Supporting Information for:

Ligand-, base-, co-catalyst-free copper fluorapatite (CuFAP) as a versatile, ecofriendly, heterogeneous and reusable catalyst for an efficient homocoupling of arylboronic acid at ambient reaction condition

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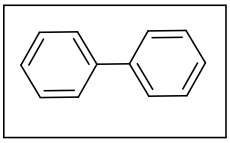
CONTENTS

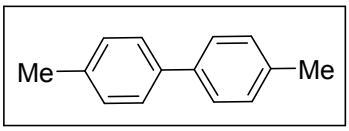
General Remarks: The Copper fluoroapatite catalyst was prepared according to reported procedures[1] and prepared catalyst was tested by one example with standard reaction condition of published paper. All reagents and solvents were obtained from commercial suppliers and Aldrich, Merck Millipore. All these commercially available chemicals were used without further purification. Reactions were monitored by thin layer chromatography (TLC) on silica gel plates (GF 254) using UV light to visualize the course of the reactions. ^{1}H NMR spectra and ^{13}C NMR spectra were respectively recorded at 200, 400, 500 MHz and 50 MHz spectrometer using $CDCl_3$ as solvent at room temperature. Chemical shifts (δ) are reported in ppm with TMS as internal standard. Abbreviations for signal couplings are: s, singlet; d, doublet; t, triplet; m, multiplet. Routine monitoring of reaction was performed by TLC using 0.25 mm E. Merck precoated silica gel TLC plates (60 F254).

General experimental procedure for homo-coupling reaction of arylboronic acid: Arylboronic acid (1 mmol), methanol (5 mL) were taken in 10 mL round bottom flask and it was stirred for 5-10 minutes, after that CuFAP catalyst (100 mg) were added into the reaction mixture and the reaction mixture was stirred at ambient temperature for 2 h (Table 2 and 3) and the progress of the reaction was monitored by TLC. After the completion of the reaction, the reaction mixture was diluted with 10 ml methanol followed by filtration to recover the catalyst. The filtrate was dried under vacuum, thereafter added 10 ml water and 10 ml ethyl acetate to separate out the organic layer and dried over anhydrous Na_2SO_4 to concentrate in vacuum to get the crude homo-coupling products; all compounds were isolated without column chromatography and confirmed by NMR.

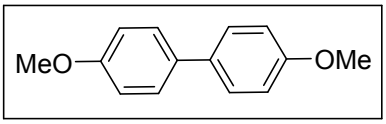
3) Spectral data of the products are given below:



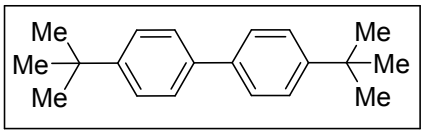
(**2a) 1,1'-biphenyl.** White solid (mp 68-70 ^{o}C); Yield: 92%; ^{1}H NMR (200 MHz, $CDCl_3$) d ppm: 7.08 - 7.15 (m, 3 H) 7.30-738 (m, 3 H) 7.69 - 7.74 (m, 4 H); ^{13}C NMR (50 MHz, $CDCl_3$) δ ppm: 141.9.129.4, 127.8,



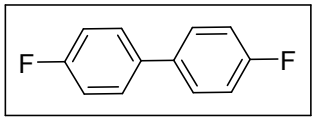
(2b), 4,4'-dimethyl-1,1'-biphenyl. Colorless solid (mp 120 ^{o}C); Yield: 94%; ^{1H} NMR (200 MHz, $CDCl_3$) δ ppm: 2.41 (s, 3 H) 7.21-7.25 (d, *J*=7.96 Hz, 4 H) 7.45 - 7.49 (d, 4 H); ^{13}C NMR (50 MHz, $CDCl_3$) δ ppm: 137.7, 135.9, 128.8, 126.2, 20.5,



(2c), 4,4'-dimethoxy-1,1'-biphenyl. White powder (mp 178-180 ^{o}C); Yield: 93%; ^{1H} NMR (400 MHz, $CDCl_3$) d ppm: 7.45-7.47 (d, 4 H) 6.93-7.95 (d, *4H* H) 3.85 (s, 6 H); ^{13}C NMR (50 MHz, $CDCl_3$) δ 158.7, 133.6, 129.4, 127.7, 114.2, 55.2.

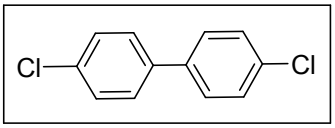


(2d) 4,4'-di-tert-butyl-1,1'-biphenyl. Light brown solid (mp 126-128^{o}C); Yield: 96%; ^{1}H NMR (400 MHz, CHLOROFORM-*d*) ppm 7.24 - 7.32 (d, 4 H) 6.83 - 6.85 (d, 4 H) 1.32 (s, 18 H); ^{13}C NMR (50 MHz, $CDCl_3$) δ 157.9, 150.4, 143.8, 138.9, 133.3, 127.3, 127.0, 126.8, 126.2, 115.4, 114.0, 77.58, 55.7, 34.7, 32.2.

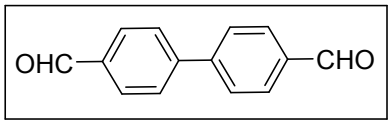


(**2e) 4,4'-difluoro-1,1'-biphenyl.** Pale yellow powder (mp 90-91^{o}C); Yield: 90%; ^{1}H NMR (400 MHz, CDCl3) d ppm 7.47 - 7.50 (d, 4 H); 7.10 - 7.14 (d, 4 H); ^{13}C NMR (400 MHz, $CDCl_3$) δ

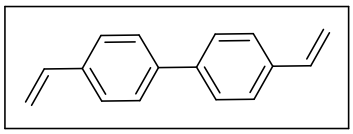
163.4, 160.9, 136.1, 128.3, 128.2, 115.5, 115.3.



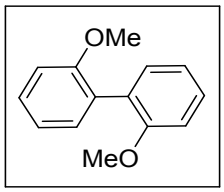
(2f), 4,4'-dichloro-1,1'-biphenyl. Colorless solid (mp 152-154 °C); Yield: 93; ^{1}H NMR (400 MHz, CDCl3) d ppm 7.34 - 7.38 (d, 4 H) 7.55 - 7.59 (d, 4 H); ^{13}C NMR (50 MHz, $CDCl_3$) δ ppm 140.9, 132.4, 129.9, 127.2.



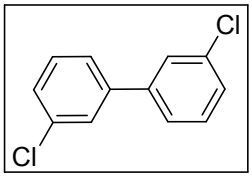
(2g), [1,1'-biphenyl]-4,4'-dicarbaldehyde. Colorless solid (mp 148-150 °C); Yield: 85%; ^{1}H NMR (200 MHz, $CDCl_3$) d ppm: 7.78-782 (d, *J*=8.3 Hz, 4 H) 7.99-8.03 (d, *J*=8.3 Hz, 4 H) 10.09 (s, 2 H); ^{13}C NMR (50 MHz, $CDCl_3$) δ ppm: 190.4, 145.2, 140.2, 138.6, 132.5, 131.7, 129.3,



(2h) 4,4'-divinyl-1,1'-biphenyl. Colorless solid (mp 138-140) °C; Yield: 92%; ^{1}H NMR (400 MHz, $CDCl_3$) ppm 7.56 - 7.58 (d, 4 H) 7.46 - 7.48 (d, 4 H) 6.72-6.79 (dd, *J*=17.63, 10.76 Hz, 2 H) 5.77 - 5.81 (d, 2 H) 5.26-5.29 (d, *J*=10.99 Hz, 2 H); ^{13}C NMR (50 MHz, $CDCl_3$) δ 140.3, 136.5, 131.8, 130.4, 128.2, 127.0, 126.7, 113.8, 77.0.

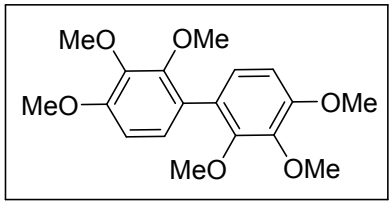


(**2i), 2,2'-dimethoxy-1,1'-biphenyl.** Colorless solid (mp 154-156 °C); Yield: 95%; ^{1H} NMR (200 MHz, $CDCl_3$) d ppm: 3.84 (s, 6 H) 7.00 - 7.05 (m, 4 H) 7.27 - 7.41 (m, 4 H); ^{13}C NMR (50 MHz, $CDCl_3$) δ ppm: 132.1, 129.2, 121.6, 111.6, 56.2.

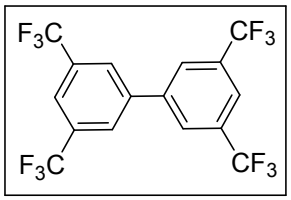


(2j) 3,3'-dichloro-1,1'-biphenyl. Colorless solid (mp 30-32 °C); Yield: 95%; ^{1}H NMR (400

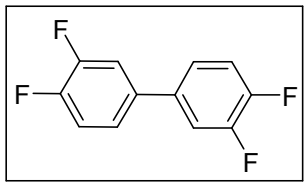
MHz, $CDCl_3$) ppm 7.55 (s, 2 H) 7.42 - 7.44 (m, 2 H) 7.35 - 7.42 (m, 4 H); ^{13}C NMR (50 MHz, $CDCl_3$) δ 141.7, 134.9, 130.4, 130.1, 127.9, 127.3, 125.2.



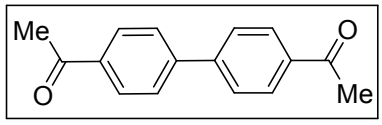
(2k), 2,2',3,3',4,4'-hexamethoxy-1,1'-biphenyl. White solid (mp 123-126 °C); Yield: 98%; ^{1H} NMR (200 MHz, $CDCl_3$) δ ppm: 3.85 (s, 6 H) 3.87 (s, 12 H) 6.54-658 (d, *J*=8.34 Hz, 2 H) 6.93-6.97 (d, *J*=8.72 Hz, 2 H); ^{13}C NMR (50 MHz, $CDCl_3$) δ ppm: 154.1, 147.6, 139.2, 138.8, 124.1, 123.3, 105.8, 61.5, 56.5.



(2m), 3,3',5,5'-tetrakis(trifluoromethyl)-1,1'-biphenyl. White solid (mp 68-70 °C); Yield: 97%; ^1H NMR (200 MHz, $CDCl_3$) d ppm: 7.76 - 8.14 (m, 6 H); ^{13}C NMR (50 MHz, $CDCl_3$) δ ppm: 139.7, 137.0, 133.4, 130.2, 129.8, 123.0, 125.2,

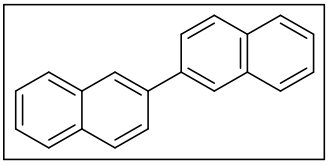


(2n) 3,3',4,4'-tetrafluoro-1,1'-biphenyl. Colorless solid (mp 81-82 °C); Yield: 93%; ^1H NMR (400 MHz, $CDCl_3$) ppm 7.27 - 7.34 (m, 2 H) 6.93 - 7.98 (m, 4 H); ^{13}C NMR (50 MHz, $CDCl_3$) δ 160.7, 158.8, 132.9, 131.9, 118.6, 111.3, 111.1, 104.3, 104.1, 103.9.

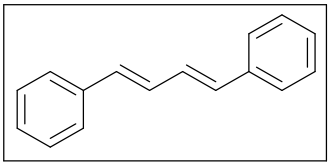


(2o) 1,1'-([1,1'-biphenyl]-4,4'-diyl)bis(ethan-1-one). Colorless solid (mp 193-194 °C); Yield: 88%; ^1H NMR (400 MHz, $CDCl_3$) ppm 7.75 – 7.87 (d, 4 H) 6.74 - 7.53 (d, 4 H) 2.48 (s, 3 H)

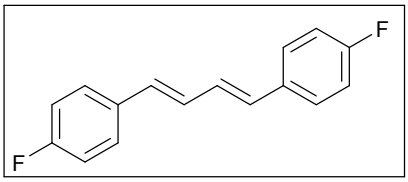
2.37 (s, 3 H); ^{13}C NMR (50 MHz, $CDCl_3$) δ 197.1, 146.9, 144.4, 136.6, 130.9, 130.6, 129.0, 127.4, 115.3, 113.7, 26.6.



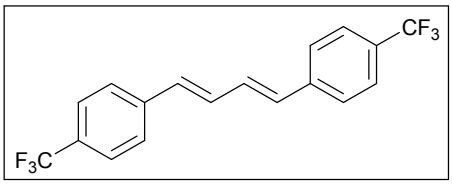
(2p) 2,2'-binaphthalene. White powder (mp 180-181 °C); Yield: 90%; ^{1}H NMR (400 MHz, $CDCl_3$) ppm 7.76 - 7.96 (m, 4 H) 7.74- 7.76 (m, 3 H) 7.49 - 7.51 (m, 4 H) 7.48 (t, 1 H) 7.14 (m, 2 H); ^{13}C NMR (126 MHz, $CDCl_3$) d ppm 138.7, 134.9, 134.1, 133.7, 133.0, 131.6, 129.6, 129.3, 128.8, 128.7, 128.5, 128.1, 127.9, 127.9, 127.7, 127.0, 126.6, 126.4, 126.2, 126.0, 126.0, 124.5, 124.2, 123.9, 123.8, 123.7.



(2q) (1E,3E)-1,4-diphenylbuta-1,3-diene. Pale yellow solid irritated (mp 152 °C); Yield: 96%; ^{1}H NMR (400 MHz, $CDCl_3$) δ ppm 7.39 - 7.41 (m, 4 H) 7.27 - 7.31 (m, 4 H) 7.17 - 7.21 (m, 2 H) 6.89 - 6.93 (dd, 2 H) 6.61 - 6.65 (dd, 2 H); ^{13}C NMR (126 MHz, $CDCl_3$) δ ppm 135.9, 133.6, 132.2, 131.1, 129.7, 129.1, 128.9, 128.7, 127.8.

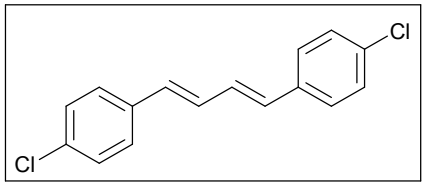


(2r) (1E,3E)-1,4-bis(4-fluorophenyl)buta-1,3-diene. Black color solid (mp 140-142 °C); Yield: 93%; ^{1}H NMR (400 MHz, $CDCl_3$) δ ppm 7.39 - 7.45 (m, 28 H) 7.31 - 7.35 (d, 4 H) 7.21- 7.25 (dd, 2 H) 6.93 - 6.99 (dd, 2 H); ^{13}C NMR (126 MHz, $CDCl_3$) δ ppm 135.9, 135.0, 134.5, 134.1, 133.6, 132.2, 131.8, 131.5, 131.1, 130.8, 129.7, 129.1, 128.9, 128.1, 128.1, 127.8.

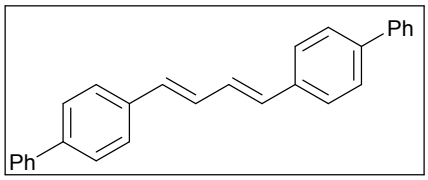


(2s) (1E,3E)-1,4-bis(4-(trifluoromethyl)phenyl)buta-1,3-diene. Pale yellow solid irritated (mp

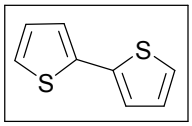
147-149 °C); Yield: 90%; ^{1}H NMR (400 MHz, $CDCl_3$) δ ppm 7.59 - 7.61 (d, 4 H) 7.53 - 7.55 (d, 4 H) 7.01 - 7.05 (dd, 2 H) 6.73 - 6.77 (dd, 2 H); ^{13}C NMR (126 MHz, CDCl3) δ ppm 132.7, 131.2, 130.9, 129.8, 129.3, 128.7, 127.1, 126.6, 125.7.



