$PhI(OCOCF_3)_2$-Mediated Cyclization of *o*-(1-Alkynyl)benzamides: Metal-free Synthesis of 3-Hydroxy-2,3-dihydroisoquinoline-1,4-dione

Chao Yang,[†] Xiang Zhang,[†] Daisy Zhang-Negrerie,[†] and Yunfei Du*[,†,‡] and Kang Zhao*[,†]

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Supporting Information

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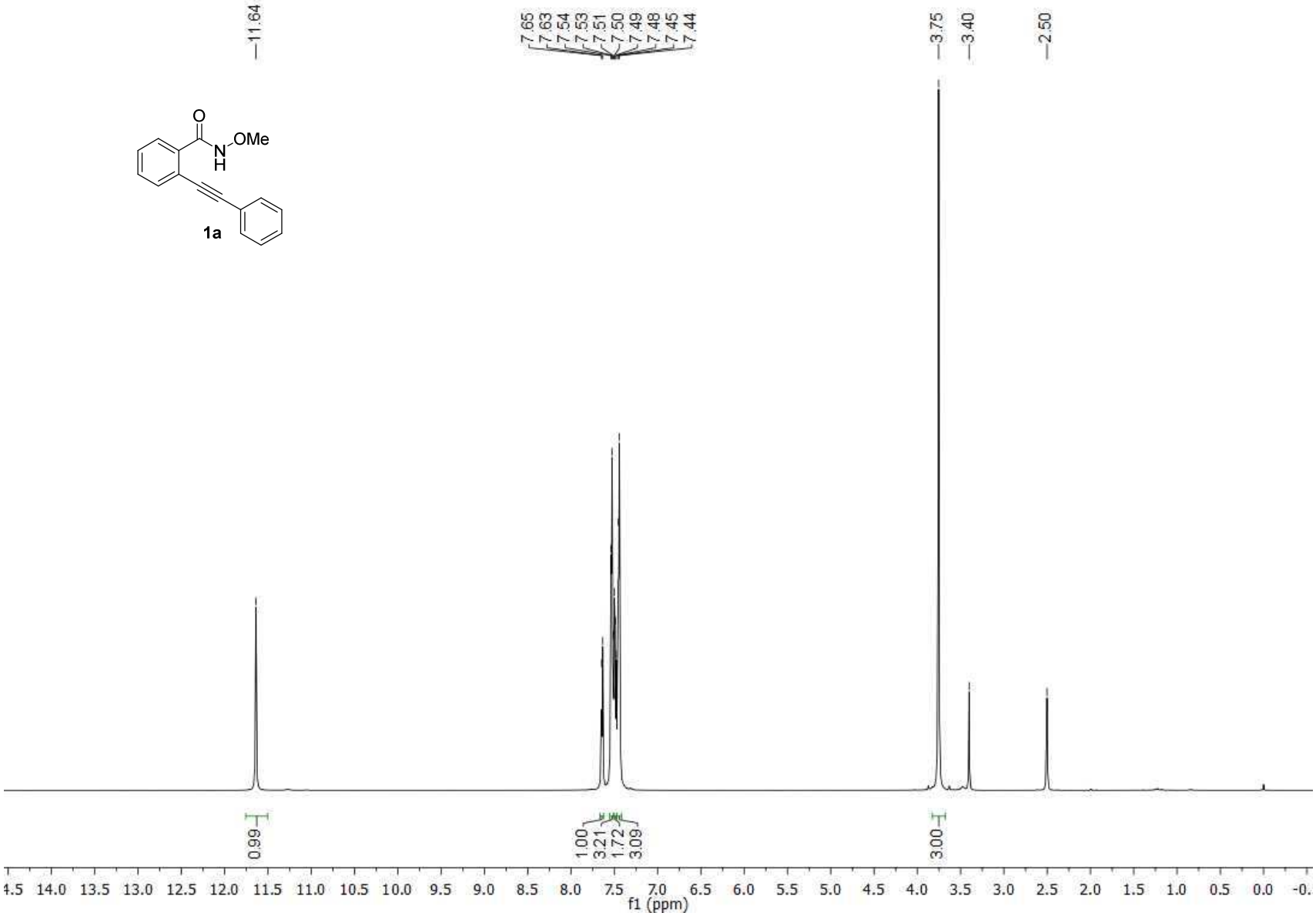
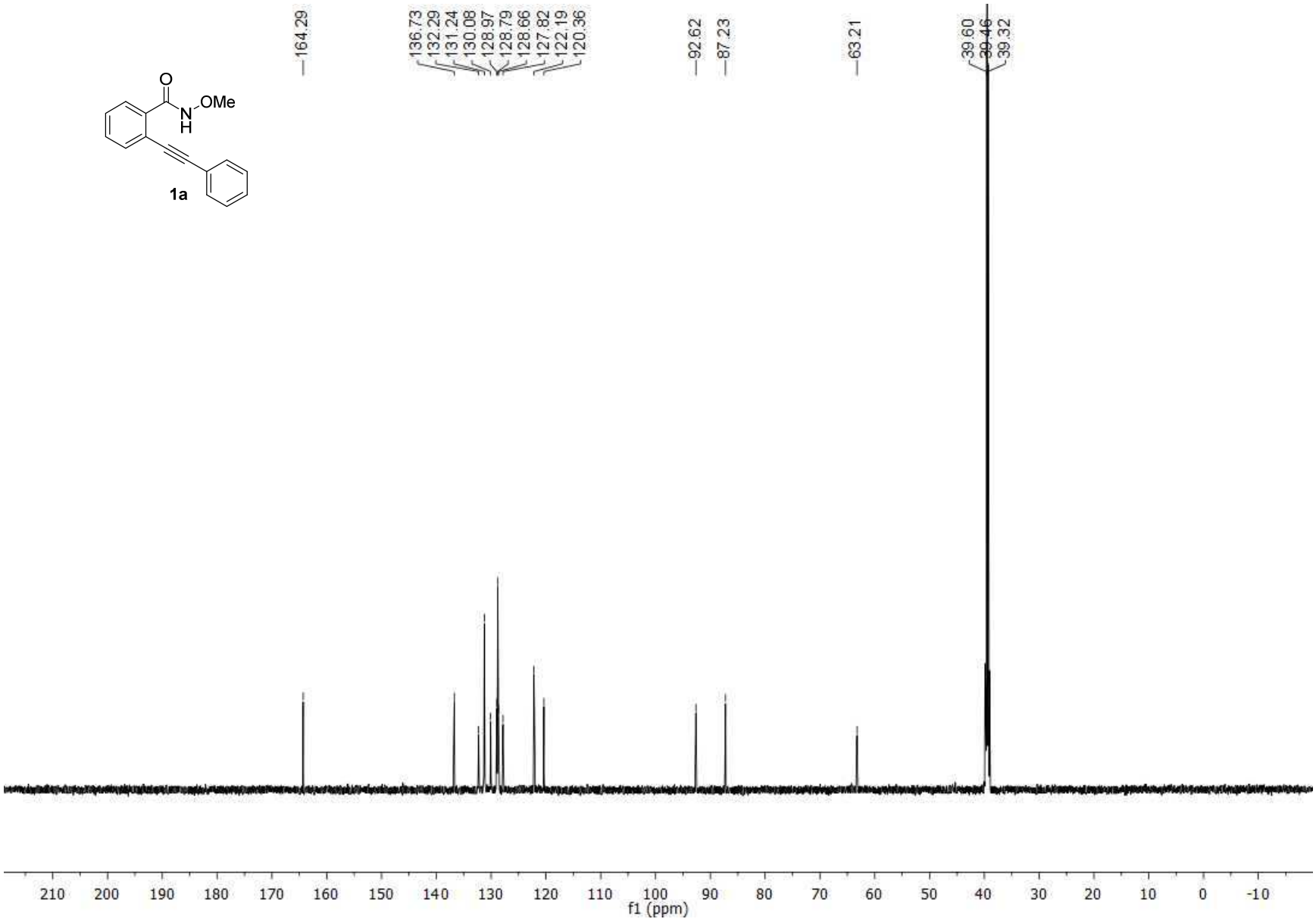
^{1}H and ^{13}C NMR Spectra

