

Supporting Information for:

Synthesis of phenoxathiins using an iron-catalysed C–H thioarylation

Amy C. Dodds and Andrew Sutherland*

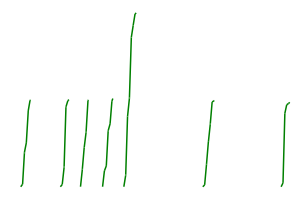
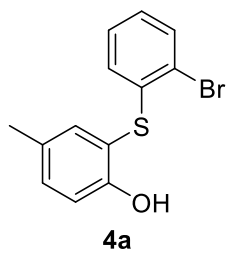
*School of Chemistry, The Joseph Black Building, University of Glasgow, Glasgow G12 8QQ,
UK. Email: Andrew.Sutherland@glasgow.ac.uk.*

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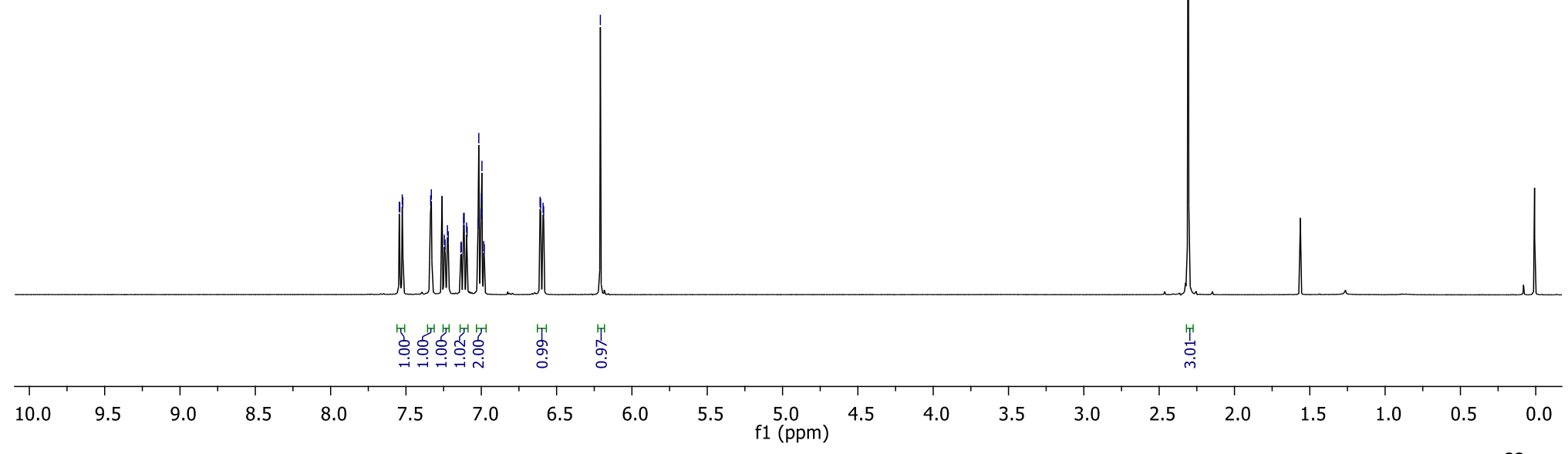
1. ^1H and $^{13}\text{C}\{^1\text{H}\}$ NMR Spectra for all Compounds	S2–S61
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¹H NMR (400 MHz, CDCl₃)

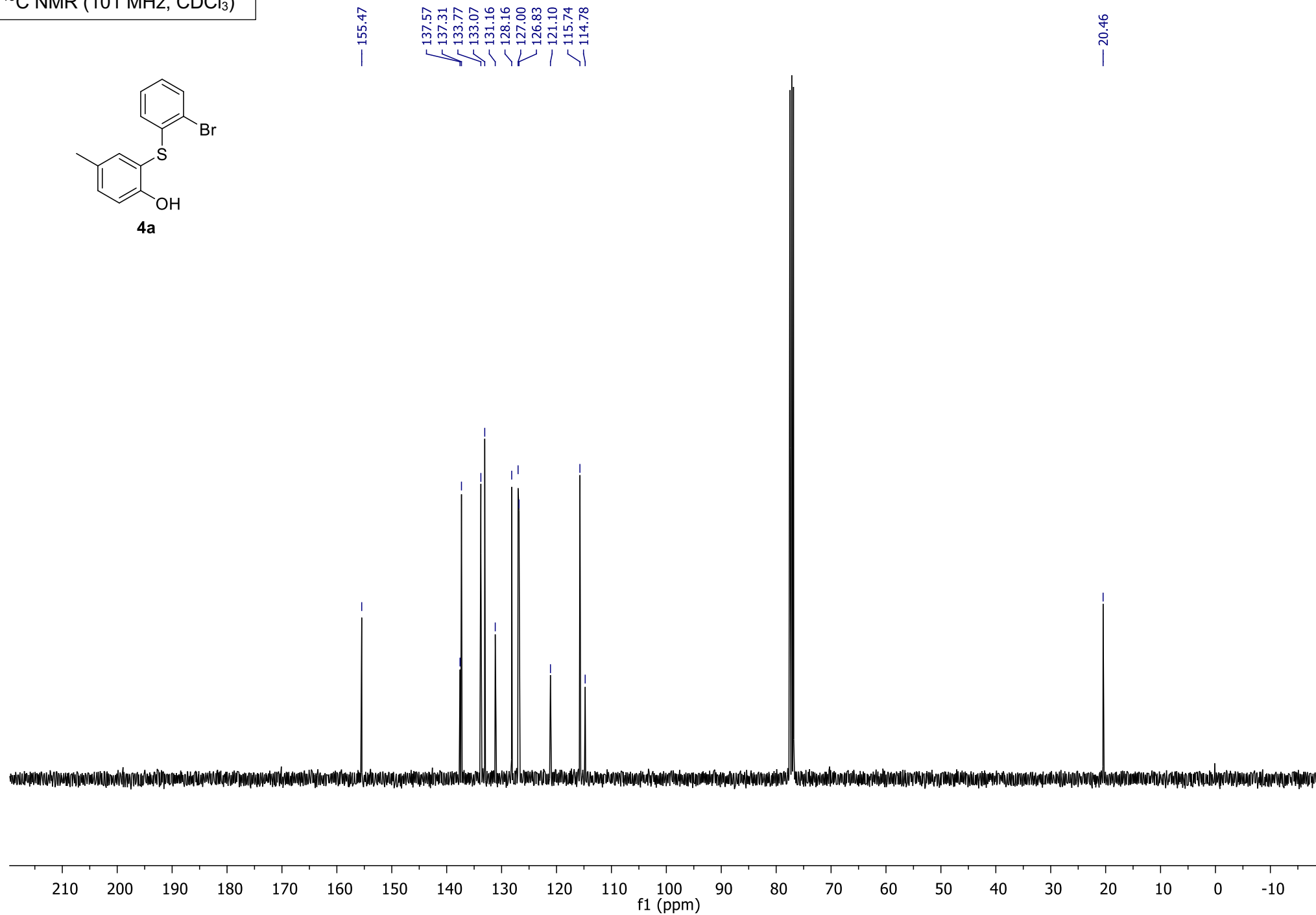
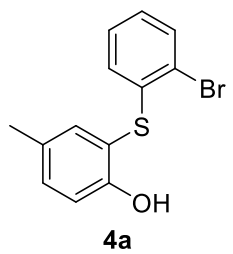
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7.33
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7.24
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7.22
7.14
7.13
7.12
7.12
7.11
7.10
7.09
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6.98
6.98
6.61
6.61
6.59
6.59
6.21



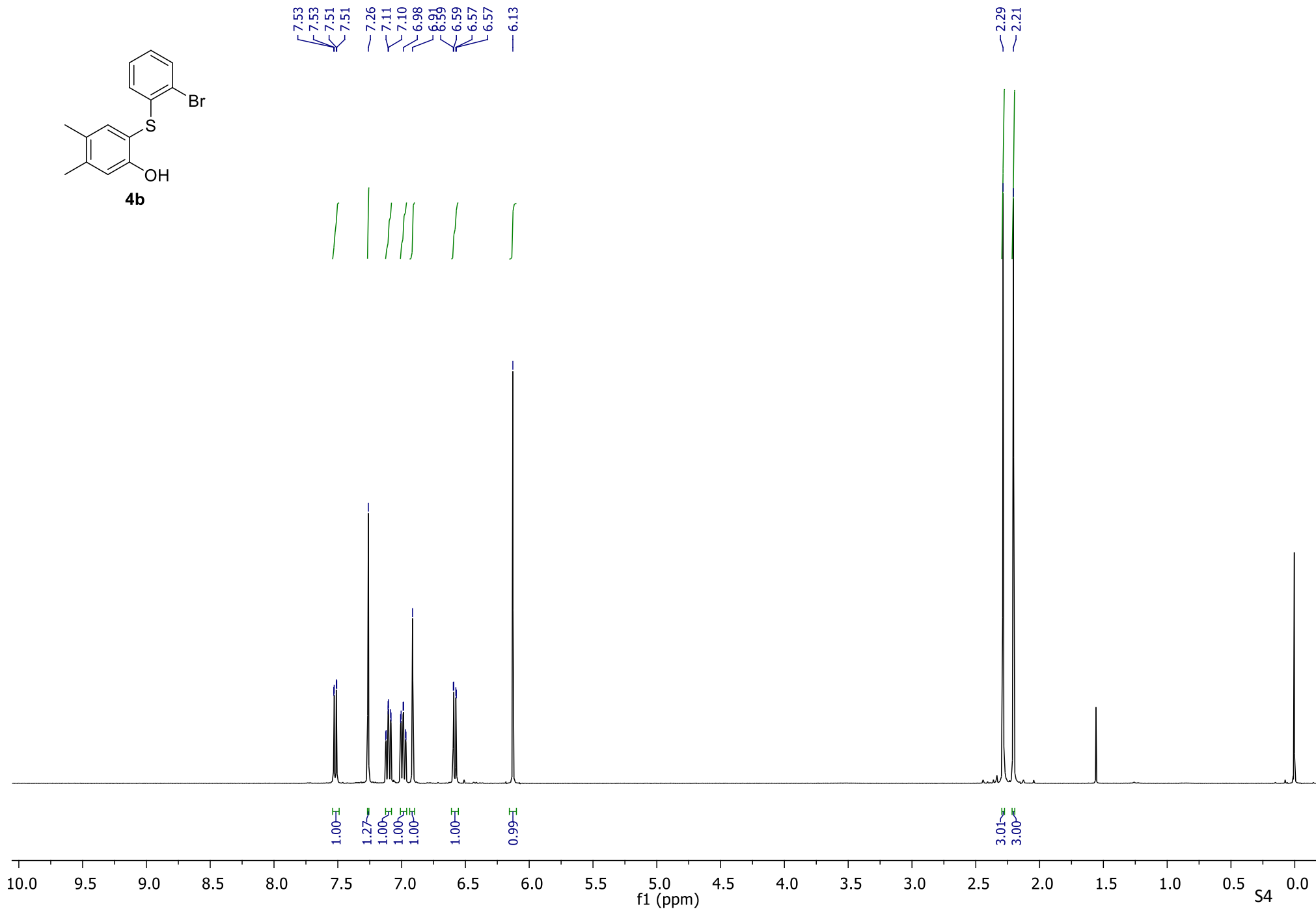
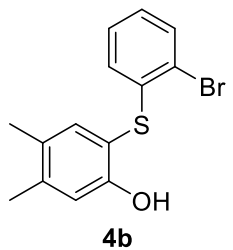
2.31



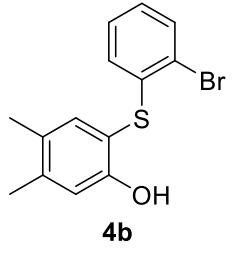
^{13}C NMR (101 MHz, CDCl_3)



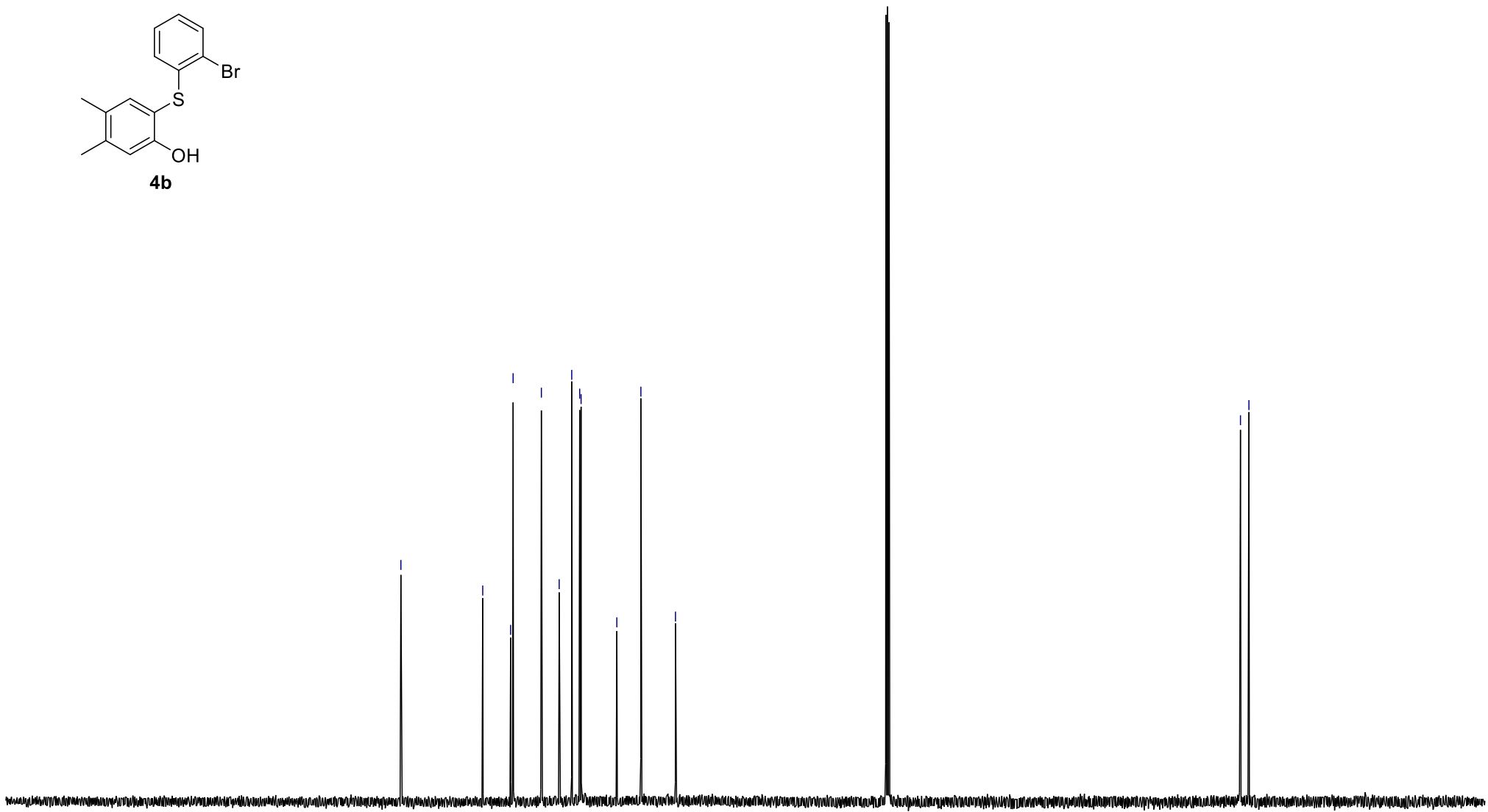
¹H NMR (400 MHz, CDCl₃)



¹³C NMR (101 MHz, CDCl₃)

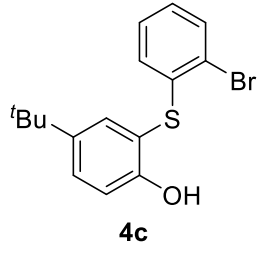


- 155.63
- 142.44
- 137.93
- 137.53
- 132.97
- 130.11
- 128.08
- 126.80
- 126.57
- 120.81
- 116.93
- 111.35
- 20.23
- 18.87



210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

¹H NMR (400 MHz, CDCl₃)



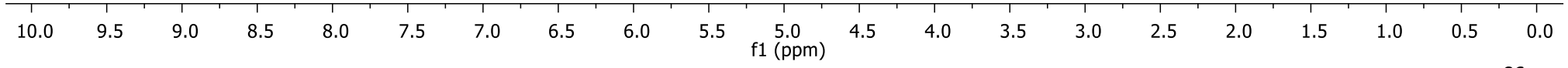
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7.55
7.54
7.53
7.49
7.48
7.46
7.46
7.12
7.12
7.06
7.04
6.58
6.57
6.55

//
///
/
/

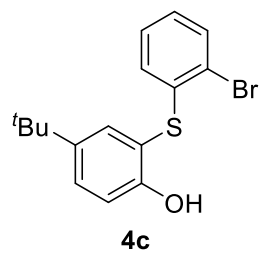
1.32

2.00
1.01
1.00
1.00
1.02
1.00
1.00

9.00

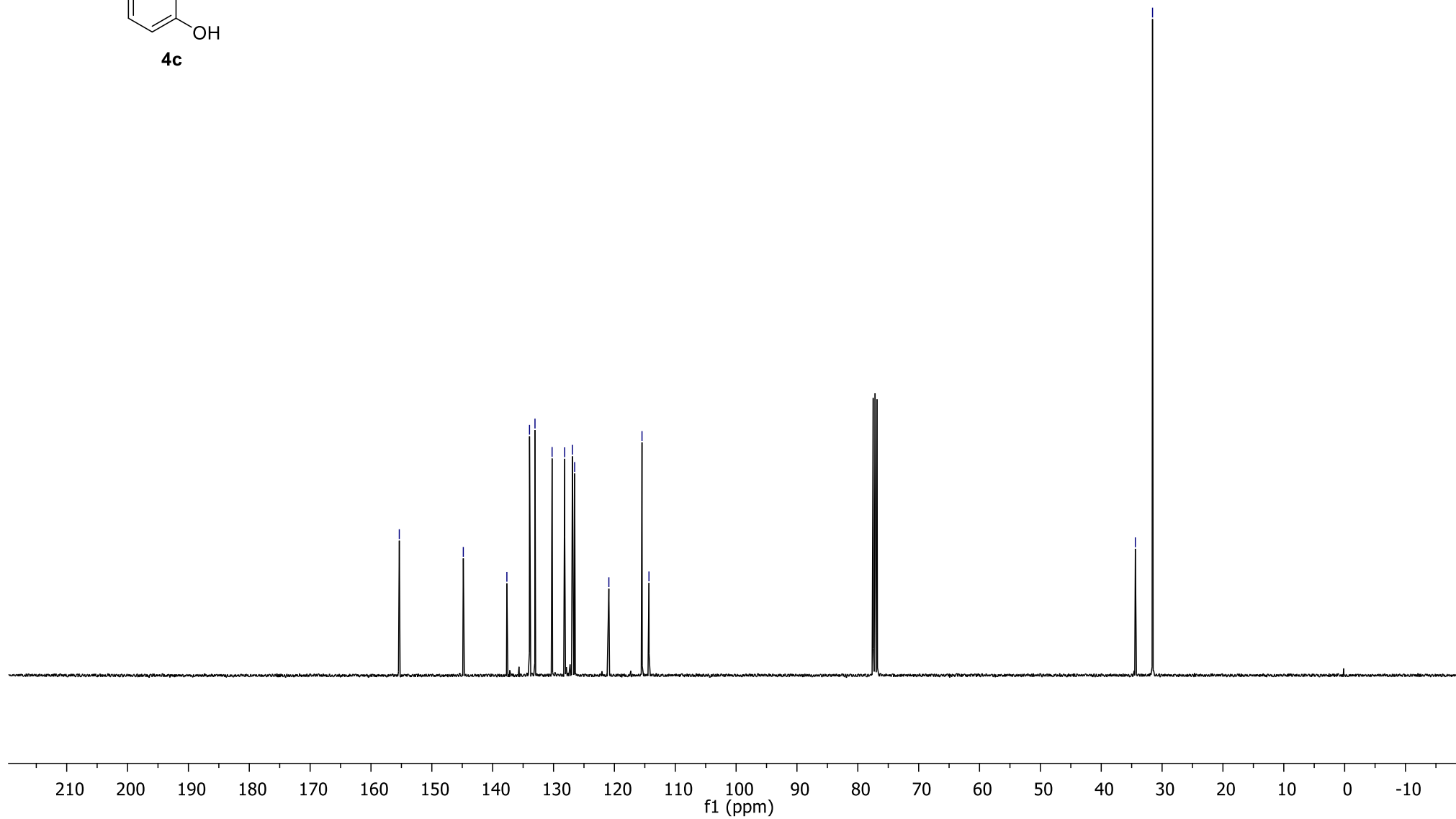


^{13}C NMR (101 MHz, CDCl_3)

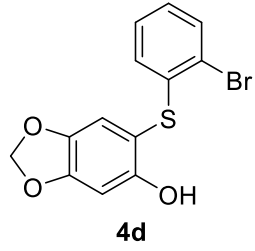


— 155.35
— 144.83
— 137.67
— 133.95
— 133.05
— 130.24
— 128.17
— 126.89
— 126.53
— 120.91
— 115.46
— 114.32

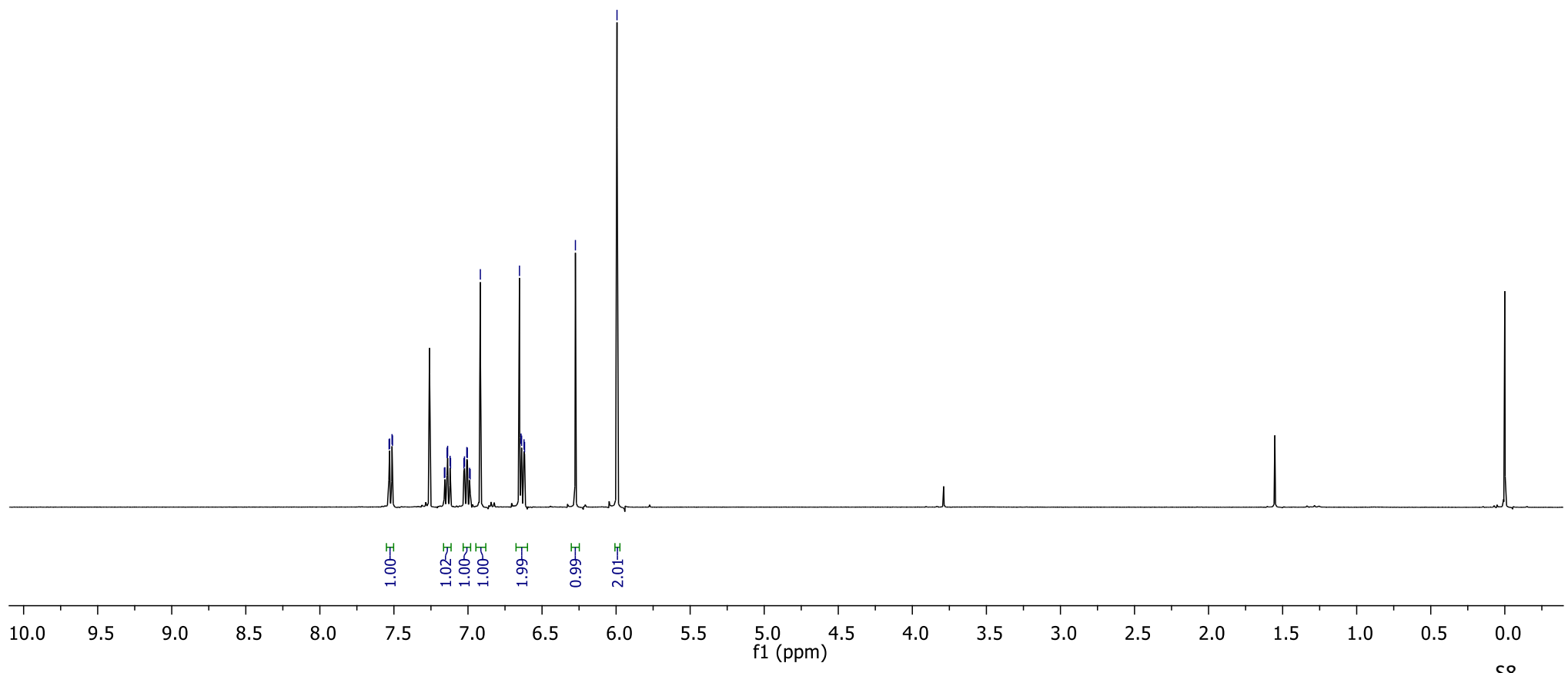
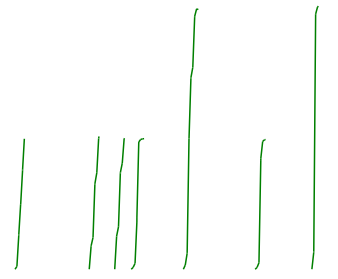
— 34.39
— 31.57



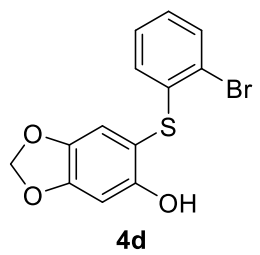
¹H NMR (400 MHz, CDCl₃)