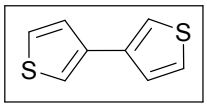
(2t) (1E,3E)-1,4-bis(4-chlorophenyl)buta-1,3-diene. White solid irritated (mp 143 °C); Yield: 94%; ^{1}H NMR (400 MHz, $CDCl_3$) δ ppm 7.34 - 7.37 (m, 4 H) 7.28 - 7.31 (m, 4 H) 7.21 - 7.23 (dd, 2 H) 7.13 - 7.15 (dd, 2 H); ^{13}C NMR (126 MHz, $CDCl_3$) δ ppm 135.9, 133.6, 132.2, 131.5, 131.1, 129.7, 129.1, 128.9, 127.8.



(2u)(1E,3E)-1,4-di([1,1'-biphenyl]-4-yl)buta-1,3-diene. Light yellow solid (mp 196 °C); Yield: 96%; ^{1}H NMR (400 MHz, $CDCl_3$) ppm 7.56 - 7.58 (d, 4 H) 7.41 - 7.50 (m, 10 H) 7.31 - 7.41 (d, 2 H) 6.73-6.80 (dd, 2 H) 5.78-5.82 (dd, *J*=17.86, 0.92 Hz, 2 H); ^{13}C NMR (126 MHz, CDCl3) d ppm 141.1, 140.9, 136.8, 136.7, 130.5, 129.3, 129.0, 128.7, 127.9, 127.6, 127.5, 127.5, 127.2, 127.0, 126.9, 125.7.

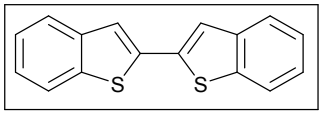


(3a) 2,2'-bithiophene. Colorless solid (mp 31-36 °C); Yield: 95%; ^{1}H NMR (200 MHz, $CDCl_3$) δ ppm 7.36 - 7.38 (m, 2 H) 7.27 - 7.34 (m, 4 H); ^{13}C NMR (50 MHz, $CDCl_3$) δ 136.8, 133.5, 129.4, 128.5, 127.9, 127.7, 126.7, 126.3, 126.1, 125.8, 123.5.

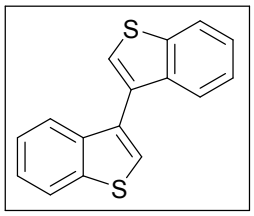


(3b) 3,3'-bithiophene. White solid (mp 130-132 °C); Yield: 96%; ^{1}H NMR (200 MHz, $CDCl_3$) δ ppm 7.17 - 7.27 (m, 4 H) 7.01 (s, 2 H); ^{13}C NMR (126 MHz, CDCl3) d ppm 144.5, 139.0, 118.5,

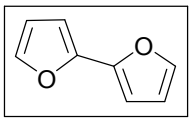
117.1.



(3c) 2,2'-bibenzo[b]thiophene. Colorless solid (mp 197-200 °C); Yield: 93%; ^{1}H NMR (500 MHz, $CDCl_3$) δ ppm 7.86 - 7.89 (m, 2 H) 7.80 - 7.83 (m, 1 H) 7.76 (dd, *J*=7.06, 1.72 Hz, 1 H) 7.51 (s, 2 H) 7.26 - 7.43 (m, 4 H);) ^{13}C NMR (126 MHz, $CDCl_3$) δ ppm 140.0, 139.2, 137.0, 133.7, 132.6, 128.0, 125.9, 125.3, 124.8, 124.6, 124.5, 124.4, 123.9, 123.9, 123.5, 123.5, 123.3, 122.2, 121.9, 121.1.



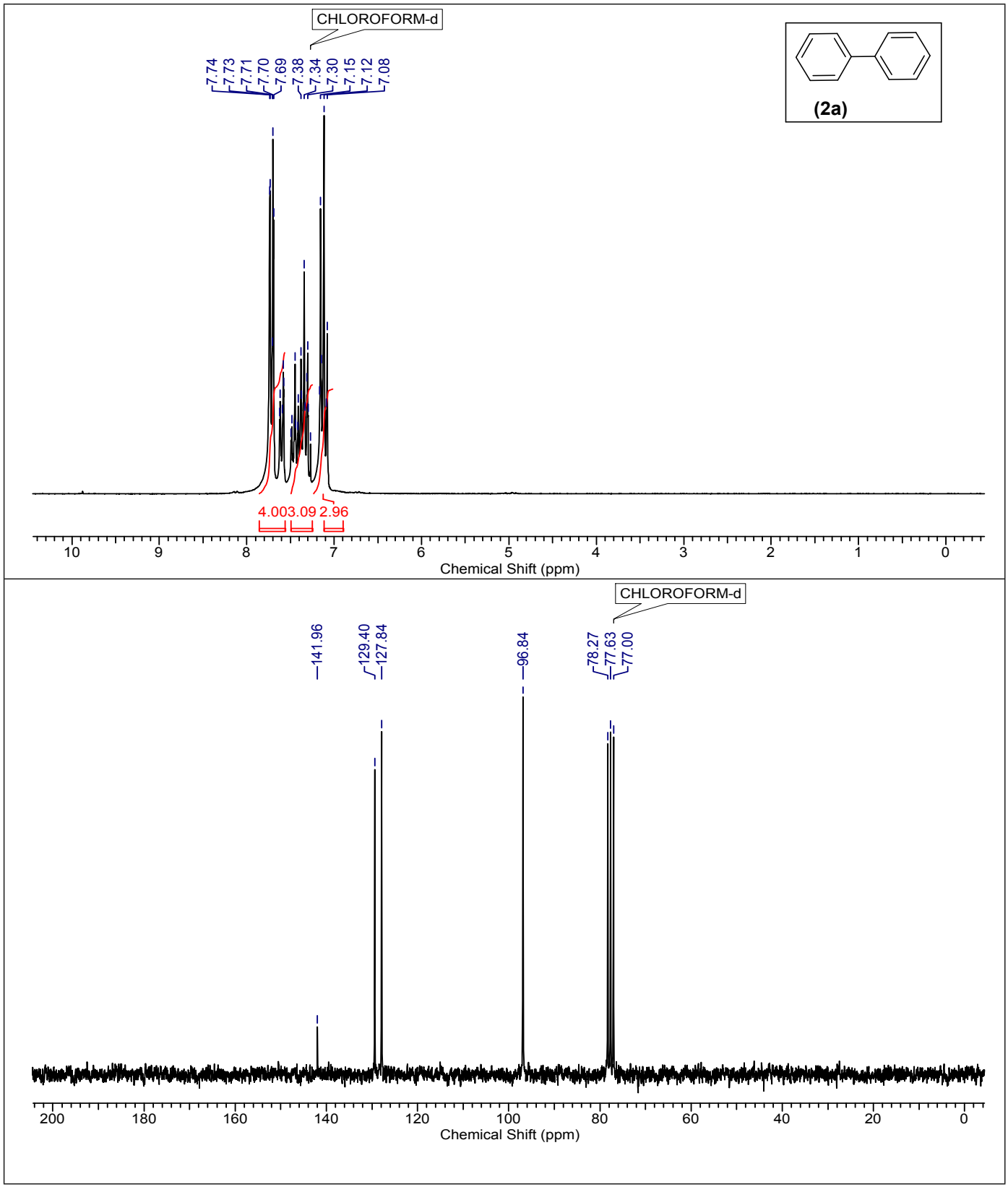
(3d) 3,3'-bibenzo[b]thiophene. Light yellow solid, mp 70 °C; Yield: 95%; ^{1}H NMR (400 MHz, $CDCl_3$) ppm 7.37 - 7.89 (m, 3 H) 7.36 (s, 1 H) 7.33 (s, 1 H) 7.33 - 7.35 (m, 4 H); ^{13}C NMR (200 MHz, $CDCl_3$) d ppm 139.6, 138.6, 134.2, 126.2, 125.2, 124.6, 124.2, 124.1, 123.8, 123.6, 122.4, 118.7.



(3e) 2,2'-bifuran. Colorless oil; Yield: 87%; ^{1}H NMR (400 MHz, $CDCl_3$) δ ppm 7.20 - 7.45 (m, 4 H) 6.02 - 6.31 (dd 14 H), ^{13}C NMR (126 MHz, $CDCl_3$) δ ppm 162.7, 138.0, 128.7, 127.8.

References:

1. Choudary, B. M.; Sridhar, C.; Kantam, M. L.; Venkanna, G. T.; Sreedhar, B. *J. Am. Chem. Soc*. **2005**, *127*, 9948-9949