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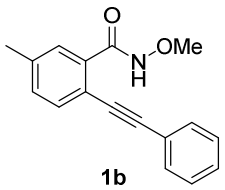
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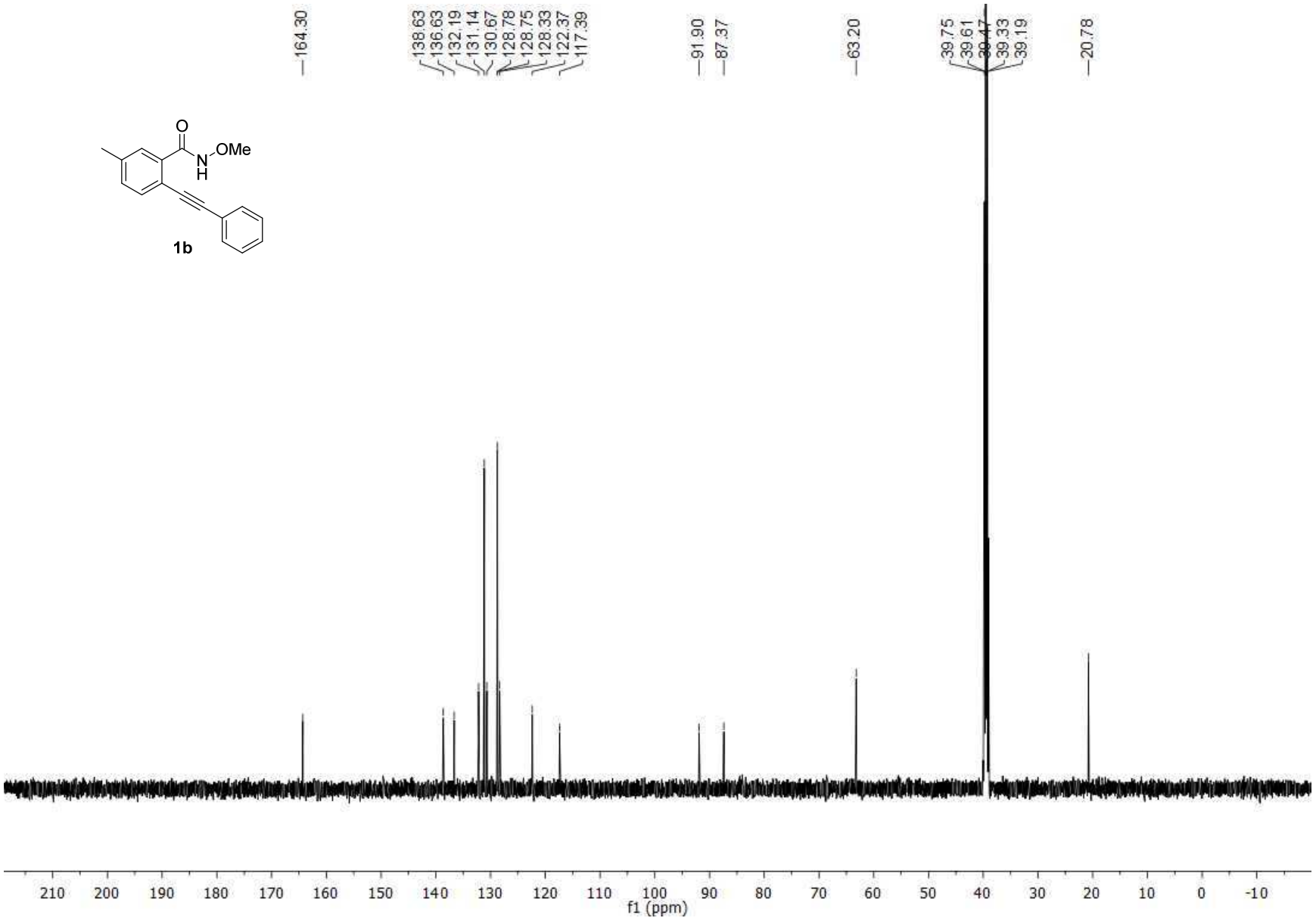
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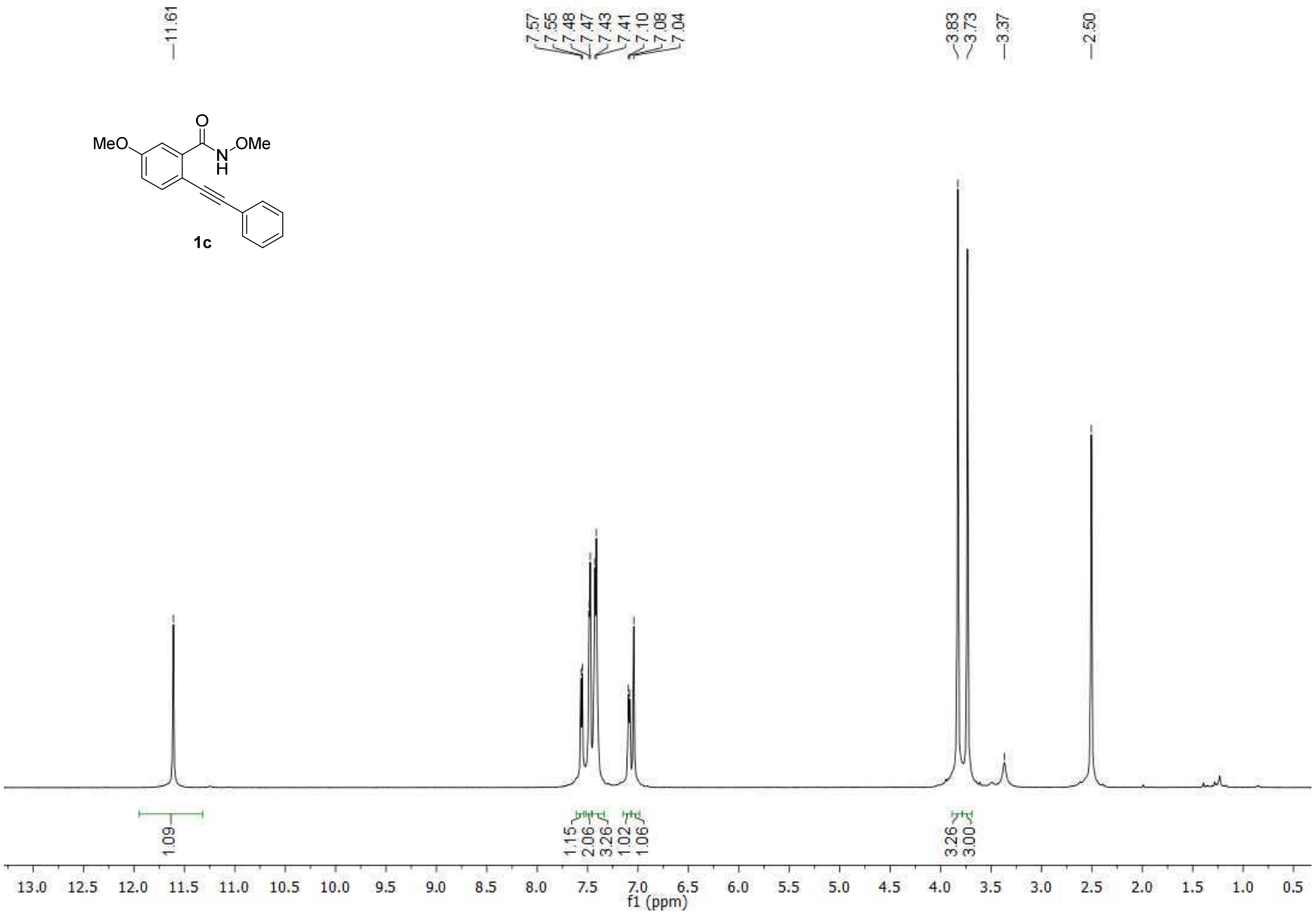
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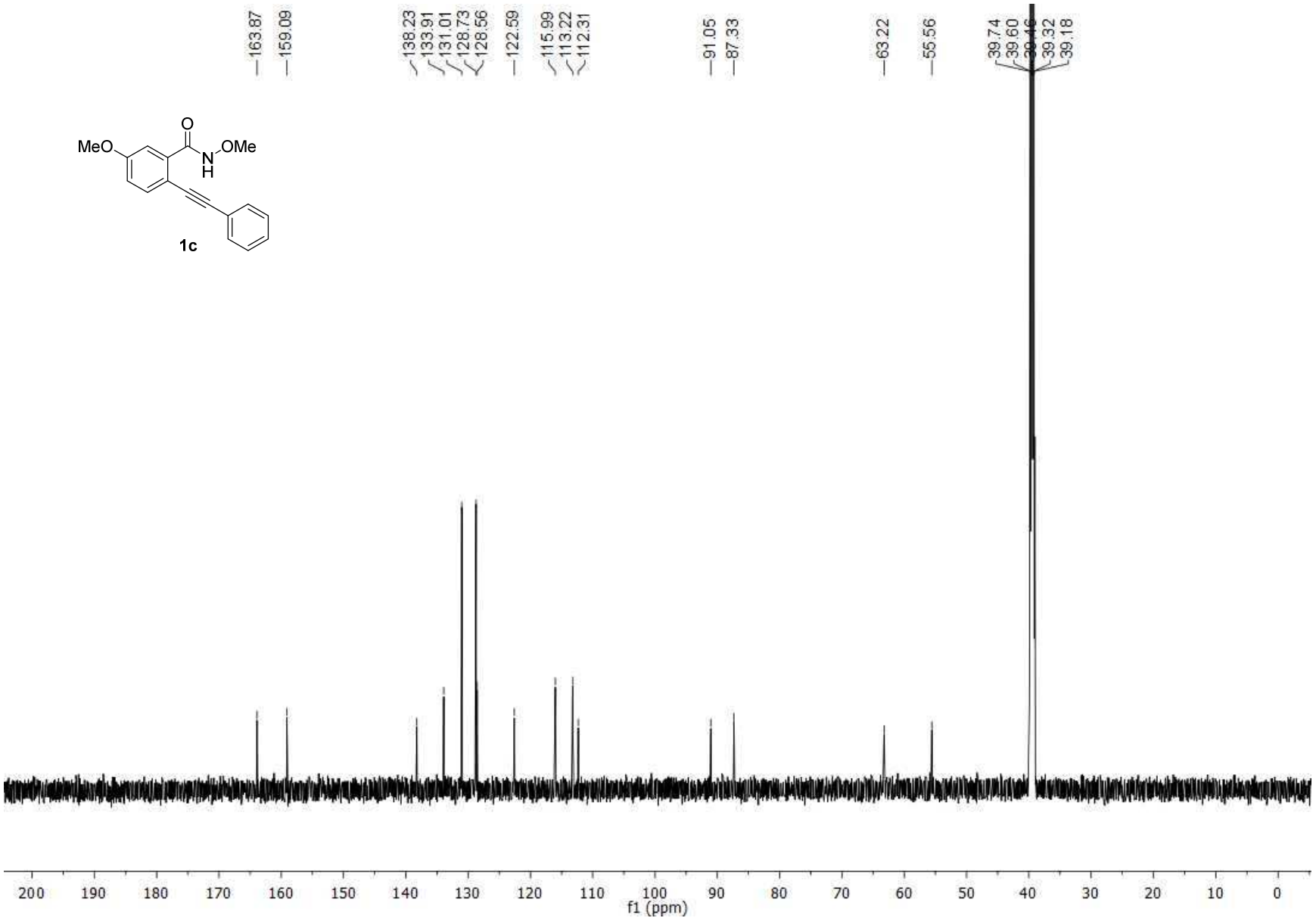
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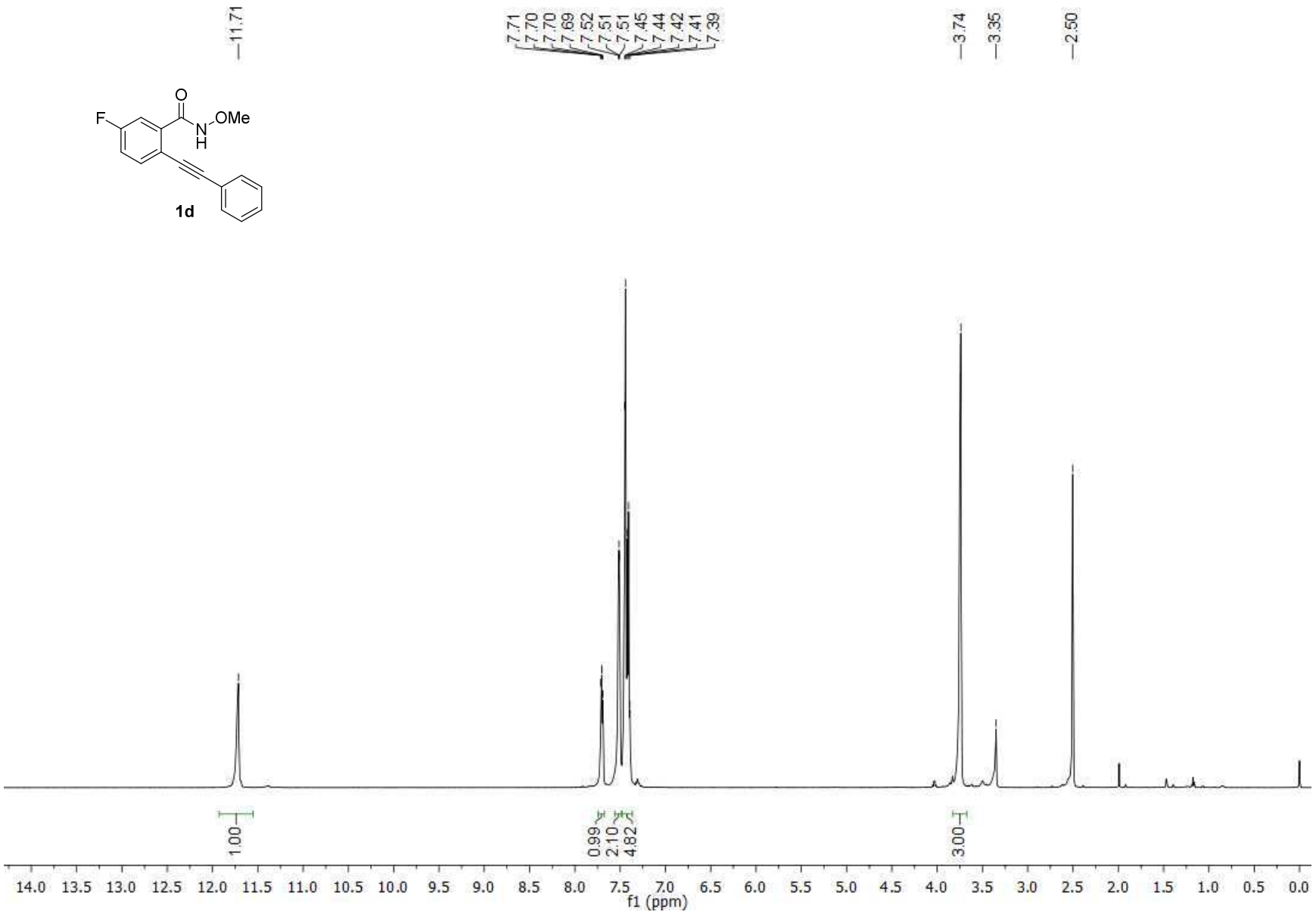
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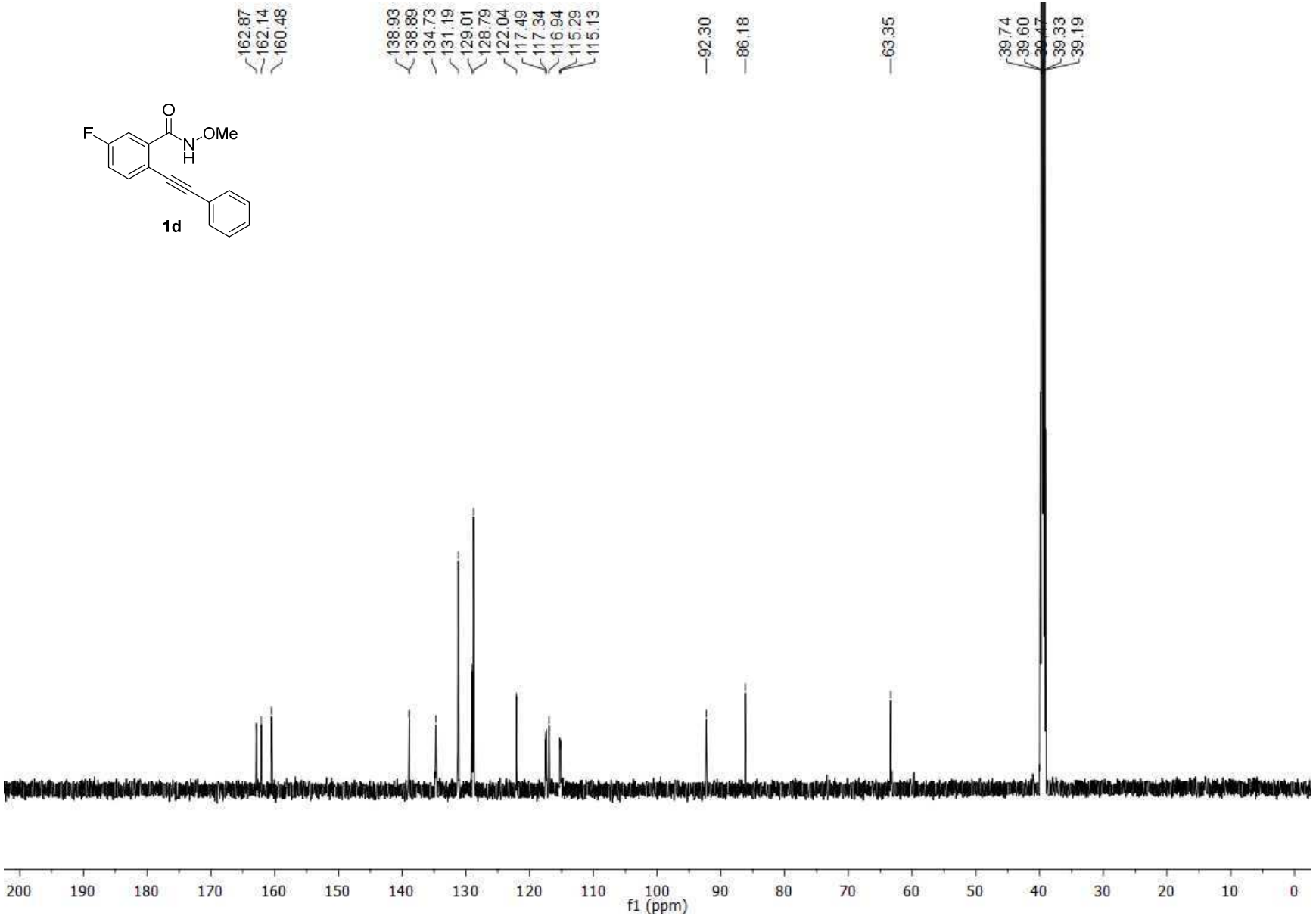
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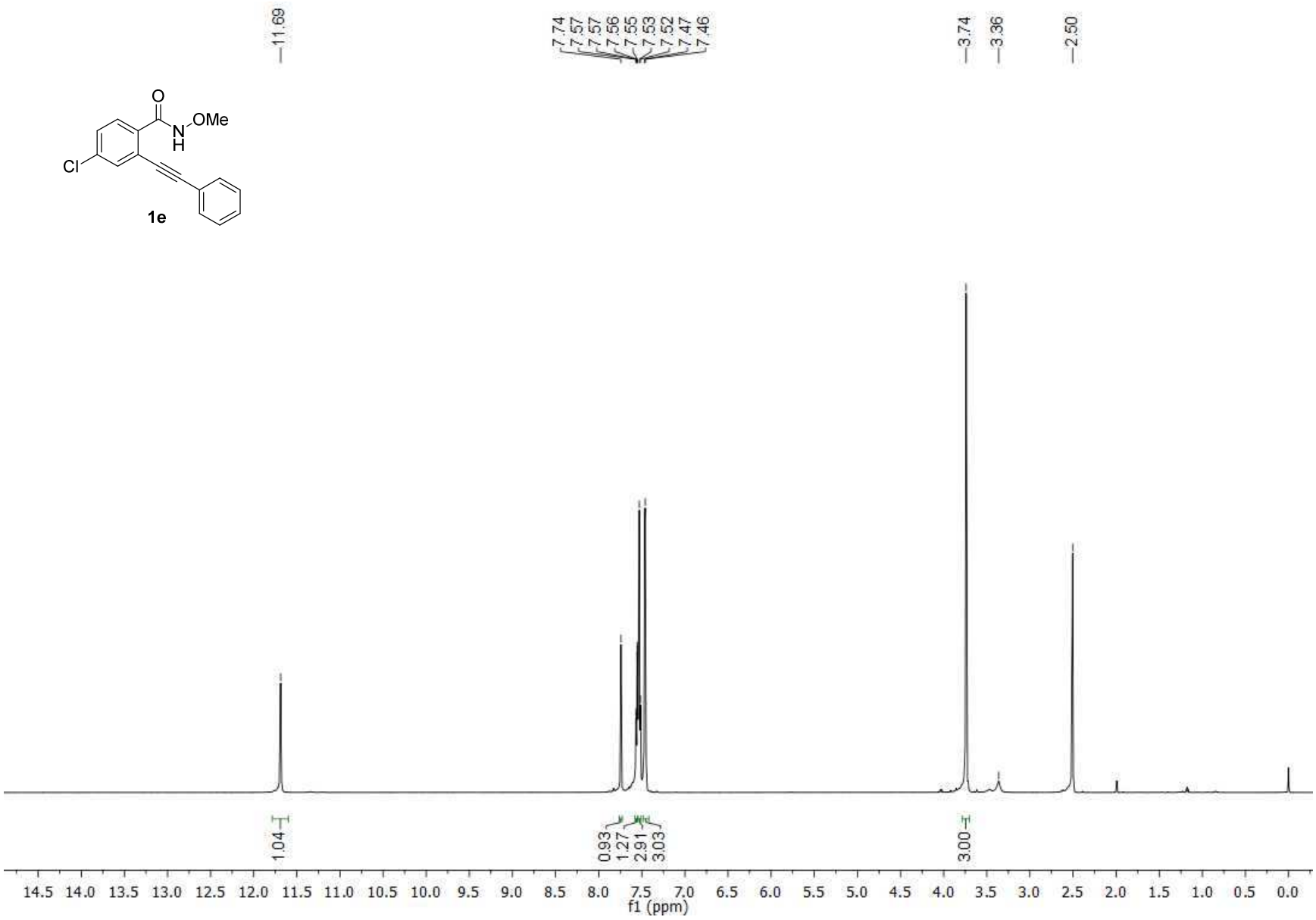
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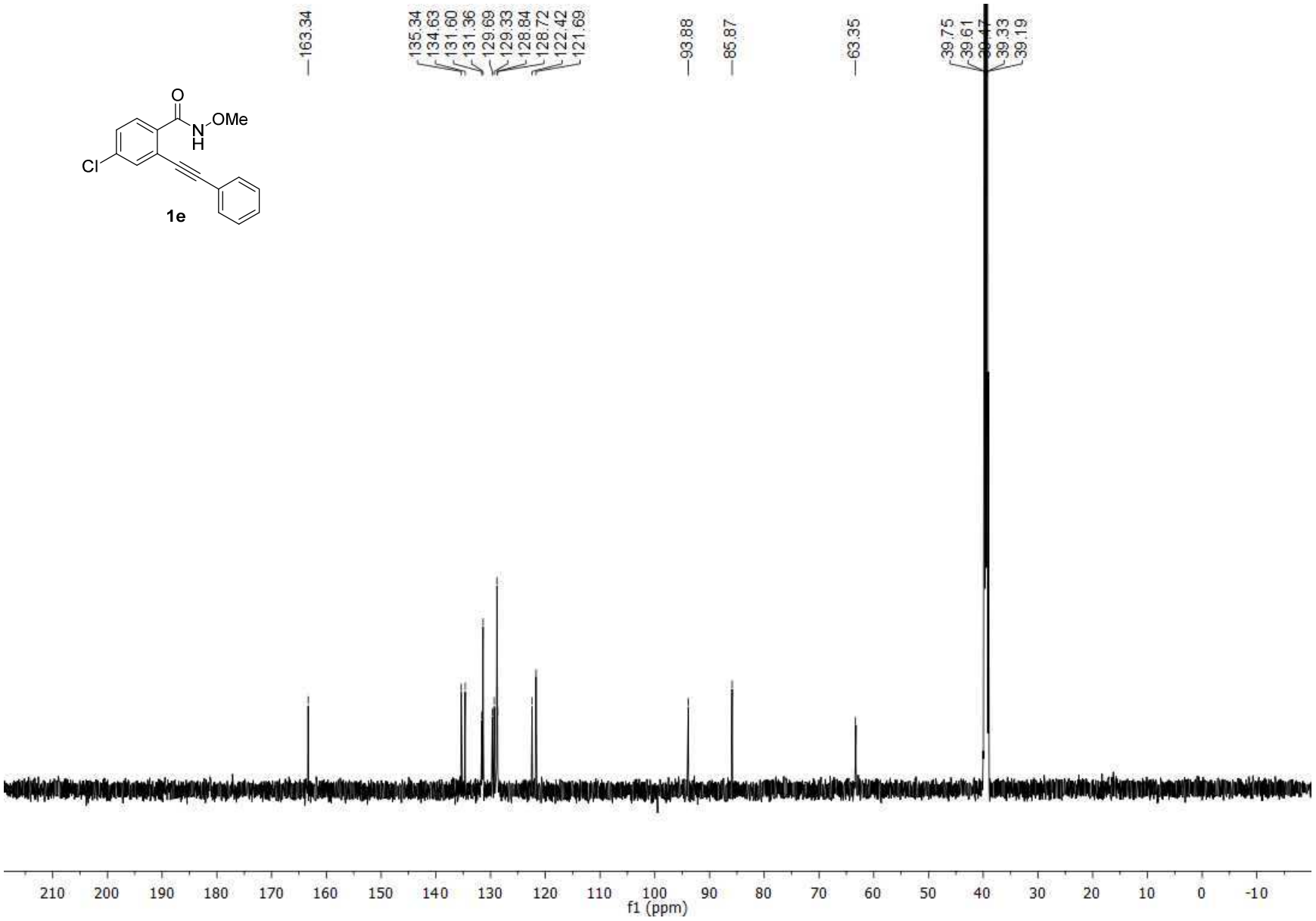
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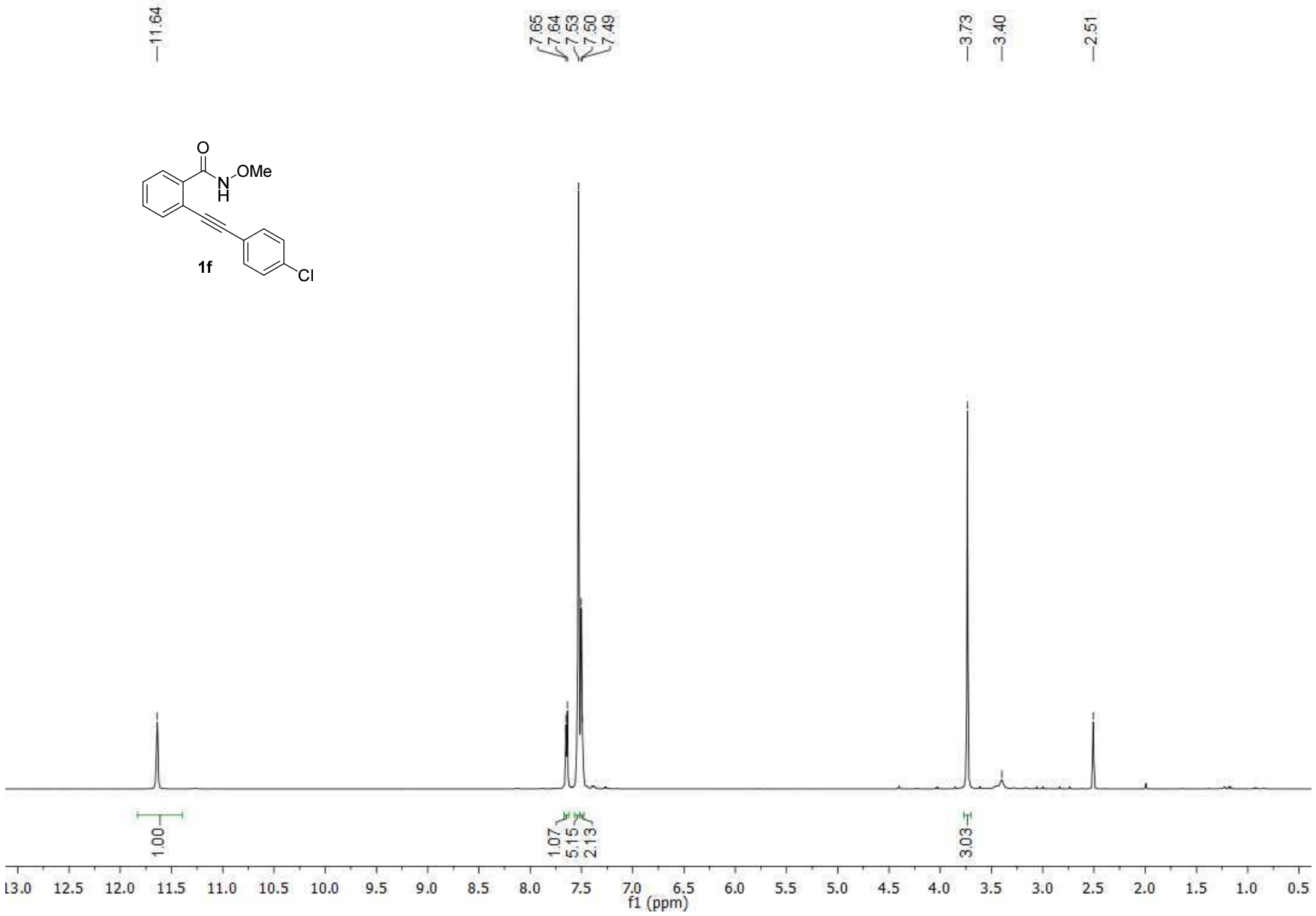
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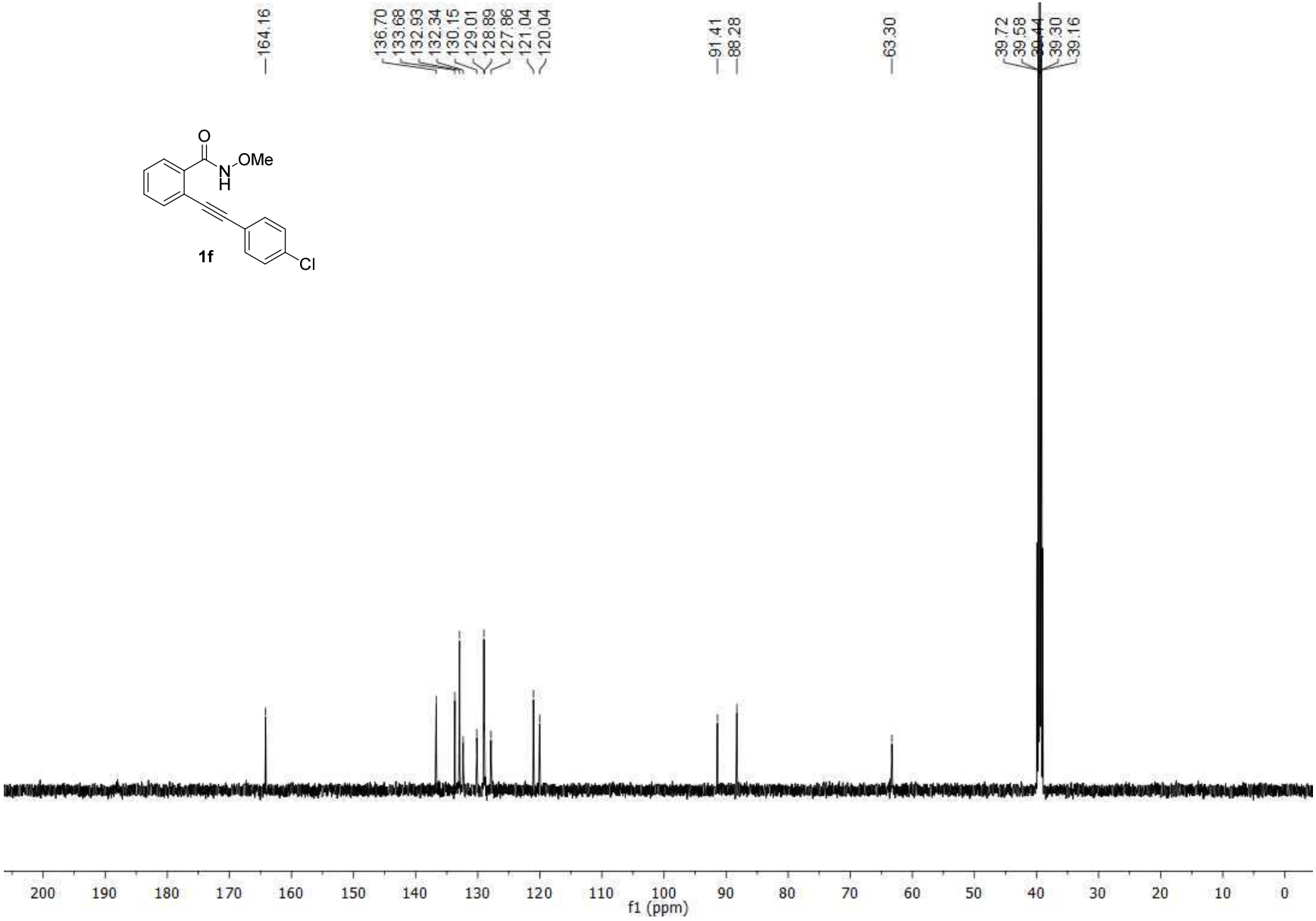
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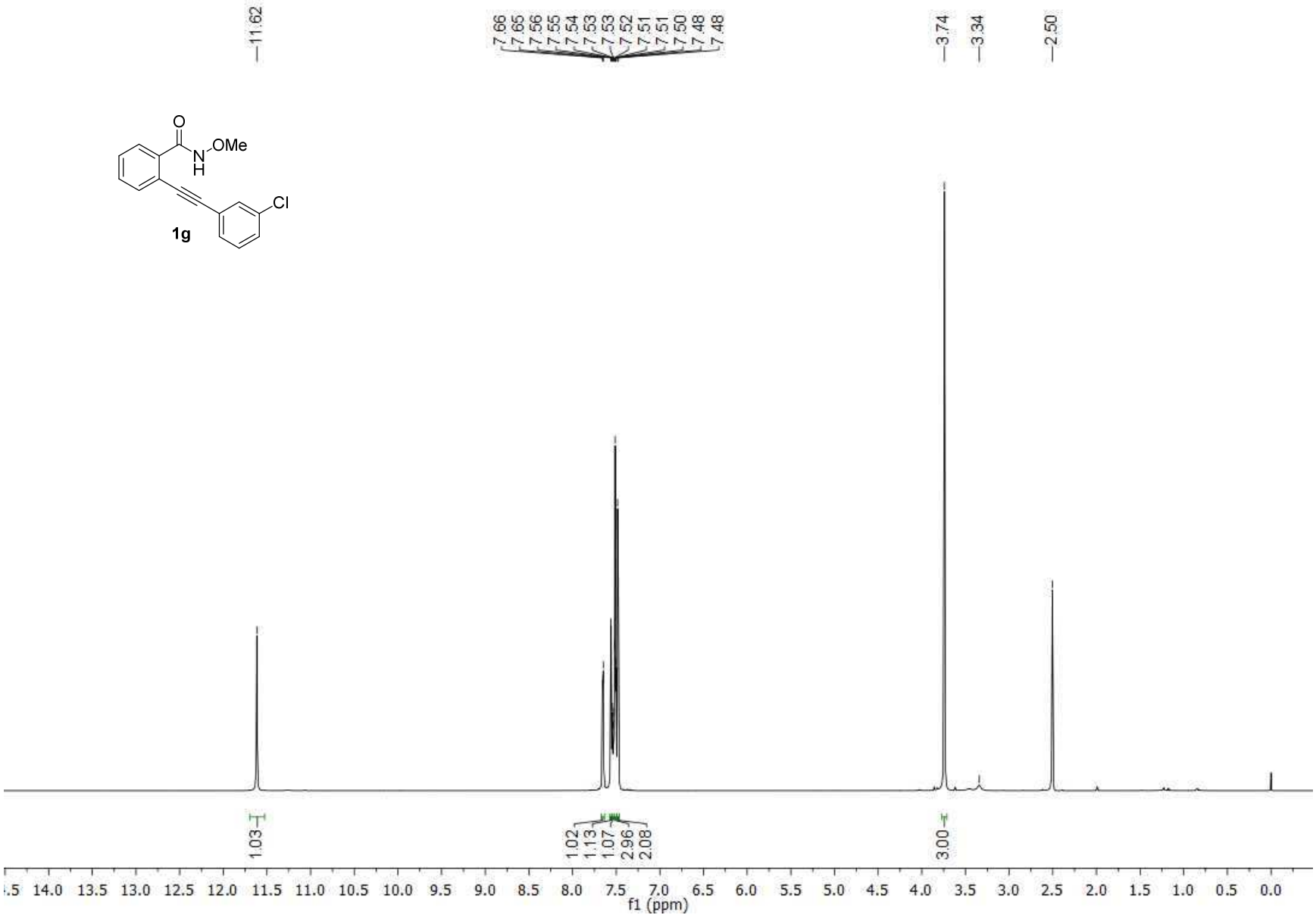
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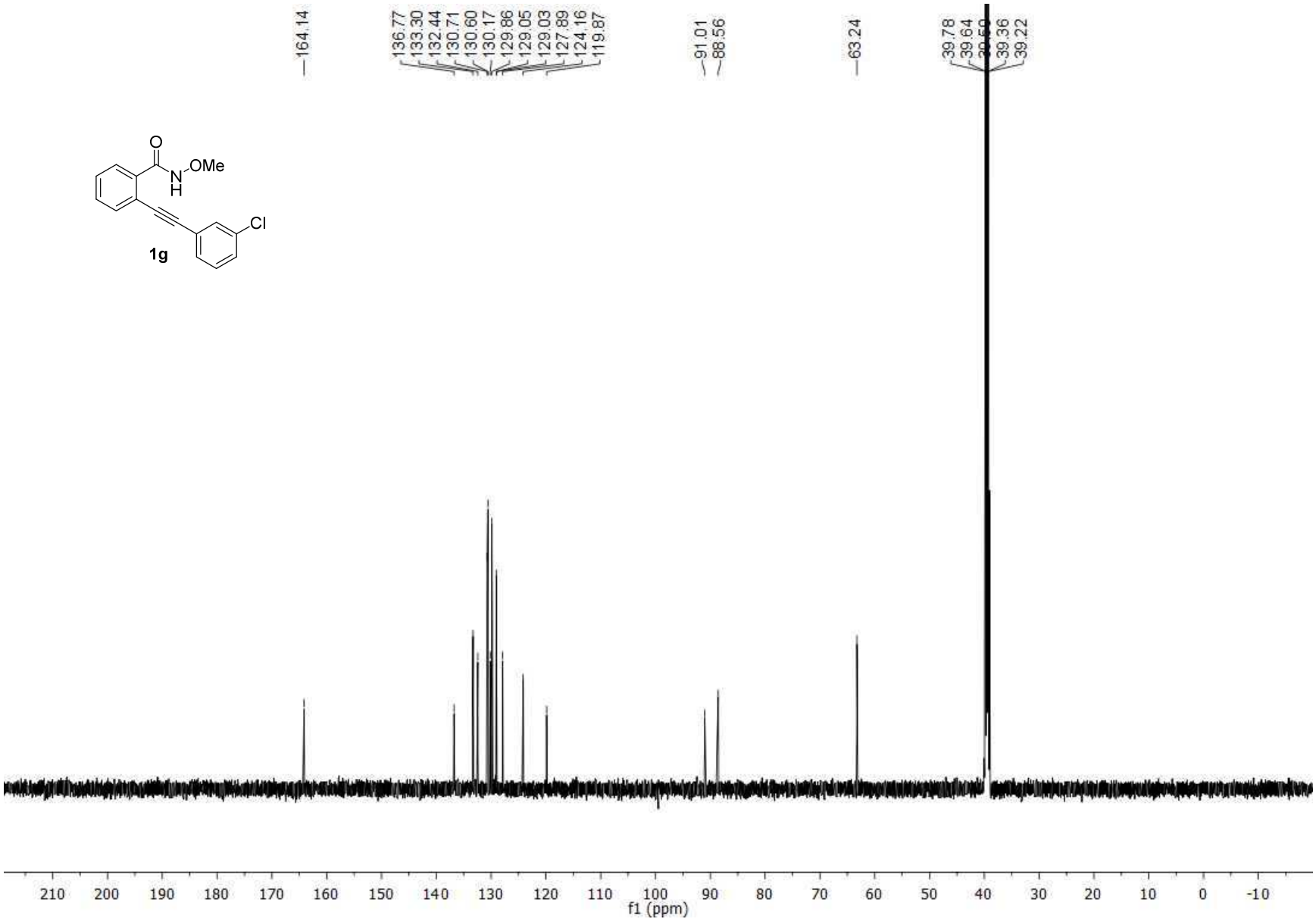
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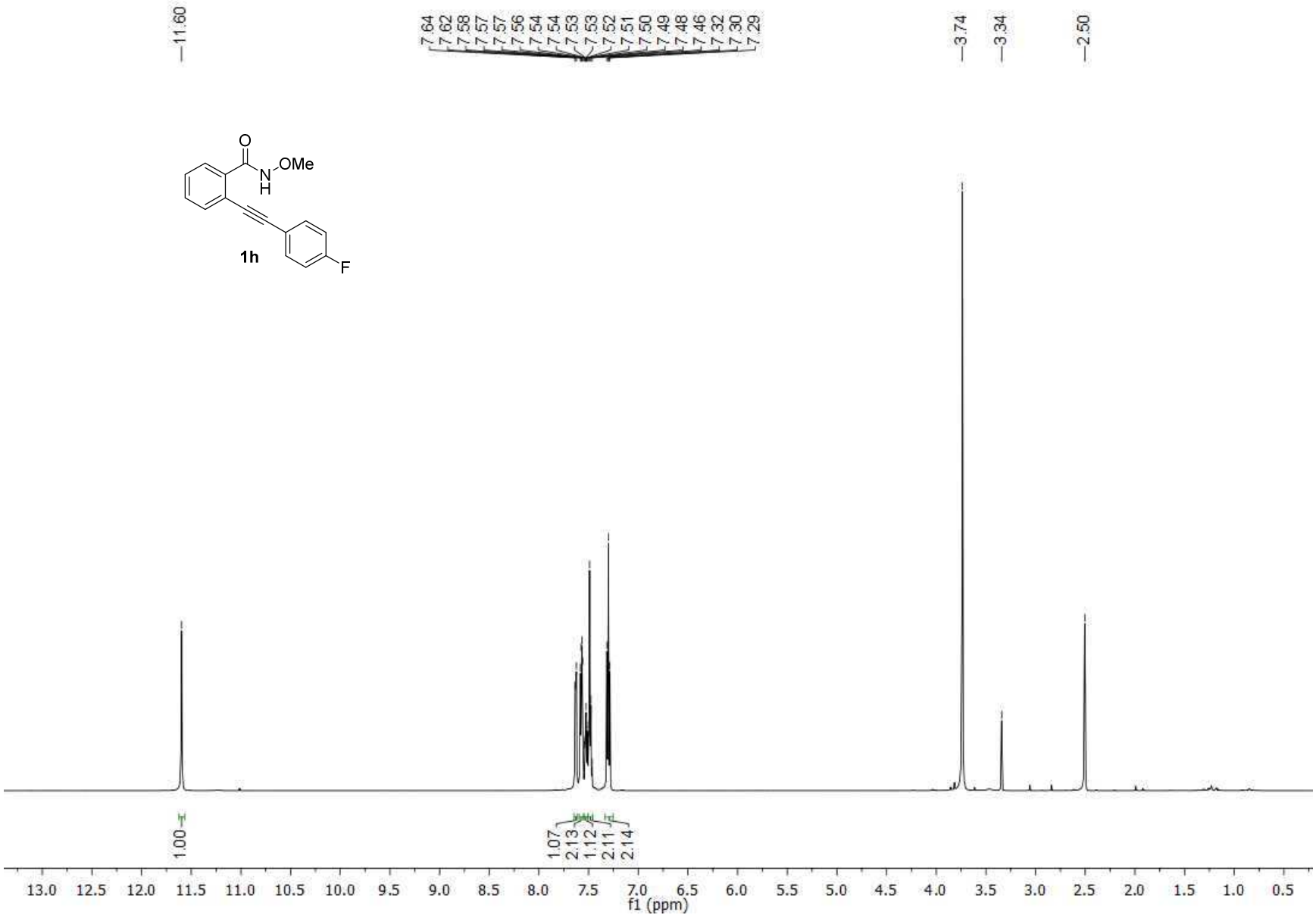
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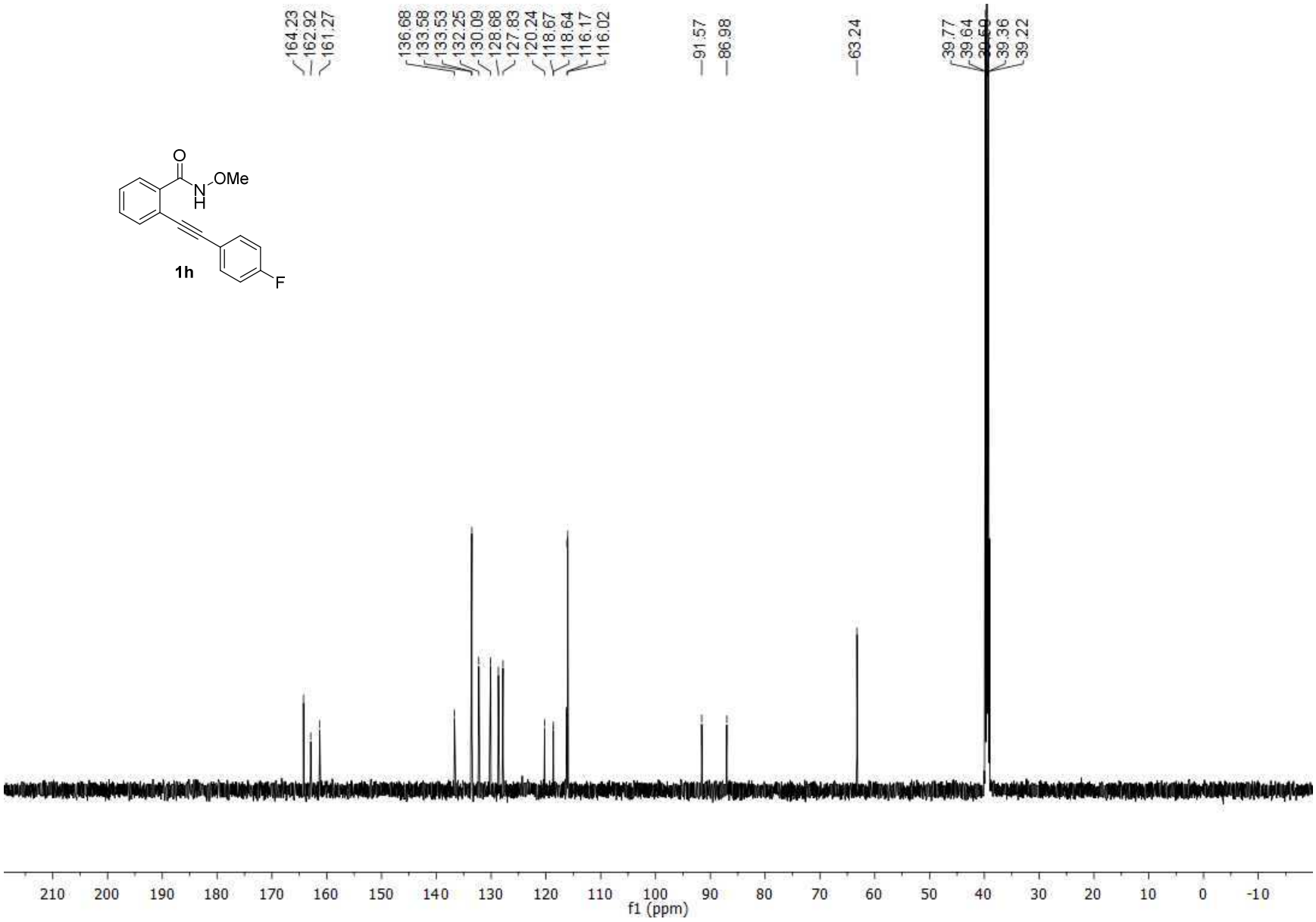
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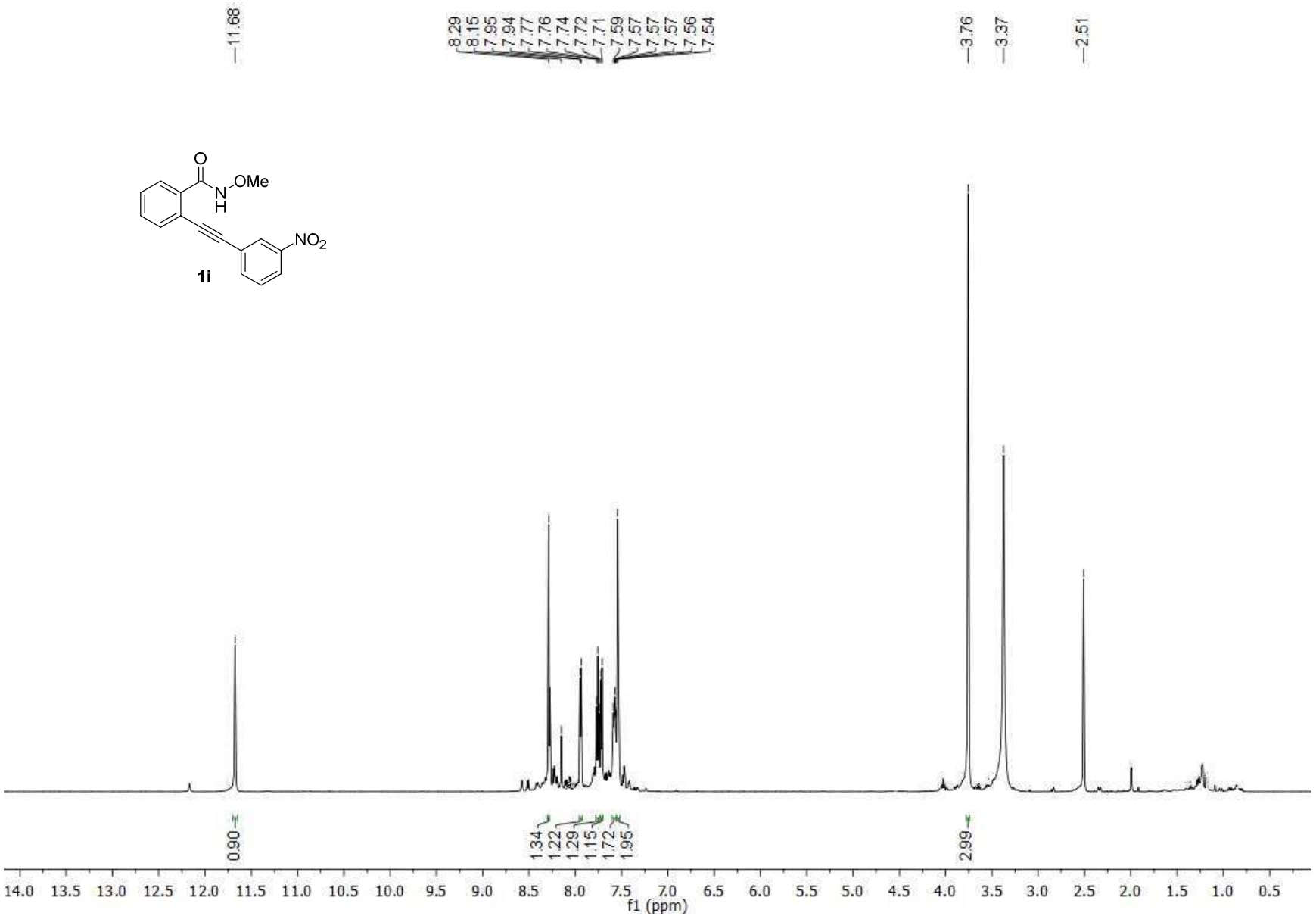
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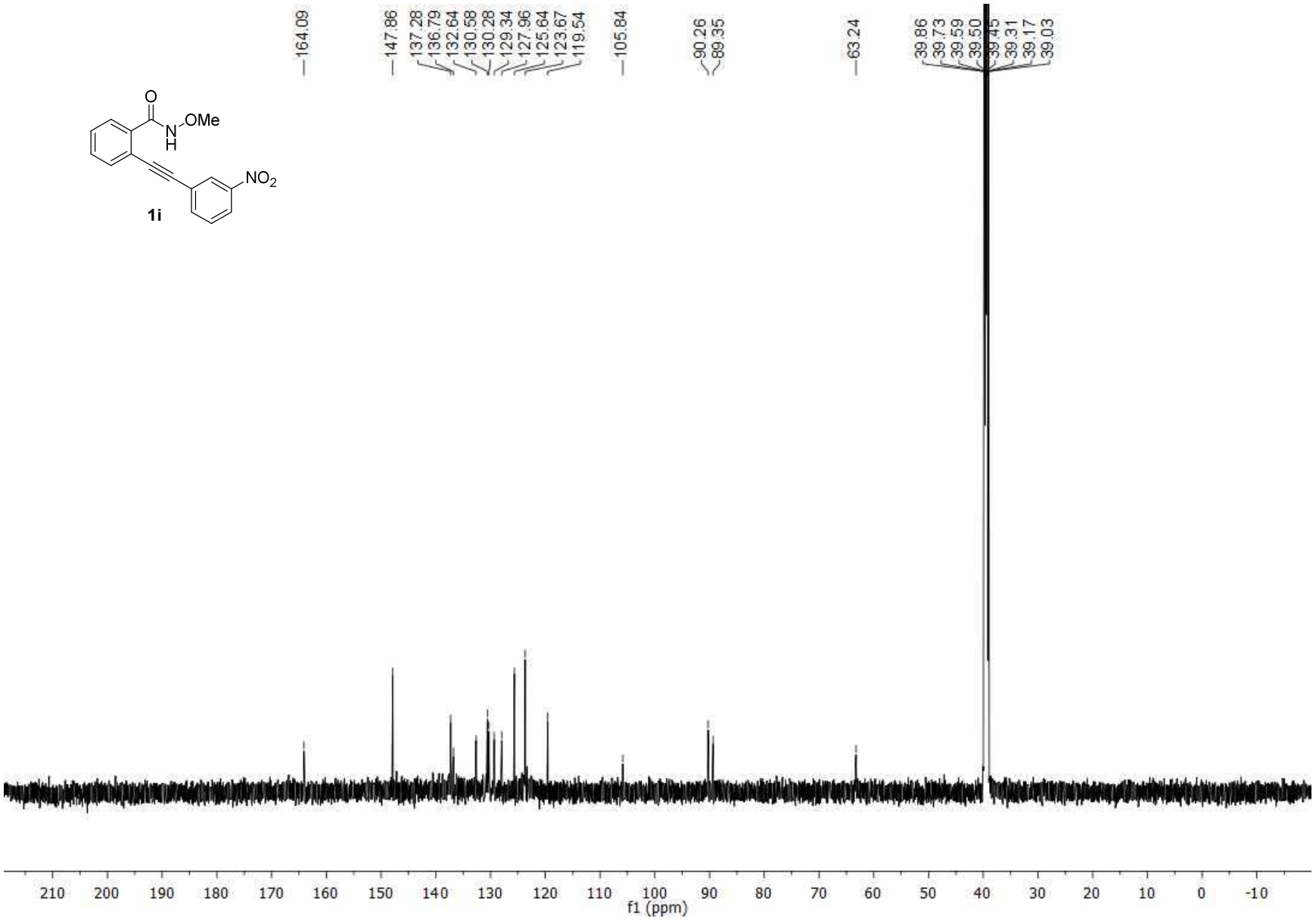
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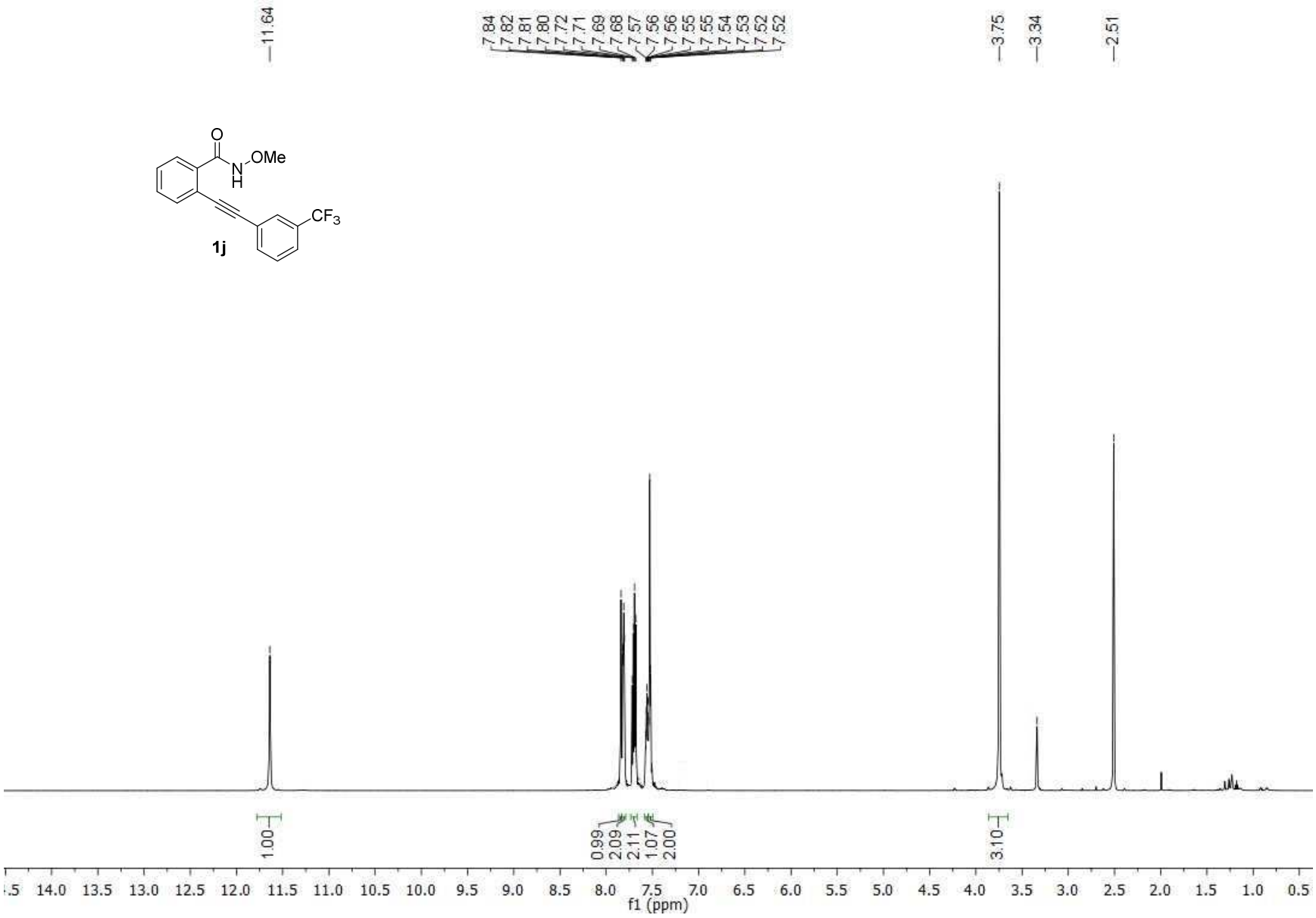
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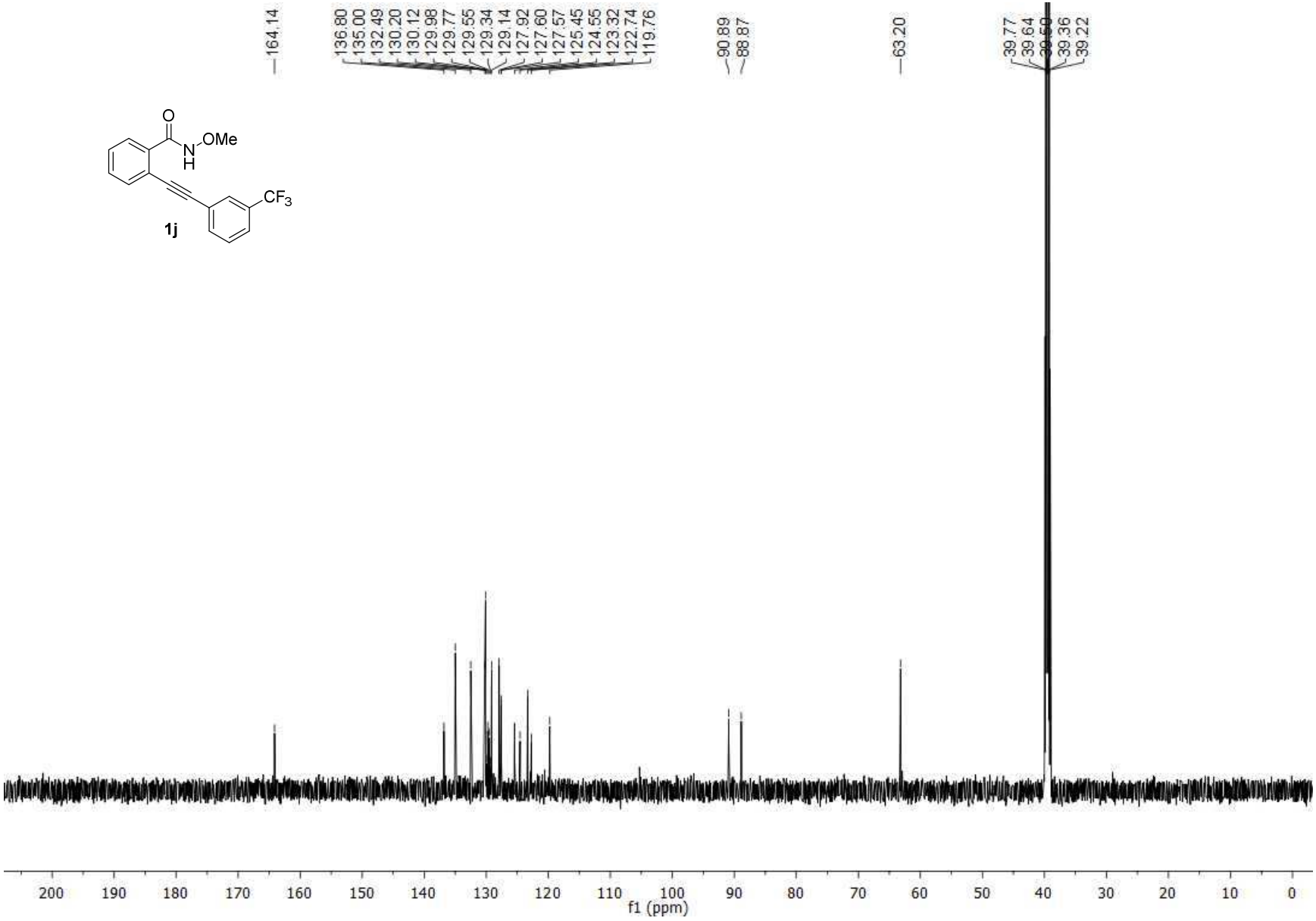
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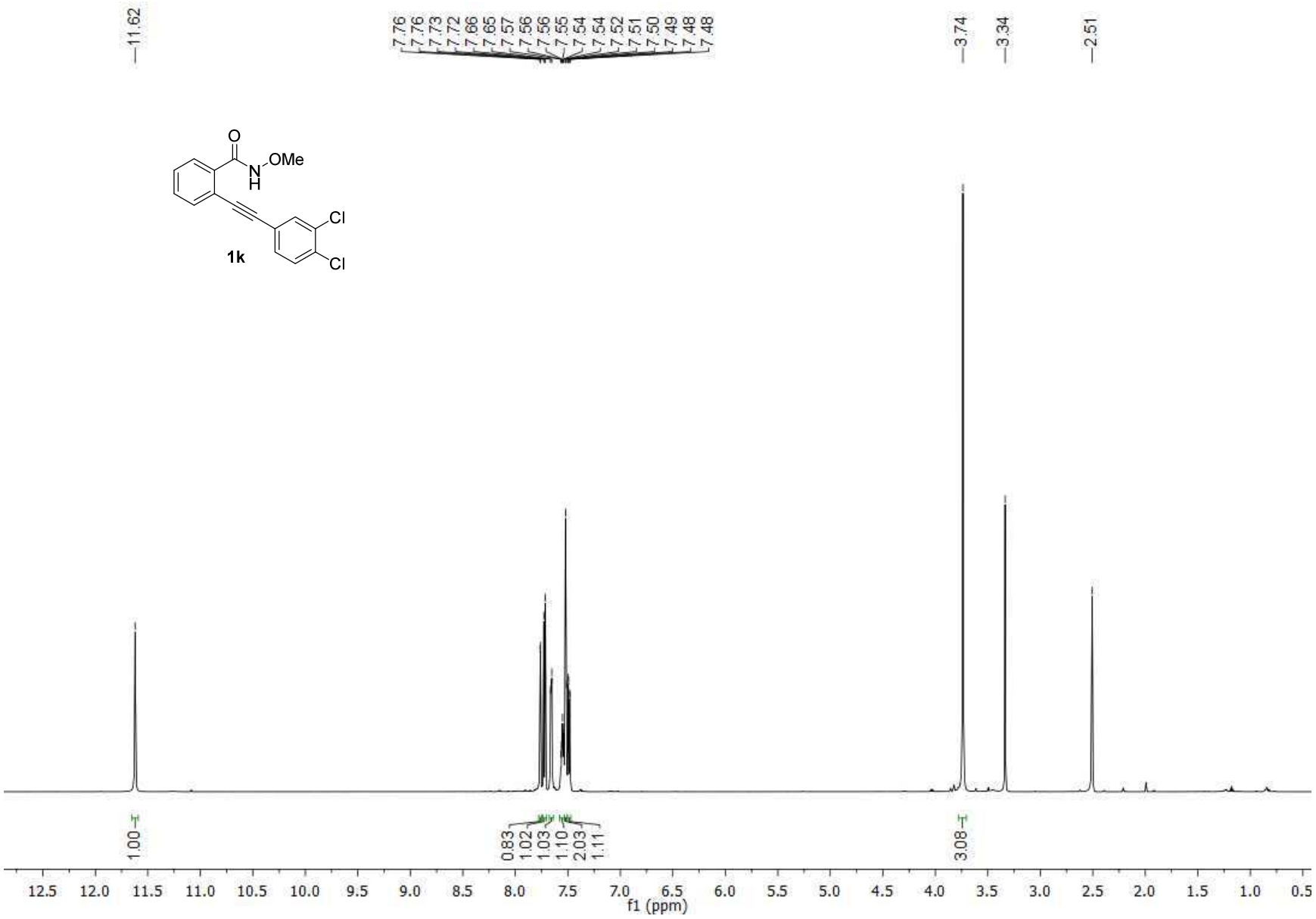
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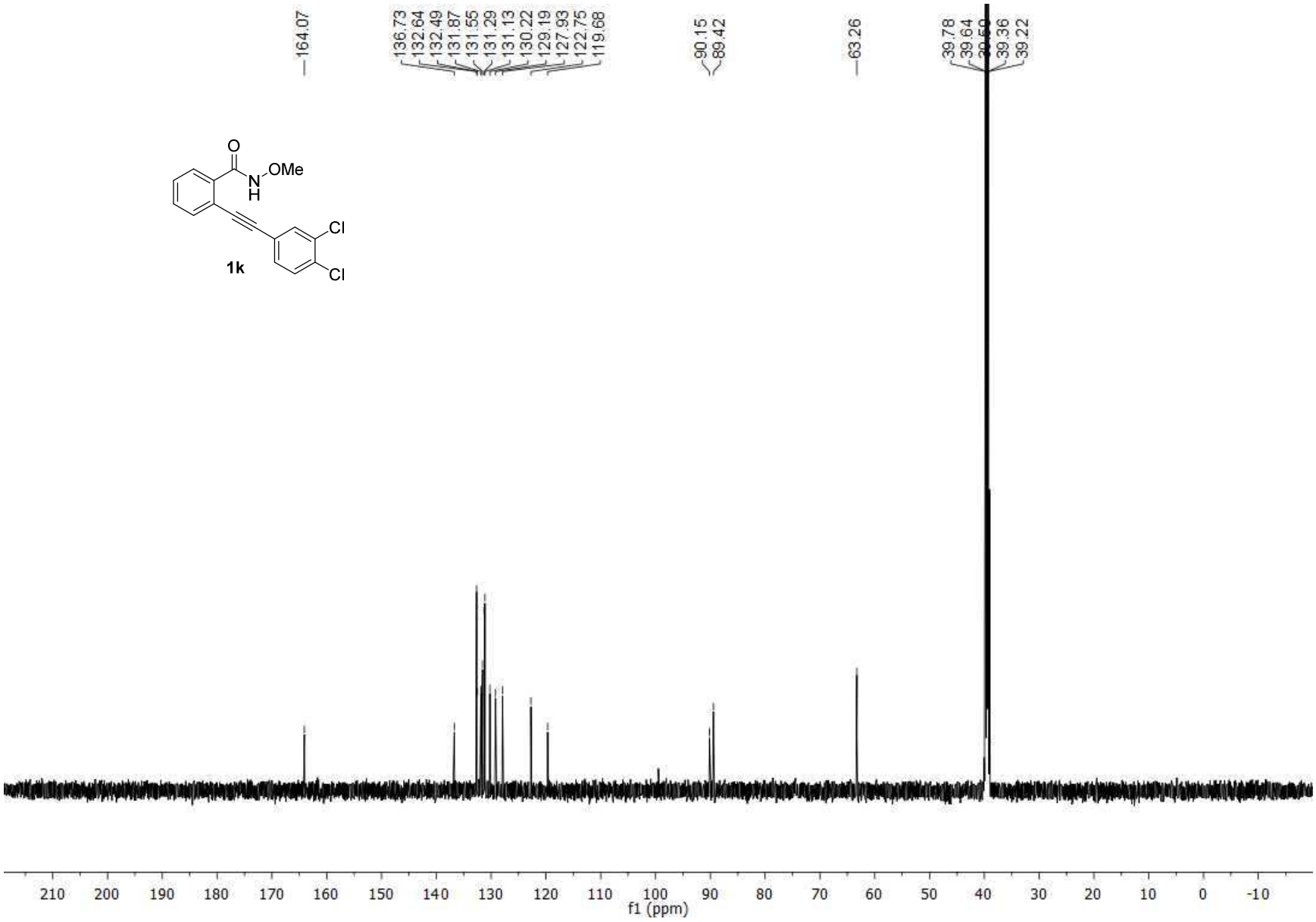
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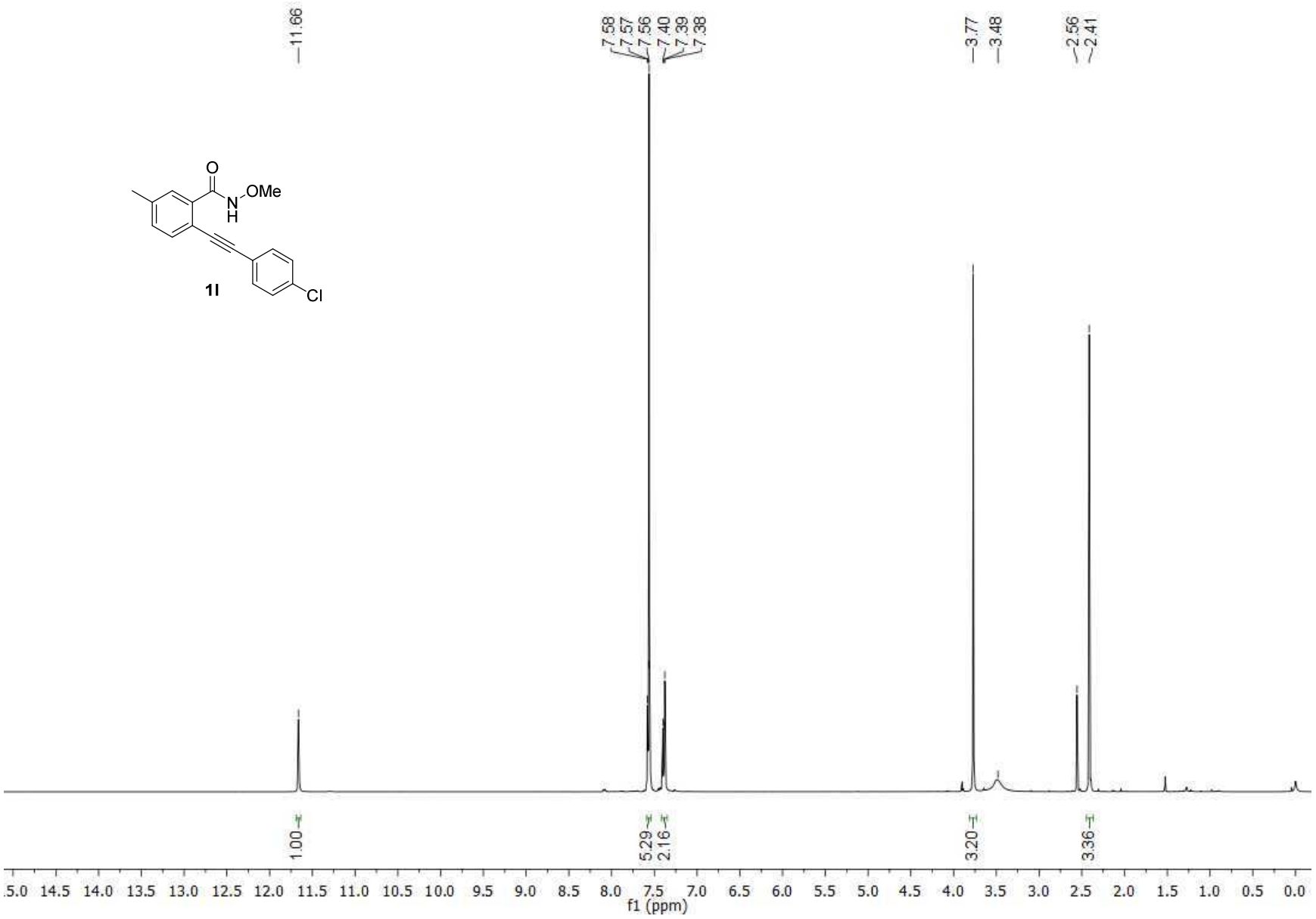
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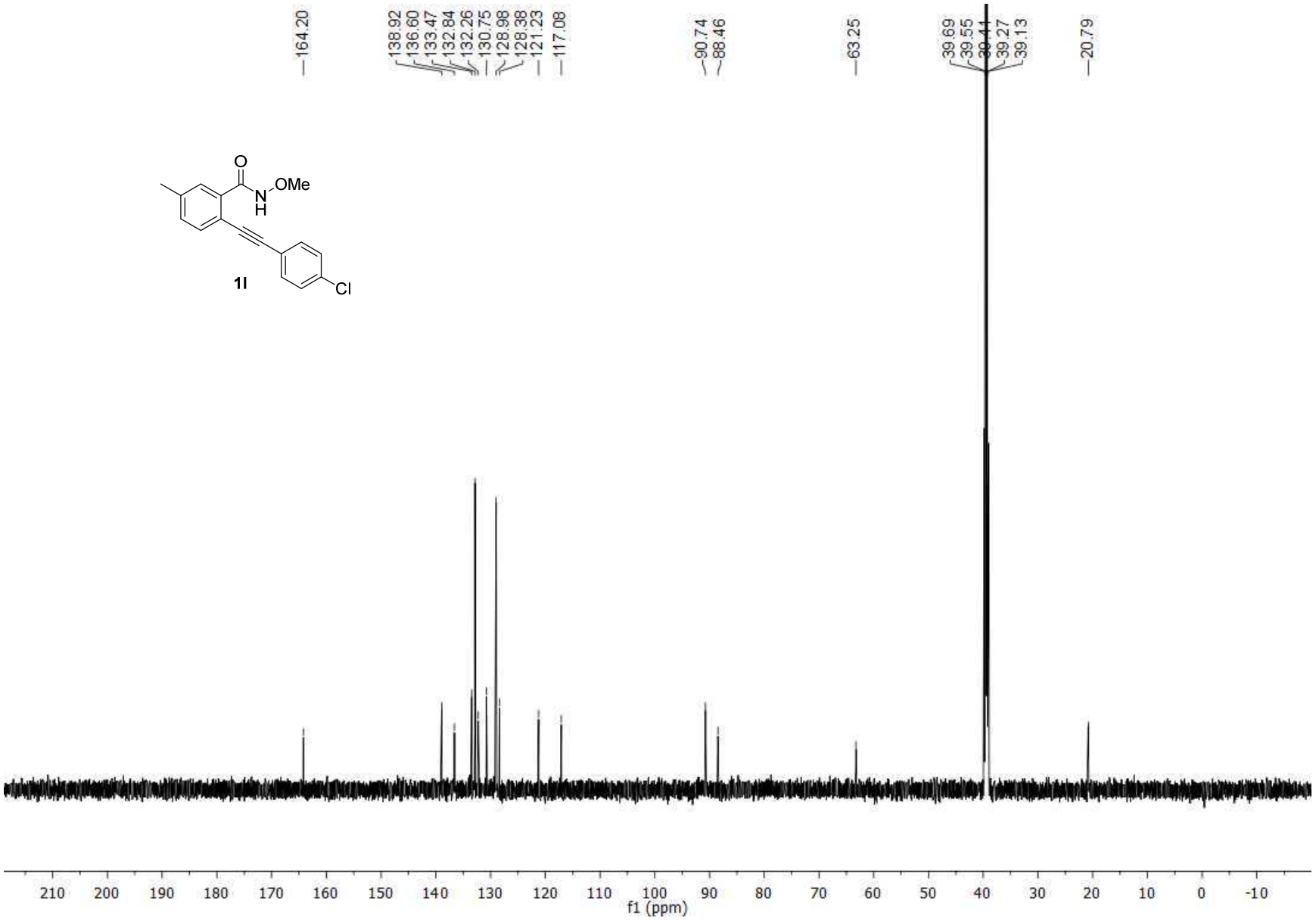
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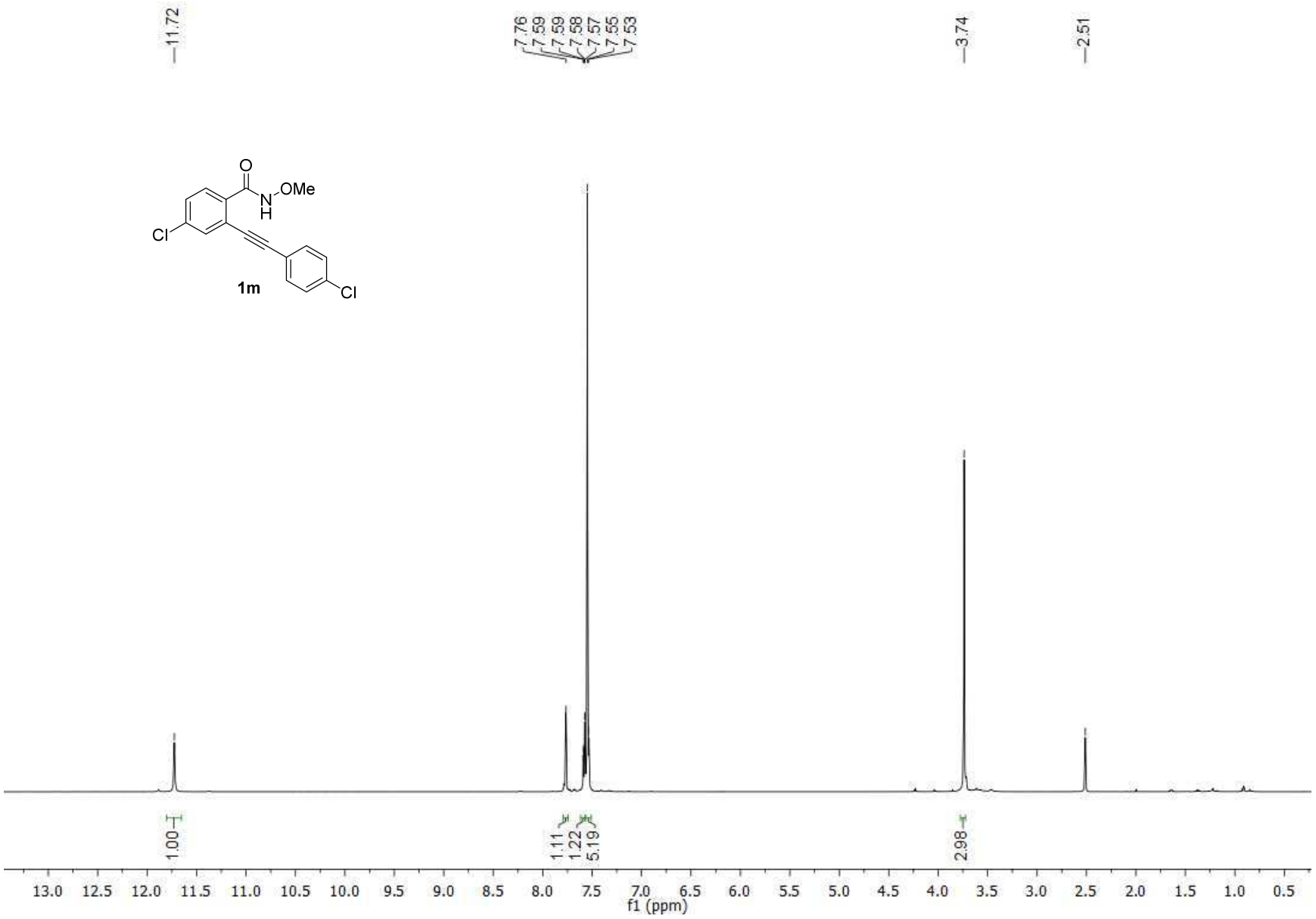
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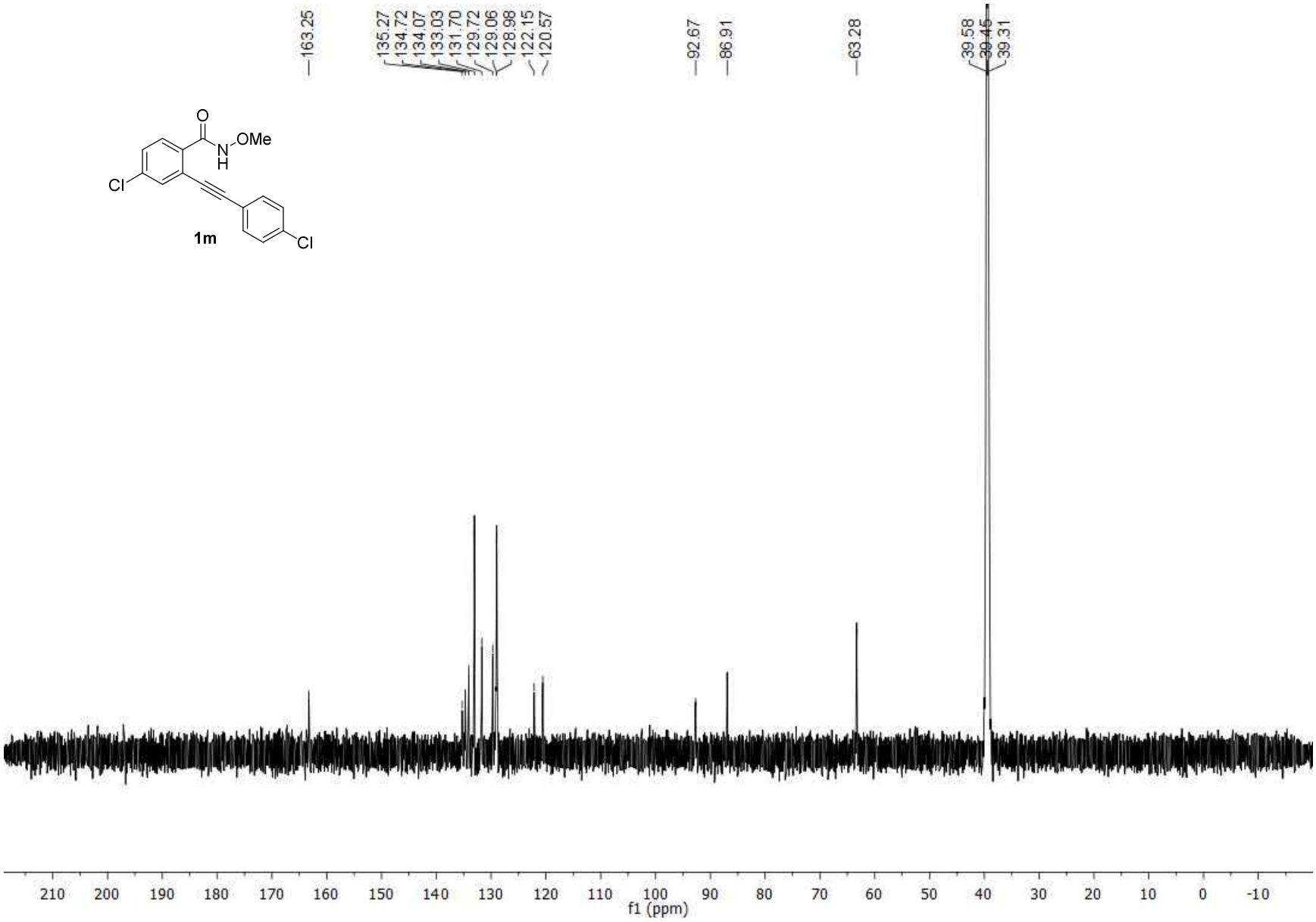
Cl
Cl
1m
1.00
1.11
1.22
5.19
2.98
13.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)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129.06
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122.15
120.57
92.67
86.91
63.28
39.58
39.45
39.31
O
N
H
OMe
Cl
Cl
1m
210
200
190
180
170
160
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100
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80
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30
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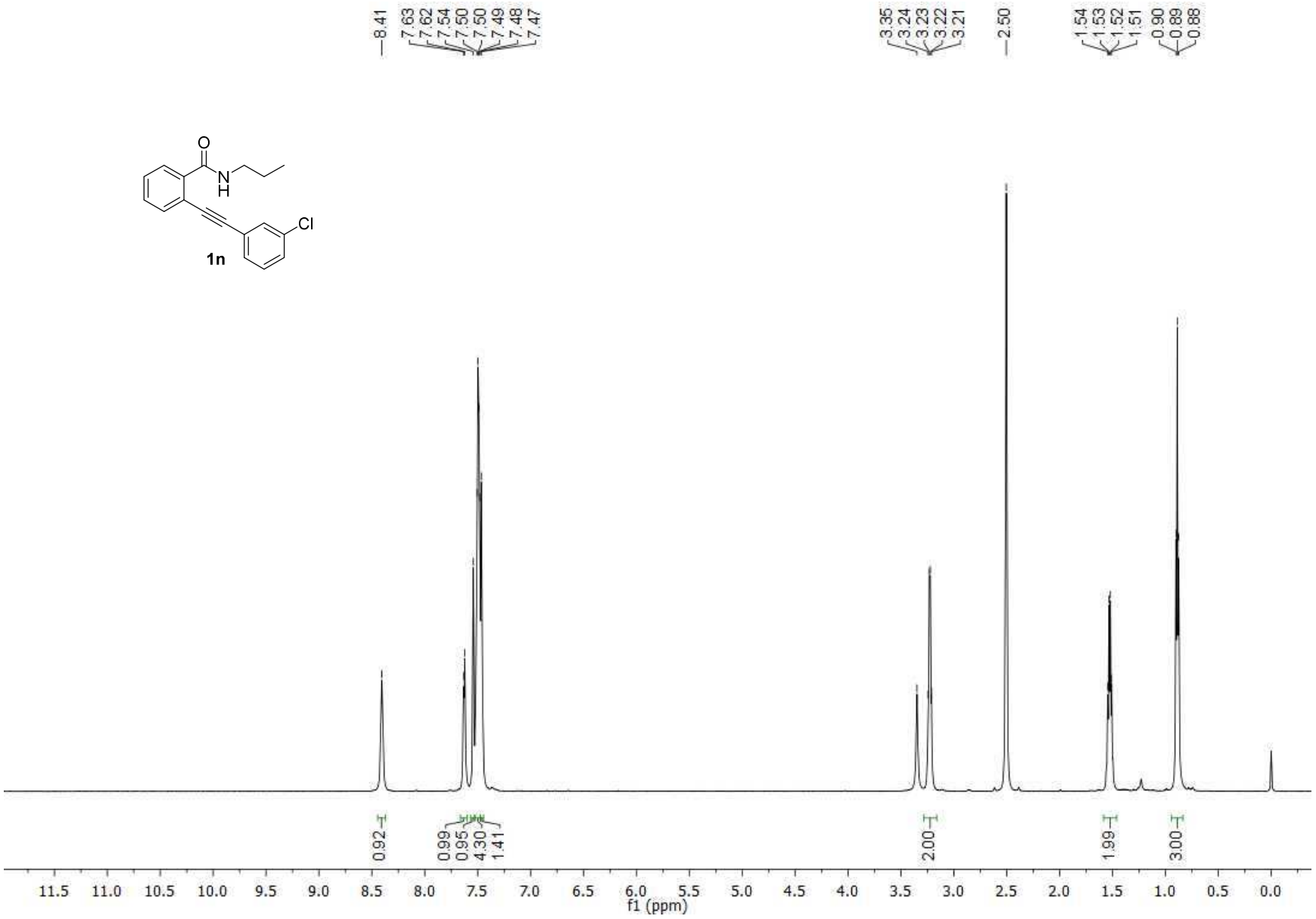
f1 (ppm)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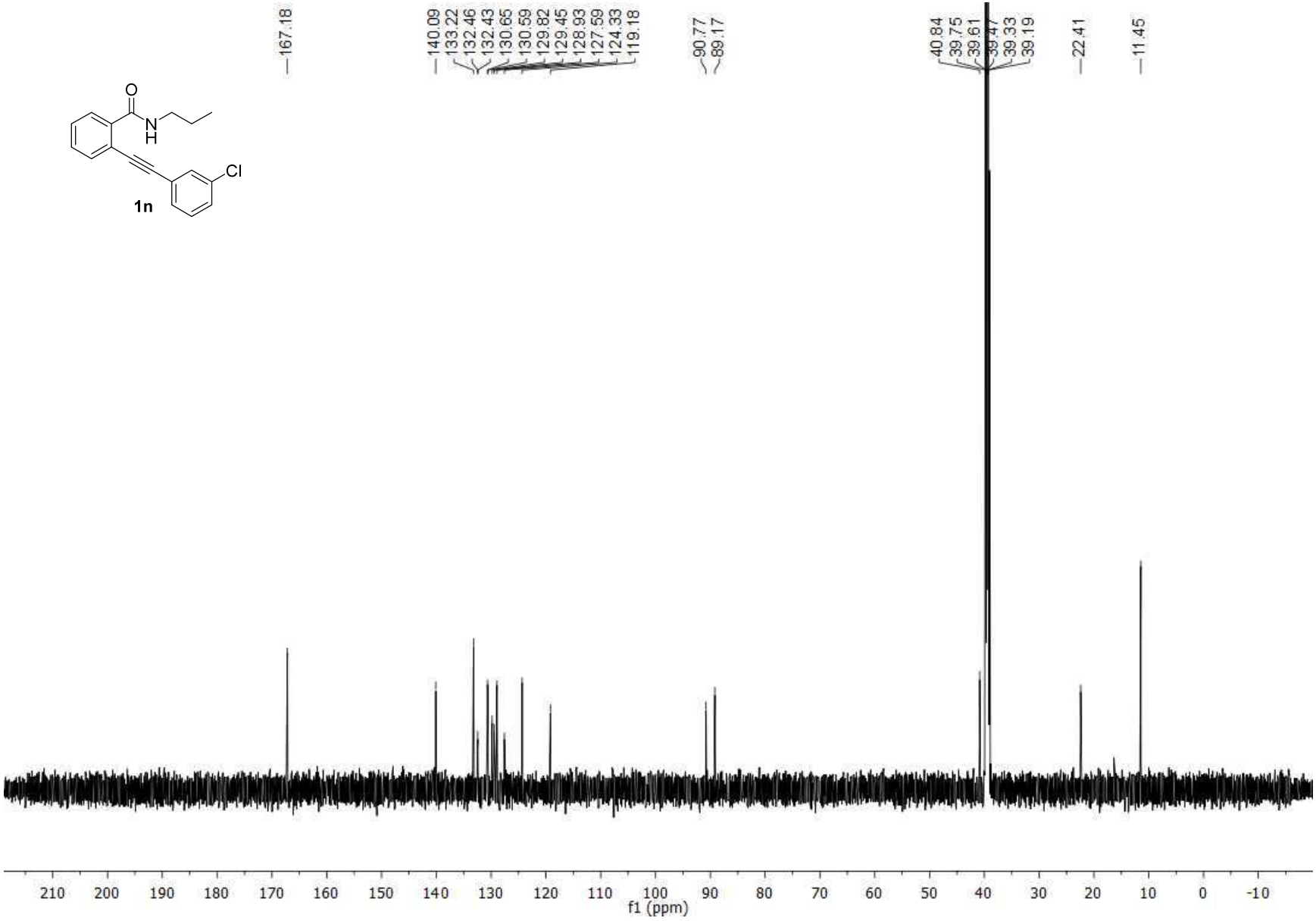
7.47
3.35
3.24
3.23
3.22
3.21
2.50
1.54
1.53
1.52
1.51
0.90
0.89
0.88
O
N
H
Cl
1n
0.92
0.99
0.95
4.30
1.41
2.00
1.99
3.00
11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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129.82
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127.59
124.33
119.18
90.77
89.17
40.84
39.75
39.61
39.47
39.33
39.19
22.41
11.45
O
N
H
Cl
1n
210
200
190
180
170
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100
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80
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40
30
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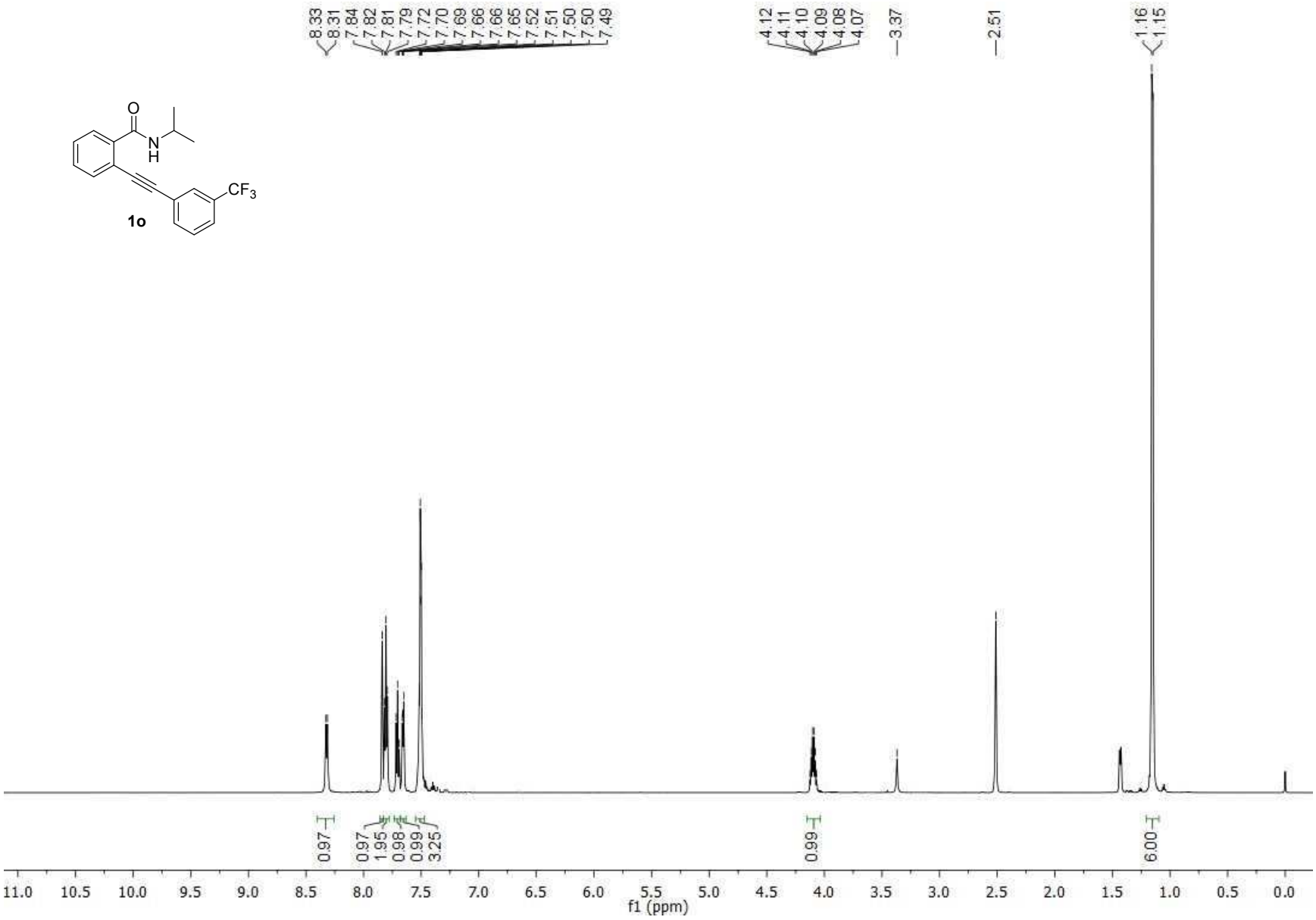
0
-10
f1 (ppm)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7.66
7.65
7.52
7.51
7.50
7.50
7.49
4.12
4.11
4.10
4.09
4.08
4.07
3.37
2.51
1.16
1.15
O
N
H
CF3
1o
0.97
0.97
1.95
0.98
0.99
3.25
0.99
6.00
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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3.5
3.0
2.5
2.0
1.5
1.0
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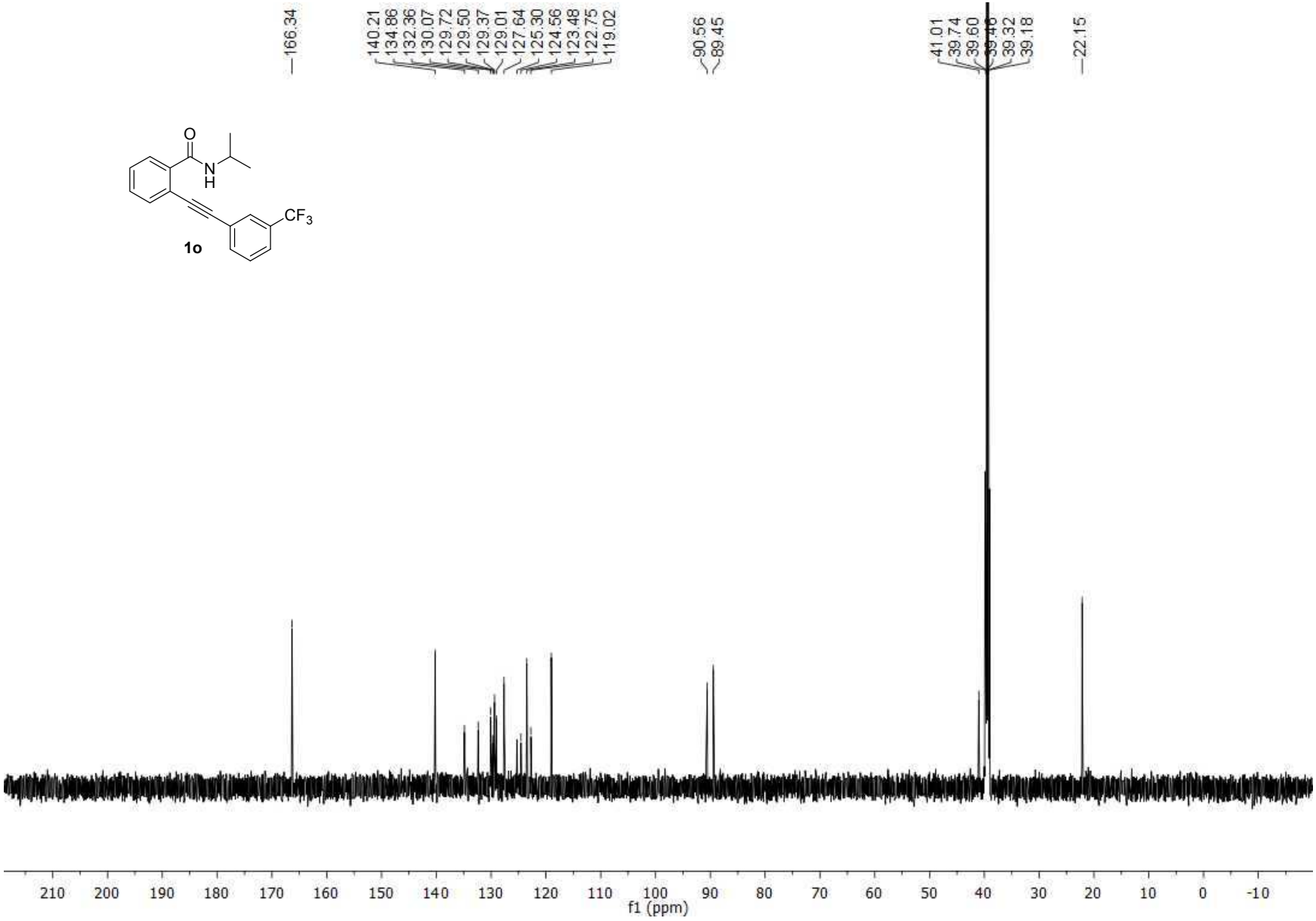
0.0
f1 (ppm)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123.48
122.75
119.02
90.56
89.45
41.01
39.74
39.60
39.46
39.32
39.18
22.15
O
N
H
CF3
1o
210
200
190
180
170
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120
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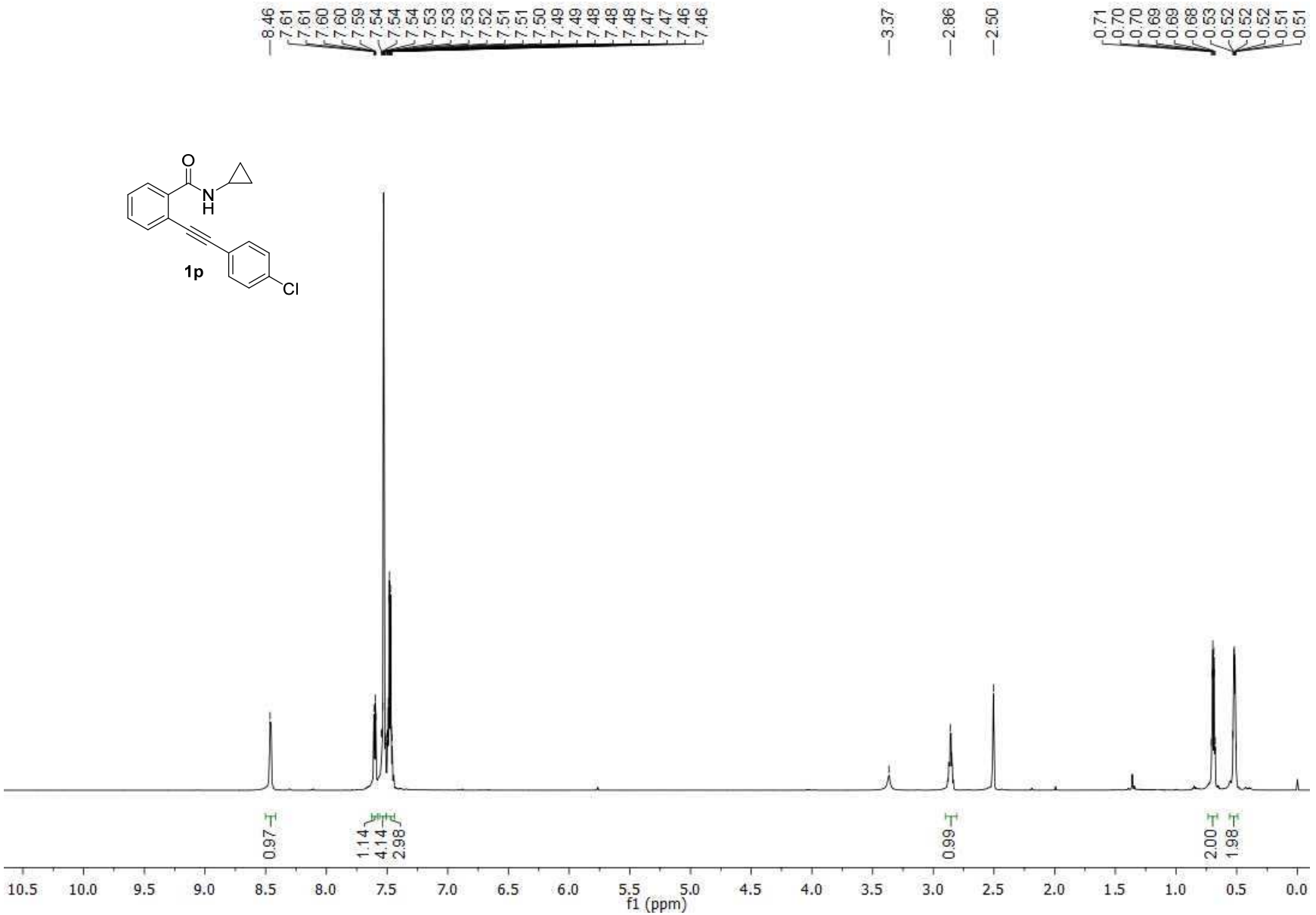
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)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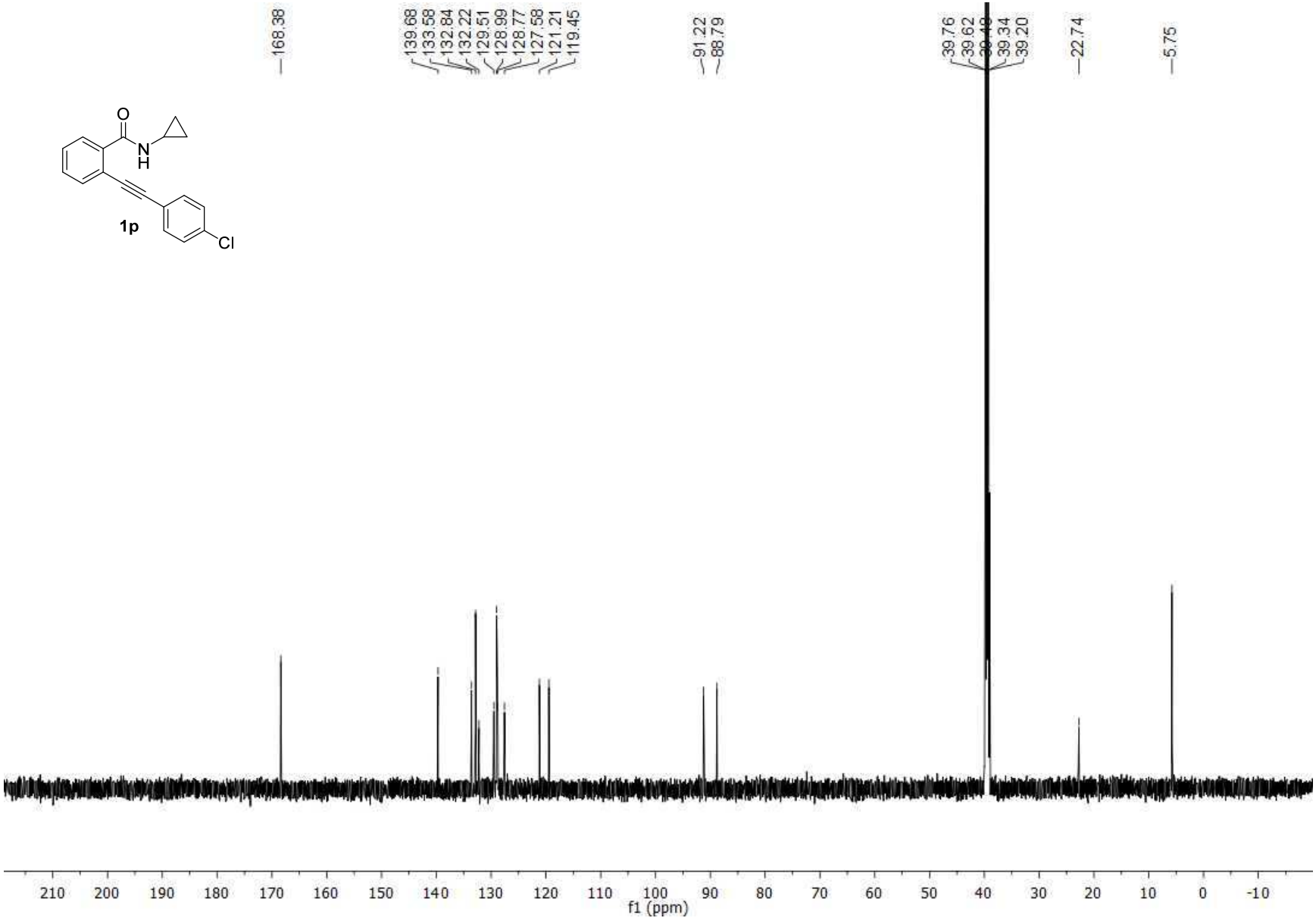
7.50
7.49
7.49
7.48
7.48
7.48
7.47
7.47
7.46
7.46
3.37
2.86
2.50
0.71
0.70
0.70
0.69
0.69
0.68
0.53
0.52
0.52
0.52
0.51
0.51
O
N
H
1p
Cl
0.97
1.14
4.14
2.98
0.99
2.00
1.98
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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129.51
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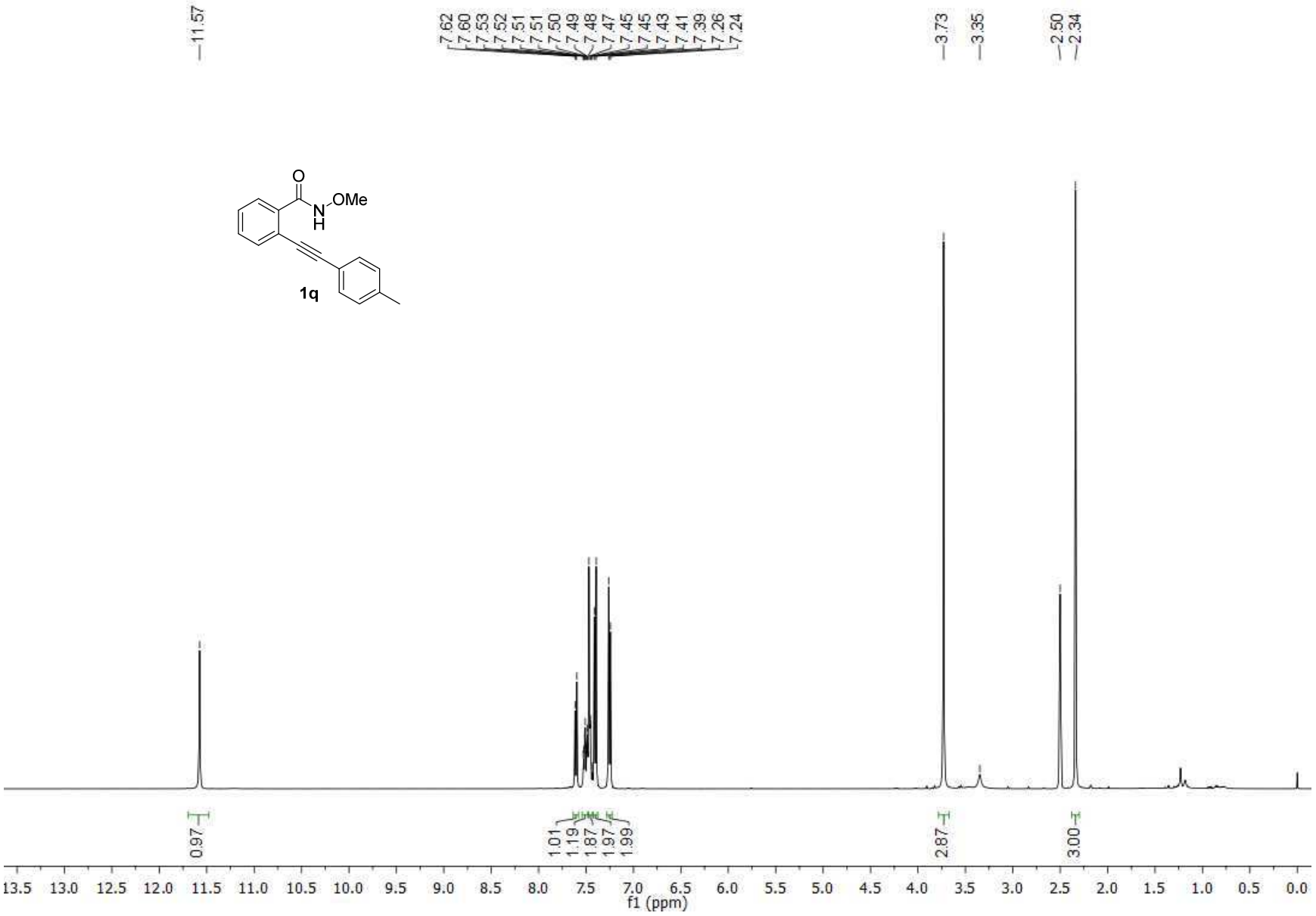
128.77
127.58
121.21
119.45
91.22
88.79
39.76
39.62
39.48
39.34
39.20
22.74
5.75
O
N
H
1p
Cl
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)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7.51
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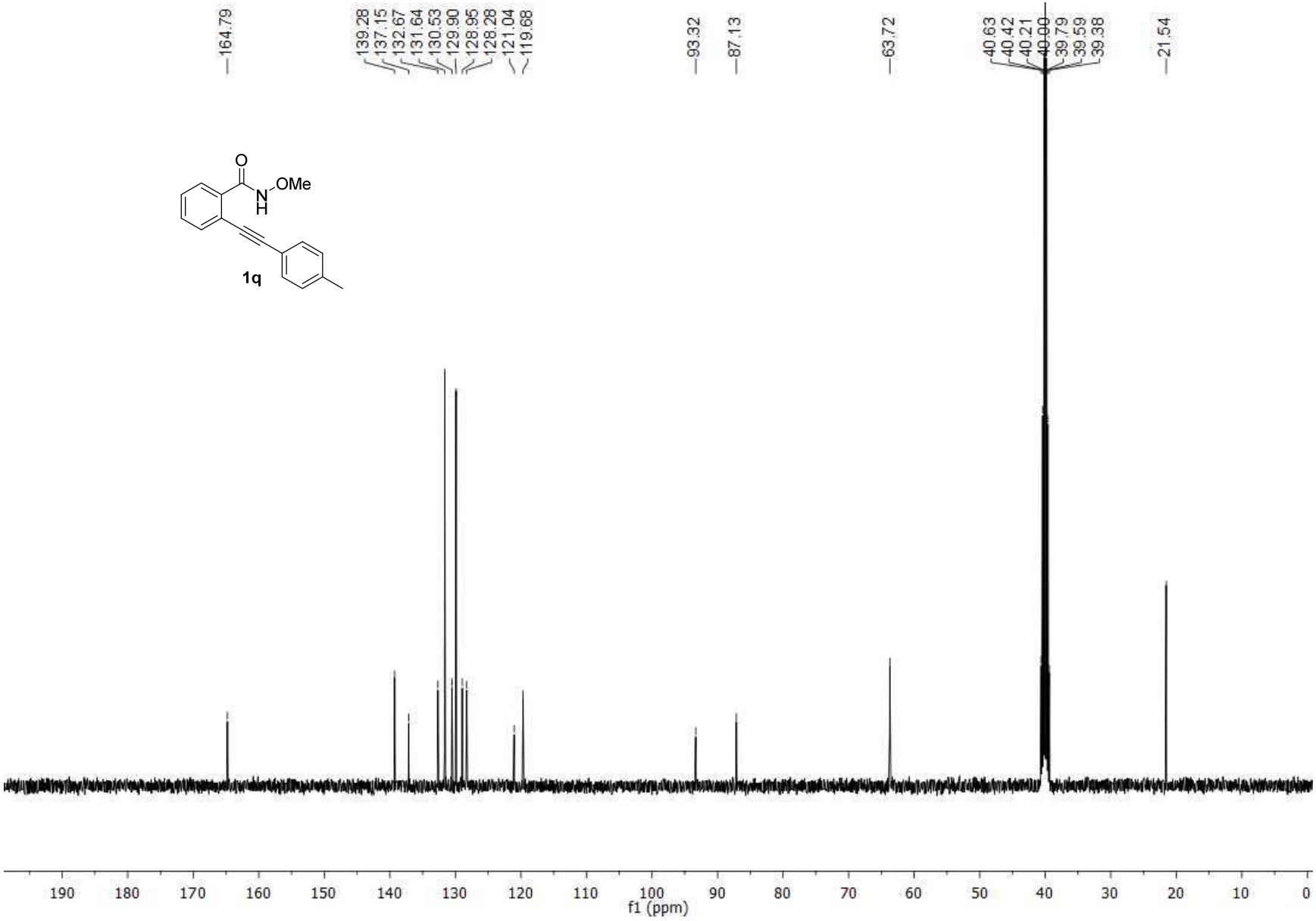
7.50
7.49
7.48
7.47
7.45
7.43
7.41
7.39
7.26
7.24
3.73
3.35
2.50
2.34
O
N
H
OMe
1q
0.97
1.01
1.19
1.87
1.97
1.99
2.87
3.00
13.5 13.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)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128.95
128.28
121.04
119.68
93.32
87.13
