Supporting Online Material for

Tandem Synthesis of 3-Chloro-4-iodoisoxazoles from 1-Copper(I) Alkynes, Dichloroformaldoxime and Molecular Iodine

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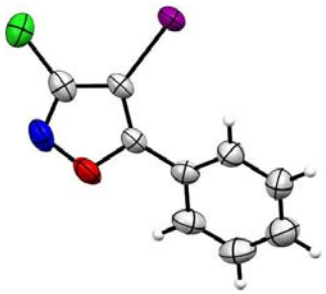
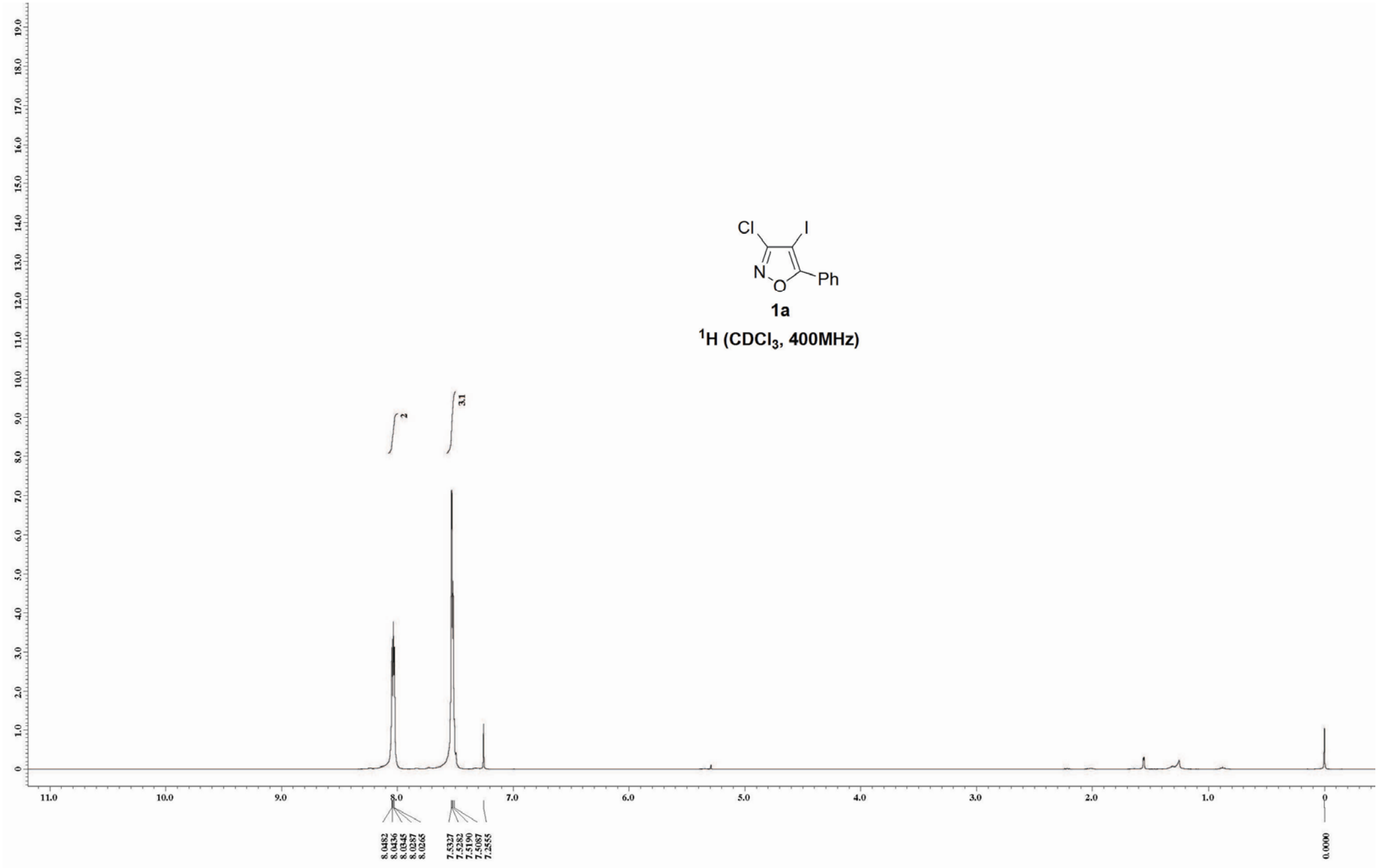
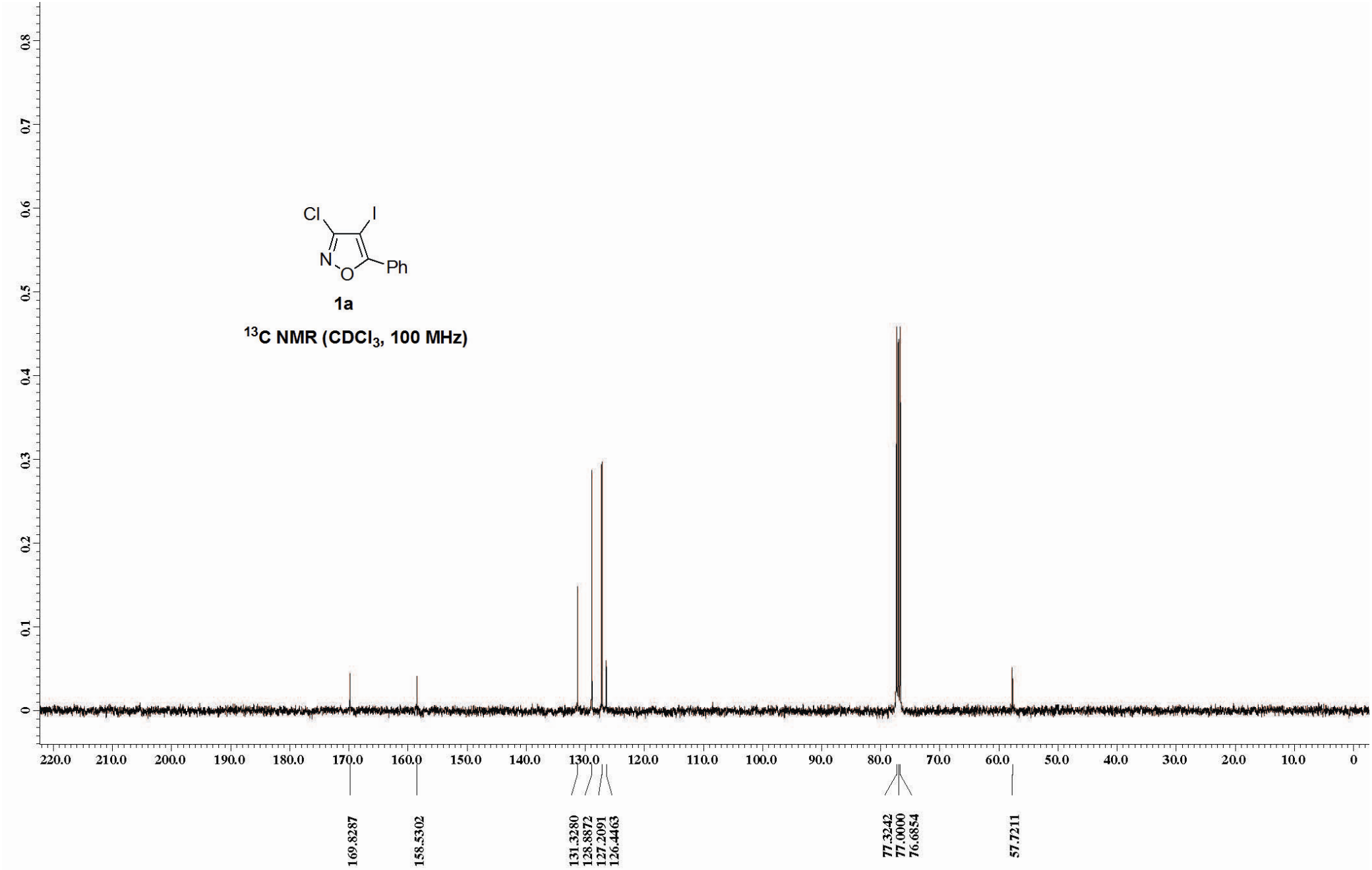
The structure of **1a** was confirmed by its single crystal X-ray diffraction analysis.

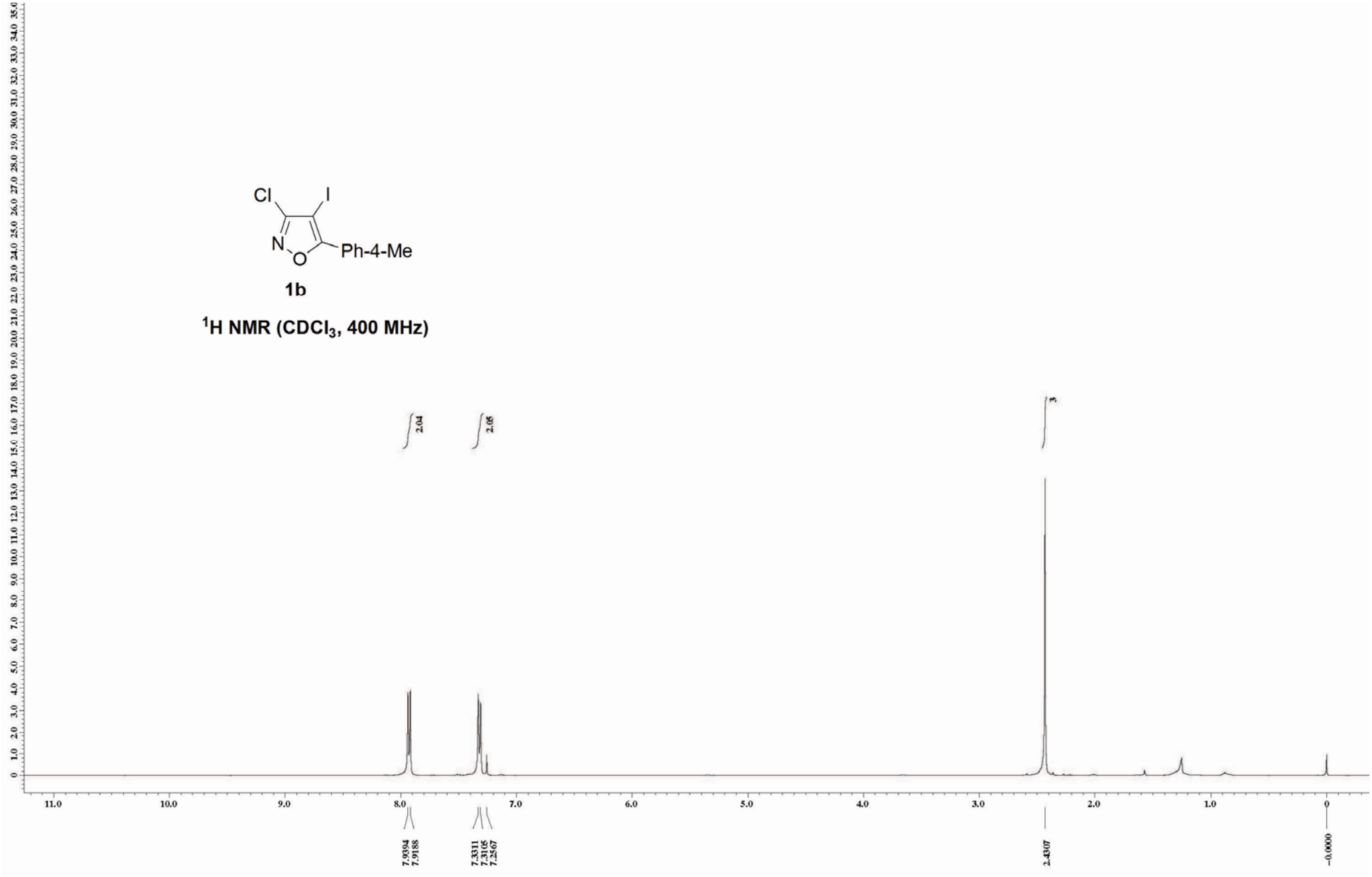
The key metrics for the single crystal X-ray structure can be found in CIF file.