CHLOROFORM-d
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7.73
7.71
7.70
7.69
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7.30
7.15
7.12
7.08
(2a)
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3.09
2.96
10
9
8
7
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5
4
3
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Chemical Shift (ppm)
CHLOROFORM-d
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127.84
96.84
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77.00
200
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160
140
120
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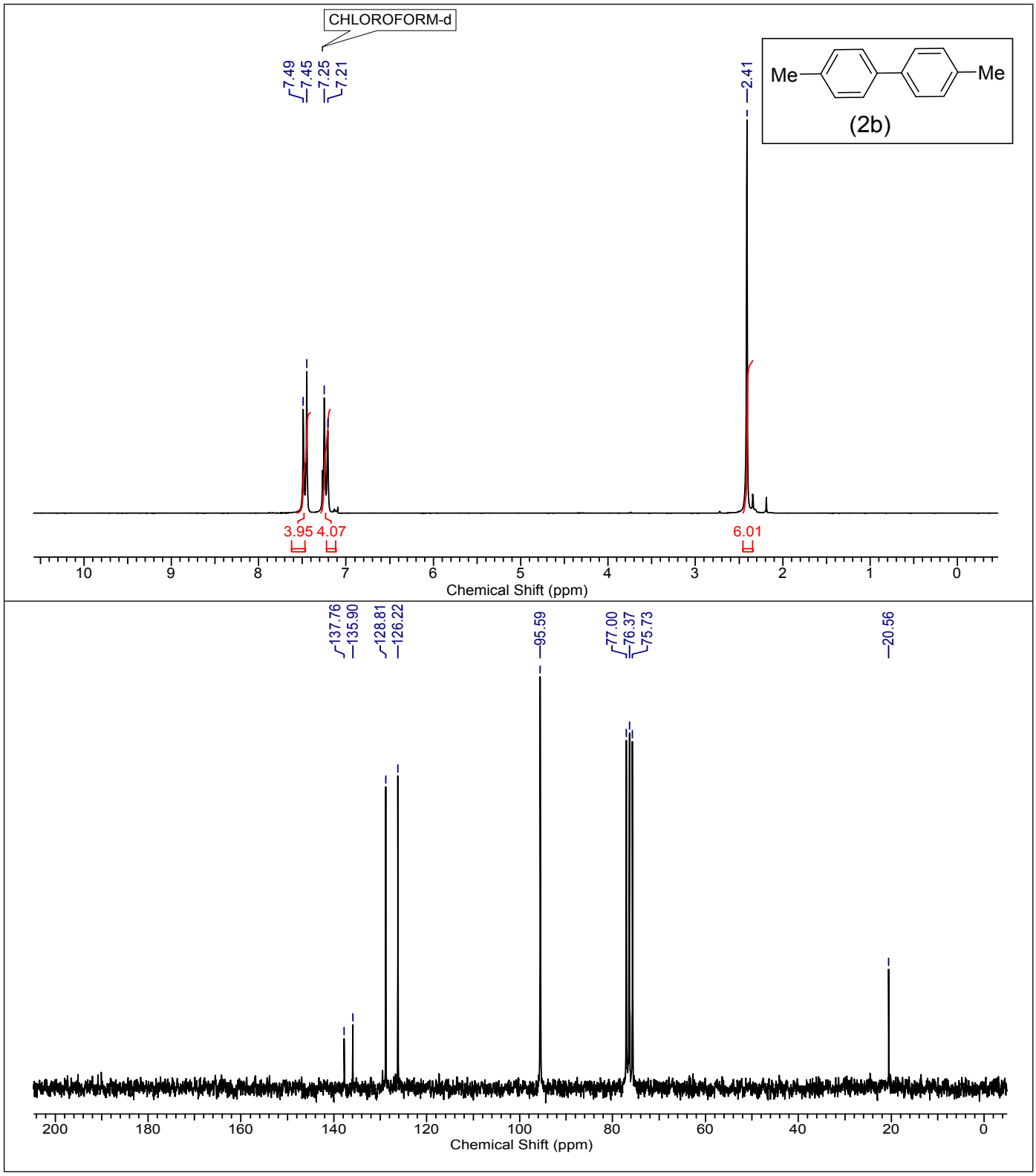
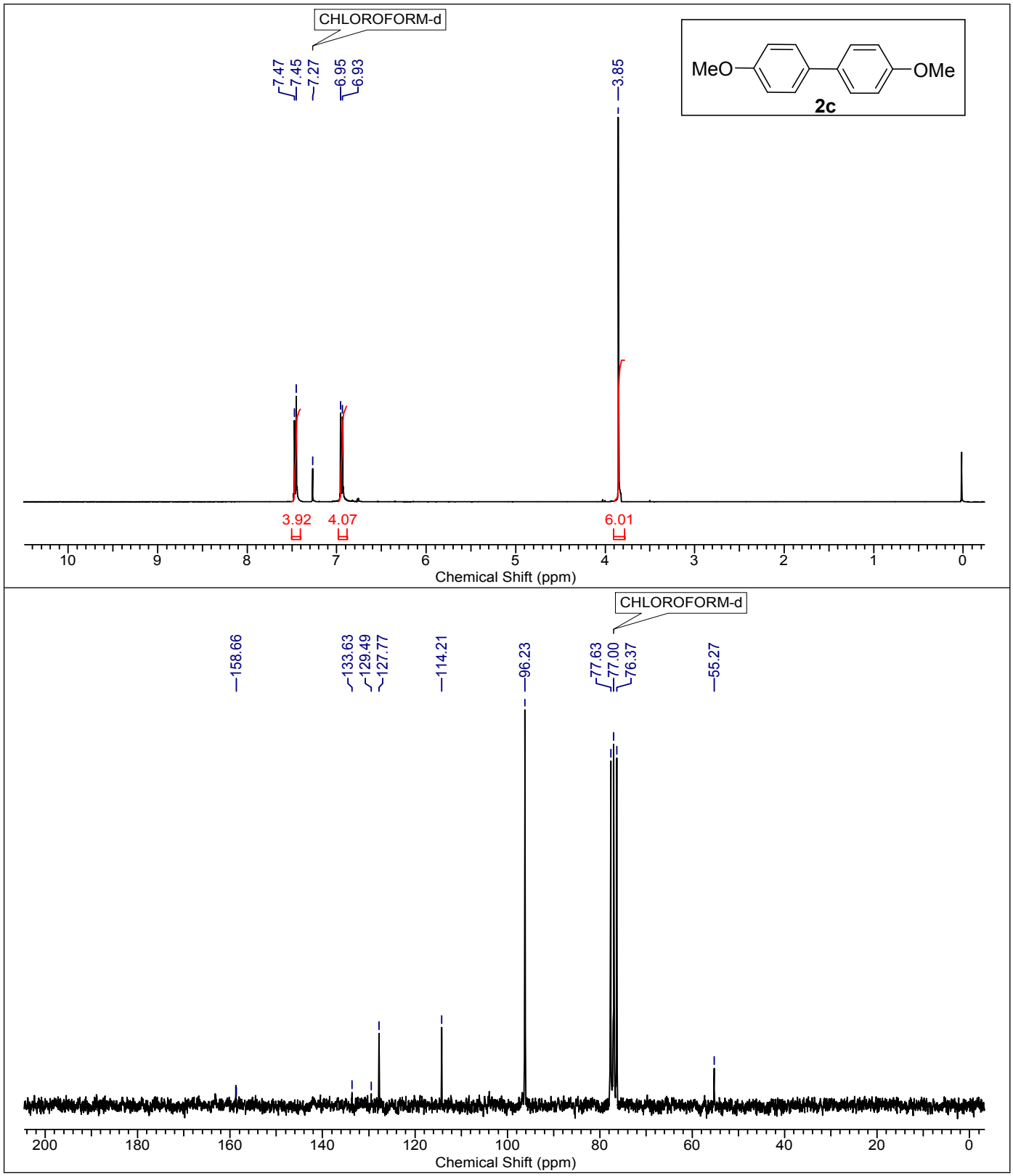
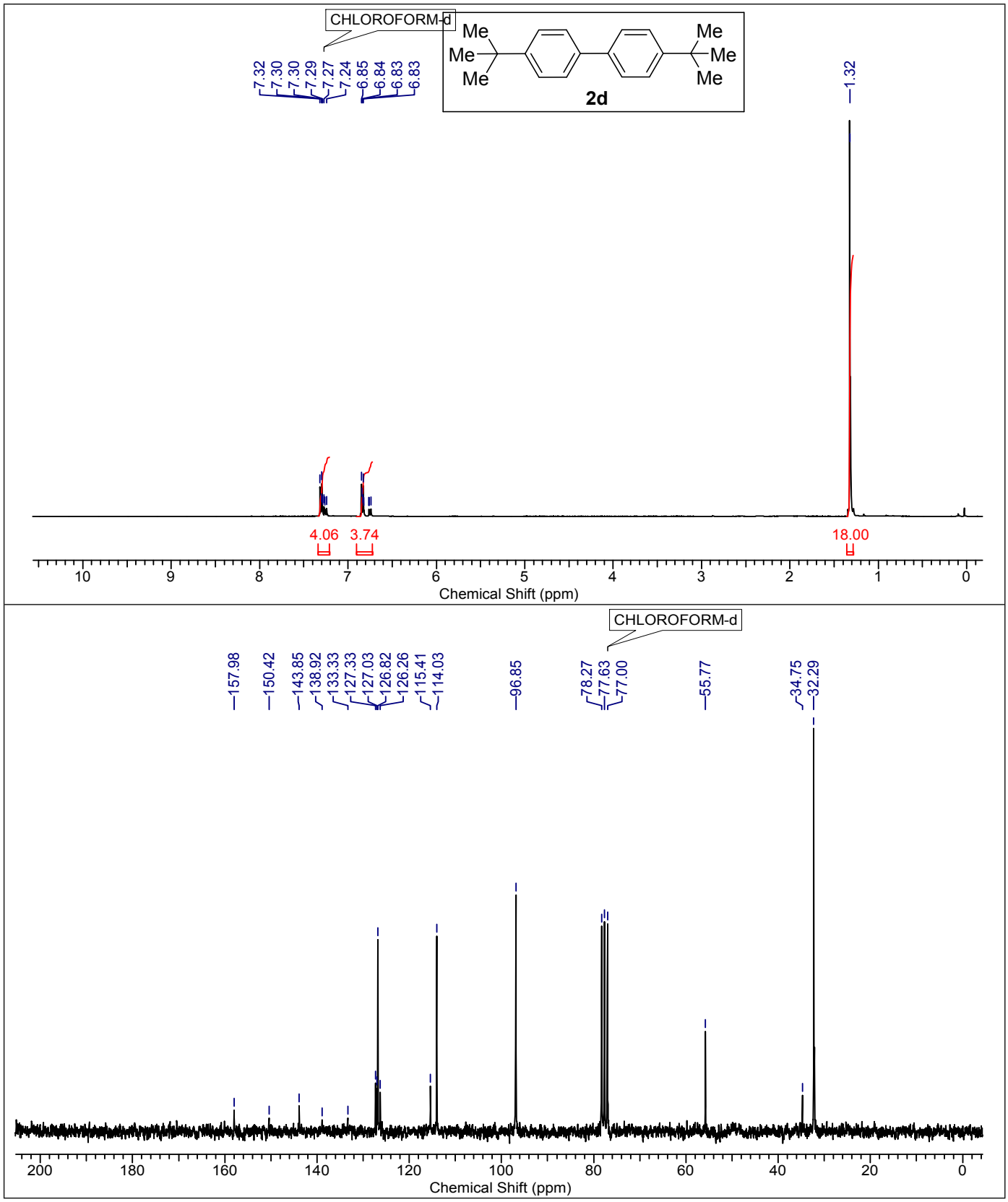
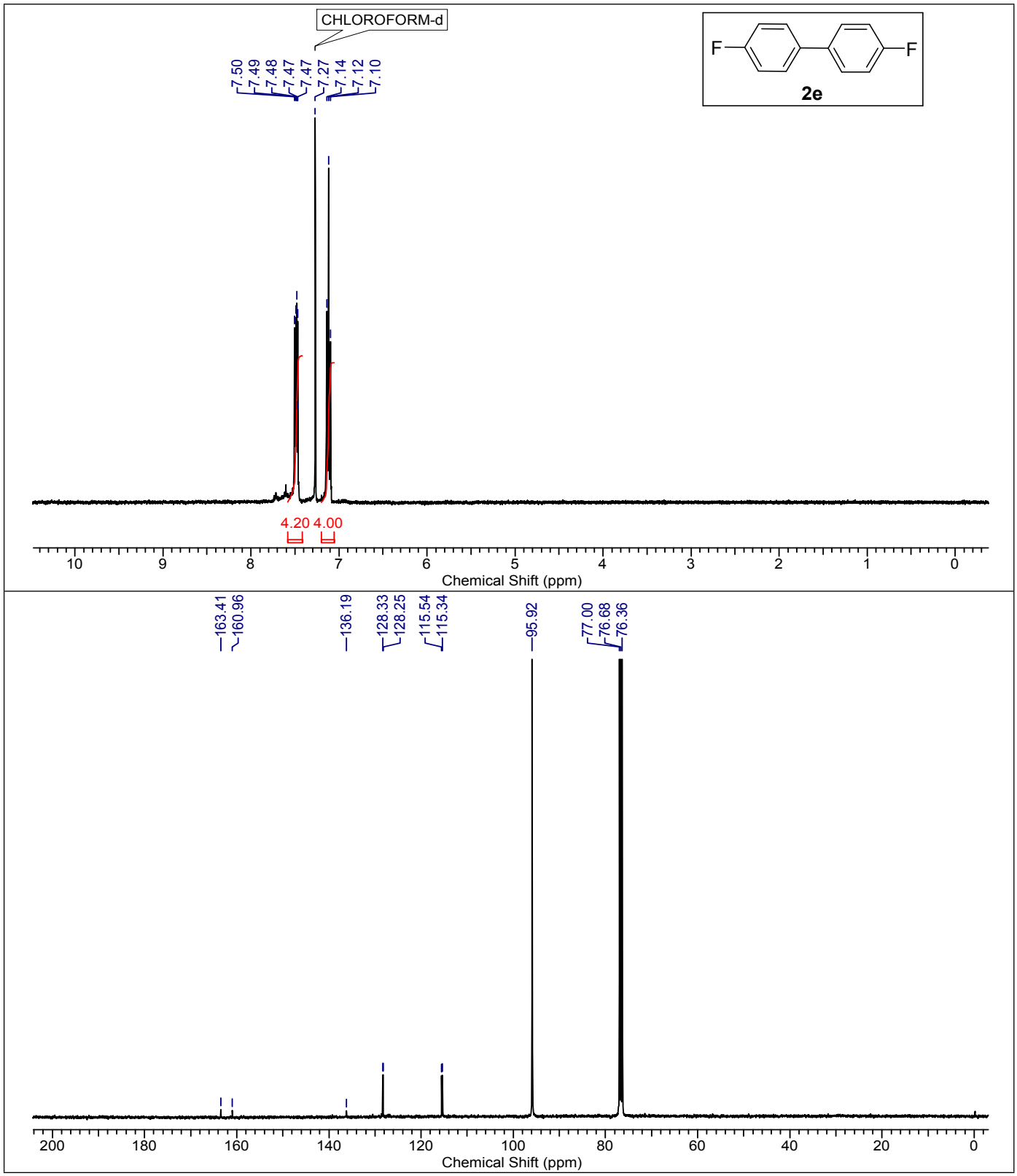
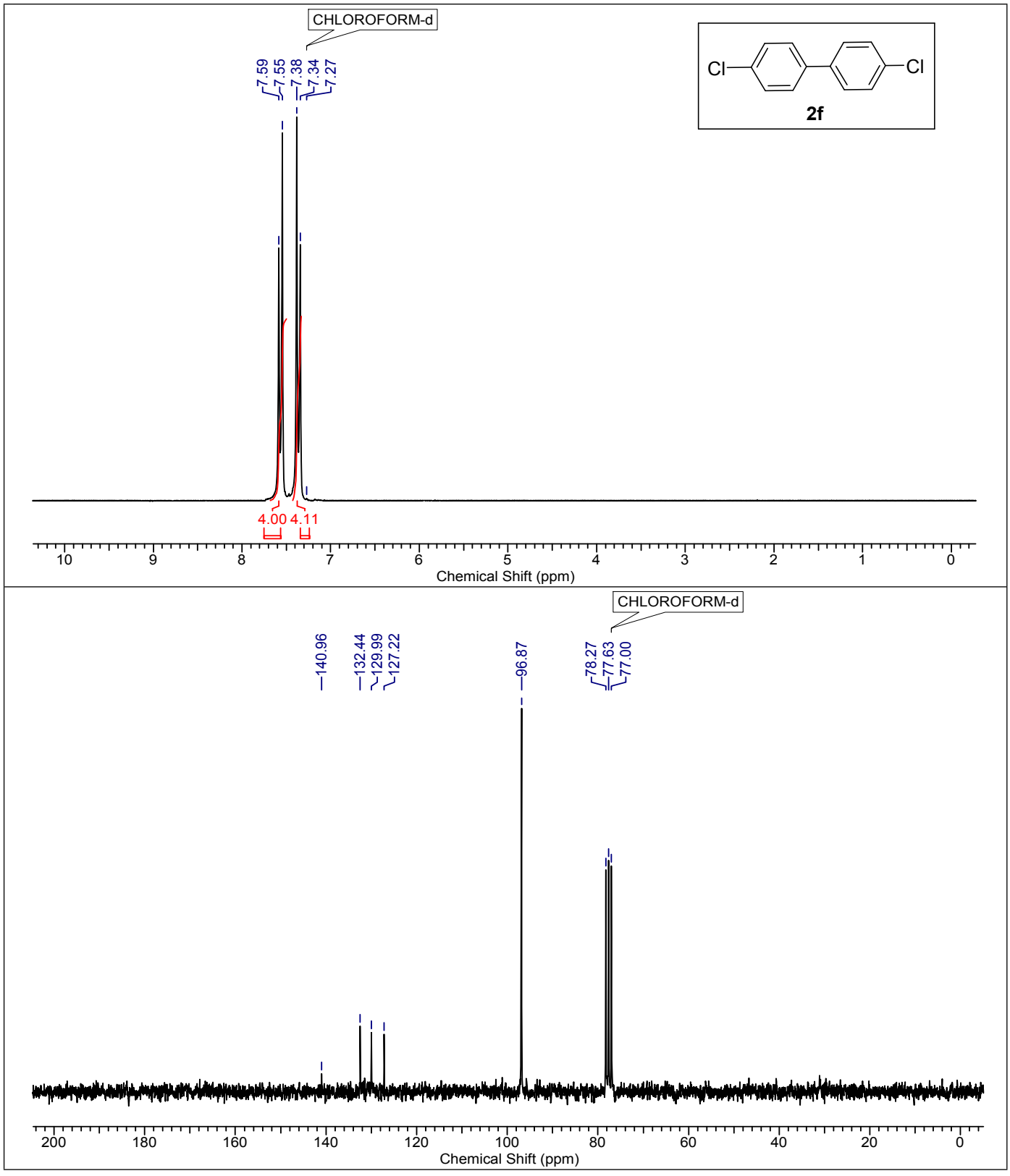
80
60
40
20
0
Chemical Shift (ppm)

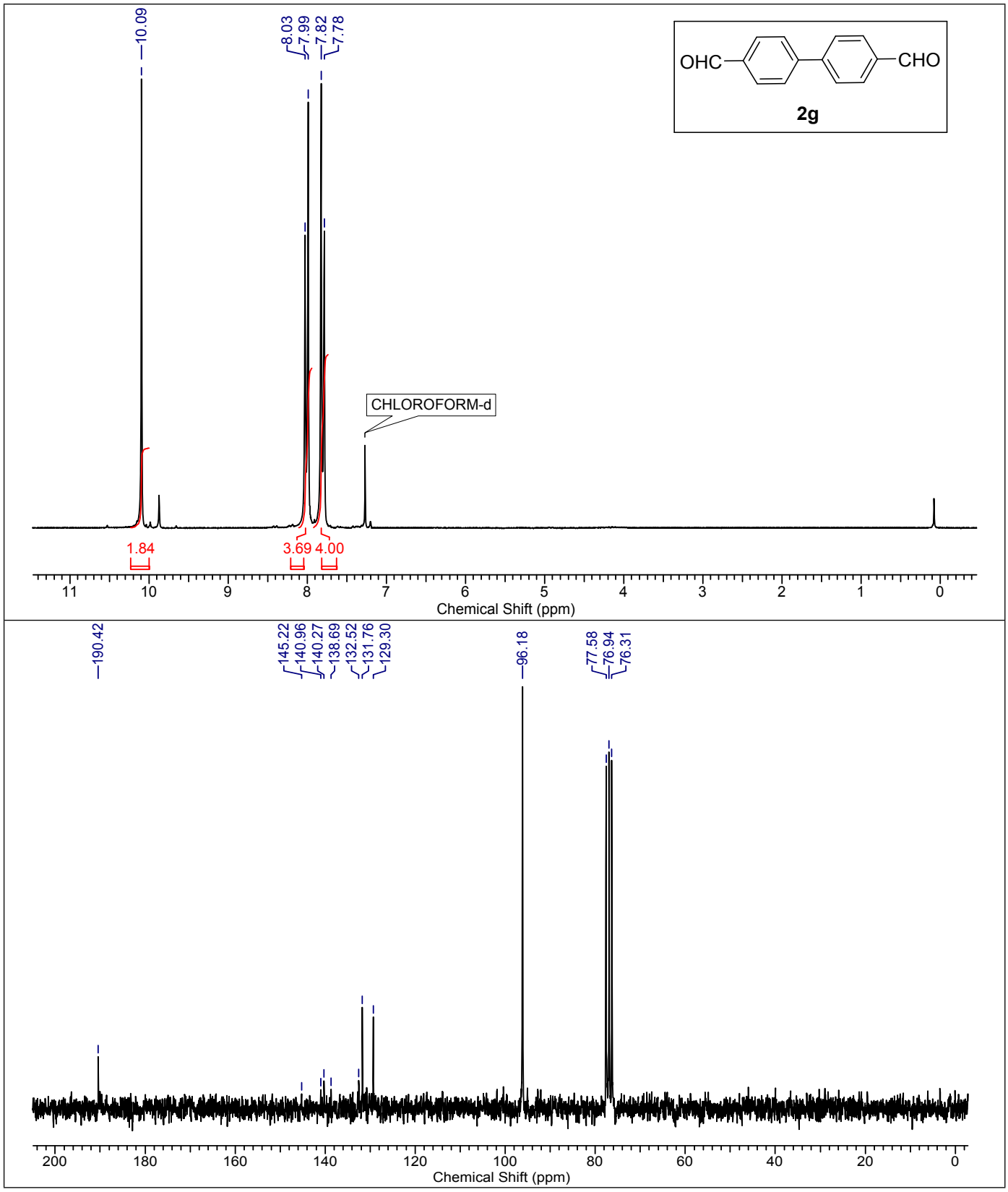

CHLOROFORM-d
7.49
7.45
7.25
7.21
2.41
Me
Me
(2b)
3.95
4.07
6.01
Chemical Shift (ppm)
137.76
135.90
128.81
126.22
95.59
77.00
76.37
75.73
20.56
Chemical Shift (ppm)