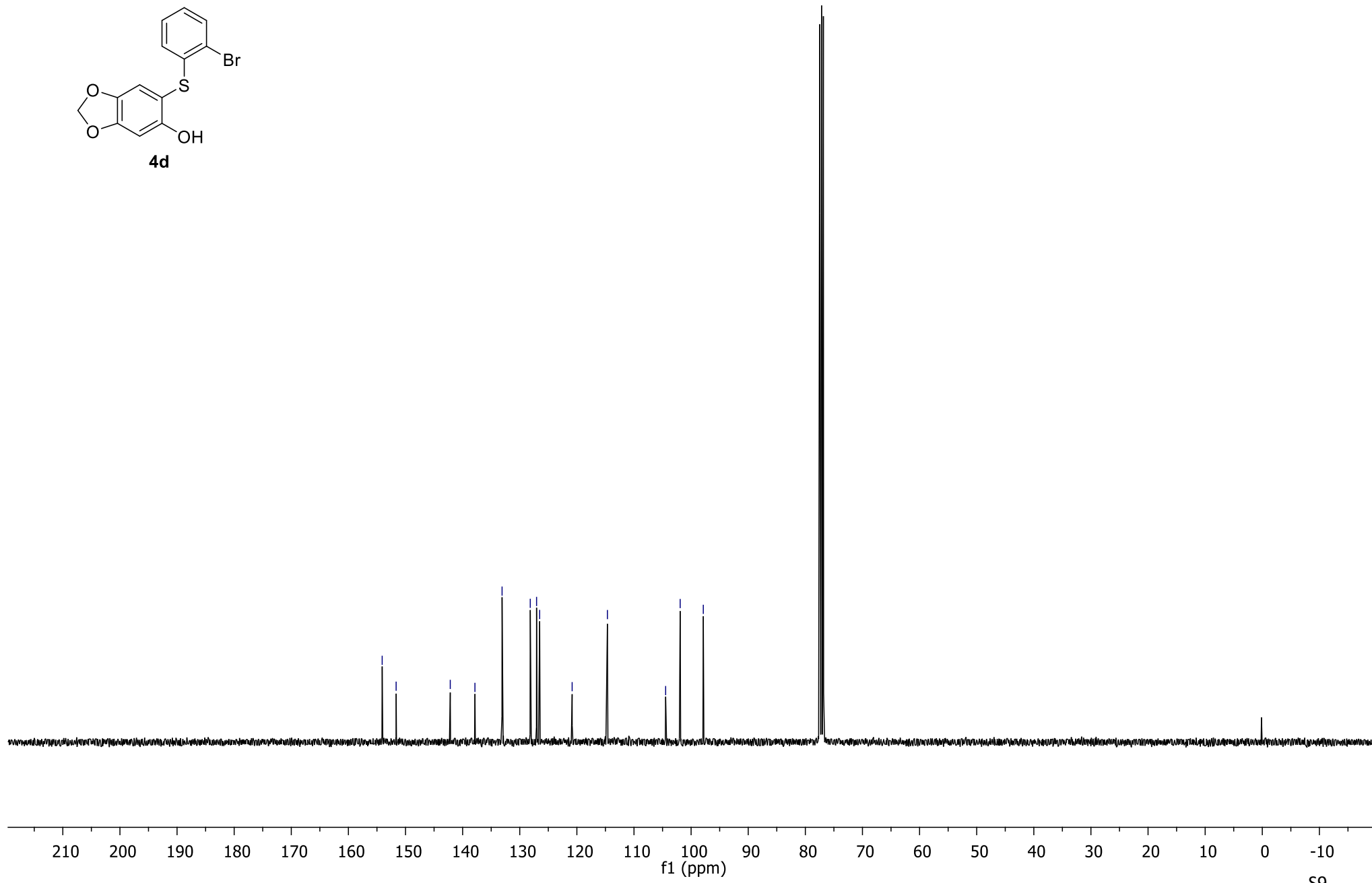
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7.53
7.51
7.51
7.14
7.14
7.01
6.92
6.65
6.64
6.64
6.64
5.99



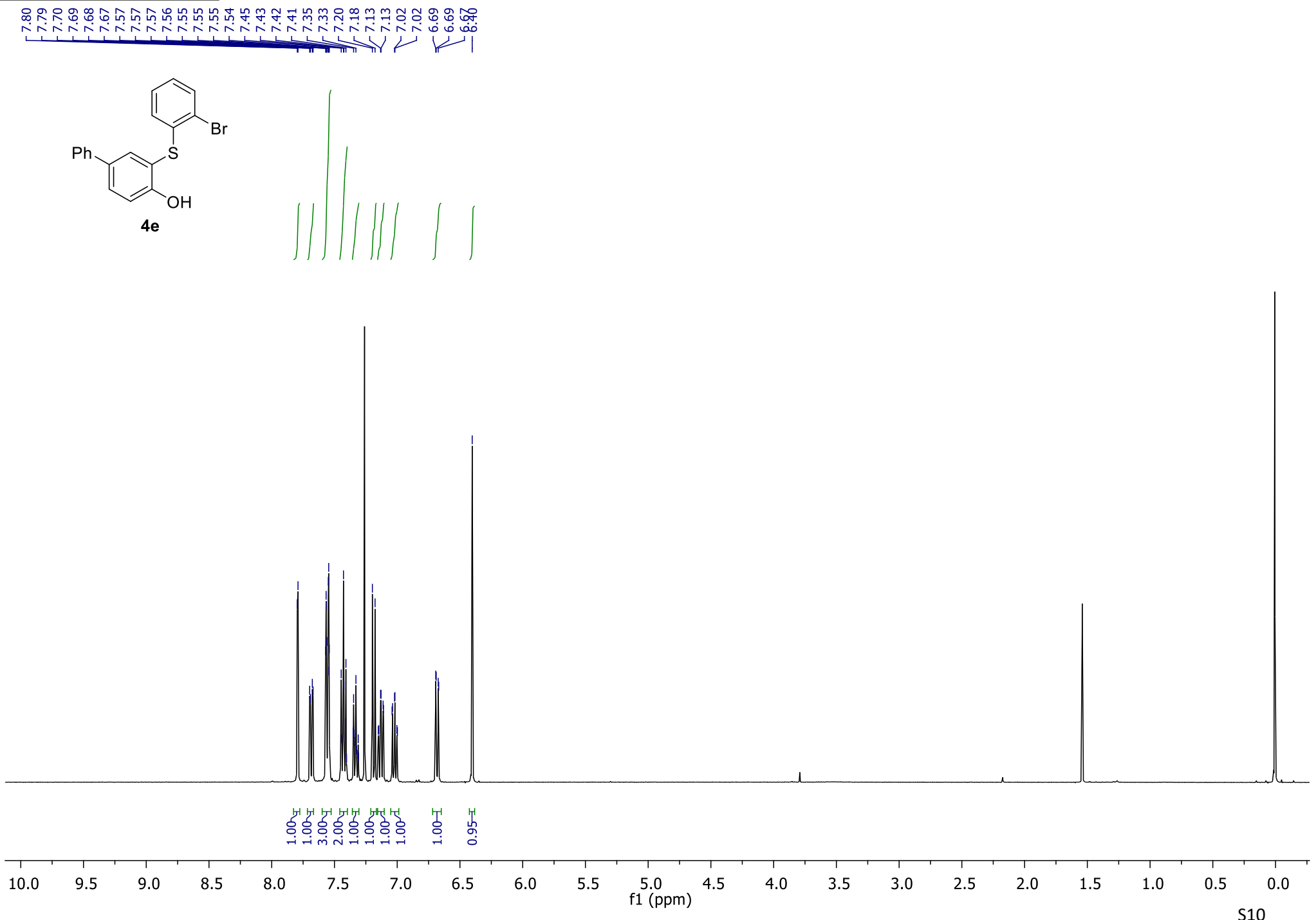
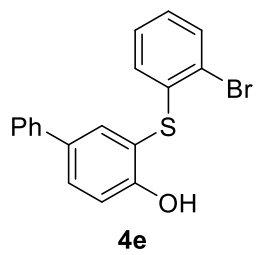
¹³C NMR (101 MHz, CDCl₃)



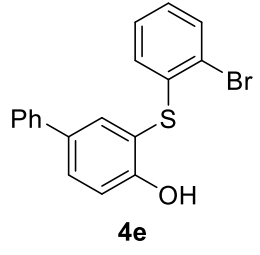
154.09
151.64
142.17
137.85
133.09
128.16
127.05
126.51
120.83
114.65
104.46
101.91
97.86



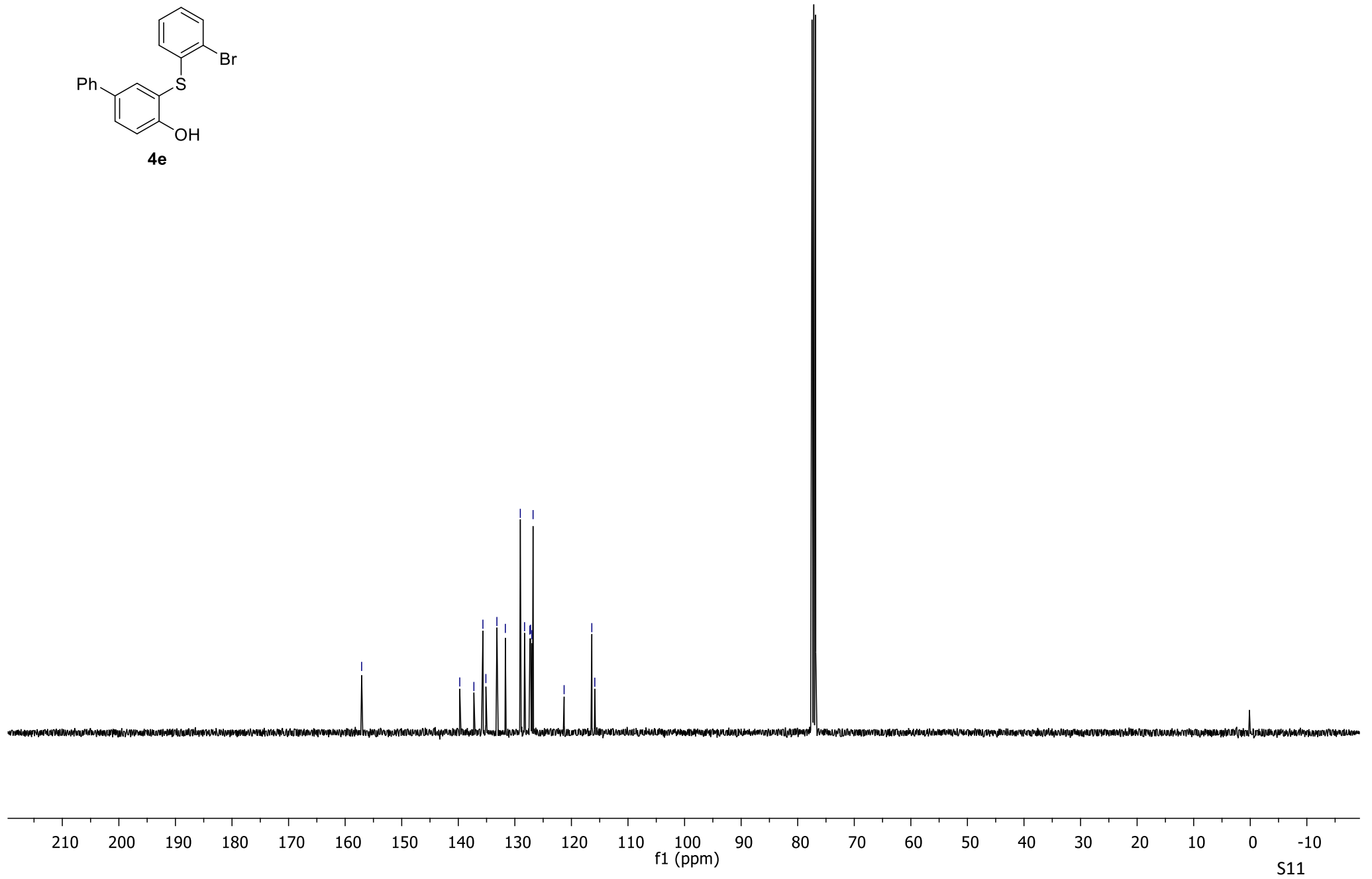
¹H NMR (400 MHz, CDCl₃)



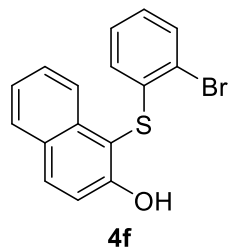
^{13}C NMR (101 MHz, CDCl_3)



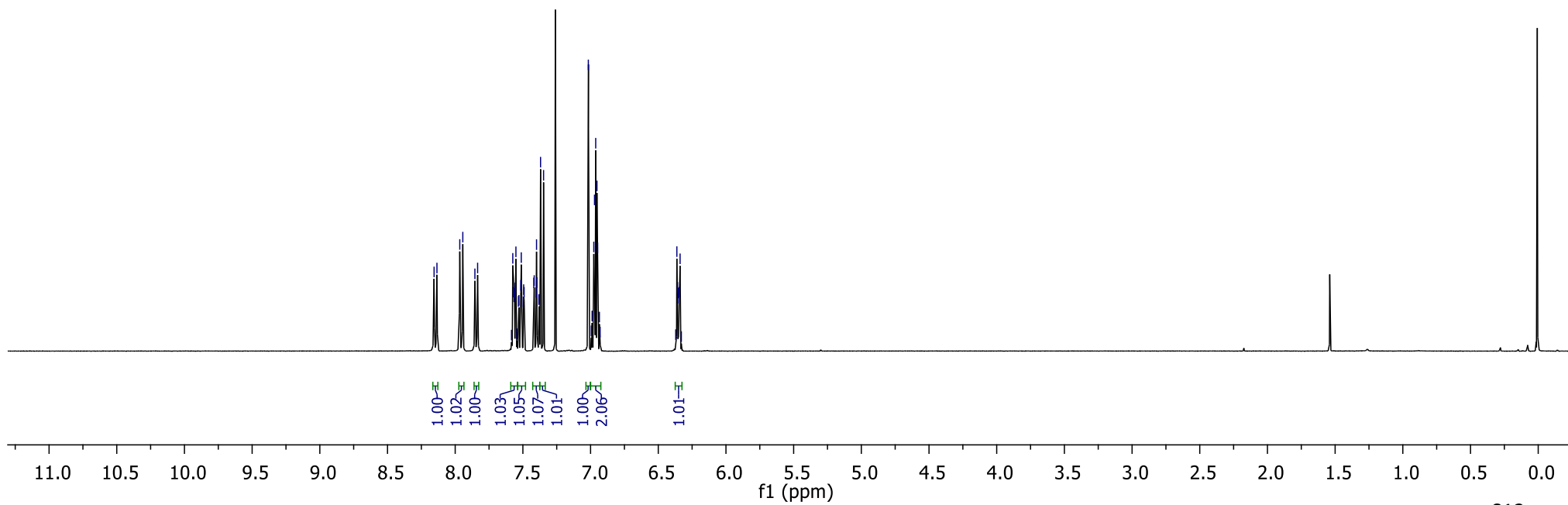
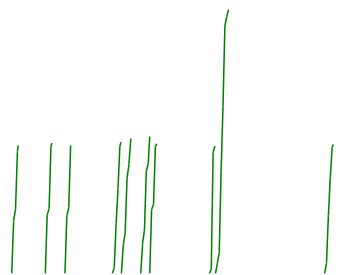
- 157.08
- 139.74
- 137.24
- 135.65
- 135.12
- 133.17
- 131.68
- 129.04
- 128.28
- 127.34
- 127.23
- 127.00
- 126.78
- 121.29
- 116.40
- 115.87



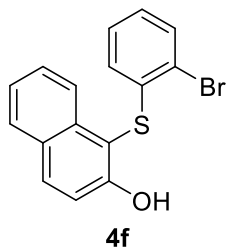
¹H NMR (400 MHz, CDCl₃)



8.16
8.13
7.97
7.94
7.85
7.83
7.57
7.57
7.57
7.56
7.55
7.54
7.53
7.53
7.52
7.51
7.51
7.49
7.49
7.42
7.42
7.40
7.40
7.40
7.38
7.38
7.37
7.35
7.02
6.96
6.95
6.37
6.36
6.36
6.35
6.35
6.35
6.34
6.33

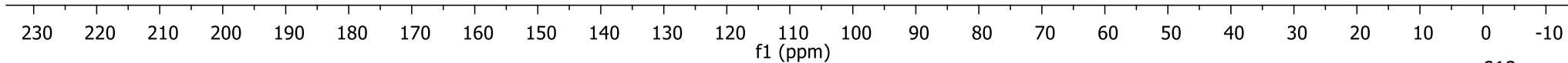


^{13}C NMR (101 MHz, CDCl_3)



157.42
136.59
135.44
133.46
133.12
129.71
128.79
128.35
128.14
126.96
126.53
124.67
124.24
121.22
117.22
107.32

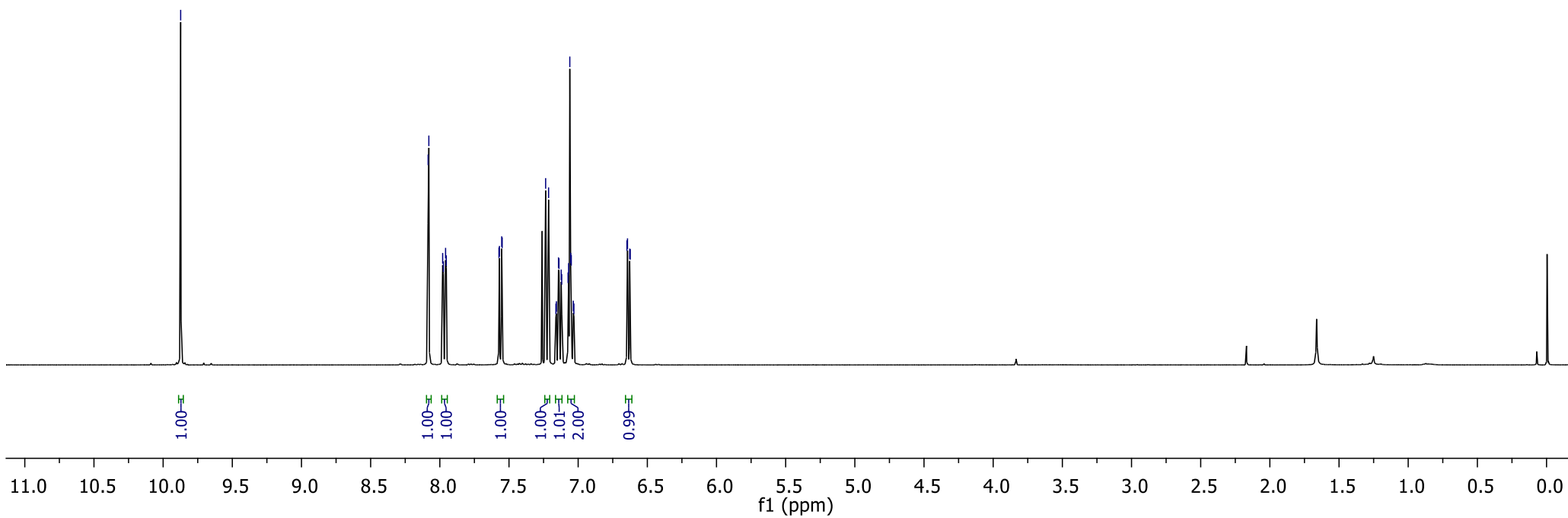
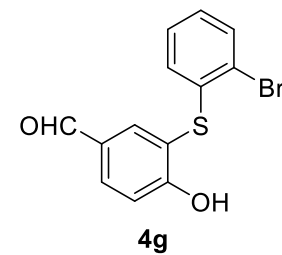
77.48
77.16
76.84



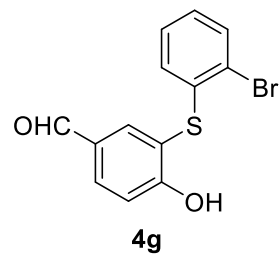
¹H NMR (400 MHz, CDCl₃)

9.87

8.08
7.98
7.97
7.96
7.95
7.55
7.55
7.24
7.21
7.06
7.05
6.65
6.64
6.63
6.62



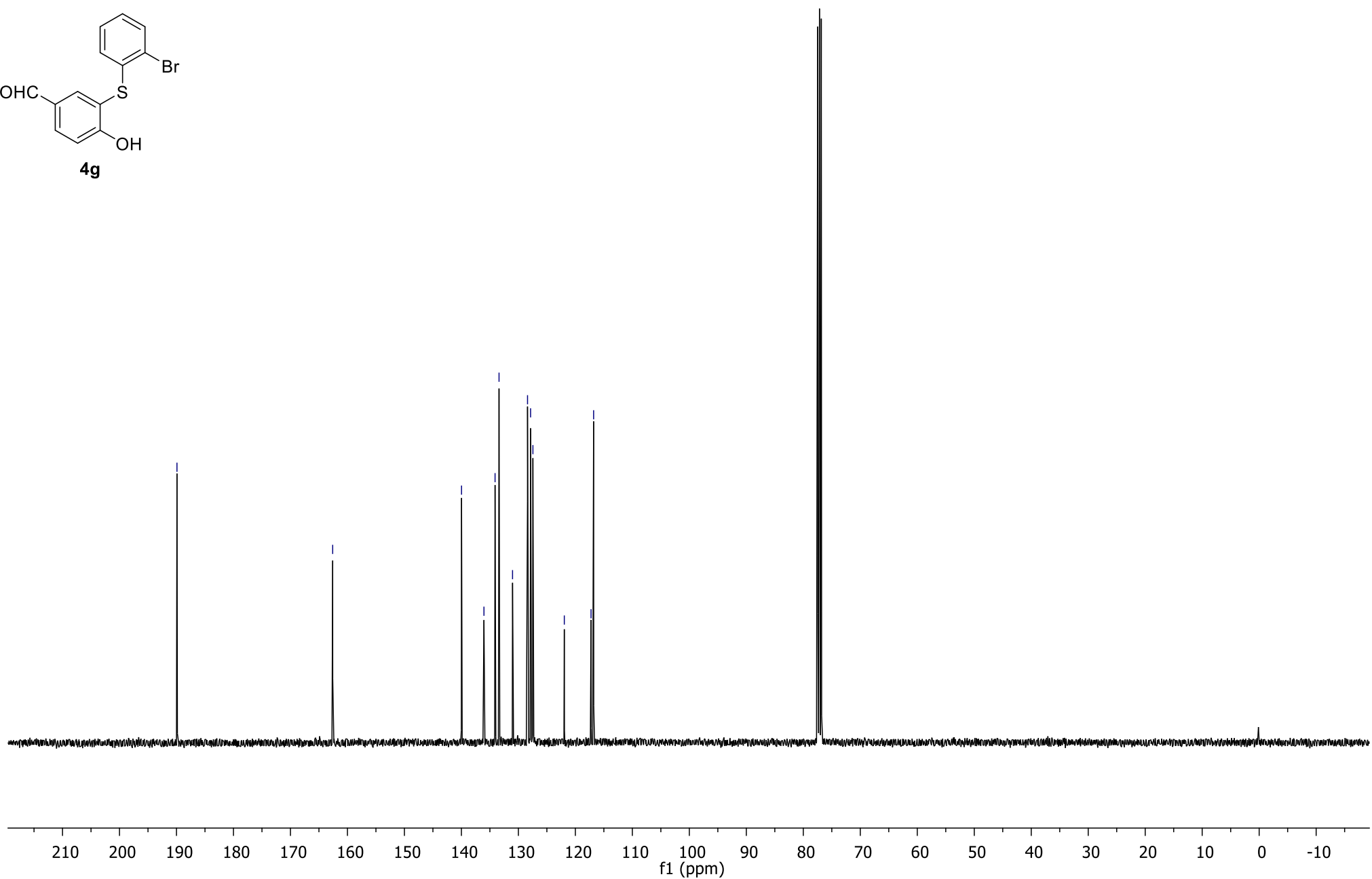
¹³C NMR (101 MHz, CDCl₃)



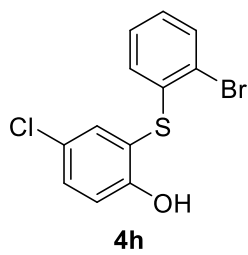
189.91

162.59

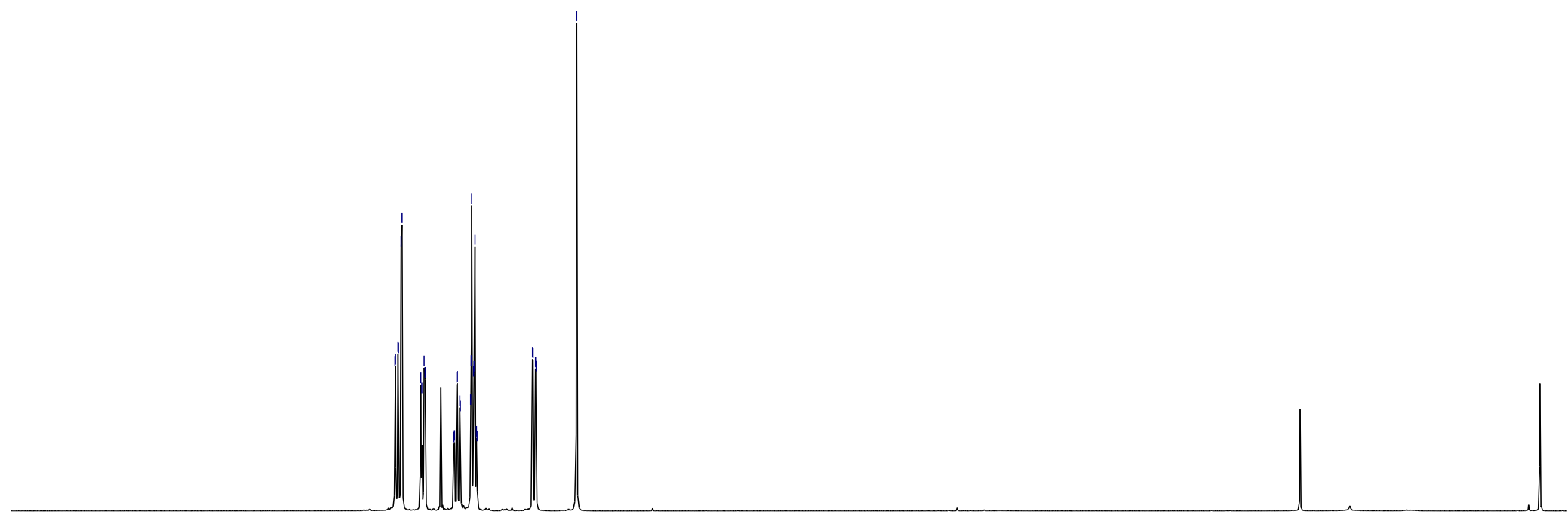
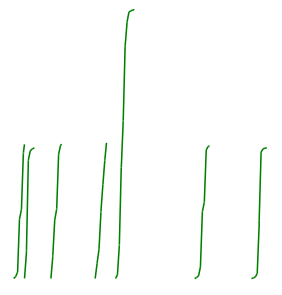
139.98
136.04
134.09
133.40
131.02
128.39
127.86
127.45
121.94
117.25
116.80