63.72
40.63
40.42
40.21
40.00
39.79
39.59
39.38
21.54
O
N
H
OMe
1q
190
180
170
160
150
140
130
120
110
100
90
80
70
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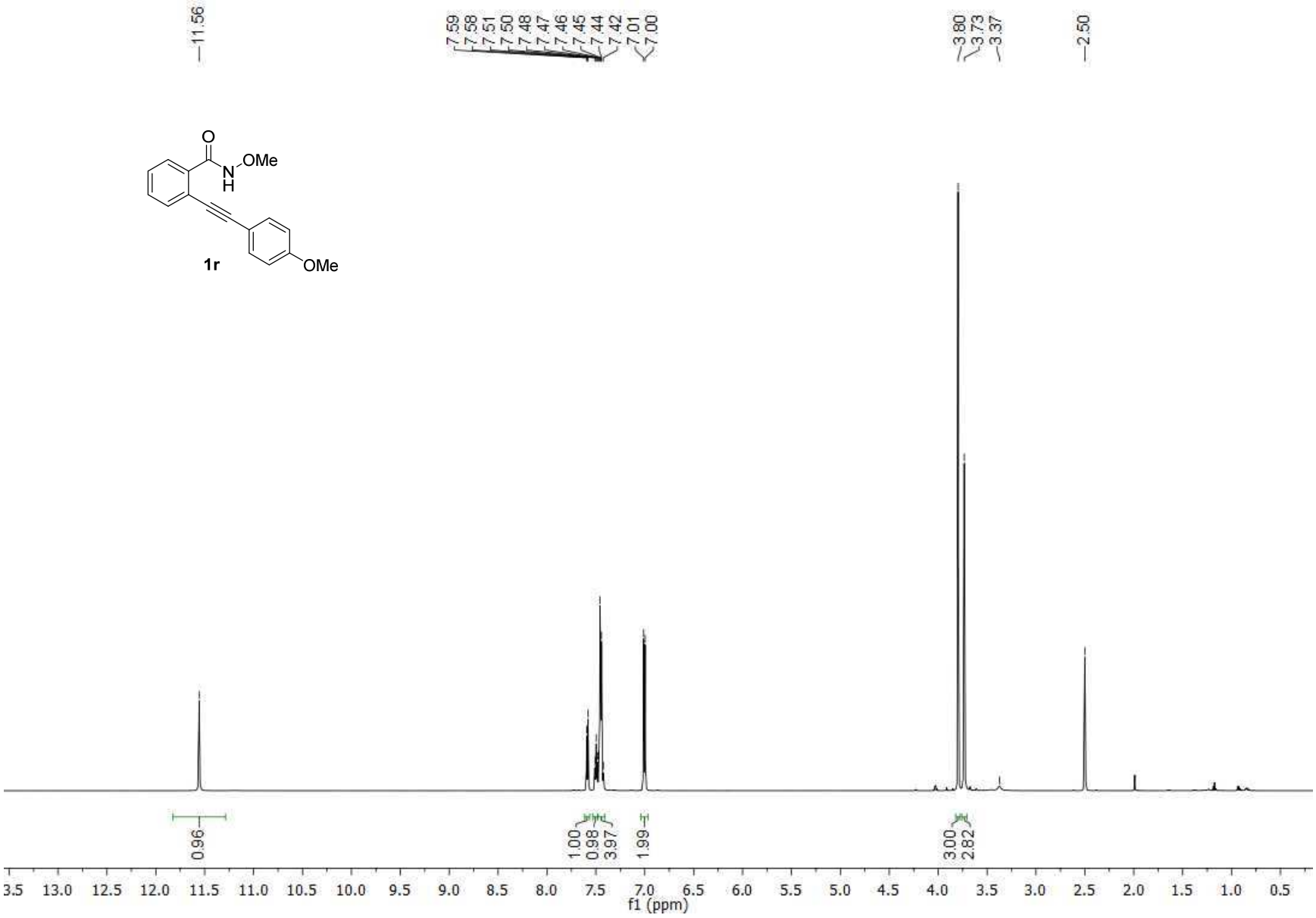
50
40
30
20
10
0
f1 (ppm)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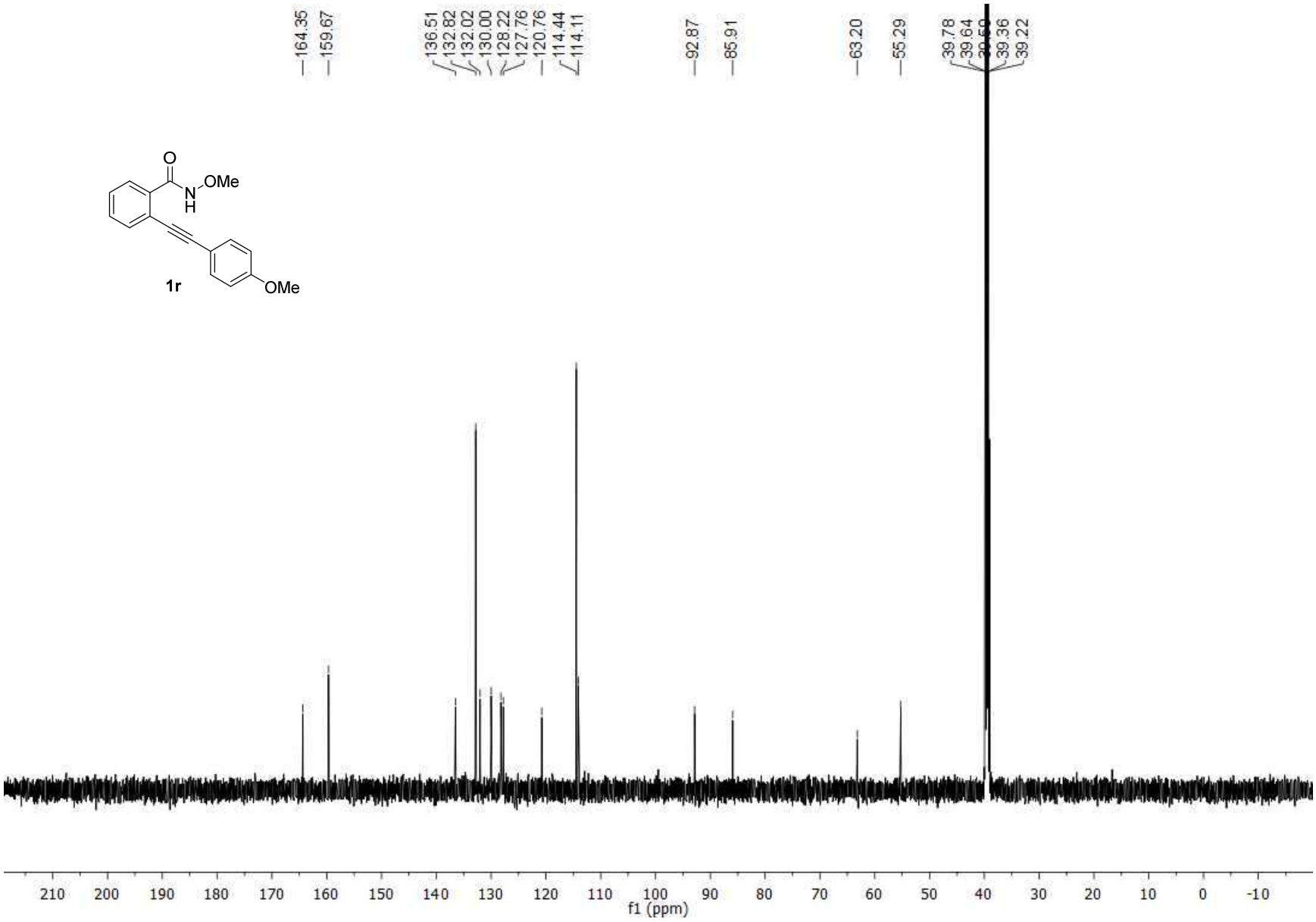
7.48
7.47
7.46
7.45
7.44
7.42
7.01
7.00
3.80
3.73
3.37
2.50
O
N
H
OMe
1r
OMe
0.96
1.00
0.98
3.97
1.99
3.00
2.82
f1 (ppm)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130.00
128.22
127.76
120.76
114.44
114.11
92.87
85.91
63.20
55.29
39.78
39.64
39.36
39.22
O
N
H
OMe
1r
OMe
210
200
190
180
170
160
150
140
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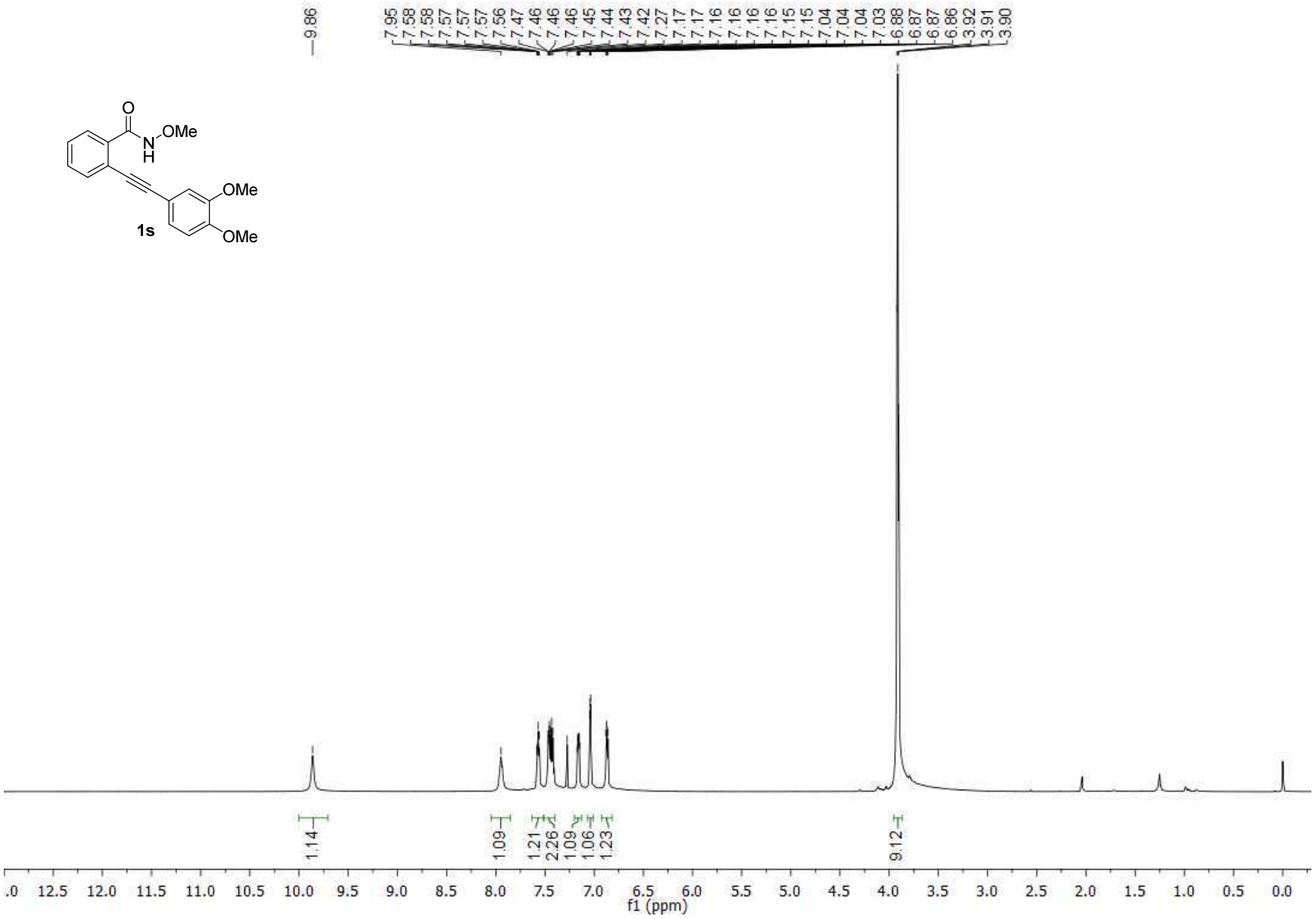
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)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7.56
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7.46
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7.46
7.45
7.44
7.43
7.42
7.27
7.17
7.17
7.16
7.16
7.16
7.16
7.15
7.15
7.04
7.04
7.04
7.03
6.88
6.87
6.87
6.86
3.92
3.91
3.90
O
N
H
OMe
OMe
OMe
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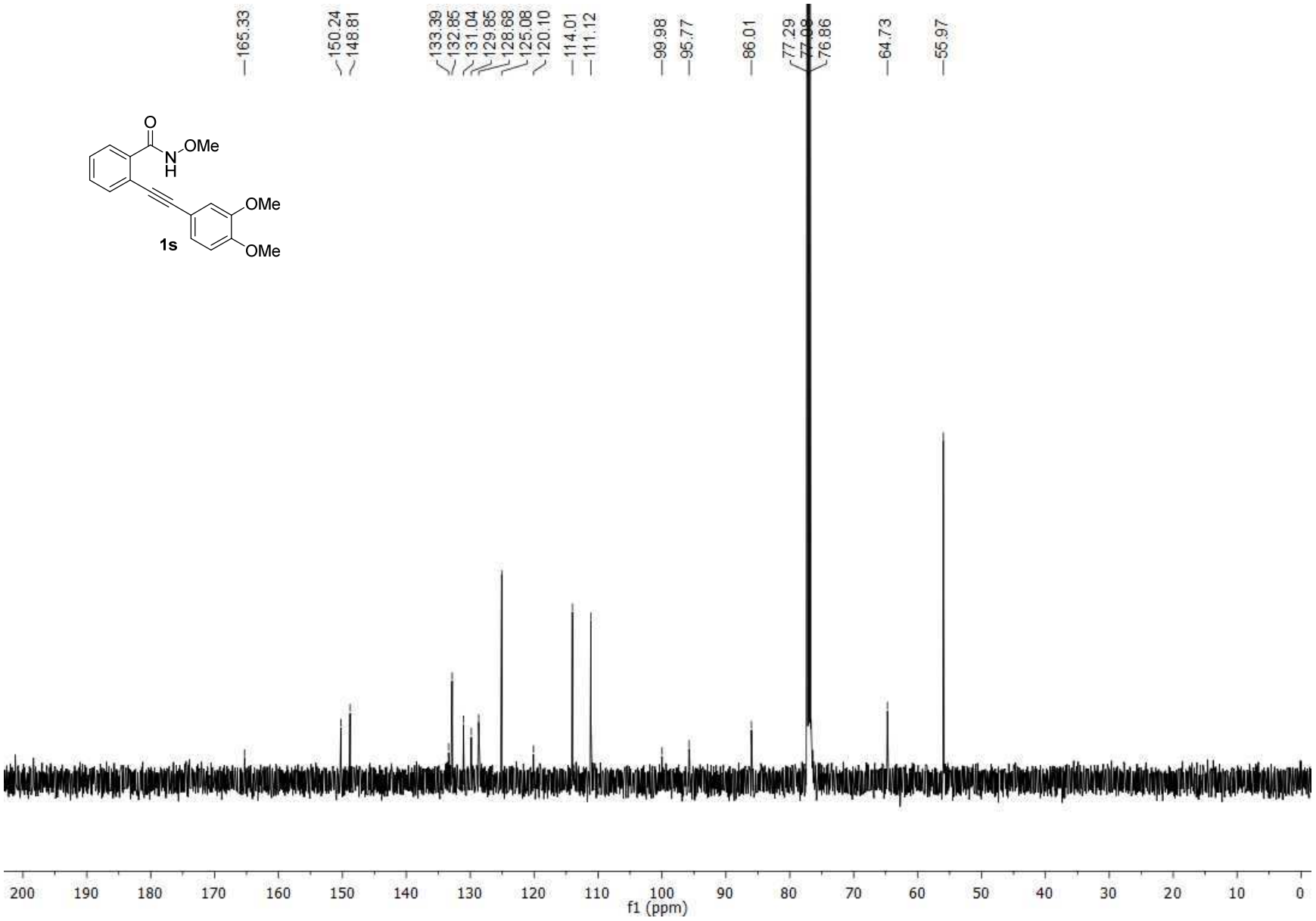
1.14
1.09
1.21
2.26
1.09
1.06
1.23
9.12
f1 (ppm)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131.04
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125.08
120.10
114.01
111.12
99.98
95.77
86.01
77.29
77.08
76.86
64.73
55.97
O
N
H
OMe
OMe
OMe
1s
200
190
180
170
160
150
140
130
120
110
100
90
80
70
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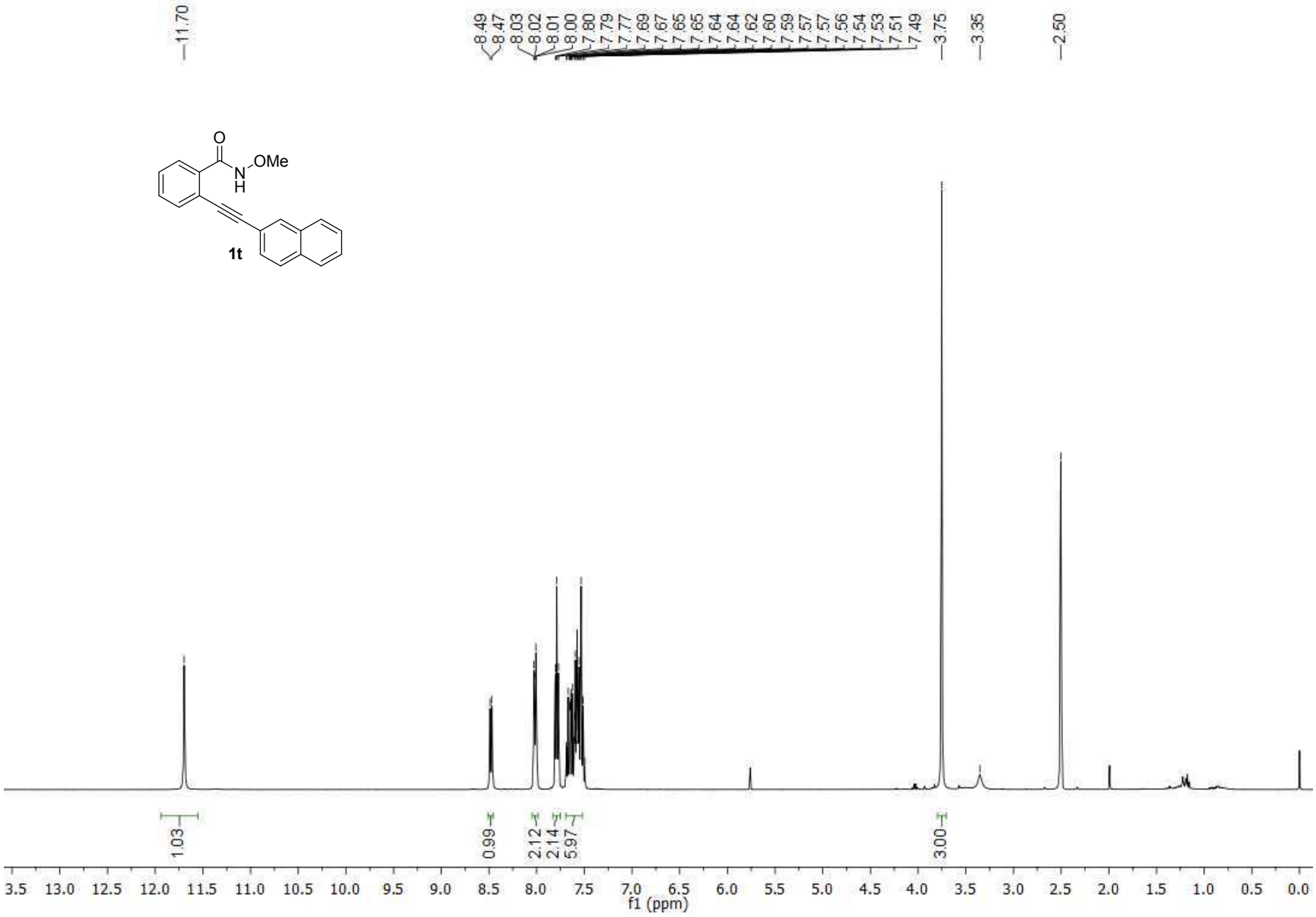
50
40
30
20
10
0
f1 (ppm)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8.49
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7.77
7.69
7.67
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7.64
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7.60
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7.57
7.57
7.56
7.54
7.53
7.51
7.49
3.75
3.35
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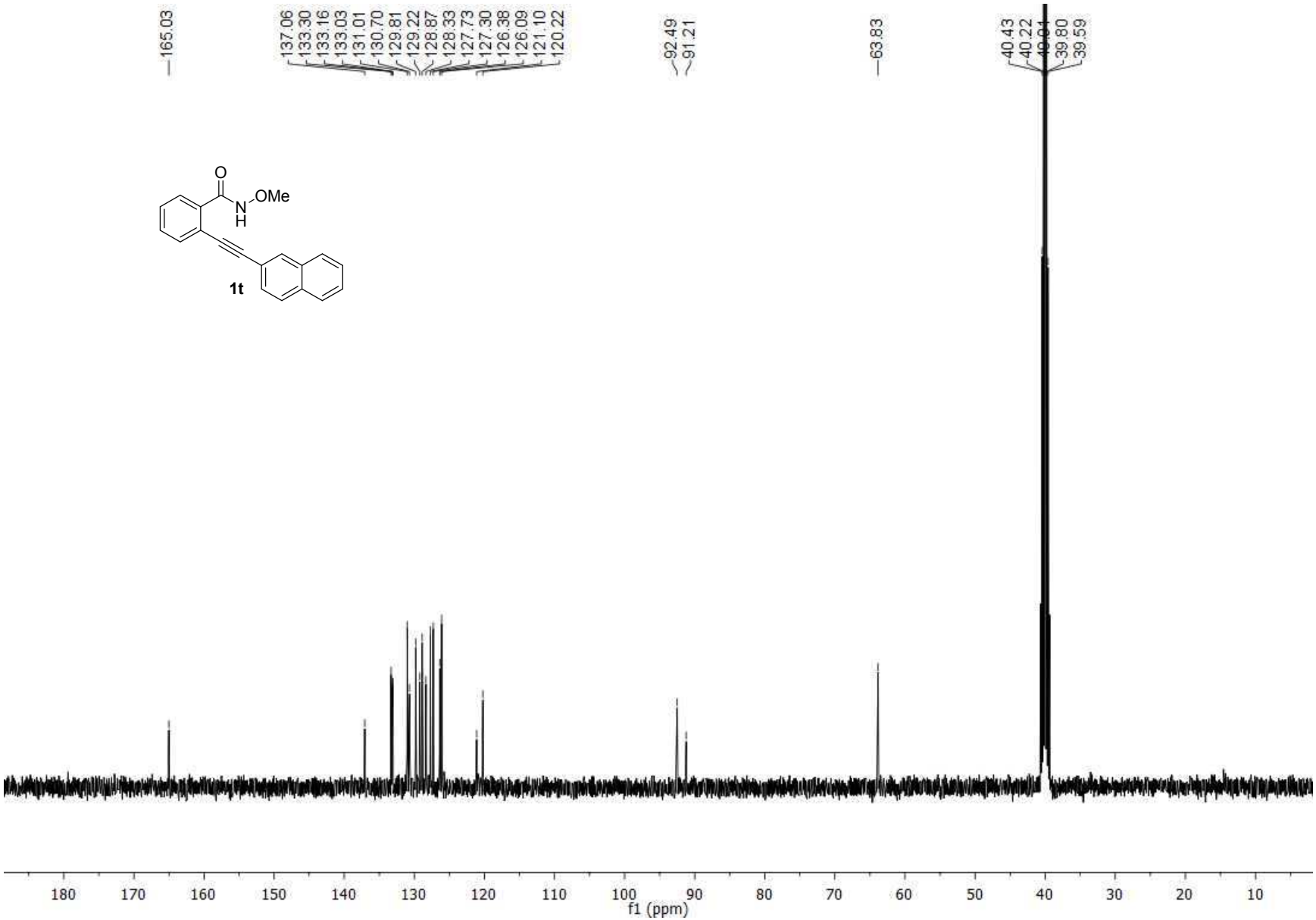
N
H
OMe
1t
1.03
0.99
2.12
2.14
5.97
3.00
f1 (ppm)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128.33
127.73
127.30
126.38
126.09
121.10
120.22
92.49
91.21
63.83
40.43
40.22
40.01
39.80
39.59
O
N
H
OMe
1t
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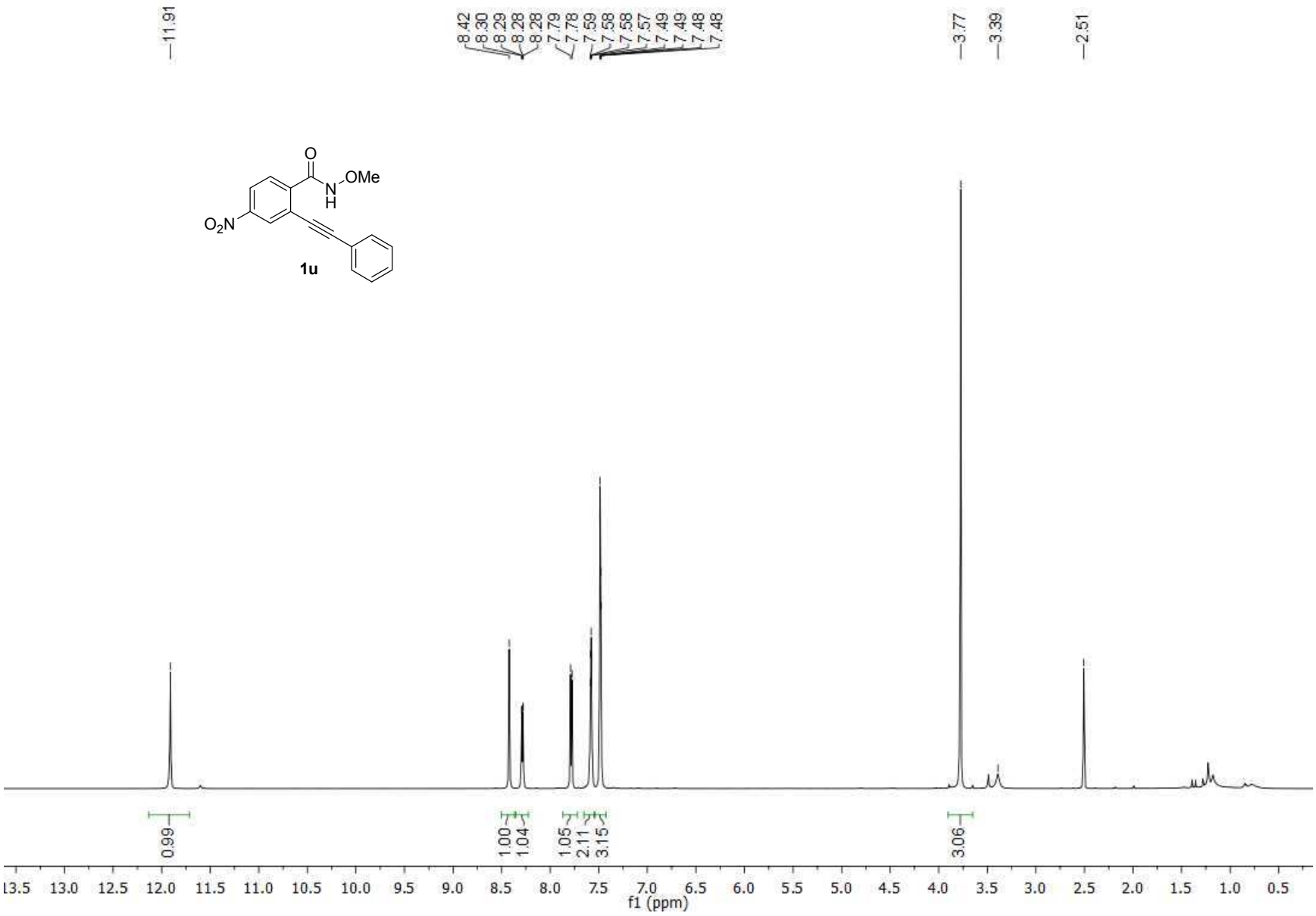
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)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7.58
7.58
7.57
7.49
7.49
7.48
7.48
3.77
3.39
2.51
O
N
OMe
H
O2N
1u
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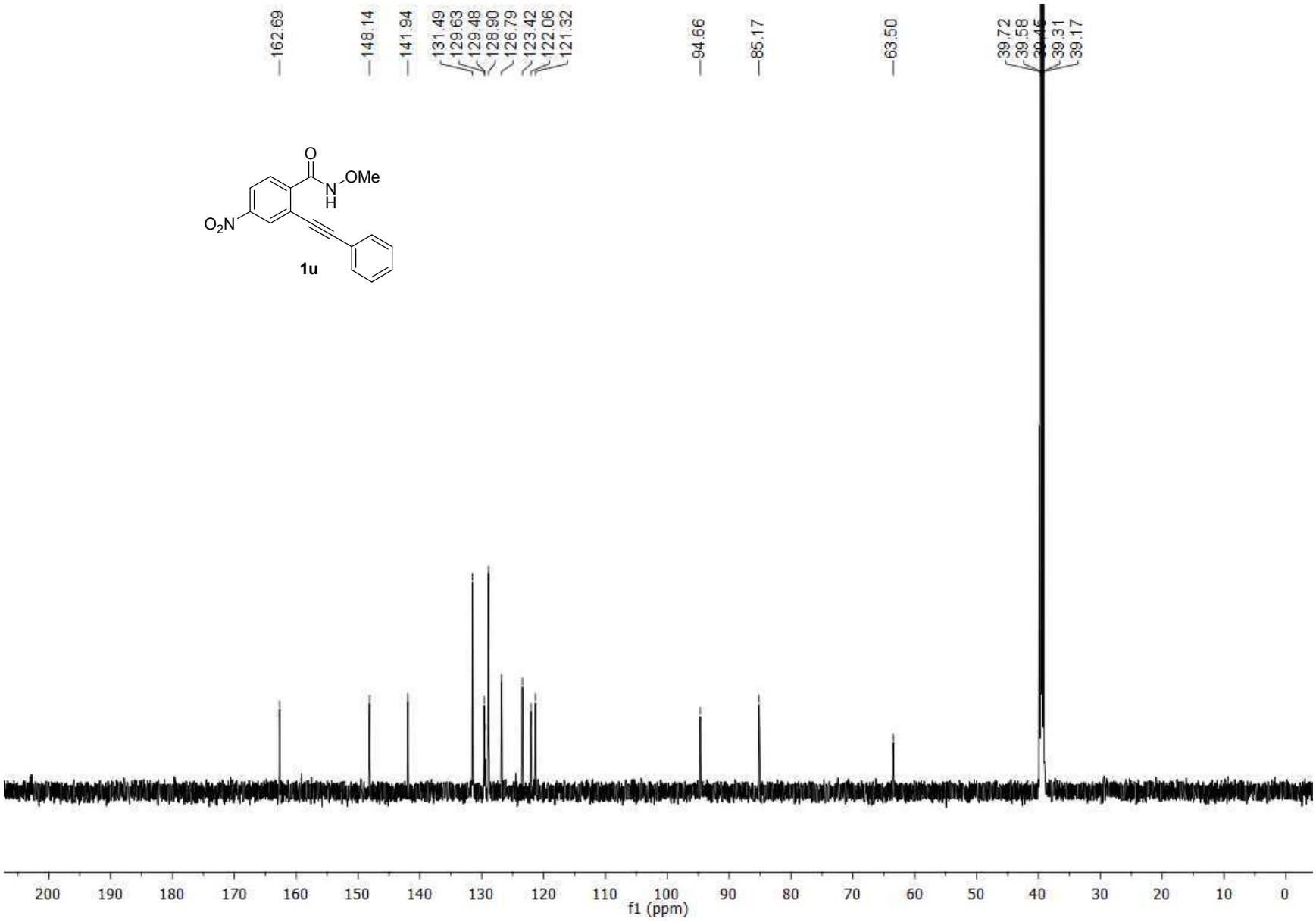
1.00
1.04
1.05
2.11
3.15
3.06
f1 (ppm)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126.79
123.42
122.06
121.32
94.66
85.17
63.50
39.72
39.58
39.46
39.31
39.17
O
N
H
OMe
O2N
1u
200
190
180
170
160
150
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130
120
110
100
90
80
70
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50
40
30
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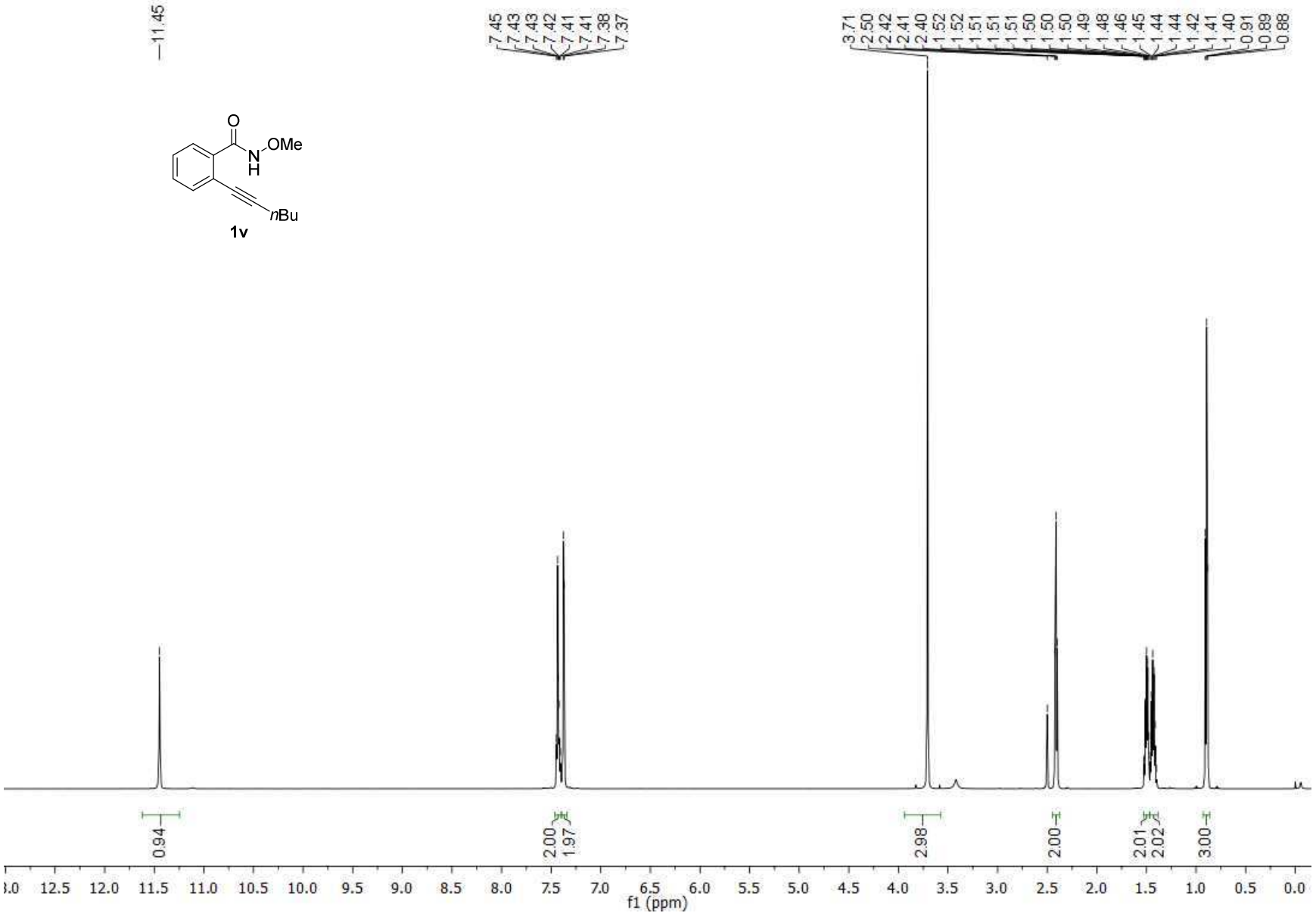
10
0
f1 (ppm)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7.38
7.37
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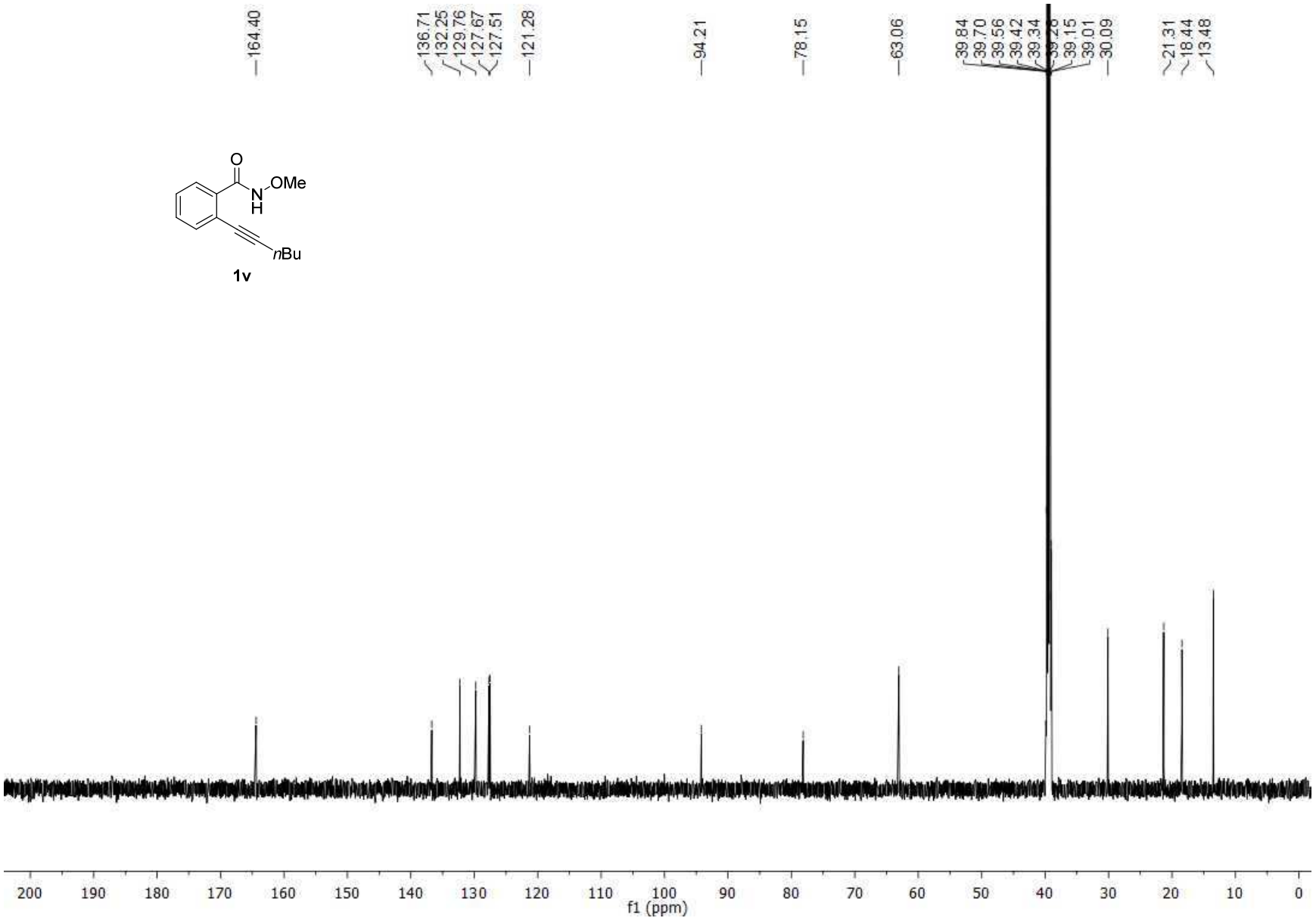
2.42
2.41
2.40
1.52
1.52
1.51
1.51
1.51
1.50
1.50
1.50
1.49
1.48
1.46
1.45
1.44
1.44
1.42
1.41
1.40
0.91
0.89
0.88
O
N
H
OMe
nBu
1v
0.94
2.00
1.97
2.98
2.00
2.01
2.02
3.00
f1 (ppm)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39.70
39.56
39.42
39.34
39.28
39.15
39.01
30.09
21.31
18.44
13.48
O
N
H
OMe
nBu
1v
200
190
180
170
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150
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130
120
110
100
90
80
70
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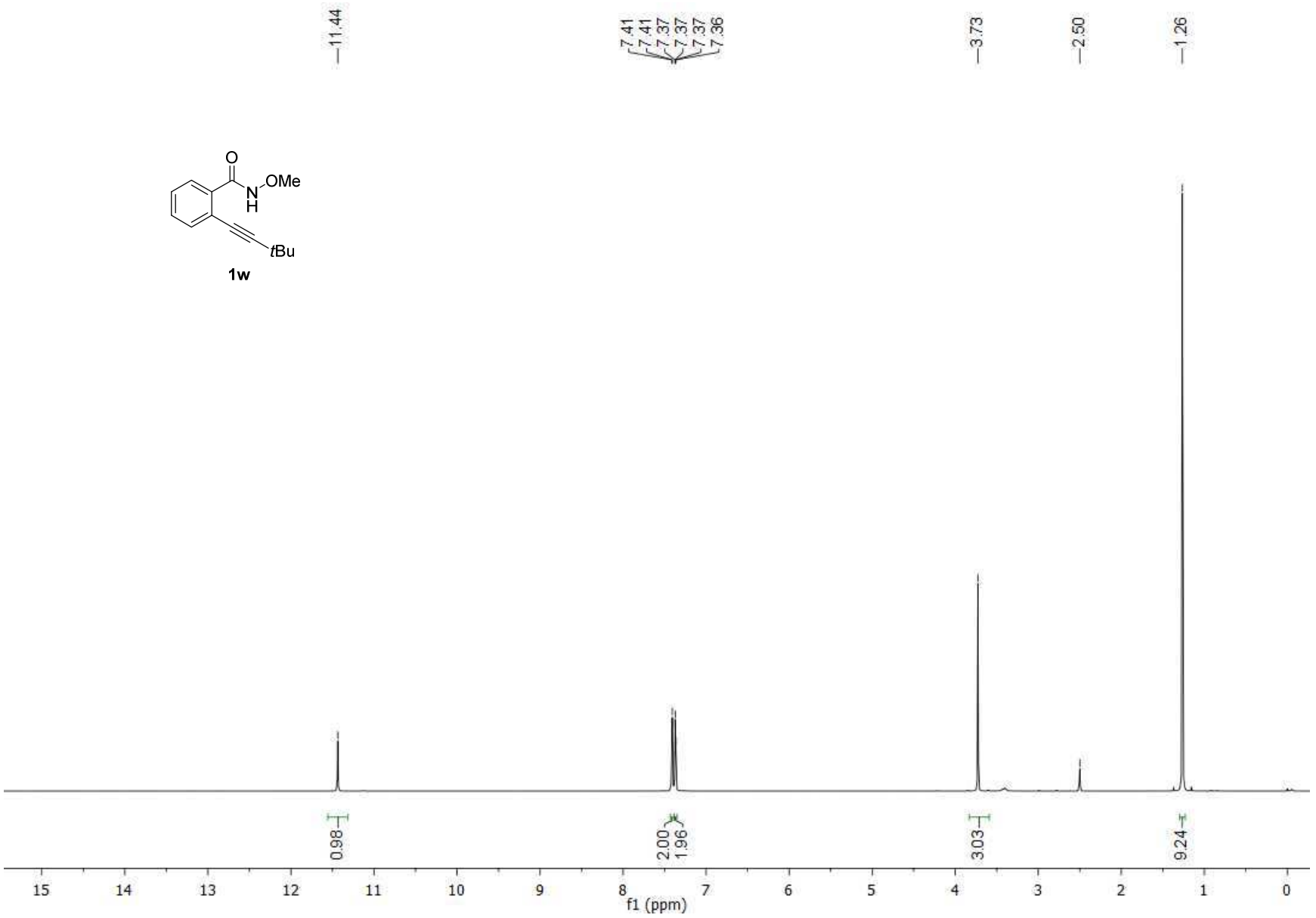
50
40
30
20
10
0
f1 (ppm)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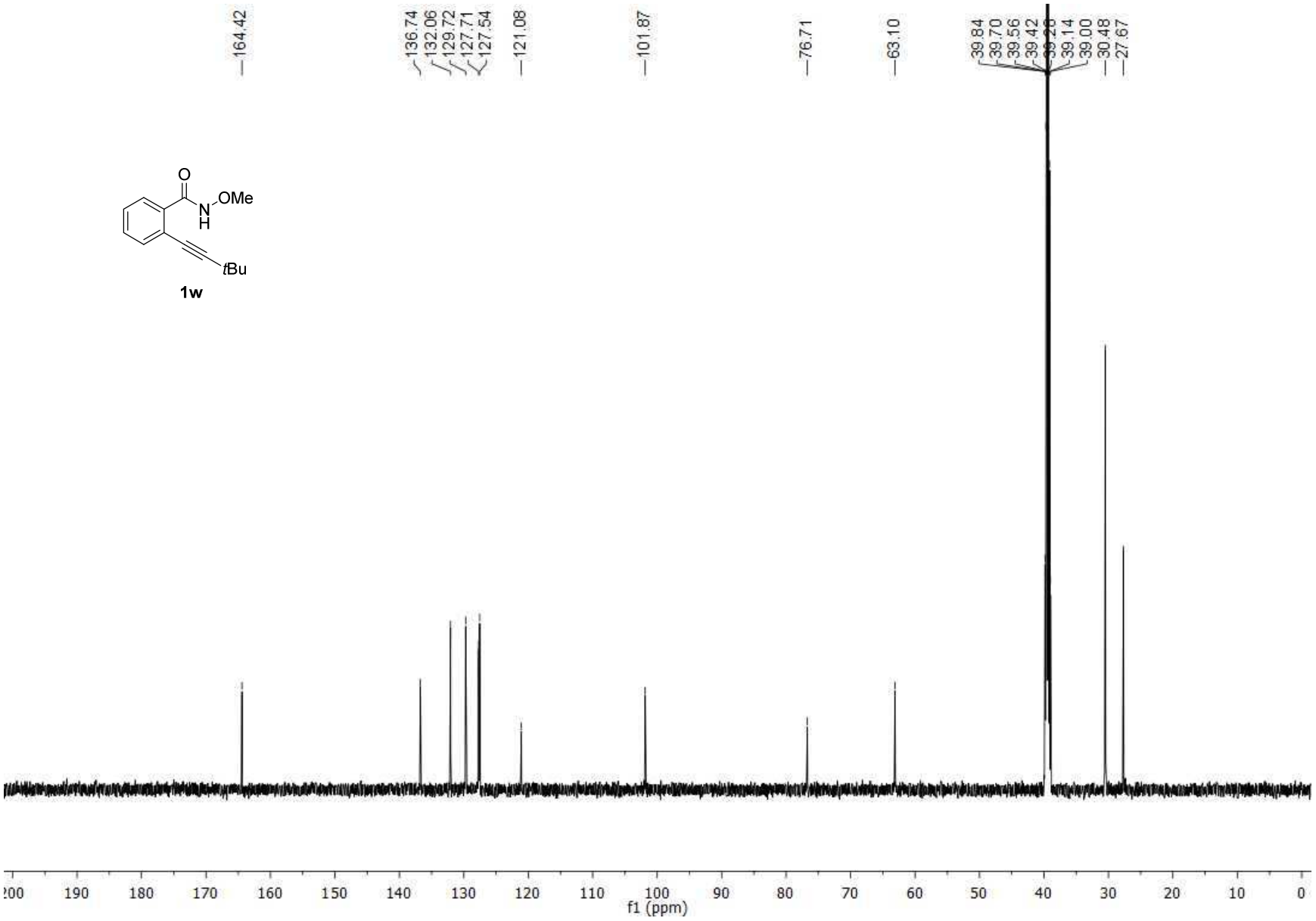
7.37
7.37
7.36
3.73
2.50
1.26
O
N
H
OMe
tBu
1w
0.98
2.00
1.96
3.03
9.24
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
f1 (ppm)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127.54
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101.87
76.71
63.10
39.84
39.70
39.56
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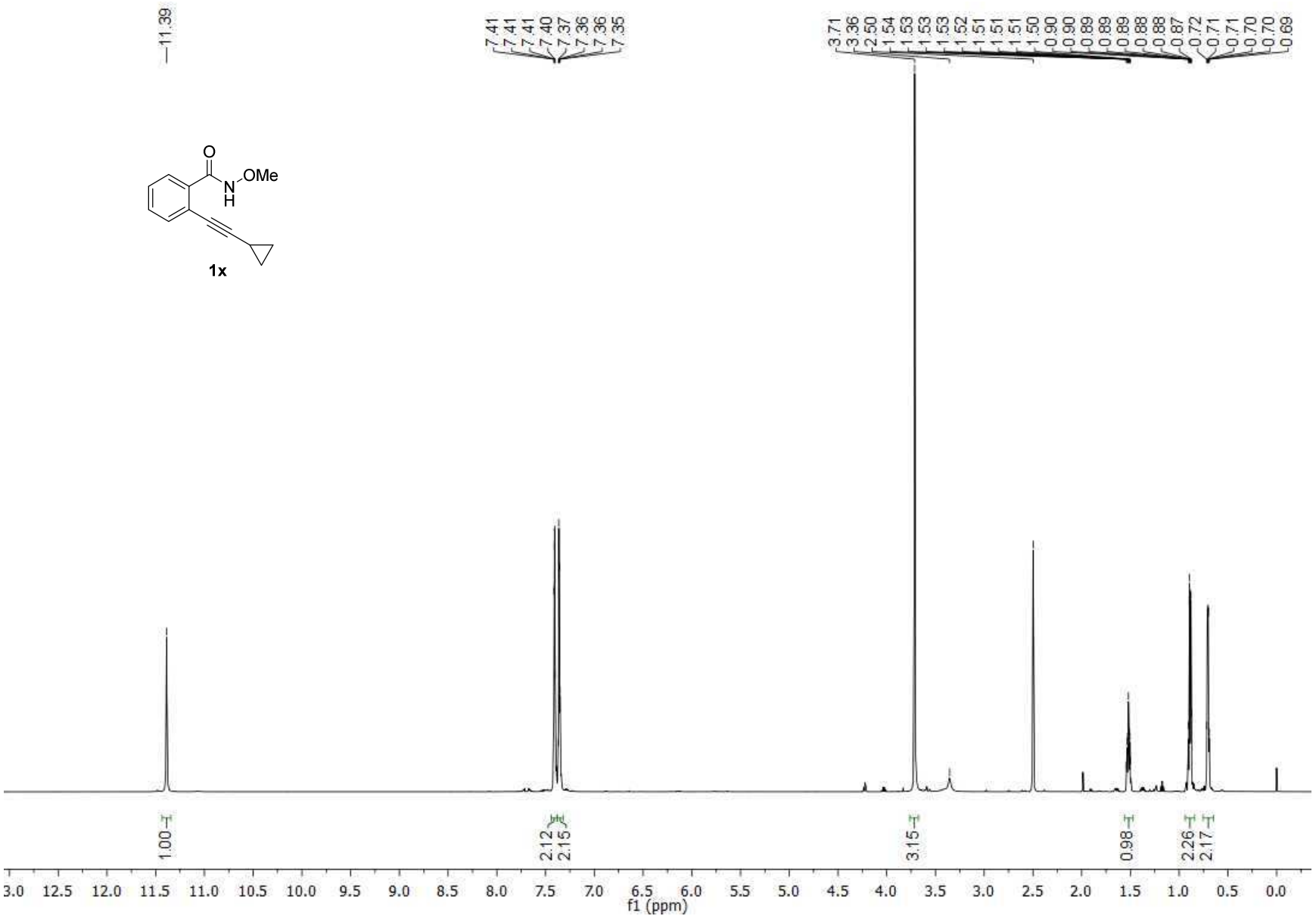
39.28
39.14
39.00
30.48
27.67
O
N
H
OMe
tBu
1w
f1 (ppm)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7.36
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3.71
3.36
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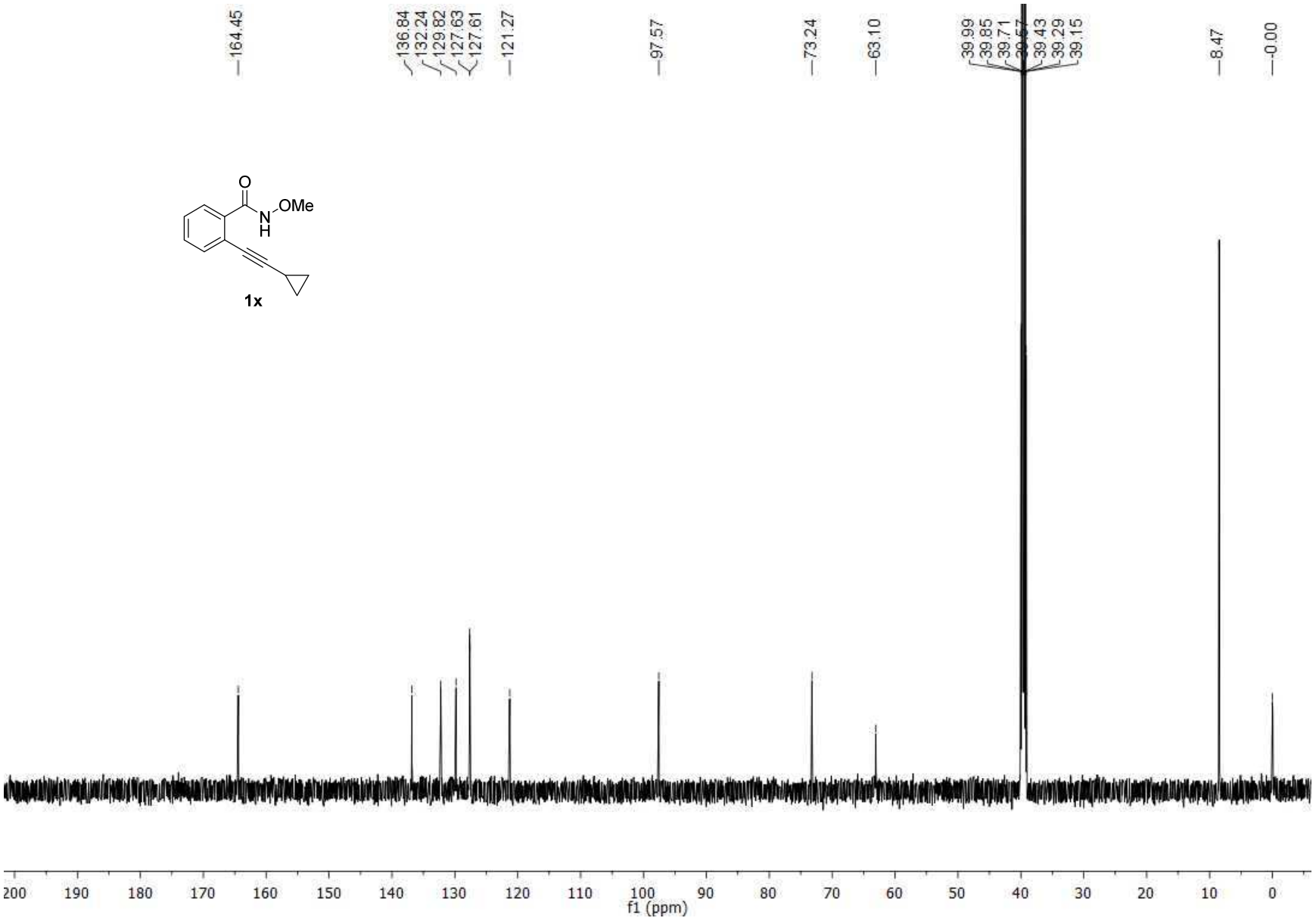
1.54
1.53
1.53
1.53
1.52
1.51
1.51
1.51
1.50
0.90
0.90
0.89
0.89
0.89
0.88
0.88
0.87
0.72
0.71
0.71
0.70
0.70
0.69
O
N
H
OMe
1x
1.00
2.12
2.15
3.15
0.98
2.26
2.17
f1 (ppm)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127.61
121.27
97.57
73.24
63.10
39.99
39.85
39.71
39.43
39.29
39.15
8.47
0.00
O
N
H
OMe
1x
200
190
180
170
160
150
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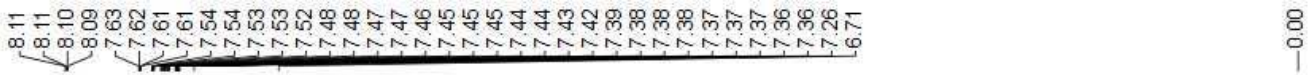
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.61
7.61
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7.54
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7.53
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7.45
7.45
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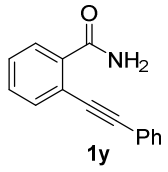
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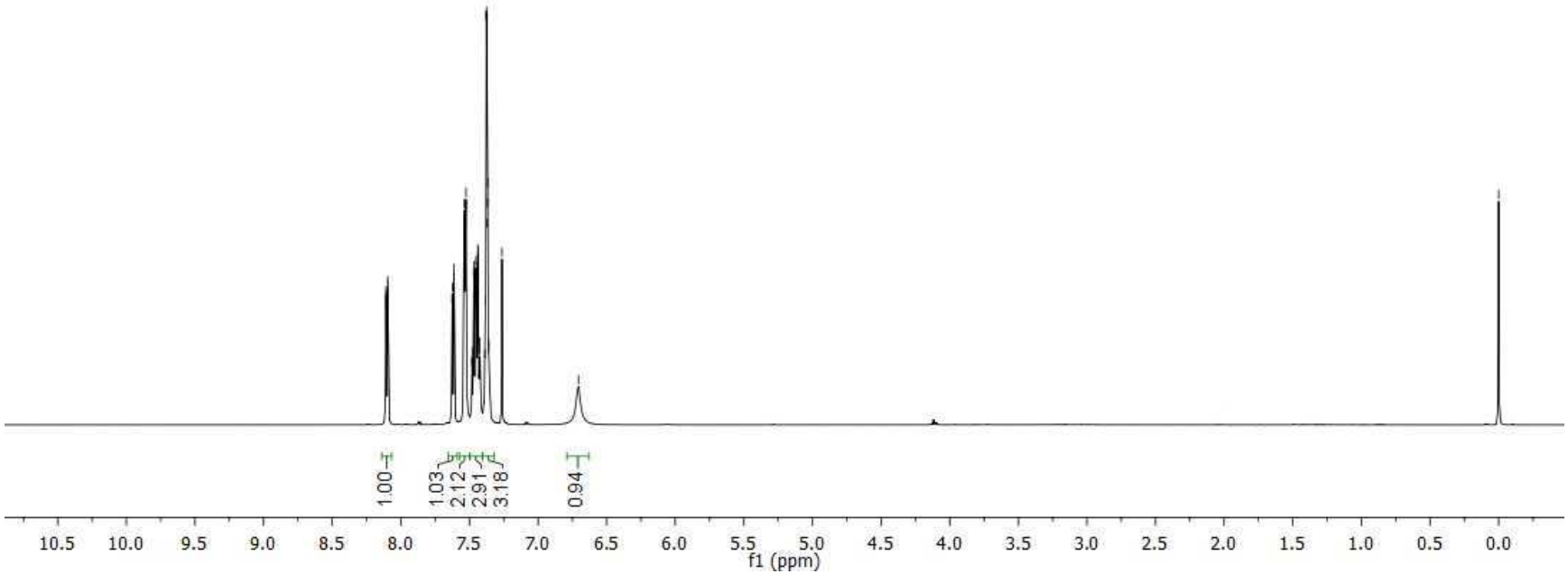
NH2
Ph