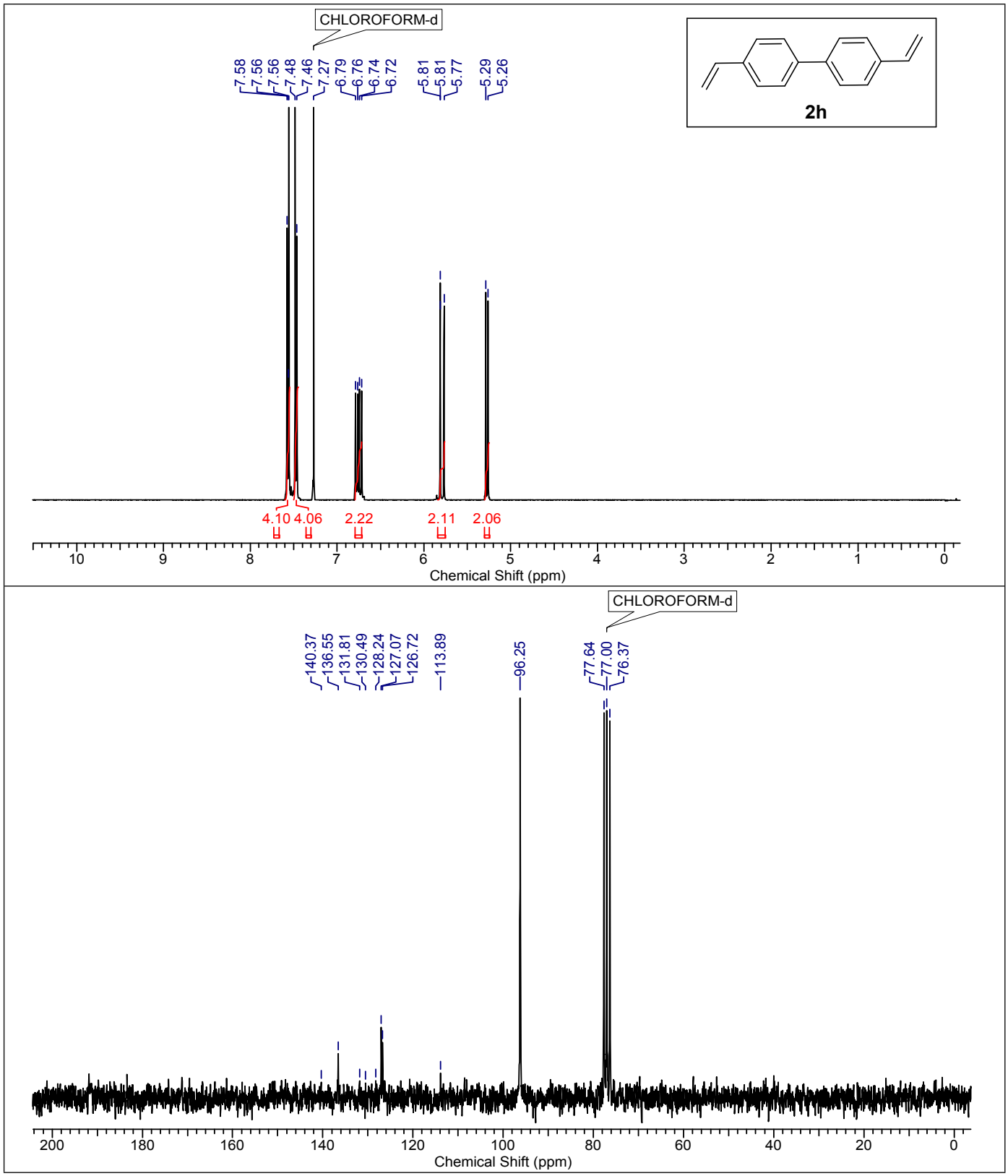

CHLOROFORM-d
7.47
7.45
7.27
6.95
6.93
3.85
MeO
OMe
2c
3.92
4.07
6.01
10
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)
CHLOROFORM-d
158.66
133.63
129.49
127.77
114.21
96.23
77.63
77.00
76.37
55.27
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

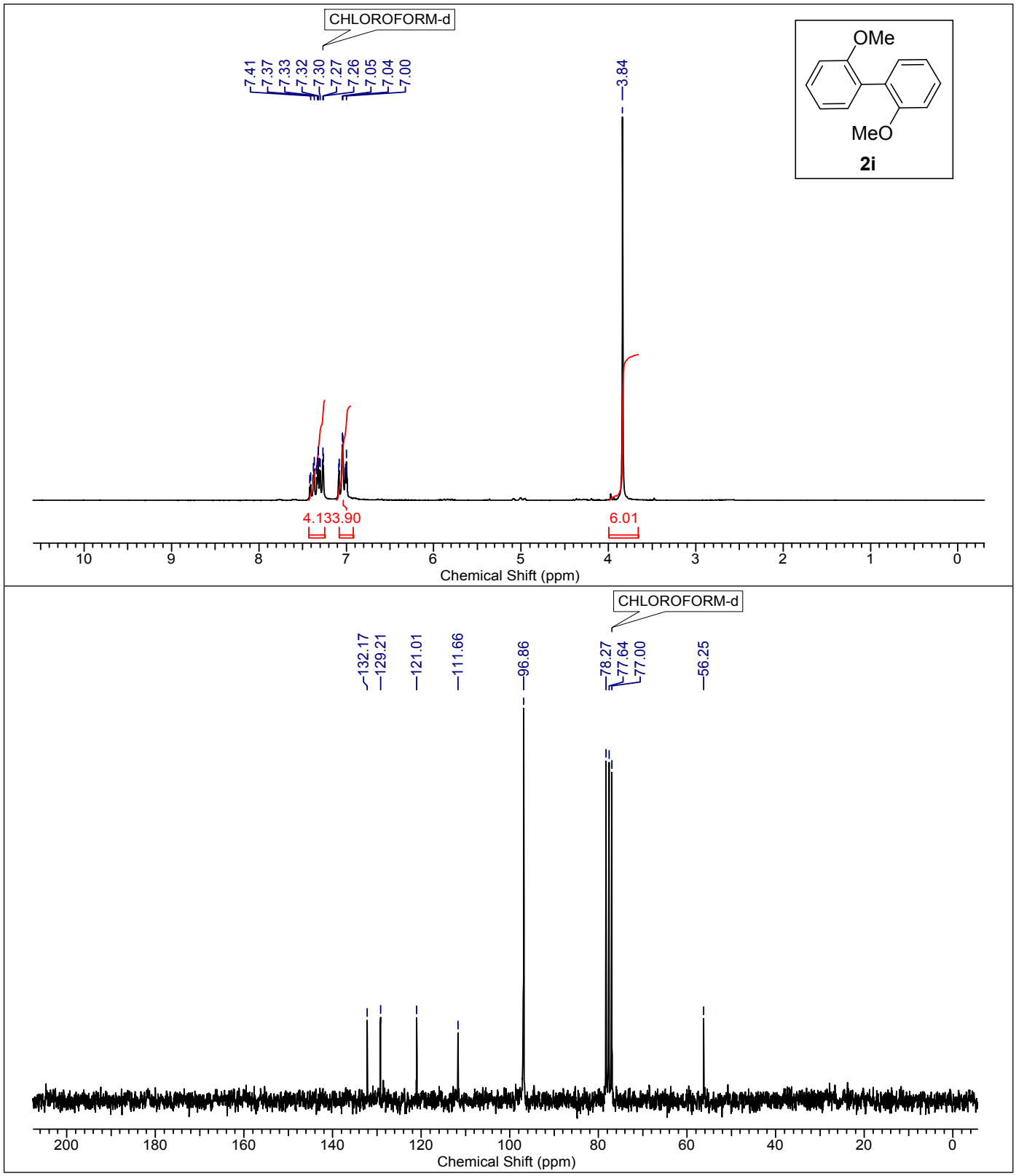

CHLOROFORM-d
7.32
7.30
7.30
7.29
7.27
7.24
6.85
6.84
6.83
6.83
Me
Me
Me
Me
Me
Me
2d
1.32
4.06
3.74
18.00
10
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)
CHLOROFORM-d
157.98
150.42
143.85
138.92
133.33
127.33
127.03
126.82
126.26
115.41
114.03
96.85
78.27
77.63
77.00
55.77
34.75
32.29
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

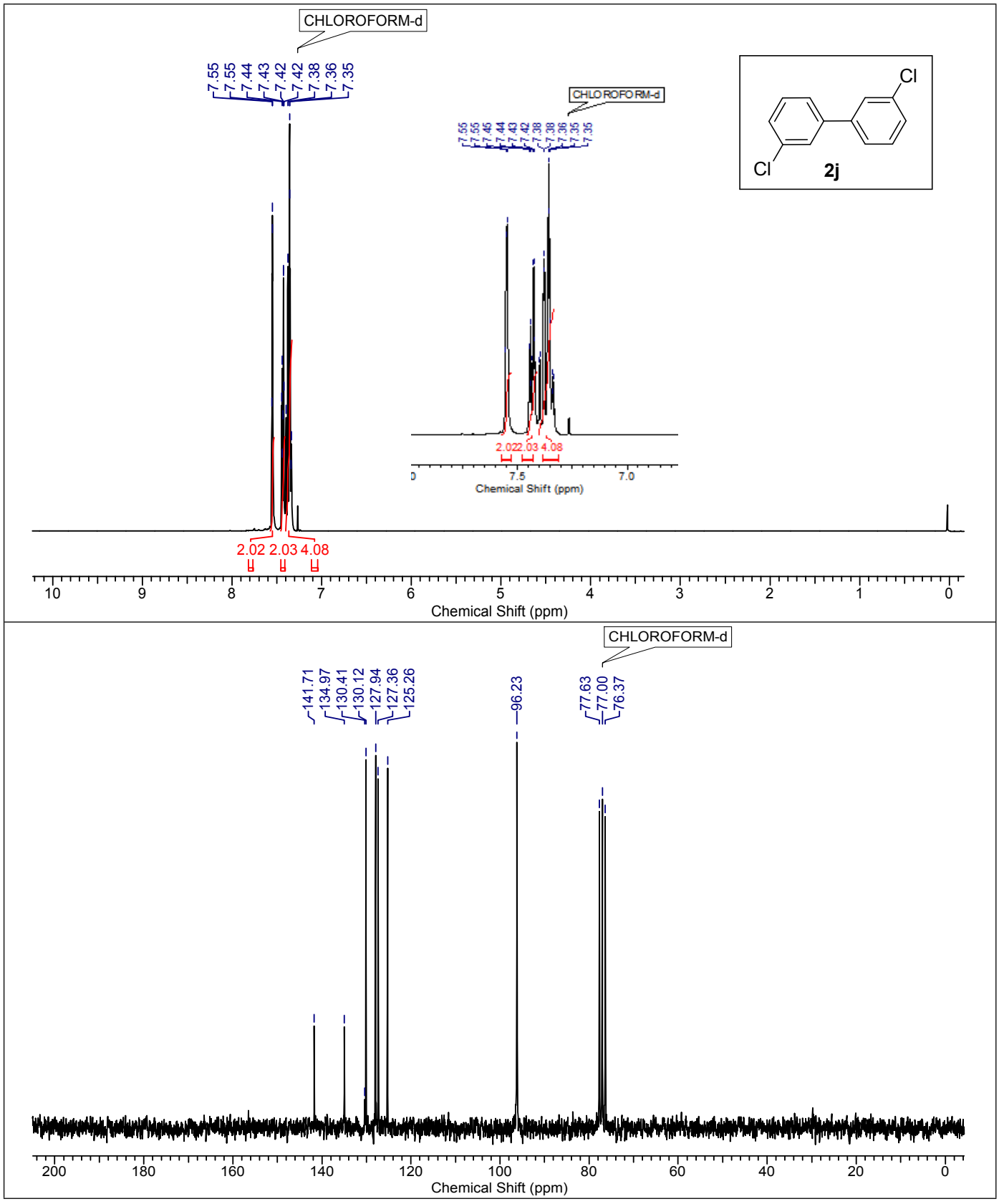

CHLOROFORM-d
7.50
7.49
7.48
7.47
7.47
7.27
7.14
7.12
7.10
F
F
2e
4.20
4.00
10
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)
163.41
160.96
136.19
128.33
128.25
115.54
115.34
95.92
77.00
76.68
76.36
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

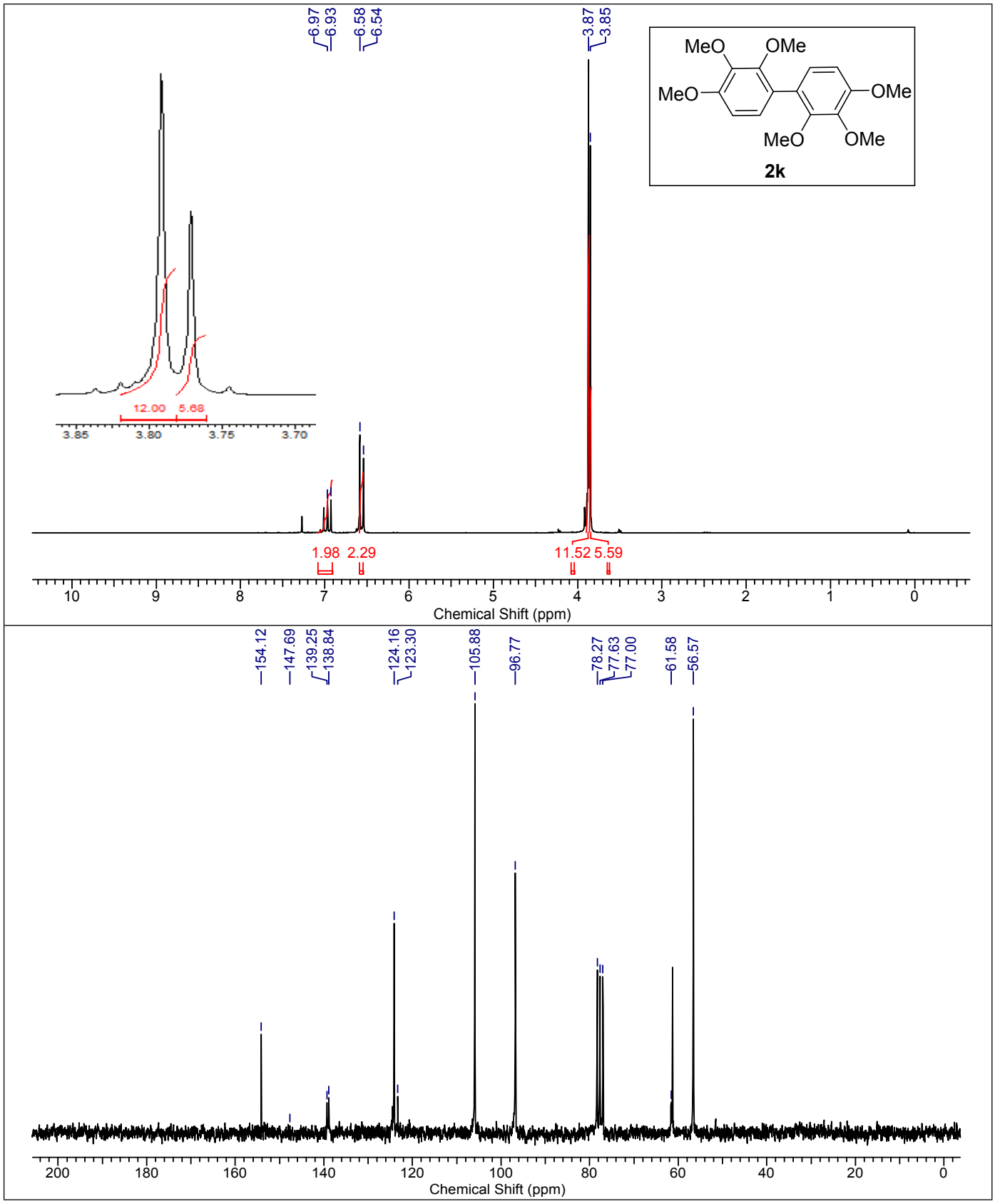

CHLOROFORM-d
7.59
7.55
7.38
7.34
7.27
Cl
Cl
2f
4.00
4.11
10
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)
CHLOROFORM-d
140.96
132.44
129.99
127.22
96.87
78.27
77.63
77.00
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