¹H NMR (400 MHz, CDCl₃)



7.56
7.56
7.54
7.54
7.52
7.52
7.39
7.39
7.37
7.36
7.17
7.17
7.15
7.15
7.13
7.13
7.06
7.06
7.06
7.04
7.04
7.03
7.03
7.02
6.65
6.65
6.63

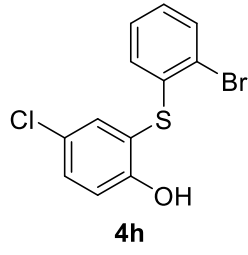


1.00
0.97
1.00
1.01
2.00
0.99
0.97

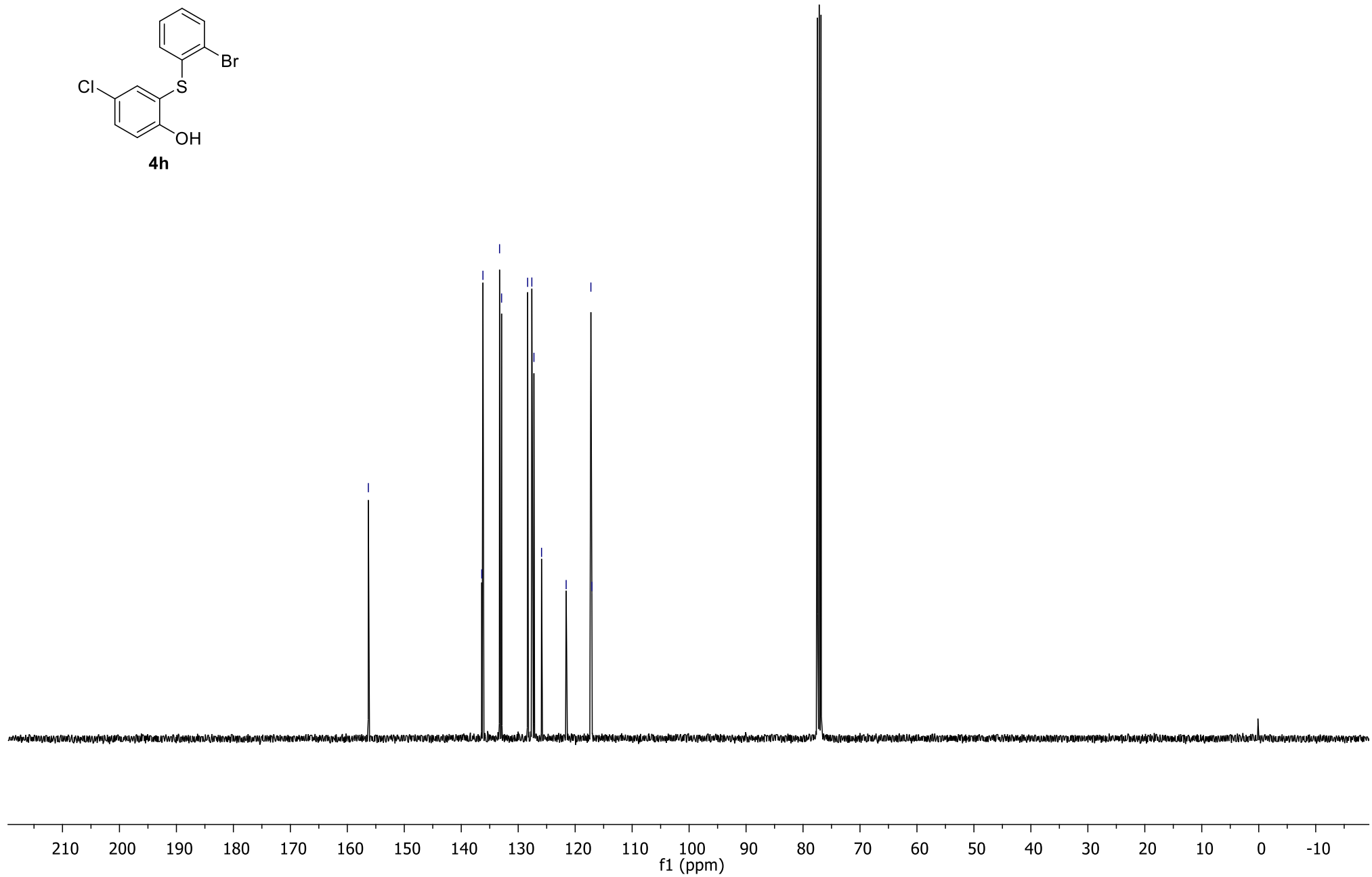
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0

f1 (ppm)

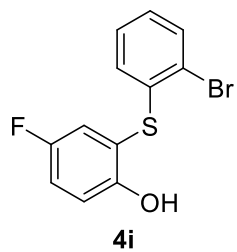
^{13}C NMR (101 MHz, CDCl_3)



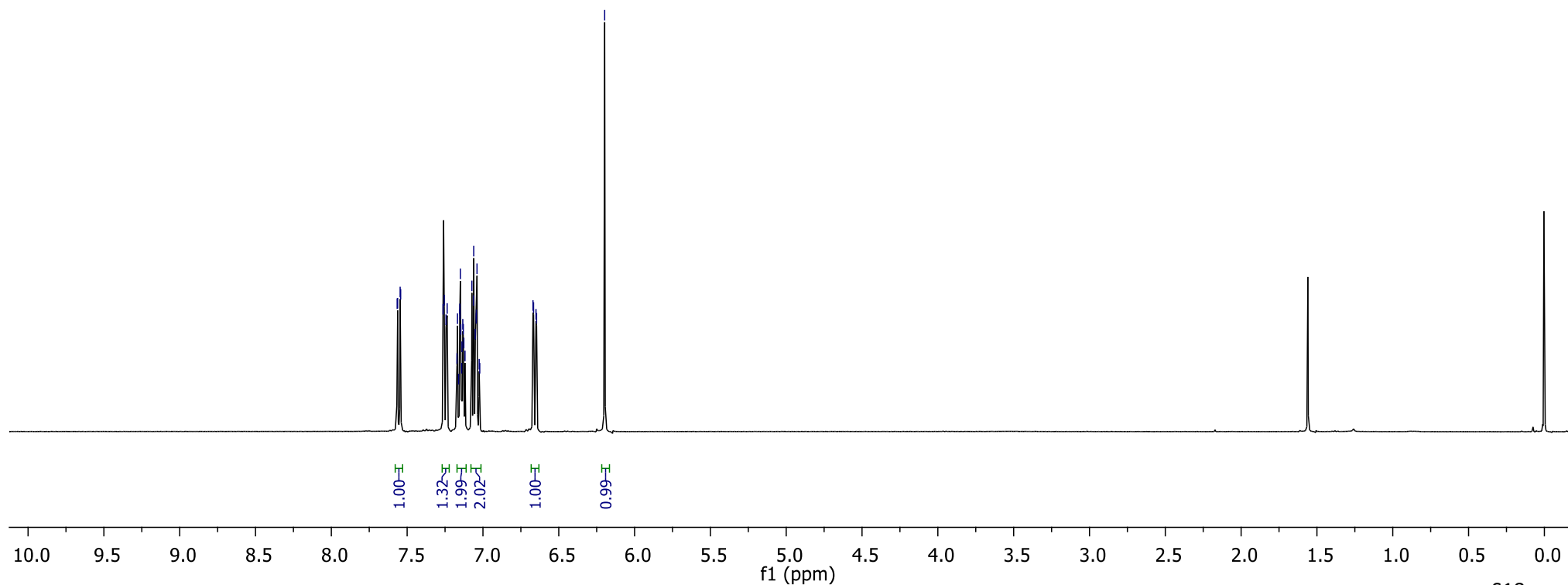
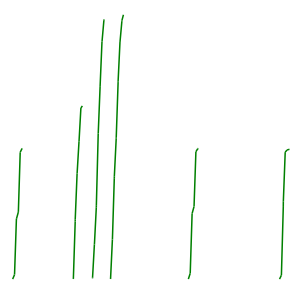
- 156.31
- 136.40
- 136.19
- 133.27
- 132.92
- 128.35
- 127.61
- 127.24
- 125.90
- 121.58
- 117.24
- 117.07



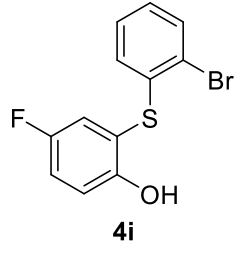
¹H NMR (400 MHz, CDCl₃)



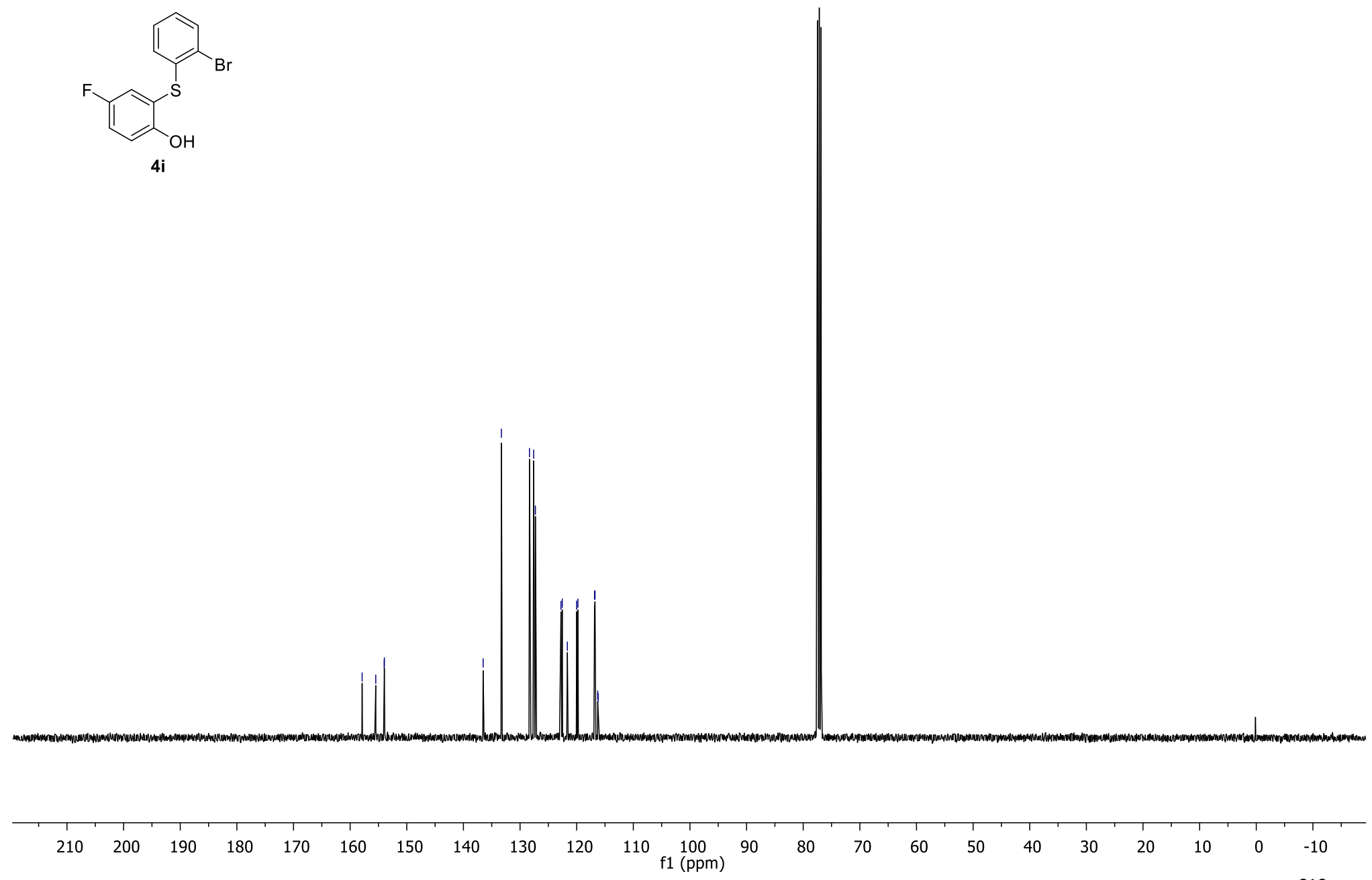
7.57
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7.07
7.06
6.84
6.67
6.65
6.65
6.20



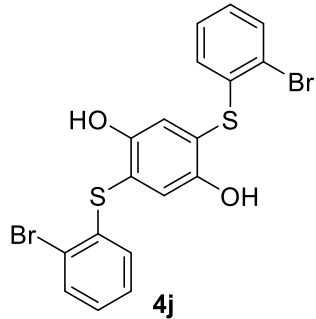
¹³C NMR (101 MHz, CDCl₃)



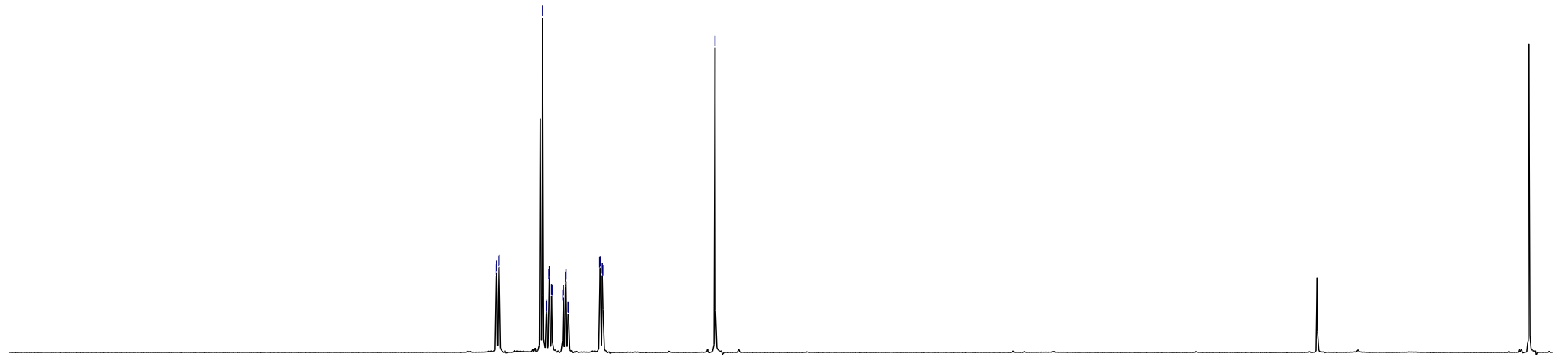
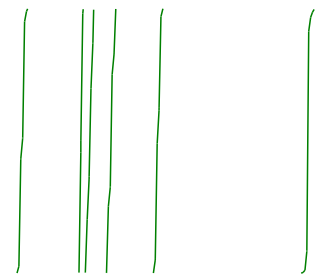
- 157.88
- 155.48
- 153.98
- 153.96
- 136.52
- 133.29
- 128.33
- 127.60
- 127.29
- 122.77
- 122.54
- 121.65
- 120.01
- 119.78
- 116.84
- 116.77
- 116.29
- 116.20



¹H NMR (400 MHz, CDCl₃)



7.59
7.58
7.57
7.56
7.24
7.20
7.19
7.18
7.07
7.07
6.82
6.81
8.88



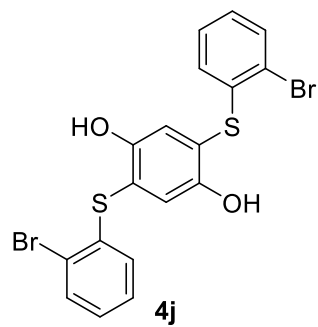
2.00
2.00
1.99
2.00
2.00
2.00

11.0 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

f1 (ppm)

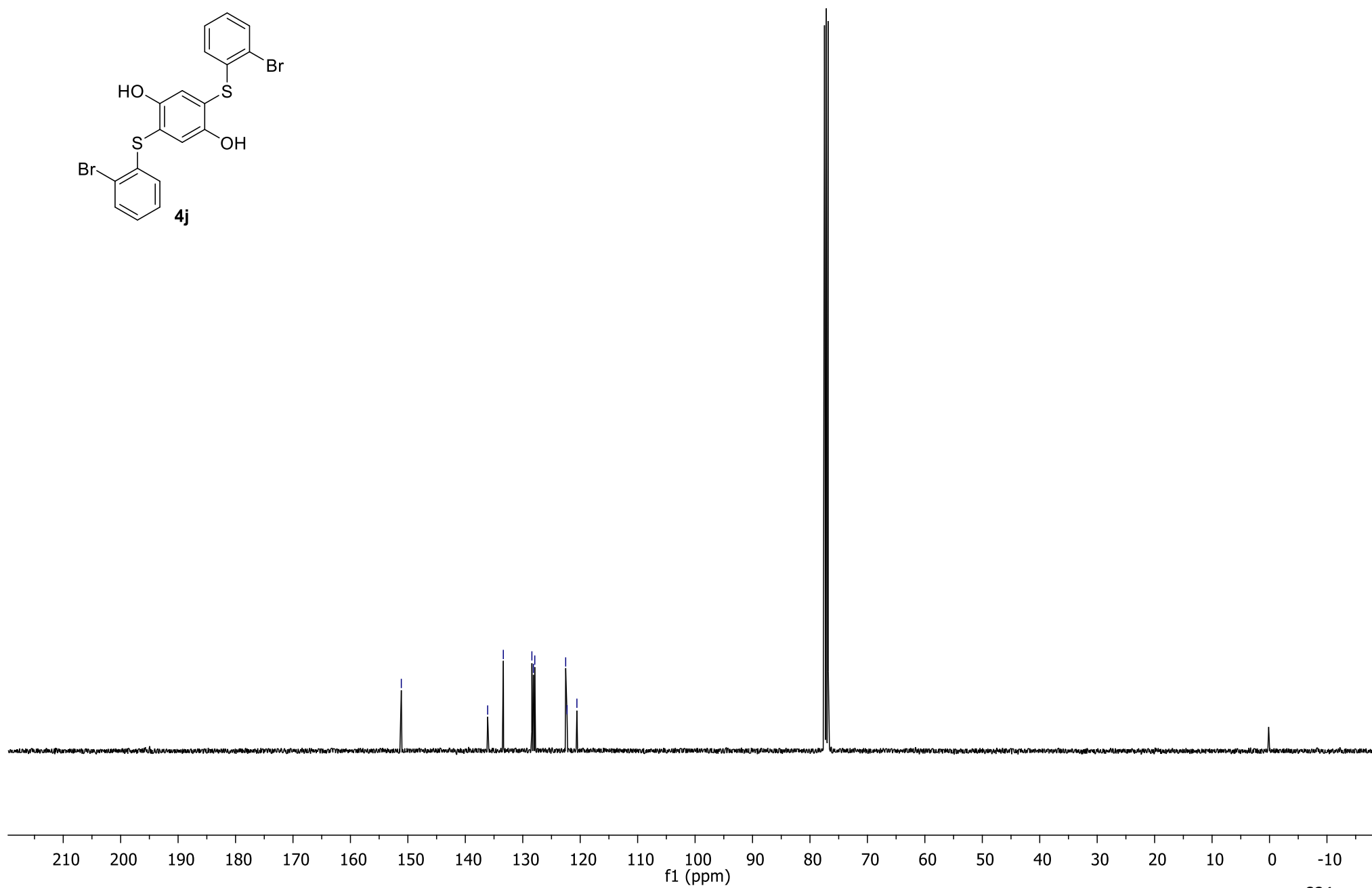
S20

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

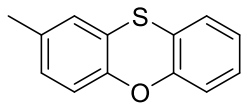


151.14

136.11
133.38
128.41
128.14
127.91
122.53
122.29
120.57



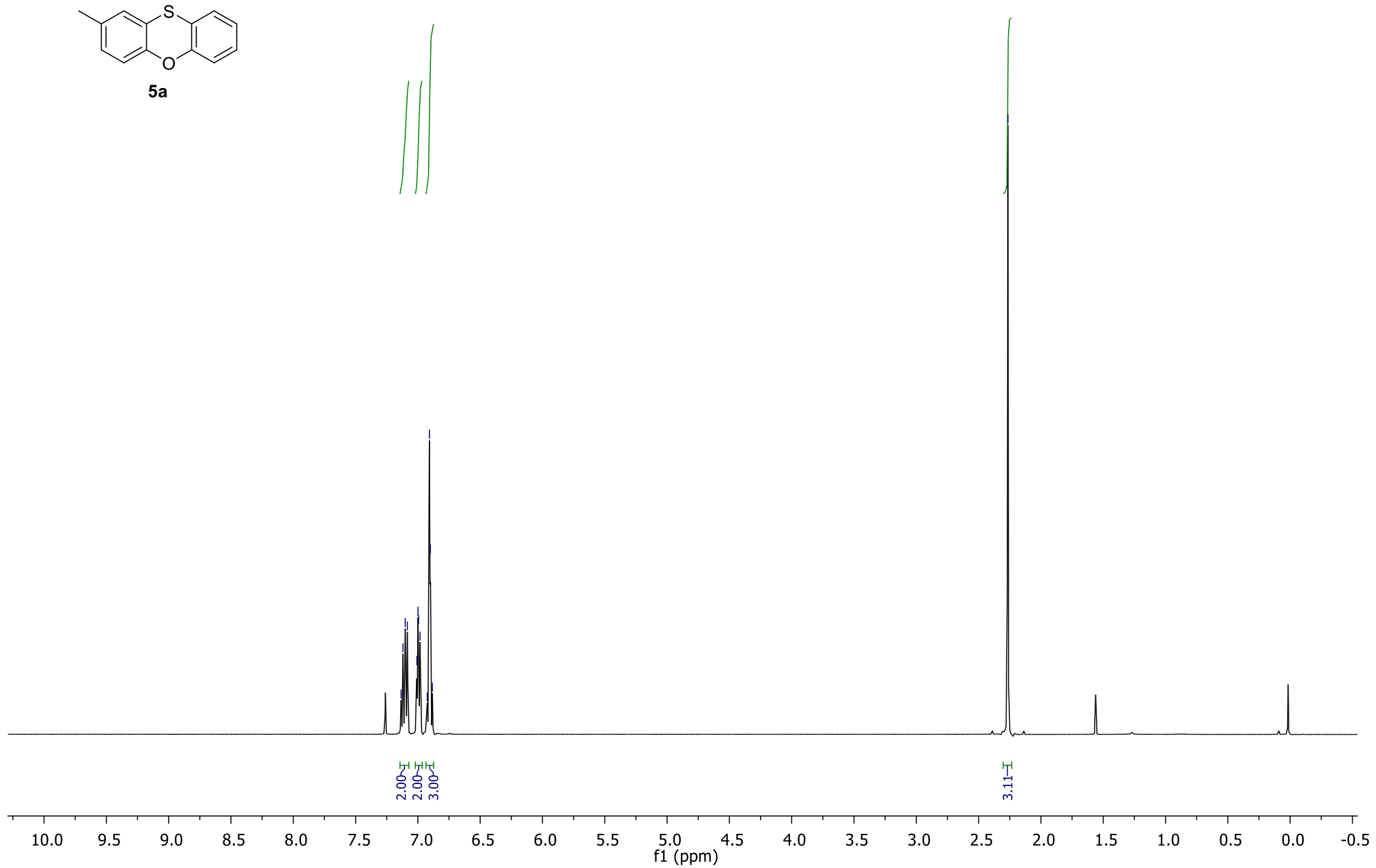
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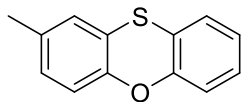
5a

7.13
7.12
7.10
7.08
7.01
7.00
6.99
6.98
6.92
6.91
6.90
6.88

2.26



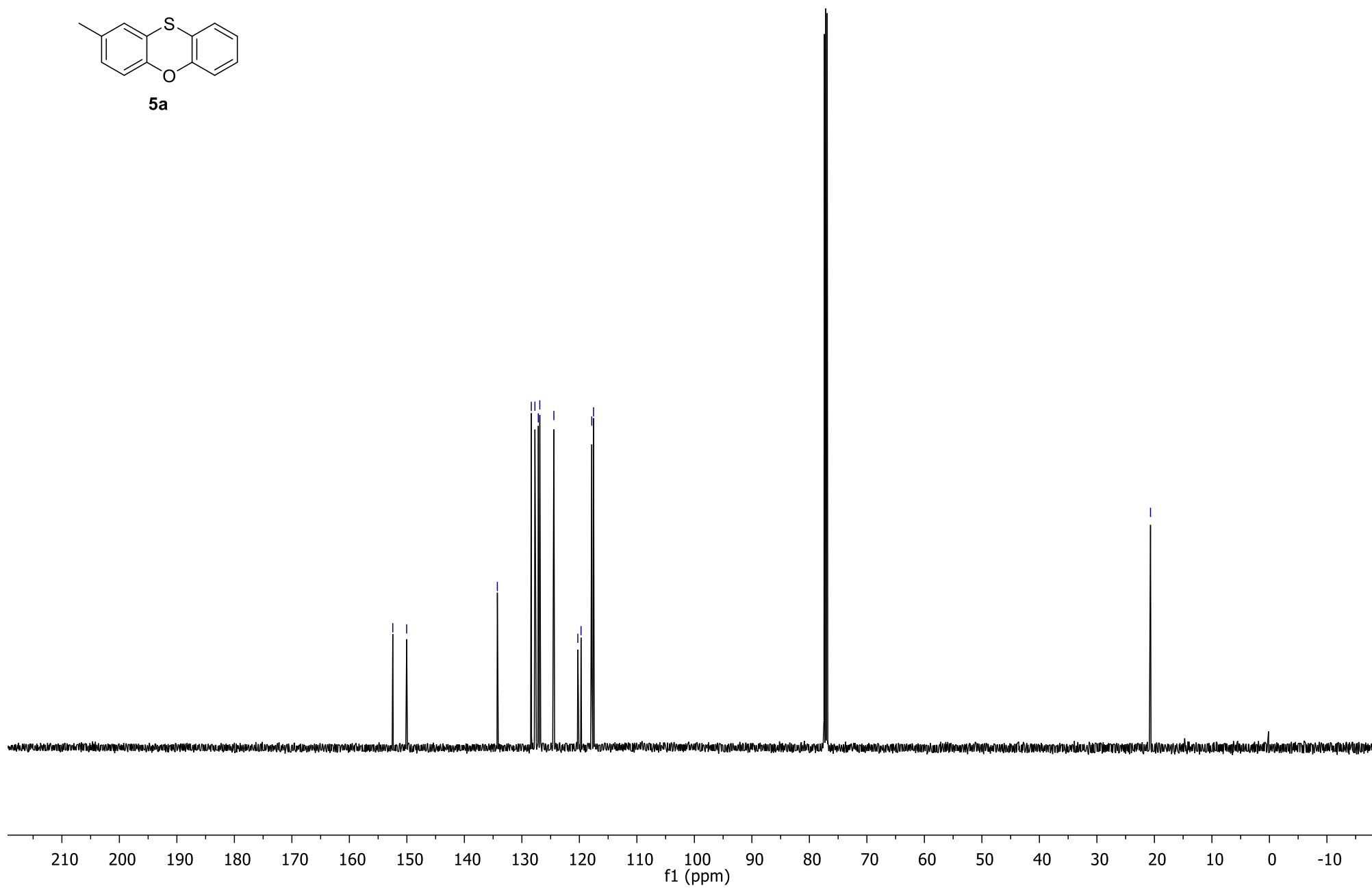
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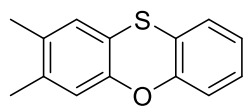
5a