1y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7.0
6.5
6.0
5.5
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2.5
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1.0
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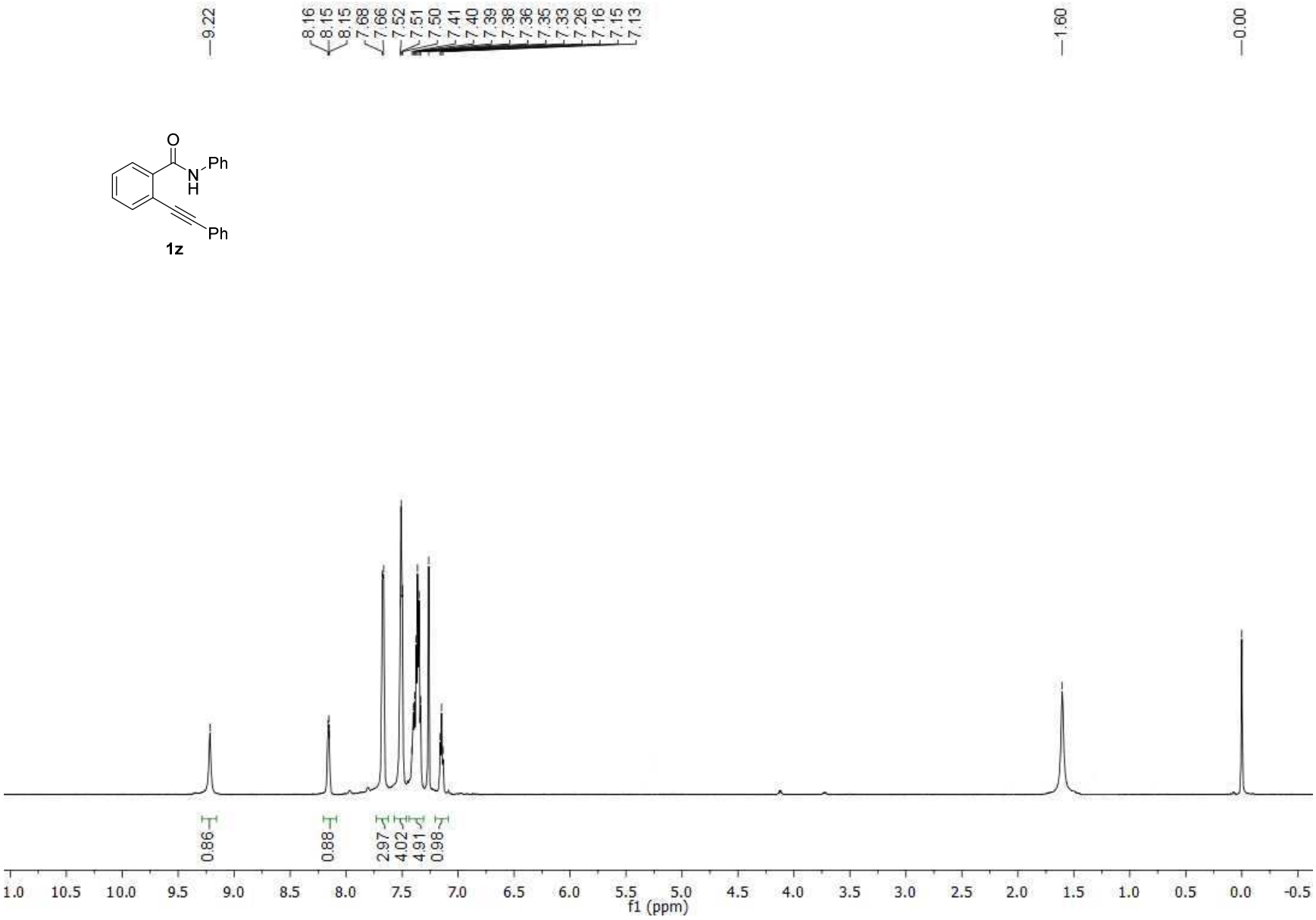
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7.38
7.36
7.35
7.33
7.26
7.16
7.15
7.13
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0.00
O
N
H
Ph
Ph
1z
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0.88
2.97
4.02
4.91
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9.5
9.0
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8.0
7.5
7.0
6.5
6.0
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5.0
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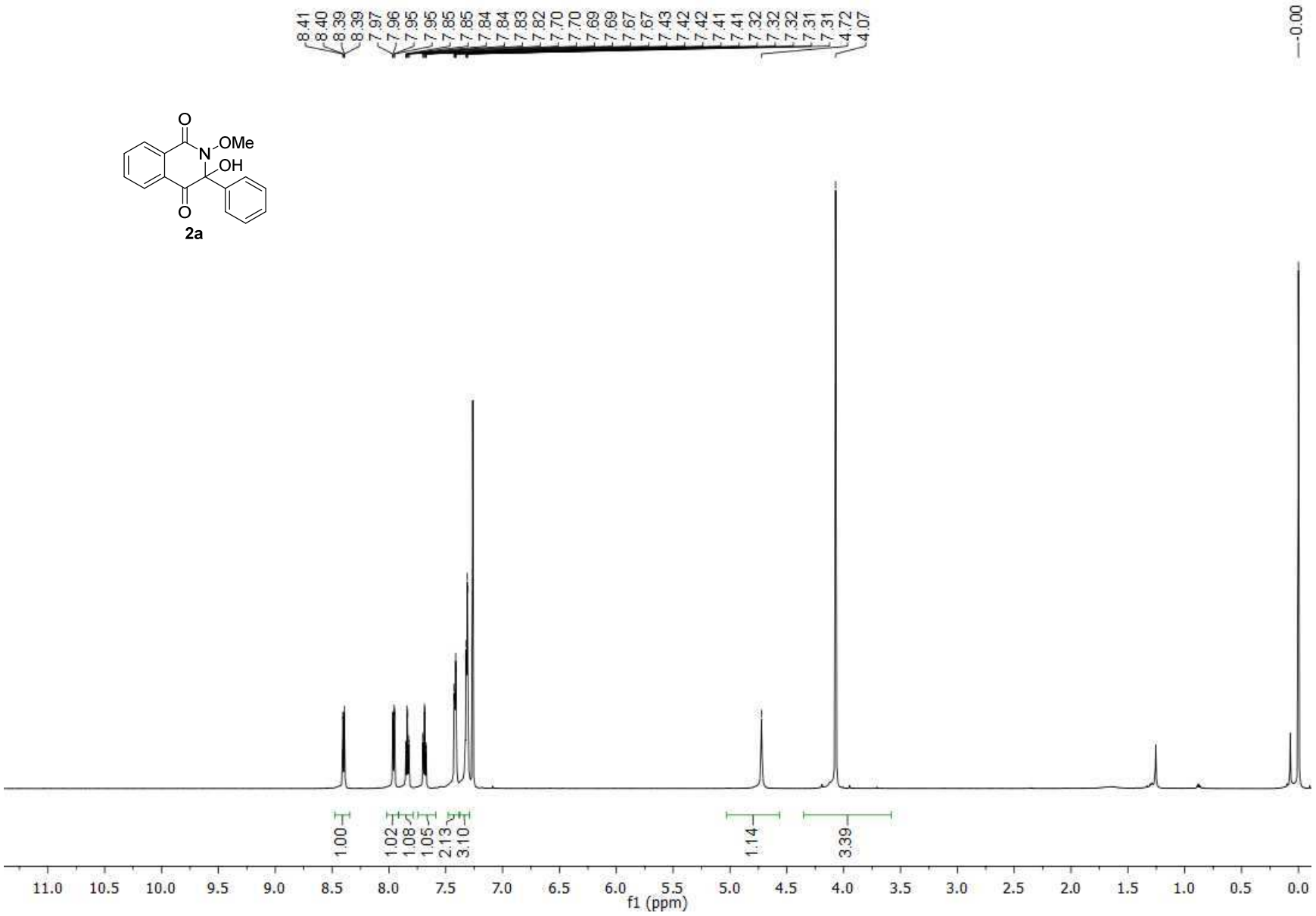
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7.83
7.82
7.70
7.70
7.69
7.69
7.67
7.67
7.43
7.42
7.42
7.41
7.41
7.32
7.32
7.31
7.31
4.72
4.07
-0.00
O
N
OMe
OH
O
2a
1.00
1.02
1.08
1.05
2.13
3.10
1.14
3.39
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
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6.0
5.5
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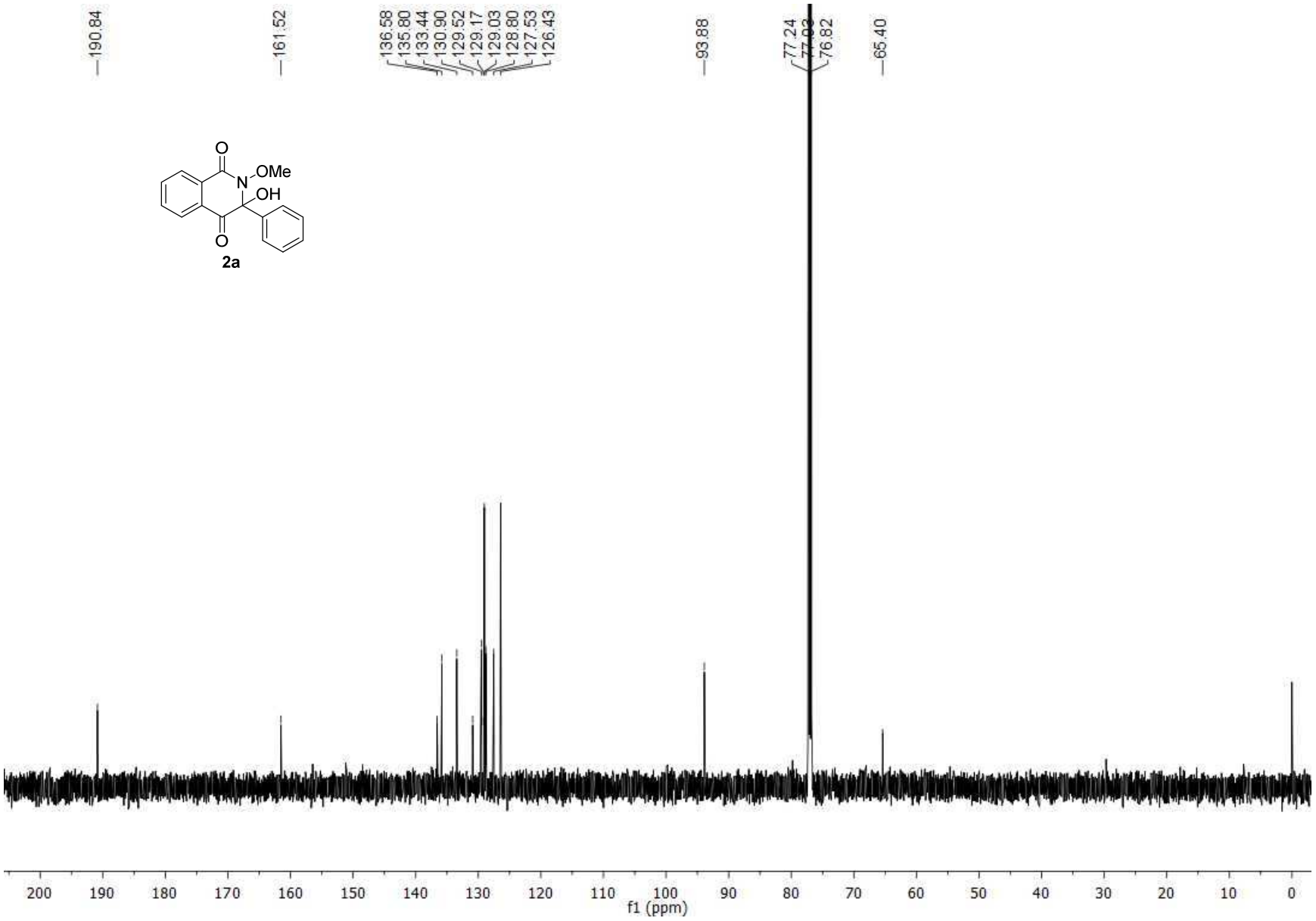
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127.53
126.43
93.88
77.24
77.03
76.82
65.40
O
N
OMe
OH
O
2a
200
190
180
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150
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130
120
110
100
90
80
70
60
50
40
30
20
10
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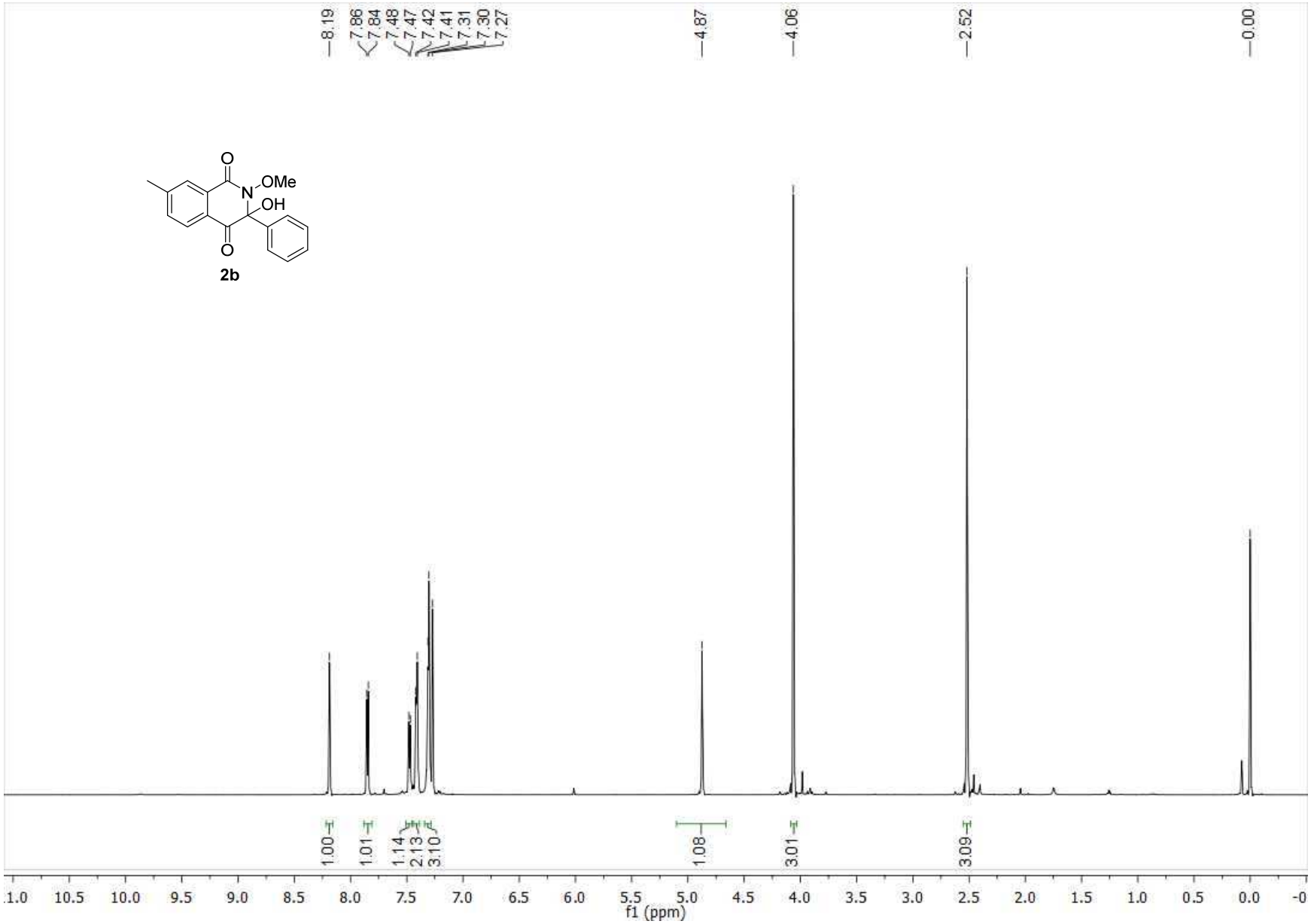
f1 (ppm)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O
N
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OH
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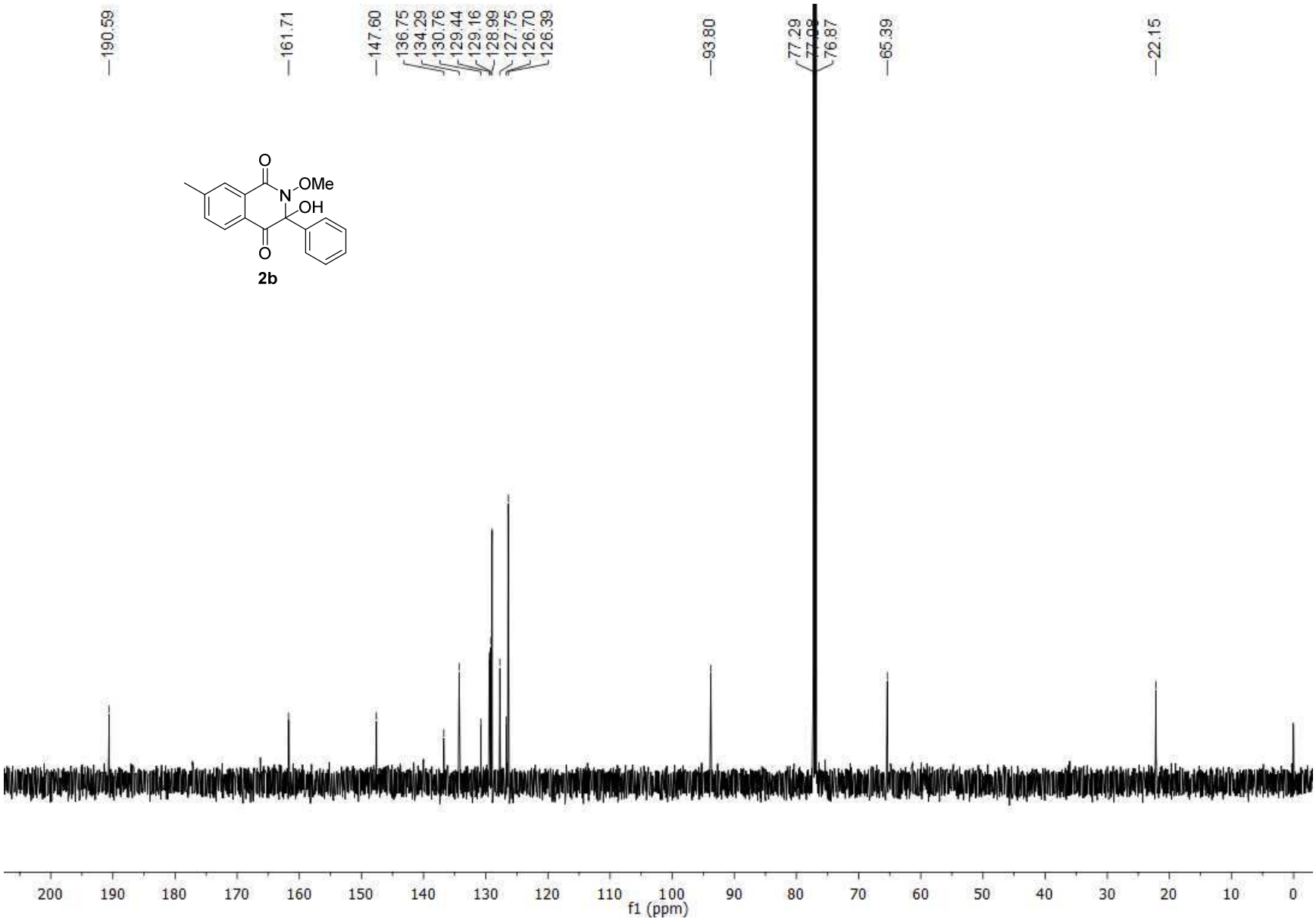
1.01
1.14
2.13
3.10
1.08
3.01
3.09
f1 (ppm)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126.39
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77.29
76.87
65.39
22.15
O
N
OMe
OH
O
2b
200
190
180
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150
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130
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110
100
90
80
70
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50
40
30
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10
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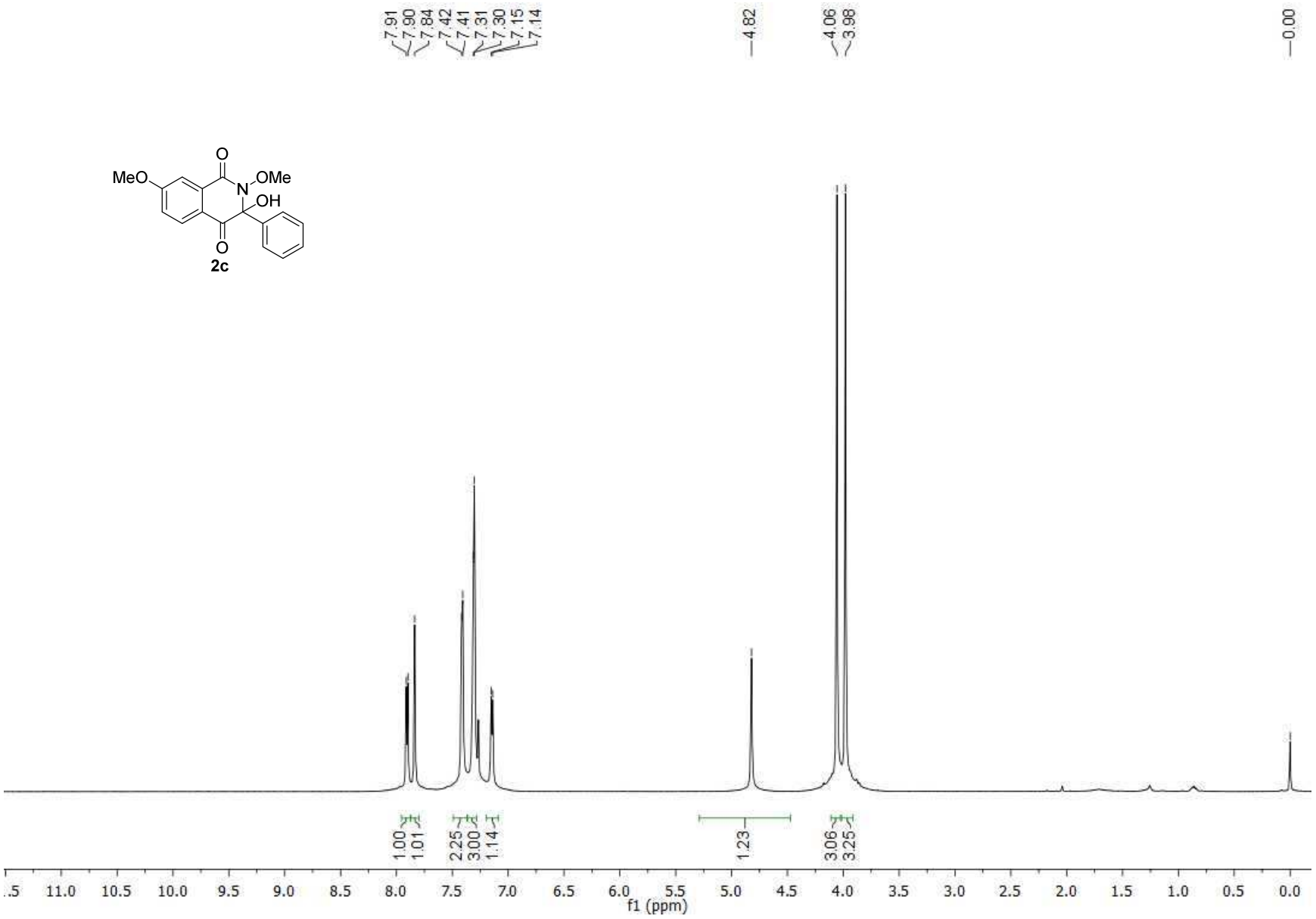
f1 (ppm)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7.15
7.14
4.82
4.06
3.98
0.00
MeO
OMe
N
OH
O
O
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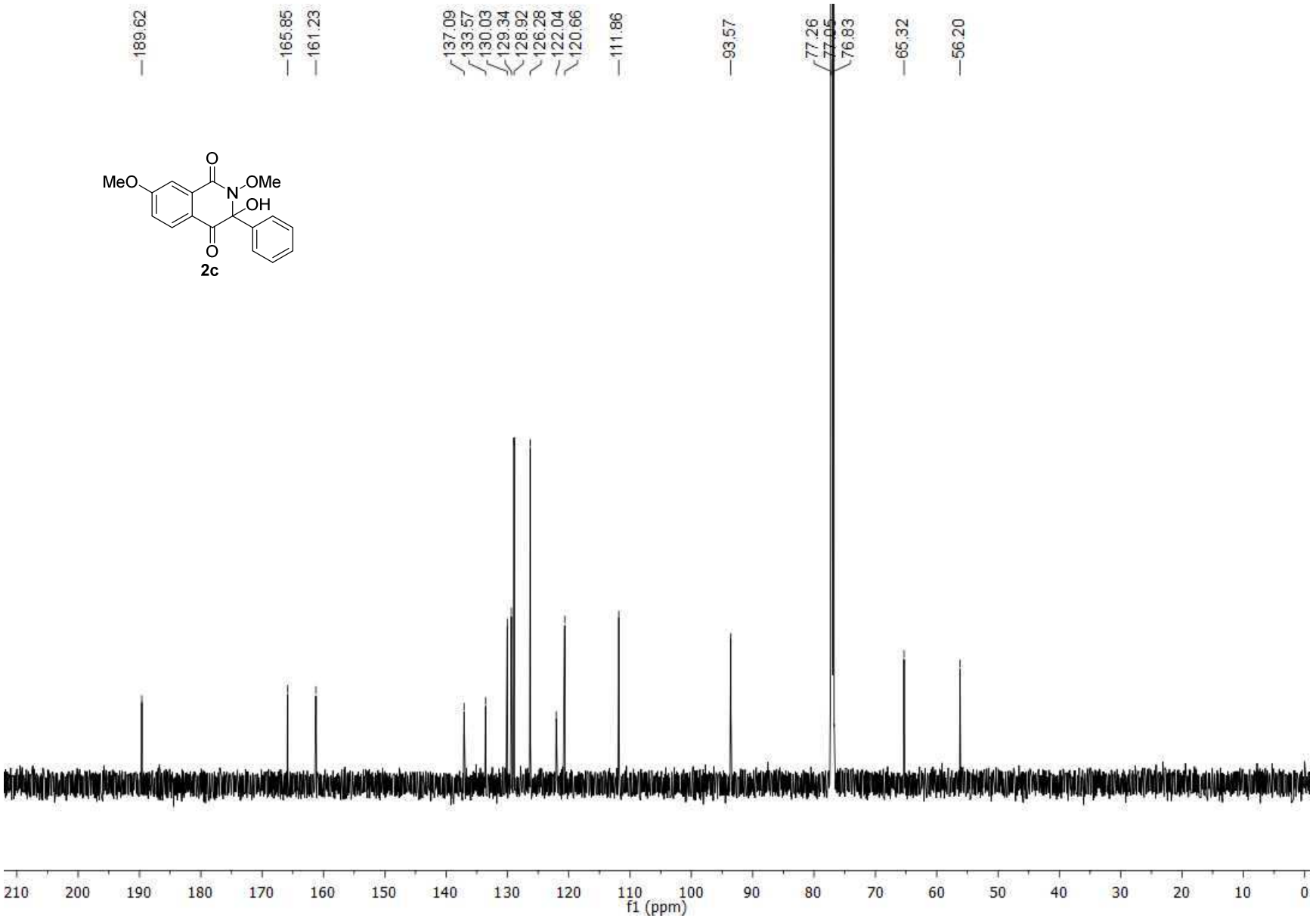
1.00
1.01
2.25
3.00
1.14
1.23
3.06
3.25
f1 (ppm)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122.04
120.66
111.86
93.57
77.26
77.05
76.83
65.32
56.20
MeO
OMe
N
OH
O
O
2c
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
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50
40
30
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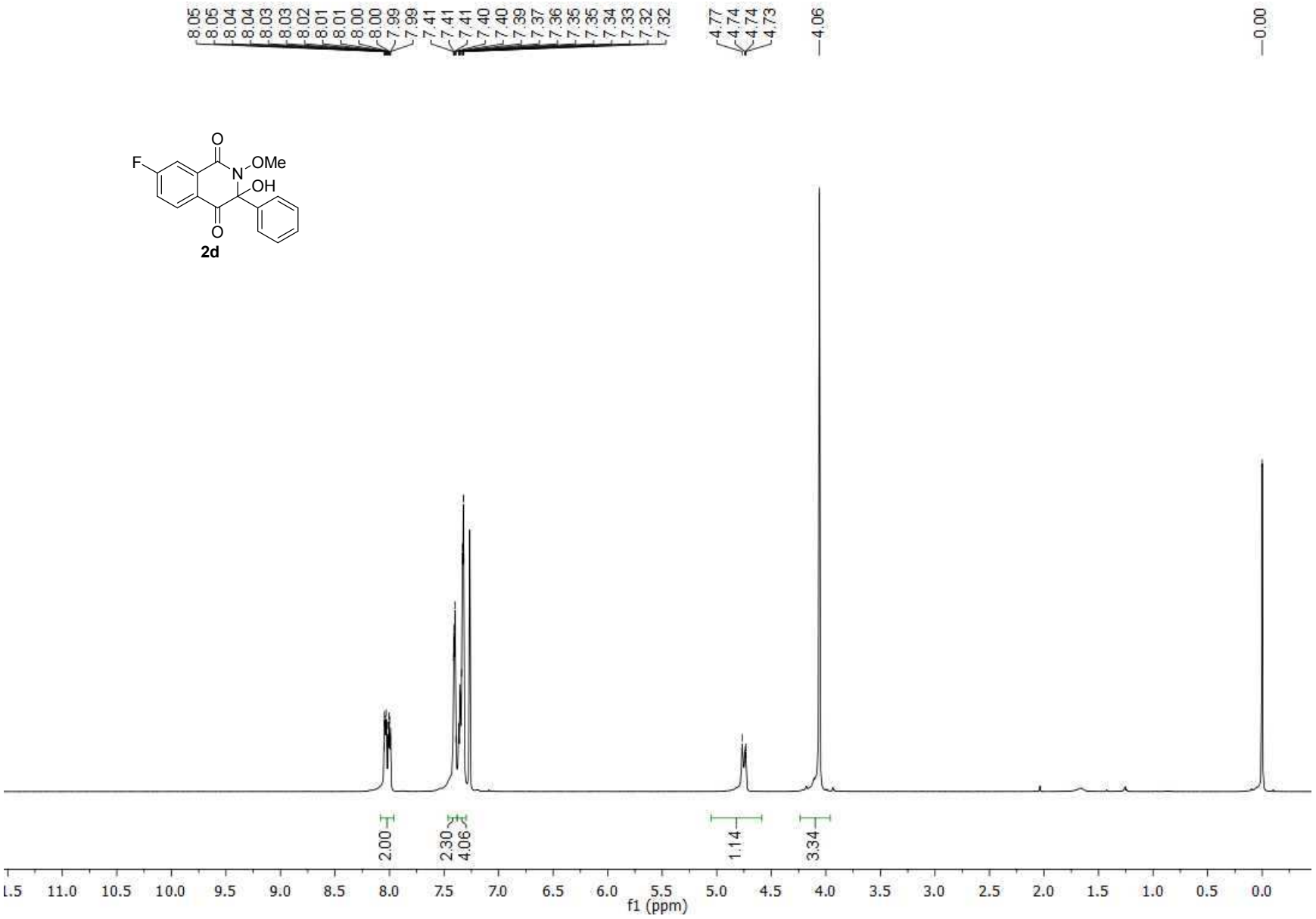
10
0
f1 (ppm)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8.00
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7.41
7.41
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7.40
7.39
7.37
7.36
7.35
7.35
7.34
7.33
7.32
7.32
4.77
4.74
4.74
4.73
4.06
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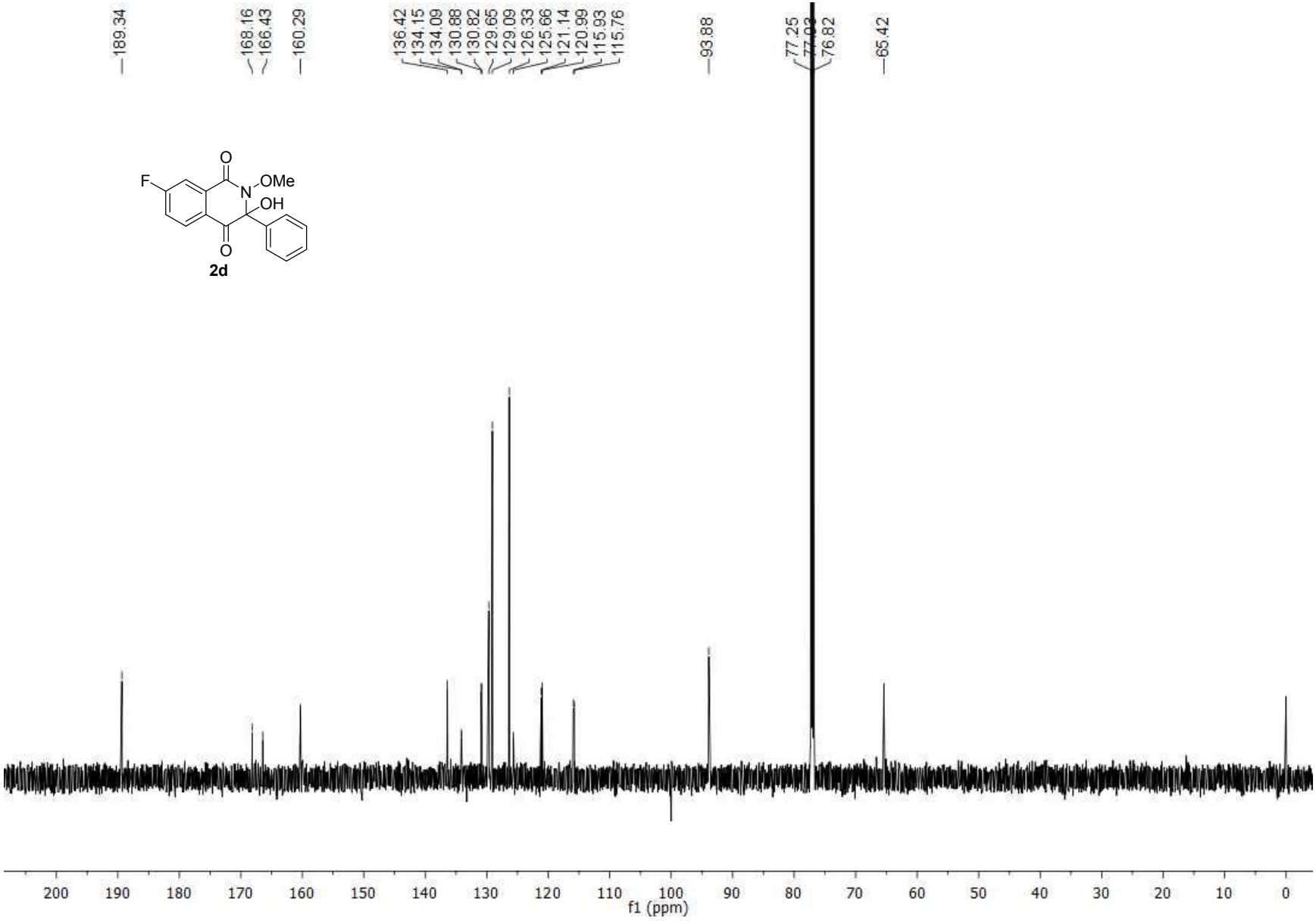
N
OMe
OH
O
2d
2.00
2.30
4.06
1.14
3.34
f1 (ppm)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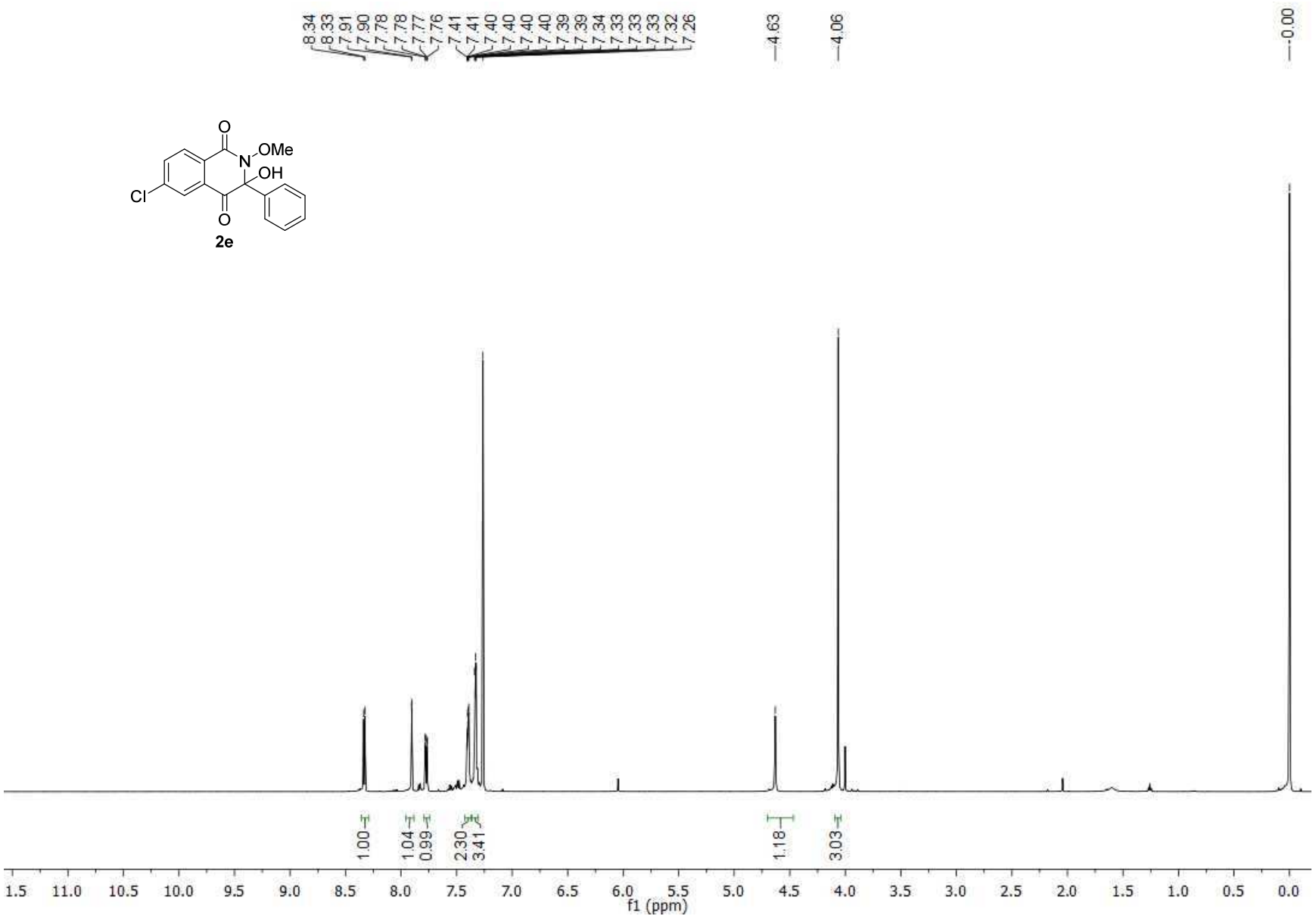
121.14
120.99
115.93
115.76
93.88
77.25
77.03
76.82
65.42
F
O
N
OMe
OH
O
2d
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.41
7.40
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7.39
7.39
7.34
7.33
7.33
7.33
7.32
7.26
4.63
4.06
0.00
O
N
OMe
OH
Cl
O
2e
1.00
1.04
0.99
2.30
3.41
1.18
3.03
1.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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4.5
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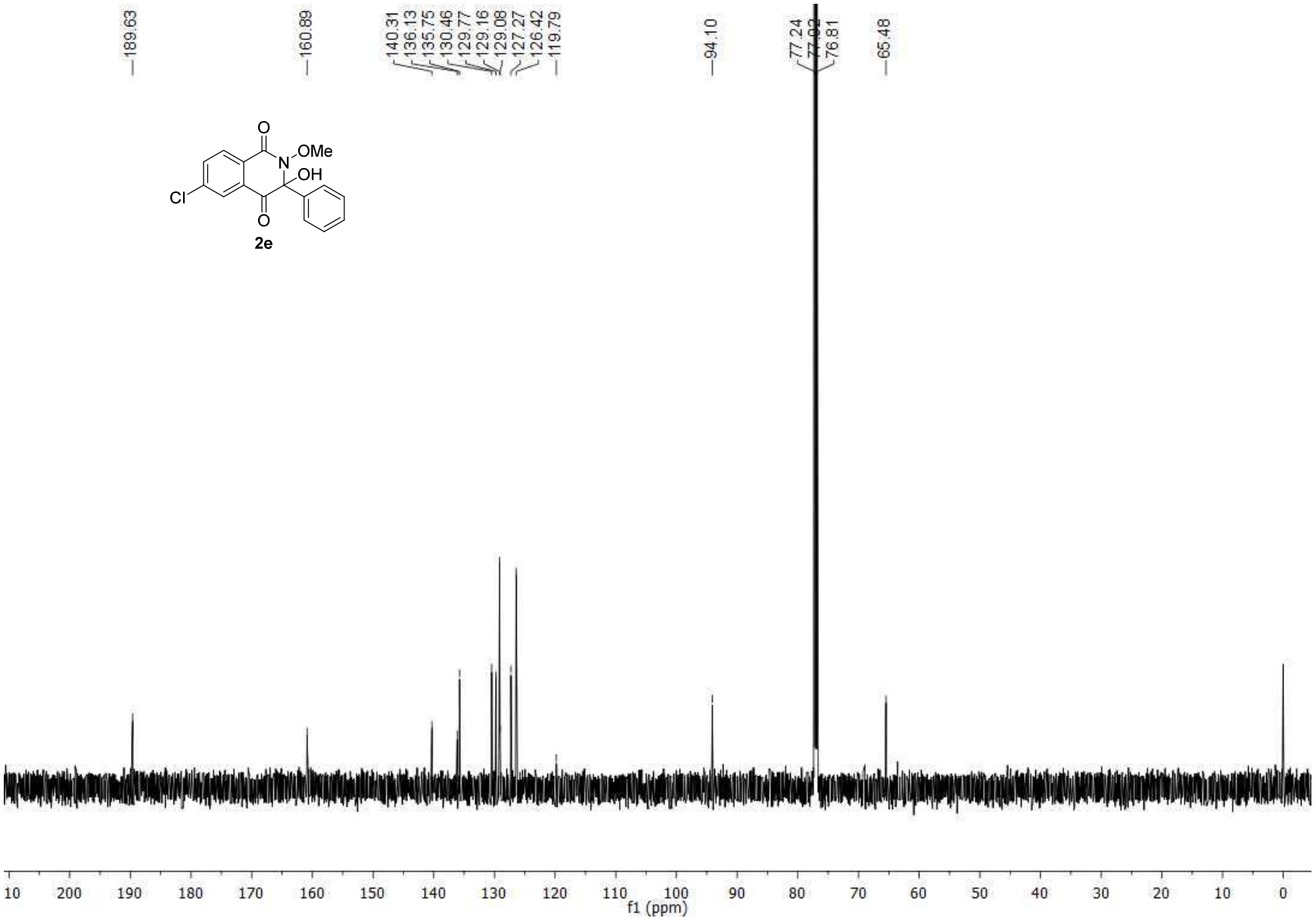
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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127.27
126.42
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94.10
77.24
77.02
76.81
65.48
O
N
OMe
OH
Cl
O
2e
10
200
190
180
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150
140
130
120
110
100
90
80
70
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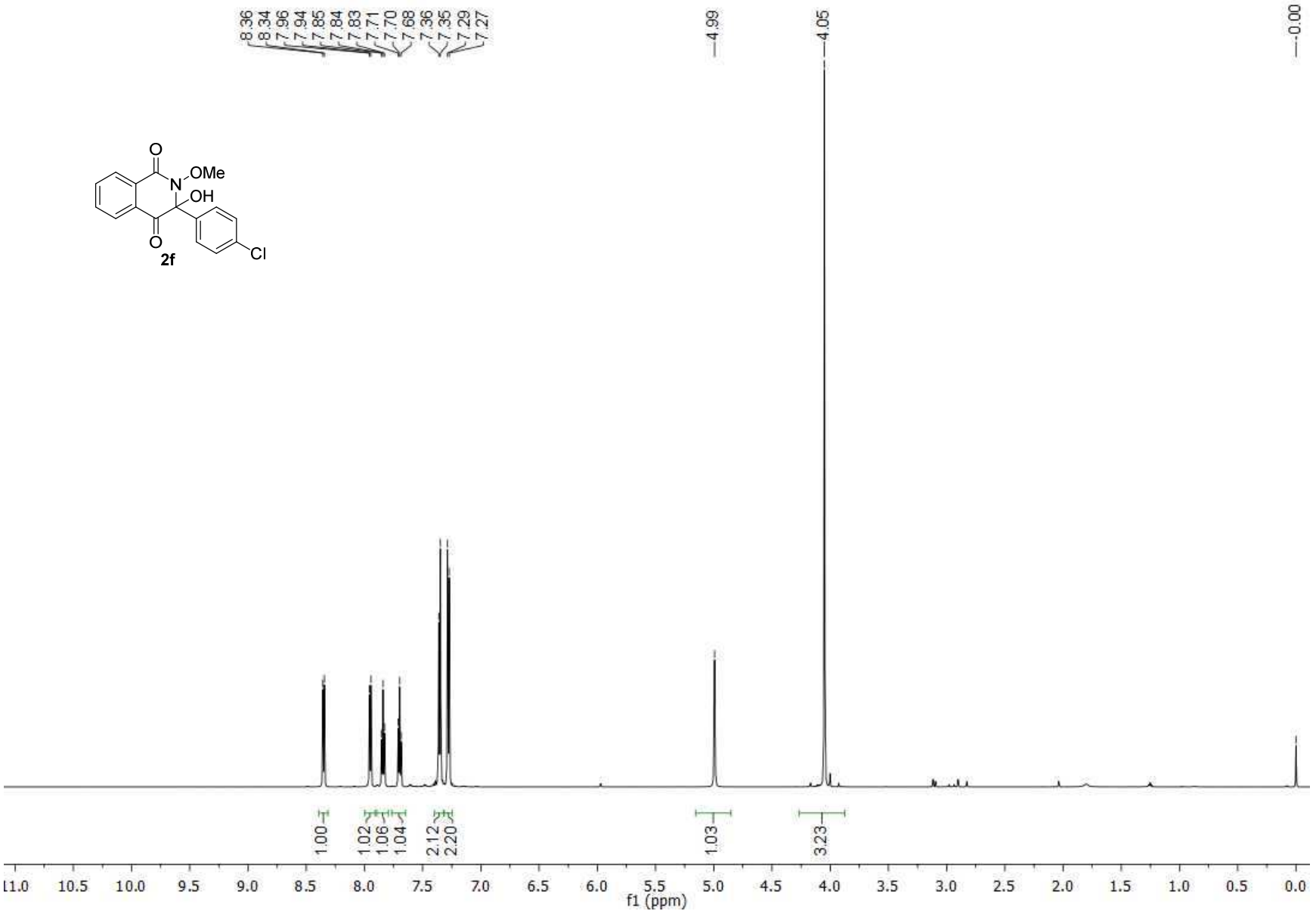
50
40
30
20
10
0
f1 (ppm)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7.29
7.27
4.99
4.05
0.00
O
N
OMe
OH
O
Cl
2f
1.00
1.02
1.06
1.04
2.12
2.20
1.03
3.23
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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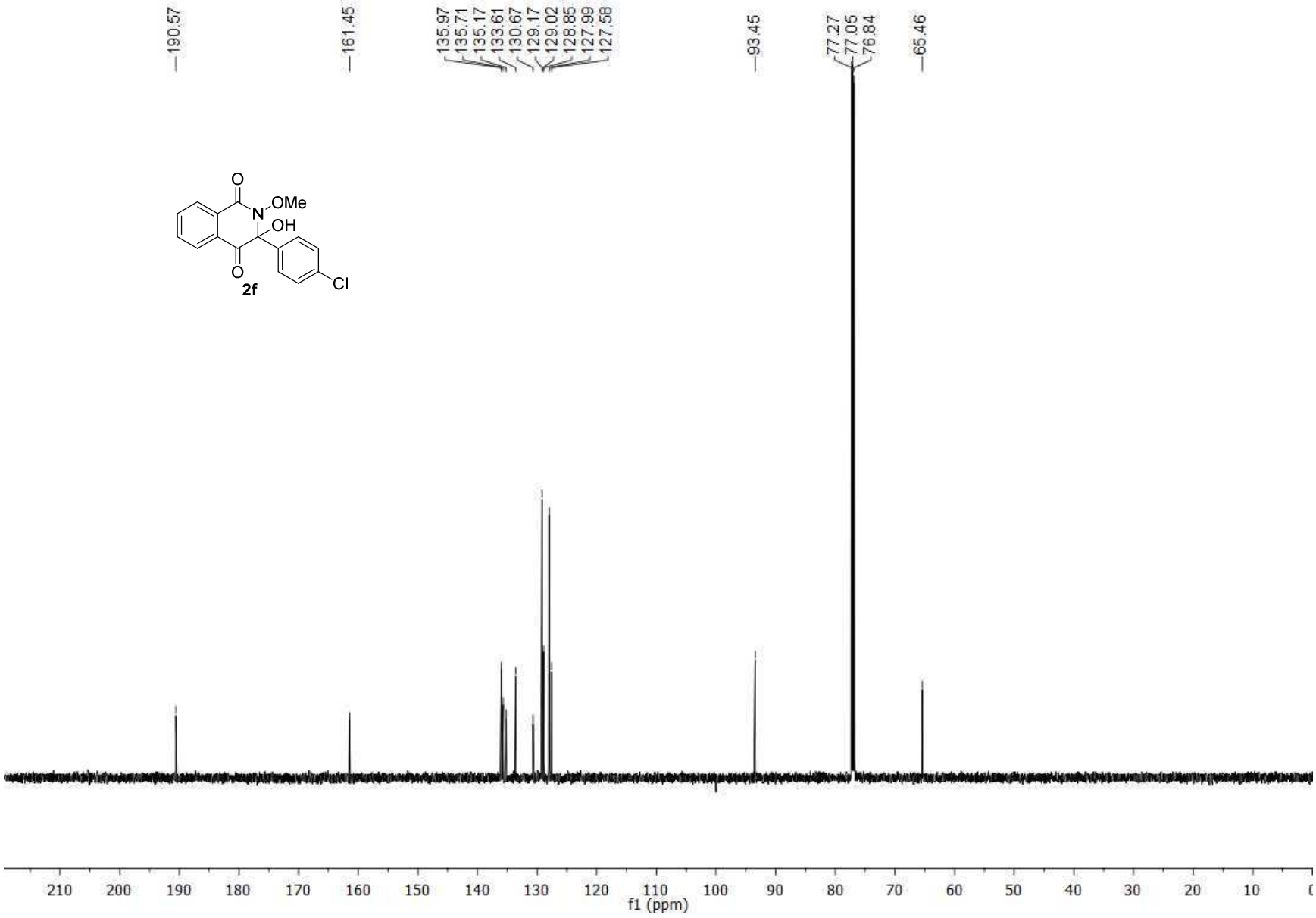
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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77.05
76.84
65.46
O
N
OMe
OH
O
Cl
2f
210
200
190
180
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150
140
130
120
110
100
90
80
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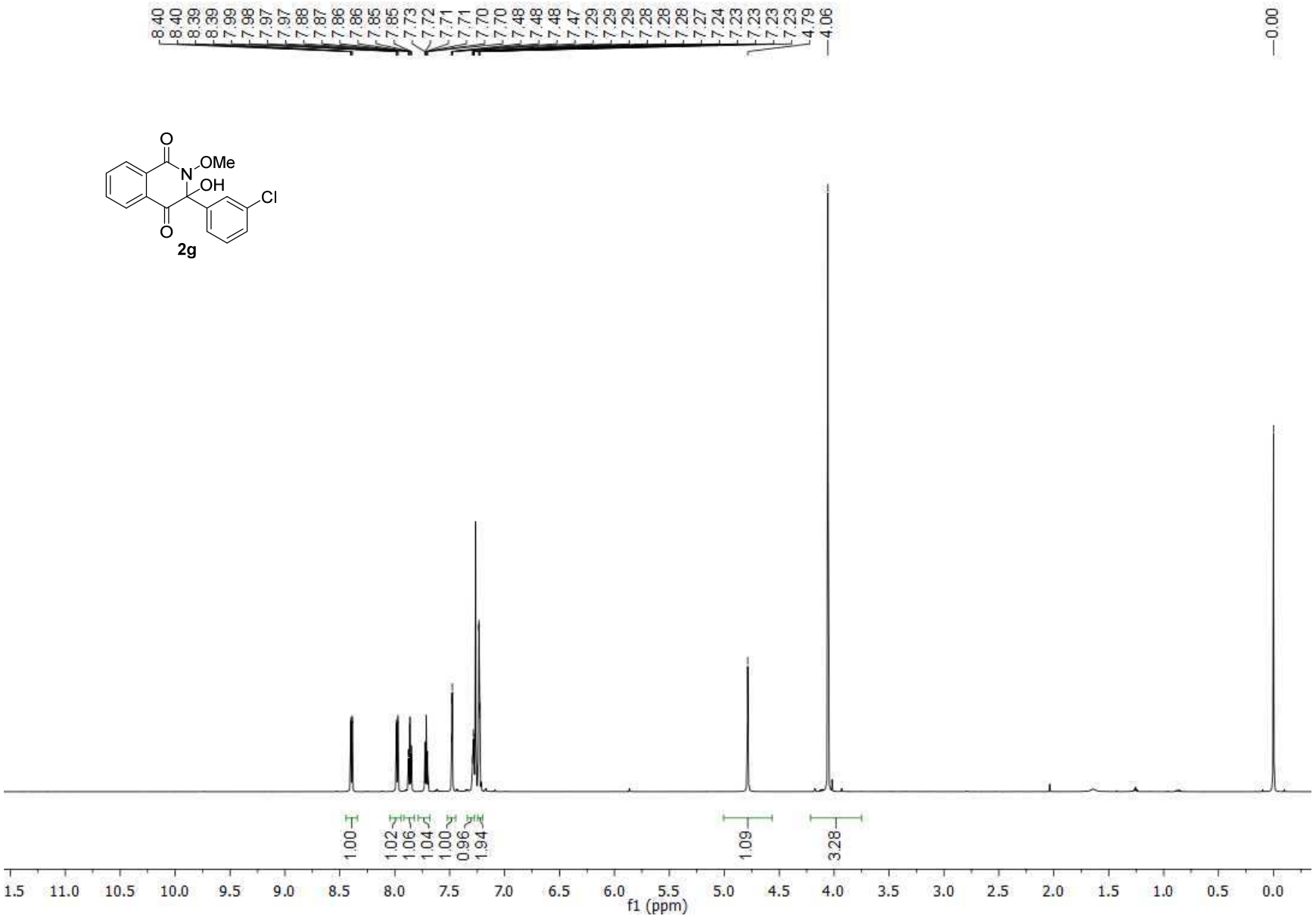
60
50
40
30
20
10
f1 (ppm)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7.85
7.85
7.73
7.72
7.71
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7.70
7.70
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7.48
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7.29
7.29
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7.28
7.28
7.27
7.24
7.23
7.23
7.23
4.79
4.06
0.00
O
N
OMe
OH
Cl
O
2g
1.00
1.02
1.06
1.04
1.00
0.96
1.94
1.09
3.28
1.5
11.0
10.5
10.0
9.5
9.0
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8.0
7.5
7.0
6.5
6.0
5.5
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4.5
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3.5
3.0
2.5
2.0
1.5
1.0
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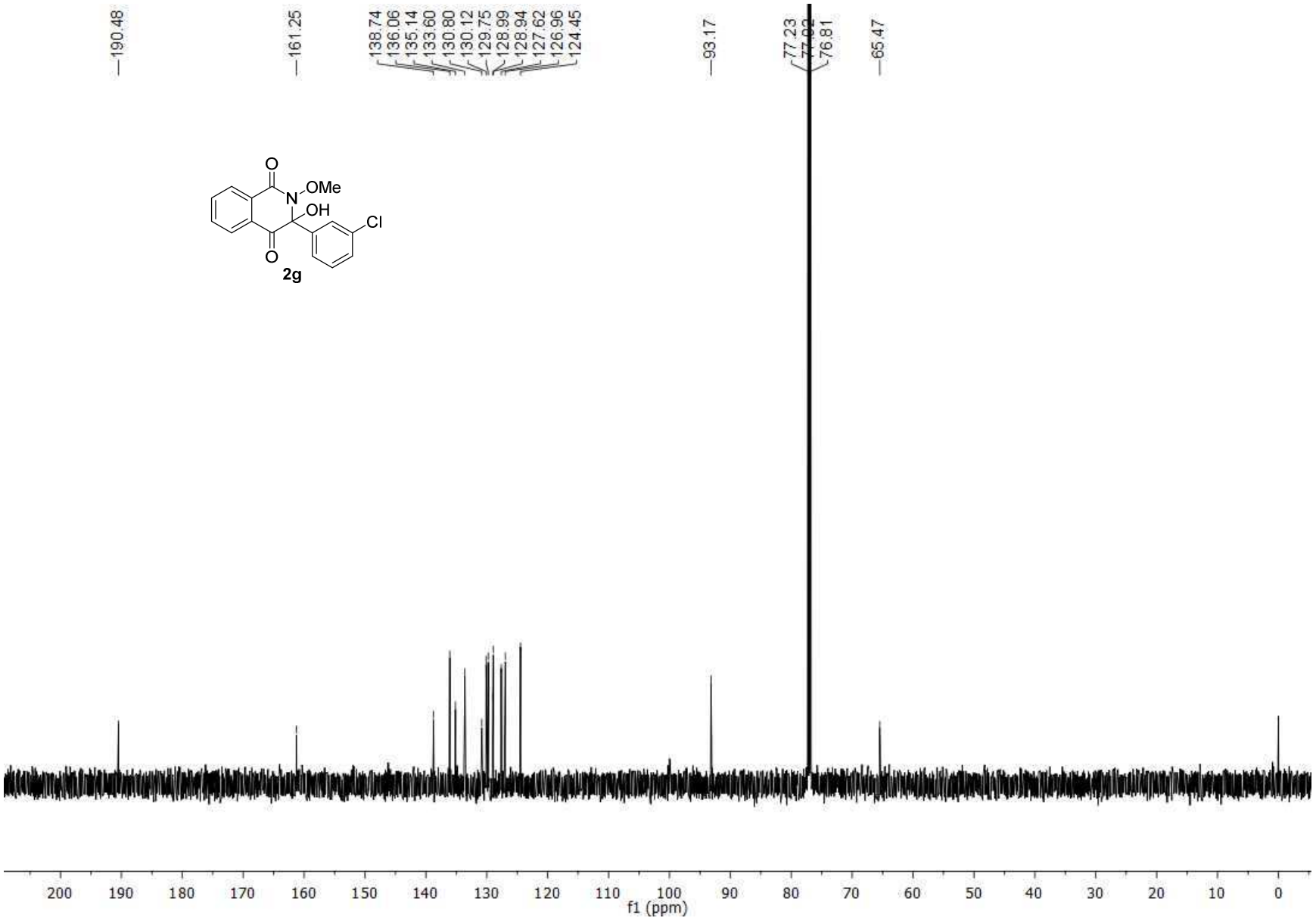
0.0
f1 (ppm)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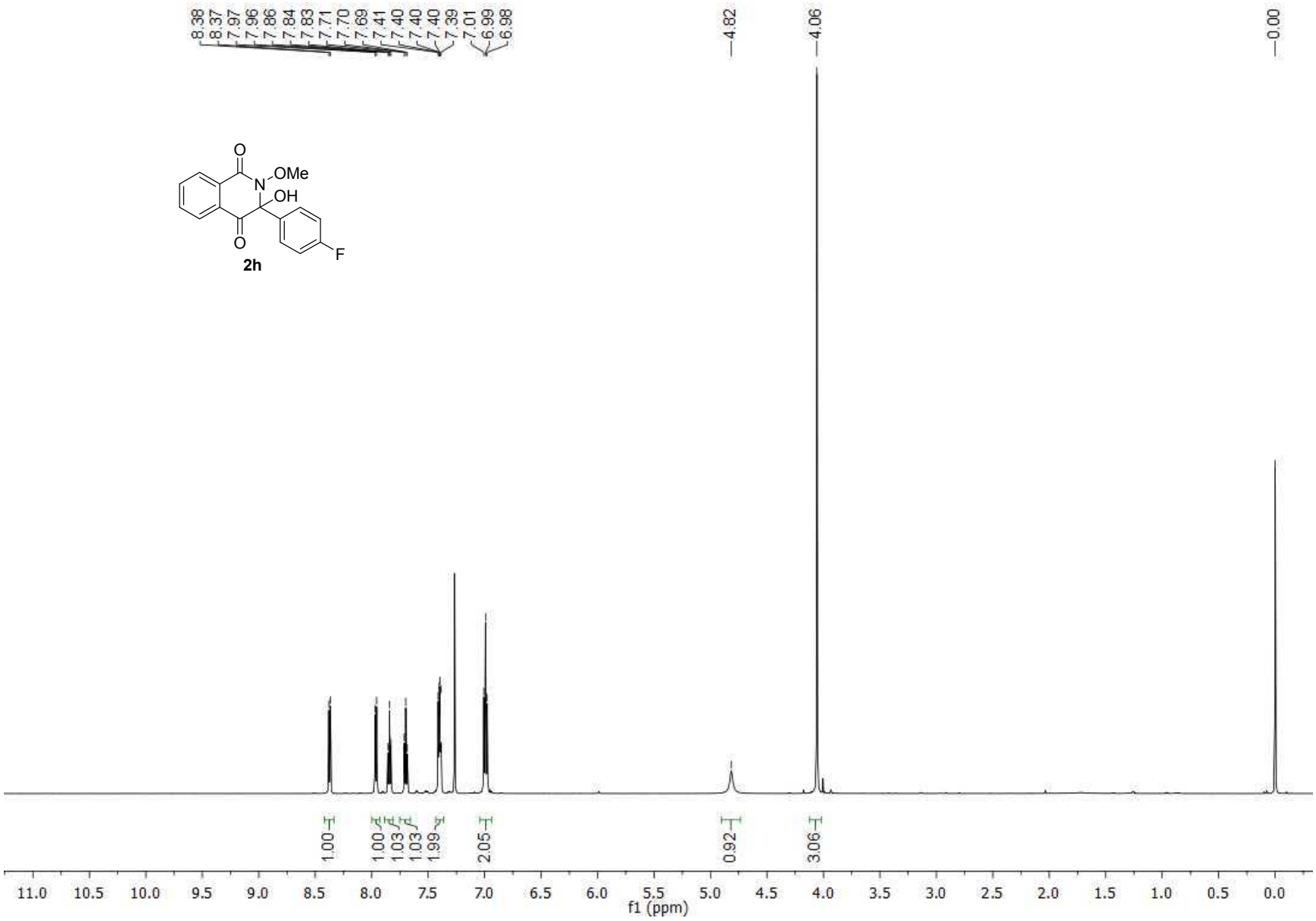
127.62
126.96
124.45
93.17
77.23
77.02
76.81
65.47
O
N
OMe
OH
Cl
O
2g
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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7.40
7.40
7.39
7.01
6.99
6.98
4.82
4.06
0.00
O
N
OMe
OH
O
F
2h
1.00
1.00
1.03
1.03
1.99
2.05
0.92
3.06
11.0
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10.0
9.5
9.0
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8.0
7.5
7.0
6.5
6.0
5.5
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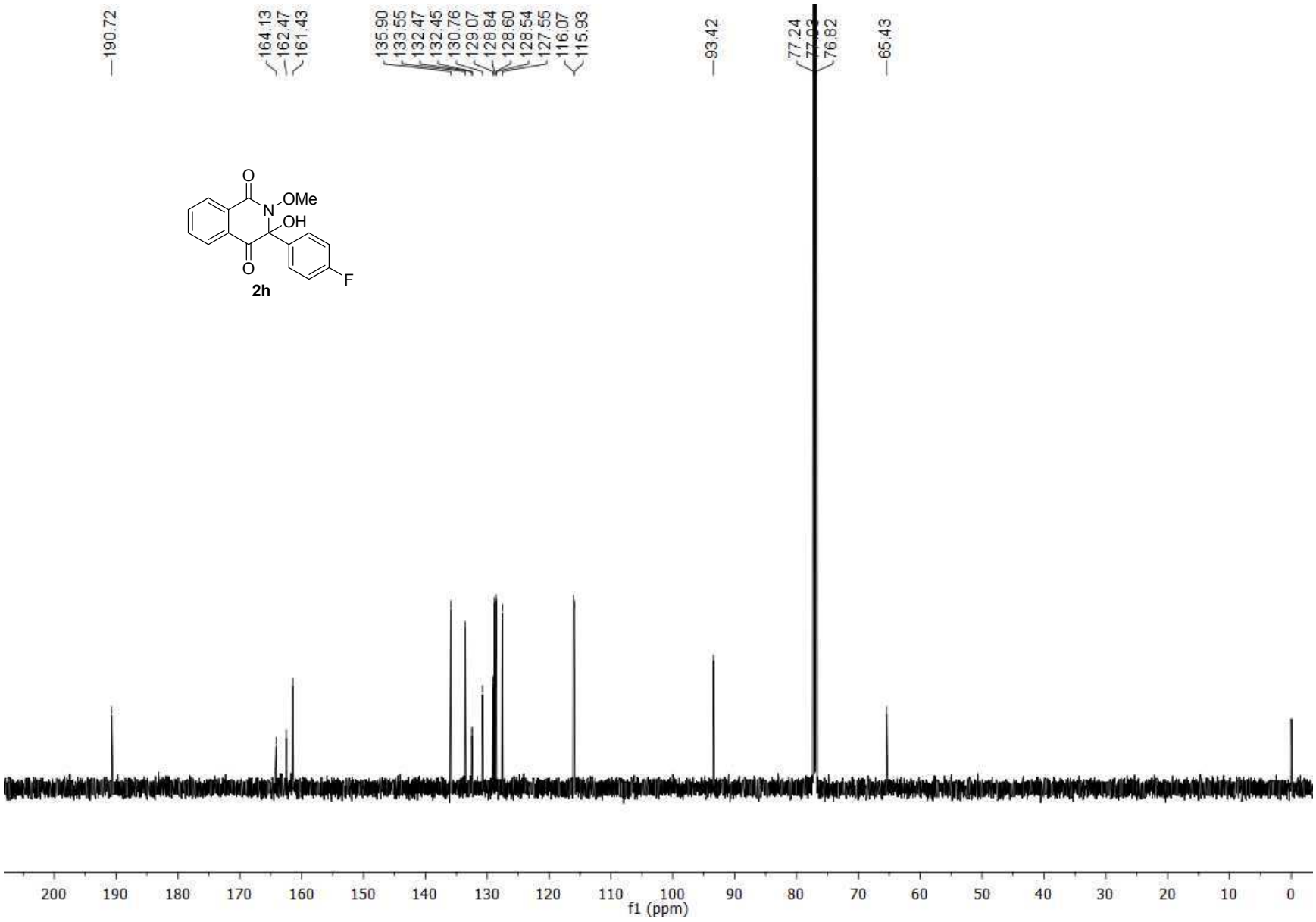
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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93.42
77.24
77.03
76.82
65.43
O
N
OMe
OH
O
F
2h
200
190
180
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150
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130
120
110
100
90
80
70
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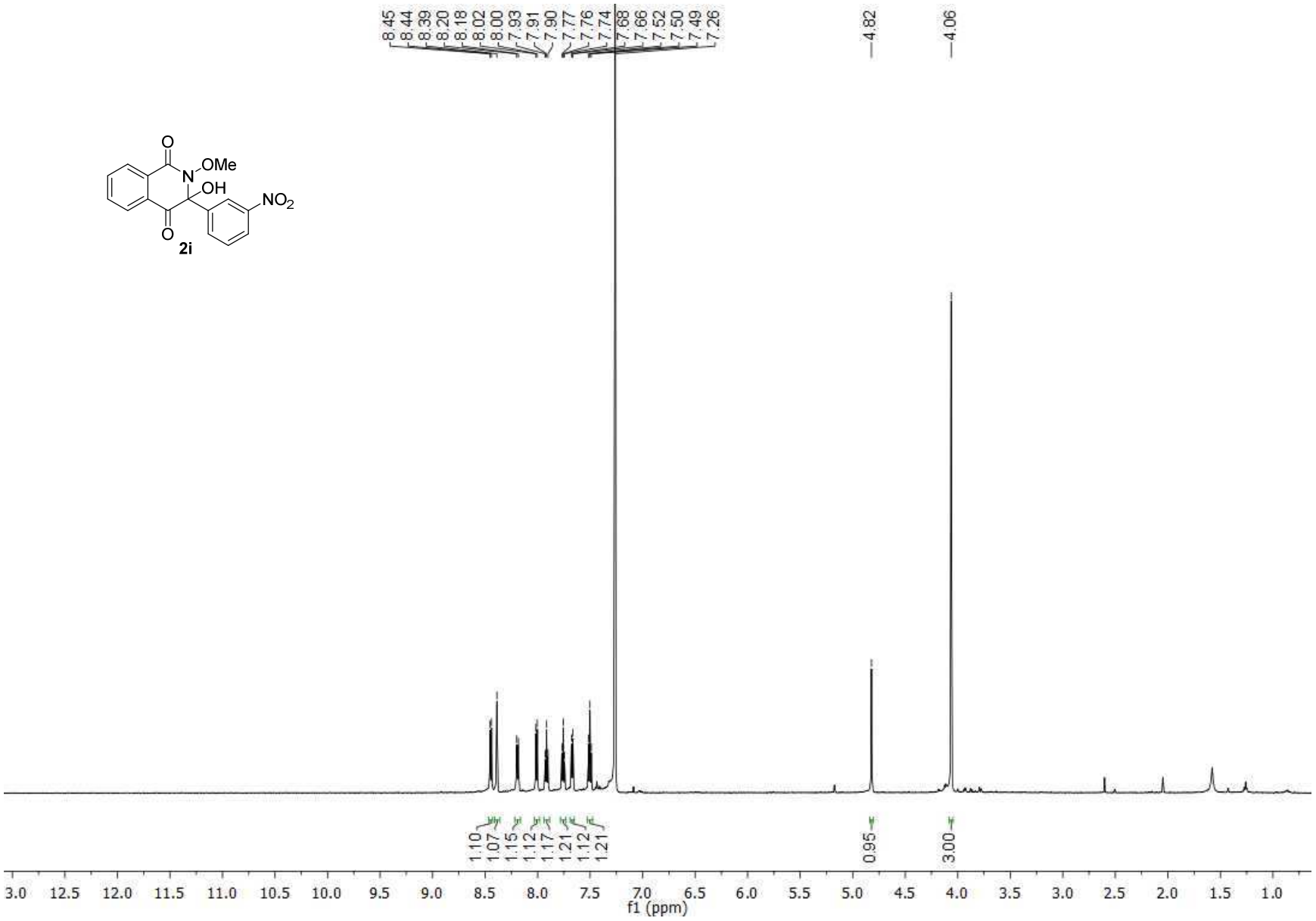
50
40
30
20
10
0
f1 (ppm)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7.76
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4.82
4.06
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N
OMe
OH
NO2
O
2i
1.10
1.07
1.15
1.12
1.17
1.21
1.12
1.21
0.95
3.00
3.0
12.5
12.0
11.5
11.0
10.5
10.0
9.5
9.0
8.5
8.0
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7.0
6.5
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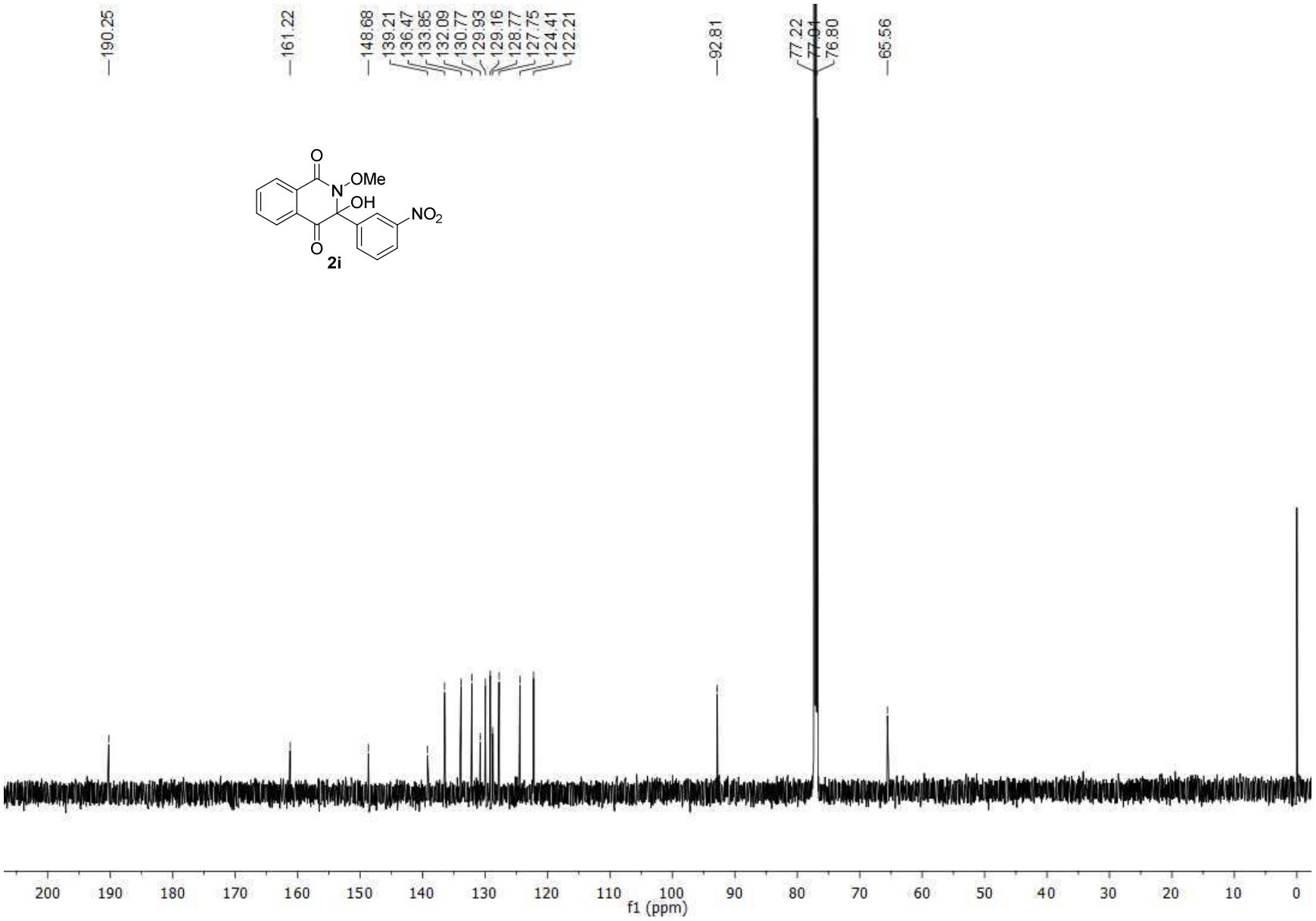
4.0
3.5
3.0
2.5
2.0
1.5
1.0
f1 (ppm)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124.41
122.21
92.81
77.22
77.01
76.80
65.56
O
N
OMe
OH
NO2
O
2i
200
190
180
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150
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130
120
110
100
90
80
70
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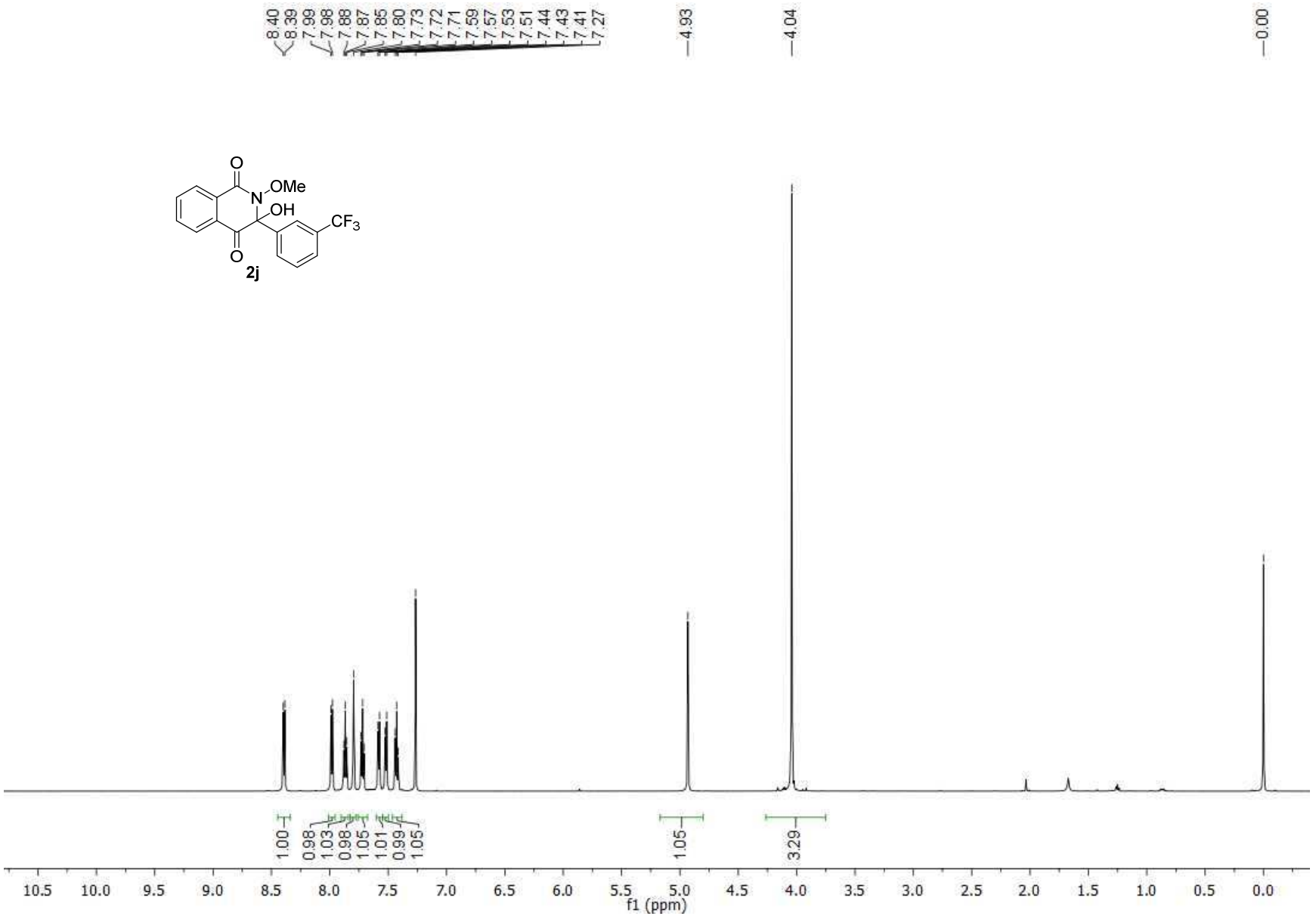
50
40
30
20
10
0
f1 (ppm)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7.53
7.51
7.44
7.43
7.41
7.27
4.93
4.04
0.00
O
N
OMe
OH
CF3
O
2j
1.00
0.98
1.03
0.98
1.05
1.01
0.99
1.05
1.05
3.29
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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3.0
2.5
2.0
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1.0
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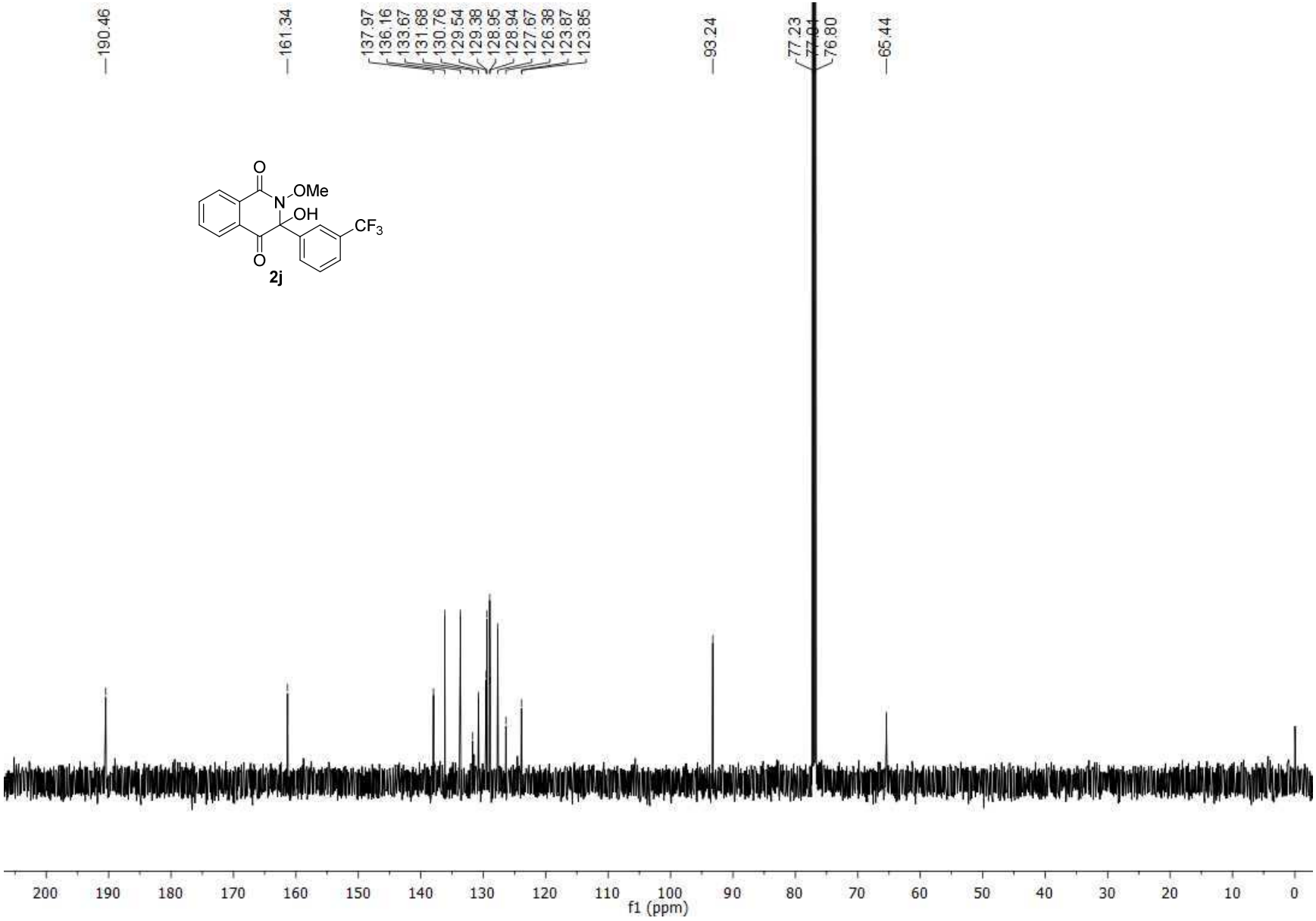
f1 (ppm)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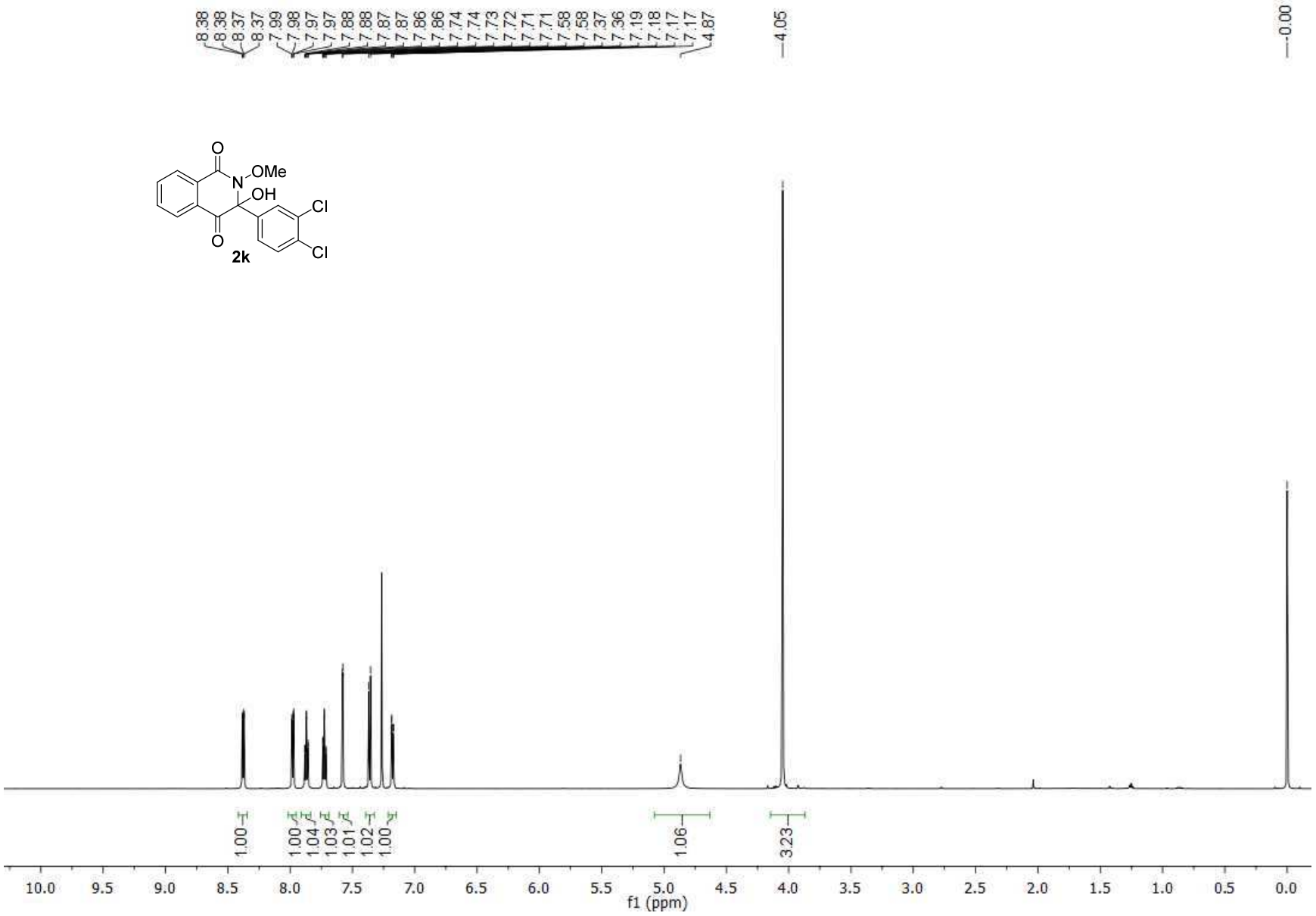
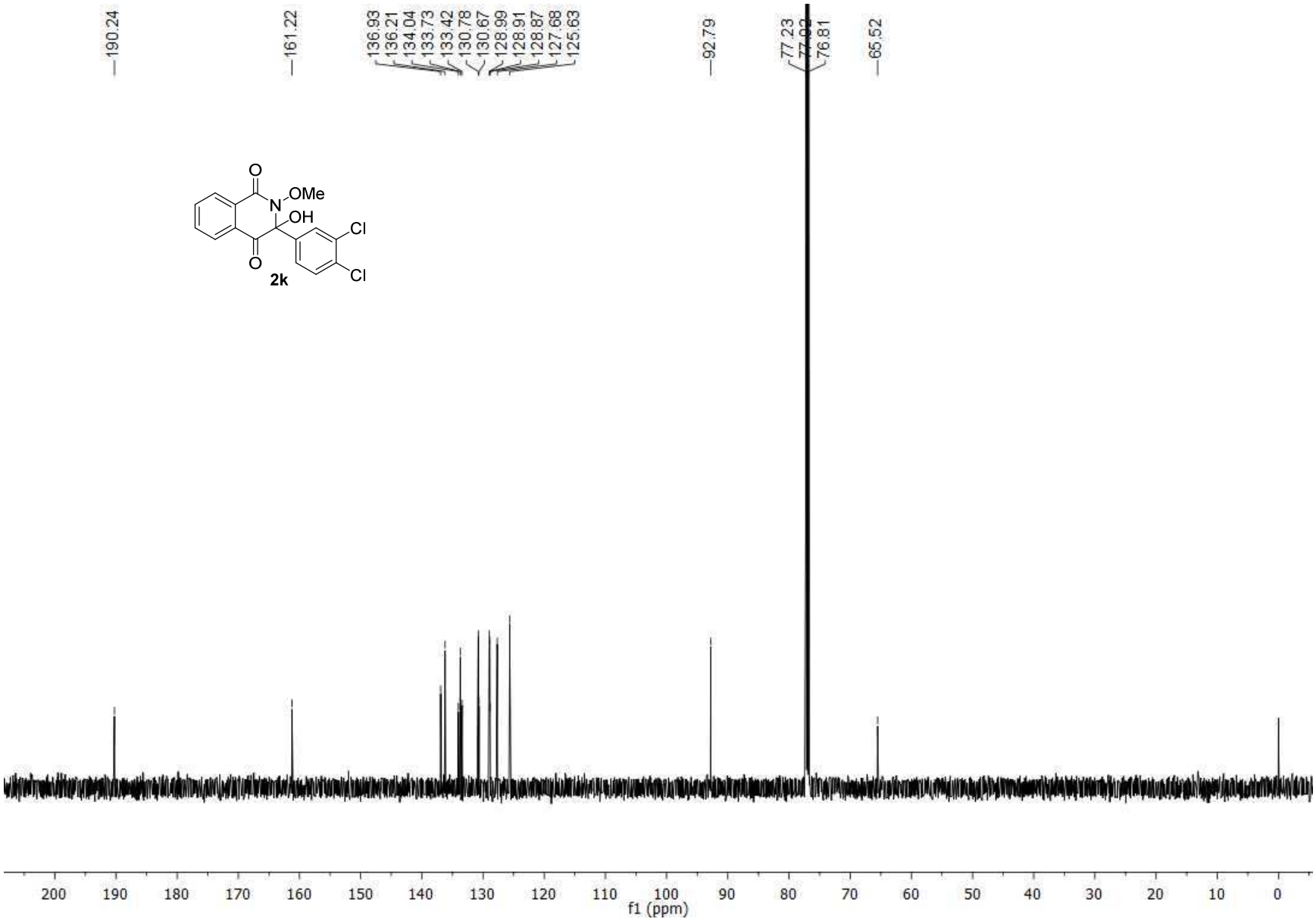
123.85
93.24
77.23
77.04
76.80
65.44
O
N
OMe
OH
CF3
O
2j
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


