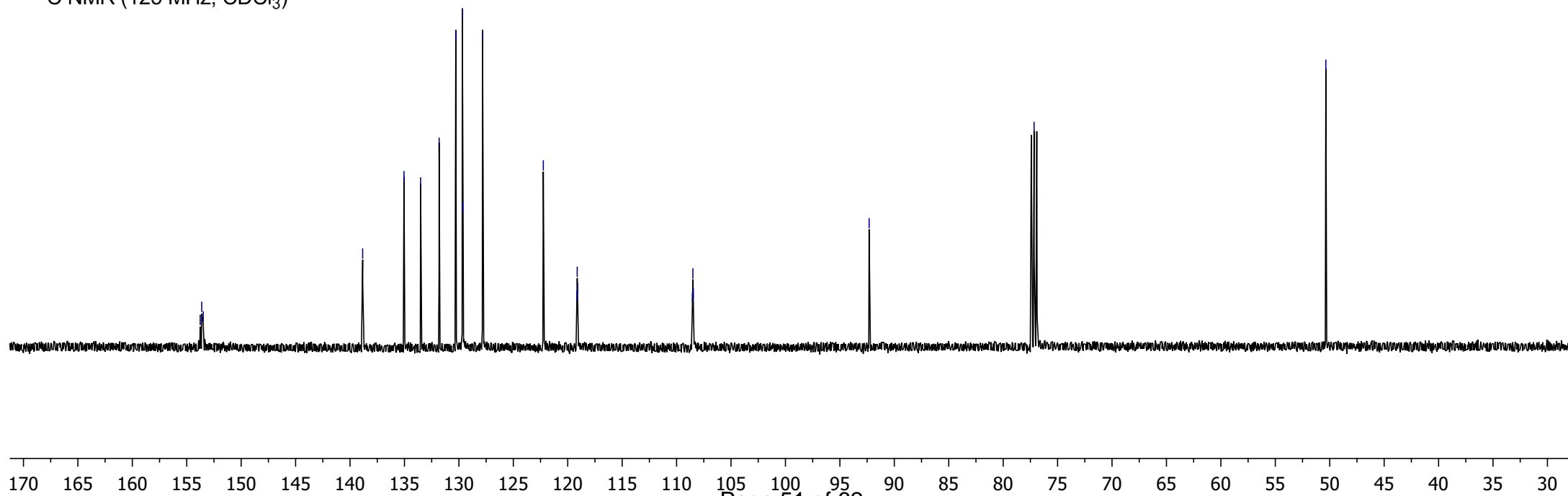
¹⁹F NMR (282 MHz; (CD₃)₂CO)

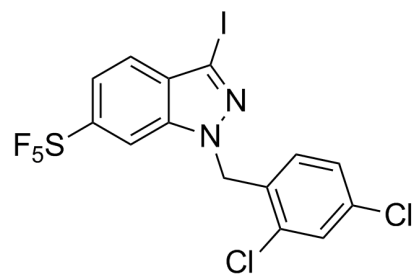




18

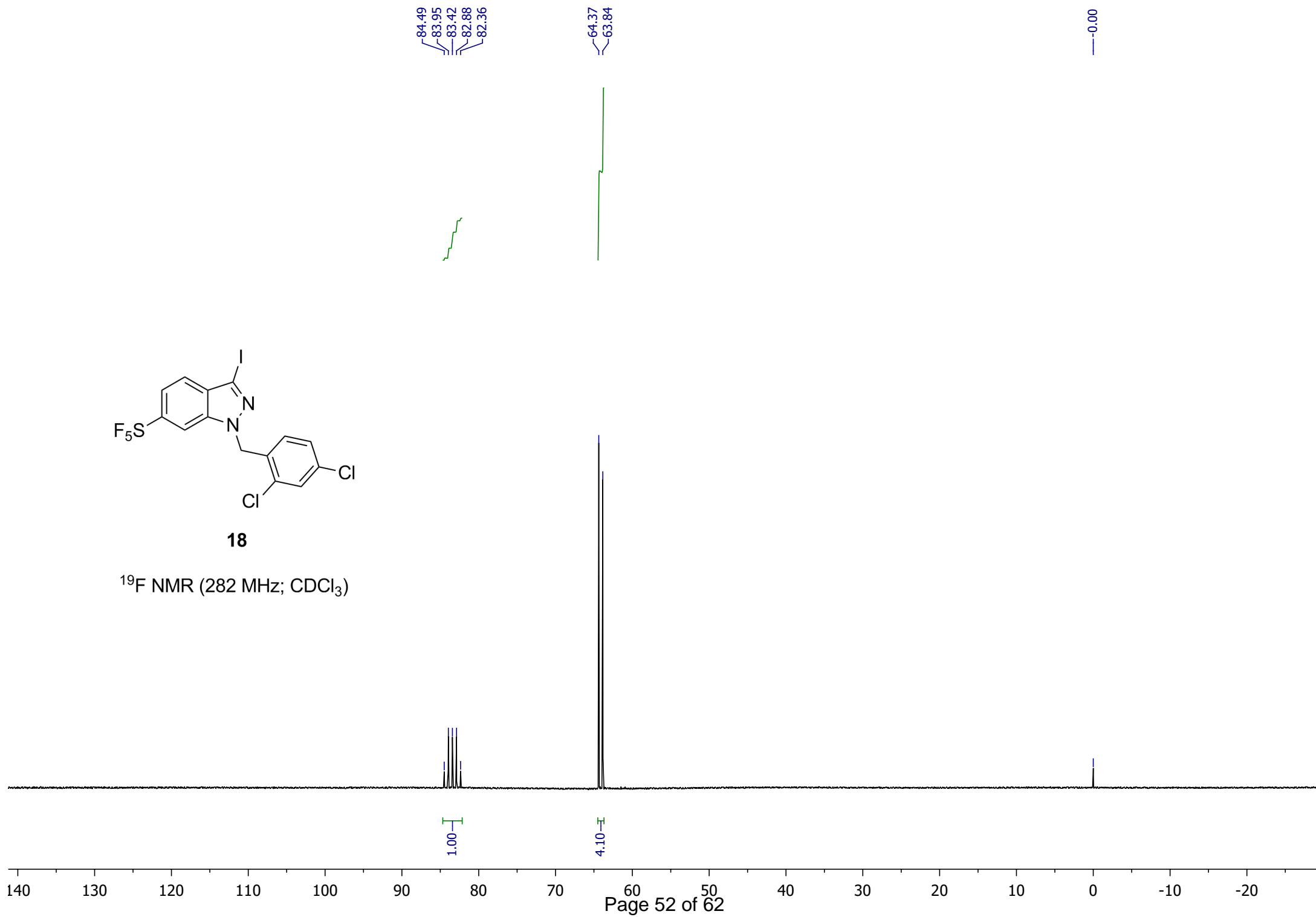
^{13}C NMR (125 MHz; CDCl_3)