152.45
150.04
134.27
128.36
127.73
127.15
126.89
124.44
120.27
119.71
117.85
117.54

20.68



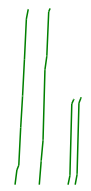
¹H NMR (400 MHz, CDCl₃)



5b

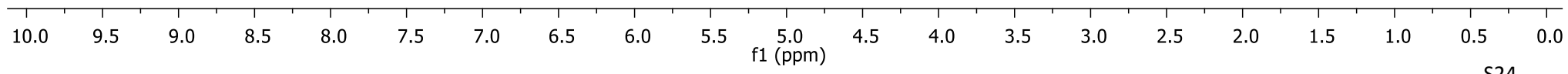
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7.12
7.11
7.10
7.10
7.09
7.09
7.08
7.07
7.07
7.00
7.00
6.99
6.98
6.98
6.97
6.96
6.96
6.85
6.81

2.20
2.17

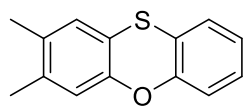


2.00
2.01
0.98
1.00

3.00
3.00



¹³C NMR (101 MHz, CDCl₃)

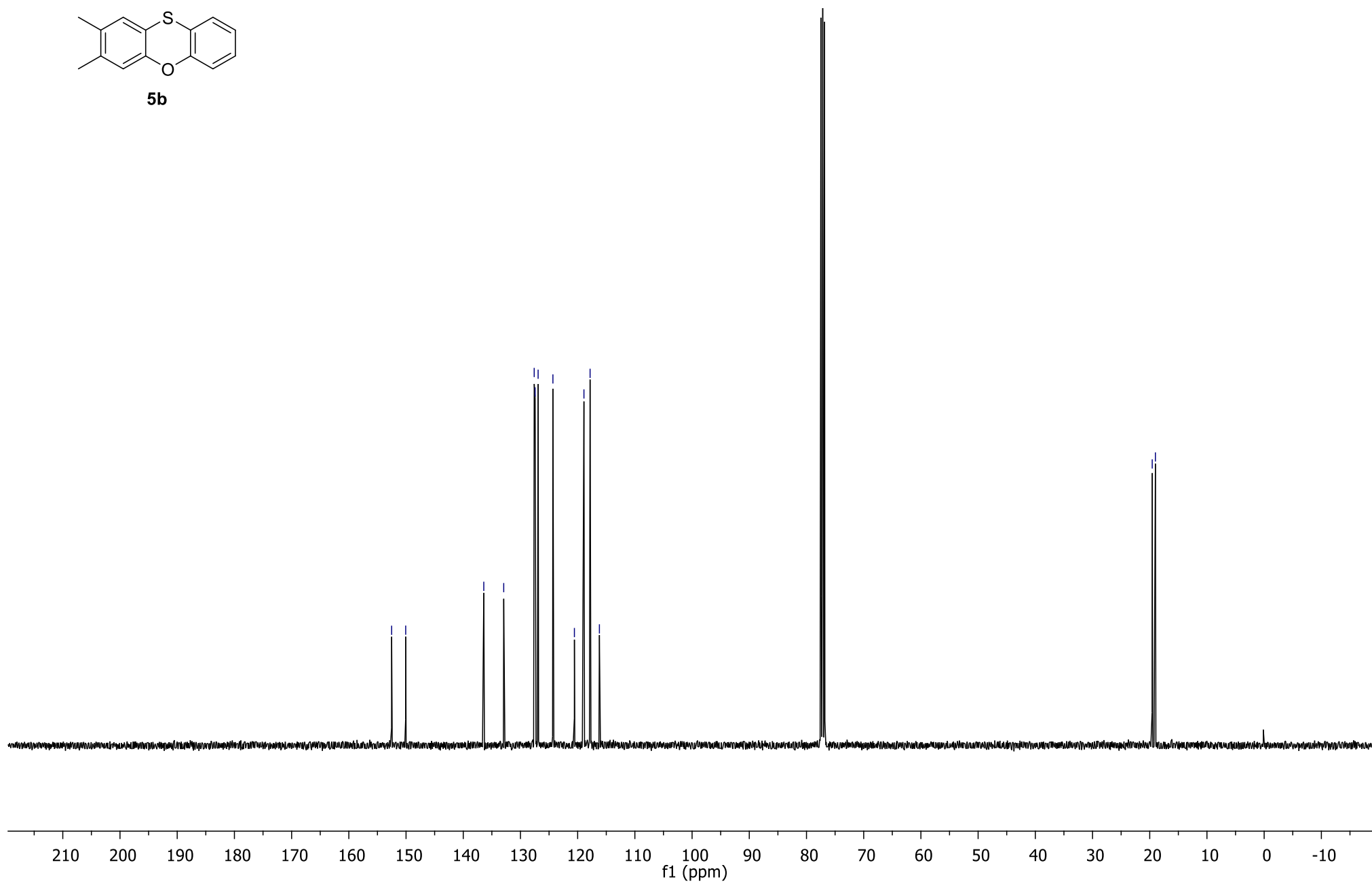


5b

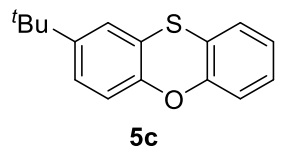
152.52
150.06

136.41
132.92
127.60
127.44
126.91
124.33
120.57
118.90
117.83
116.20

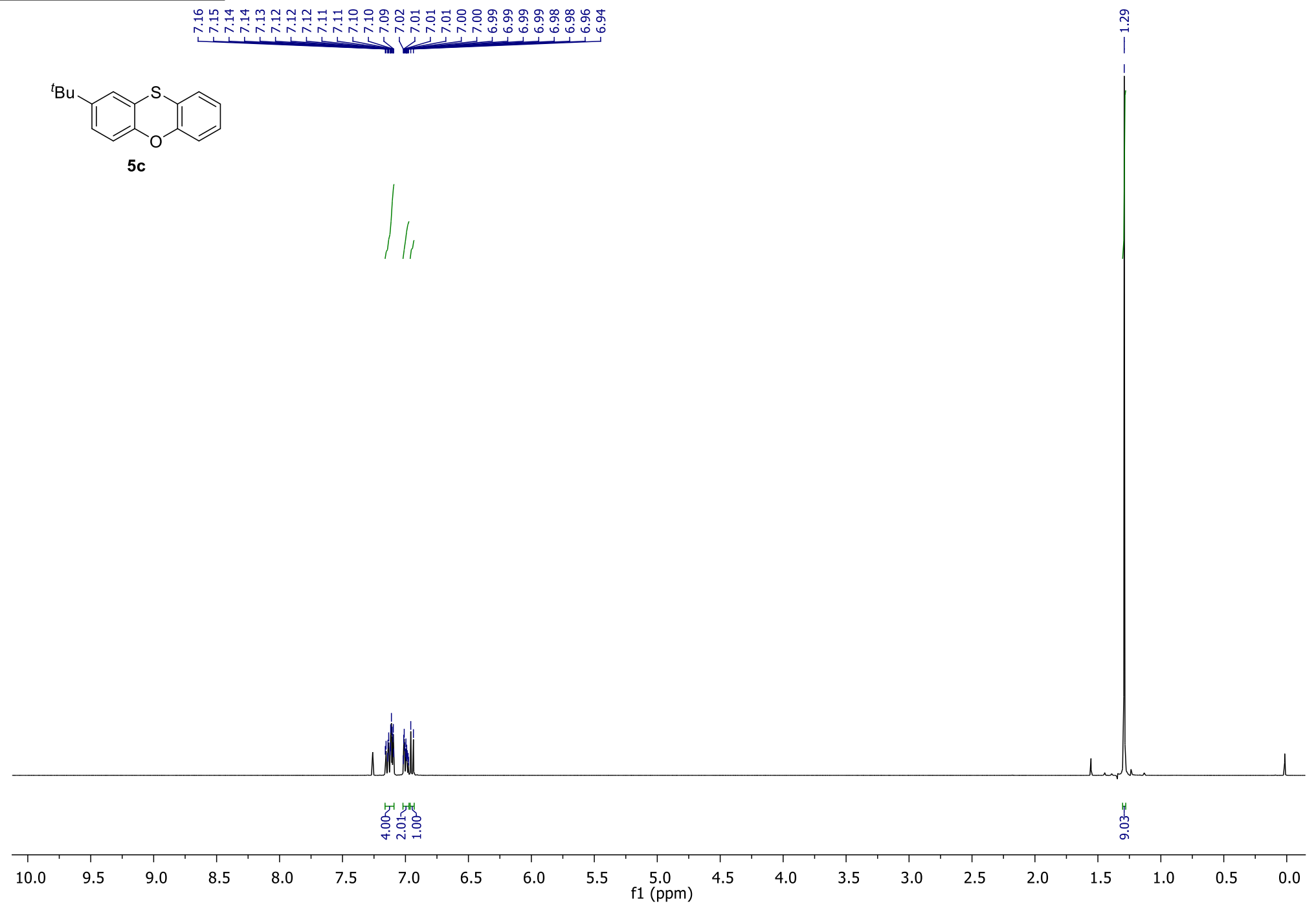
19.56
19.01



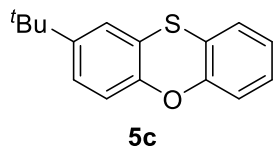
¹H NMR (400 MHz, CDCl₃)



7.16
7.15
7.14
7.14
7.13
7.12
7.12
7.11
7.11
7.10
7.10
7.09
7.02
7.01
7.01
7.01
7.00
7.00
6.99
6.99
6.99
6.98
6.98
6.96
6.94



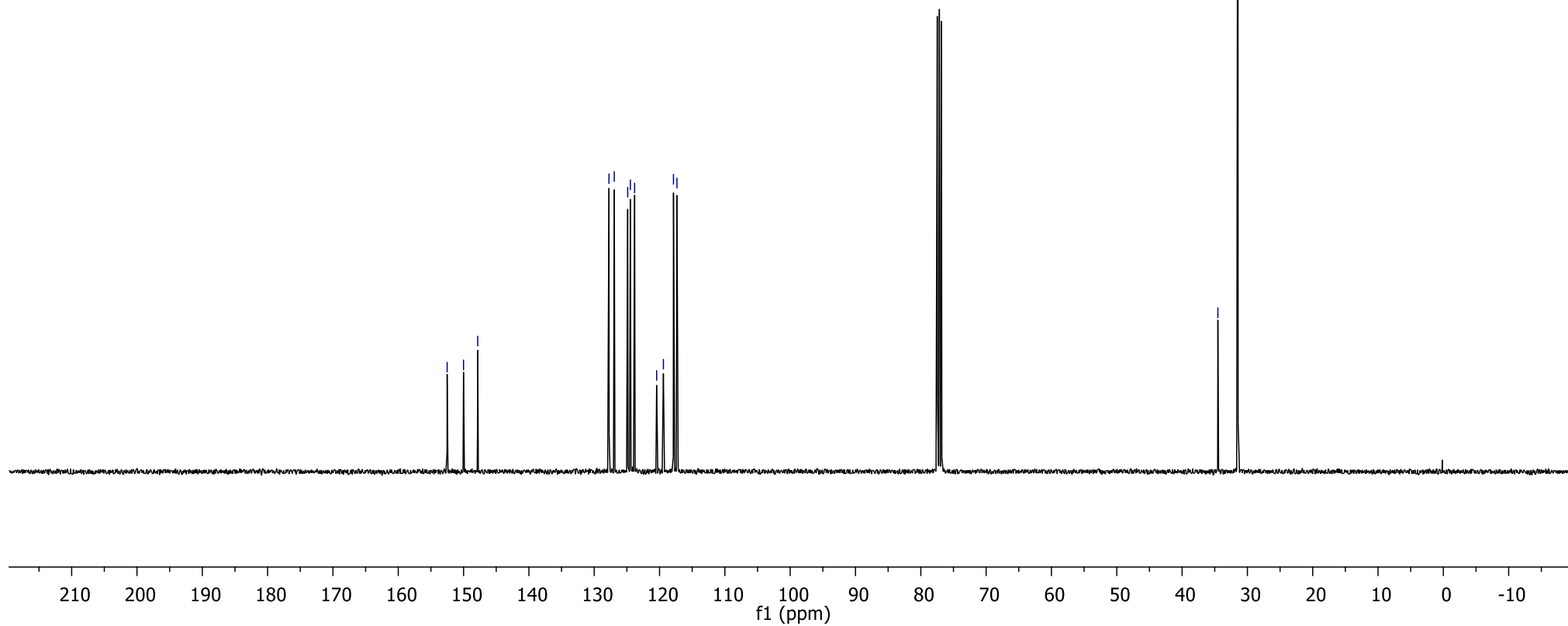
^{13}C NMR (101 MHz, CDCl_3)



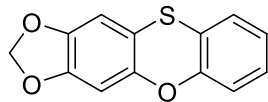
152.52
150.01
147.83

127.74
126.93
124.87
124.46
123.81
120.44
119.41
117.87
117.34

34.52
31.45

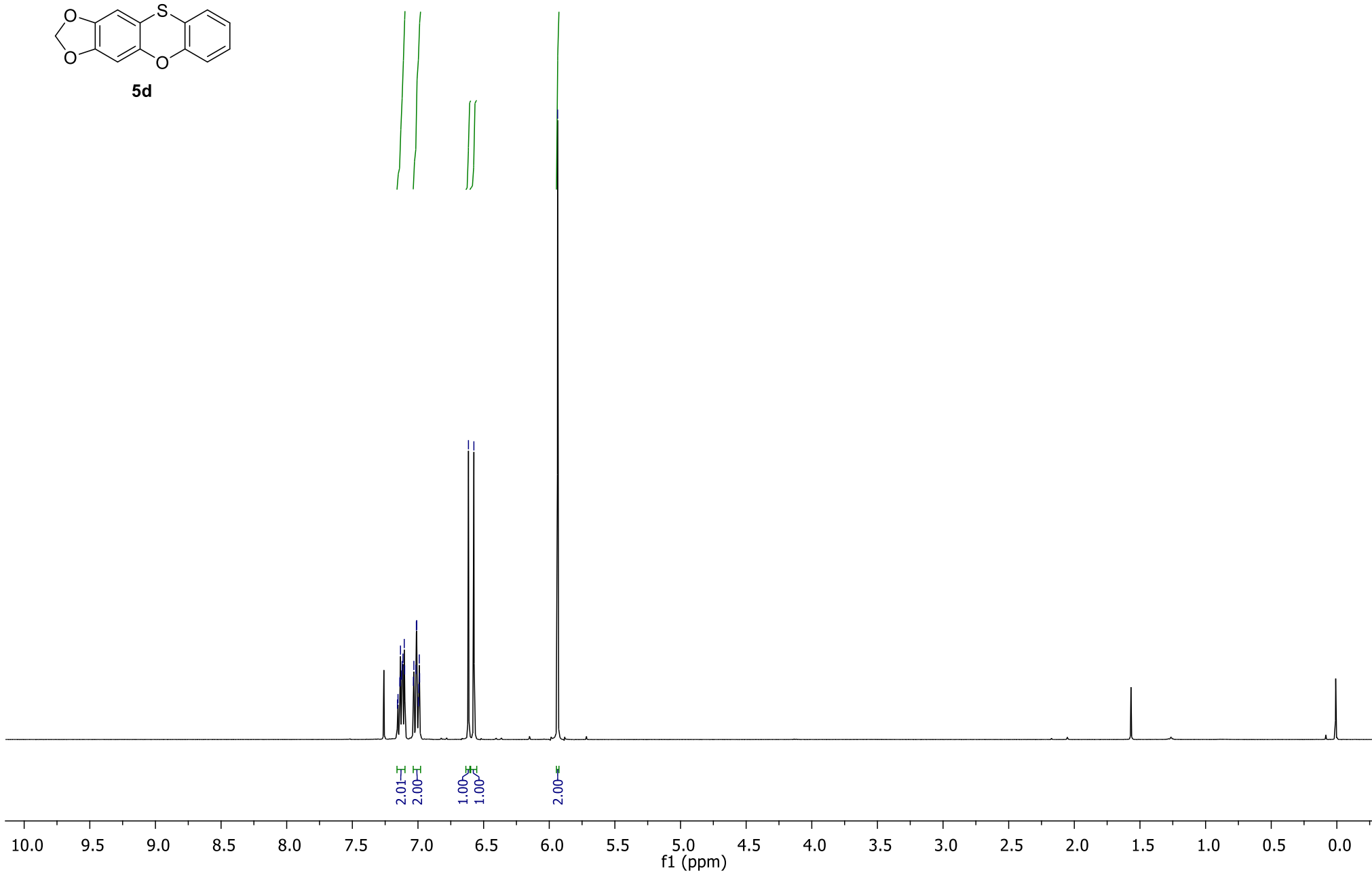


¹H NMR (400 MHz, CDCl₃)

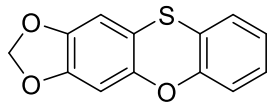


5d

7.16
7.15
7.14
7.14
7.13
7.12
7.12
7.11
7.10
7.10
7.03
7.03
7.02
7.01
7.01
7.01
7.00
6.99
6.99
6.62
6.57



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

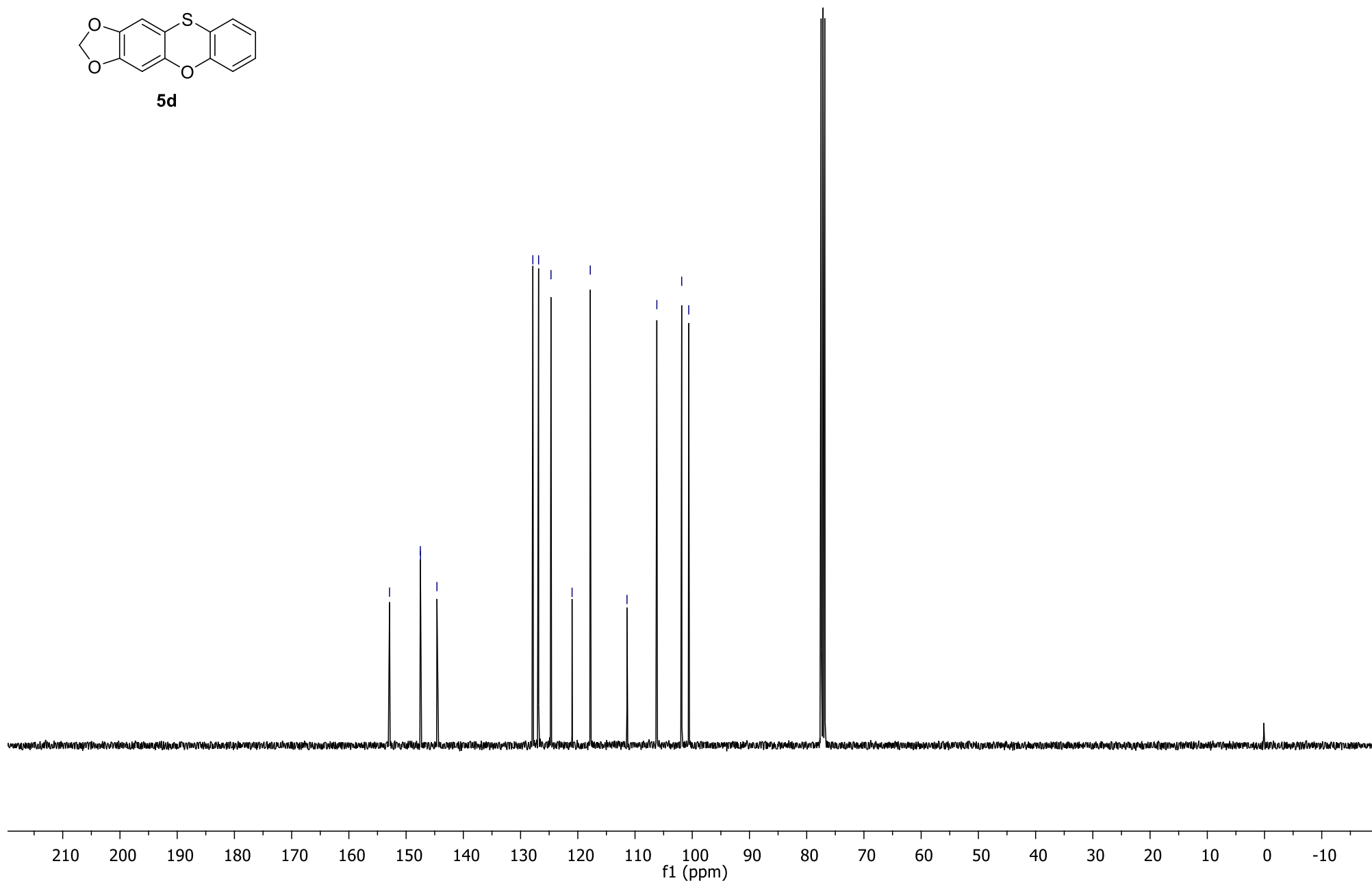


5d

152.89
147.50
147.49
144.61

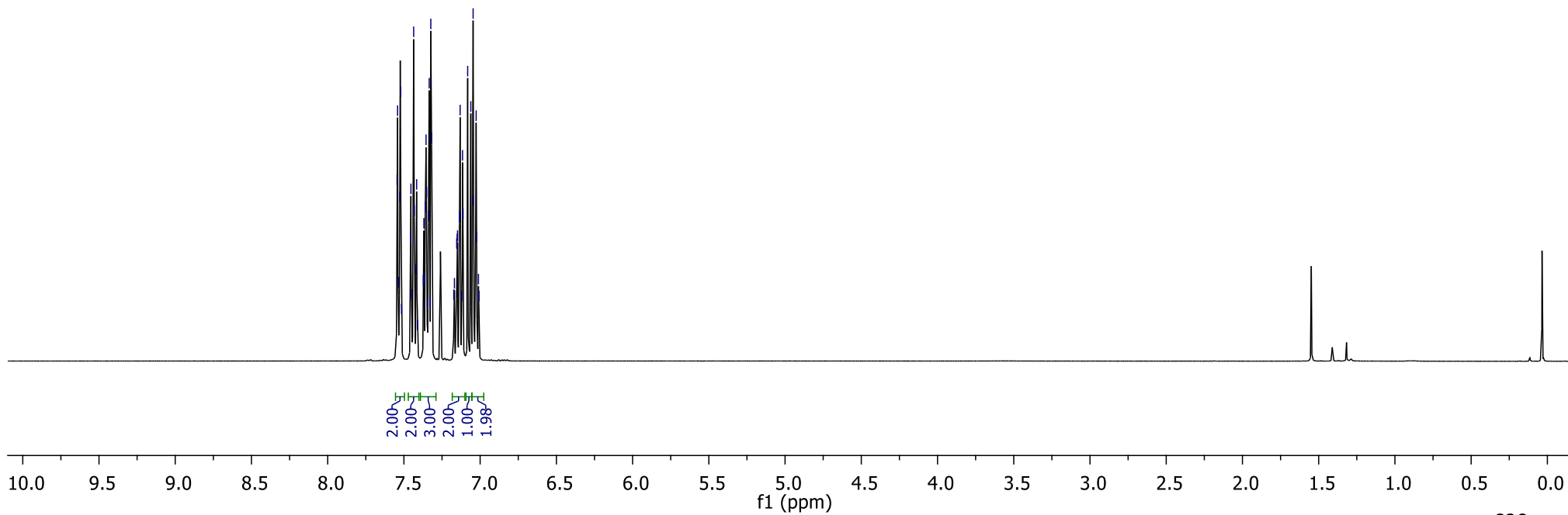
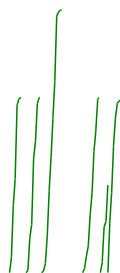
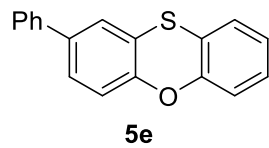
127.85
126.85
124.68
121.00
117.82

111.40
106.19
101.84
100.61

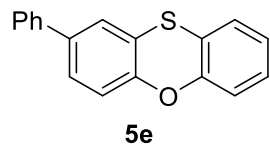


¹H NMR (400 MHz, CDCl₃)