8.38
8.38
8.37
8.37
7.99
7.98
7.97
7.97
7.88
7.88
7.87
7.87
7.86
7.86
7.74
7.74
7.73
7.72
7.71
7.71
7.58
7.58
7.37
7.36
7.19
7.18
7.17
7.17
4.87
4.05
0.00
O
N
OMe
OH
Cl
Cl
O
2k
1.00
1.00
1.04
1.03
1.01
1.02
1.00
1.06
3.23
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)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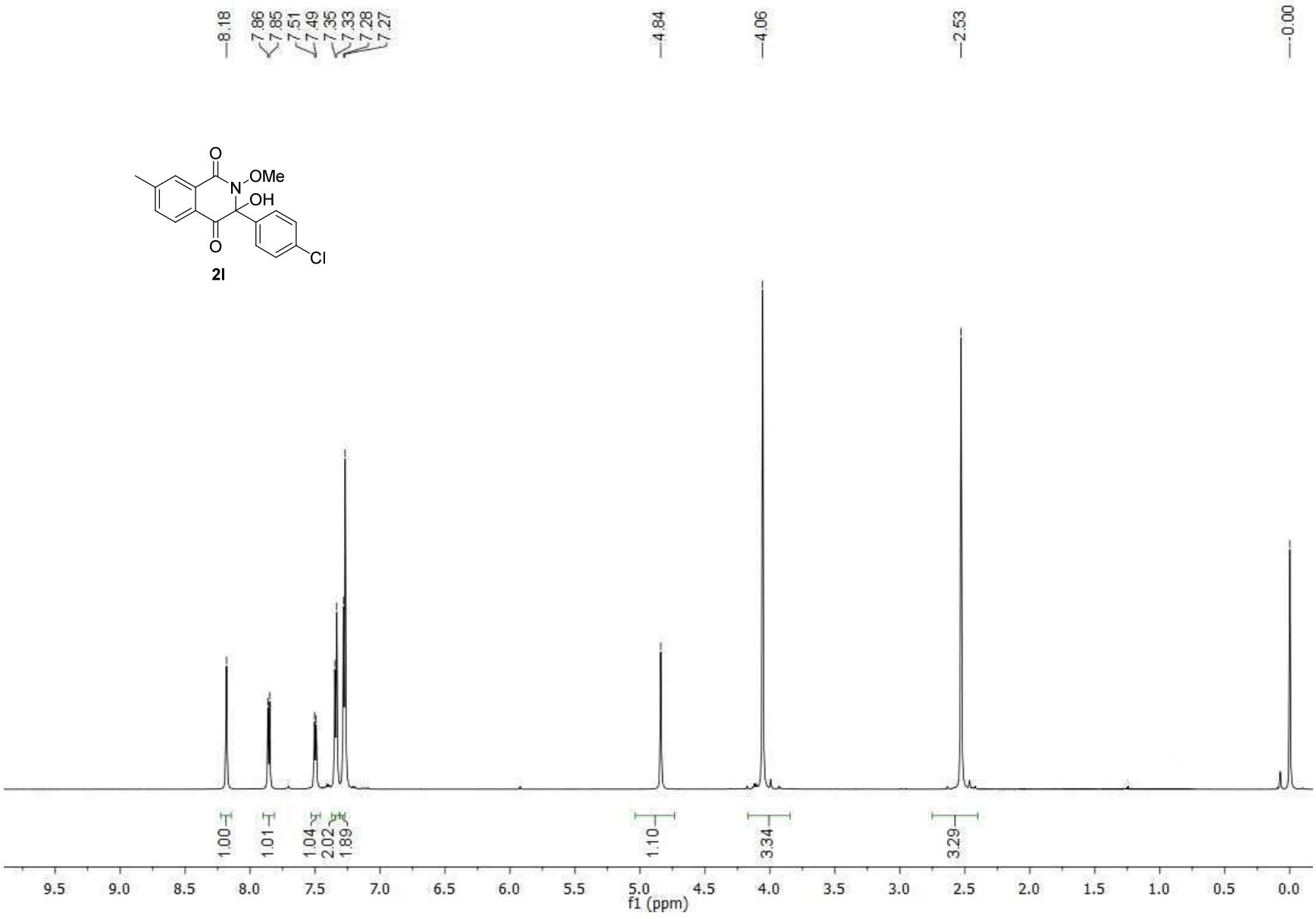
161.22
136.93
136.21
134.04
133.73
133.42
130.78
130.67
128.99
128.91
128.87
127.68
125.63
92.79
77.23
77.02
76.81
65.52
O
N
OMe
OH
Cl
Cl
O
2k
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)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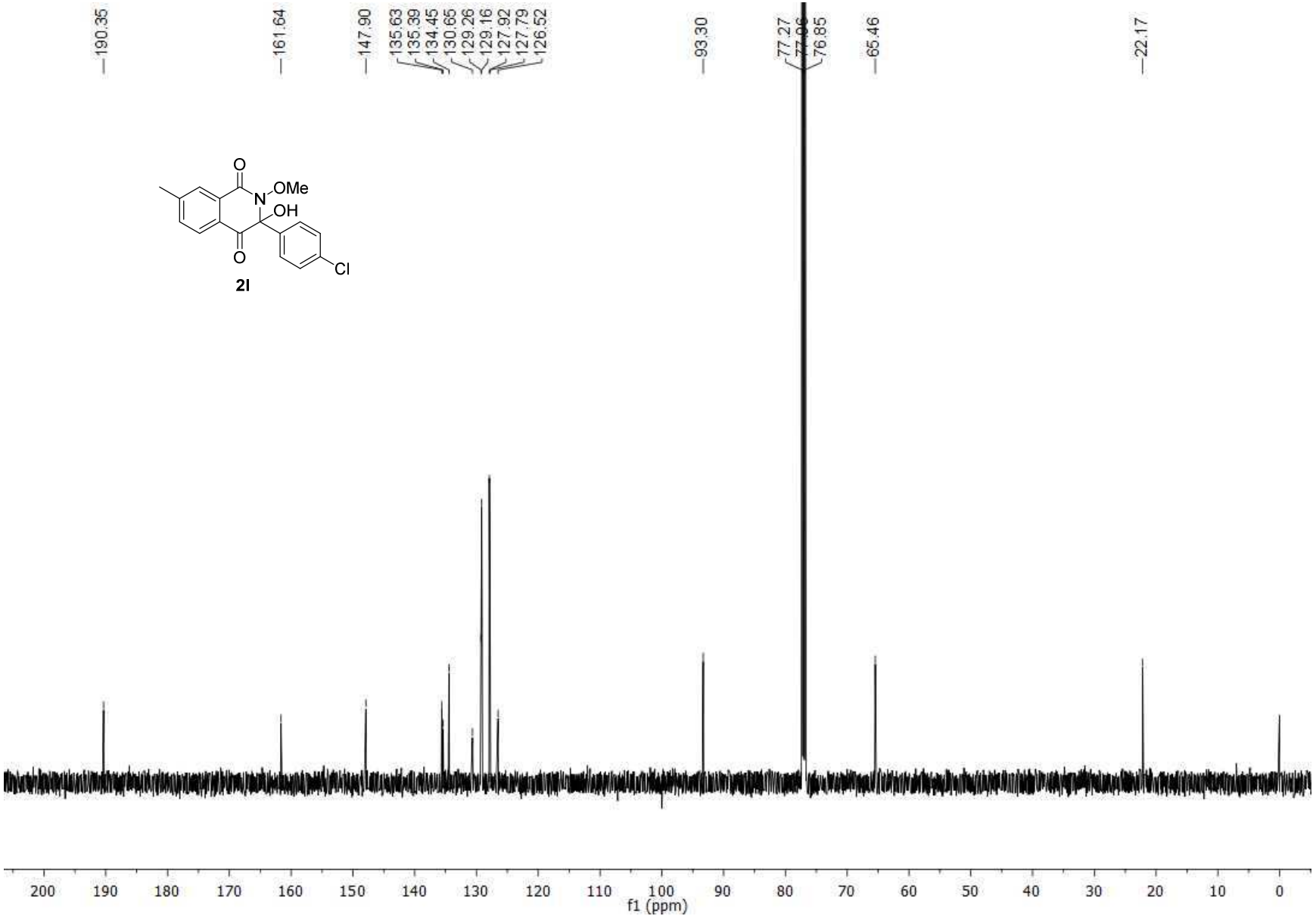
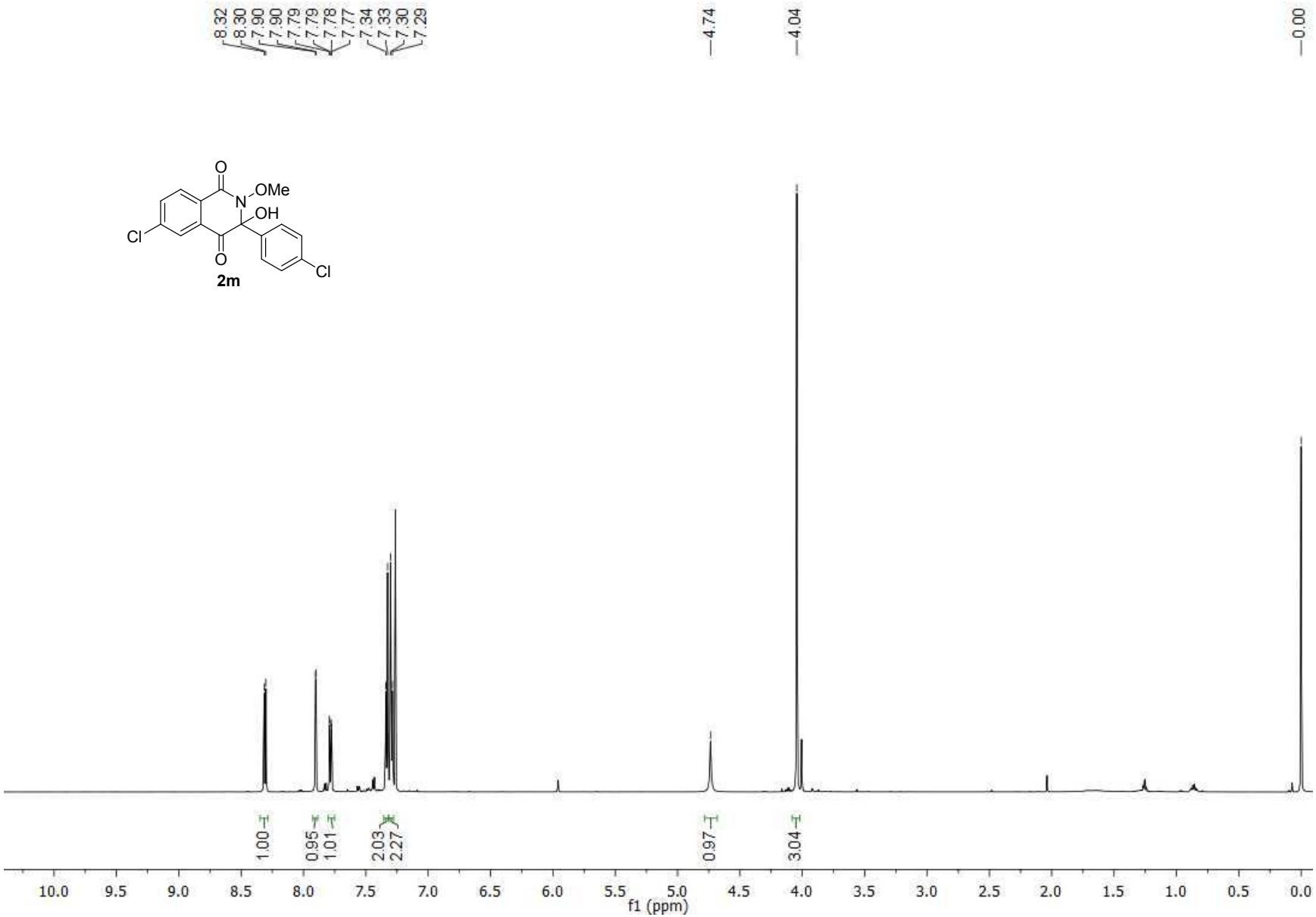
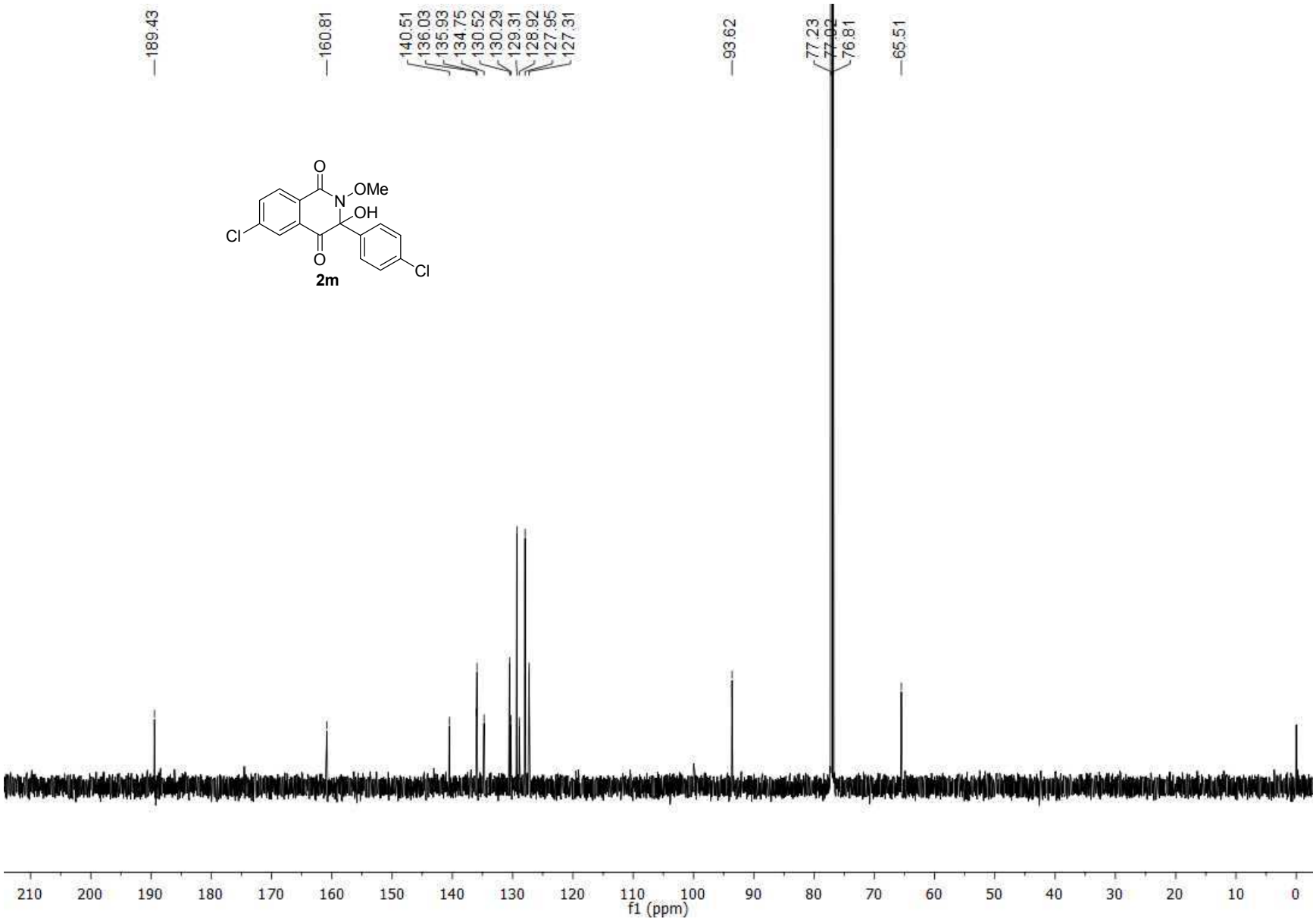
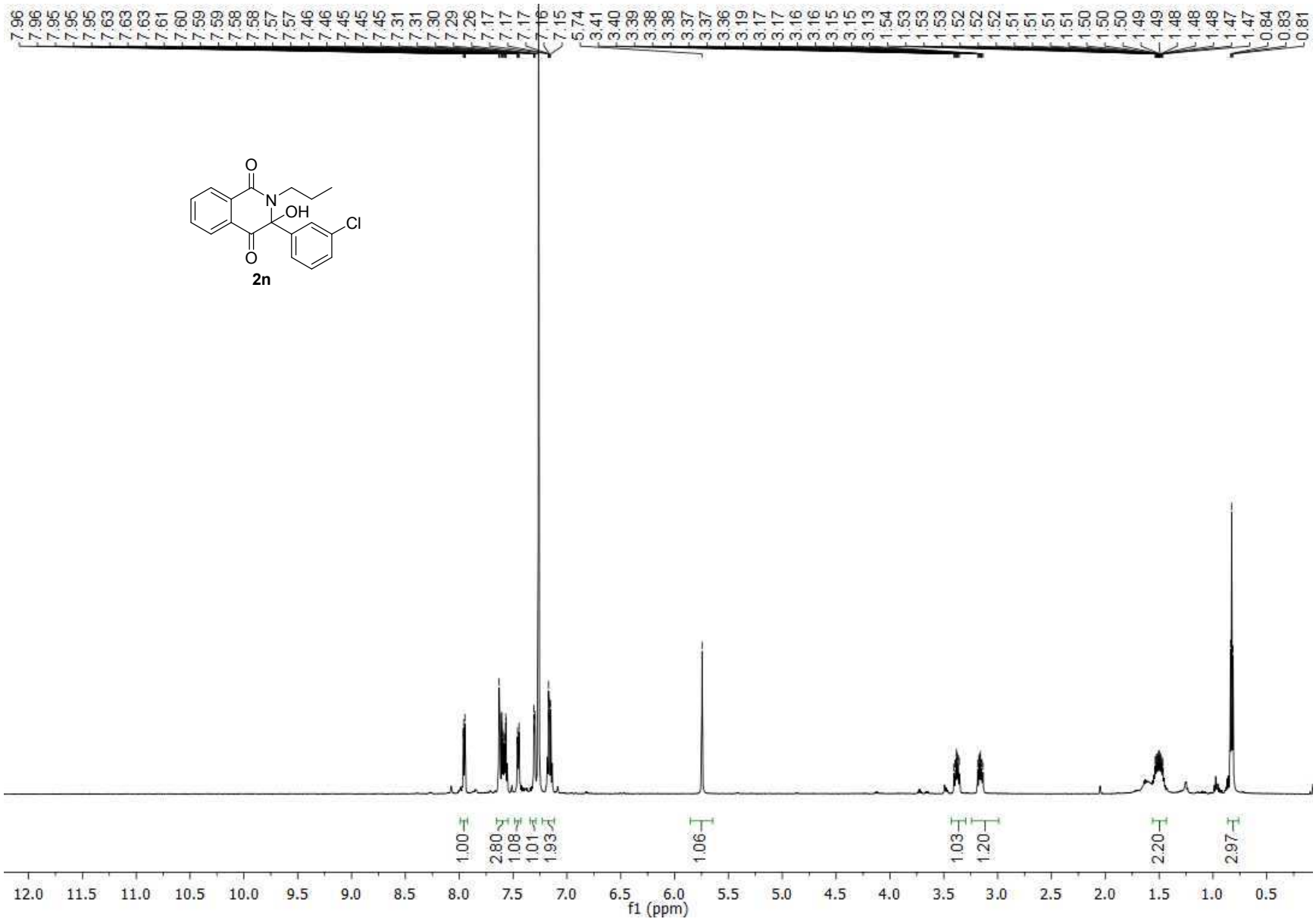
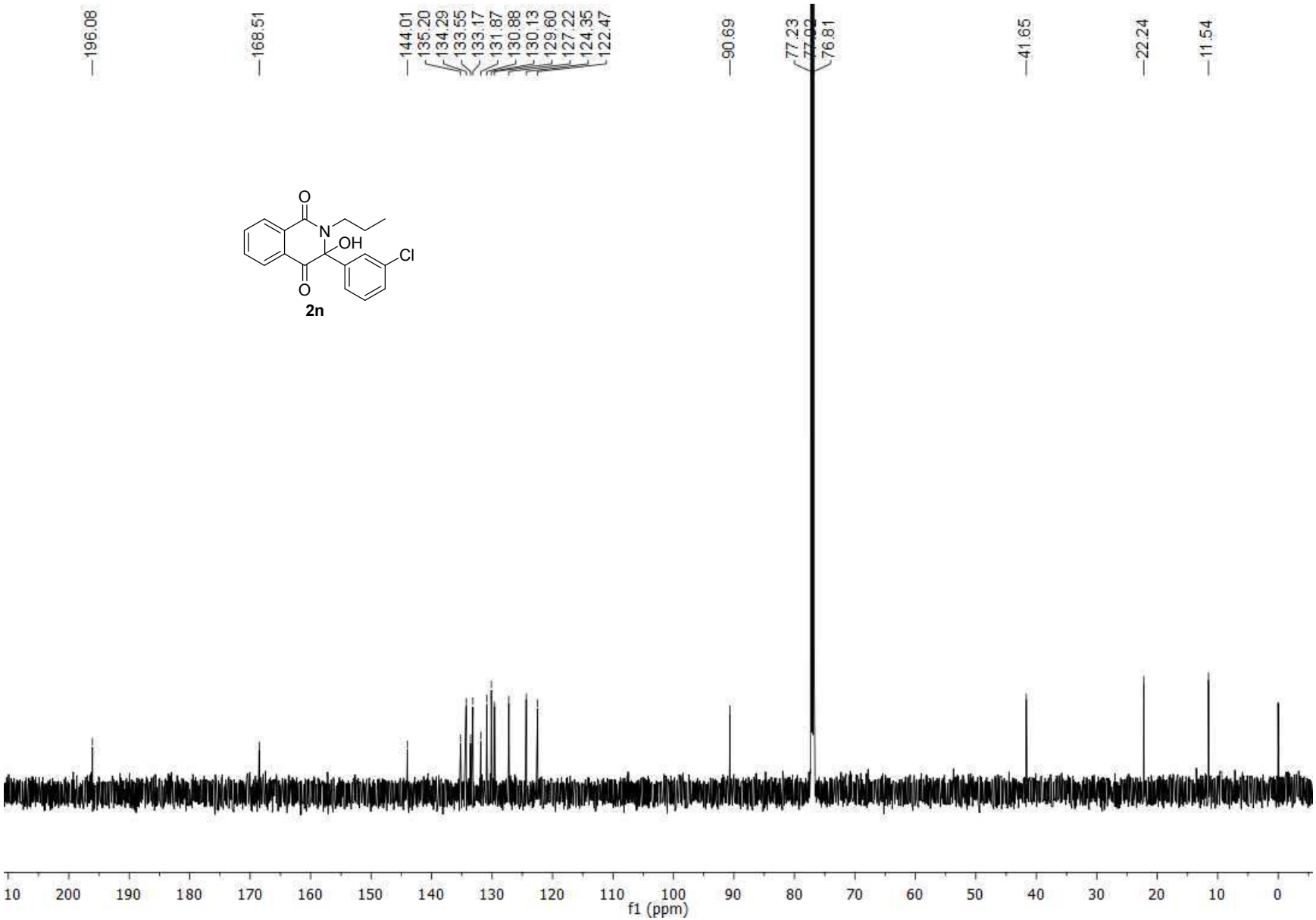
7.86
7.85
7.51
7.49
7.35
7.33
7.28
7.27
4.84
4.06
2.53
0.00
O
N
OMe
OH
O
Cl
2l
1.00
1.01
1.04
2.02
1.89
1.10
3.34
3.29
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