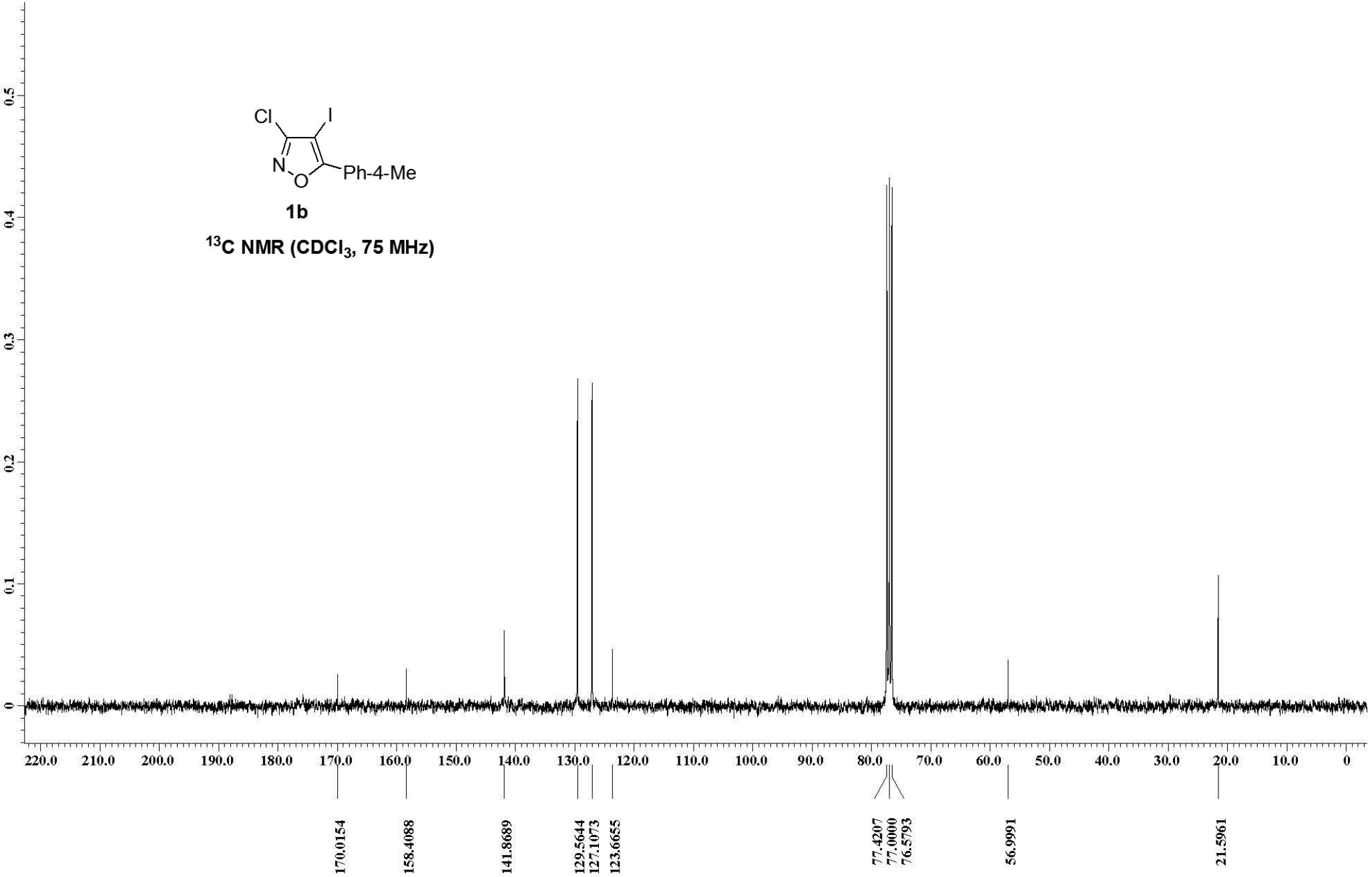


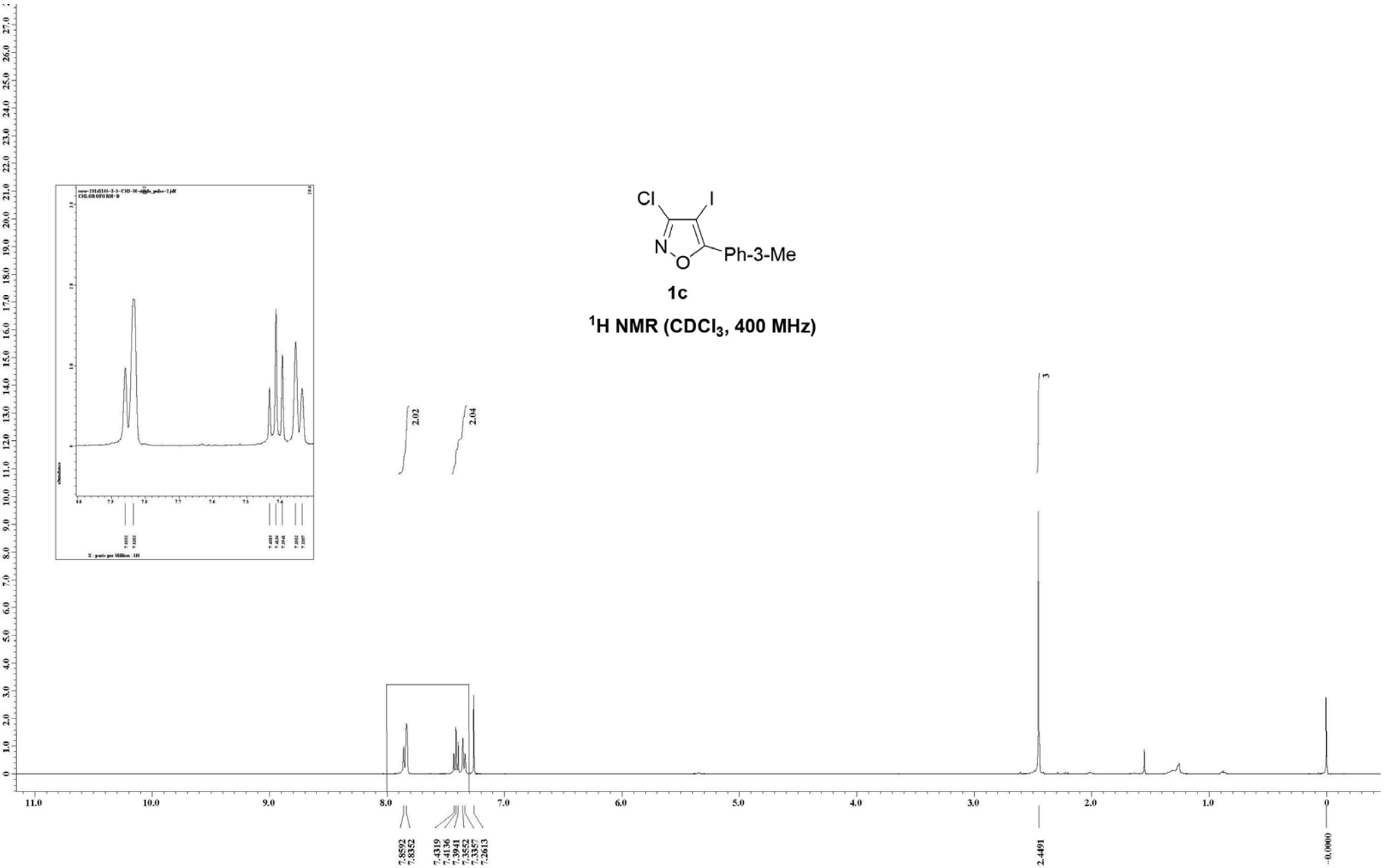
The structure of **1a**.