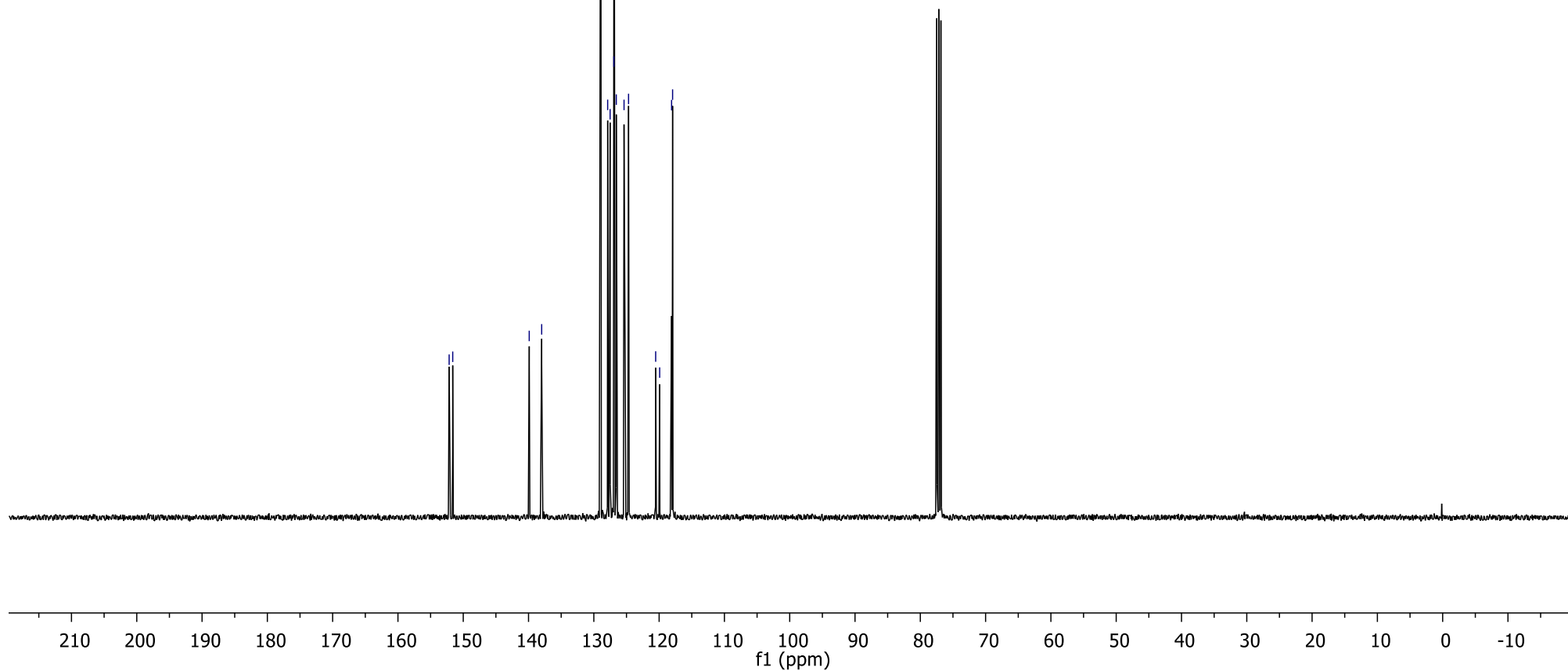
7.55
7.54
7.54
7.53
7.52
7.52
7.46
7.45
7.45
7.44
7.43
7.42
7.42
7.41
7.37
7.37
7.37
7.36
7.35
7.35
7.35
7.34
7.34
7.33
7.33
7.32
7.32
7.17
7.17
7.15
7.15
7.15
7.14
7.14
7.13
7.12
7.12
7.11
7.11
7.08
7.06
7.05
7.04
7.03
7.02
7.01
7.01



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

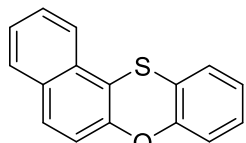


152.16
151.61
139.89
137.99
128.95
127.88
127.51
126.93
126.90
126.56
125.37
124.68
120.53
119.92
118.09
117.94

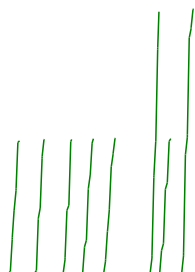


¹H NMR (400 MHz, CDCl₃)

7.93 7.93 7.91 7.91 7.80 7.78 7.66 7.64 7.57 7.57 7.55 7.55 7.55 7.53 7.53 7.46 7.46 7.44 7.44 7.44 7.44 7.42 7.42 7.42 7.23 7.22 7.22 7.20 7.20 7.20 7.19 7.19 7.18 7.17 7.16 7.16 7.15 7.15 7.14 7.14 7.07 7.06 7.06 7.05 7.05 7.04 7.04 7.03 7.03



5f

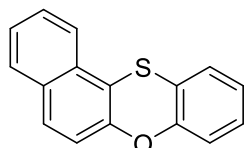


1.00 1.01 1.01 1.02 1.02 1.98 1.02 2.00

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

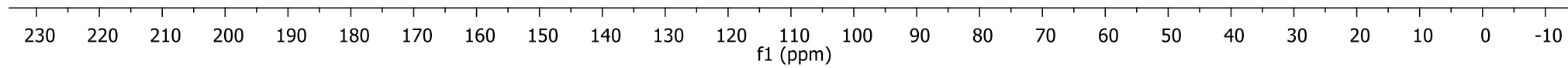
f1 (ppm)

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

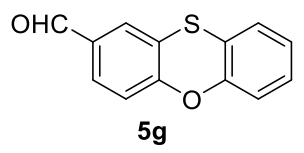


5f

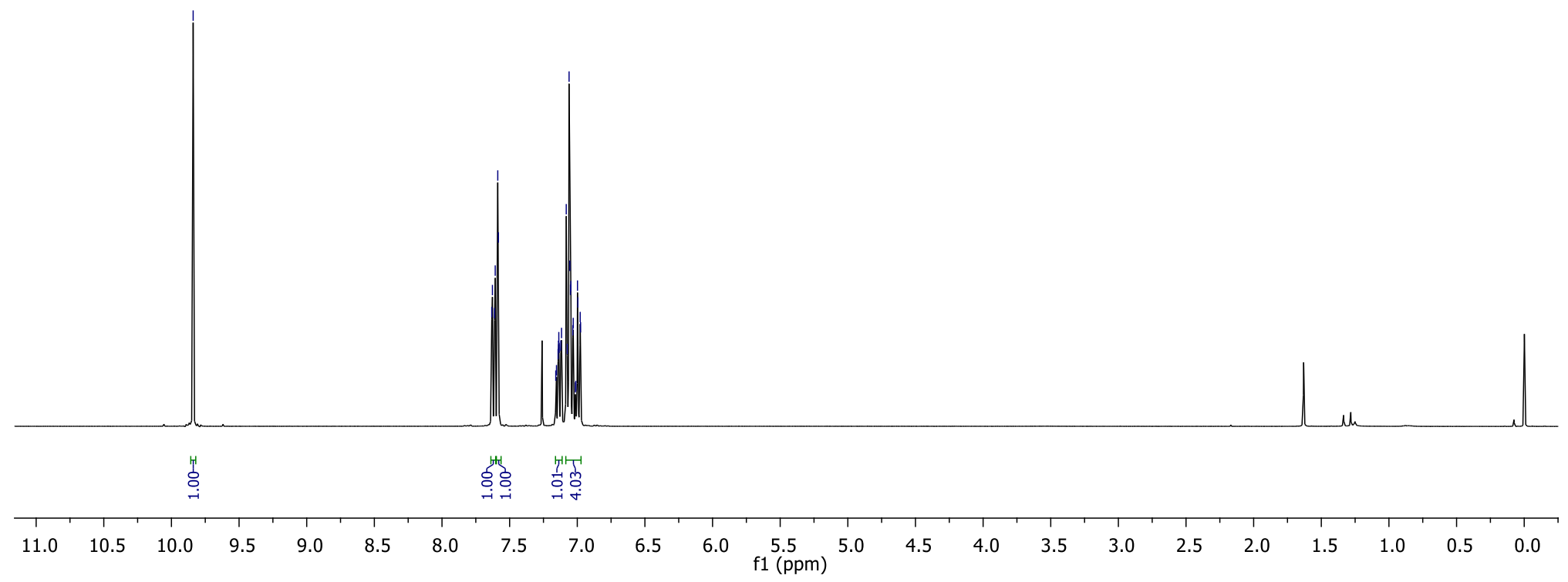
152.52
149.75
131.13
130.33
128.48
128.05
127.78
127.16
126.97
125.14
124.82
123.06
119.65
118.47
117.80
113.60
77.48
77.16
76.84



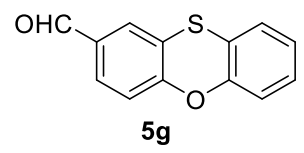
¹H NMR (400 MHz, CDCl₃)



7.63
7.63
7.61
7.61
7.59
7.58
7.16
7.15
7.14
7.14
7.14
7.13
7.12
7.12
7.08
7.07
7.06
7.05
7.05
7.03
7.03
7.01
7.00
7.00
6.98



¹³C NMR (101 MHz, CDCl₃)

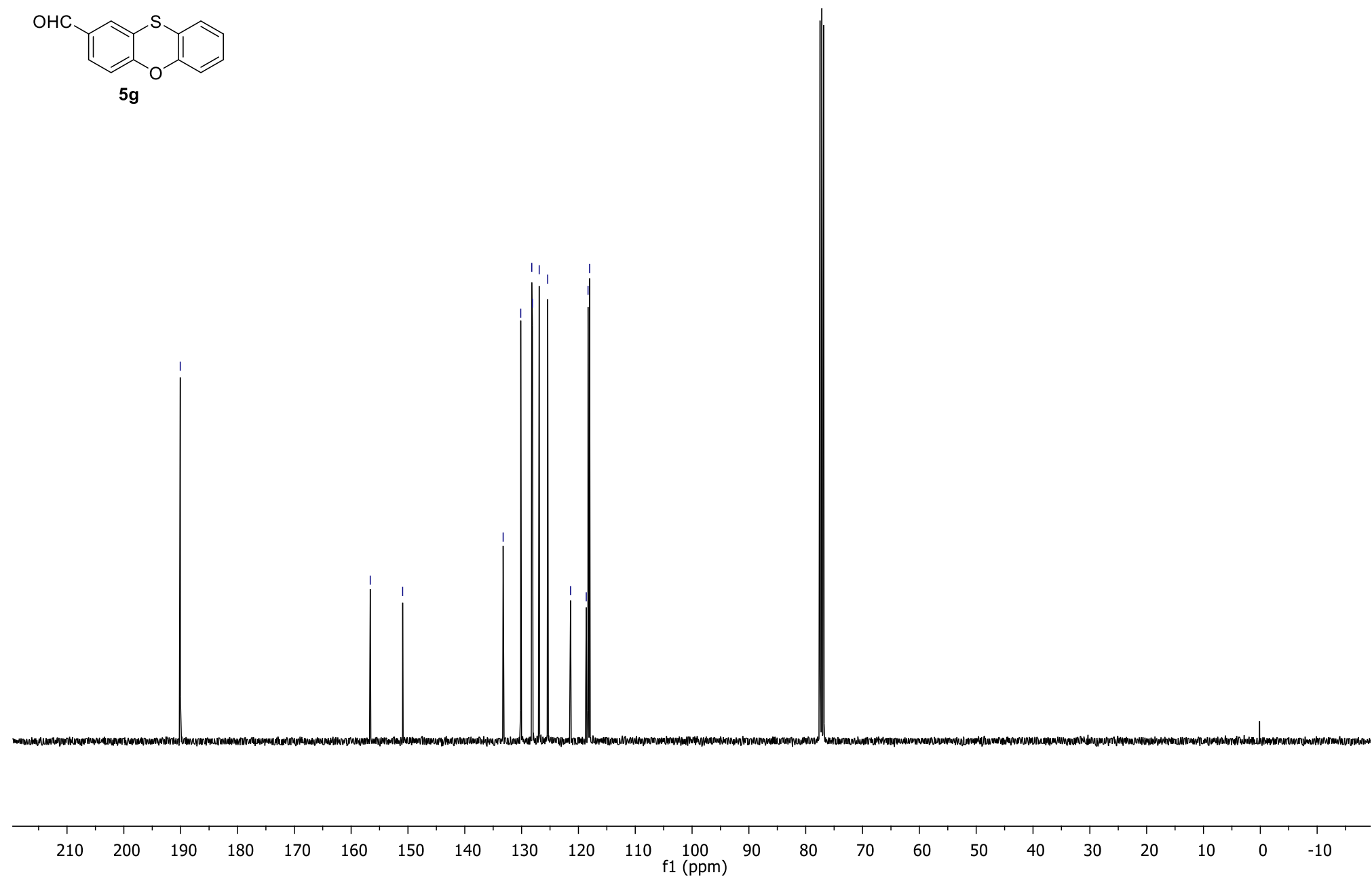


190.07

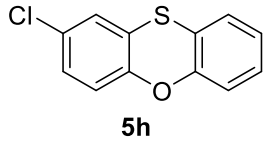
156.64

150.93

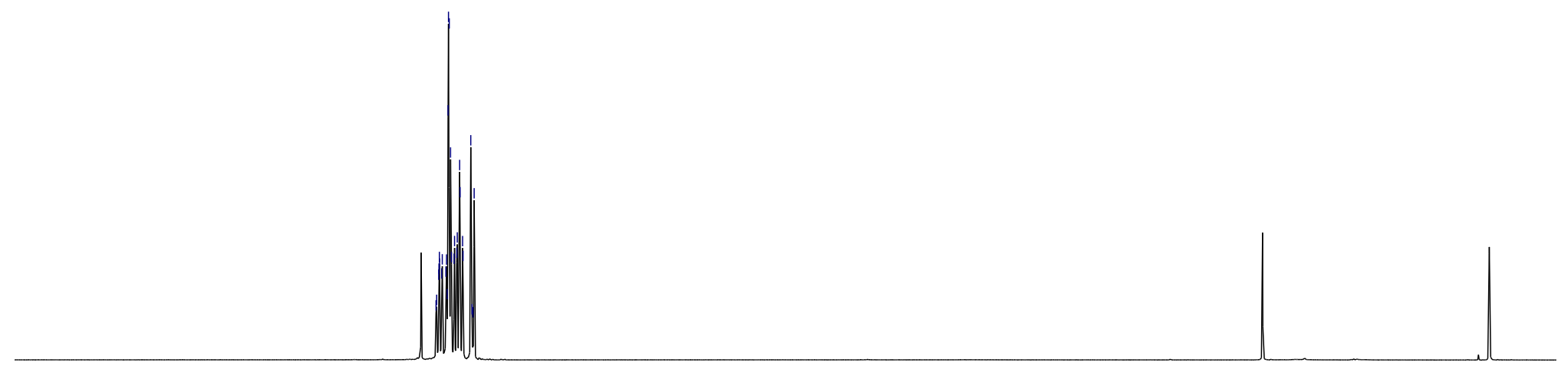
133.25
130.15
128.21
128.10
126.90
125.41
121.38
118.62
118.30
118.03



¹H NMR (400 MHz, CDCl₃)



7.16
7.15
7.14
7.14
7.14
7.13
7.12
7.12
7.09
7.09
7.08
7.08
7.07
7.07
7.06
7.05
7.04
7.03
7.02
7.01
7.00
7.00
6.98
6.98
6.92
6.91
6.91
6.90

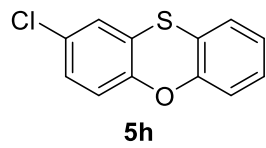


1.03
5.00
1.00

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

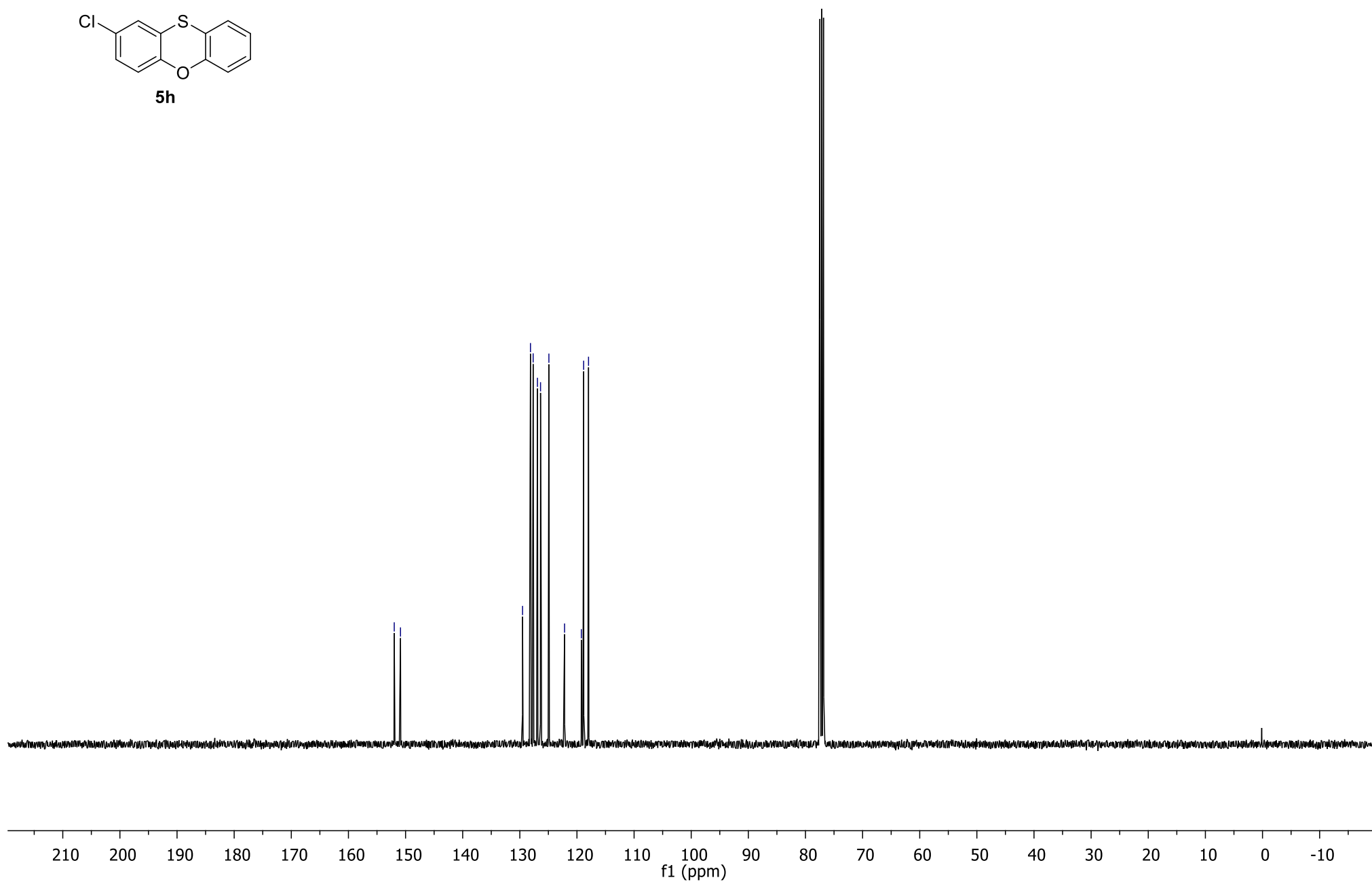
f1 (ppm)

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



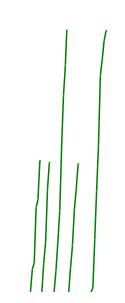
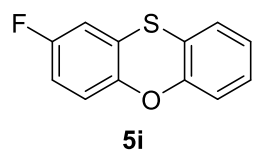
151.98
150.89

129.52
128.13
127.67
126.91
126.38
124.91
122.17
119.23
118.83
117.97



¹H NMR (400 MHz, CDCl₃)

7.17
7.16
7.15
7.15
7.14
7.14
7.13
7.13
7.11
7.10
7.09
7.08
7.04
7.04
7.02
7.02
7.01
7.01
7.00
7.00
6.99
6.99
6.97
6.97
6.96
6.95
6.95
6.95
6.94
6.93
6.85
6.84
6.84
6.84
6.83
6.83
6.82
6.82
6.81
6.80
6.79

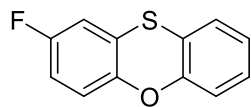


1.01
0.99
2.00
0.98
1.99

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

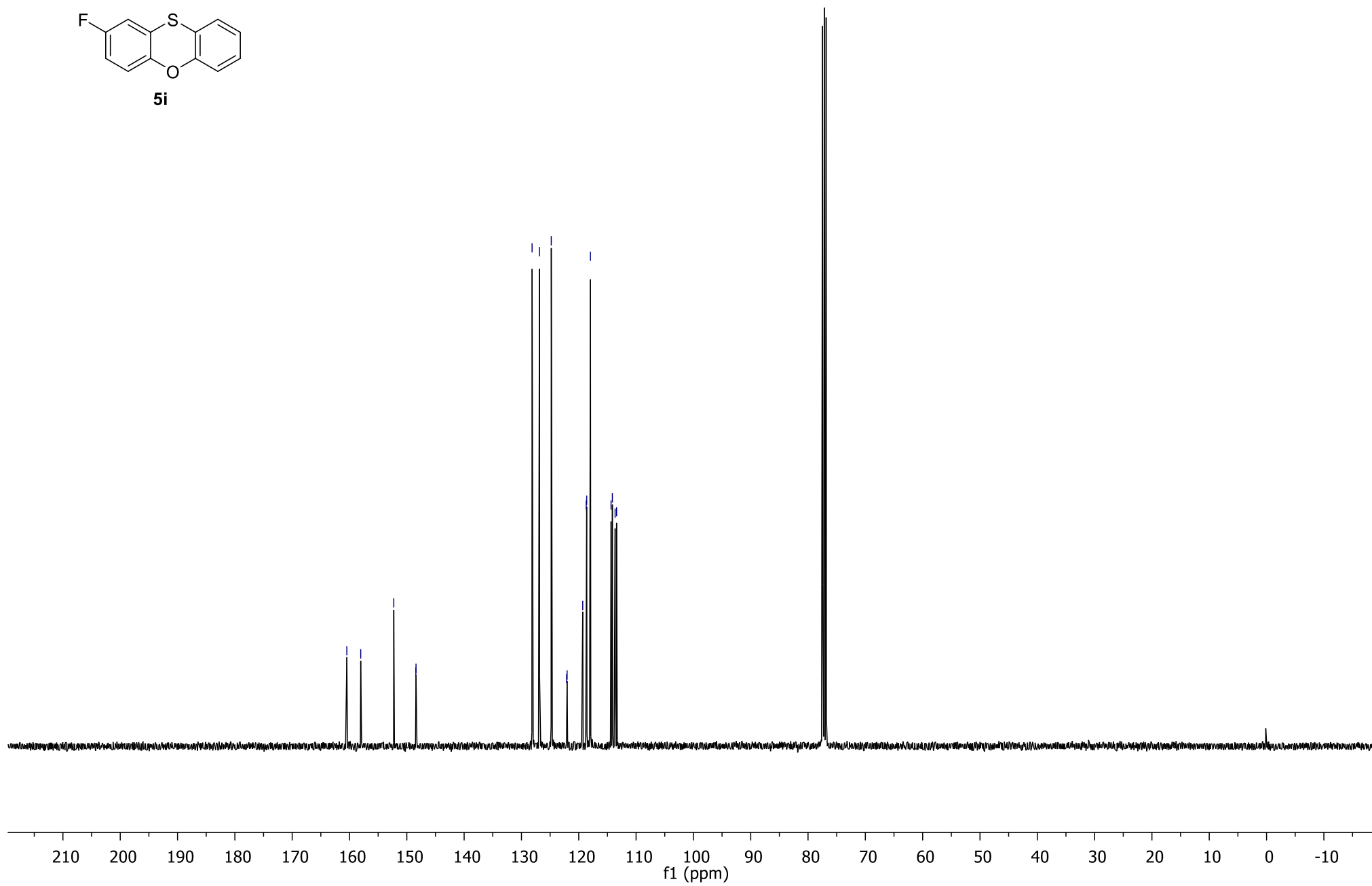
f1 (ppm)

¹³C NMR (101 MHz, CDCl₃)

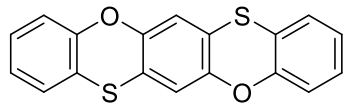


5i

160.47
158.04
152.27
148.42
148.40
128.14
126.85
124.80
122.12
122.03
119.31
118.70
118.61
117.96
114.37
114.13
113.66
113.40

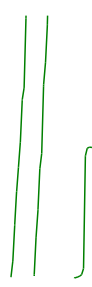


¹H NMR (400 MHz, CDCl₃)