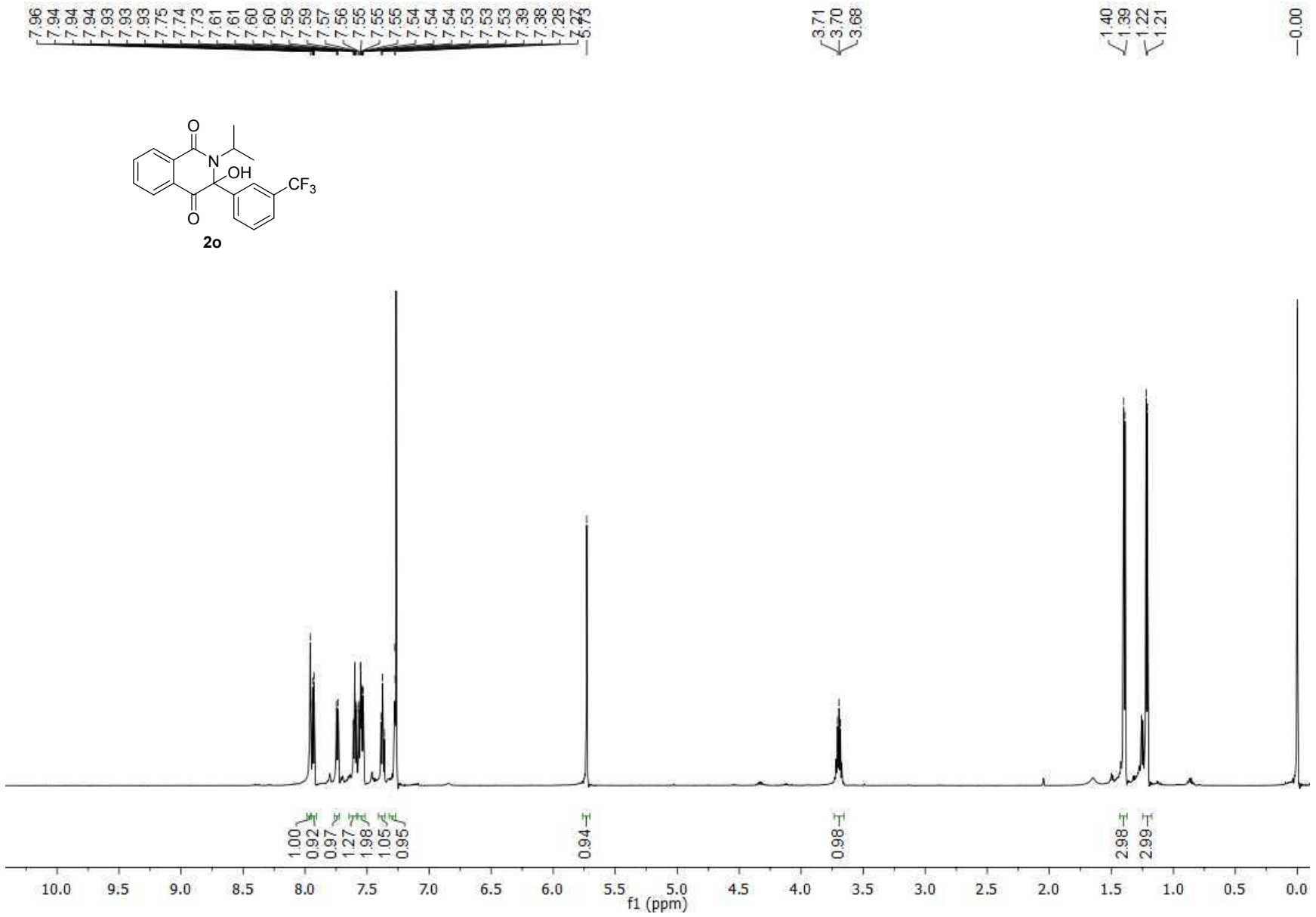

190.35
161.64
147.90
135.63
135.39
134.45
130.65
129.26
129.16
127.92
127.79
126.52
93.30
77.27
76.85
65.46
22.17
O
N
OMe
OH
O
Cl
2l
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

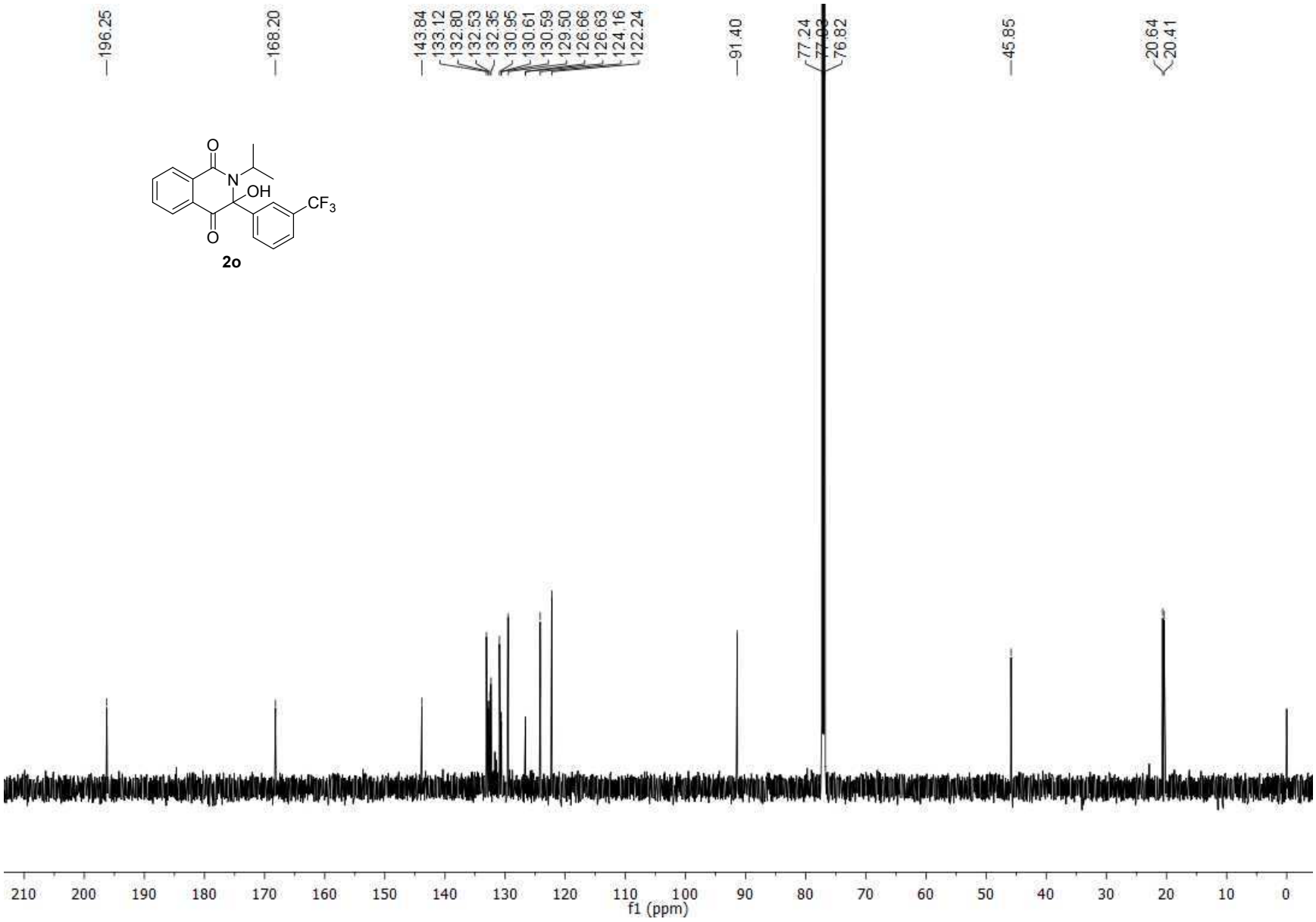

8.32
8.30
7.90
7.90
7.79
7.79
7.78
7.77
7.34
7.33
7.30
7.29
4.74
4.04
0.00
O
N
OMe
OH
Cl
O
Cl
2m
1.00
0.95
1.01
2.03
2.27
0.97
3.04
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

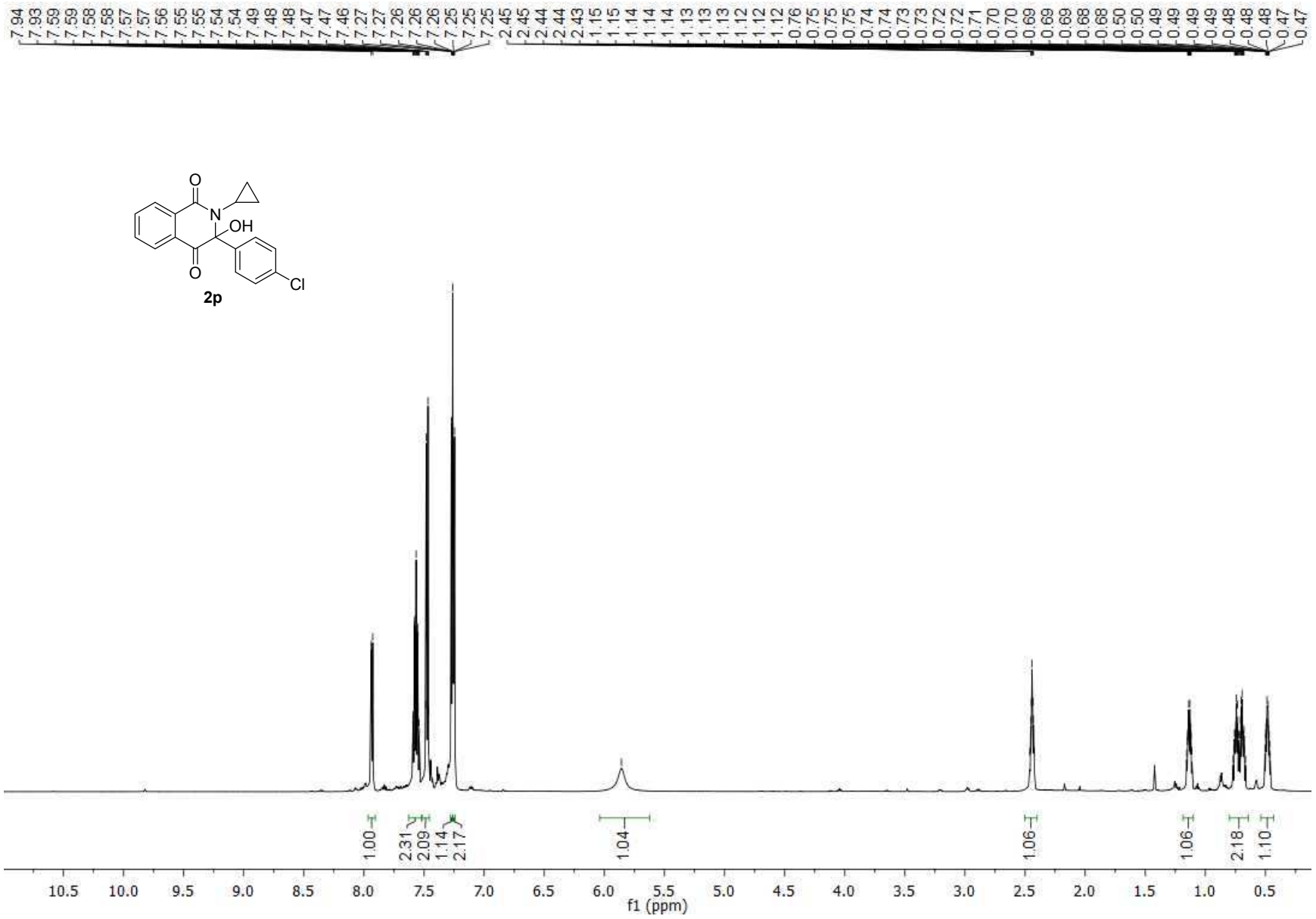

189.43
160.81
140.51
136.03
135.93
134.75
130.52
130.29
129.31
128.92
127.95
127.31
93.62
77.23
77.02
76.81
65.51
O
N
OMe
OH
Cl
O
Cl
2m
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

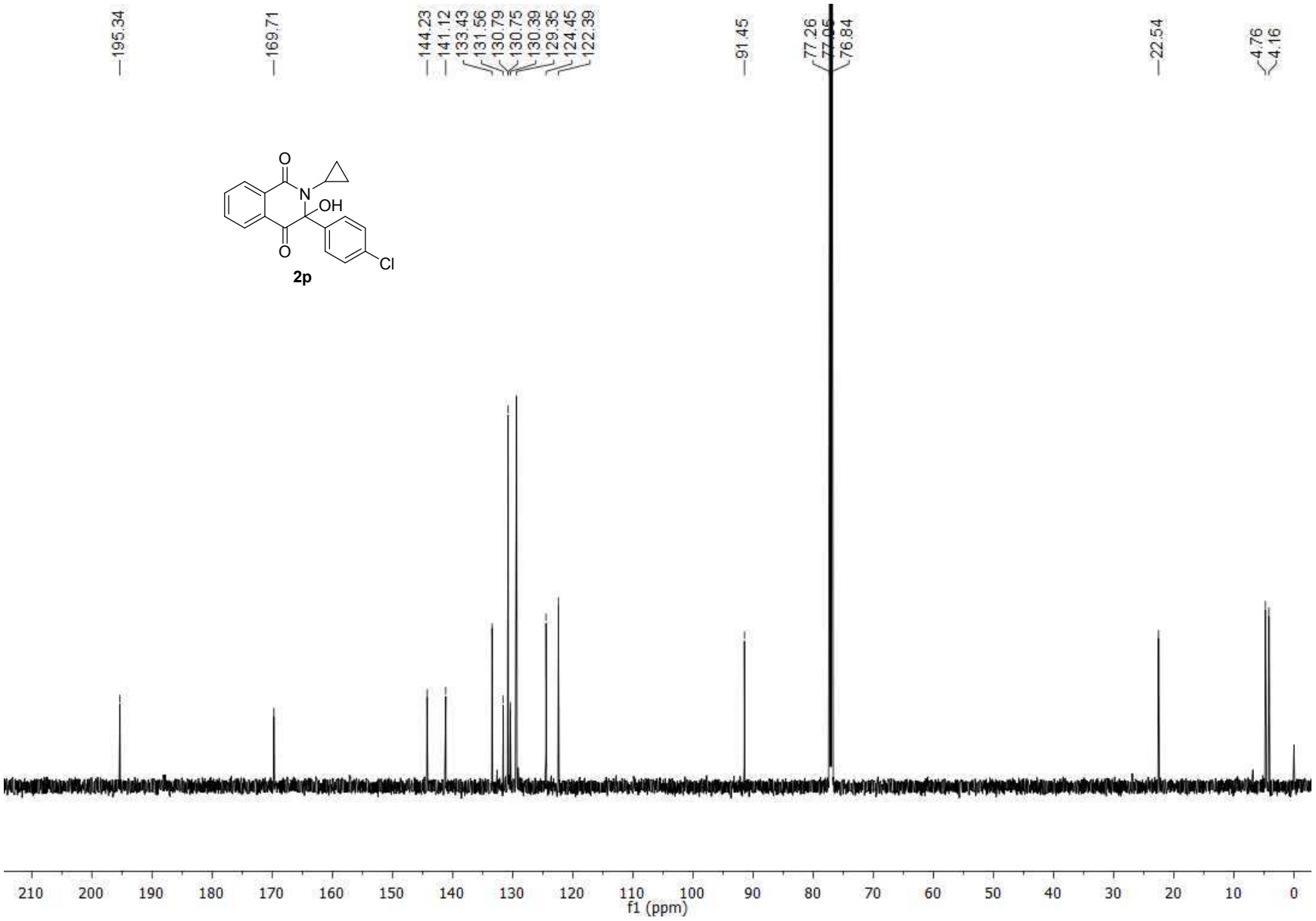

7.96 7.96 7.95 7.95 7.95 7.63 7.63 7.63 7.61 7.60 7.59 7.59 7.58 7.58 7.57 7.57 7.46 7.46 7.45 7.45 7.45 7.31 7.31 7.30 7.29 7.26 7.17 7.17 7.17 7.16 7.15 5.74 3.41 3.40 3.39 3.38 3.38 3.37 3.37 3.36 3.19 3.17 3.17 3.16 3.16 3.15 3.15 3.13 1.54 1.53 1.53 1.53 1.52 1.52 1.52 1.51 1.51 1.51 1.51 1.50 1.50 1.50 1.49 1.49 1.48 1.48 1.48 1.47 1.47 0.84 0.83 0.81
O
N
OH
Cl
O
2n
1.00 2.80 1.08 1.01 1.93 1.06 1.03 1.20 2.20 2.97
12.0 11.5 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
f1 (ppm)

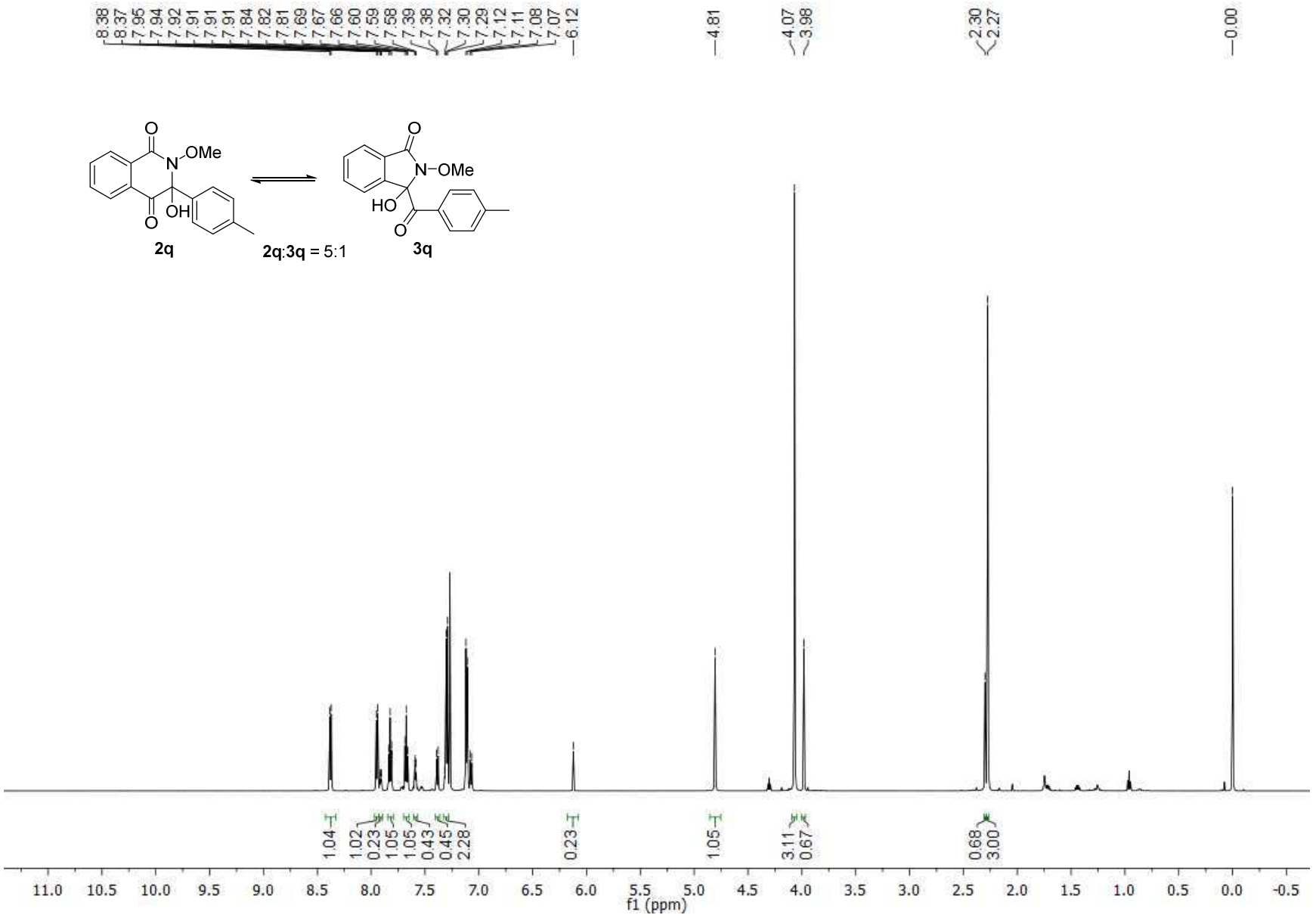

196.08
168.51
144.01
135.20
134.29
133.55
133.17
131.87
130.88
130.13
129.60
127.22
124.35
122.47
90.69
77.23
77.02
76.81
41.65
22.24
11.54
O
N
OH
Cl
O
2n
10
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