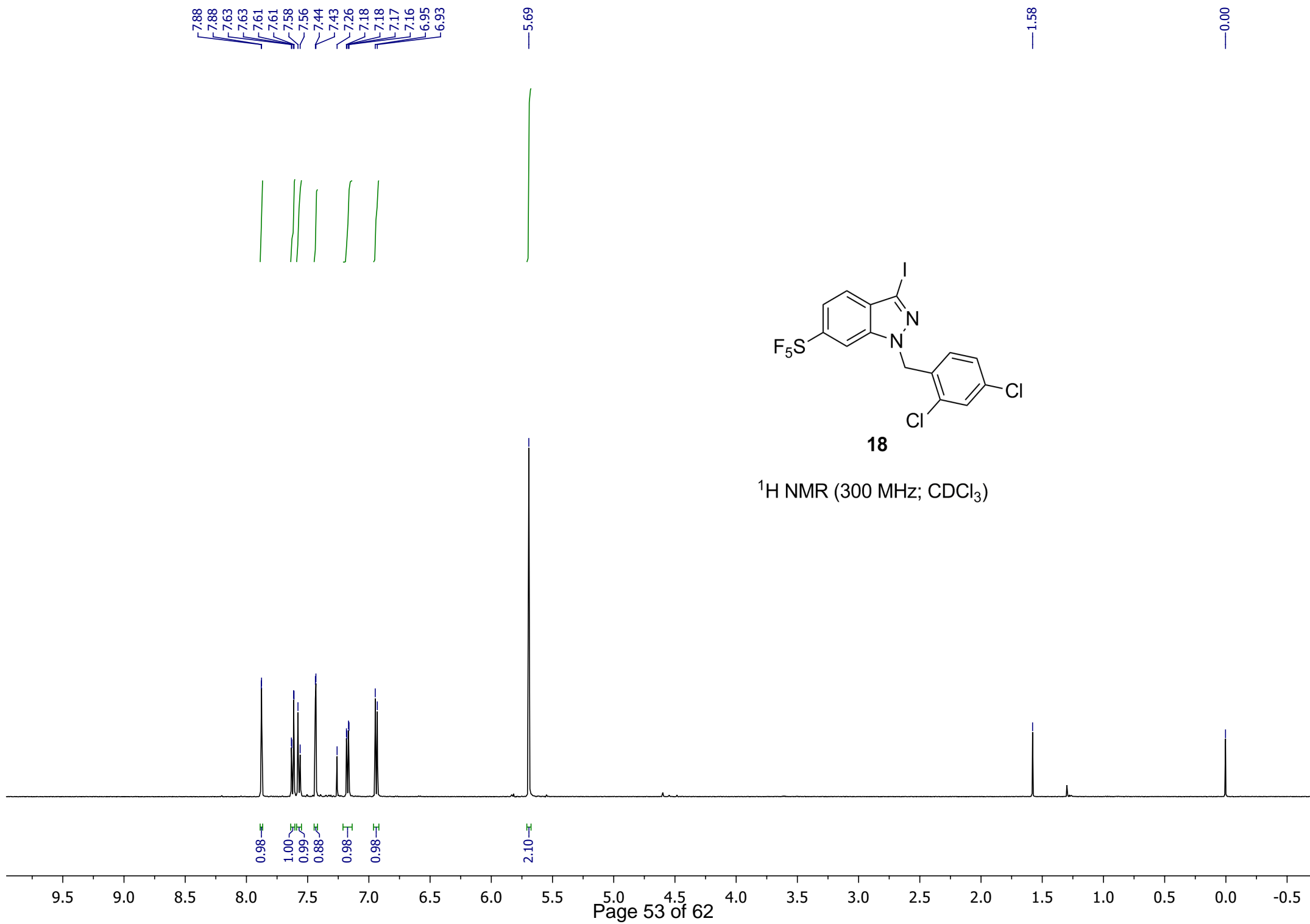


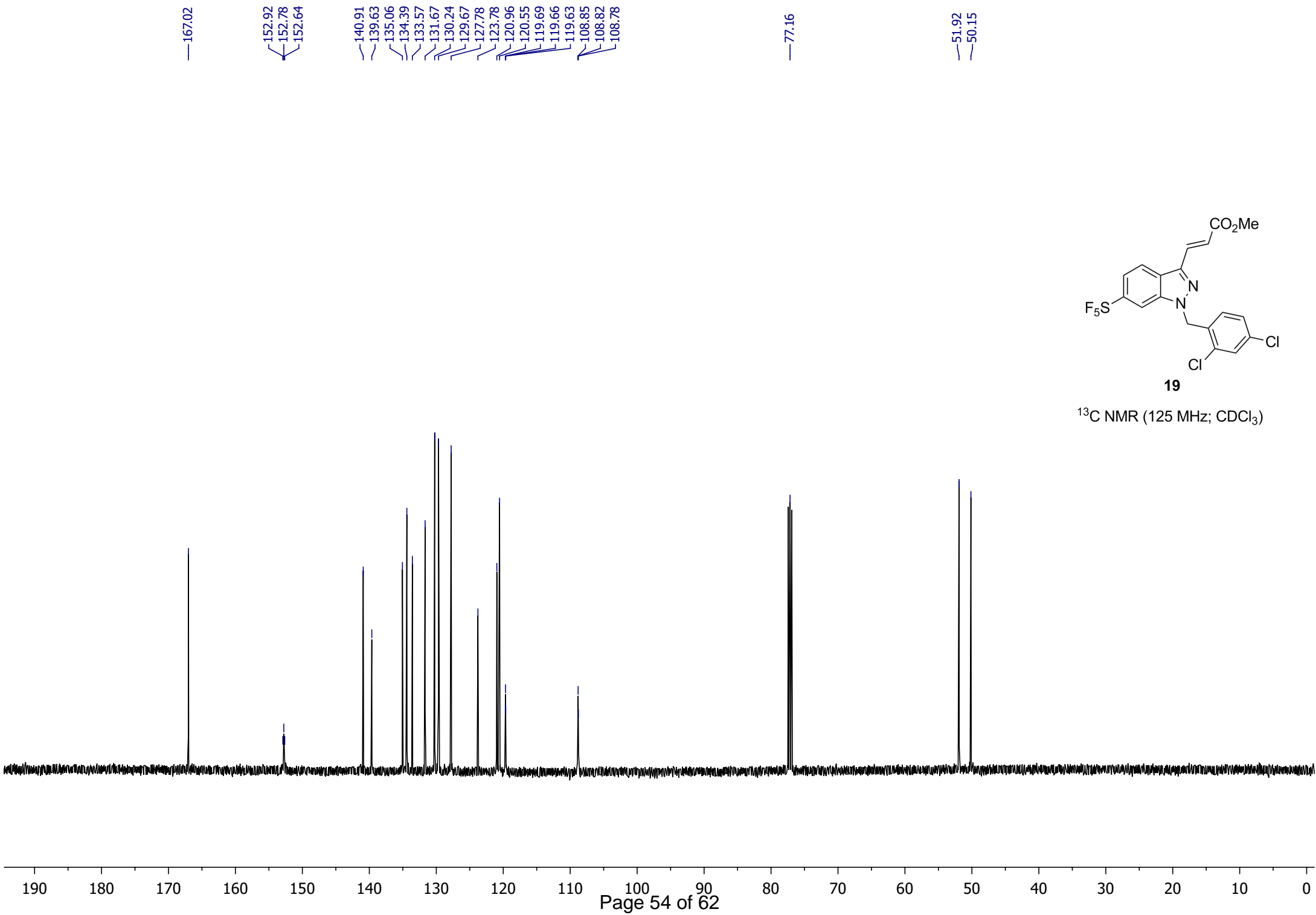


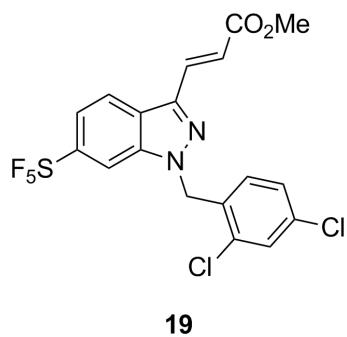
18

¹⁹F NMR (282 MHz; CDCl₃)