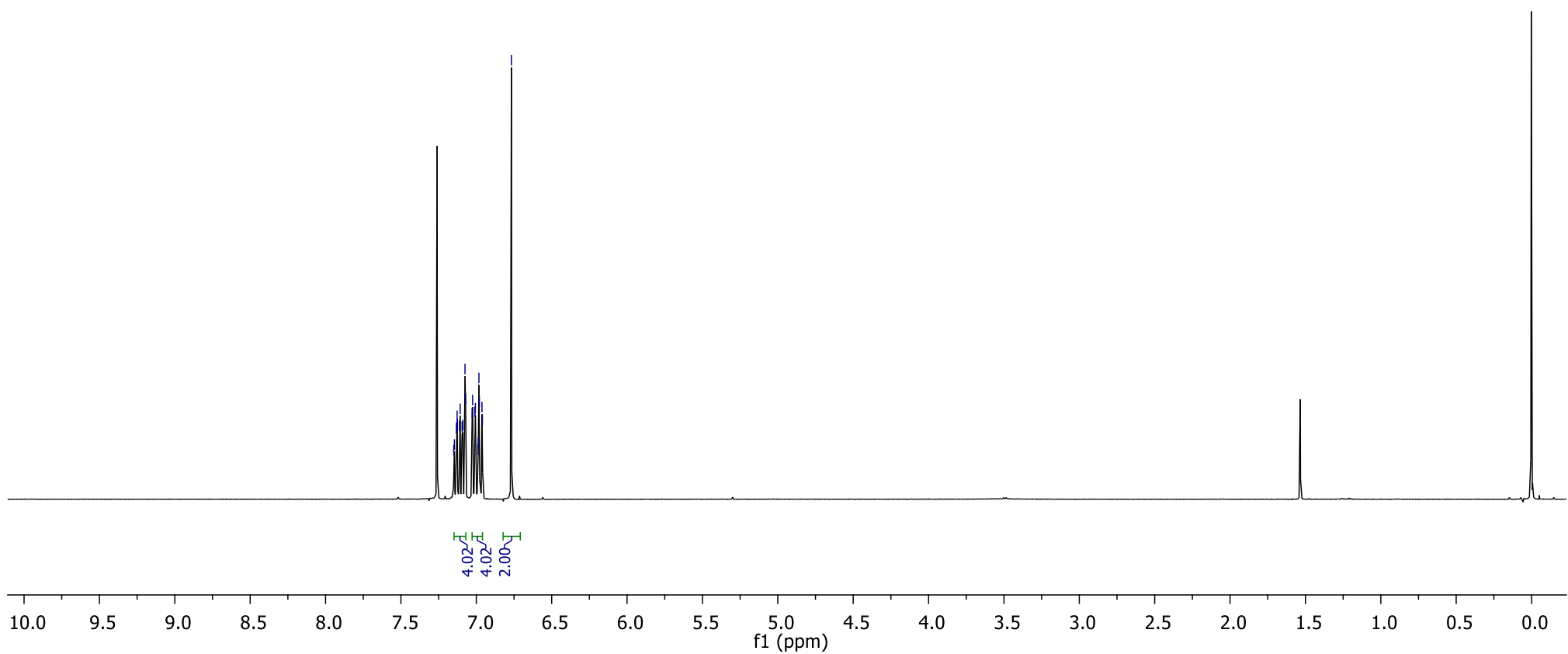


5j

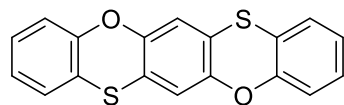
7.15
7.15
7.13
7.13
7.13
7.11
7.11
7.09
7.07
7.07
7.03
7.02
7.01
7.01
6.99
6.99
6.98
6.98
6.96
6.96
6.77



4.02
4.02
2.00



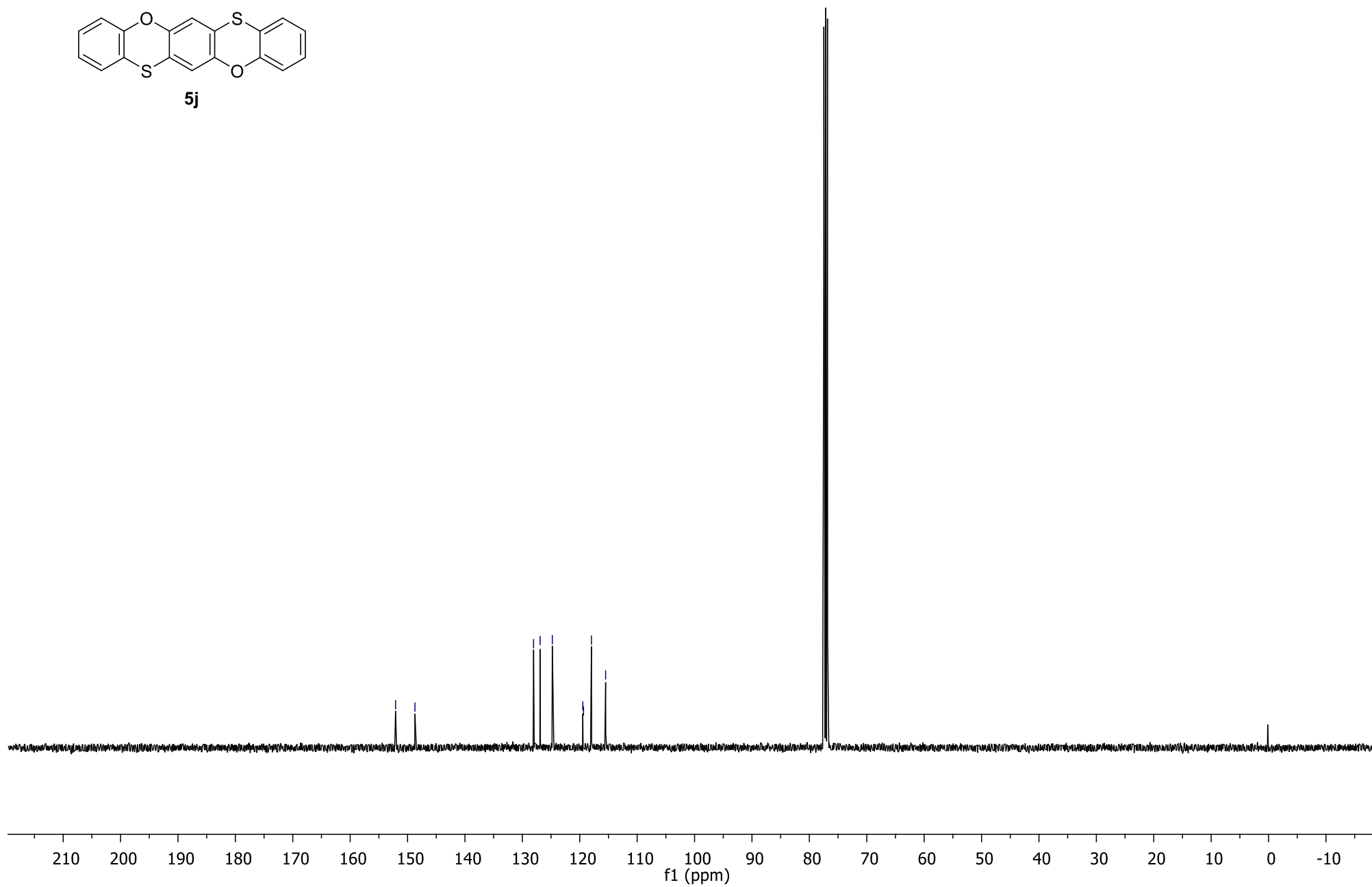
^{13}C NMR (101 MHz, CDCl_3)



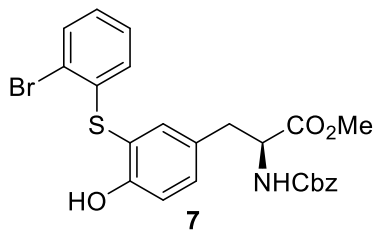
5j

— 152.09
— 148.71

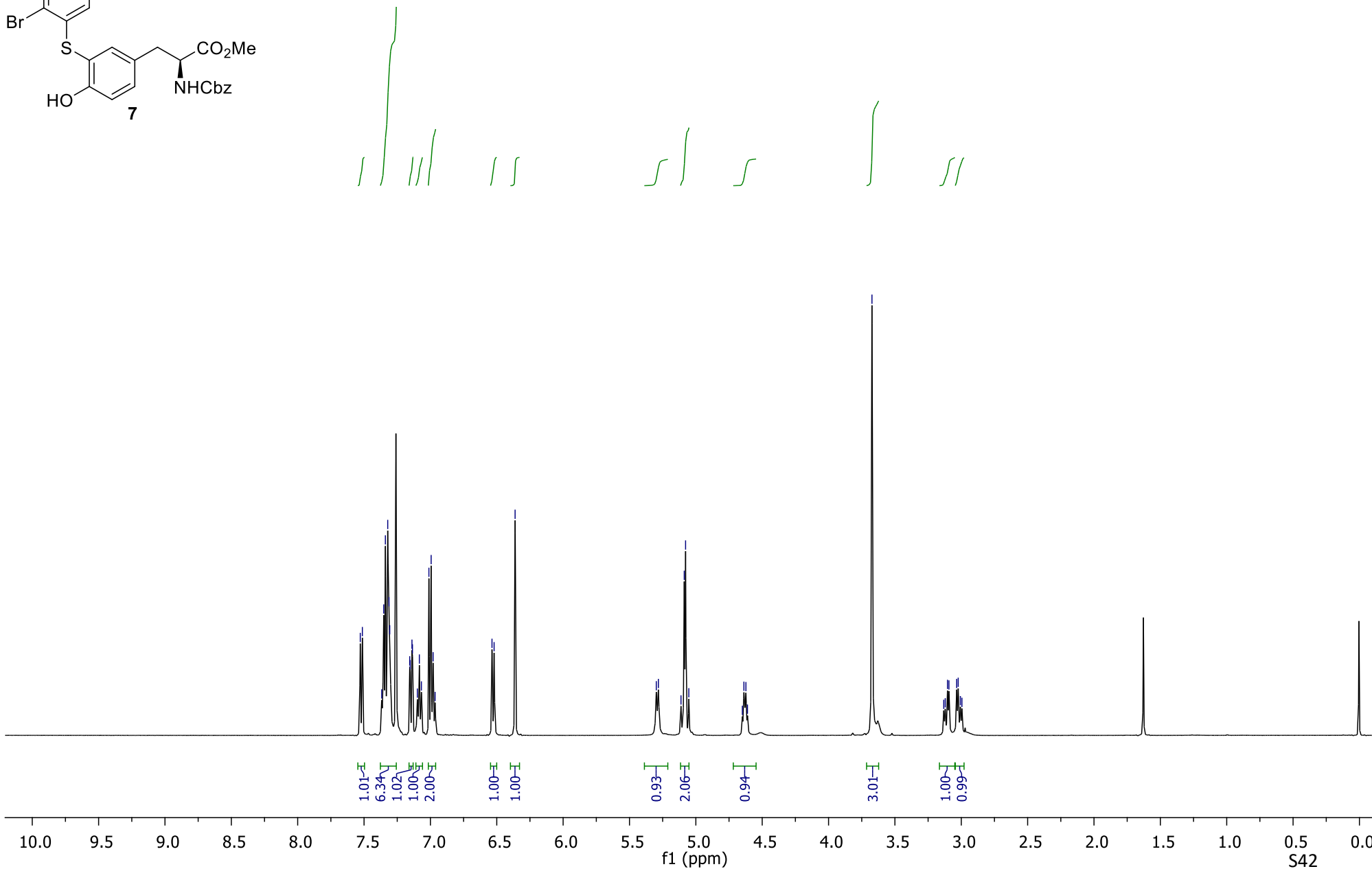
— 128.05
— 126.90
— 124.78
— 119.47
— 119.34
— 117.96
— 115.49



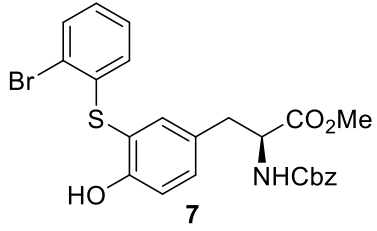
¹H NMR (400 MHz, CDCl₃)



7.53
7.51
7.37
7.35
7.34
7.32
7.31
7.31
7.16
7.14
7.14
7.10
7.08
7.07
7.01
6.99
6.96
6.54
6.52
6.36
5.30
5.28
5.11
5.09
5.08
5.05
4.65
4.64
4.62
4.61
3.67
3.13
3.12
3.10
3.09
3.03
3.02
3.01
3.00



¹³C NMR (101 MHz, CDCl₃)



— 171.84

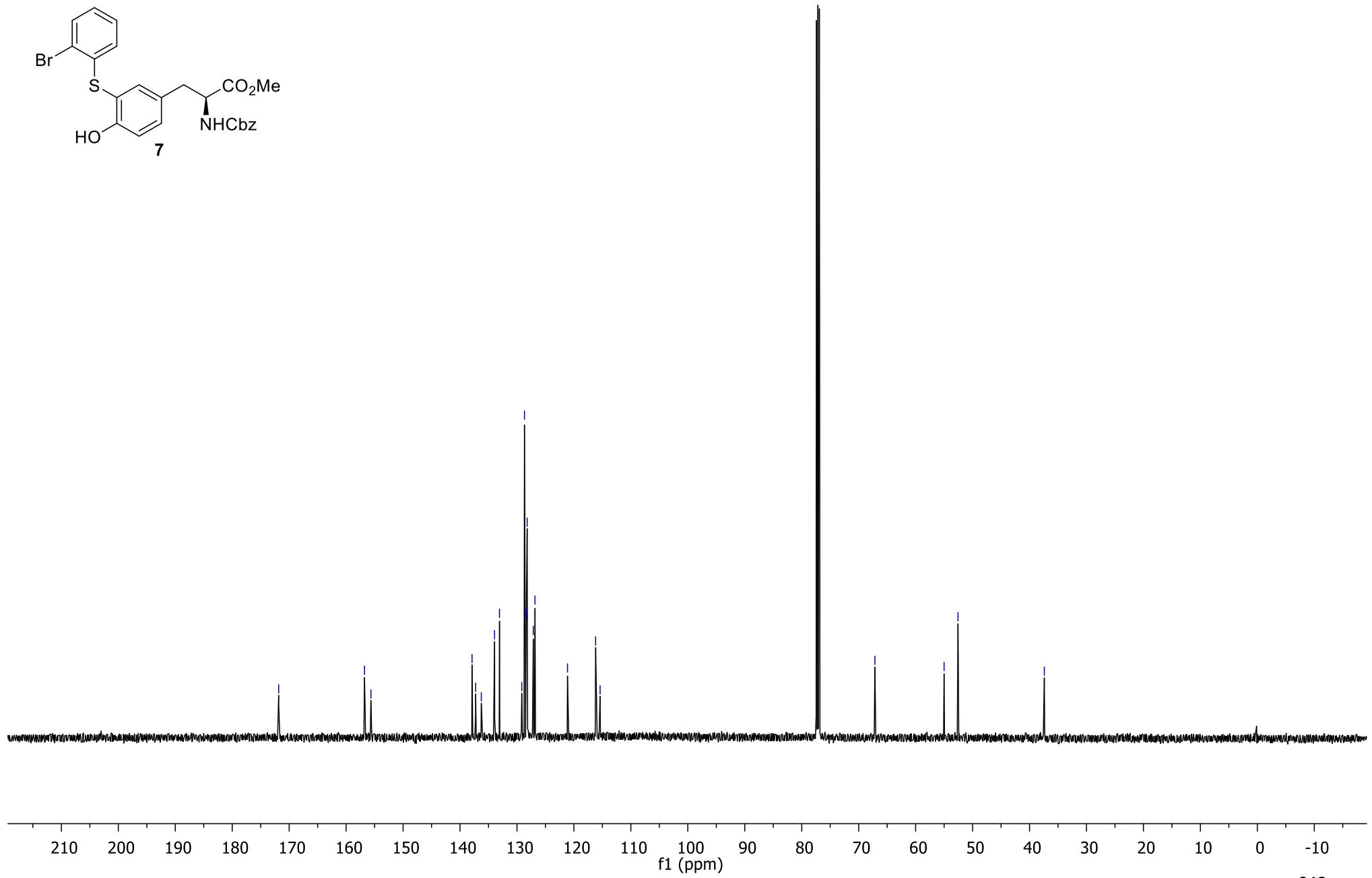
— 156.77
— 155.64

— 137.89
— 137.25
— 136.27
— 133.93
— 133.07
— 129.16
— 128.68
— 128.37
— 128.24
— 128.21
— 127.14
— 126.84
— 121.13
— 116.19
— 115.40

— 67.16

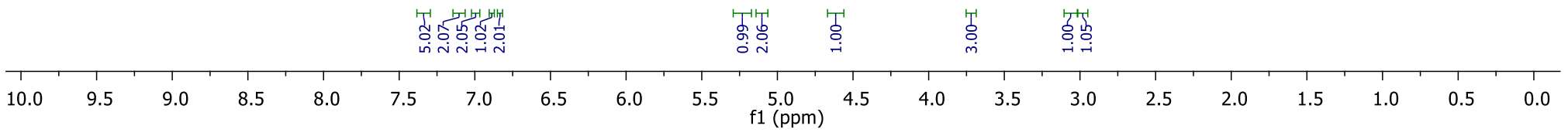
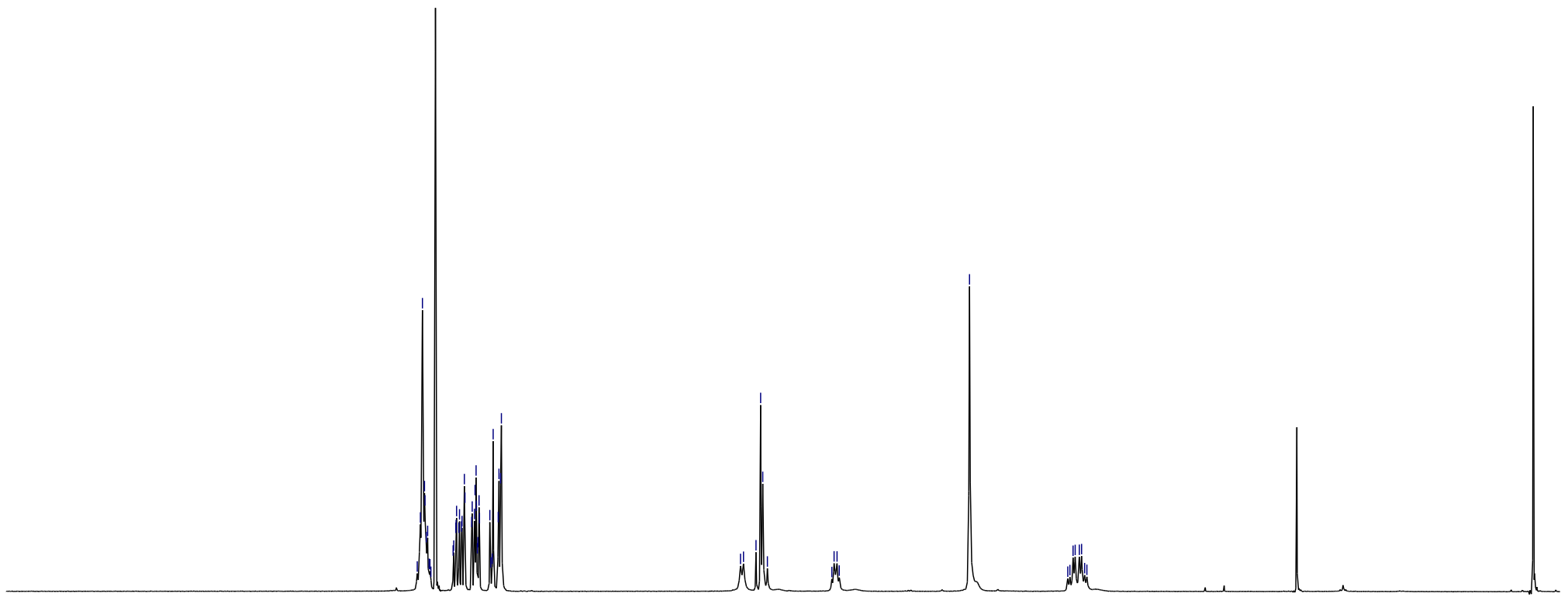
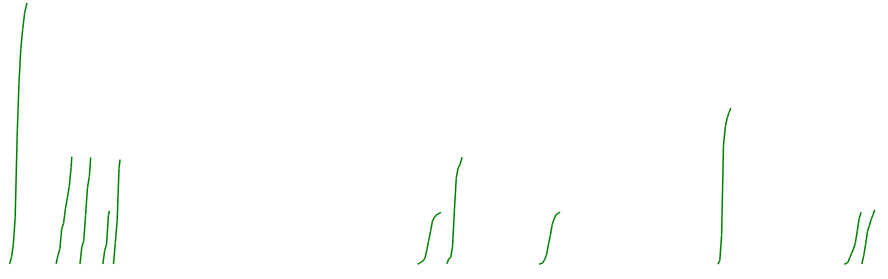
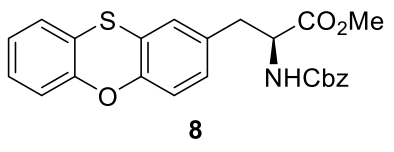
— 55.00
— 52.56

— 37.40

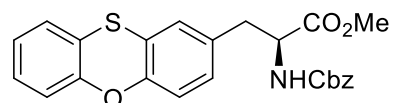


¹H NMR (400 MHz, CDCl₃)

7.36, 7.35, 7.33, 7.33, 7.32, 7.31, 7.30, 7.14, 7.14, 7.13, 7.12, 7.12, 7.11, 7.10, 7.09, 7.08, 7.07, 7.07, 7.02, 7.02, 7.00, 7.00, 6.99, 6.99, 6.98, 6.98, 6.97, 6.97, 6.90, 6.89, 6.89, 6.88, 6.88, 6.84, 6.83, 6.82, 6.82, 5.24, 5.22, 5.14, 5.11, 5.10, 5.07, 5.07, 4.64, 4.63, 4.61, 4.59, 3.73, 3.08, 3.07, 3.05, 3.03, 3.00, 2.99, 2.97, 2.95



¹³C NMR (101 MHz, CDCl₃)



8

171.87

155.72

152.18

151.38

136.34

132.29

128.70

128.65

128.36

128.23

127.90

127.49

126.92

124.67

120.48

119.91

117.95

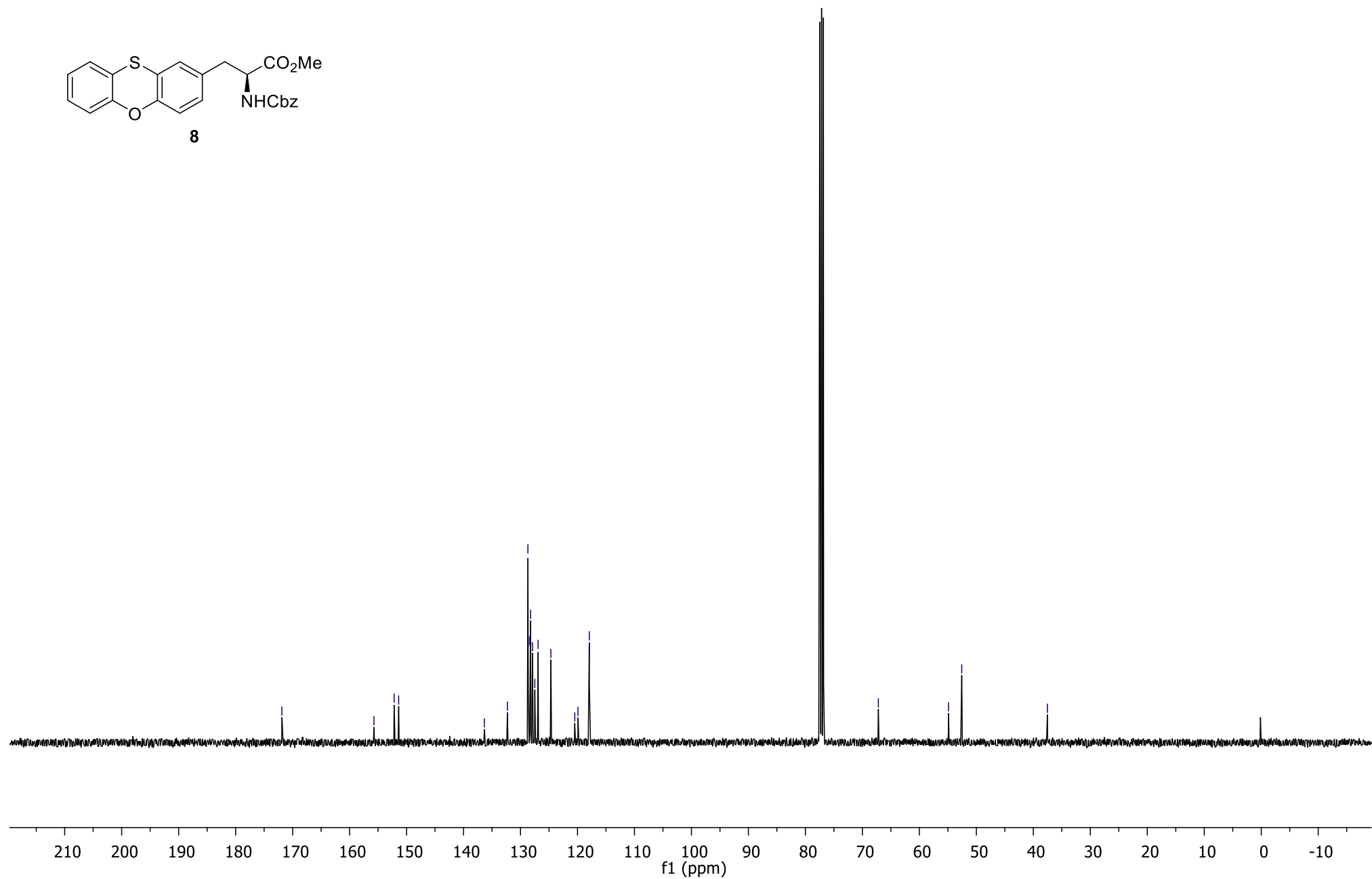
117.92

67.20

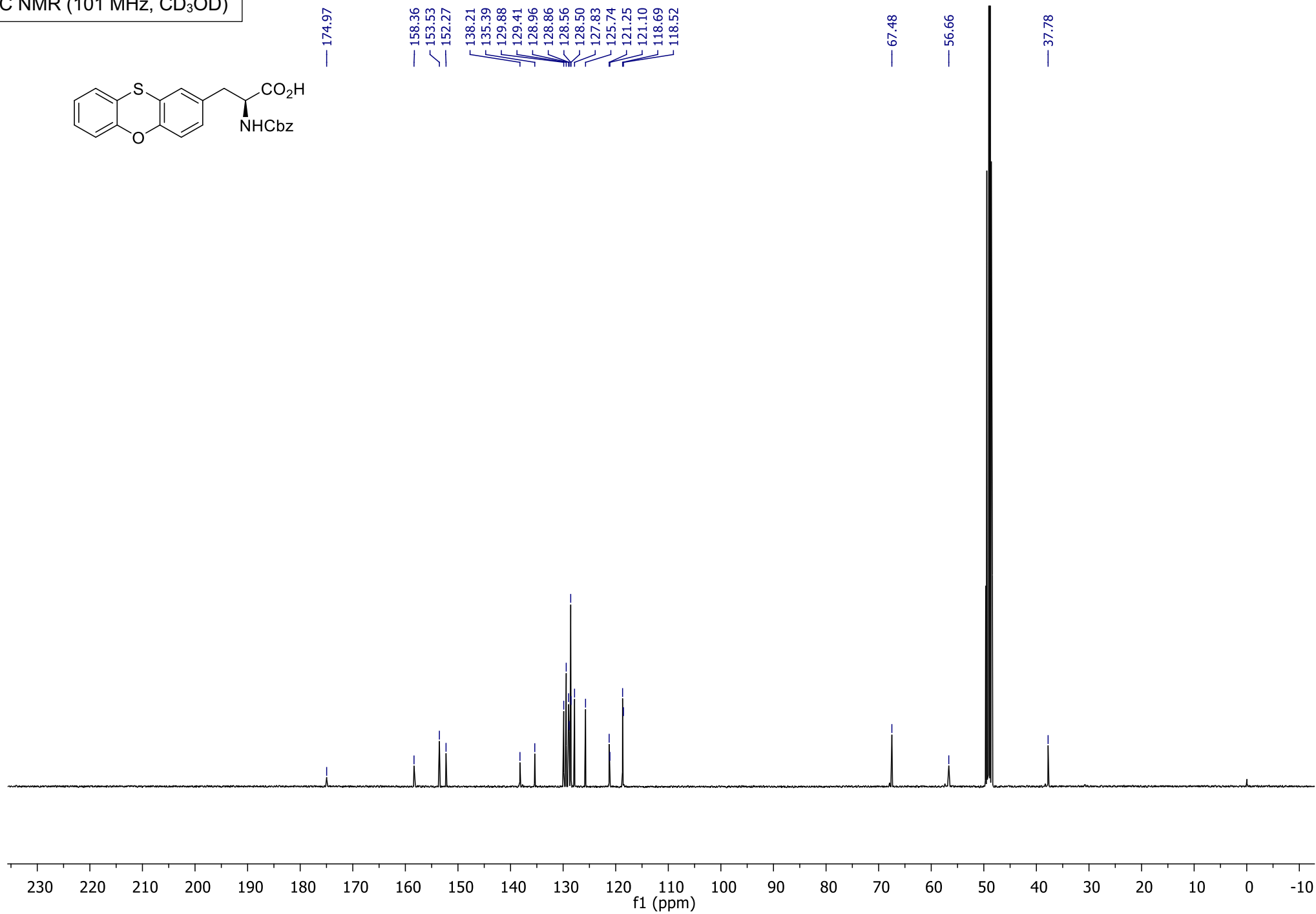
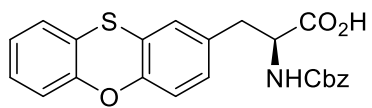
54.88