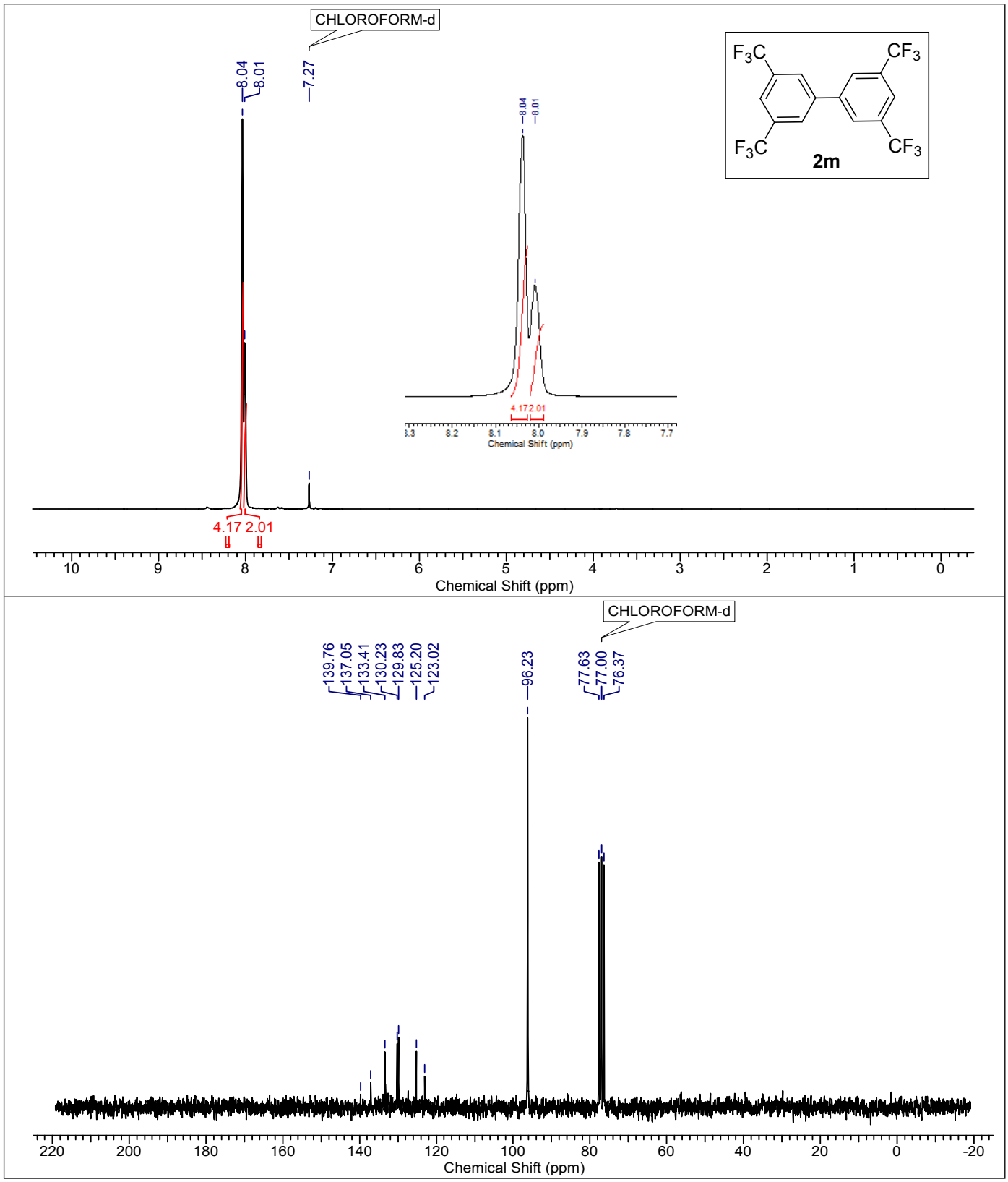

10.09
8.03
7.99
7.82
7.78
OHC
CHO
2g
CHLOROFORM-d
1.84
3.69
4.00
11
10
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)
190.42
145.22
140.96
140.27
138.69
132.52
131.76
129.30
96.18
77.58
76.94
76.31
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

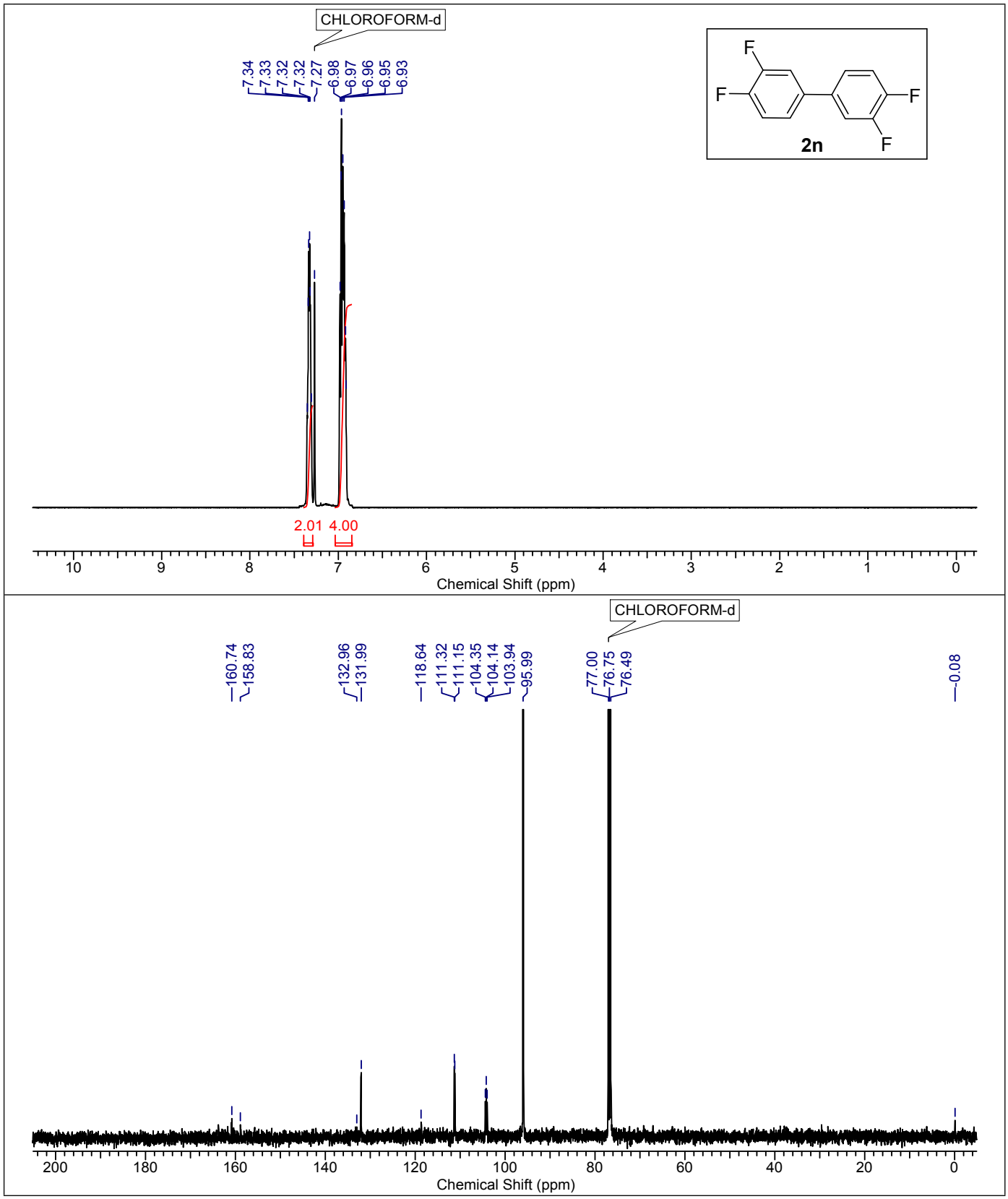

CHLOROFORM-d
7.58
7.56
7.56
7.48
7.46
7.27
6.79
6.76
6.74
6.72
5.81
5.81
5.77
5.29
5.26
2h
4.10
4.06
2.22
2.11
2.06
Chemical Shift (ppm)
CHLOROFORM-d
140.37
136.55
131.81
130.49
128.24
127.07
126.72
113.89
96.25
77.64
77.00
76.37
Chemical Shift (ppm)

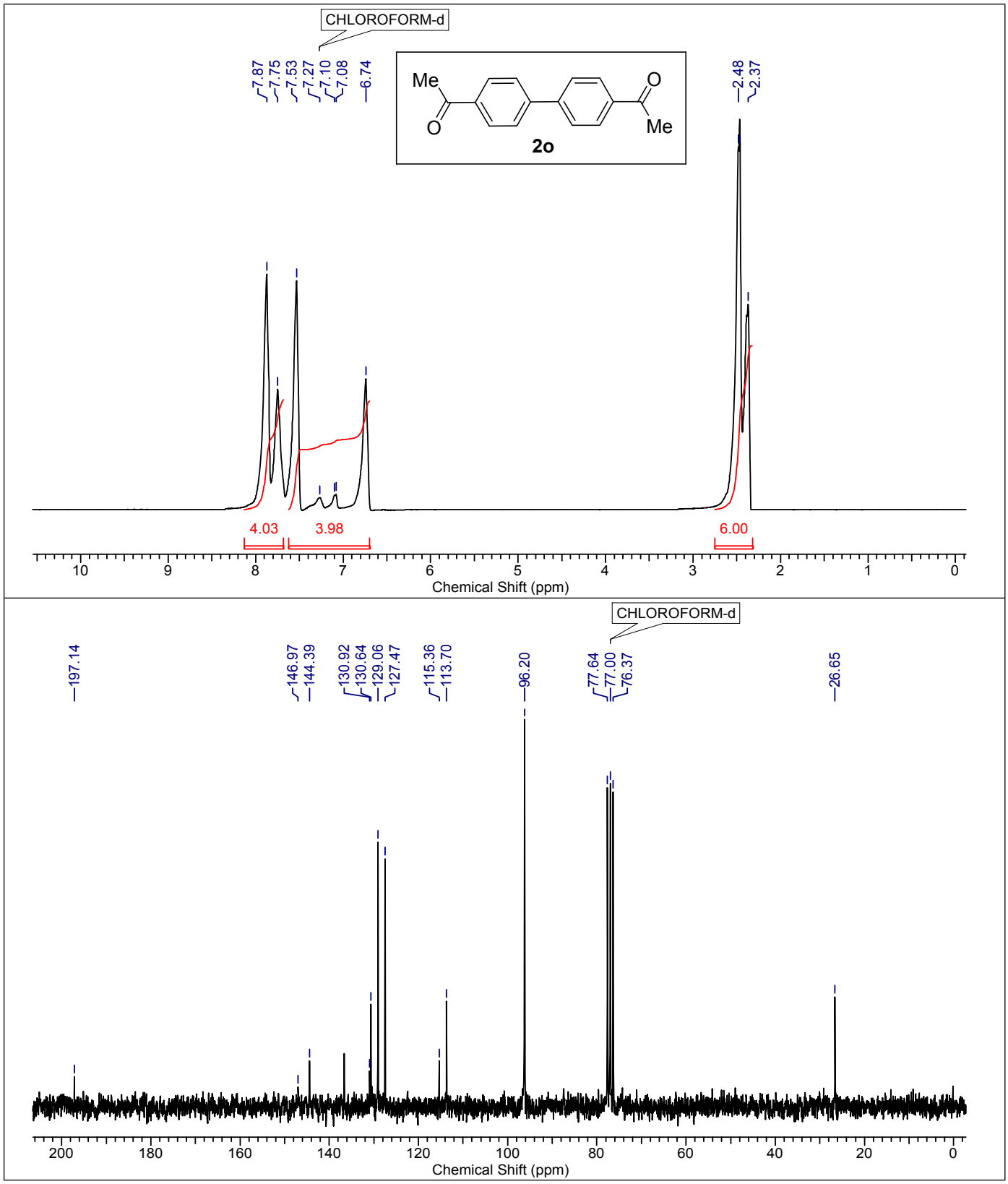

CHLOROFORM-d
7.41
7.37
7.33
7.32
7.30
7.27
7.26
7.05
7.04
7.00
3.84
OMe
MeO
2i
4.13
3.90
6.01
10
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)
CHLOROFORM-d
132.17
129.21
121.01
111.66
96.86
78.27
77.64
77.00
56.25
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

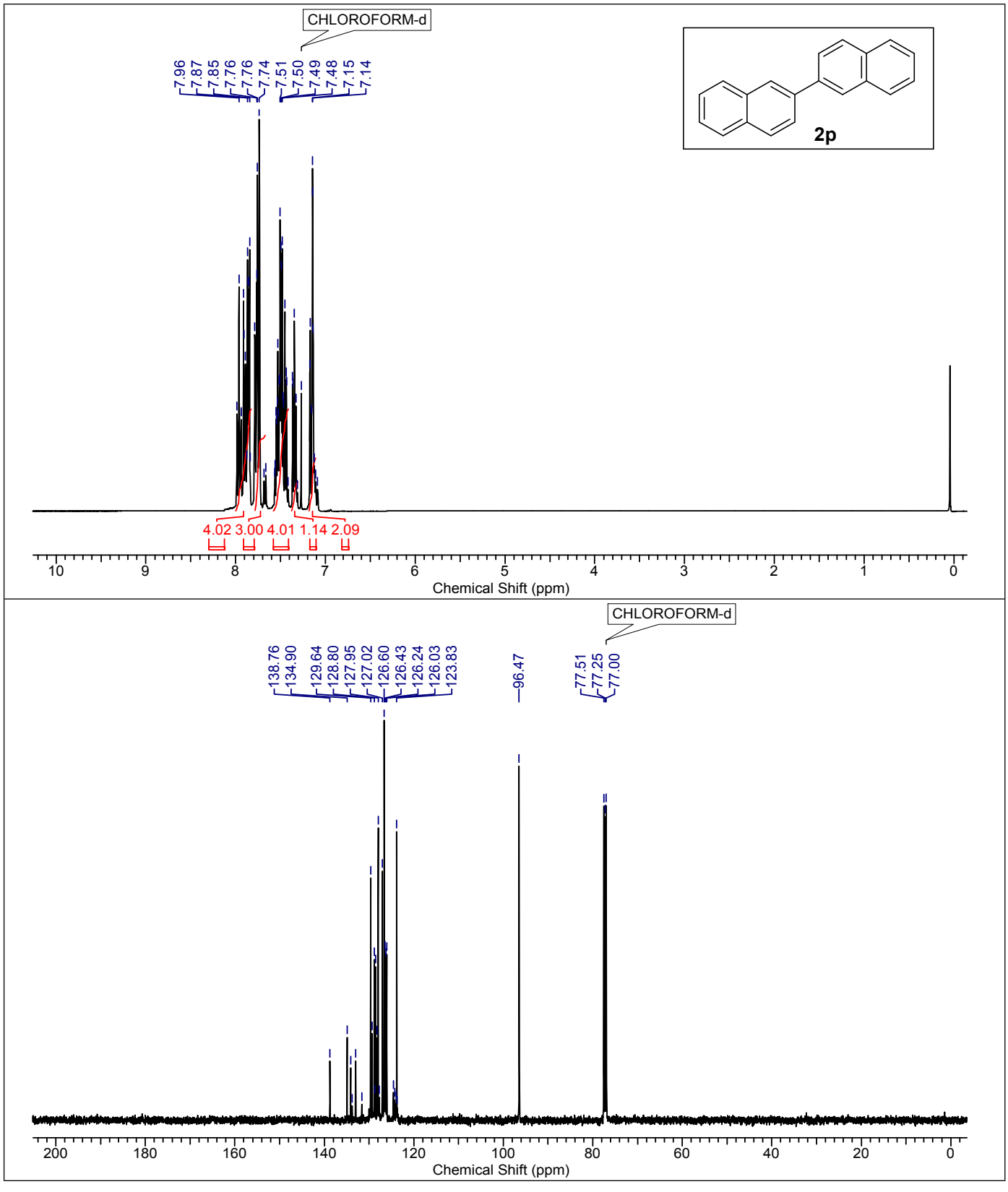

CHLOROFORM-d
7.55
7.55
7.44
7.43
7.42
7.42
7.38
7.36
7.35
2.02 2.03 4.08
10
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)
CHLOROFORM-d
2.02 2.03 4.08
7.5
7.0
Chemical Shift (ppm)
Cl
Cl
2j
CHLOROFORM-d
141.71
134.97
130.41
130.12
127.94
127.36
125.26
96.23
77.63
77.00
76.37
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