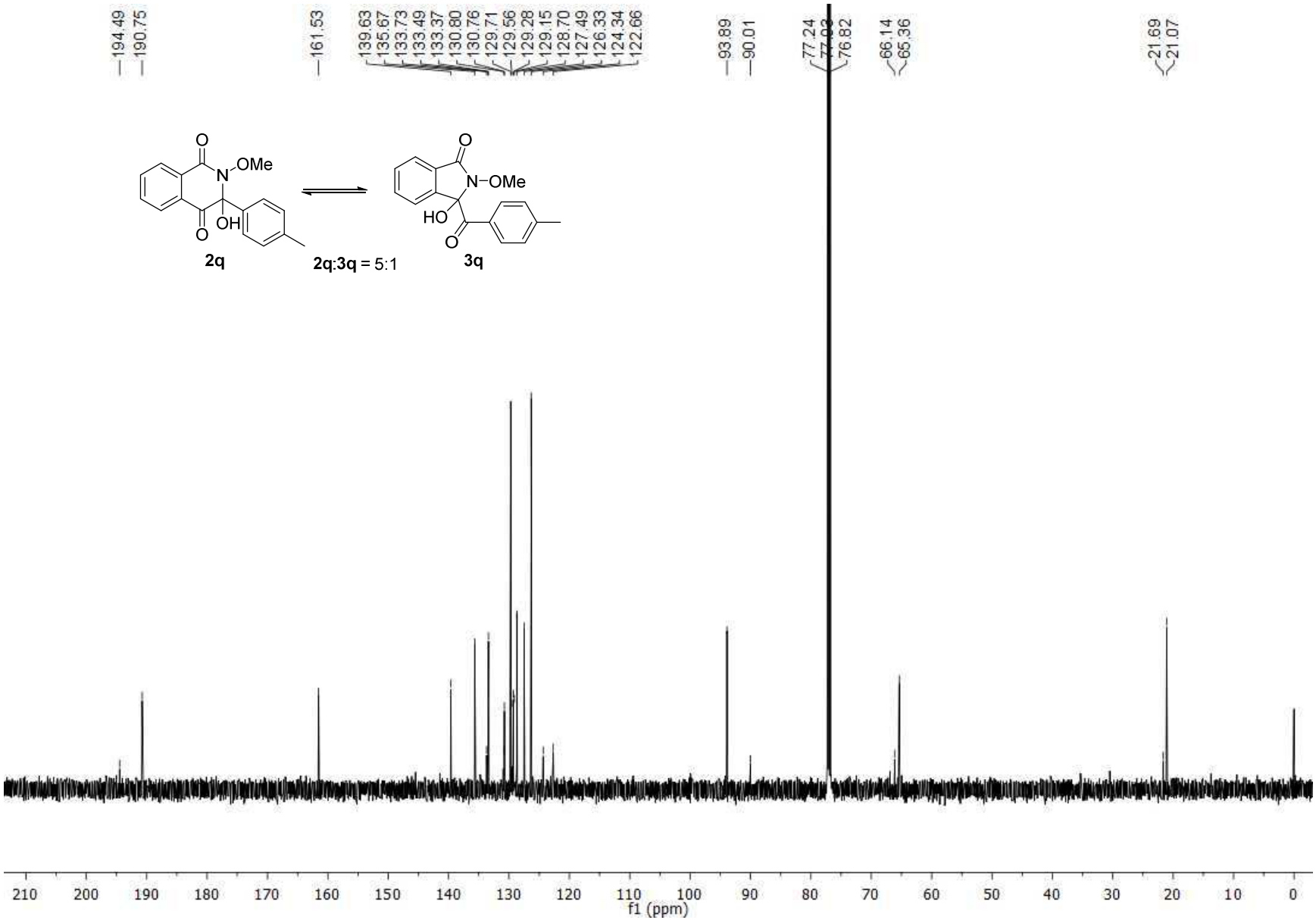

7.96
7.94
7.94
7.94
7.93
7.93
7.93
7.75
7.74
7.73
7.61
7.61
7.60
7.60
7.59
7.59
7.57
7.56
7.55
7.55
7.55
7.54
7.54
7.54
7.53
7.53
7.53
7.39
7.38
7.28
5.73
3.71
3.70
3.68
1.40
1.39
1.22
1.21
0.00
O
N
OH
CF3
O
2o
1.00
0.92
0.97
1.27
1.98
1.05
0.95
0.94
0.98
2.98
2.99
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
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)


196.25
168.20
143.84
133.12
132.80
132.53
132.35
130.95
130.61
130.59
129.50
126.66
126.63
124.16
122.24
91.40
77.24
77.03
76.82
45.85
20.64
20.41
O
N
OH
CF3
O
2o
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.94 7.93 7.59 7.59 7.58 7.58 7.57 7.57 7.56 7.55 7.54 7.54 7.49 7.48 7.48 7.47 7.47 7.46 7.27 7.27 7.26 7.26 7.26 7.25 7.25 7.25
2.45 2.45 2.44 2.44 2.43 1.15 1.15 1.14 1.14 1.14 1.13 1.13 1.13 1.12 1.12 1.12 0.76 0.75 0.75 0.75 0.74 0.74 0.73 0.73 0.72 0.72 0.71 0.70 0.70 0.69 0.69 0.69 0.68 0.68 0.50 0.50 0.49 0.49 0.49 0.49 0.48 0.48 0.48 0.47 0.47
O
N
OH
O
Cl
2p
1.00
2.31
2.09
1.14
2.17
1.04
1.06
1.06
2.18
1.10
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
f1 (ppm)


195.34
169.71
144.23
141.12
133.43
131.56
130.79
130.75
130.39
129.35
124.45
122.39
91.45
77.26
77.05
76.84
22.54
4.76
4.16
O
N
OH
O
Cl
2p
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


8.38
8.37
7.95
7.94
7.92
7.91
7.91
7.91
7.84
7.82
7.81
7.69
7.67
7.66
7.60
7.59
7.58
7.39
7.38
7.32
7.30
7.29
7.12
7.11
7.08
7.07
6.12
4.81
4.07
3.98
2.30
2.27
0.00
O
N-OMe
OH
O
2q
2q:3q = 5:1
O
N-OMe
HO
O
3q
1.04
1.02
0.23
1.05
1.05
0.43
0.45
2.28
0.23
1.05
3.11
0.67
0.68
3.00
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
-0.5
f1 (ppm)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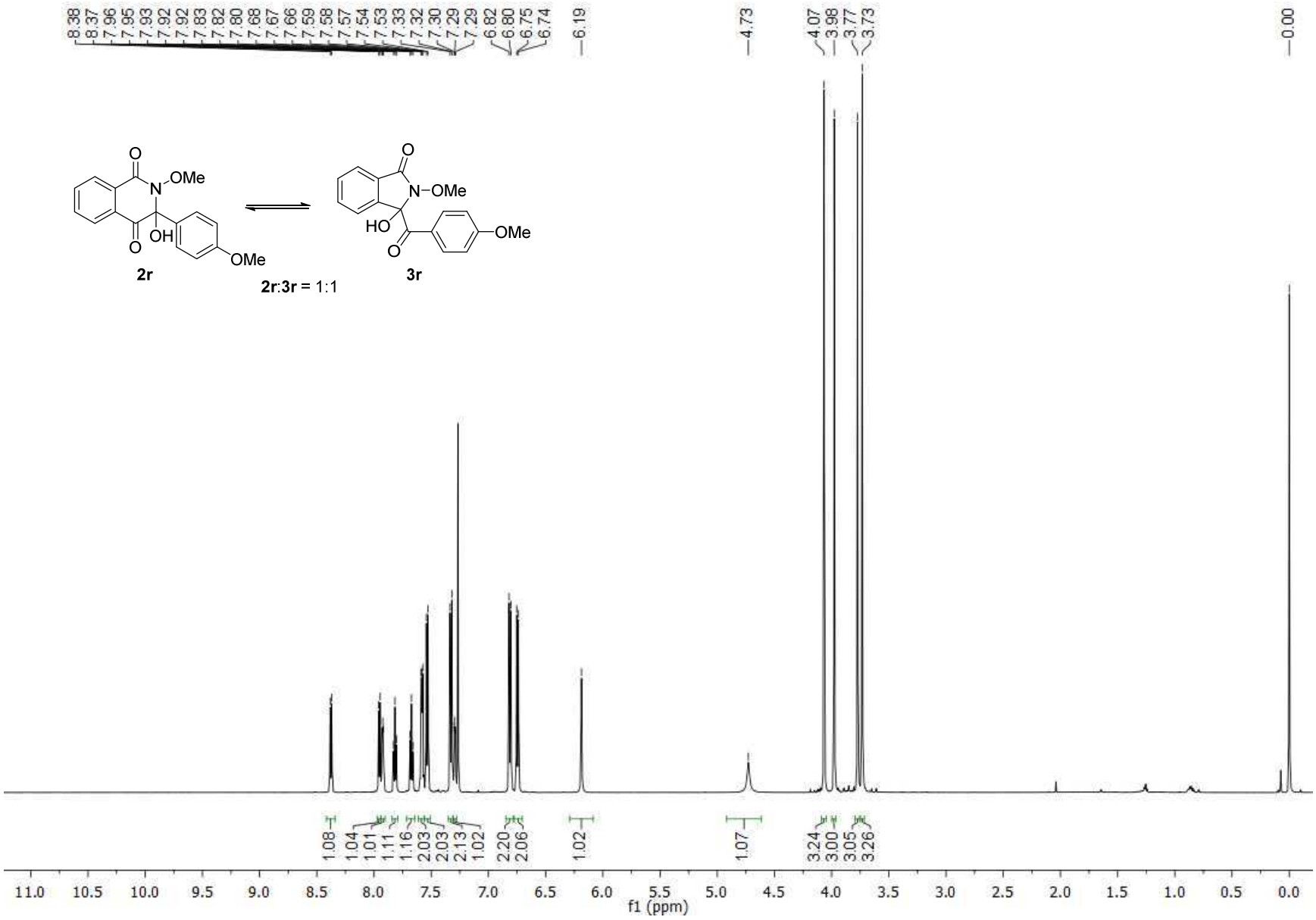
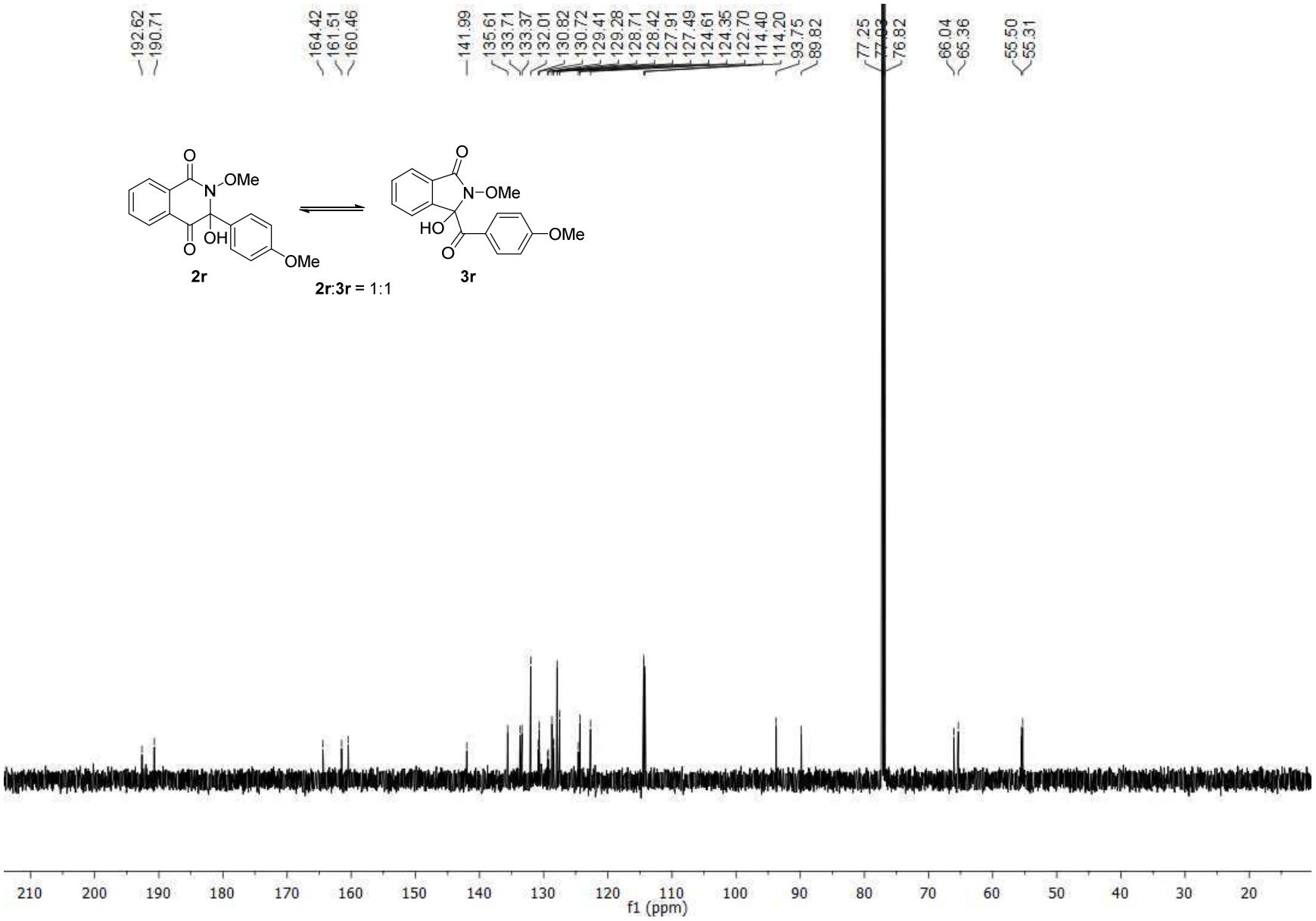
139.63
135.67
133.73
133.49
133.37
130.80
130.76
129.71
129.56
129.28
129.15
128.70
127.49
126.33
124.34
122.66
93.89
90.01
77.24
77.03
76.82
66.14
65.36
21.69
21.07
O
N
OMe
OH
O
2q
2q:3q = 5:1
O
N–OMe
HO
O
3q
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


8.38
8.37
7.96
7.95
7.93
7.92
7.92
7.83
7.82
7.80
7.68
7.67
7.66
7.59
7.58
7.57
7.54
7.53
7.33
7.32
7.30
7.29
7.29
6.82
6.80
6.75
6.74
6.19
4.73
4.07
3.98
3.77
3.73
0.00
O
N
OMe
OH
O
OMe
2r
2r:3r = 1:1
O
N–OMe
HO
O
OMe
3r
1.08
1.04
1.01
1.11
1.16
2.03
2.03
2.13
1.02
2.20
2.06
1.02
1.07
3.24
3.00
3.05
3.26
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)


192.62
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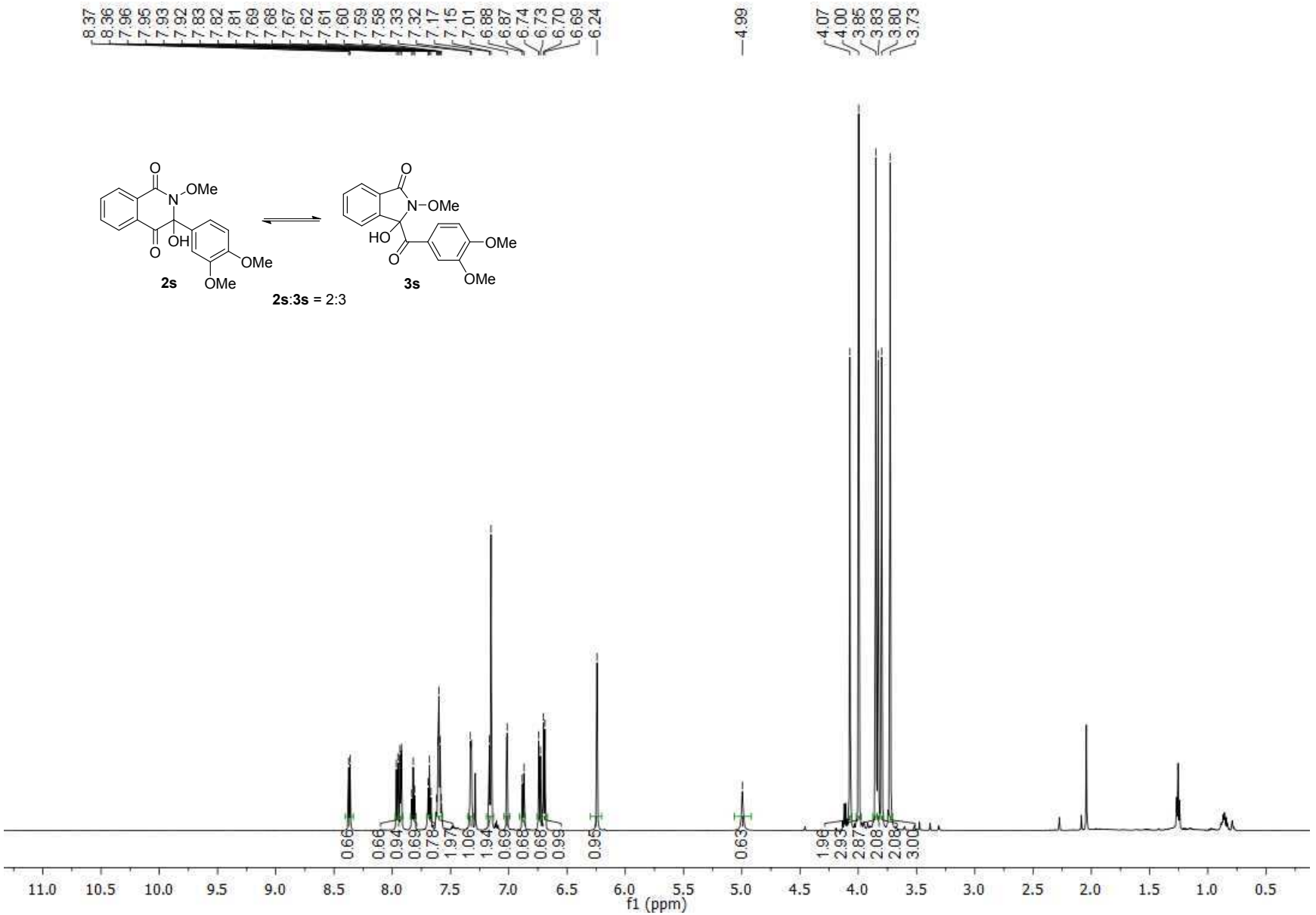
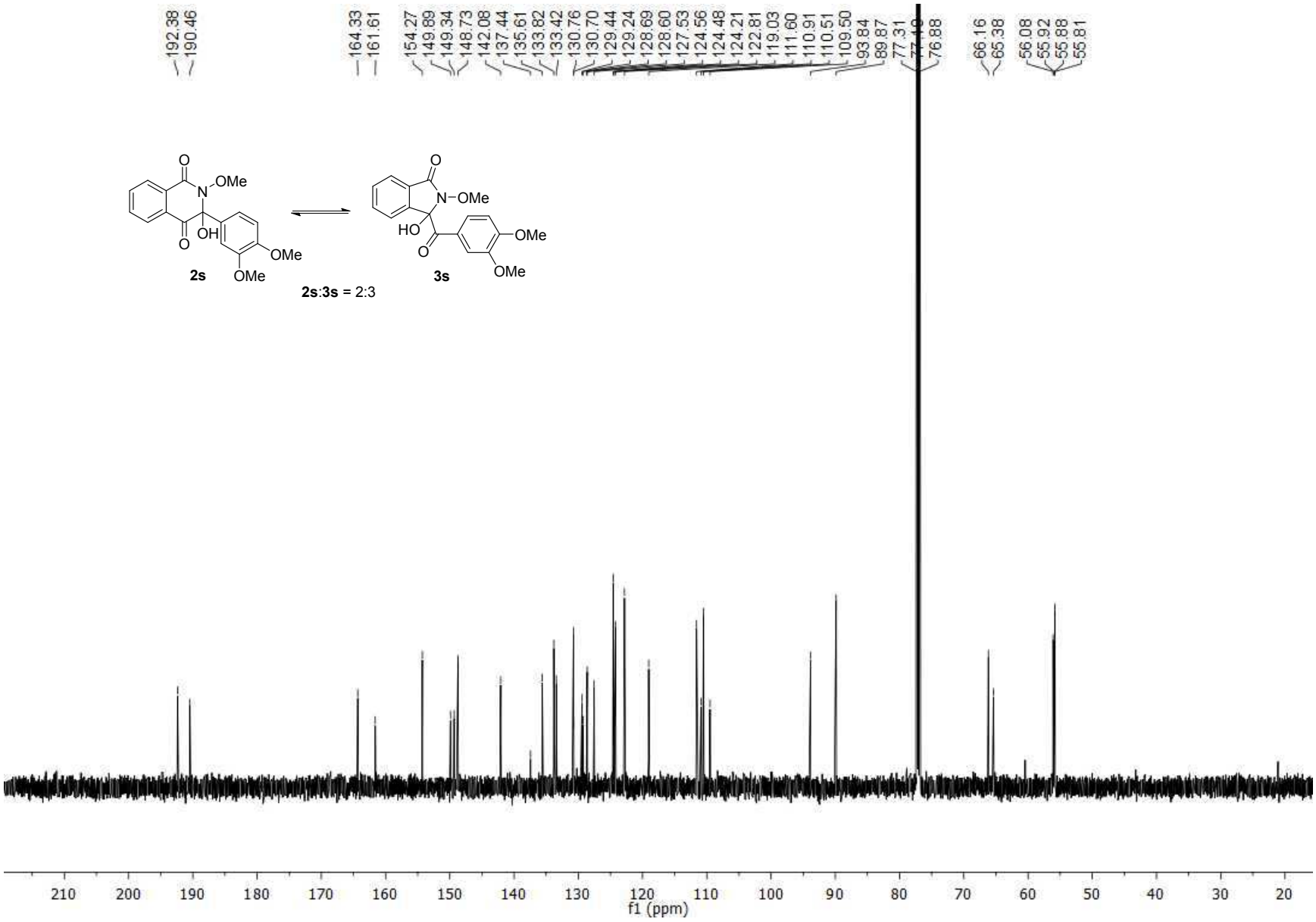
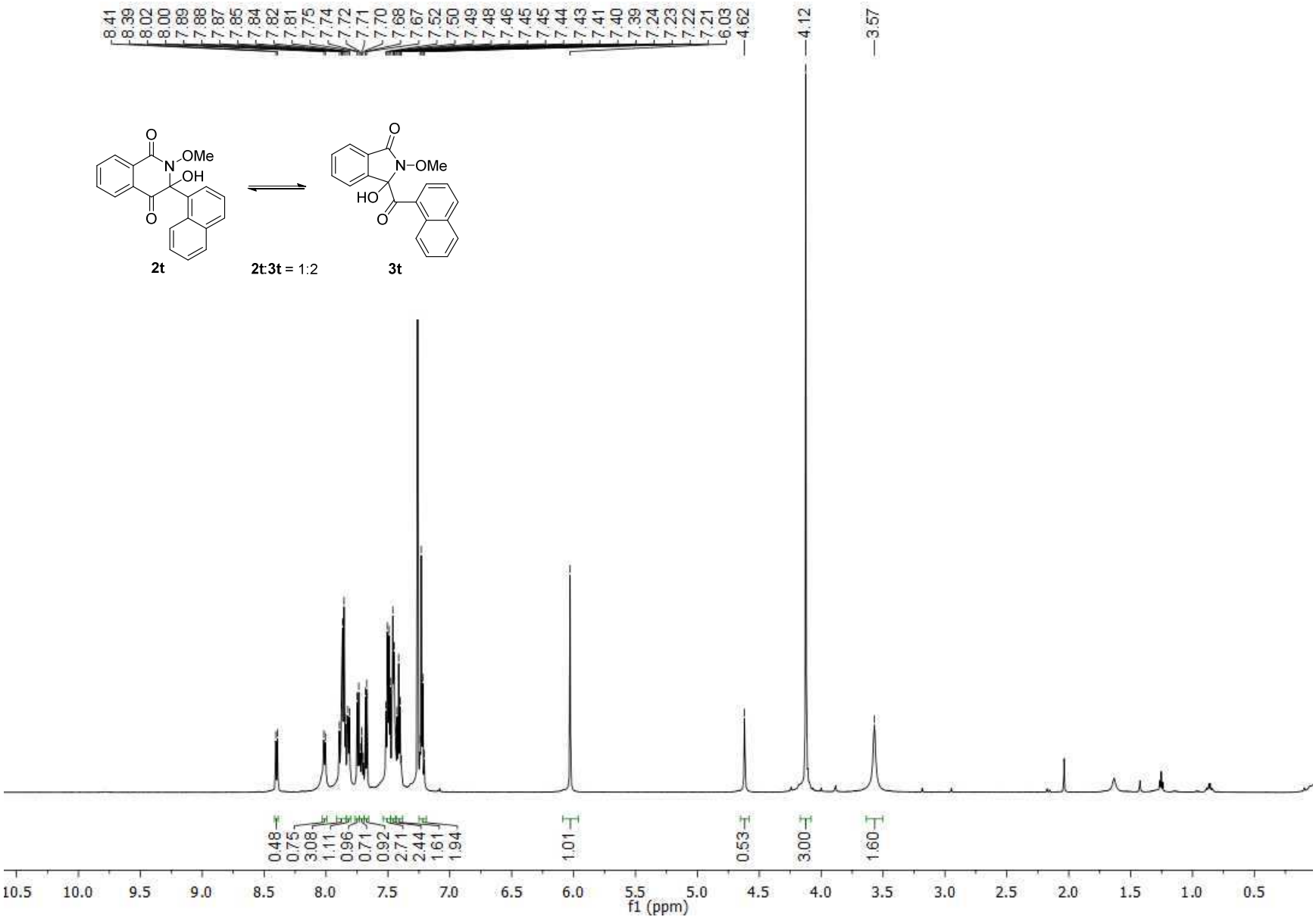
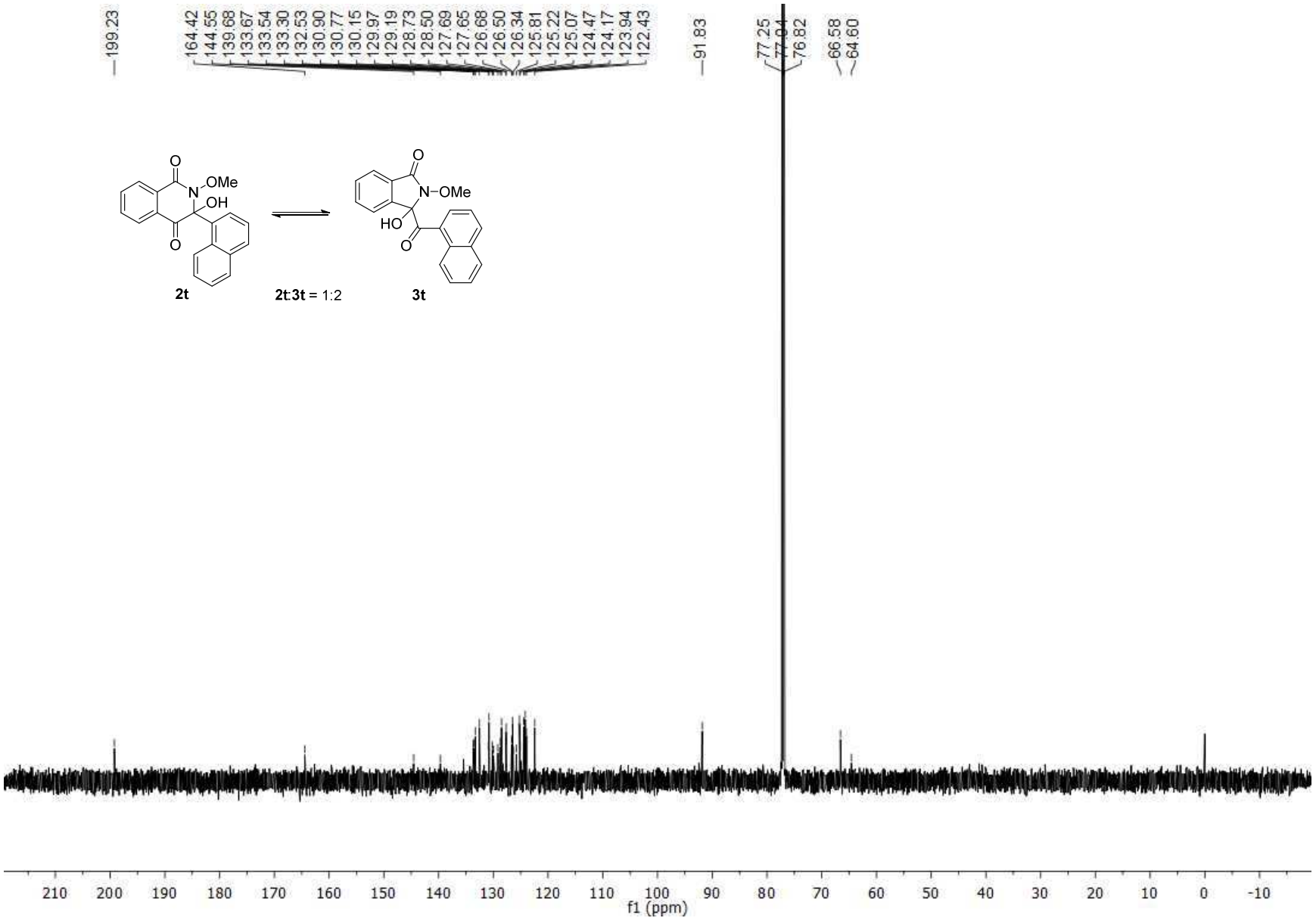
164.42
161.51
160.46
141.99
135.61
133.71
133.37
132.01
130.82
130.72
129.41
129.28
128.71
128.42
127.91
127.49
124.61
124.35
122.70
114.40
114.20
93.75
89.82
77.25
77.03
76.82
66.04
65.36
55.50
55.31
O
N
OMe
OH
O
OMe
2r
2r:3r = 1:1
O
N–OMe
HO
O
OMe
3r
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
f1 (ppm)


8.37
8.36
7.96
7.95
7.93
7.92
7.83
7.82
7.81
7.69
7.68
7.67
7.62
7.61
7.60
7.59
7.58
7.33
7.32
7.17
7.15
7.01
6.88
6.87
6.74
6.73
6.70
6.69
6.24
4.99
4.07
4.00
3.85
3.83
3.80
3.73
O
N
OMe
OH
O
OMe
OMe
2s
HO
N−OMe
O
O
OMe
OMe
3s
2s:3s = 2:3
0.66
0.66
0.94
0.69
0.78
1.97
1.06
1.94
0.69
0.68
0.68
0.99
0.95
0.63
1.96
2.93
2.87
2.08
2.08
3.00
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
f1 (ppm)


192.38
190.46
164.33
161.61
154.27
149.89
149.34
148.73
142.08
137.44
135.61
133.82
133.42
130.76
130.70
129.44
129.24
128.69
128.60
127.53
124.56
124.48
124.21
122.81
119.03
111.60
110.91
110.51
109.50
93.84
89.87
77.31
77.10
76.88
66.16
65.38
56.08
55.92
55.88
55.81
OMe
OH
OMe
OMe
2s
HO
N–OMe
OMe
OMe
3s
2s:3s = 2:3
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
f1 (ppm)


8.41
8.39
8.02
8.00
7.89
7.88
7.87
7.85
7.84
7.82
7.81
7.75
7.74
7.72
7.71
7.70
7.68
7.67
7.52
7.50
7.49
7.48
7.46
7.45
7.45
7.44
7.43
7.41
7.40
7.39
7.24
7.23
7.22
7.21
6.03
4.62
4.12
3.57
O
N
OMe
OH
O
2t
2t:3t = 1:2
O
N–OMe
HO
O
3t
0.48
0.75
3.08
1.11
0.96
0.71
0.92
2.71
2.44
1.61
1.94
1.01
0.53
3.00
1.60
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
f1 (ppm)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132.53
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129.19
128.73
128.50
127.69
127.65
126.68
126.50
126.34
125.81
125.22
125.07
124.47
124.17
123.94
122.43
91.83
77.25
77.04
76.82
66.58
64.60
O
N
OMe
OH
O
2t
2t:3t = 1:2
O
N–OMe
HO
O
3t
210
200
190
180
170
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150
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100
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60
50
40
30
20
10
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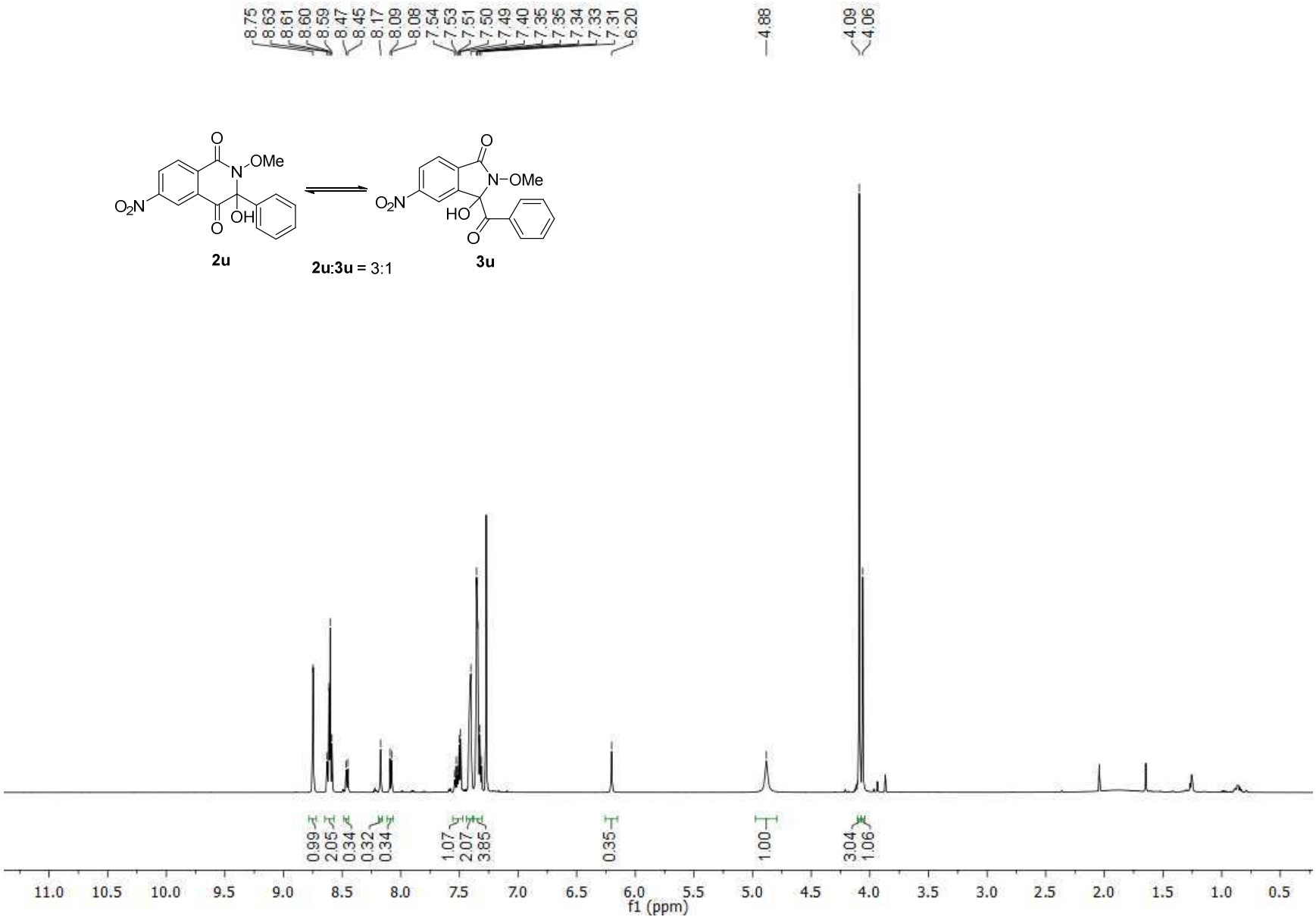
-10
f1 (ppm)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8.61
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8.47
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8.17
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8.08
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7.51
7.50
7.49
7.40
7.35
7.35
7.34
7.33
7.31
6.20
4.88
4.09
4.06
O
N
OMe
O2N
OH
O
2u
2u:3u = 3:1
O
N
OMe
O2N
HO
O
3u
0.99
2.05
0.34
0.32
0.34
1.07
2.07
3.85
0.35
1.00
3.04
1.06
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
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5.0
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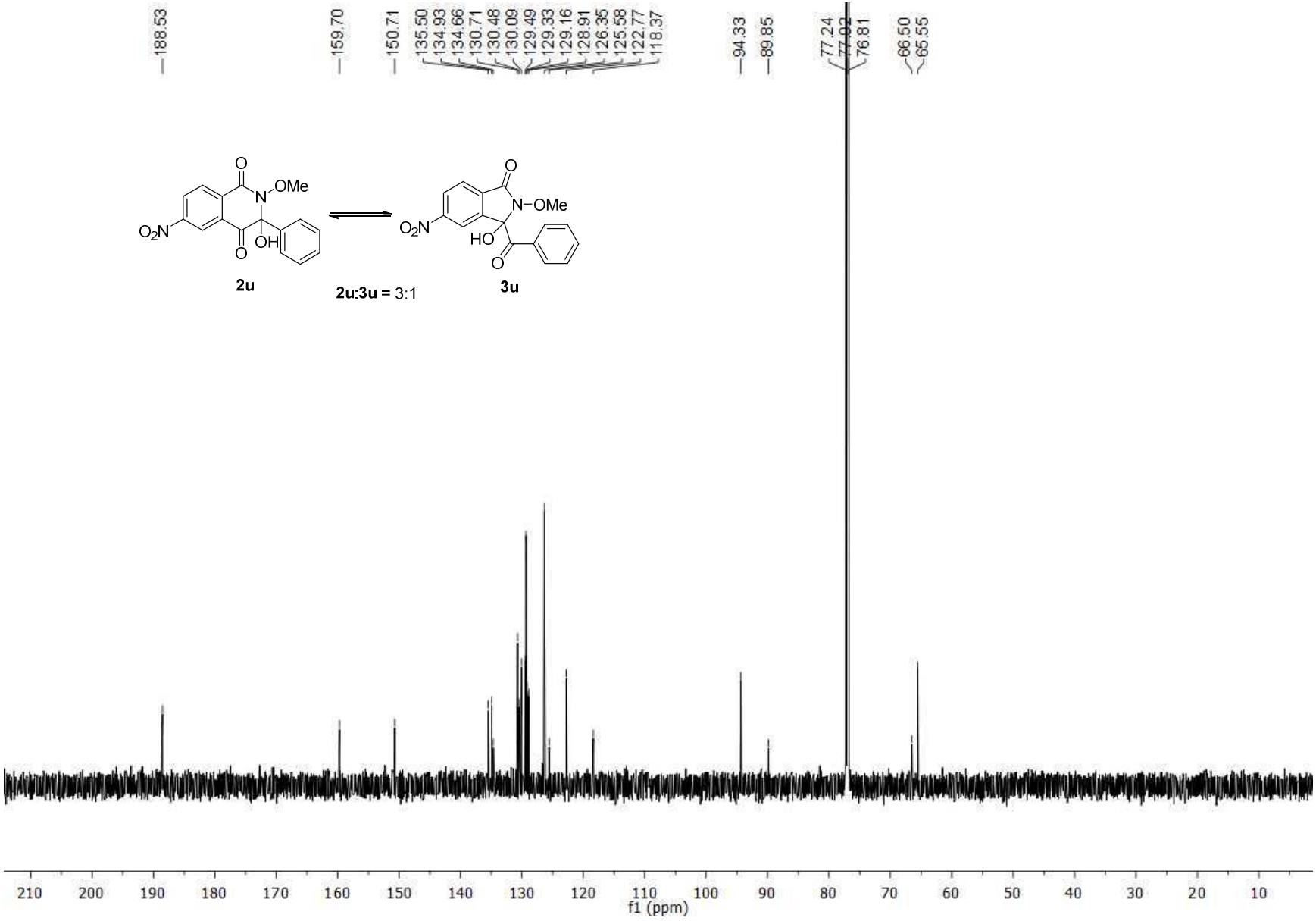
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)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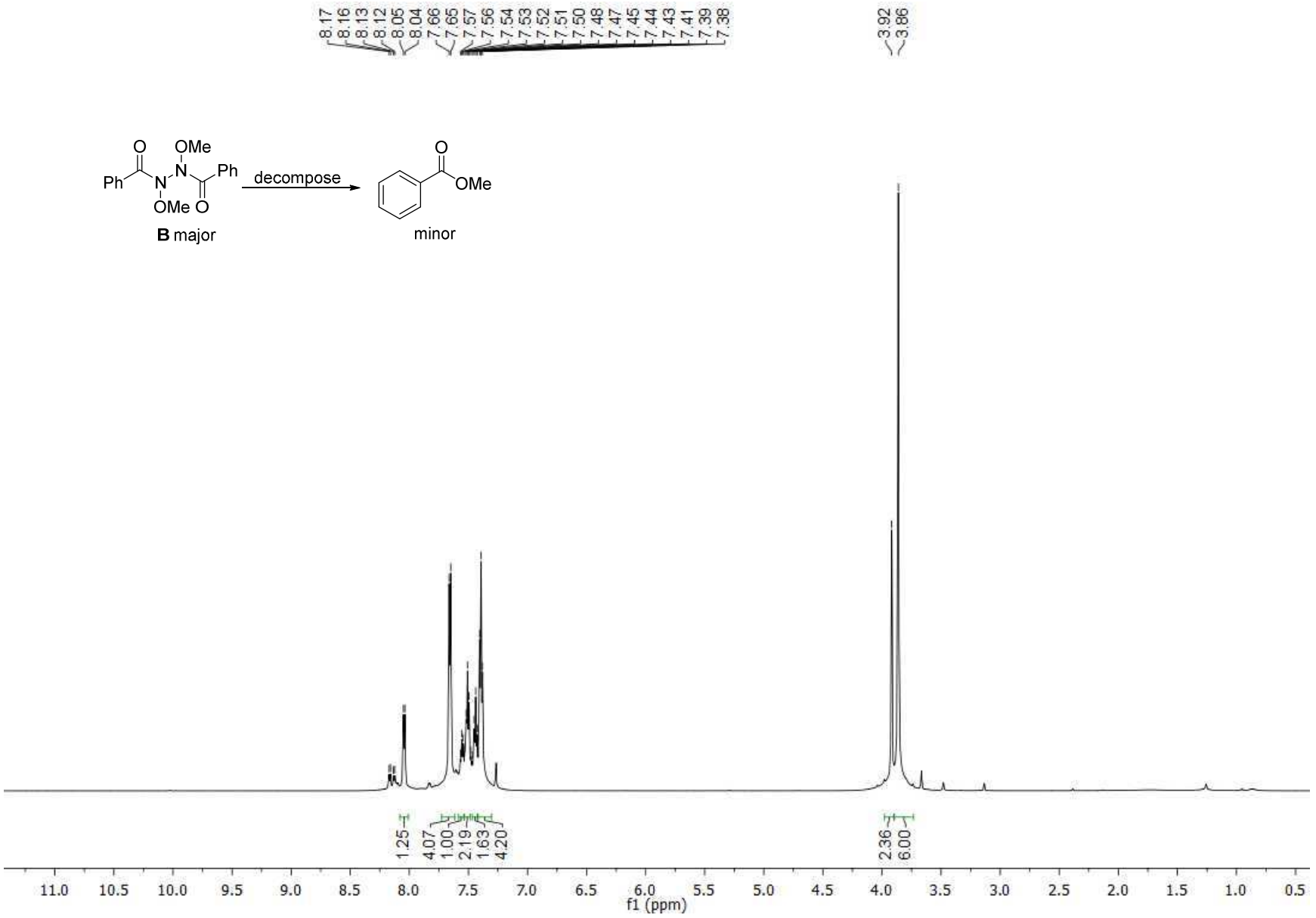
130.48
130.09
129.49
129.33
129.16
128.91
126.35
125.58
122.77
118.37
94.33
89.85
77.24
77.02
76.81
66.50
65.55
O
N
OMe
O2N
OH
O
2u
2u:3u = 3:1
O
N
OMe
O2N
HO
O
3u
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)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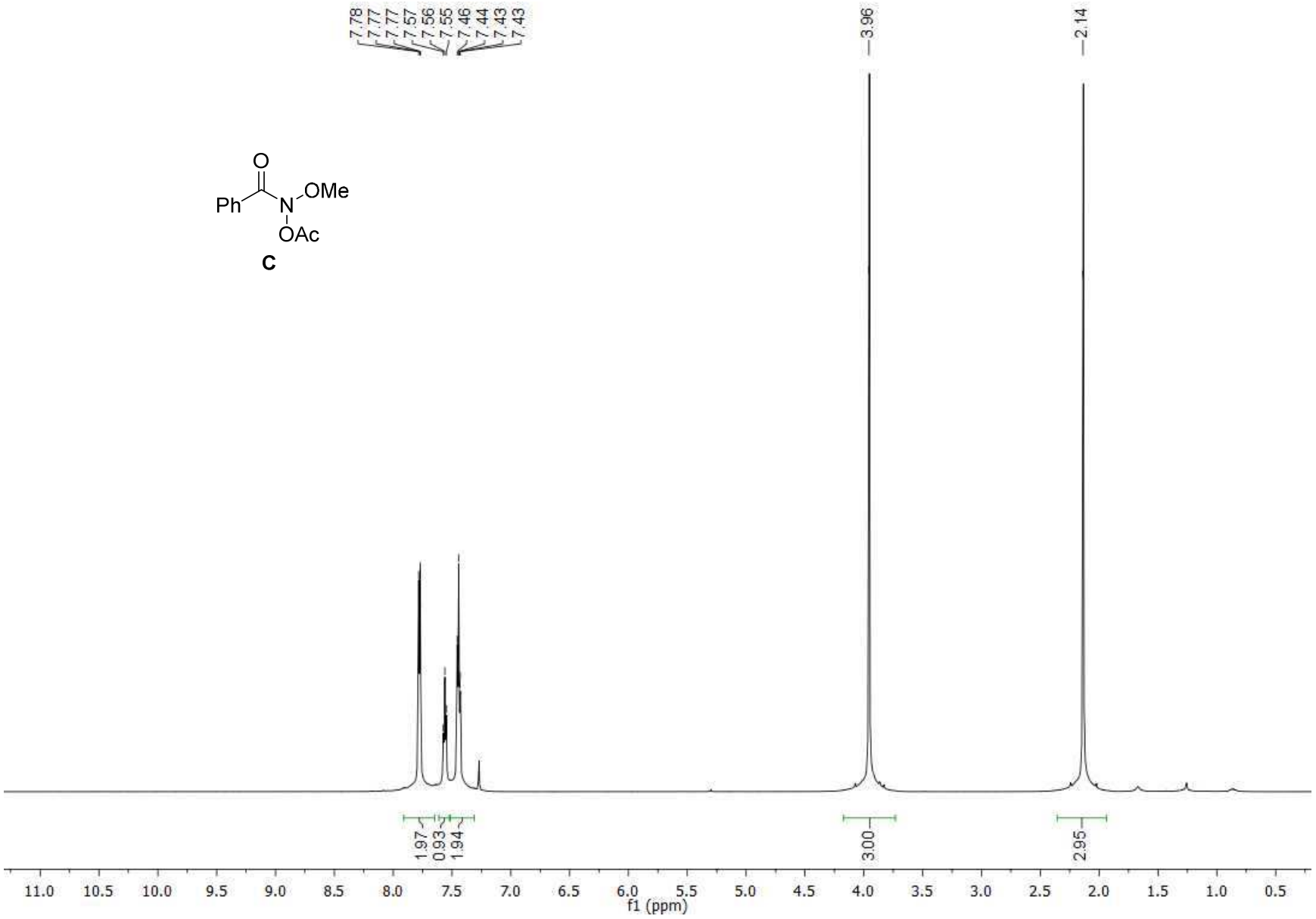
8.16
8.13
8.12
8.05
8.04
7.66
7.65
7.57
7.56
7.54
7.53
7.52
7.51
7.50
7.48
7.47
7.45
7.44
7.43
7.41
7.39
7.38
3.92
3.86
O
OMe
N
Ph
N
Ph
OMe O
B major
decompose
O
OMe
minor
1.25
4.07
1.00
2.19
1.63
4.20
2.36
6.00
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)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7.77
7.77
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7.55
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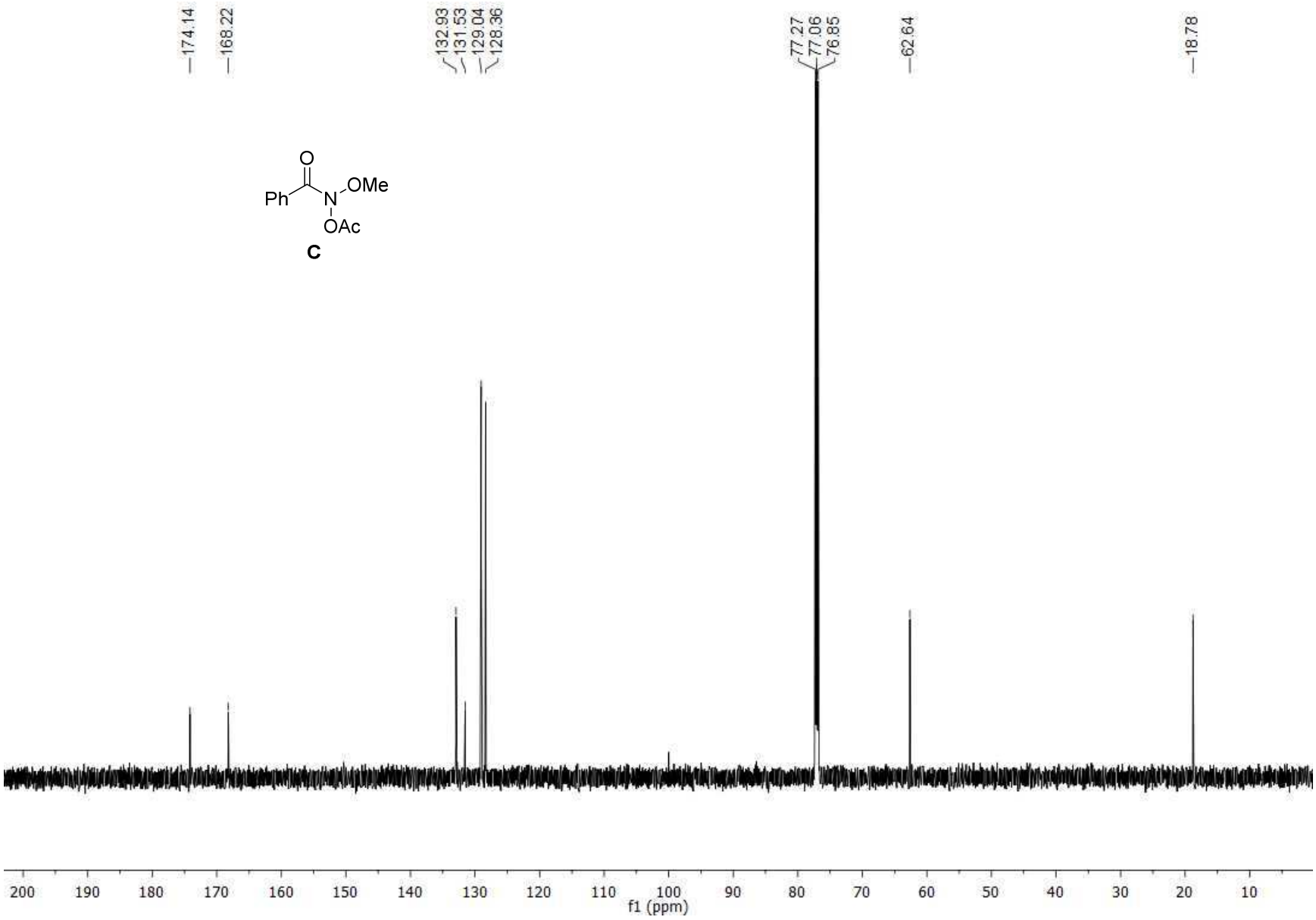
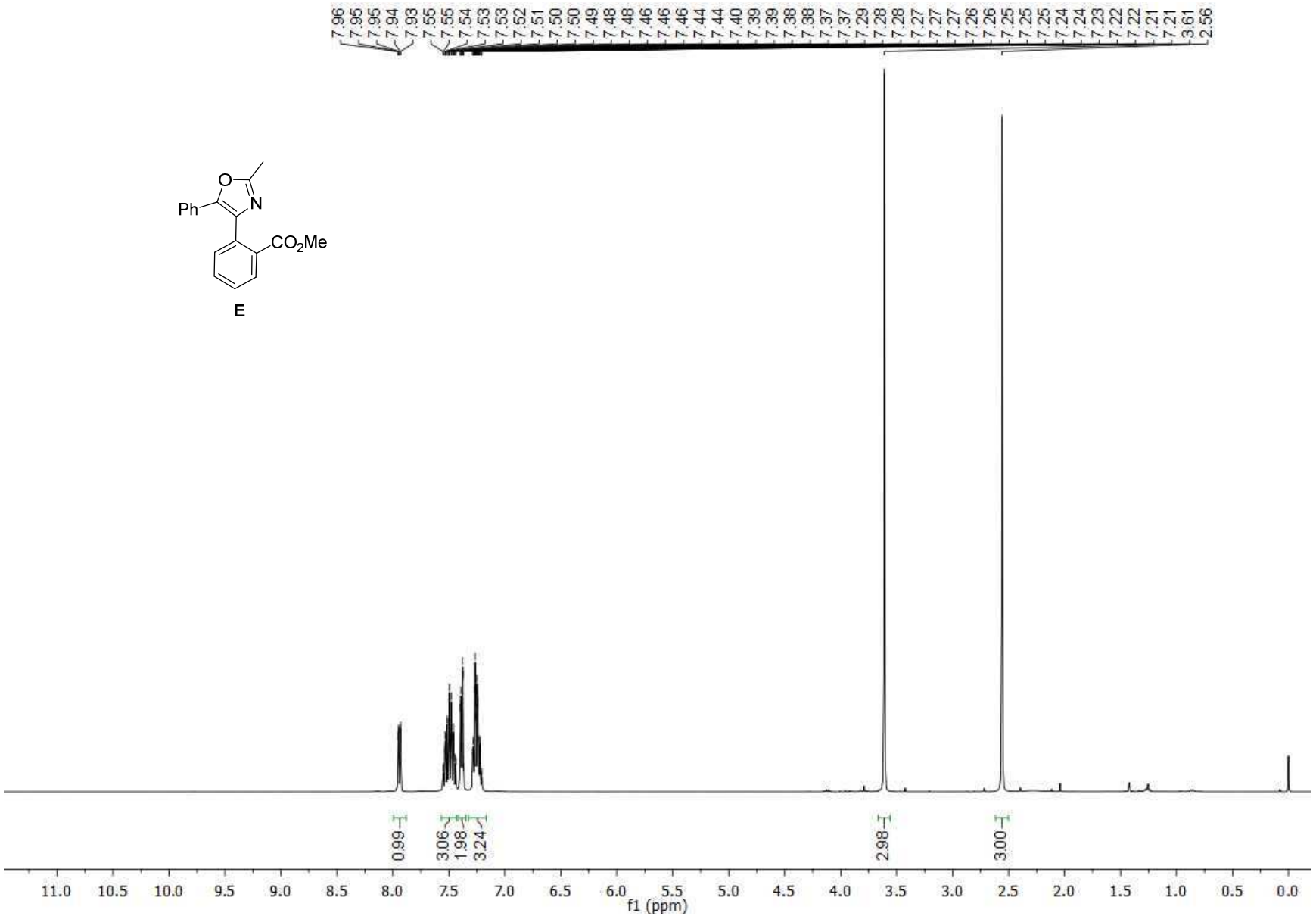
7.44
7.43
7.43
3.96
2.14
O
Ph
N
OMe
OAc
c
1.97
0.93
1.94
3.00
2.95
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)