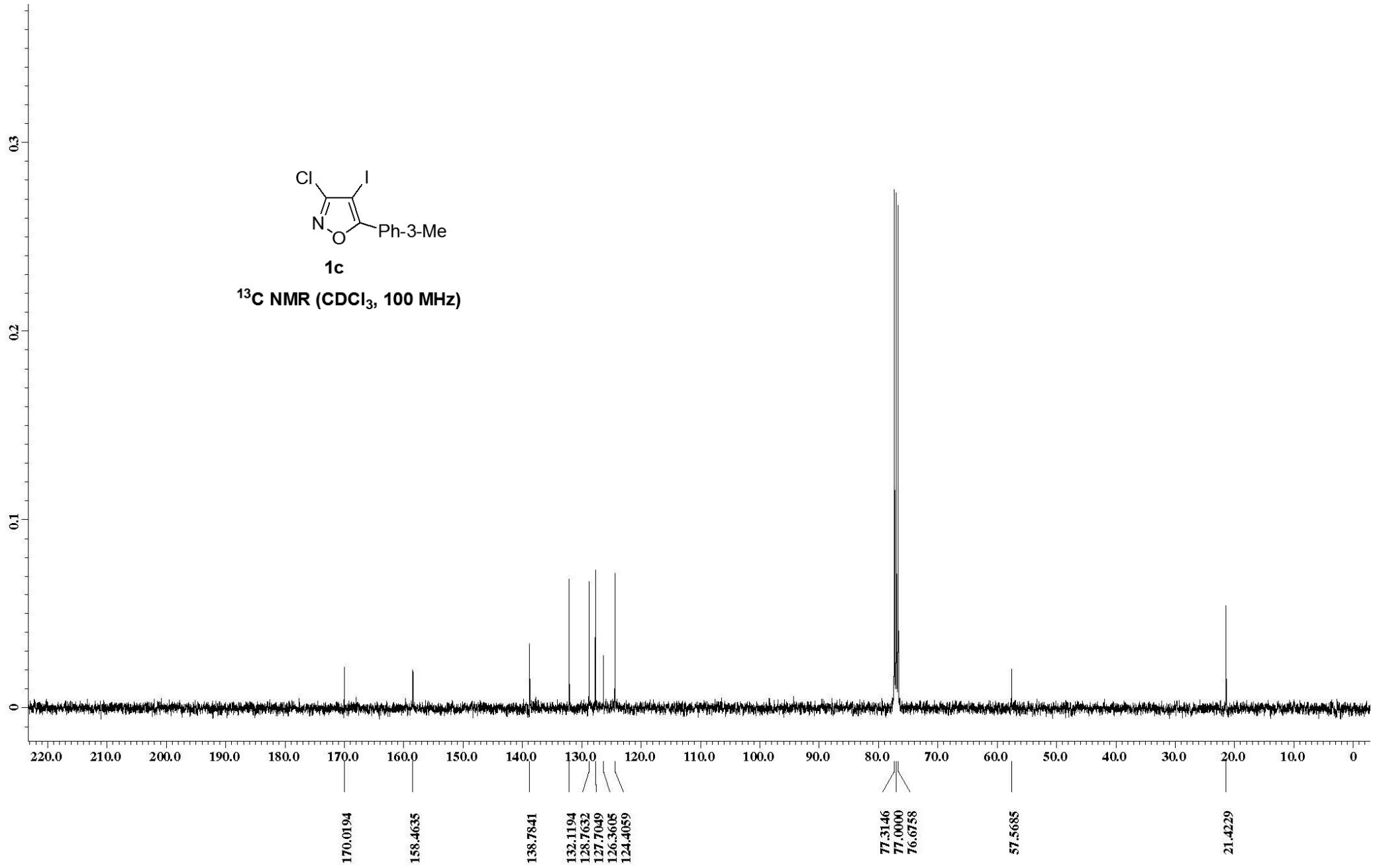

Cl
I
N
O
Ph
1a
^{1}H (CDCl$_3$, 400MHz)
2
3.1
8.0482
8.0436
8.0345
8.0287
8.0265
7.5327
7.5282
7.5190
7.5087
7.2555
0.0000

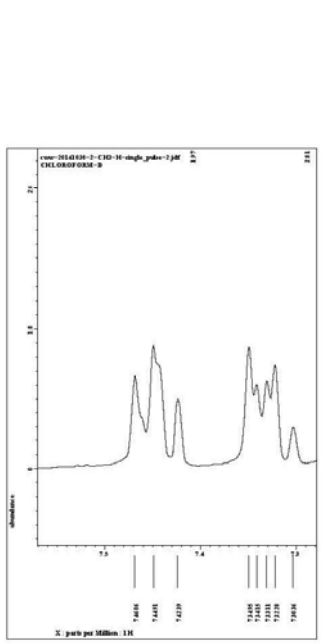

Cl
I
N
O
Ph
1a
^{13}C NMR ($CDCl_3$, 100 MHz)
169.8287
158.5302
131.3280
128.8872
127.2091
126.4463
77.3242
77.0000
76.6854
57.7211

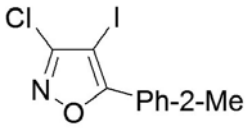

Cl
I
N
O
Ph-4-Me
1b
^{1}H NMR ($CDCl_3$, 400 MHz)
2.04
2.05
3
7.9394
7.9188
7.3311
7.3105
7.2567
2.4307
0.0000


Cl
I
N
O
Ph-4-Me
1b
13C NMR (CDCl3, 75 MHz)
0.5
0.4
0.3
0.2
0.1
0
220.0
210.0
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
170.0154
158.4088
141.8689
129.5644
127.1073
123.6655
77.4207
77.0000
76.5793
56.9991
21.5961


Cl
I
N
O
Ph-3-Me
1c
^{1}H NMR ($CDCl_3$, 400 MHz)
2.02
2.04
3
7.8592
7.8352
7.4319
7.4136
7.3941
7.3552
7.3357
7.2613
2.4491
0.0000

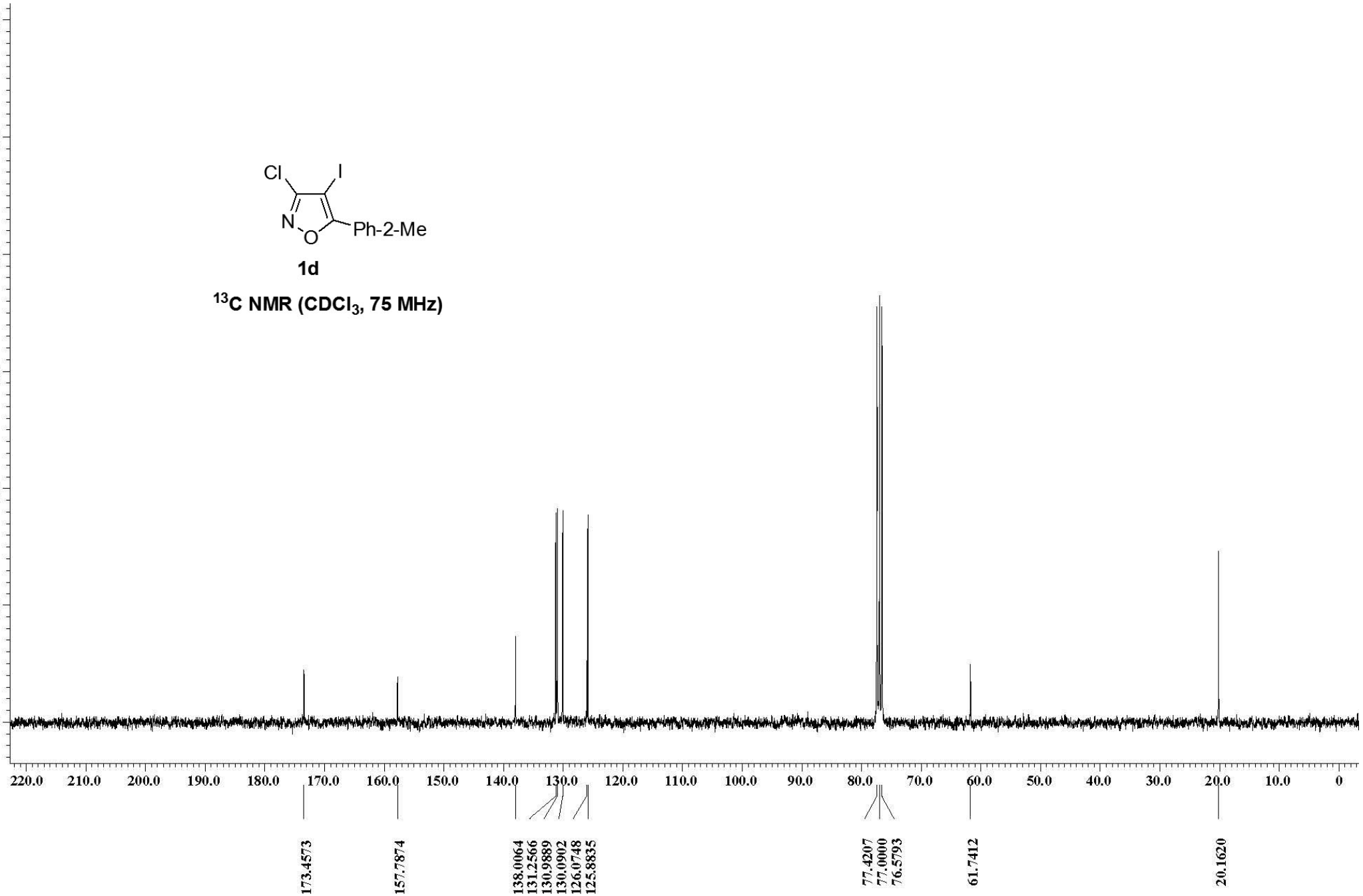

Cl
I
N
O
Ph-3-Me
1c
13C NMR (CDCl3, 100 MHz)
0.3
0.2
0.1
0
220.0
210.0
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
170.0194
158.4635
138.7841
132.1194
128.7632
127.7049
126.3605
124.4059
77.3146
77.0000
76.6758
57.5685
21.4229