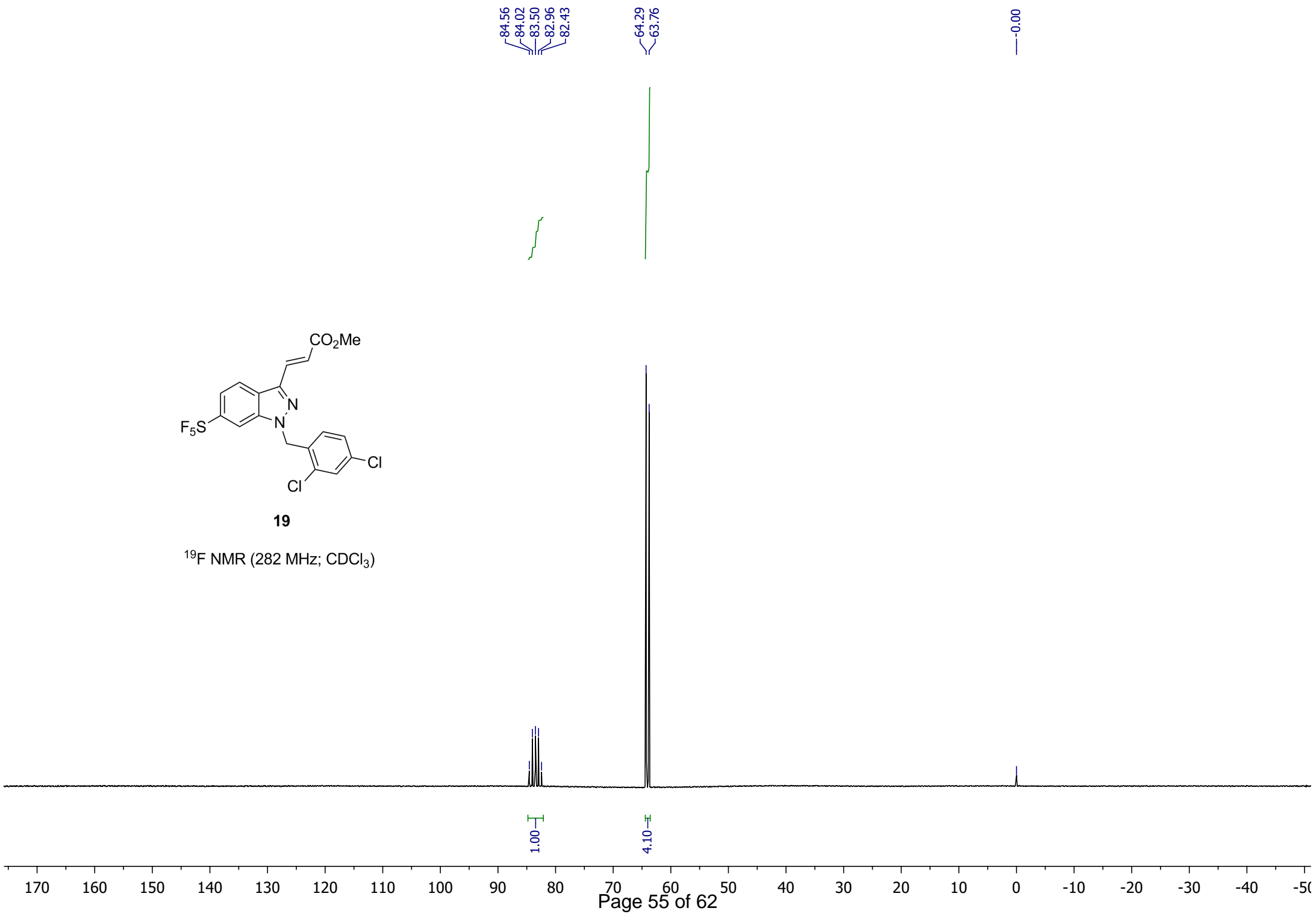


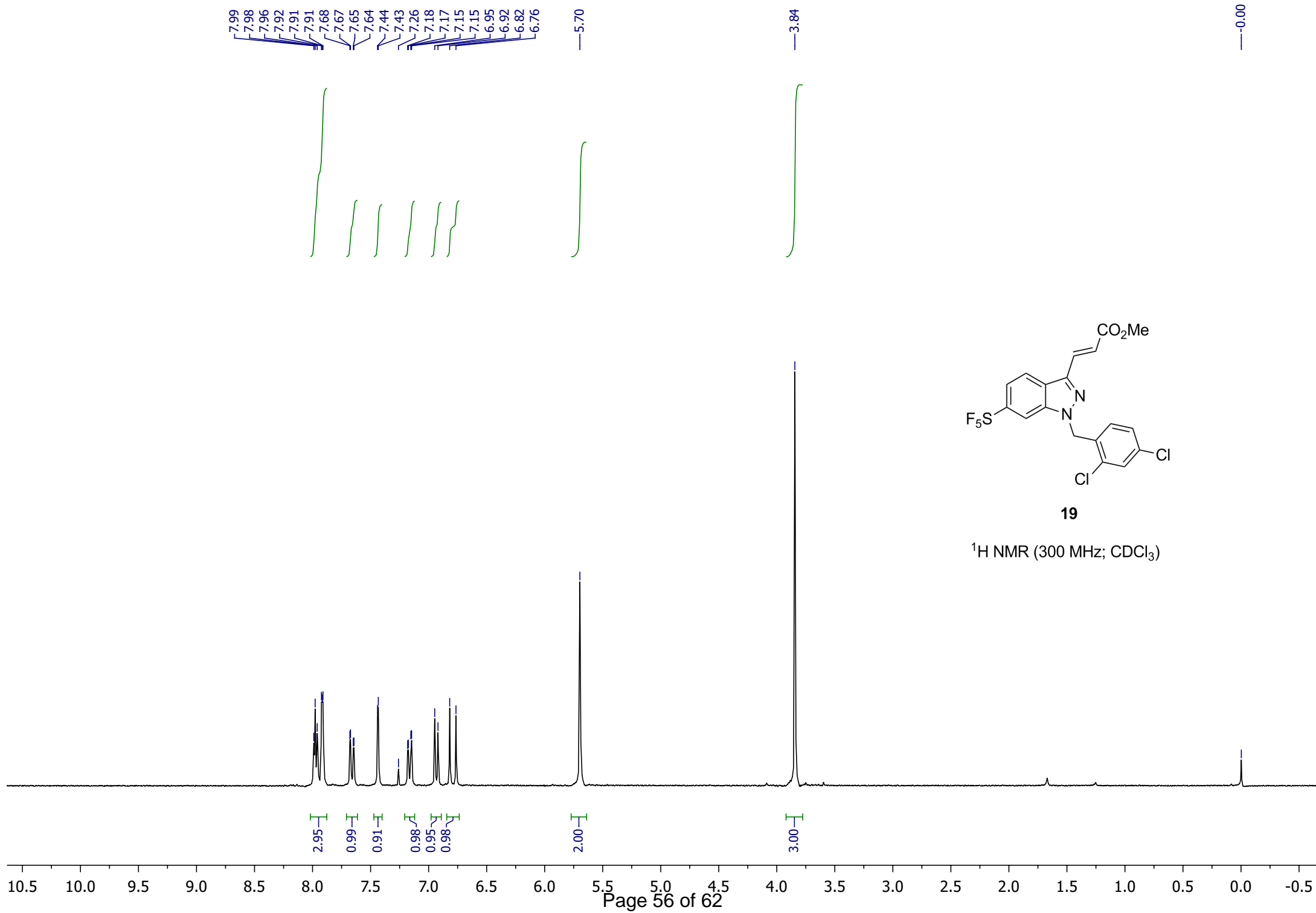






^{19}F NMR (282 MHz; CDCl_3)