52.56

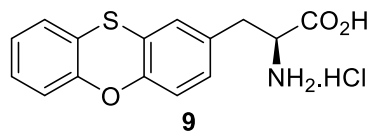
37.53



¹³C NMR (101 MHz, CD₃OD)



¹H NMR (400 MHz, CD₃OD)

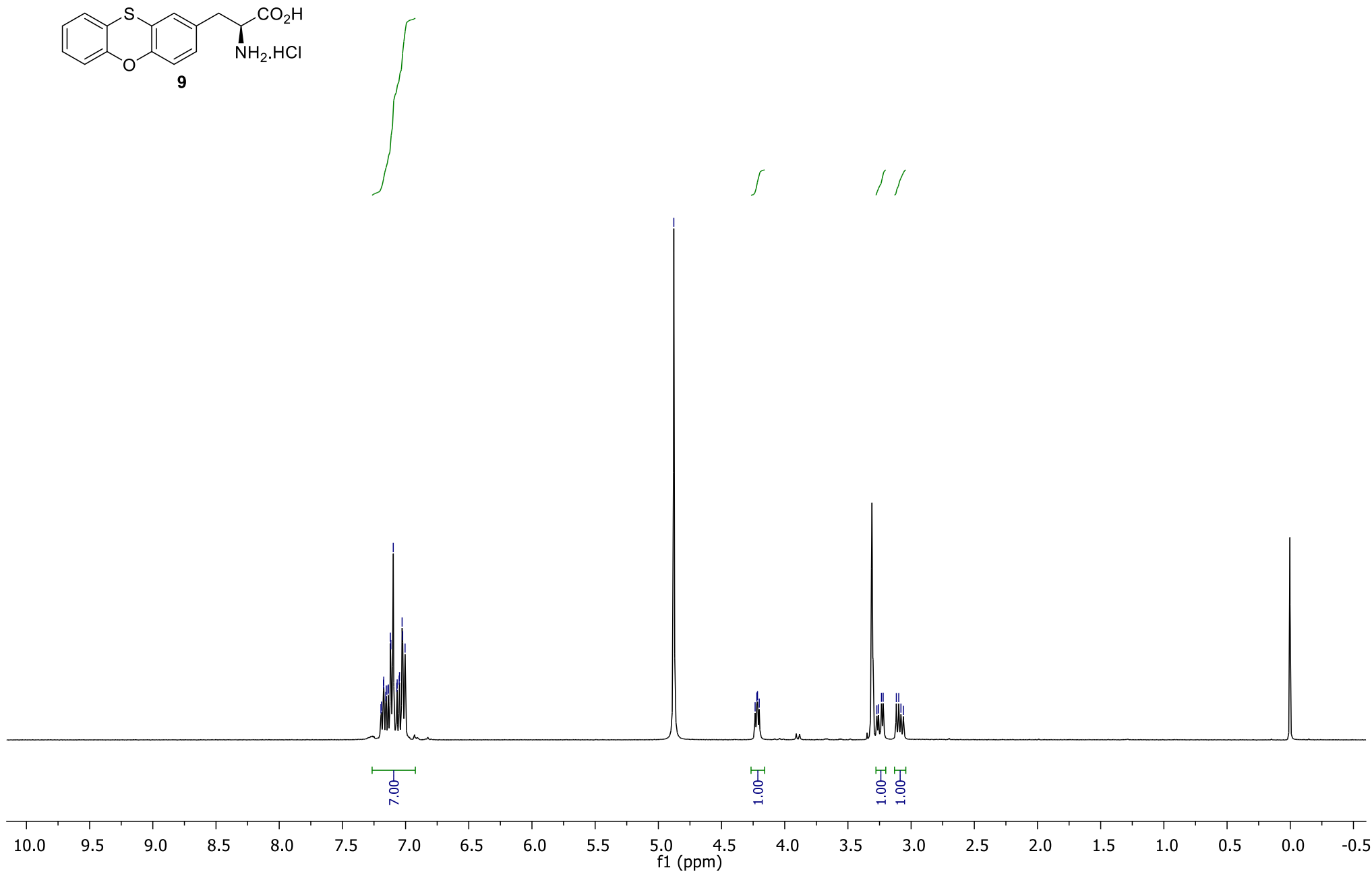


7.19
7.19
7.17
7.17
7.16
7.15
7.14
7.14
7.12
7.12
7.10
7.07
7.07
7.05
7.03
7.02
7.00

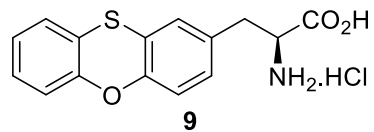
4.88

4.23
4.22
4.22
4.20

3.27
3.26
3.23
3.22
3.12
3.10
3.08
3.06



^{13}C NMR (101 MHz, CD_3OD)



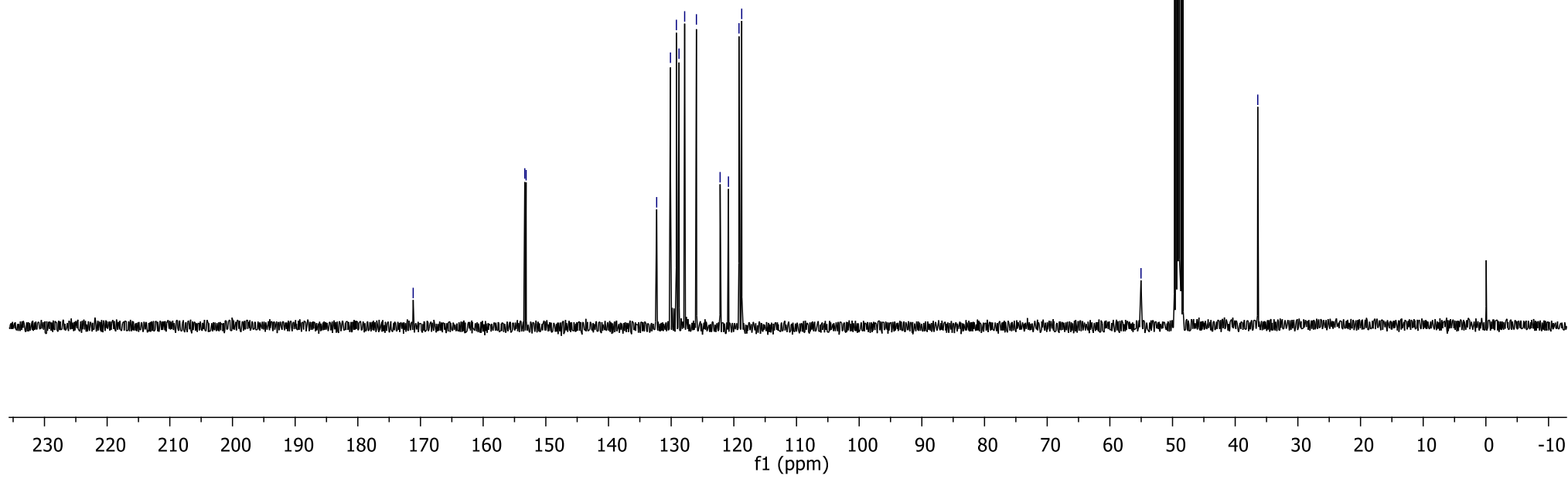
171.17

153.36
153.15

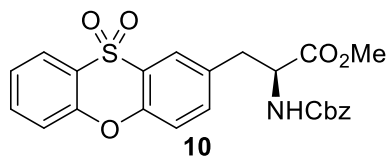
132.32
130.12
129.17
128.75
127.85
125.97
122.19
120.86
119.17
118.74

55.03

36.40



¹H NMR (400 MHz, CDCl₃)



8.05
8.04
8.03
7.79
7.79
7.65
7.65
7.64
7.62
7.62
7.41
7.39
7.38
7.36
7.34
7.34
7.31
7.31
7.30
7.29
7.27

5.43
5.42

5.10

4.69
4.68
4.67
4.66

3.75

3.28

3.27

3.25

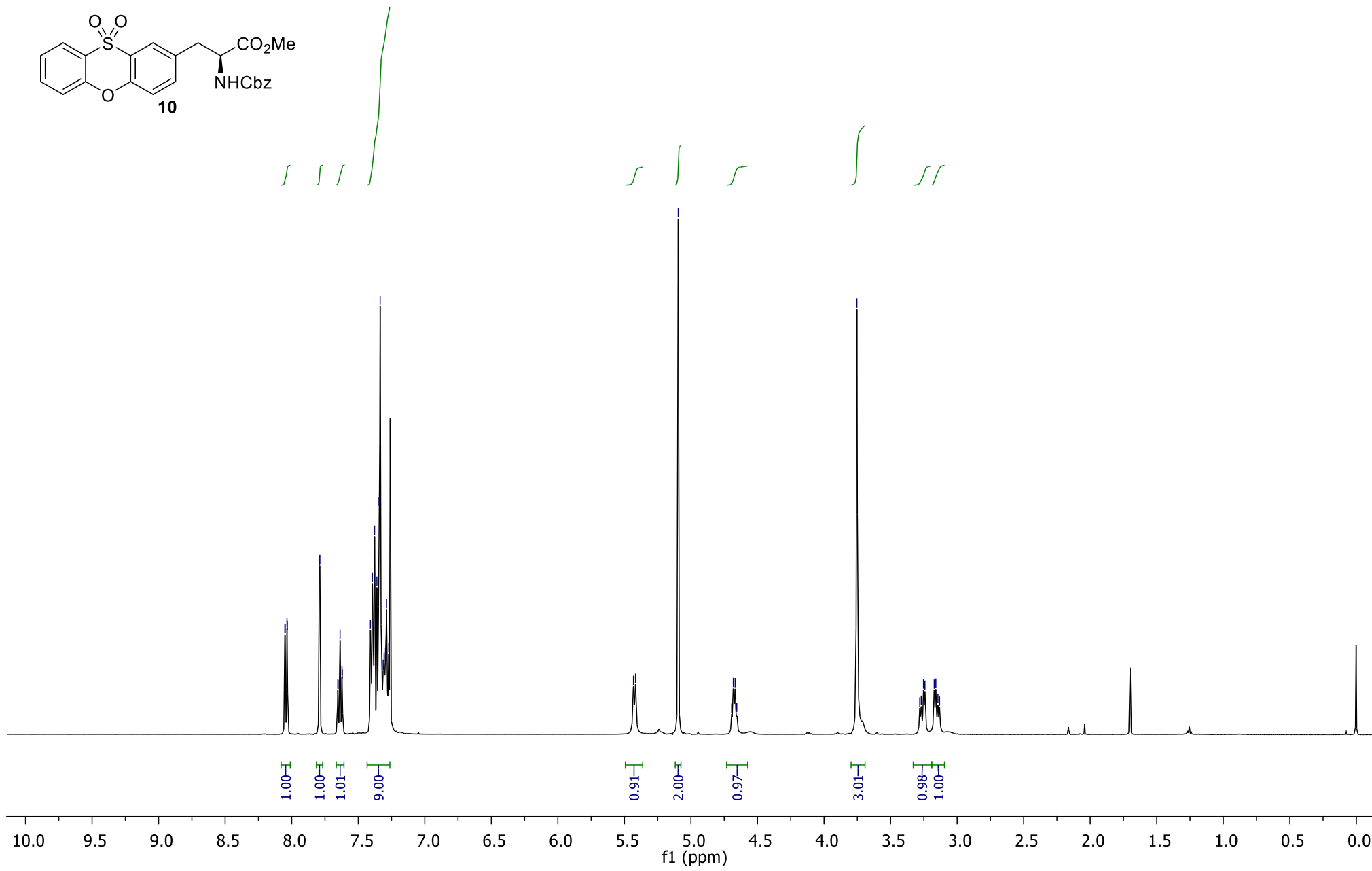
3.24

3.17

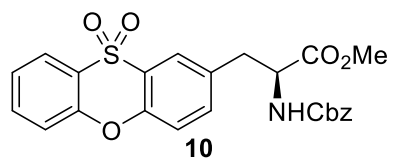
3.16

3.14

3.13



¹³C NMR (101 MHz, CDCl₃)



171.44

155.67

151.57

150.64

136.20

135.20

134.26

133.40

128.64

128.32

128.24

124.98

124.93

124.92

123.76

123.49

119.23

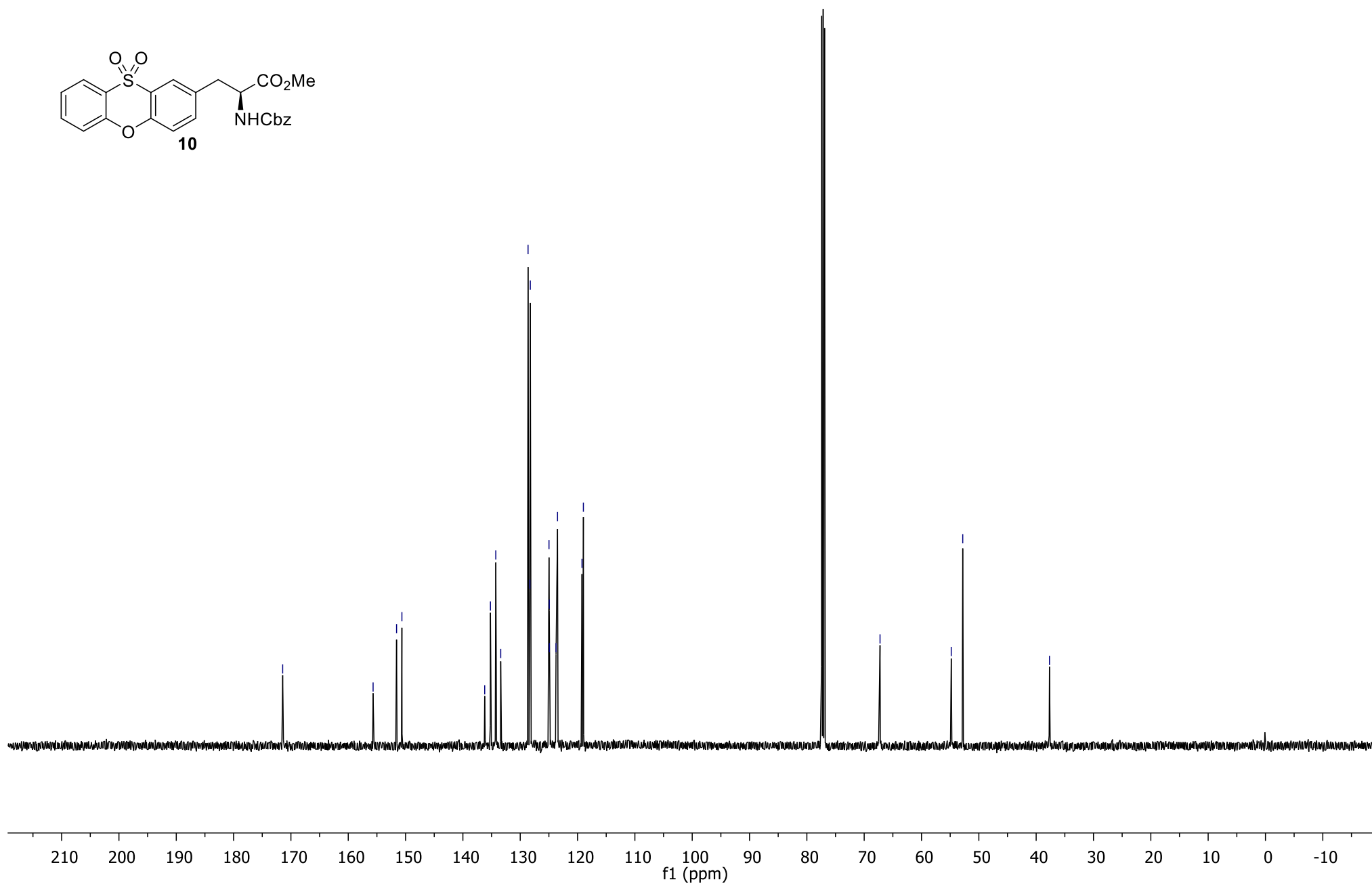
118.98

67.24

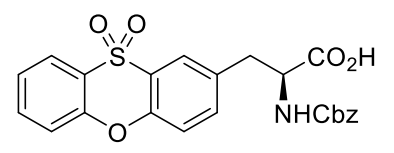
54.81

52.81

37.67



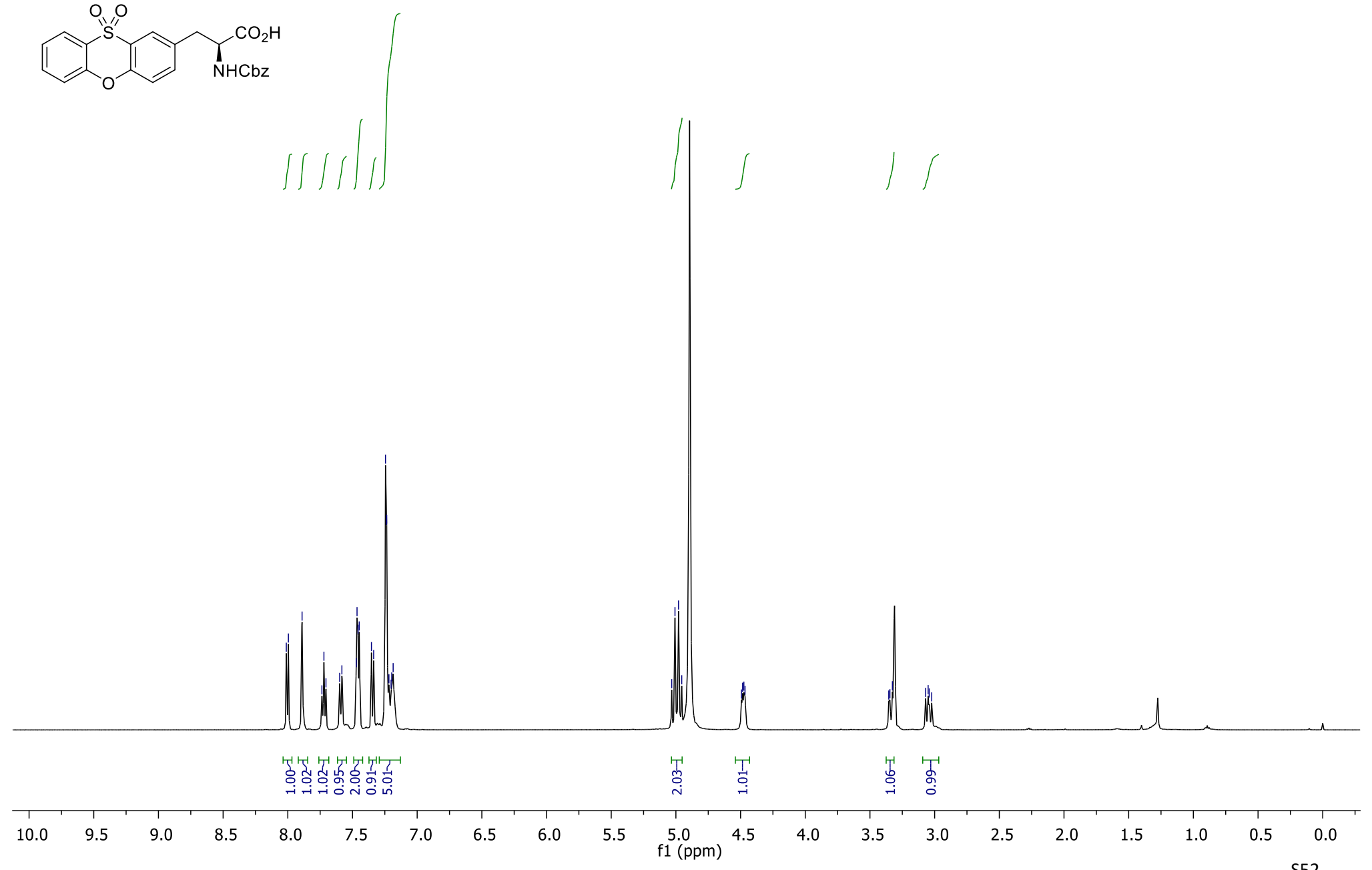
¹H NMR (500 MHz, CD₃OD)



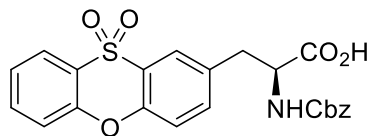
8.01
8.00
7.89
7.74
7.72
7.71
7.60
7.58
7.47
7.46
7.46
7.45
7.35
7.34
7.24
7.24
7.24
7.22
7.20
7.20
7.19

5.03
5.01
4.98
4.95
4.49
4.48
4.47
4.47

3.35
3.35
3.33
3.07
3.05
3.04
3.02



^{13}C NMR (126 MHz, CD_3OD)



174.59

158.36

152.98

151.77

138.08

136.73

136.50

135.72

129.36

128.84

128.69

126.28

126.22

125.90

124.44

124.09

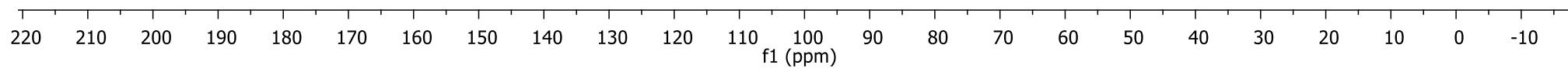
120.11

120.10

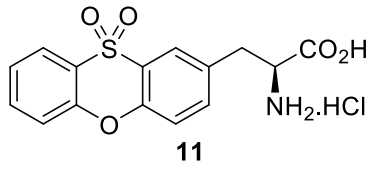
67.60

56.52

37.82



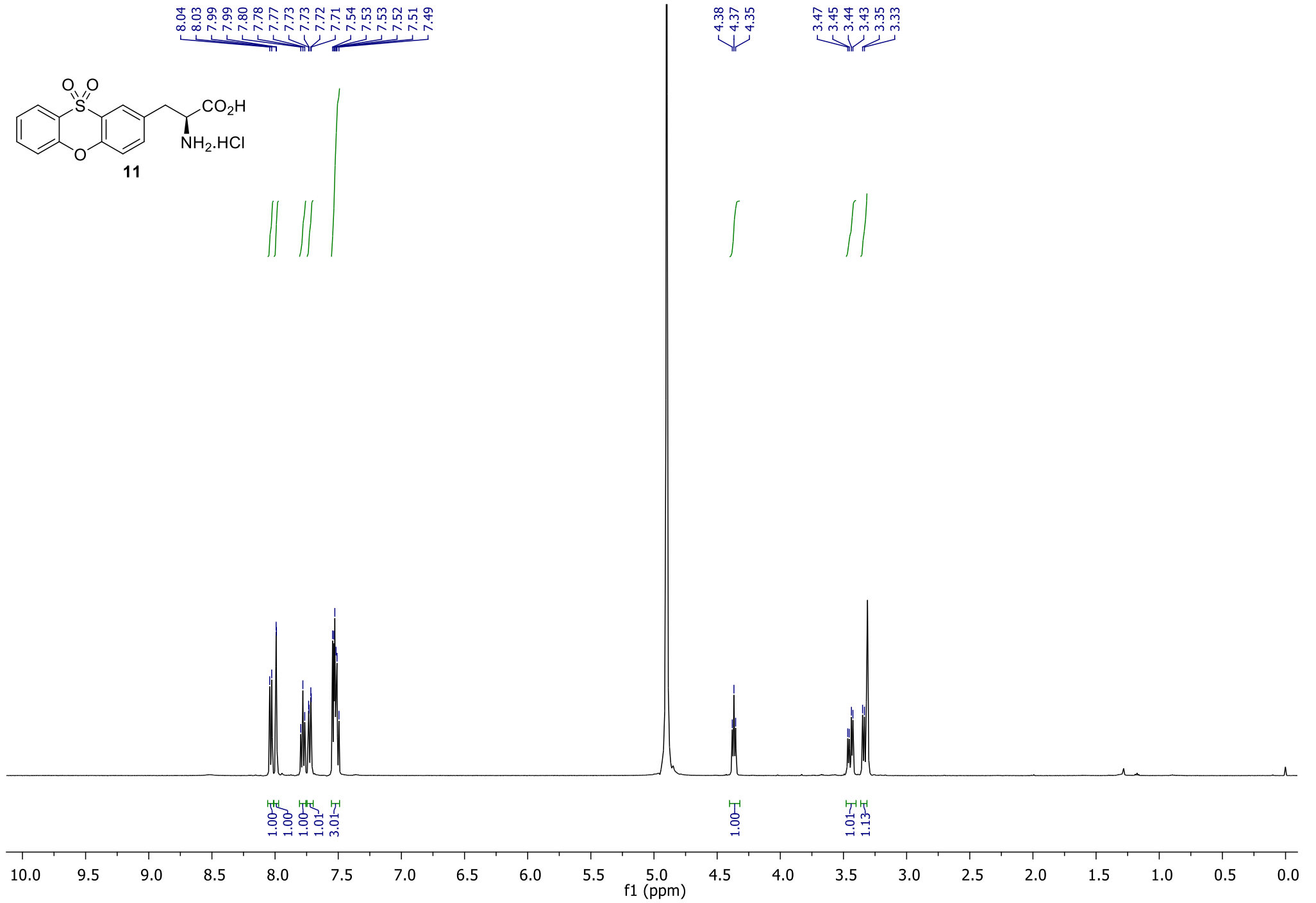
¹H NMR (500 MHz, CD₃OD)