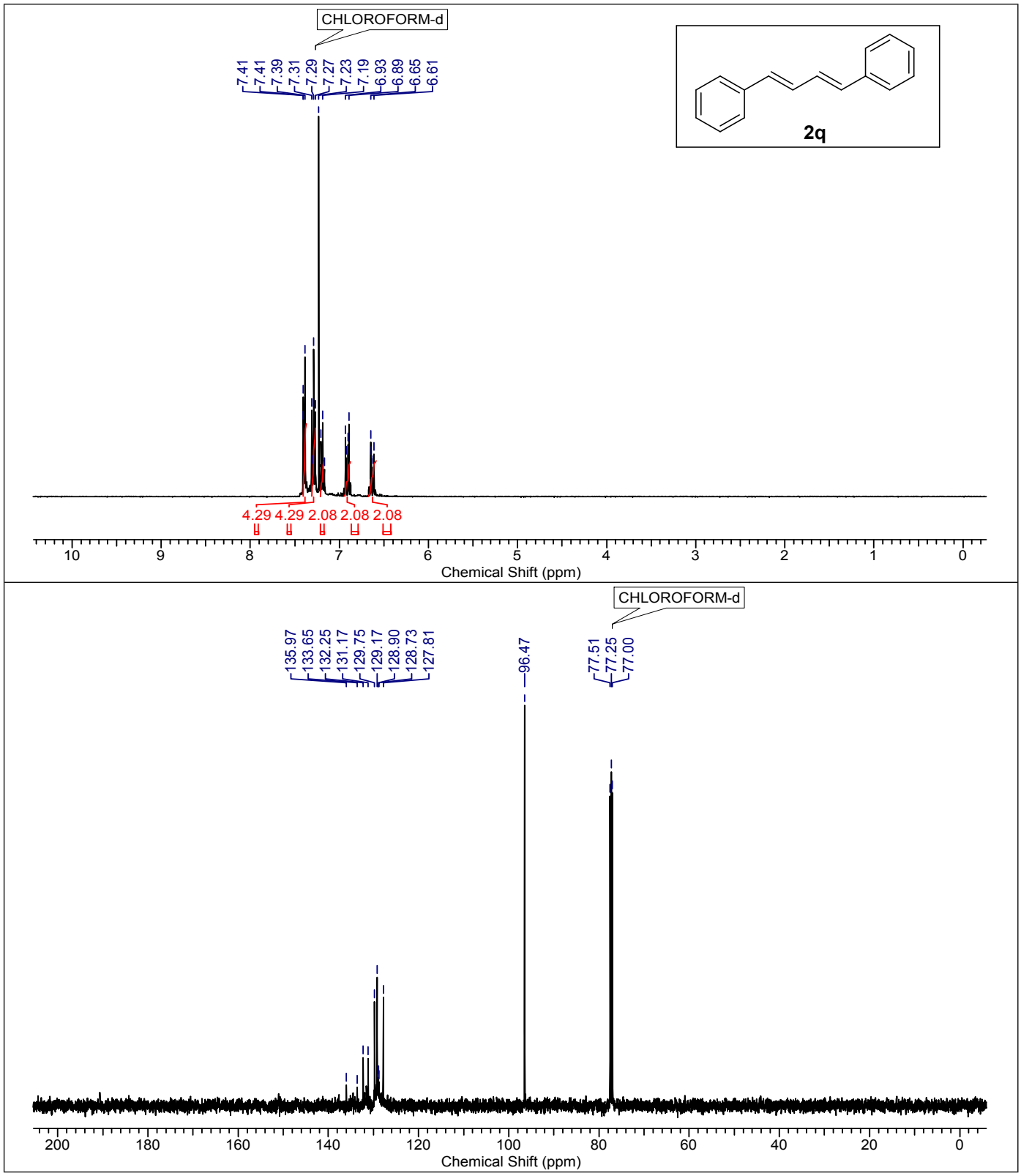

6.97
6.93
6.58
6.54
3.87
3.85
MeO
OMe
MeO
OMe
MeO
OMe
2k
12.00
5.68
3.85
3.80
3.75
3.70
1.98
2.29
11.52
5.59
Chemical Shift (ppm)
154.12
147.69
139.25
138.84
124.16
123.30
105.88
96.77
78.27
77.63
77.00
61.58
56.57
Chemical Shift (ppm)

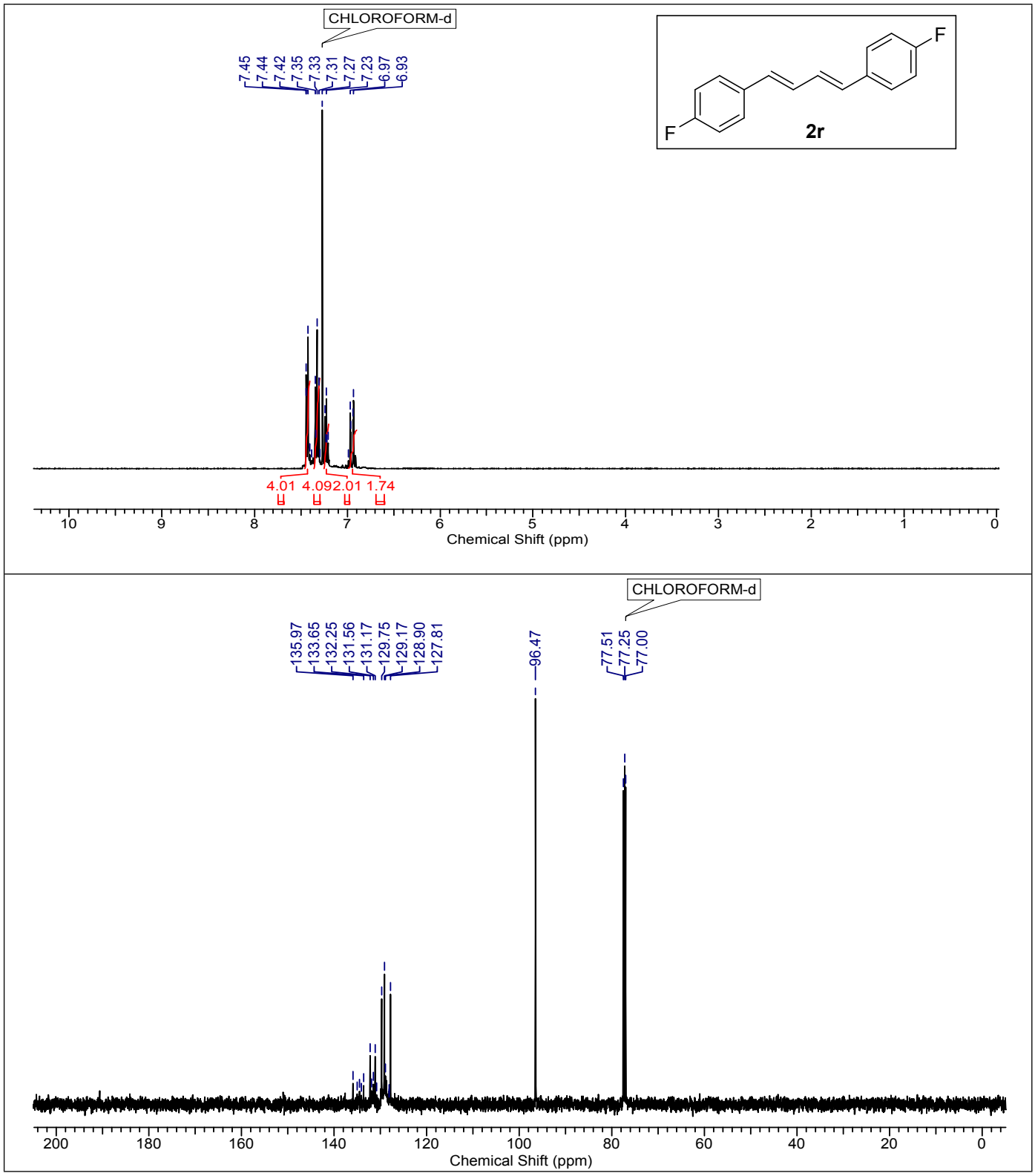

CHLOROFORM-d
8.04
8.01
7.27
4.17
2.01
Chemical Shift (ppm)
F3C
CF3
F3C
CF3
2m
CHLOROFORM-d
139.76
137.05
133.41
130.23
129.83
125.20
123.02
96.23
77.63
77.00
76.37
Chemical Shift (ppm)

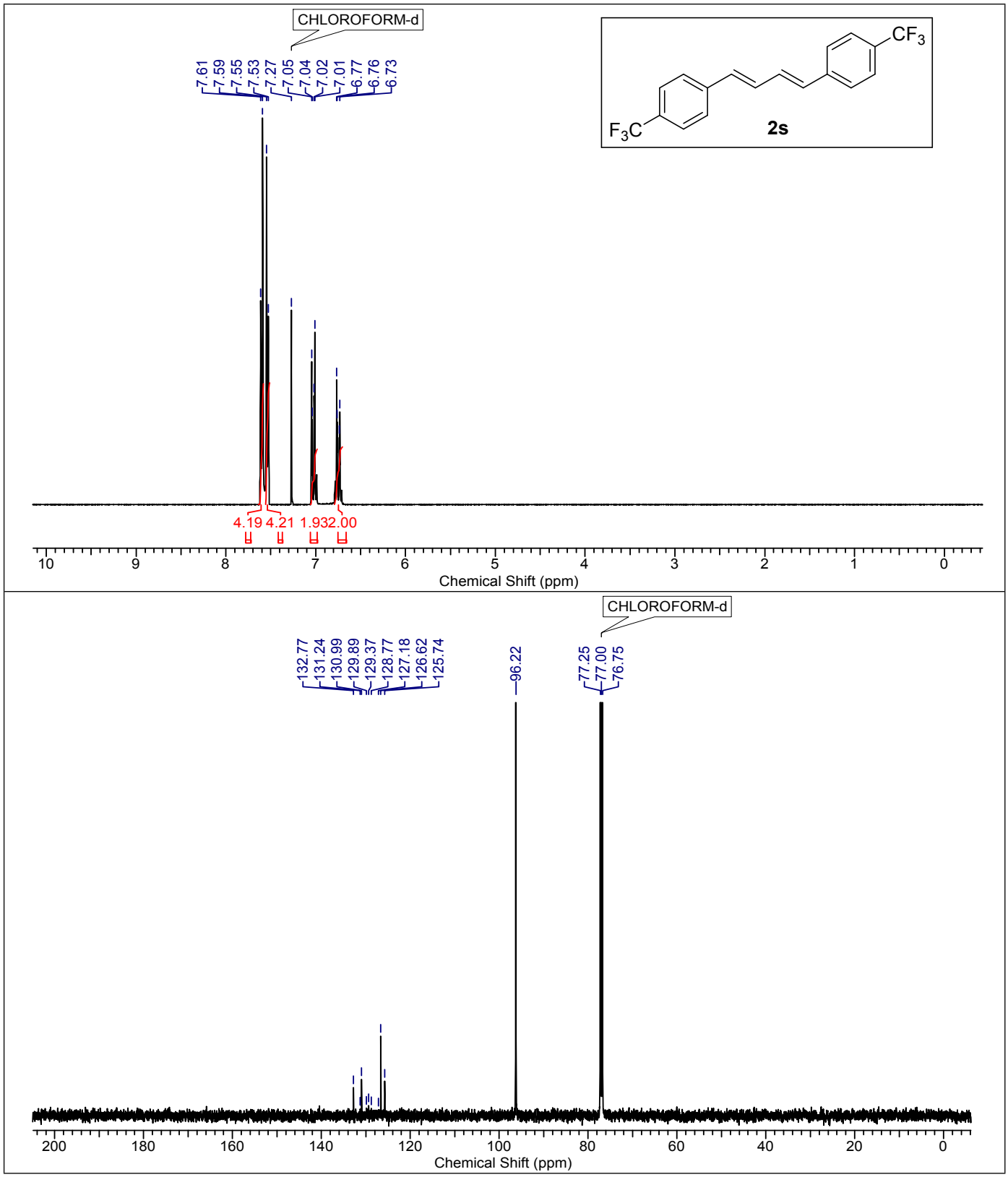

CHLOROFORM-d
7.34
7.33
7.32
7.32
7.27
6.98
6.97
6.96
6.95
6.93
F
F
F
F
2n
2.01
4.00
10
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)
CHLOROFORM-d
160.74
158.83
132.96
131.99
118.64
111.32
111.15
104.35
104.14
103.94
95.99
77.00
76.75
76.49
-0.08
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

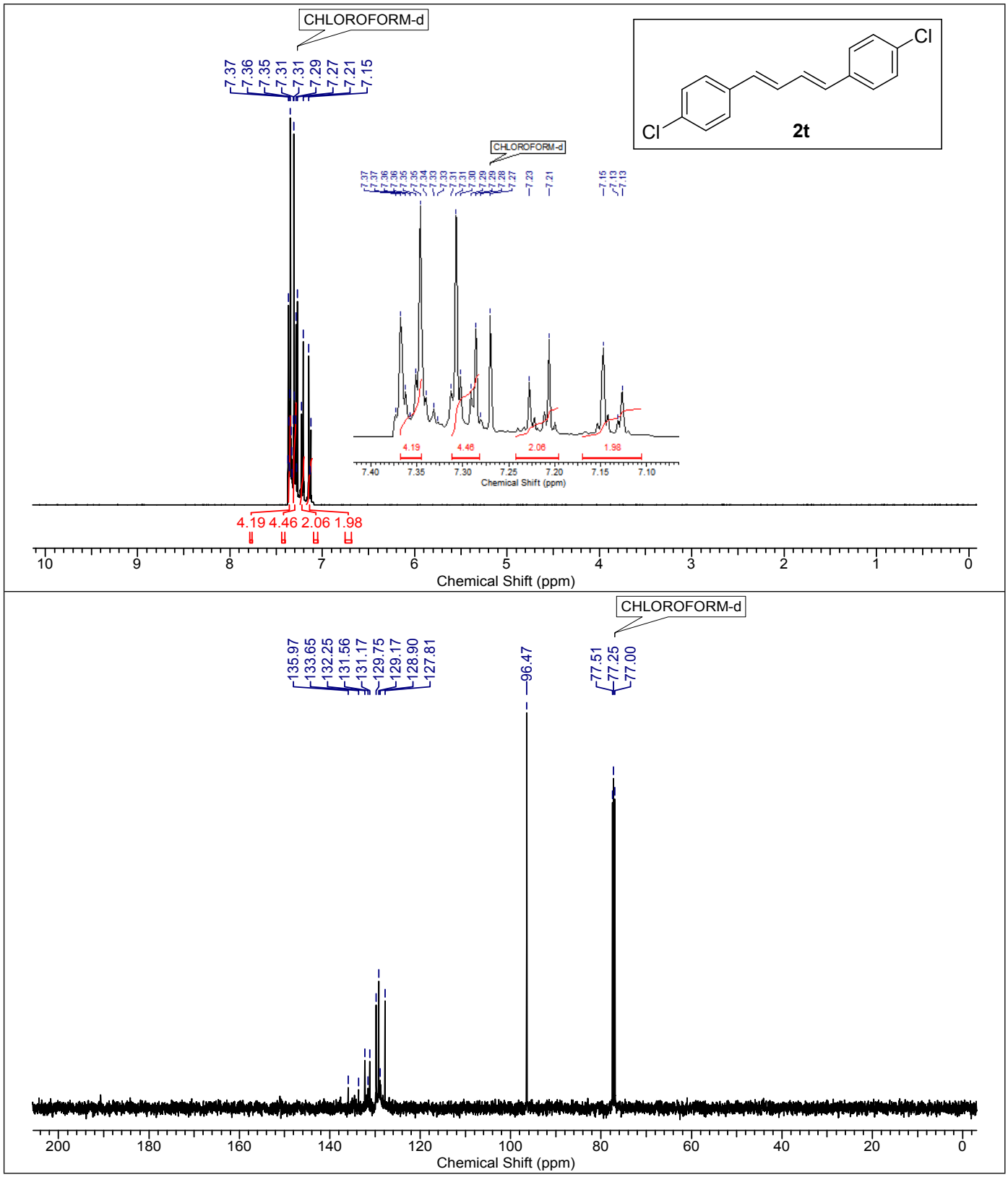

CHLOROFORM-d
7.87
7.75
7.53
7.27
7.10
7.08
6.74
2.48
2.37
Me
O
O
Me
2o
4.03
3.98
6.00
Chemical Shift (ppm)
CHLOROFORM-d
197.14
146.97
144.39
130.92
130.64
129.06
127.47
115.36
113.70
96.20
77.64
77.00
76.37
26.65
Chemical Shift (ppm)