—206.28

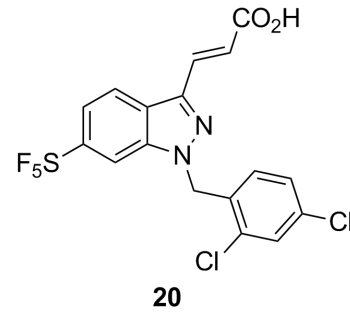
—167.75

152.84
152.71
152.57

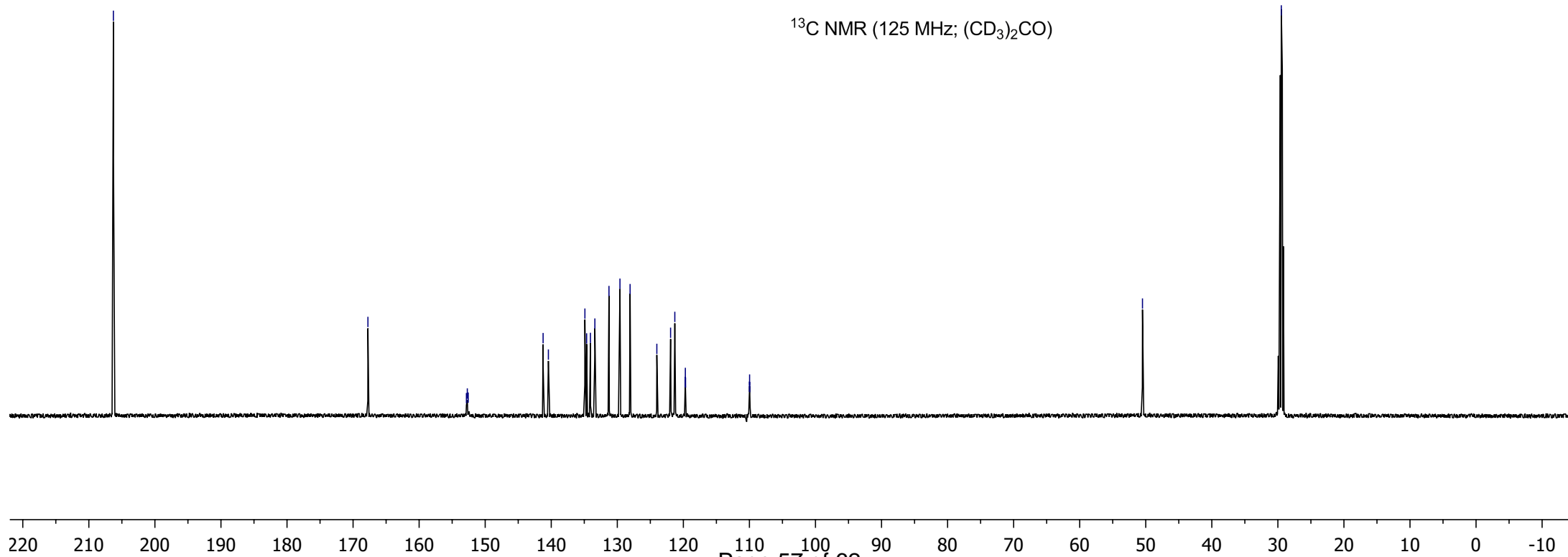
141.23
140.43
134.90
134.64
134.07
133.39
131.26
129.60
128.06
124.01
121.93
121.29
119.73
119.70
119.67
110.01
109.97
109.94