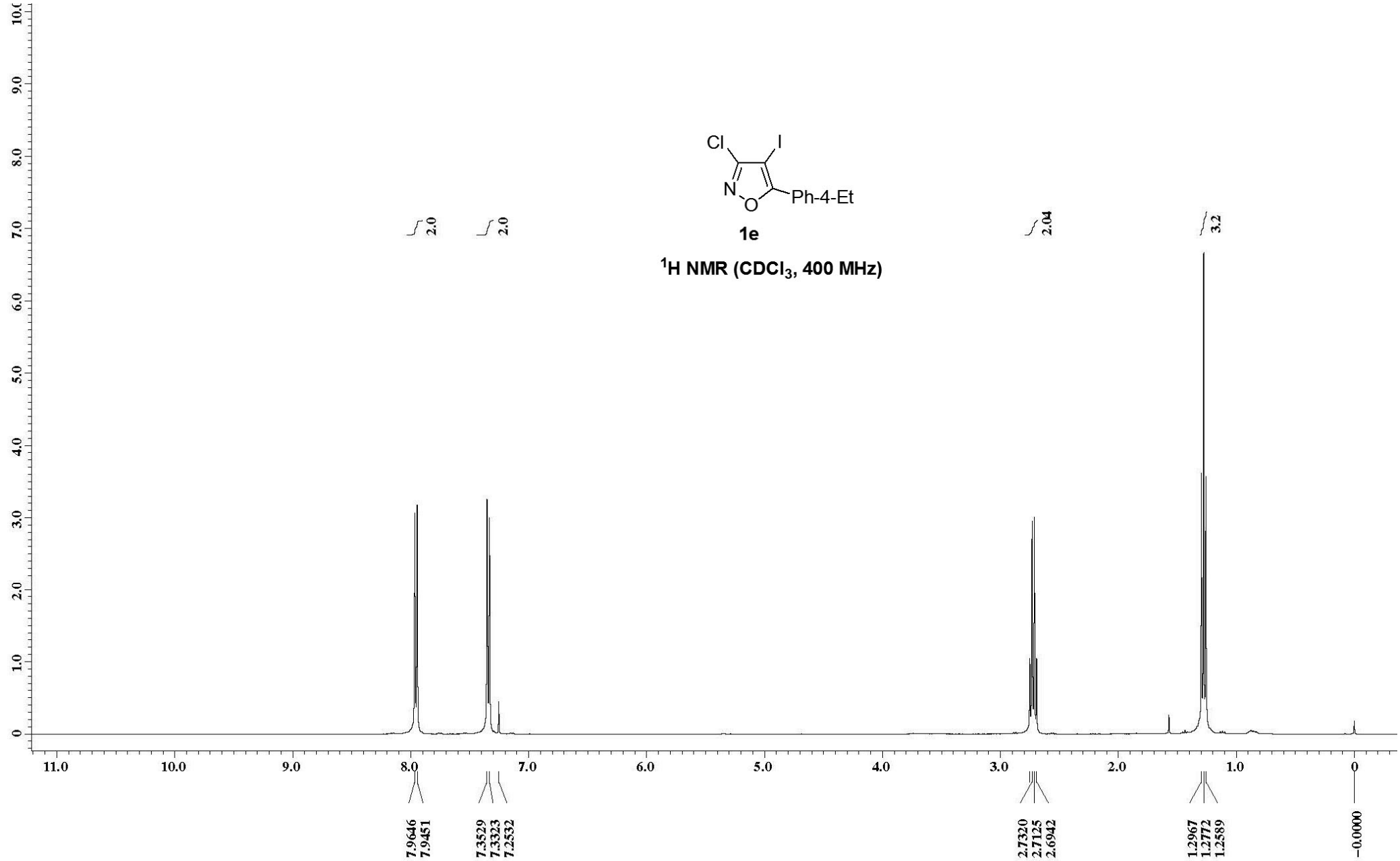


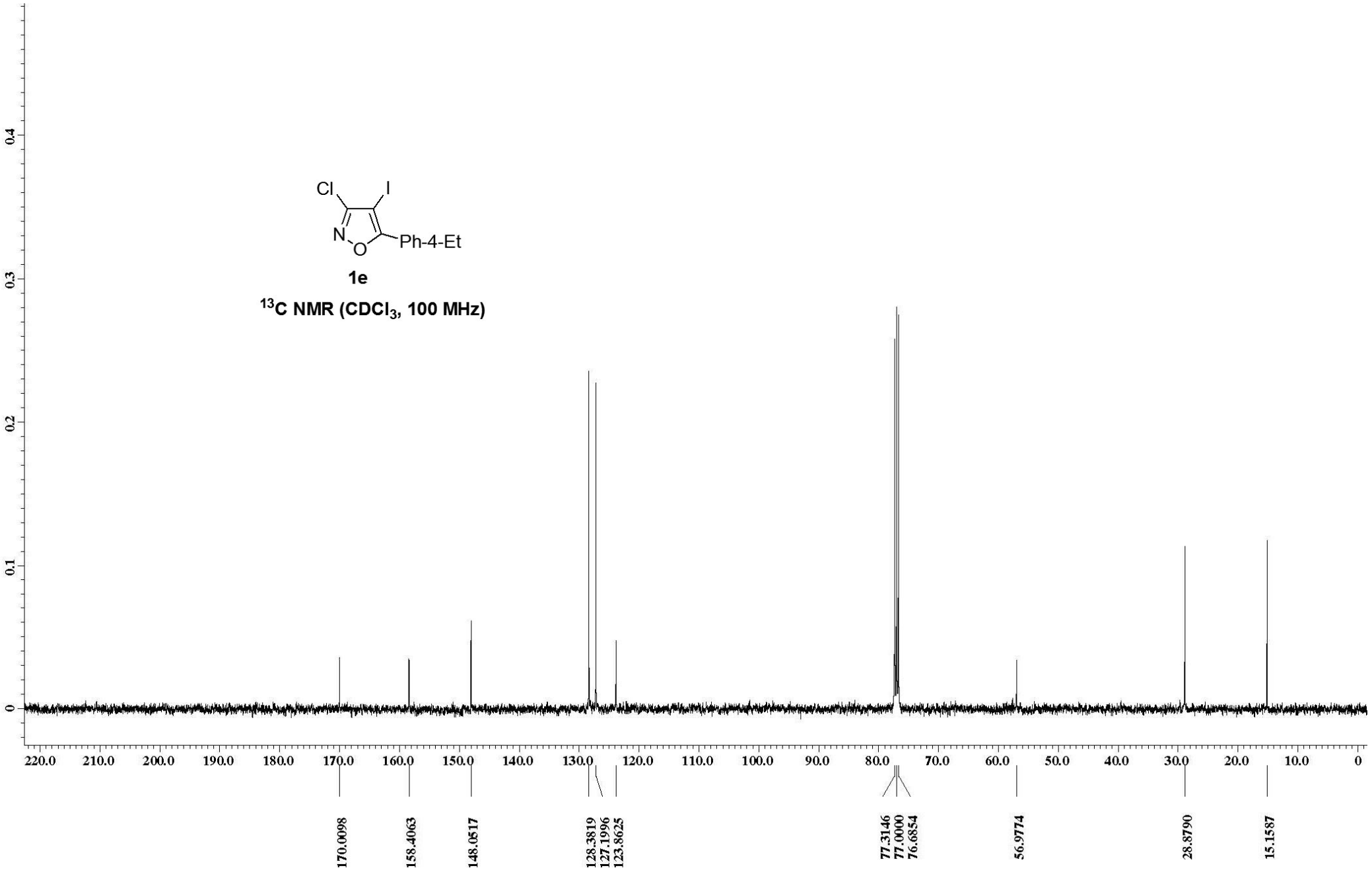


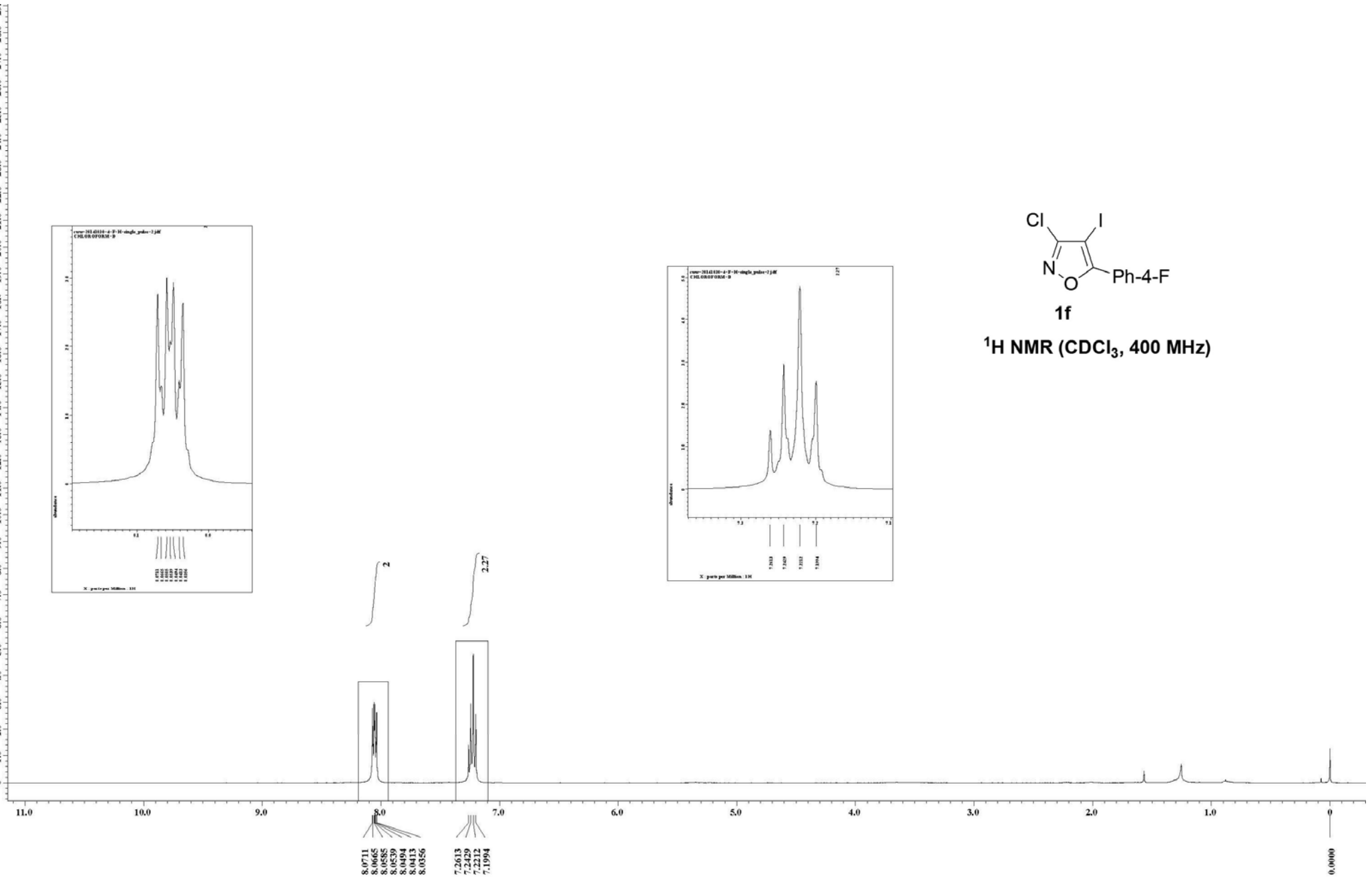
1d

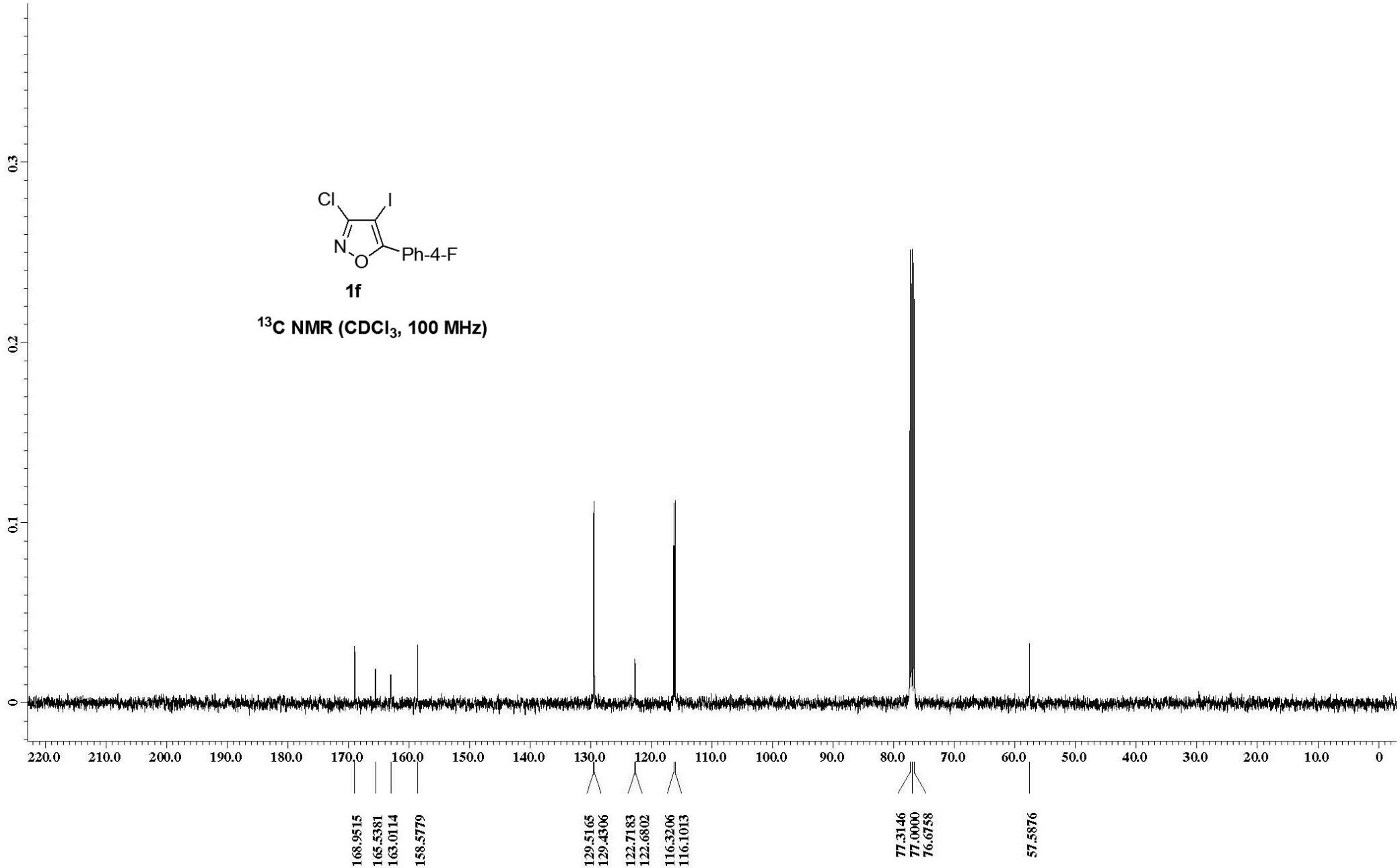
^{1}H NMR ($CDCl_3$, 400 MHz)

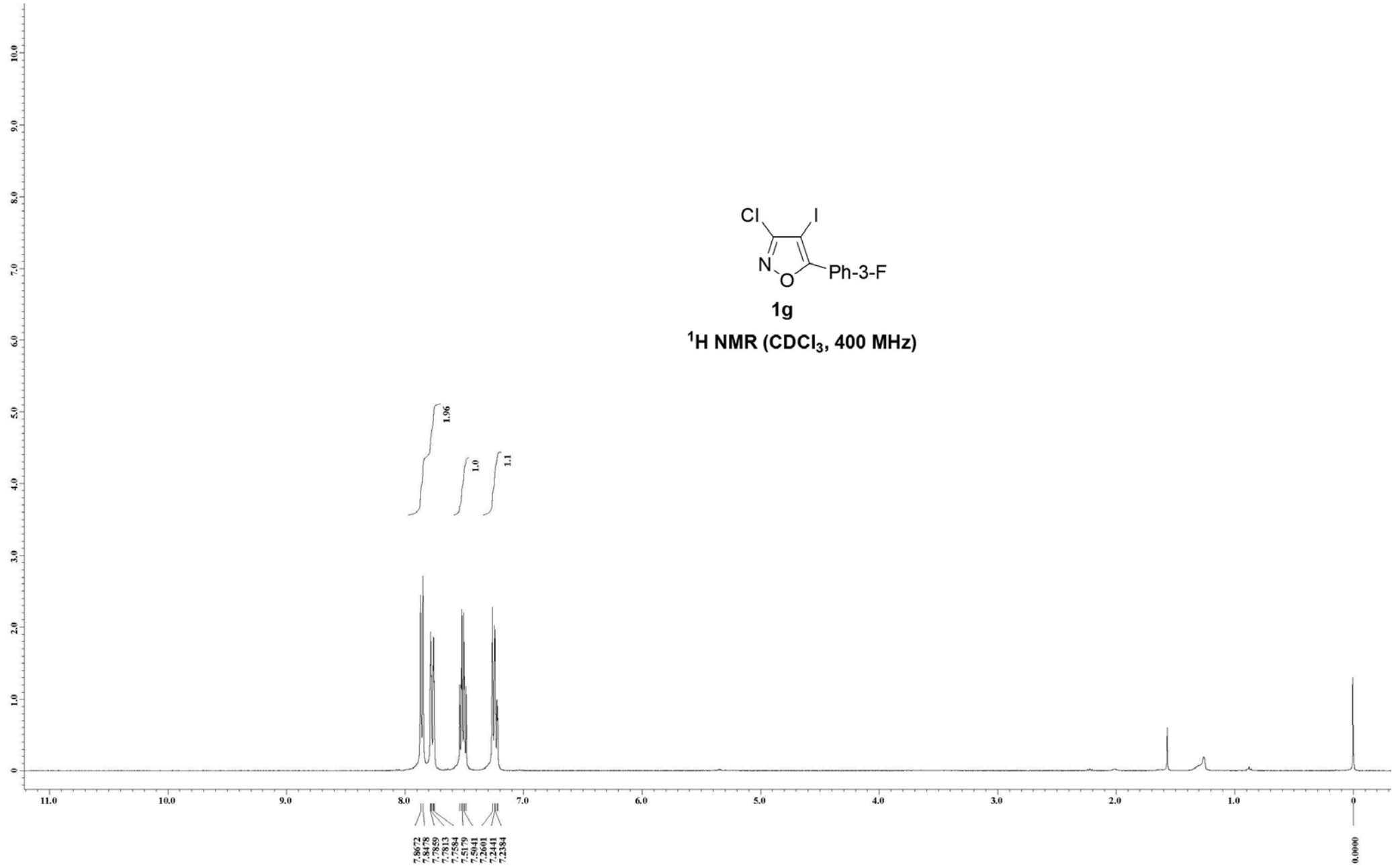

Cl
I
N
O
Ph-2-Me
1d
13C NMR (CDCl3, 75 MHz)
0.6
0.5
0.4
0.3
0.2
0.1
0
220.0
210.0