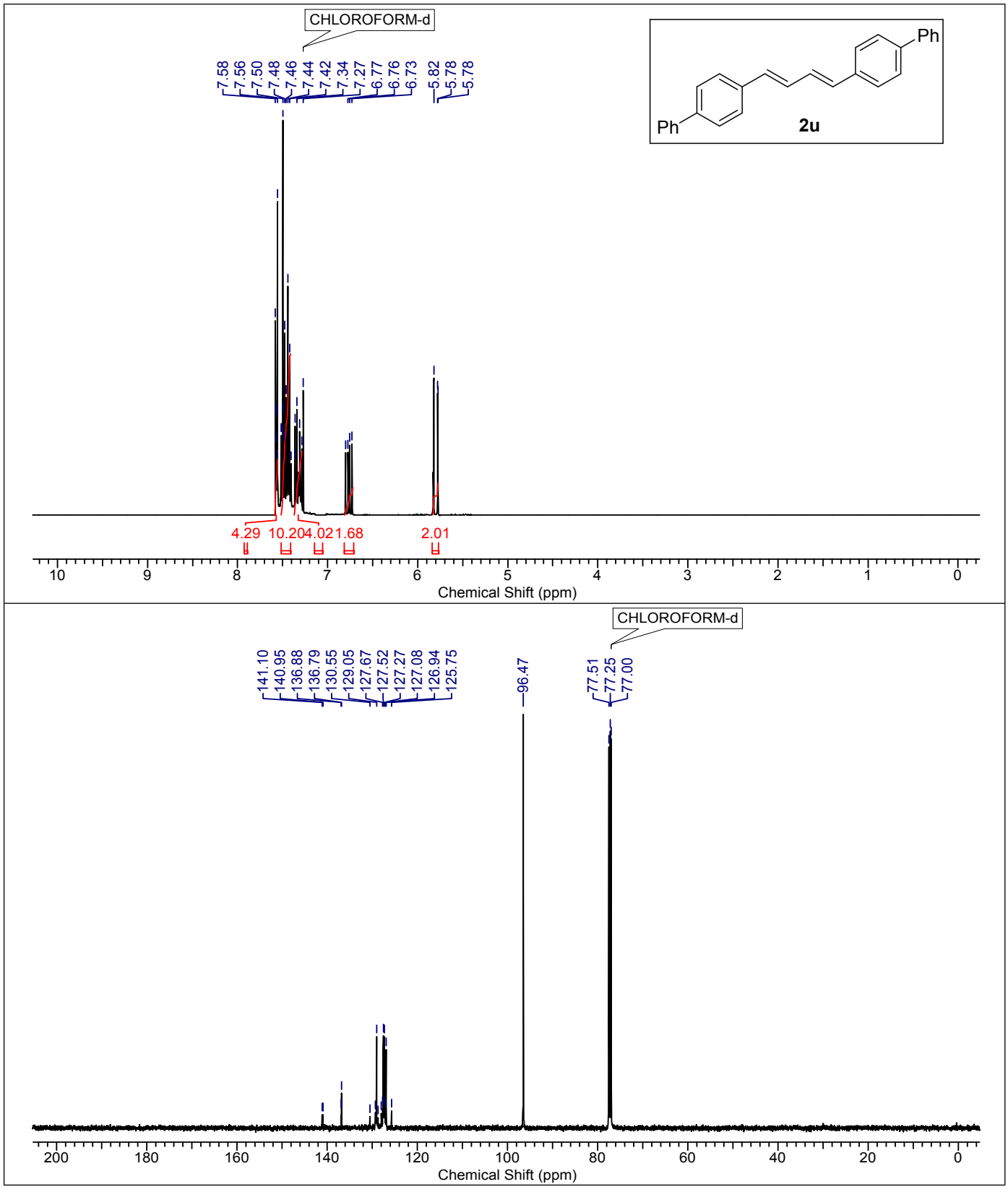

CHLOROFORM-d
7.96
7.87
7.85
7.76
7.76
7.74
7.51
7.50
7.49
7.48
7.15
7.14
2p
4.02 3.00 4.01 1.14 2.09
10 9 8 7 6 5 4 3 2 1 0
Chemical Shift (ppm)
CHLOROFORM-d
138.76
134.90
129.64
128.80
127.95
127.02
126.60
126.43
126.24
126.03
123.83
96.47
77.51
77.25
77.00
200 180 160 140 120 100 80 60 40 20 0
Chemical Shift (ppm)

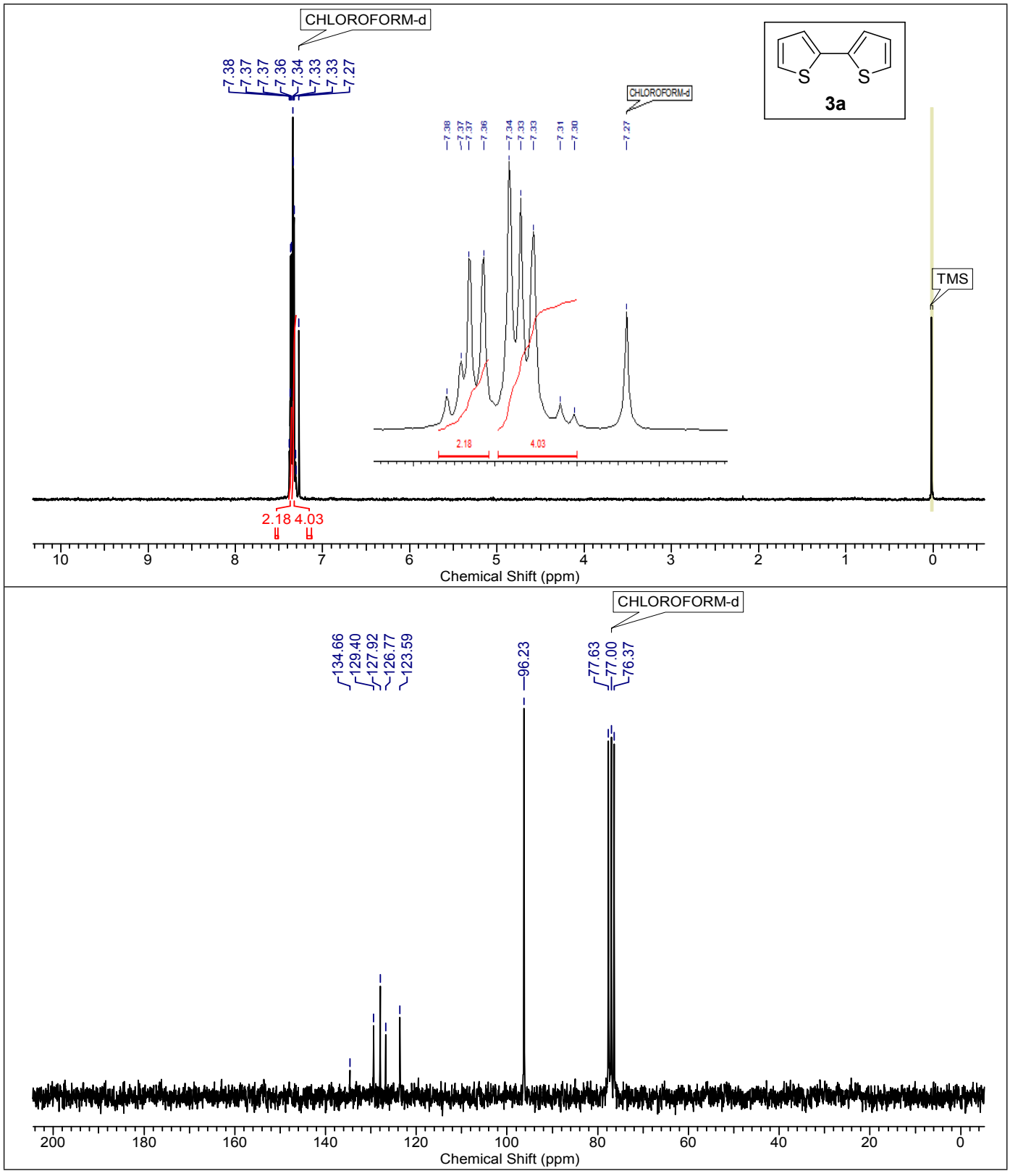

CHLOROFORM-d
7.41
7.41
7.39
7.31
7.29
7.27
7.23
7.19
6.93
6.89
6.65
6.61
2q
4.29 4.29 2.08 2.08 2.08
10 9 8 7 6 5 4 3 2 1 0
Chemical Shift (ppm)
CHLOROFORM-d
135.97
133.65
132.25
131.17
129.75
129.17
128.90
128.73
127.81
96.47
77.51
77.25
77.00
200 180 160 140 120 100 80 60 40 20 0
Chemical Shift (ppm)

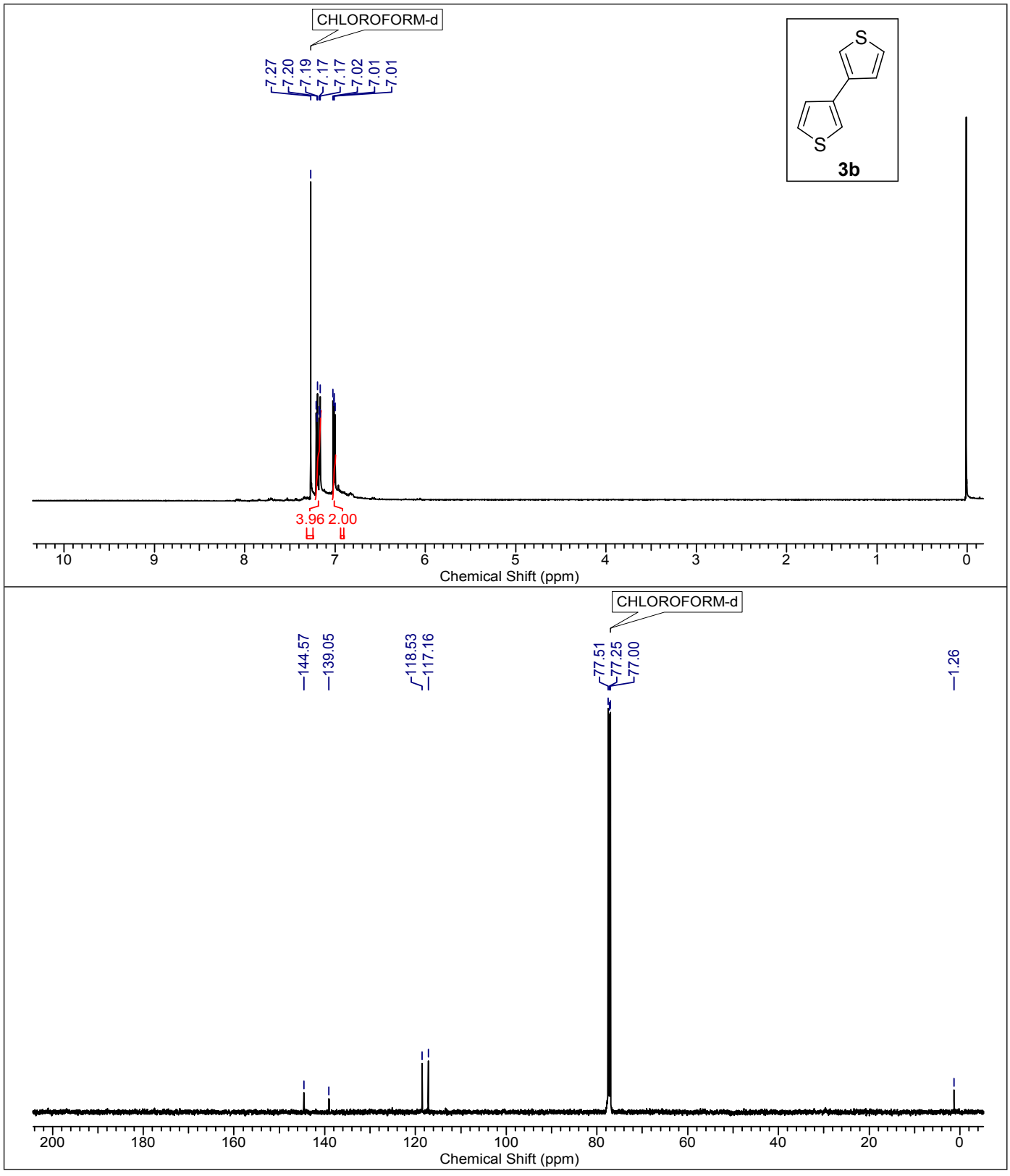

CHLOROFORM-d
7.45
7.44
7.42
7.35
7.33
7.31
7.27
7.23
6.97
6.93
F
F
2r
4.01
4.09
2.01
1.74
Chemical Shift (ppm)
CHLOROFORM-d
135.97
133.65
132.25
131.56
131.17
129.75
129.17
128.90
127.81
96.47
77.51
77.25
77.00
Chemical Shift (ppm)