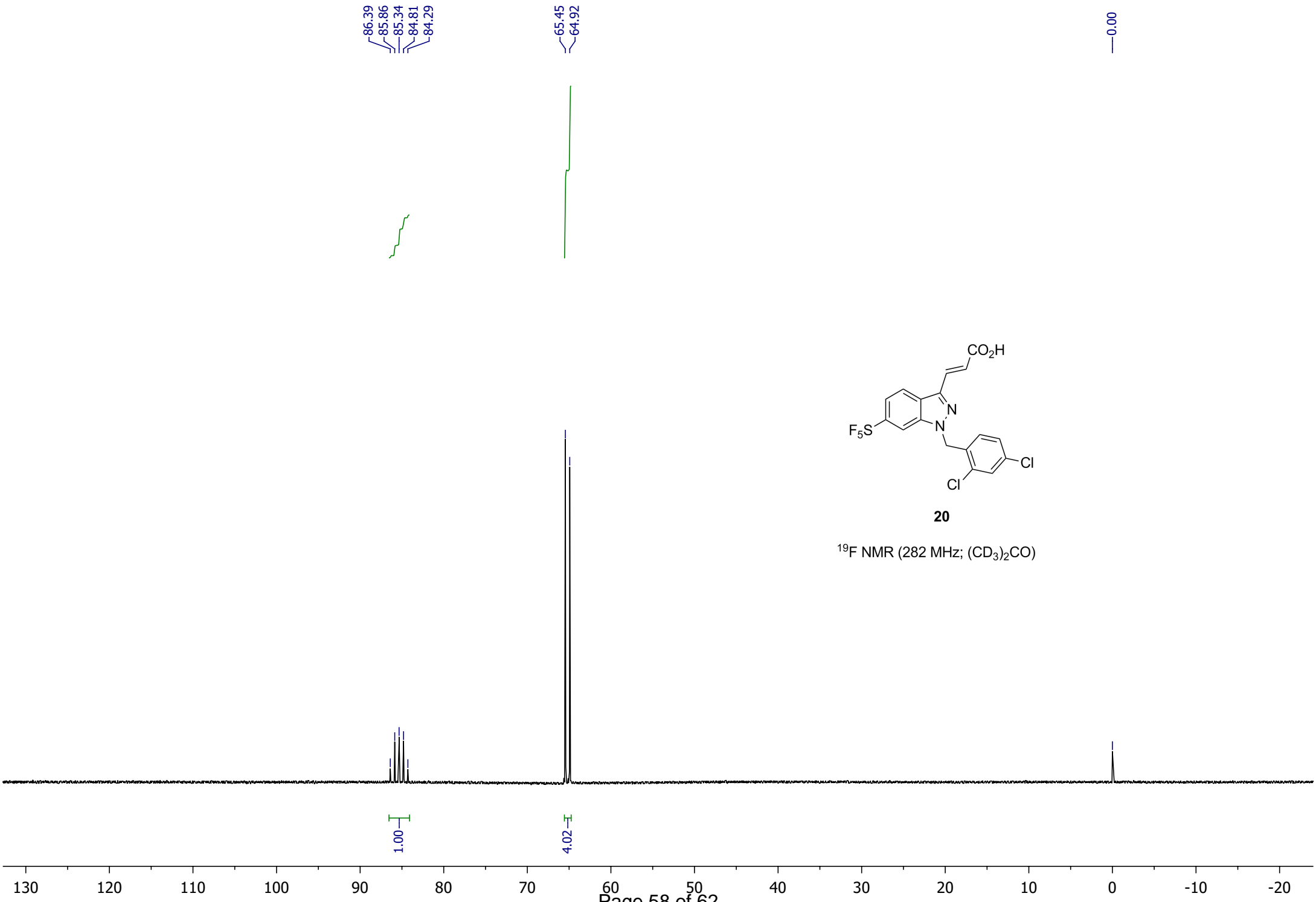
—50.50

—29.48

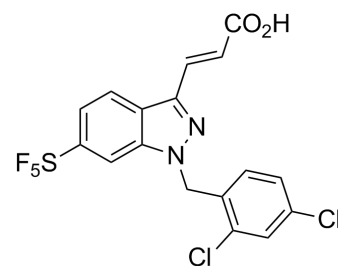
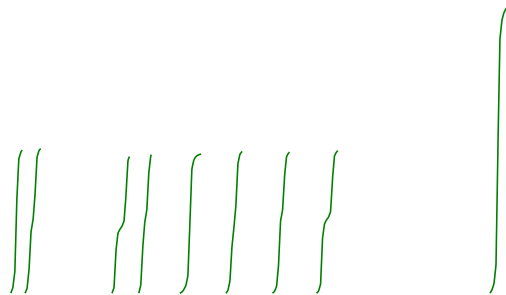


^{13}C NMR (125 MHz; $(\text{CD}_3)_2\text{CO}$)





8.47
8.47
8.40
8.37
7.96
7.90
7.82
7.81
7.79
7.78
7.57
7.57
7.36
7.35
7.33
7.32
7.11
7.08
6.88
6.83
5.97



20

¹H NMR (300 MHz; (CD₃)₂CO)