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
173.4573
157.7874
138.0064
131.2566
130.9889
130.0902
126.0748
125.8835
77.4207
77.0000
76.5793
61.7412
20.1620

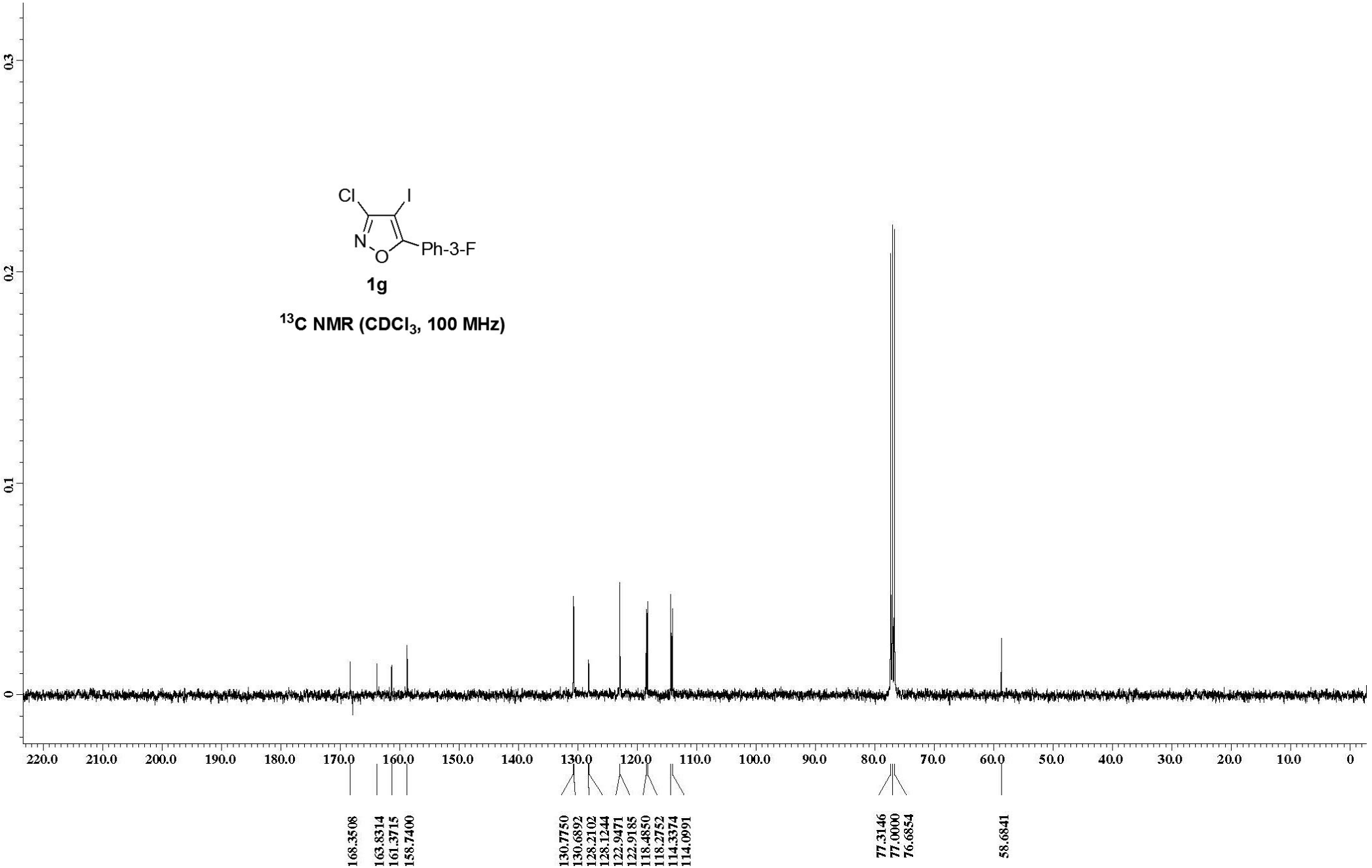

Cl
I
N
O
Ph-4-Et
1e
^{1}H NMR (CDCl$_3$, 400 MHz)
2.0
2.0
2.04
3.2
7.9646
7.9451
7.3529
7.3323
7.2532
2.7320
2.7125
2.6942
1.2967
1.2772
1.2589
−0.0000