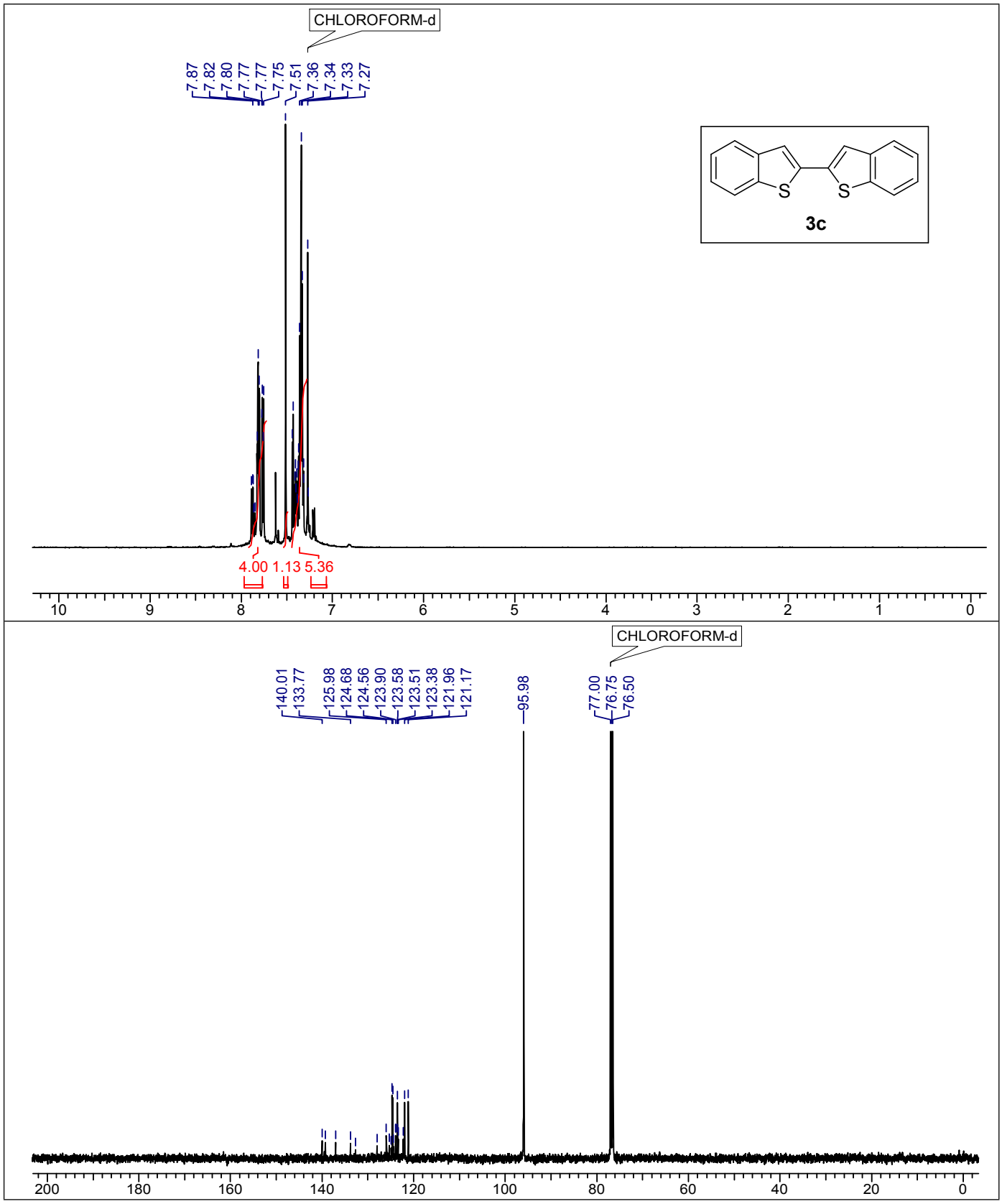

CHLOROFORM-d
7.61
7.59
7.55
7.53
7.27
7.05
7.04
7.02
7.01
6.77
6.76
6.73
CF3
F3C
2s
4.19 4.21 1.93 2.00
10 9 8 7 6 5 4 3 2 1 0
Chemical Shift (ppm)
CHLOROFORM-d
132.77
131.24
130.99
129.89
129.37
128.77
127.18
126.62
125.74
96.22
77.25
77.00
76.75
200 180 160 140 120 100 80 60 40 20 0
Chemical Shift (ppm)

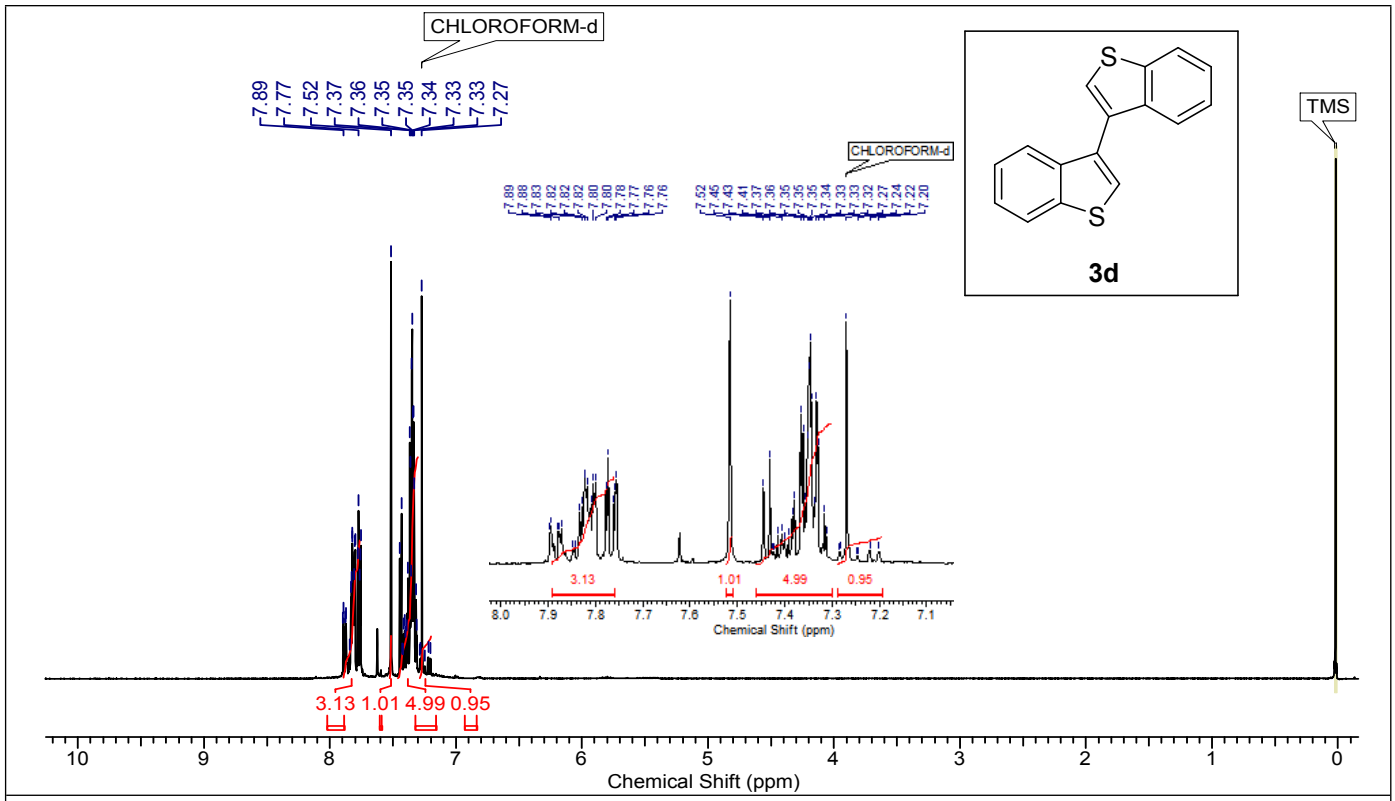

CHLOROFORM-d
7.37
7.36
7.35
7.31
7.31
7.29
7.27
7.21
7.15
Cl
Cl
2t
4.19 4.46 2.06 1.98
Chemical Shift (ppm)
CHLOROFORM-d
135.97
133.65
132.25
131.56
131.17
129.75
129.17
128.90
127.81
96.47
77.51
77.25
77.00
Chemical Shift (ppm)

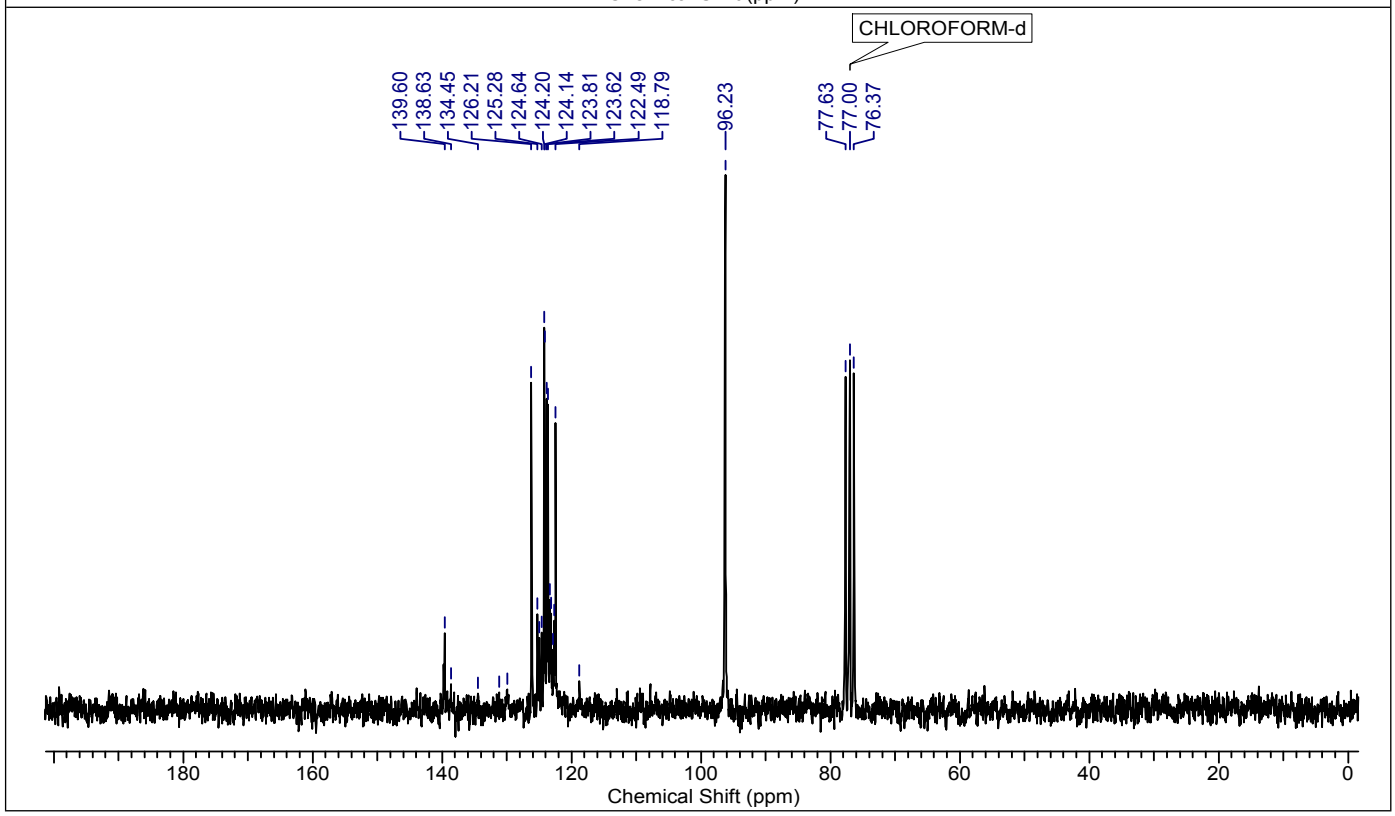

CHLOROFORM-d
7.58
7.56
7.50
7.48
7.46
7.44
7.42
7.34
7.27
6.77
6.76
6.73
5.82
5.78
5.78
Ph
Ph
2u
4.29
10.20
4.02
1.68
2.01
10
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)
CHLOROFORM-d
141.10
140.95
136.88
136.79
130.55
129.05
127.67
127.52
127.27
127.08
126.94
125.75
96.47
77.51
77.25
77.00
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

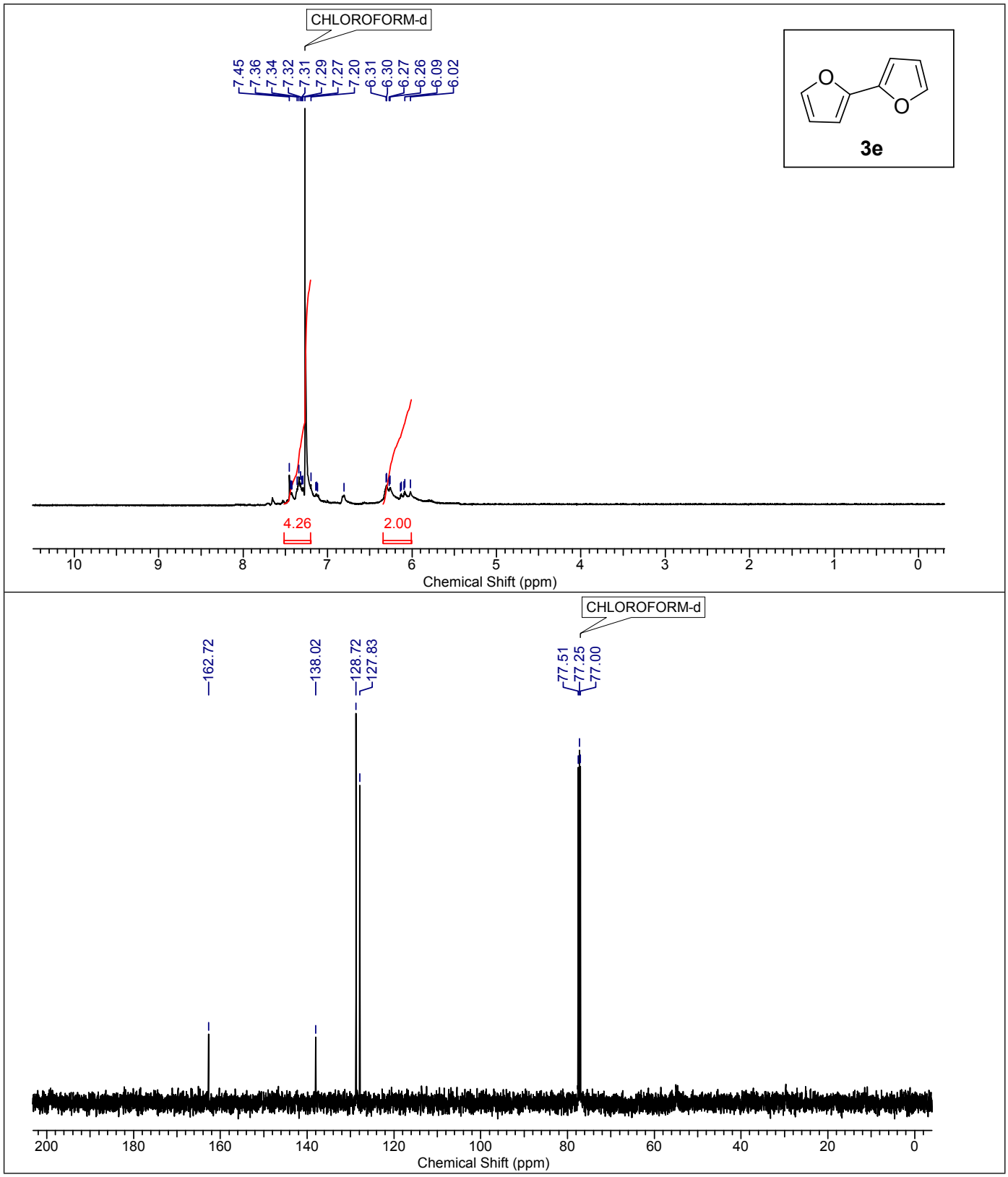

CHLOROFORM-d
7.38
7.37
7.37
7.36
7.34
7.33
7.33
7.27
S
S
3a
CHLOROFORM-d
7.38
7.37
7.37
7.36
7.34
7.33
7.33
7.31
7.30
7.27
2.18
4.03
TMS
2.18
4.03
10
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)
CHLOROFORM-d
134.66
129.40
127.92
126.77
123.59
96.23
77.63
77.00
76.37
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)


CHLOROFORM-d
7.27
7.20
7.19
7.17
7.17
7.02
7.01
7.01
S
S
3b
3.96
2.00
10
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)
CHLOROFORM-d
144.57
139.05
118.53
117.16
77.51
77.25
77.00
1.26
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)


CHLOROFORM-d
7.87
7.82
7.80
7.77
7.77
7.75
7.51
7.36
7.34
7.33
7.27
S
S
3c
4.00
1.13
5.36
10
9
8
7
6
5
4
3
2
1
0
CHLOROFORM-d
140.01
133.77
125.98
124.68
124.56
123.90
123.58
123.51
123.38
121.96
121.17
95.98
77.00
76.75
76.50
200
180
160
140
120
100
80
60
40
20
0


CHLOROFORM-d
7.89
7.77
7.52
7.37
7.36
7.35
7.35
7.34
7.33
7.33
7.27
TMS
3d
3.13 1.01 4.99 0.95
Chemical Shift (ppm)


CHLOROFORM-d
139.60
138.63
134.45
126.21
125.28
124.64
124.20
124.14
123.81
123.62
122.49
118.79
96.23
77.63
77.00
76.37
Chemical Shift (ppm)


CHLOROFORM-d
7.45
7.36
7.34
7.32
7.31
7.29
7.27
7.20
6.31
6.30
6.27
6.26
6.09
6.02
O
O
3e
4.26
2.00
10
9
8
7
6
5
4
3
2
1
0
Chemical Shift (ppm)
CHLOROFORM-d
162.72
138.02
128.72
127.83
77.51
77.25
77.00
200
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)