1.00
1.01
0.96
0.97
0.98
1.00
1.00
1.00

2.00

2.05

-0.01

10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5

—206.75

—174.18

—153.15

—153.02

—152.89

—146.40

—140.25

—134.66

—134.62

—134.13

—131.22

—129.85

—128.35

—124.91

—122.02

—118.07

—118.04

—118.00

—109.54

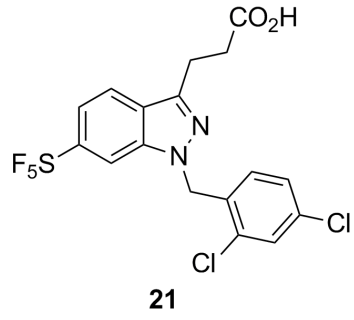
—109.50

—50.16

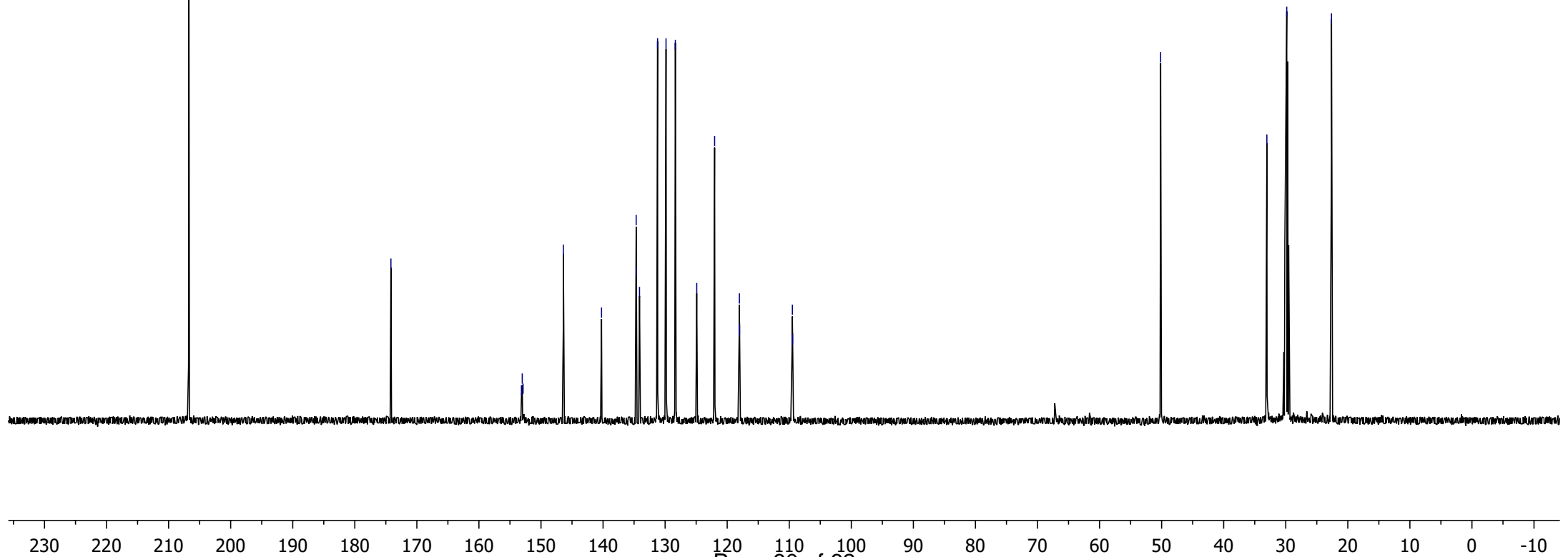
—33.04

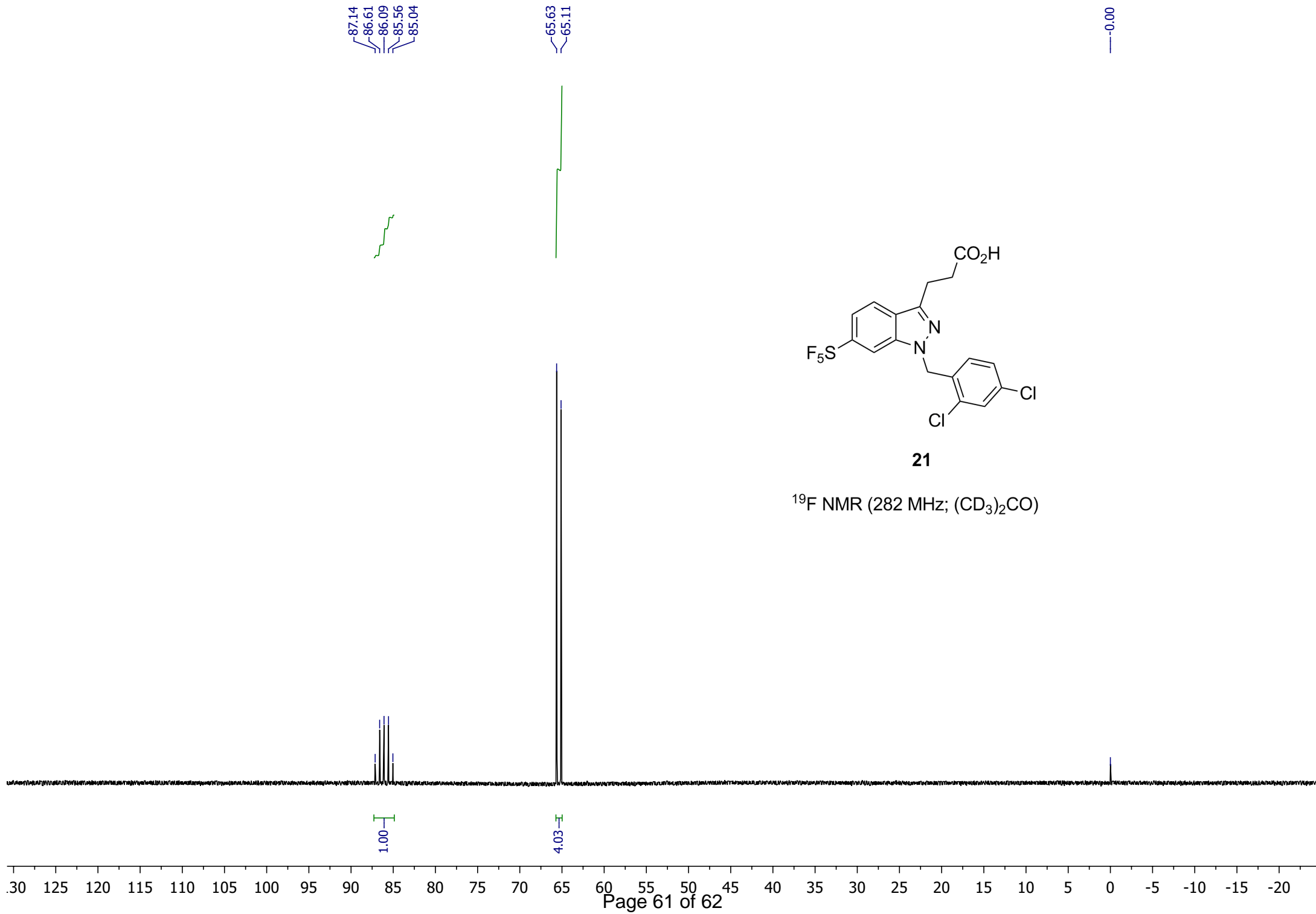
—29.84

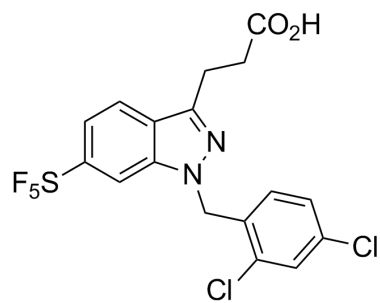
—22.63



¹³C NMR (125 MHz; (CD₃)₂CO)







21

$^1\text{H NMR}$ (300 MHz; $(\text{CD}_3)_2\text{CO}$)