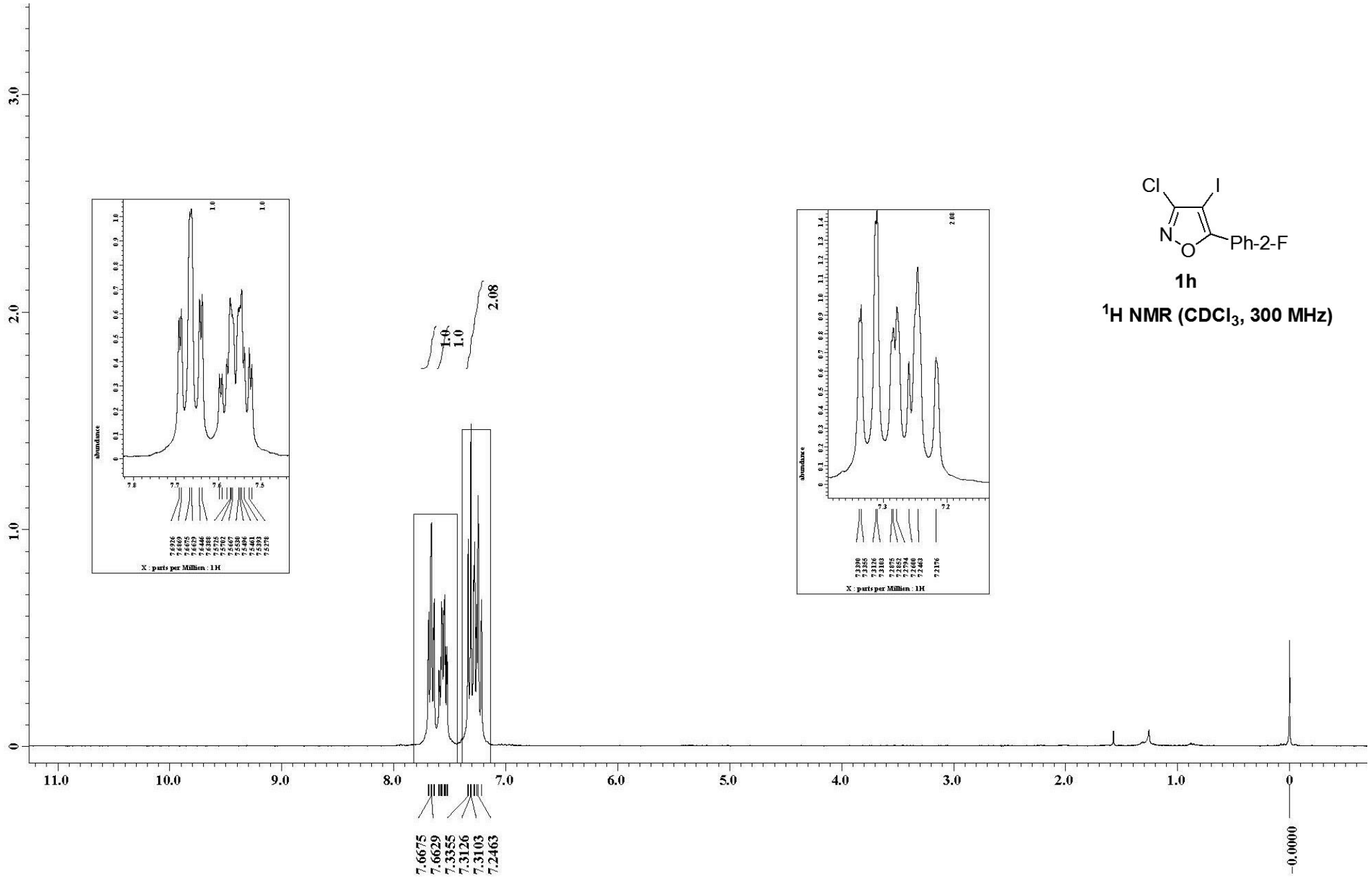

Cl
I
N
O
Ph-4-Et
1e
13C NMR (CDCl3, 100 MHz)
0.4
0.3
0.2
0.1
0
220.0
210.0
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
170.0098
158.4063
148.0517
128.3819
127.1996
123.8625
77.3146
77.0000
76.6854
56.9774
28.8790
15.1587

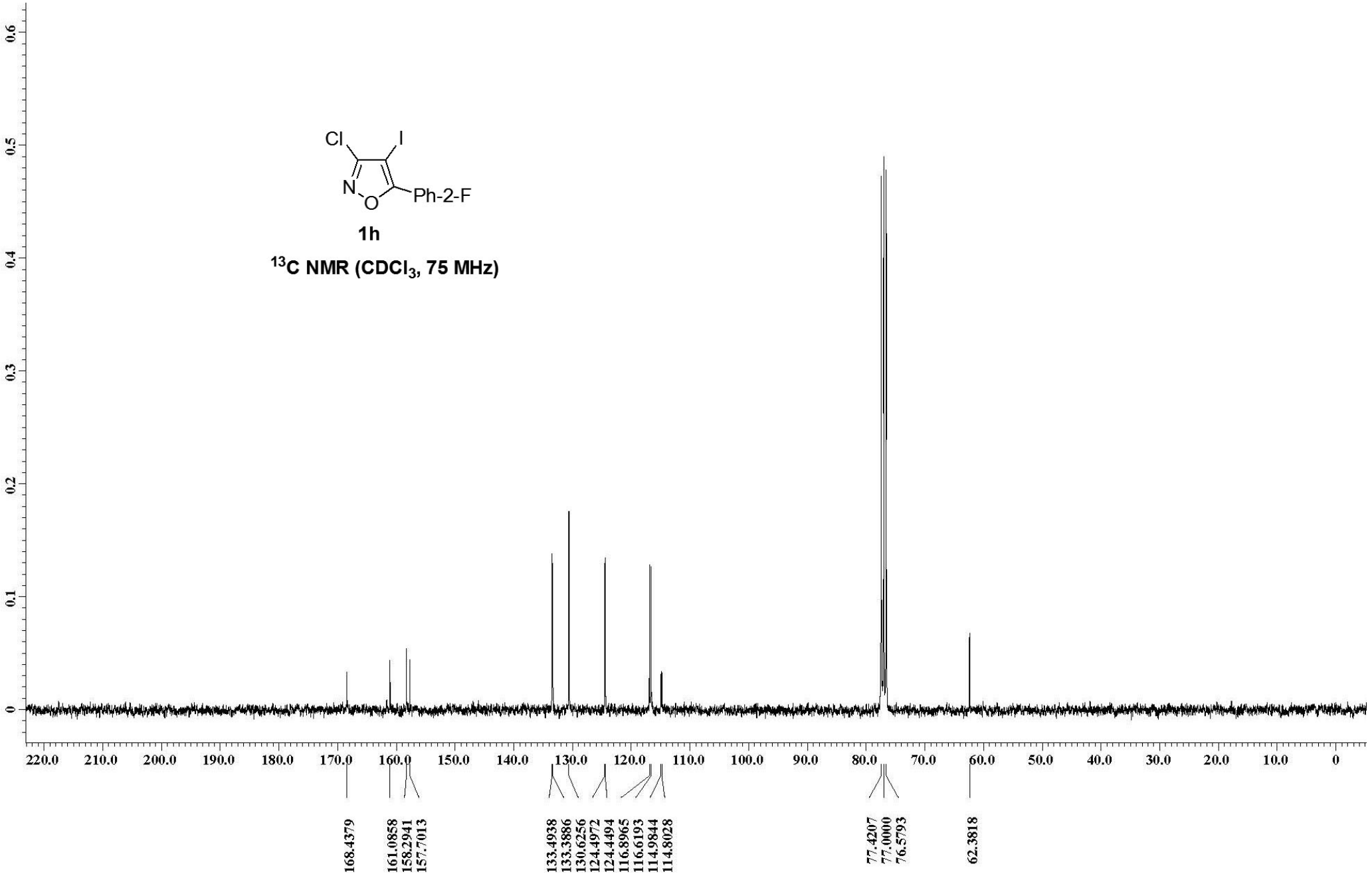

Cl
I
N
O
Ph-4-F
1f
^{1}H NMR ($CDCl_3$, 400 MHz)
2
2.27
8.0711
8.0665
8.0585
8.0539
8.0494
8.0413
8.0356
7.2613
7.2429
7.2212
7.1994
0.0000

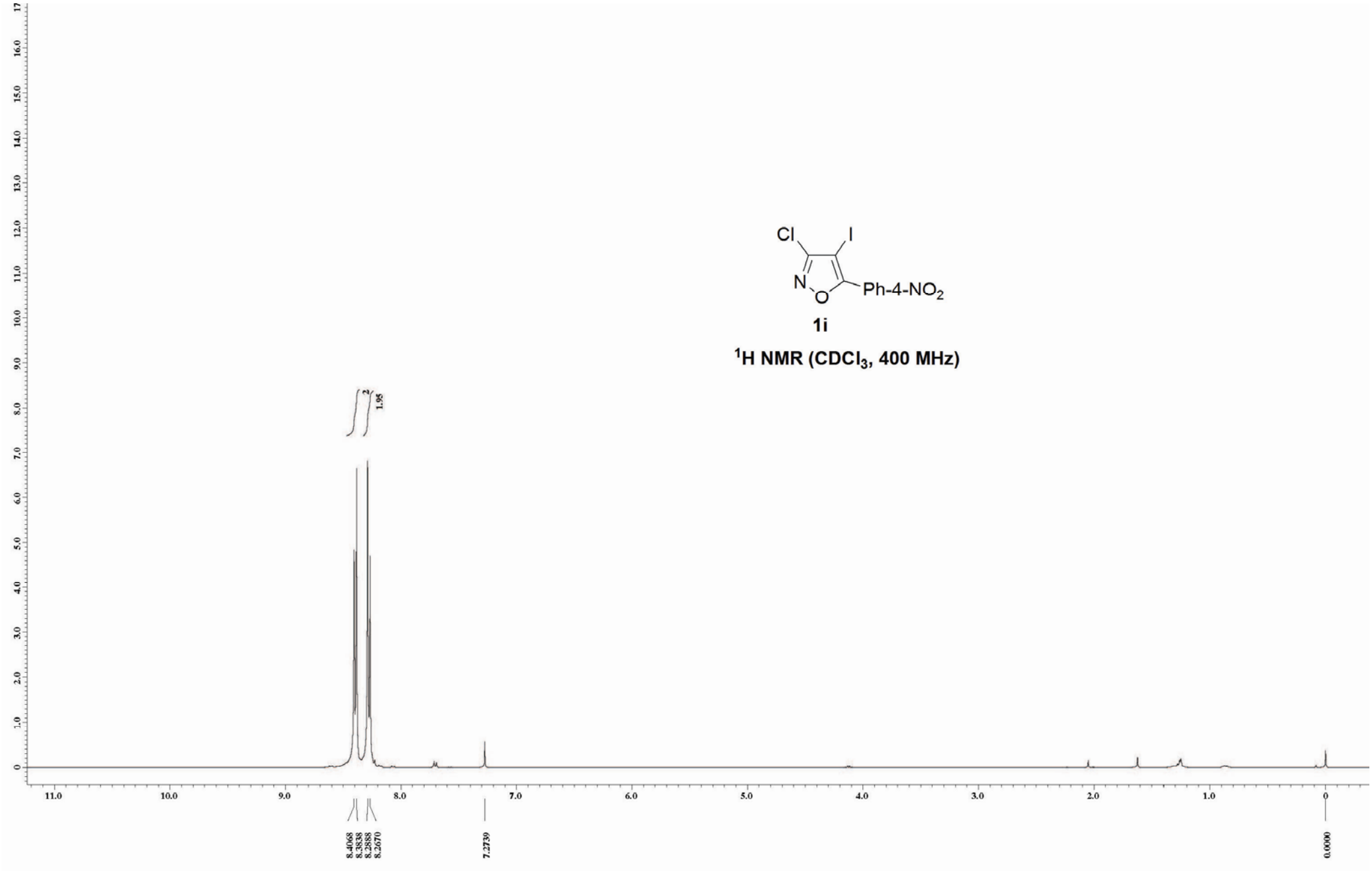

Cl
I
N
O
Ph-4-F
1f
^{13}C NMR ($CDCl_3$, 100 MHz)
0.3
0.2
0.1
0
220.0
210.0
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
168.9515
165.5381
163.0114
158.5779
129.5165
129.4306
122.7183
122.6802
116.3206
116.1013
77.3146
77.0000
76.6758
57.5876