174.14
168.22
132.93
131.53
129.04
128.36
77.27
77.06
76.85
62.64
18.78
O
Ph
N
OMe
OAc
C
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)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7.95
7.95
7.94
7.93
7.55
7.55
7.54
7.53
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7.51
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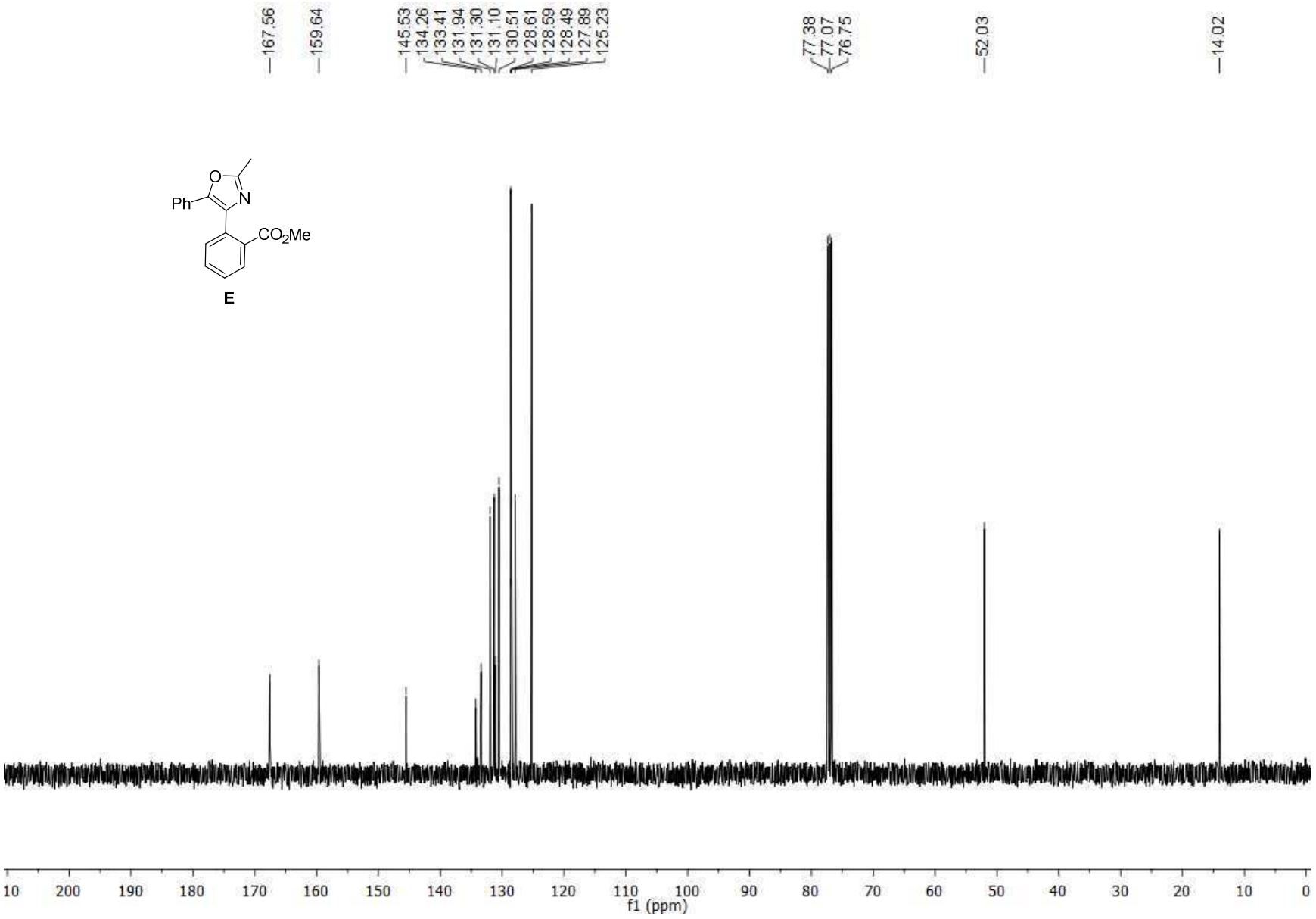
7.25
7.24
7.24
7.23
7.22
7.22
7.21
7.21
3.61
2.56
Ph
O
N
CO2Me
E
0.99
3.06
1.98
3.24
2.98
3.00
11.0
10.5
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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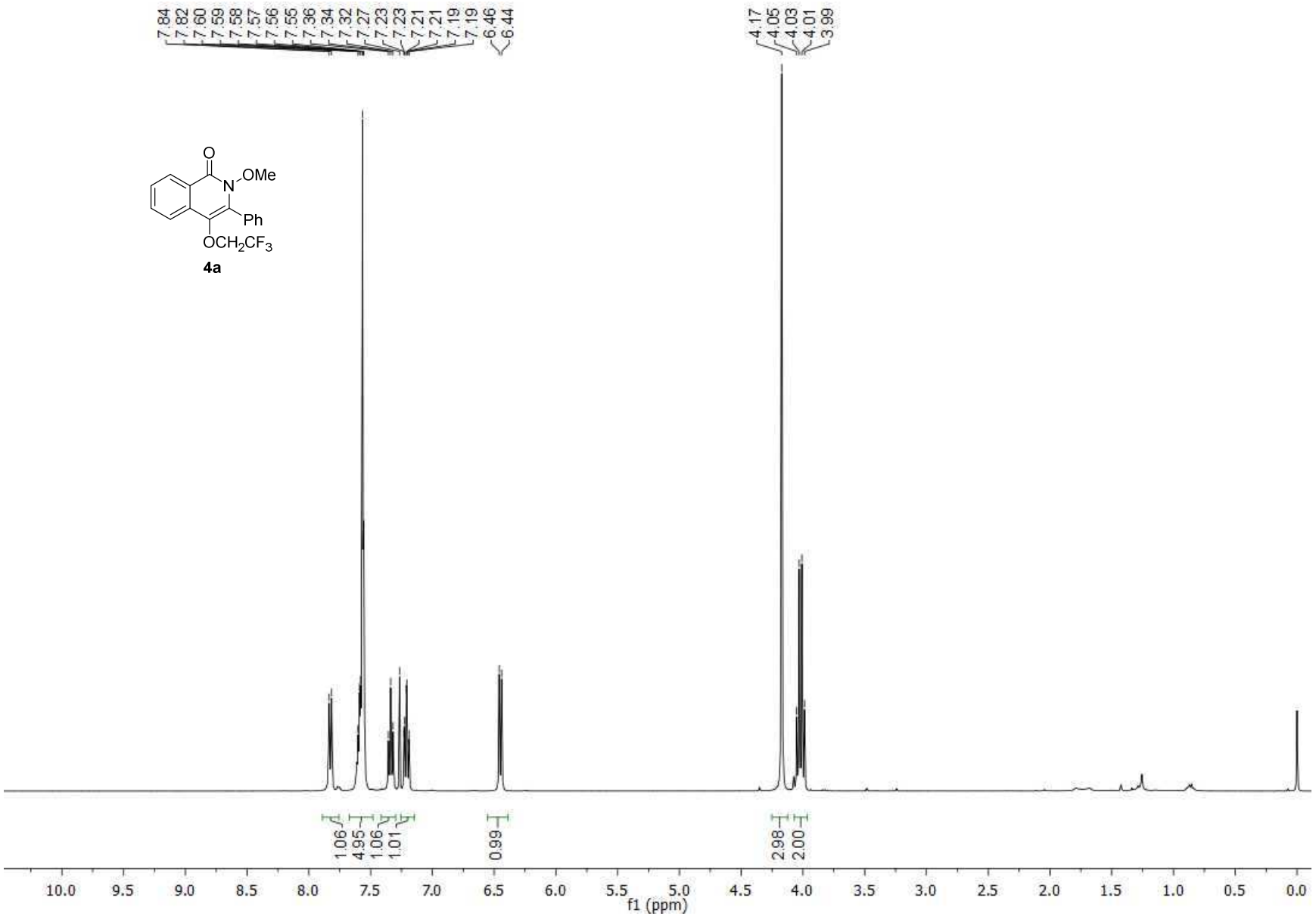
133.41
131.94
131.30
131.10
130.51
128.61
128.59
128.49
127.89
125.23
77.38
77.07
76.75
52.03
14.02
Ph
CO2Me
E
f1 (ppm)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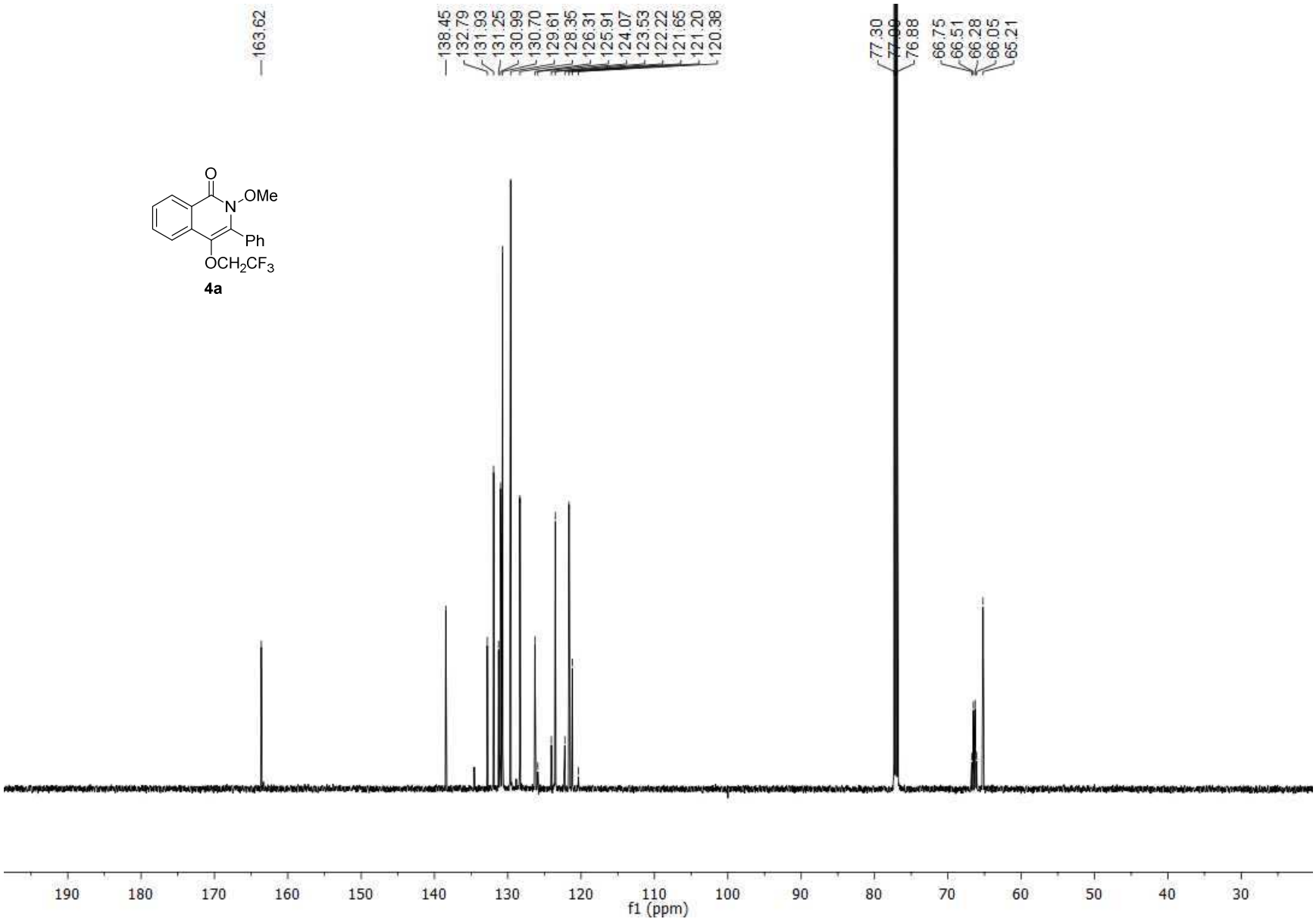
7.57
7.56
7.55
7.36
7.34
7.32
7.27
7.23
7.23
7.21
7.21
7.19
7.19
6.46
6.44
4.17
4.05
4.03
4.01
3.99
O
N
OMe
Ph
OCH2CF3
4a
1.06
4.95
1.06
1.01
0.99
2.98
2.00
10.0
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)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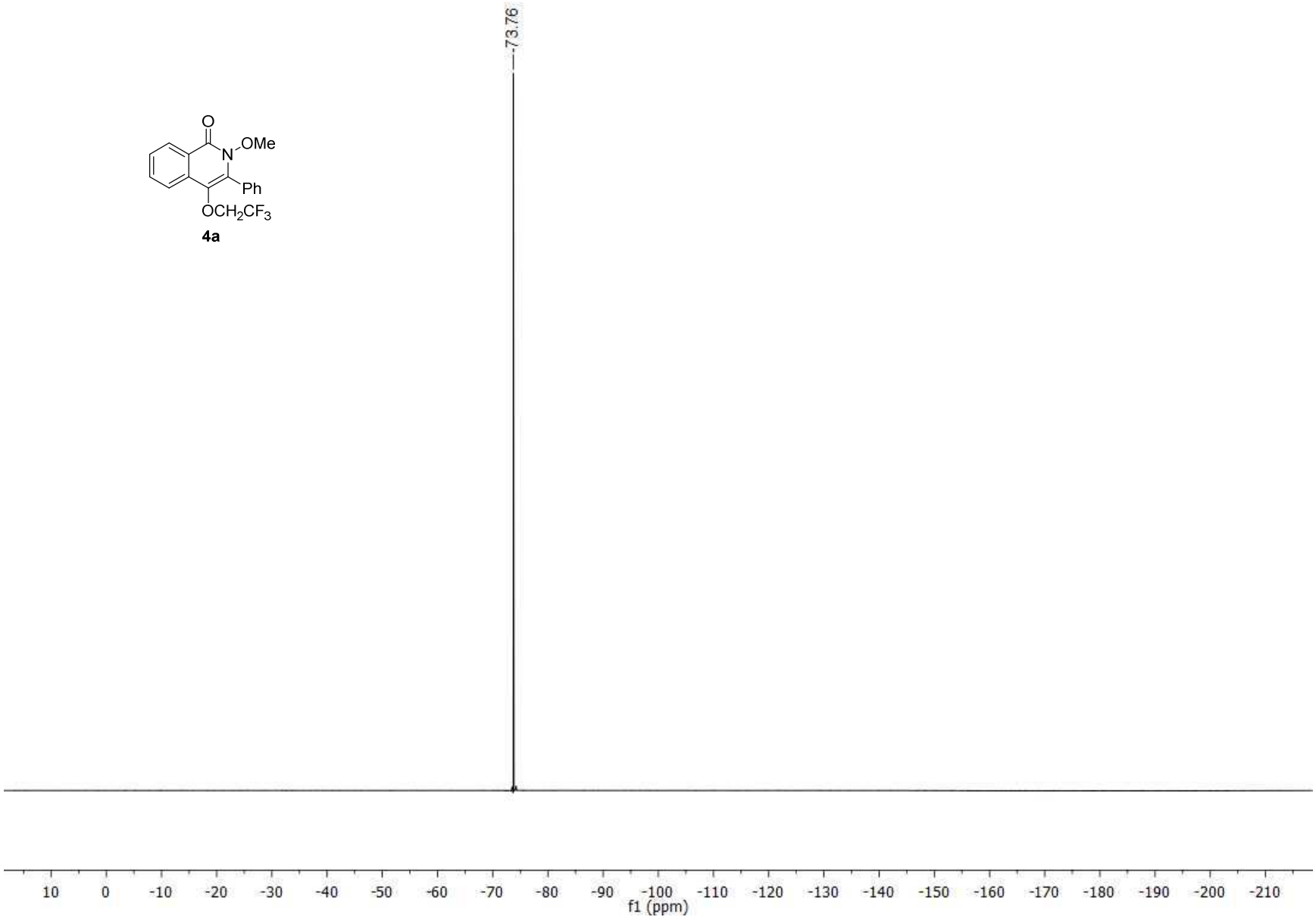
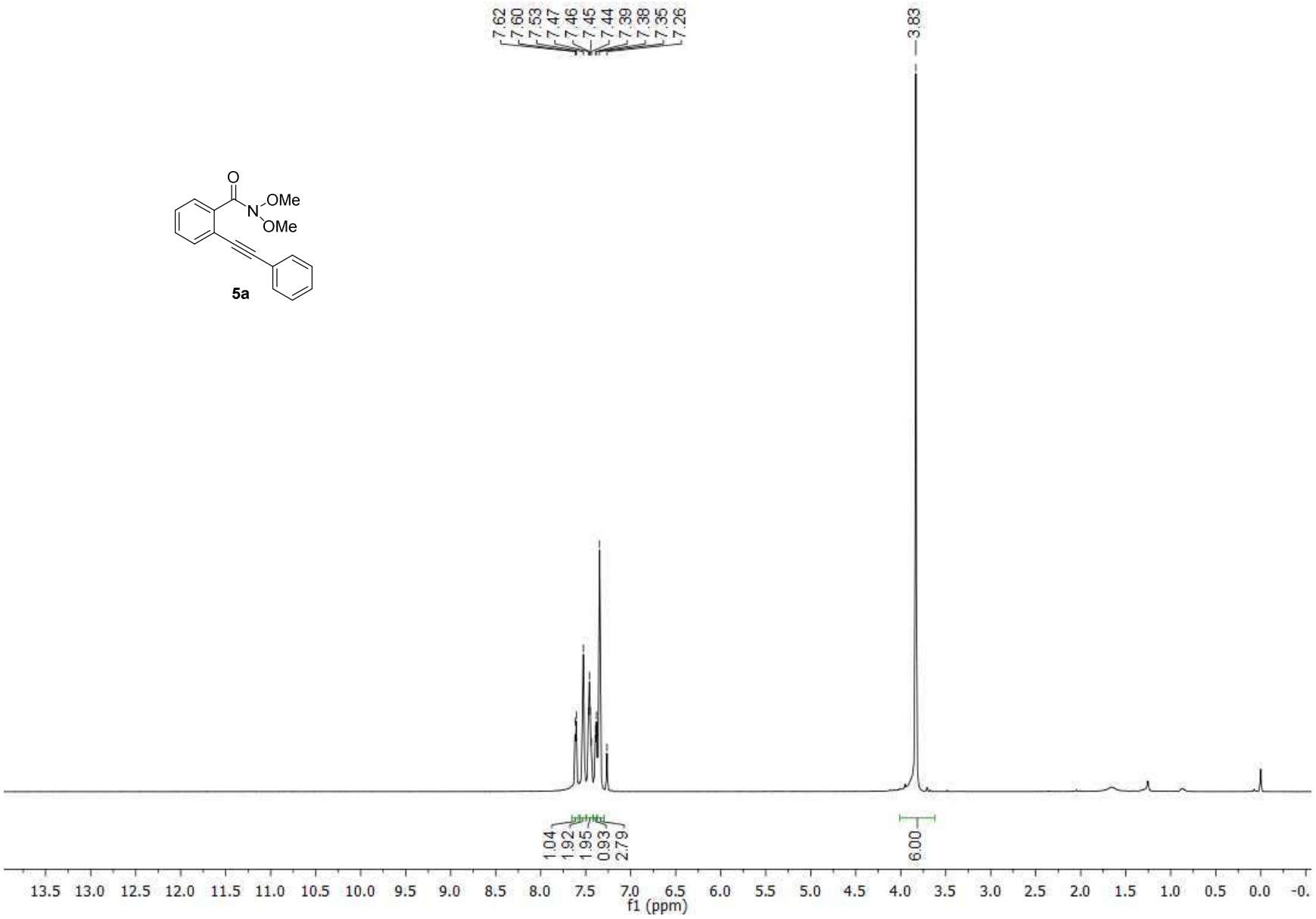
131.93
131.25
130.99
130.70
129.61
128.35
126.31
125.91
124.07
123.53
122.22
121.65
121.20
120.38
77.30
77.00
76.88
66.75
66.51
66.28
66.05
65.21
O
N
OMe
Ph
OCH2CF3
4a
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
f1 (ppm)


-73.76
O
N
OMe
Ph
OCH2CF3
4a
10
0
-10
-20
-30
-40
-50
-60
-70
-80
-90
-100
-110
-120
-130
-140
-150
-160
-170
-180
-190
-200
-210
f1 (ppm)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7.60
7.53
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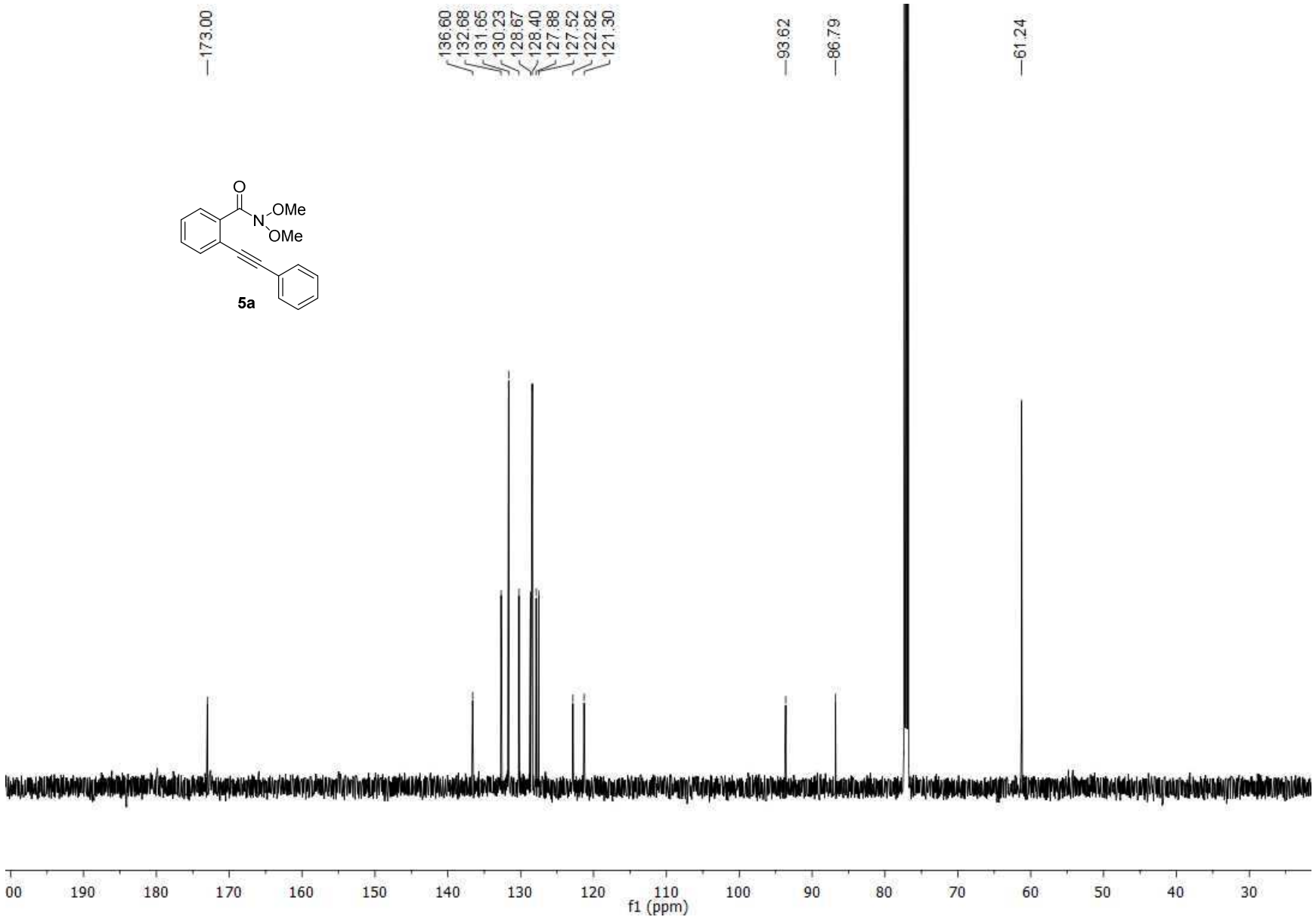
7.46
7.45
7.44
7.39
7.38
7.35
7.26
3.83
O
N
OMe
OMe
5a
1.04
1.92
1.95
0.93
2.79
6.00
13.5 13.0 12.5 12.0 11.5 11.0 10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.
f1 (ppm)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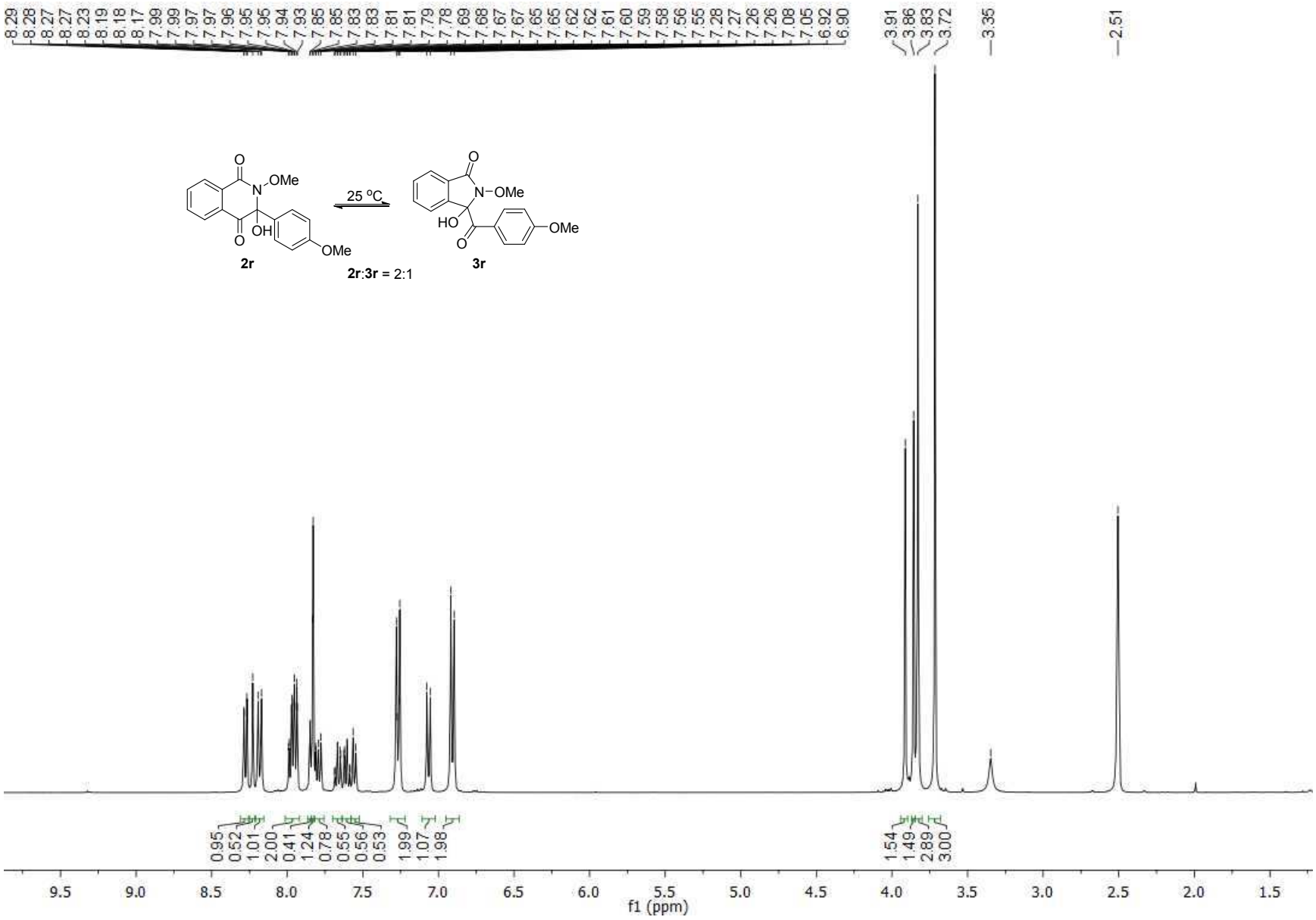
130.23
128.67
128.40
127.88
127.52
122.82
121.30
93.62
86.79
61.24
O
N
OMe
OMe
5a
00
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
f1 (ppm)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8.19
8.18
8.17
7.99
7.99
7.97
7.97
7.96
7.95
7.95
7.94
7.93
7.85
7.85
7.83
7.83
7.81
7.81
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7.78
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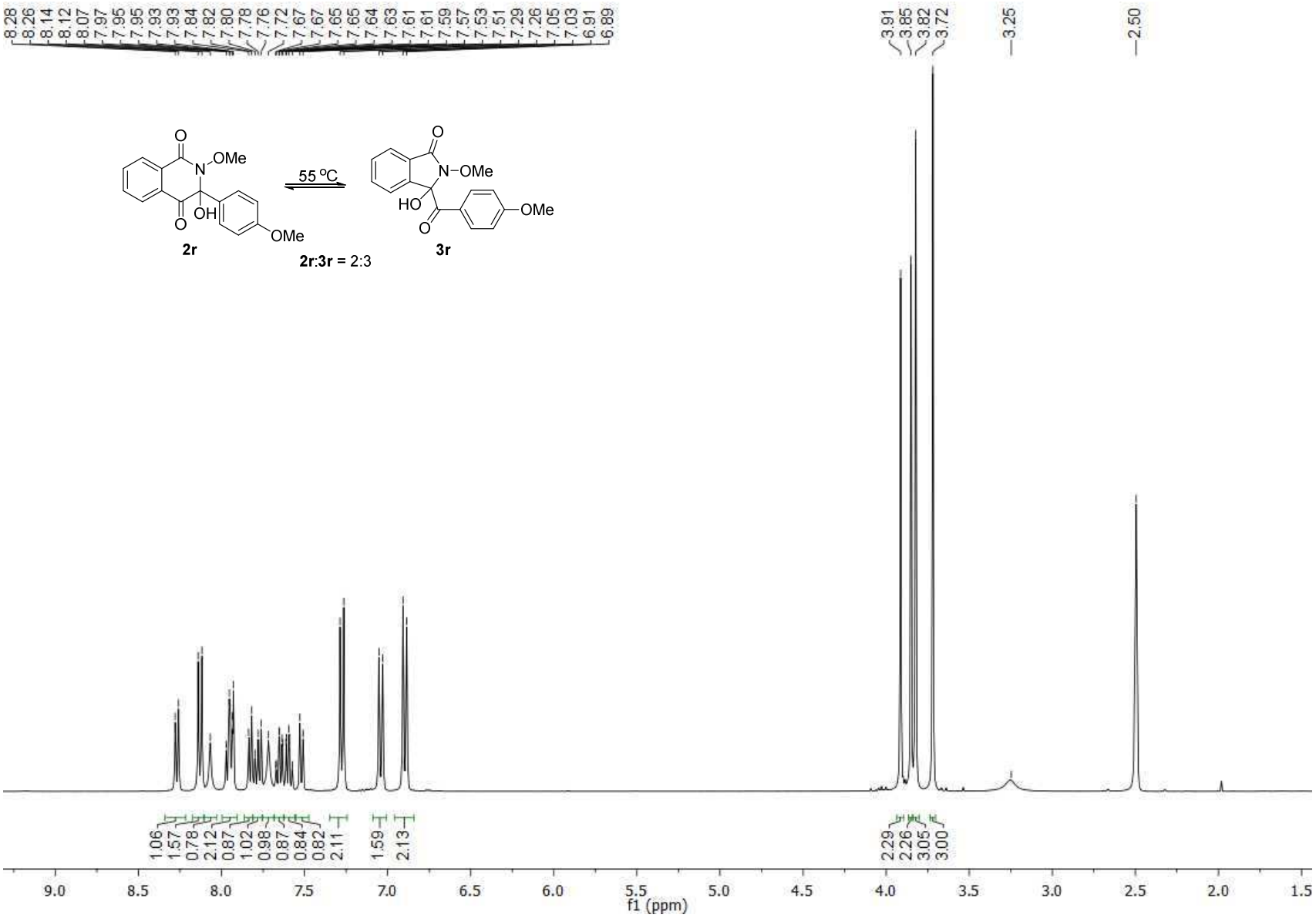
7.67
7.65
7.65
7.62
7.62
7.61
7.60
7.59
7.58
7.56
7.55
7.28
7.27
7.26
7.26
7.08
7.05
6.92
6.90
3.91
3.86
3.83
3.72
3.35
2.51
O
N
OMe
OH
O
OMe
2r
25 °C
2r:3r = 2:1
O
N−OMe
HO
O
OMe
3r
0.95
0.52
1.01
2.00
0.41
1.24
0.78
0.55
0.56
0.53
1.99
1.07
1.98
1.54
1.49
2.89
3.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
f1 (ppm)


8.28
8.26
8.14
8.12
8.07
7.97
7.95
7.95
7.93
7.93
7.84
7.82
7.80
7.78
7.76
7.72
7.67
7.67
7.65
7.65
7.64
7.63
7.61
7.61
7.59
7.57
7.53
7.51
7.29
7.26
7.05
7.03
6.91
6.89
3.91
3.85
3.82
3.72
3.25
2.50
O
N
OMe
OH
O
OMe
2r
55 °C
2r:3r = 2:3
O
N−OMe
HO
O
OMe
3r
1.06
1.57
0.78
2.12
0.87
1.02
0.98
0.87
0.84
0.82
2.11
1.59
2.13
2.29
2.26
3.05
3.00
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
f1 (ppm)

X-ray Data of Compound 2a

Structure of Compound **2a**

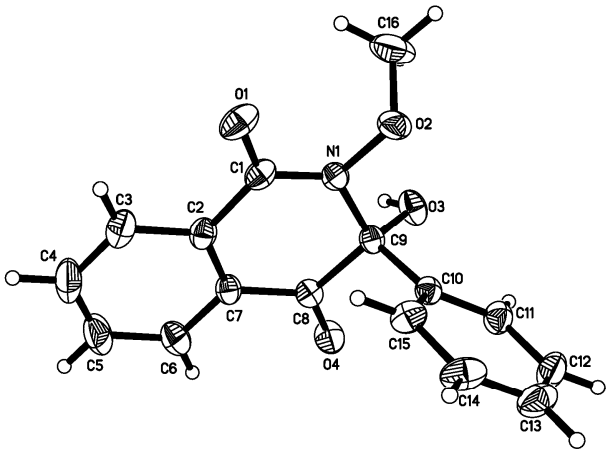


Fig.1. Molecular structure of the title compound showing the atom labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented as small spheres of arbitrary radii.

Data of Compound **2a**

Table 1. Crystal data and structure refinement for shelxl.

Identification code	**shelxl**
Empirical formula	**C16 H13 N O4**
Formula weight	**283.27**
Temperature	**293(2) K**
Wavelength	**0.71073 A**
Crystal system, space group	**Orthorhombic, P2(1)2(1)2(1)**
Unit cell dimensions	**a = 5.8370(12) A alpha = 90 deg.**
	b = 14.149(3) A beta = 90 deg.
	c = 17.650(4) A gamma = 90 deg.
Volume	**1457.7(5) A^3**
Z, Calculated density	**4, 1.291 Mg/m^3**
Absorption coefficient	**0.094 mm^-1**

F(000)	**592**
Crystal size	**0.20 x 0.18 x 0.12 mm**
Theta range for data collection	**2.72 to 25.02 deg.**
Limiting indices	**-6<=h<=6, -16<=k<=15, -21<=l<=20**
Reflections collected / unique	**13466 / 2553 [R(int) = 0.0584]**
Completeness to theta = 25.02	**99.8 %**
Absorption correction	**Semi-empirical from equivalents**
Max. and min. transmission	**0.9888 and 0.9815**
Refinement method	**Full-matrix least-squares on F^2**
Data / restraints / parameters	**2553 / 0 / 193**
Goodness-of-fit on F^2	**1.062**
Final R indices [I>2sigma(I)]	**R1 = 0.0423, wR2 = 0.0890**
R indices (all data)	**R1 = 0.0539, wR2 = 0.0946**
Absolute structure parameter	**-0.2(14)**
Extinction coefficient	**0.053(4)**
Largest diff. peak and hole	**0.136 and -0.134 e.A^-3**

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (A^2 x 10^3) for shelxl.

U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

	x	y	z	U(eq)
O(1)	9132(3)	6088(1)	8114(1)	56(1)
O(2)	6791(3)	5936(1)	6852(1)	52(1)
O(3)	2197(3)	5431(1)	7035(1)	51(1)
O(4)	1313(3)	4105(1)	8173(1)	53(1)
N(1)	5864(3)	5616(1)	7539(1)	41(1)

C(1)	7288(4)	5671(1)	8143(1)	41(1)
C(2)	6439(4)	5232(1)	8853(1)	42(1)
C(3)	7684(5)	5369(2)	9514(1)	58(1)
C(4)	6925(6)	4983(2)	10187(2)	73(1)
C(5)	4937(6)	4459(2)	10206(1)	72(1)
C(6)	3691(5)	4310(2)	9553(1)	56(1)
C(7)	4436(4)	4697(2)	8872(1)	38(1)
C(8)	3079(3)	4554(1)	8177(1)	37(1)
C(9)	3951(4)	4957(1)	7410(1)	35(1)
C(10)	4734(3)	4126(1)	6927(1)	35(1)
C(11)	3285(4)	3747(2)	6383(1)	48(1)
C(12)	3965(5)	2961(2)	5974(1)	64(1)
C(13)	6054(6)	2547(2)	6104(2)	65(1)
C(14)	7493(5)	2911(2)	6642(2)	60(1)
C(15)	6829(4)	3704(2)	7054(1)	48(1)
C(16)	6281(7)	6929(2)	6754(2)	83(1)

Table 3. Bond lengths [A] and angles [deg] for shelxl.

O(1)-C(1)	1.228(3)
O(2)-N(1)	1.403(2)
O(2)-C(16)	1.446(3)
O(3)-C(9)	1.391(2)
O(3)-H(3)	0.8200
O(4)-C(8)	1.210(2)
N(1)-C(1)	1.354(3)
N(1)-C(9)	1.472(3)
C(1)-C(2)	1.484(3)
C(2)-C(3)	1.388(3)
C(2)-C(7)	1.393(3)
C(3)-C(4)	1.381(4)

C(3)-H(3A)	0.9300
C(4)-C(5)	1.377(4)
C(4)-H(4)	0.9300
C(5)-C(6)	1.378(4)
C(5)-H(5)	0.9300
C(6)-C(7)	1.392(3)
C(6)-H(6)	0.9300
C(7)-C(8)	1.473(3)
C(8)-C(9)	1.555(3)
C(9)-C(10)	1.523(3)
C(10)-C(15)	1.379(3)
C(10)-C(11)	1.387(3)
C(11)-C(12)	1.384(3)
C(11)-H(11)	0.9300
C(12)-C(13)	1.373(4)
C(12)-H(12)	0.9300
C(13)-C(14)	1.369(4)
C(13)-H(13)	0.9300
C(14)-C(15)	1.392(3)
C(14)-H(14)	0.9300
C(15)-H(15)	0.9300
C(16)-H(16A)	0.9600
C(16)-H(16B)	0.9600
C(16)-H(16C)	0.9600

N(1)-O(2)-C(16)	109.71(17)
C(9)-O(3)-H(3)	109.5
C(1)-N(1)-O(2)	115.13(17)
C(1)-N(1)-C(9)	128.62(17)
O(2)-N(1)-C(9)	111.29(15)
O(1)-C(1)-N(1)	122.3(2)

O(1)-C(1)-C(2)	121.9(2)
N(1)-C(1)-C(2)	115.79(18)
C(3)-C(2)-C(7)	119.7(2)
C(3)-C(2)-C(1)	118.4(2)
C(7)-C(2)-C(1)	121.85(19)
C(4)-C(3)-C(2)	120.0(3)
C(4)-C(3)-H(3A)	120.0
C(2)-C(3)-H(3A)	120.0
C(5)-C(4)-C(3)	120.3(3)
C(5)-C(4)-H(4)	119.8
C(3)-C(4)-H(4)	119.8
C(4)-C(5)-C(6)	120.4(3)
C(4)-C(5)-H(5)	119.8
C(6)-C(5)-H(5)	119.8
C(5)-C(6)-C(7)	119.8(2)
C(5)-C(6)-H(6)	120.1
C(7)-C(6)-H(6)	120.1
C(6)-C(7)-C(2)	119.8(2)
C(6)-C(7)-C(8)	119.8(2)
C(2)-C(7)-C(8)	120.42(19)
O(4)-C(8)-C(7)	122.35(18)
O(4)-C(8)-C(9)	117.77(18)
C(7)-C(8)-C(9)	119.85(17)
O(3)-C(9)-N(1)	109.01(15)
O(3)-C(9)-C(10)	109.10(16)
N(1)-C(9)-C(10)	110.37(16)
O(3)-C(9)-C(8)	110.51(16)
N(1)-C(9)-C(8)	110.24(16)
C(10)-C(9)-C(8)	107.60(15)
C(15)-C(10)-C(11)	119.1(2)
C(15)-C(10)-C(9)	120.64(19)

C(11)-C(10)-C(9)	120.20(19)
C(12)-C(11)-C(10)	119.8(2)
C(12)-C(11)-H(11)	120.1
C(10)-C(11)-H(11)	120.1
C(13)-C(12)-C(11)	120.7(2)
C(13)-C(12)-H(12)	119.6
C(11)-C(12)-H(12)	119.6
C(14)-C(13)-C(12)	120.0(2)
C(14)-C(13)-H(13)	120.0
C(12)-C(13)-H(13)	120.0
C(13)-C(14)-C(15)	119.7(3)
C(13)-C(14)-H(14)	120.2
C(15)-C(14)-H(14)	120.2
C(10)-C(15)-C(14)	120.7(2)
C(10)-C(15)-H(15)	119.6
C(14)-C(15)-H(15)	119.6
O(2)-C(16)-H(16A)	109.5
O(2)-C(16)-H(16B)	109.5
H(16A)-C(16)-H(16B)	109.5
O(2)-C(16)-H(16C)	109.5
H(16A)-C(16)-H(16C)	109.5
H(16B)-C(16)-H(16C)	109.5

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (A^2 x 10^3) for shelxl.
The anisotropic displacement factor exponent takes the form:
-2 pi^2 [h^2 a*^2 U11 + ... + 2 h k a* b* U12]

U11	U22	U33	U23	U13	U12

O(1)	43(1)	54(1)	71(1)	-20(1)	10(1)	-13(1)
O(2)	70(1)	41(1)	44(1)	3(1)	20(1)	-5(1)
O(3)	47(1)	64(1)	41(1)	5(1)	0(1)	0(1)
O(4)	40(1)	70(1)	49(1)	6(1)	1(1)	-13(1)
N(1)	48(1)	41(1)	33(1)	-1(1)	7(1)	-7(1)
C(1)	37(1)	38(1)	47(1)	-12(1)	6(1)	2(1)
C(2)	41(1)	46(1)	38(1)	-8(1)	-1(1)	6(1)
C(3)	57(2)	65(2)	53(2)	-16(1)	-14(1)	3(1)
C(4)	87(2)	91(2)	42(2)	-12(1)	-20(2)	10(2)
C(5)	94(2)	88(2)	32(1)	2(1)	2(1)	11(2)
C(6)	62(2)	66(2)	41(1)	7(1)	6(1)	3(1)
C(7)	37(1)	45(1)	33(1)	-2(1)	2(1)	4(1)
C(8)	31(1)	41(1)	38(1)	0(1)	4(1)	4(1)
C(9)	33(1)	40(1)	33(1)	1(1)	-3(1)	3(1)
C(10)	34(1)	39(1)	33(1)	1(1)	3(1)	-3(1)
C(11)	47(1)	54(1)	44(1)	-3(1)	-5(1)	-5(1)
C(12)	80(2)	62(2)	48(1)	-17(1)	-1(1)	-19(2)
C(13)	79(2)	47(2)	70(2)	-14(1)	31(2)	-10(1)
C(14)	49(1)	42(1)	88(2)	-3(1)	20(1)	5(1)
C(15)	39(1)	43(1)	61(1)	-4(1)	-1(1)	0(1)
C(16)	122(3)	44(2)	82(2)	18(1)	32(2)	2(2)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (A^2 x 10^3) for shelxl.

	x	y	z	U(eq)
H(3)	1370	5694	7346	76

H(3A)	9029	5721	9503	70
H(4)	7760	5077	10629	88
H(5)	4432	4205	10662	86
H(6)	2356	3952	9569	67
H(11)	1861	4020	6294	58
H(12)	2996	2712	5606	76
H(13)	6492	2018	5826	78
H(14)	8909	2631	6732	72
H(15)	7808	3951	7419	57
H(16A)	4653	7023	6772	124
H(16B)	6858	7139	6273	124
H(16C)	6995	7285	7152	124

Table 6. Torsion angles [deg] for shelxl.

C(16)-O(2)-N(1)-C(1)	-91.4(3)
C(16)-O(2)-N(1)-C(9)	111.3(2)
O(2)-N(1)-C(1)-O(1)	9.5(3)
C(9)-N(1)-C(1)-O(1)	162.19(19)
O(2)-N(1)-C(1)-C(2)	-173.00(16)
C(9)-N(1)-C(1)-C(2)	-20.4(3)
O(1)-C(1)-C(2)-C(3)	5.5(3)
N(1)-C(1)-C(2)-C(3)	-171.93(19)
O(1)-C(1)-C(2)-C(7)	-174.92(19)
N(1)-C(1)-C(2)-C(7)	7.6(3)
C(7)-C(2)-C(3)-C(4)	-0.5(3)
C(1)-C(2)-C(3)-C(4)	179.0(2)
C(2)-C(3)-C(4)-C(5)	0.1(4)
C(3)-C(4)-C(5)-C(6)	0.4(4)
C(4)-C(5)-C(6)-C(7)	-0.5(4)
C(5)-C(6)-C(7)-C(2)	0.1(4)

C(5)-C(6)-C(7)-C(8)	-178.7(2)
C(3)-C(2)-C(7)-C(6)	0.5(3)
C(1)-C(2)-C(7)-C(6)	-179.1(2)
C(3)-C(2)-C(7)-C(8)	179.25(19)
C(1)-C(2)-C(7)-C(8)	-0.3(3)
C(6)-C(7)-C(8)-O(4)	-0.1(3)
C(2)-C(7)-C(8)-O(4)	-178.89(19)
C(6)-C(7)-C(8)-C(9)	-177.96(19)
C(2)-C(7)-C(8)-C(9)	3.2(3)
C(1)-N(1)-C(9)-O(3)	143.6(2)
O(2)-N(1)-C(9)-O(3)	-62.88(19)
C(1)-N(1)-C(9)-C(10)	-96.6(2)
O(2)-N(1)-C(9)-C(10)	56.9(2)
C(1)-N(1)-C(9)-C(8)	22.2(3)
O(2)-N(1)-C(9)-C(8)	175.65(16)
O(4)-C(8)-C(9)-O(3)	49.4(2)
C(7)-C(8)-C(9)-O(3)	-132.66(19)
O(4)-C(8)-C(9)-N(1)	169.95(17)
C(7)-C(8)-C(9)-N(1)	-12.1(2)
O(4)-C(8)-C(9)-C(10)	-69.6(2)
C(7)-C(8)-C(9)-C(10)	108.3(2)
O(3)-C(9)-C(10)-C(15)	161.63(19)
N(1)-C(9)-C(10)-C(15)	41.9(2)
C(8)-C(9)-C(10)-C(15)	-78.5(2)
O(3)-C(9)-C(10)-C(11)	-22.3(3)
N(1)-C(9)-C(10)-C(11)	-142.05(19)
C(8)-C(9)-C(10)-C(11)	97.6(2)
C(15)-C(10)-C(11)-C(12)	-0.6(3)
C(9)-C(10)-C(11)-C(12)	-176.7(2)
C(10)-C(11)-C(12)-C(13)	0.5(4)
C(11)-C(12)-C(13)-C(14)	-0.2(4)

C(12)-C(13)-C(14)-C(15)	-0.1(4)
C(11)-C(10)-C(15)-C(14)	0.3(3)
C(9)-C(10)-C(15)-C(14)	176.4(2)
C(13)-C(14)-C(15)-C(10)	0.1(4)

Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for shelxl [A and deg.].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O(3)-H(3)...O(1)#1	0.82	1.96	2.773(2)	169.3

Symmetry transformations used to generate equivalent atoms: #1 x-1, y, z