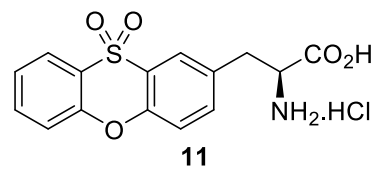
8.04
8.03
7.99
7.99
7.80
7.78
7.77
7.73
7.73
7.72
7.71
7.54
7.53
7.53
7.52
7.51
7.49

4.38
4.37
4.35

3.47
3.45
3.44
3.43
3.35
3.33



^{13}C NMR (126 MHz, CD_3OD)



— 170.85

— 152.94
— 152.56

— 136.93
— 135.93

— 133.39

— 126.48

— 126.25

— 124.95

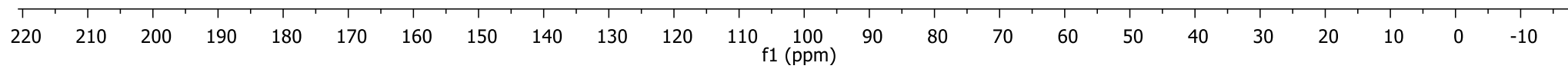
— 124.12

— 120.94

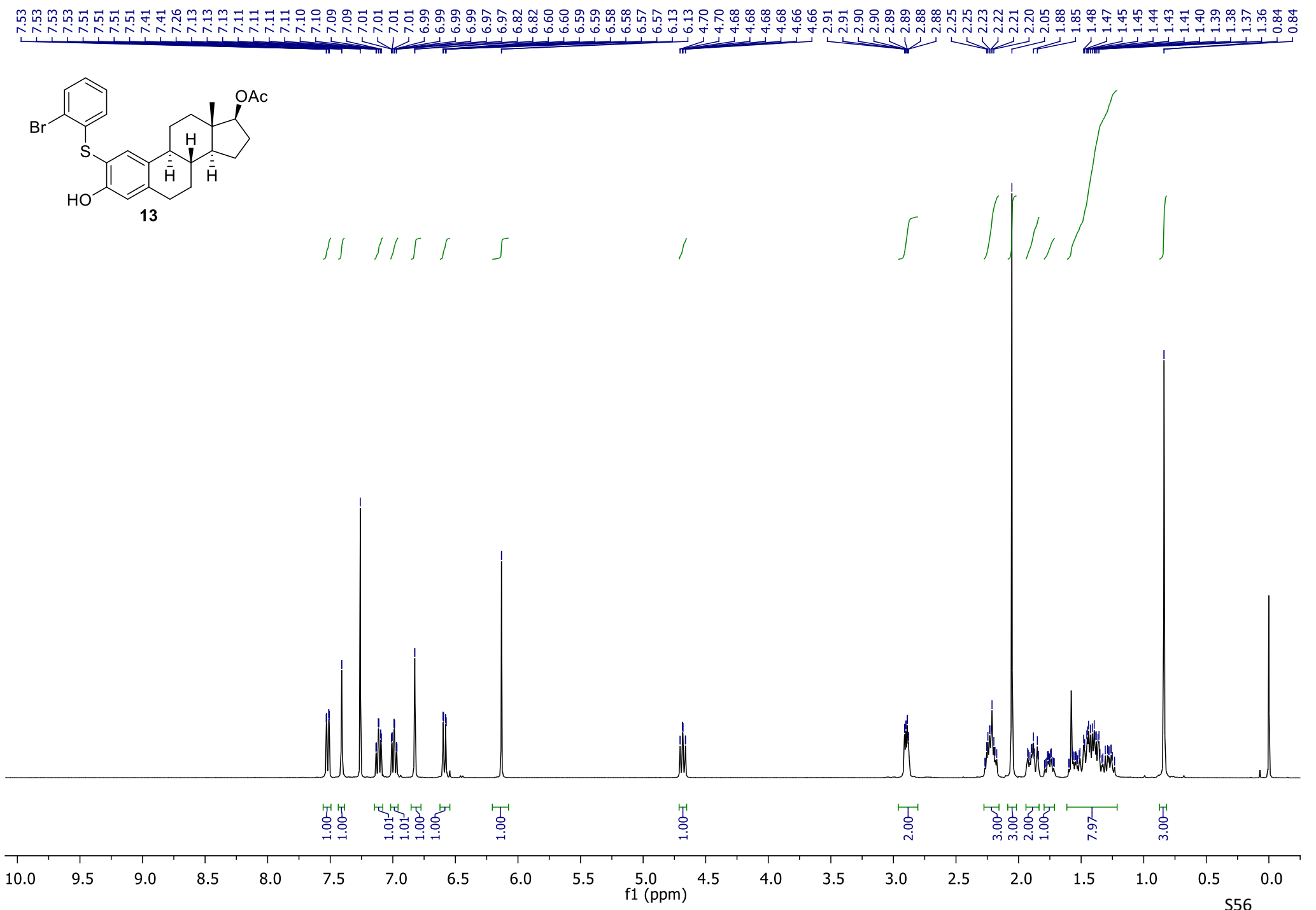
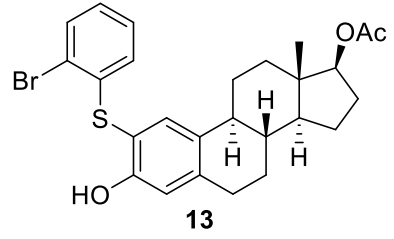
— 120.17

— 54.74

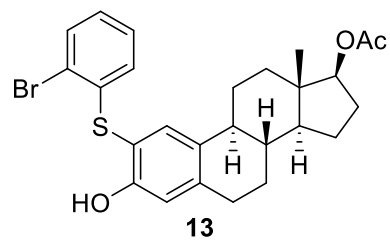
— 36.36



¹H NMR (400 MHz, CDCl₃)



¹³C NMR (101 MHz, CDCl₃)



— 171.36

— 155.29

— 142.62

— 137.95

— 134.26

— 134.19

— 133.02

— 128.12

— 126.83

— 126.60

— 120.82

— 115.66

— 111.98

— 82.75

— 49.92

— 43.78

— 43.00

— 38.42

— 36.88

— 29.80

— 27.69

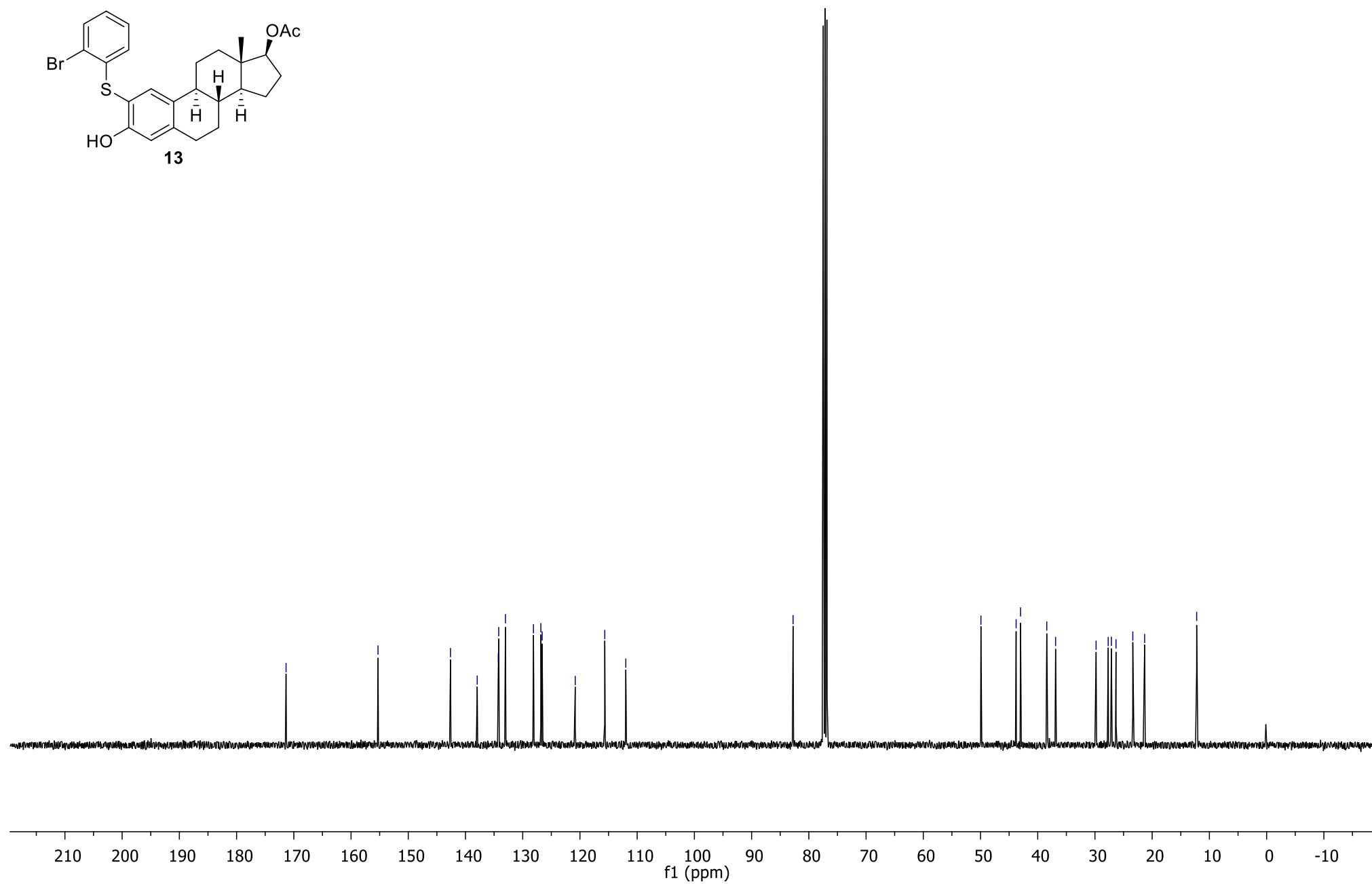
— 27.13

— 26.32

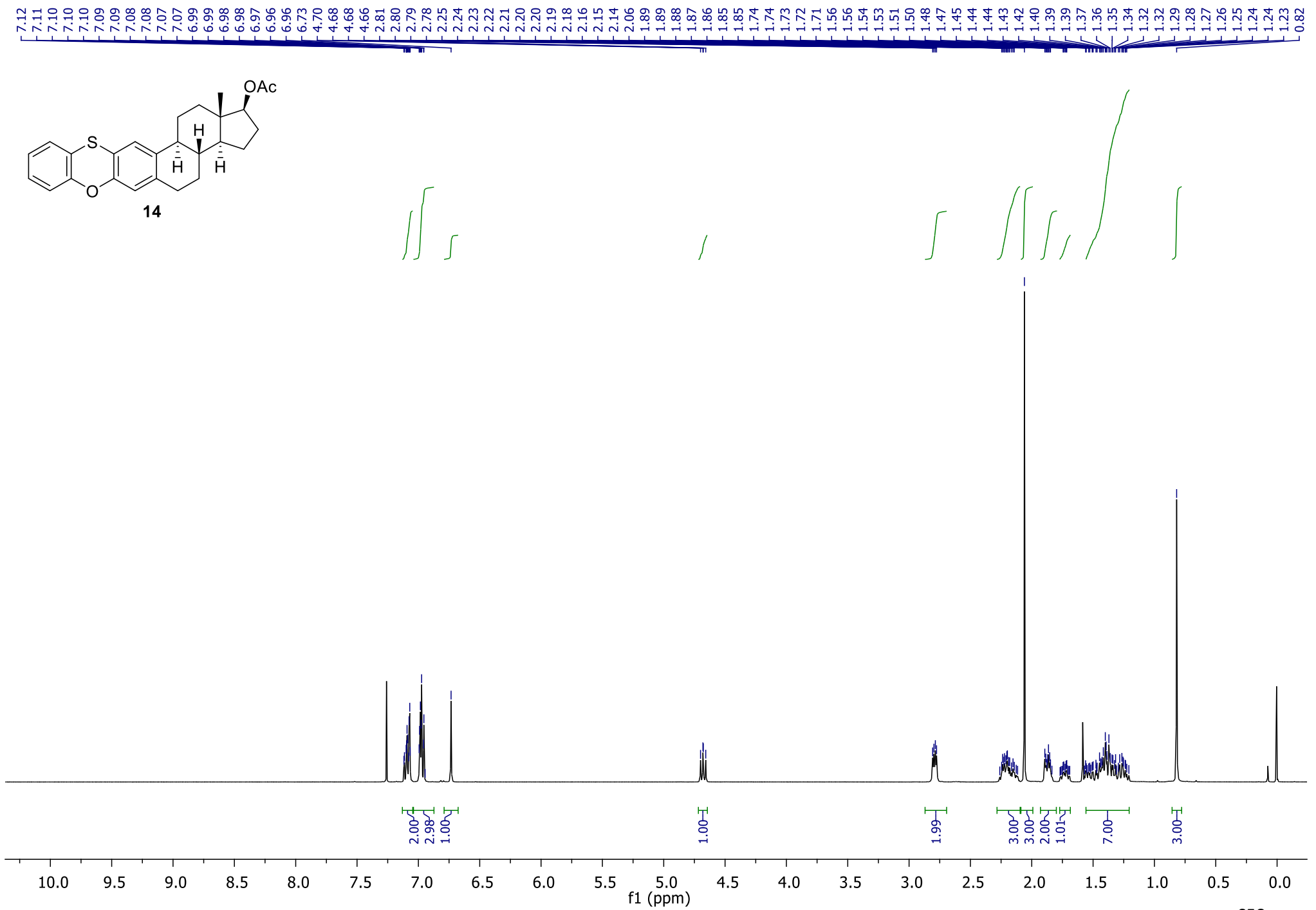
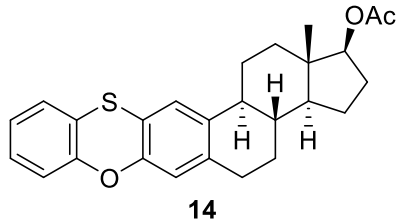
— 23.39

— 21.32

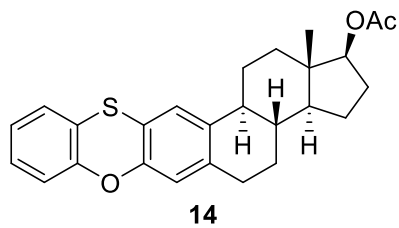
— 12.22



¹H NMR (400 MHz, CDCl₃)



¹³C NMR (101 MHz, CDCl₃)



— 171.34

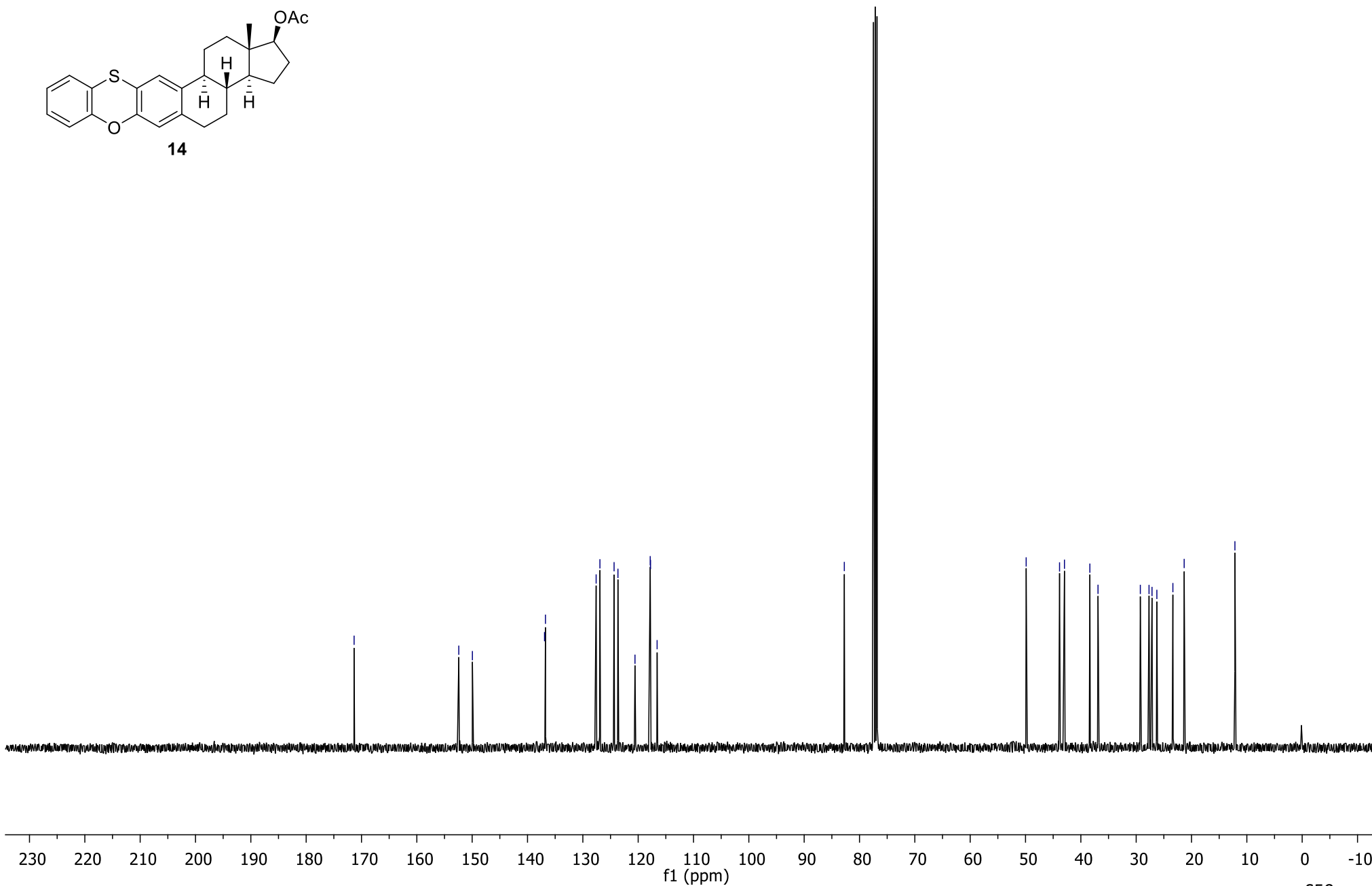
— 152.42
— 149.96

— 136.92
— 136.75
— 127.60
— 126.91
— 124.35
— 123.66
— 120.58
— 117.82
— 117.77
— 116.58

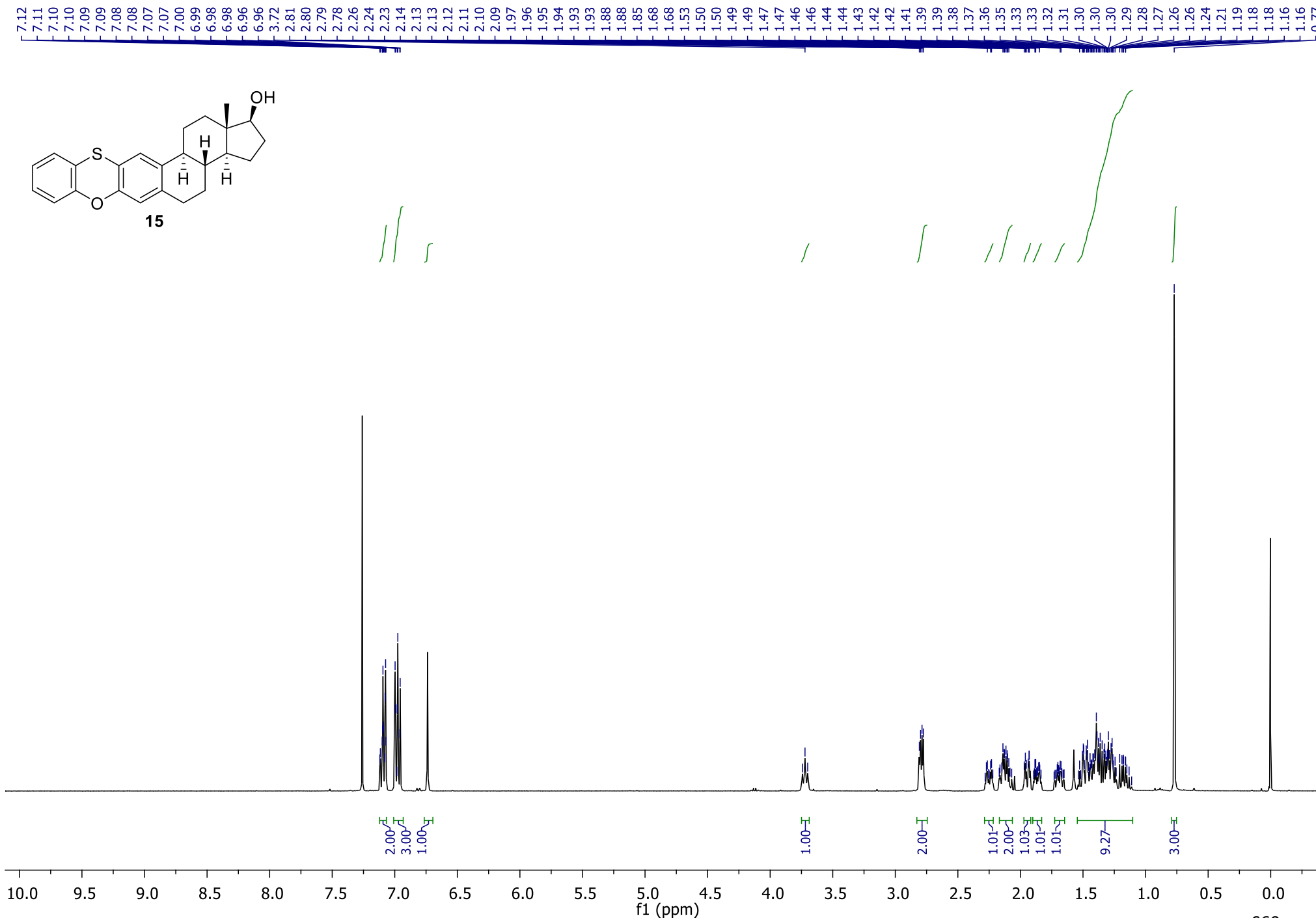
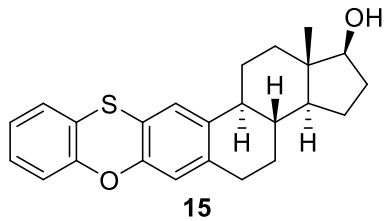
— 82.75

— 49.88
— 43.85
— 42.96
— 38.37
— 36.90
— 29.25
— 27.68
— 27.14
— 26.26
— 23.37
— 21.33

— 12.16



¹H NMR (400 MHz, CDCl₃)



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

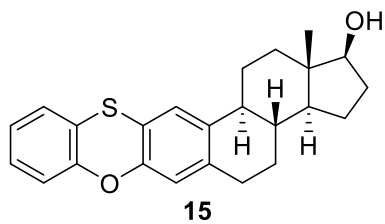
0.0

f1 (ppm)

S60

7.12
7.11
7.10
7.09
7.09
7.08
7.07
7.07
7.00
6.99
6.98
6.98
6.96
6.96
3.72
2.81
2.80
2.79
2.78
2.26
2.24
2.23
2.14
2.13
2.13
2.12
2.11
2.10
2.09
1.97
1.96
1.95
1.94
1.93
1.93
1.88
1.88
1.85
1.68
1.68
1.53
1.50
1.50
1.49
1.49
1.47
1.47
1.46
1.46
1.44
1.44
1.43
1.42
1.42
1.41
1.39
1.39
1.38
1.37
1.36
1.35
1.33
1.33
1.32
1.31
1.30
1.30
1.29
1.28
1.27
1.26
1.26
1.24
1.24
1.19
1.18
1.18
1.16
1.16
0.77

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



152.44
149.95
137.07
136.85
127.60
126.92
124.35
123.67
120.61
117.83
117.79
116.56
81.97
50.14
44.02
43.32
38.65
36.72
30.72
29.29
27.17
26.39
23.25
11.16