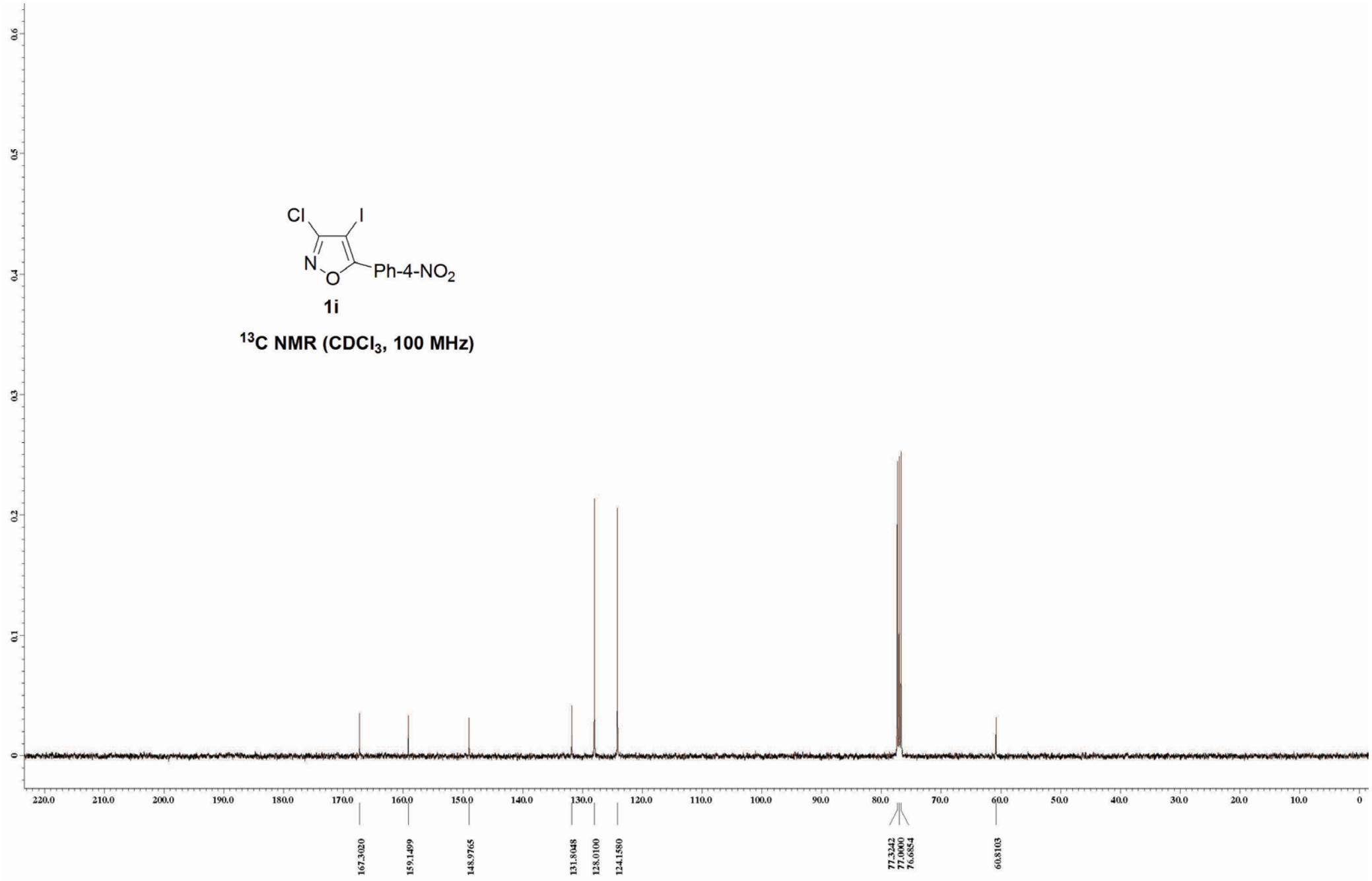

Cl
I
N
O
Ph-3-F
1g
^{1}H NMR ($CDCl_3$, 400 MHz)
1.96
1.0
1.1
7.8672
7.8478
7.7859
7.7813
7.7584
7.5179
7.5041
7.2601
7.2441
7.2384
0.0000

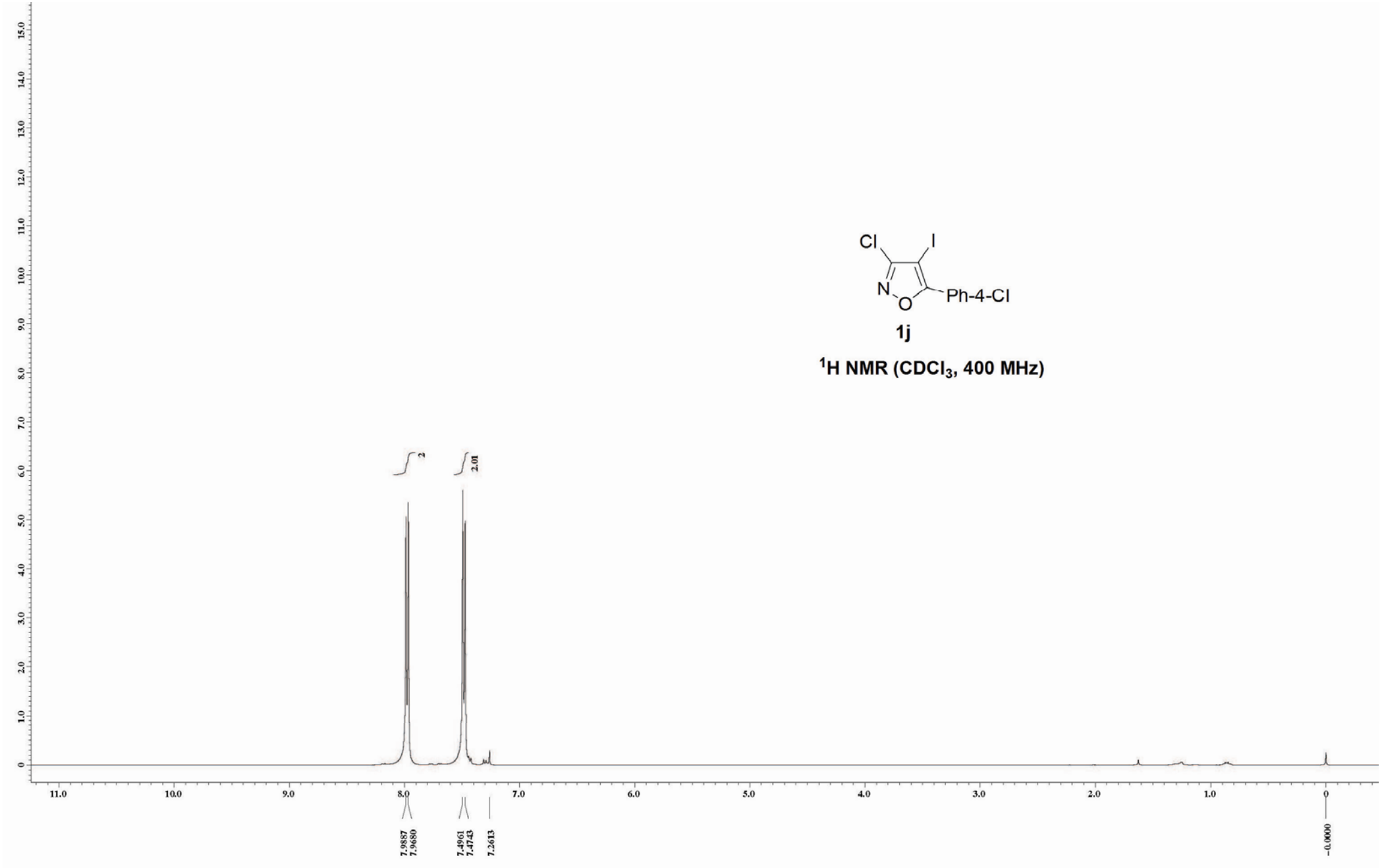

Cl
I
N
O
Ph-3-F
1g
^{13}C NMR (CDCl$_3$, 100 MHz)
0.3
0.2
0.1
0
220.0
210.0
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
168.3508
163.8314
161.3715
158.7400
130.7750
130.6892
128.2102
128.1244
122.9471
122.9185
118.4850
118.2752
114.3374
114.0991
77.3146
77.0000
76.6854
58.6841

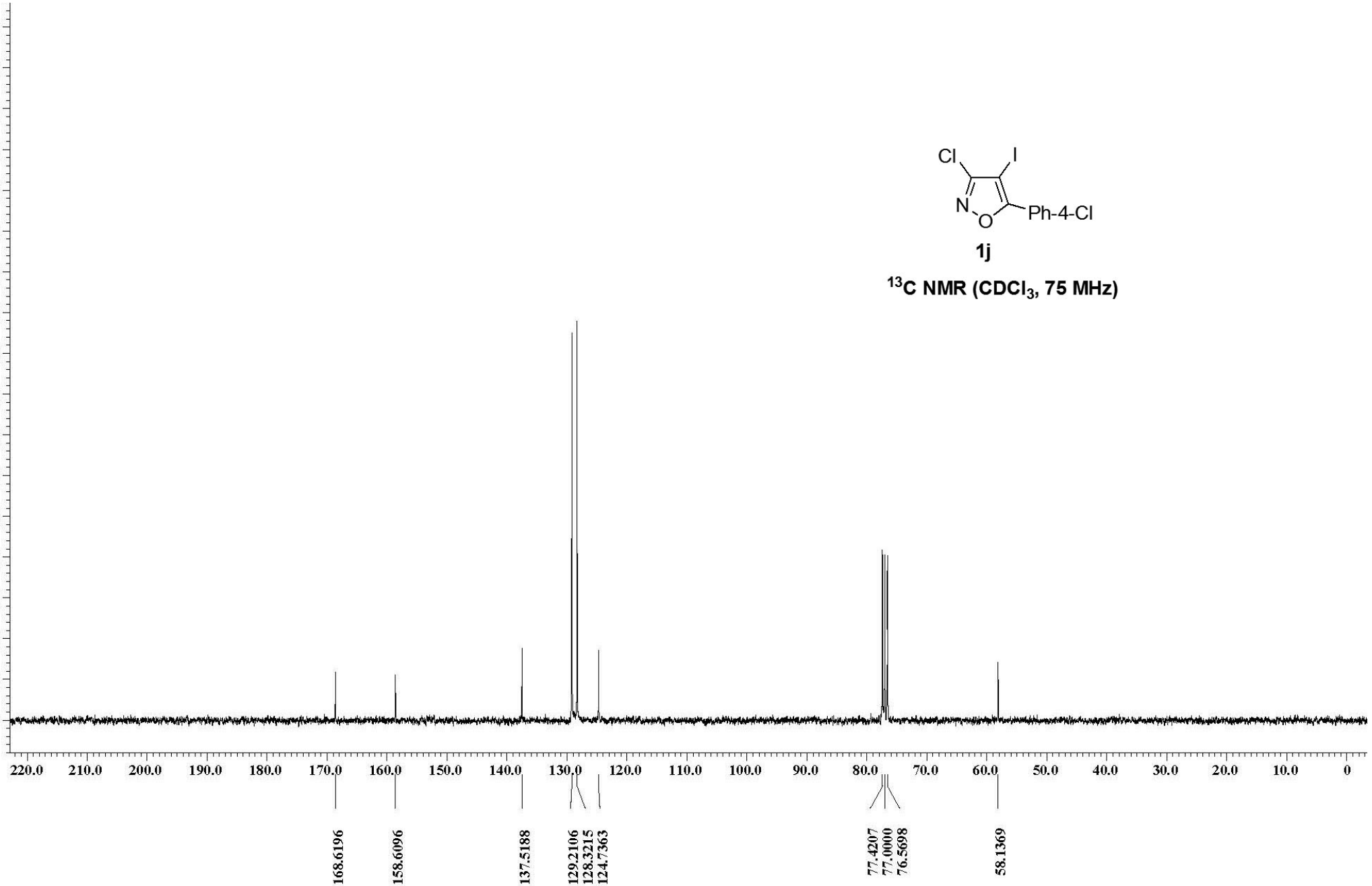

Cl
I
N
O
Ph-2-F
1h
^{1}H NMR ($CDCl_3$, 300 MHz)
1.0
1.0
2.08
7.6675
7.6629
7.3355
7.3126
7.3103
7.2463
-0.0000

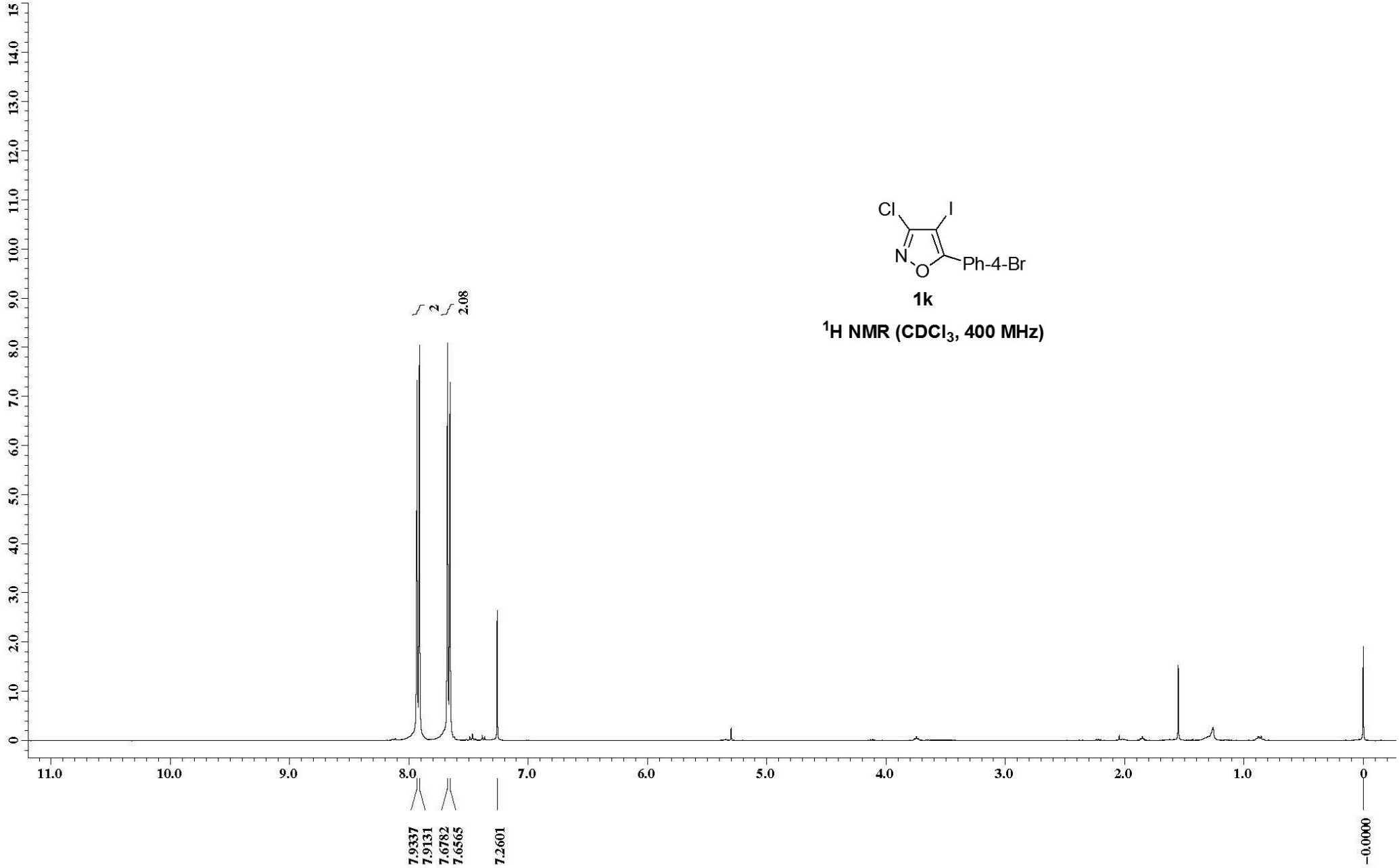

Cl
I
N
O
Ph-2-F
1h
13C NMR (CDCl3, 75 MHz)
168.4379
161.0858
158.2941
157.7013
133.4938
133.3886
130.6256
124.4972
124.4494
116.8965
116.6193
114.9844
114.8028
77.4207
77.0000
76.5793
62.3818

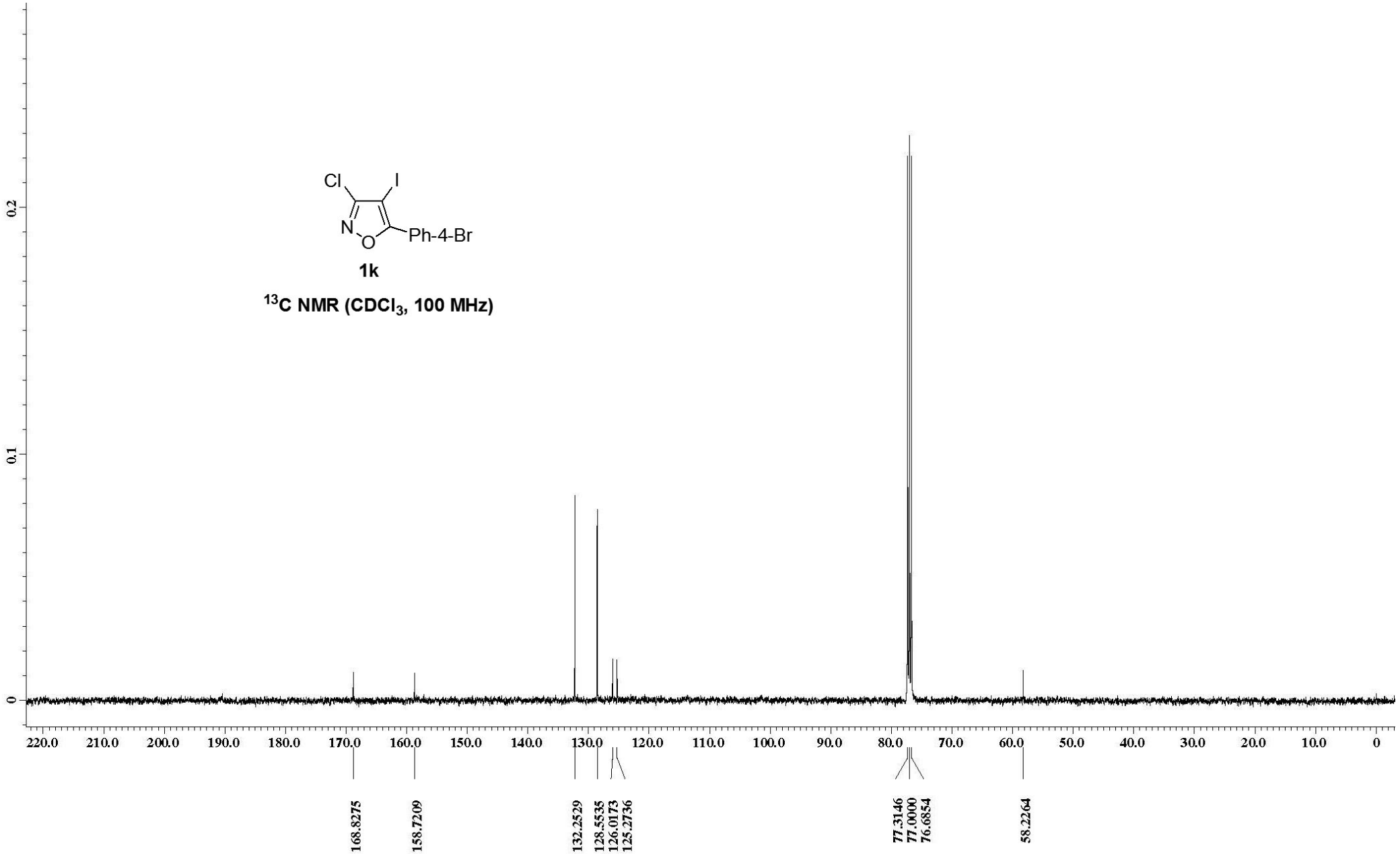

Cl
I
N
O
Ph-4-NO2
1i
1H NMR (CDCl3, 400 MHz)
2
1.95
8.4068
8.3838
8.2888
8.2670
7.2739
0.0000

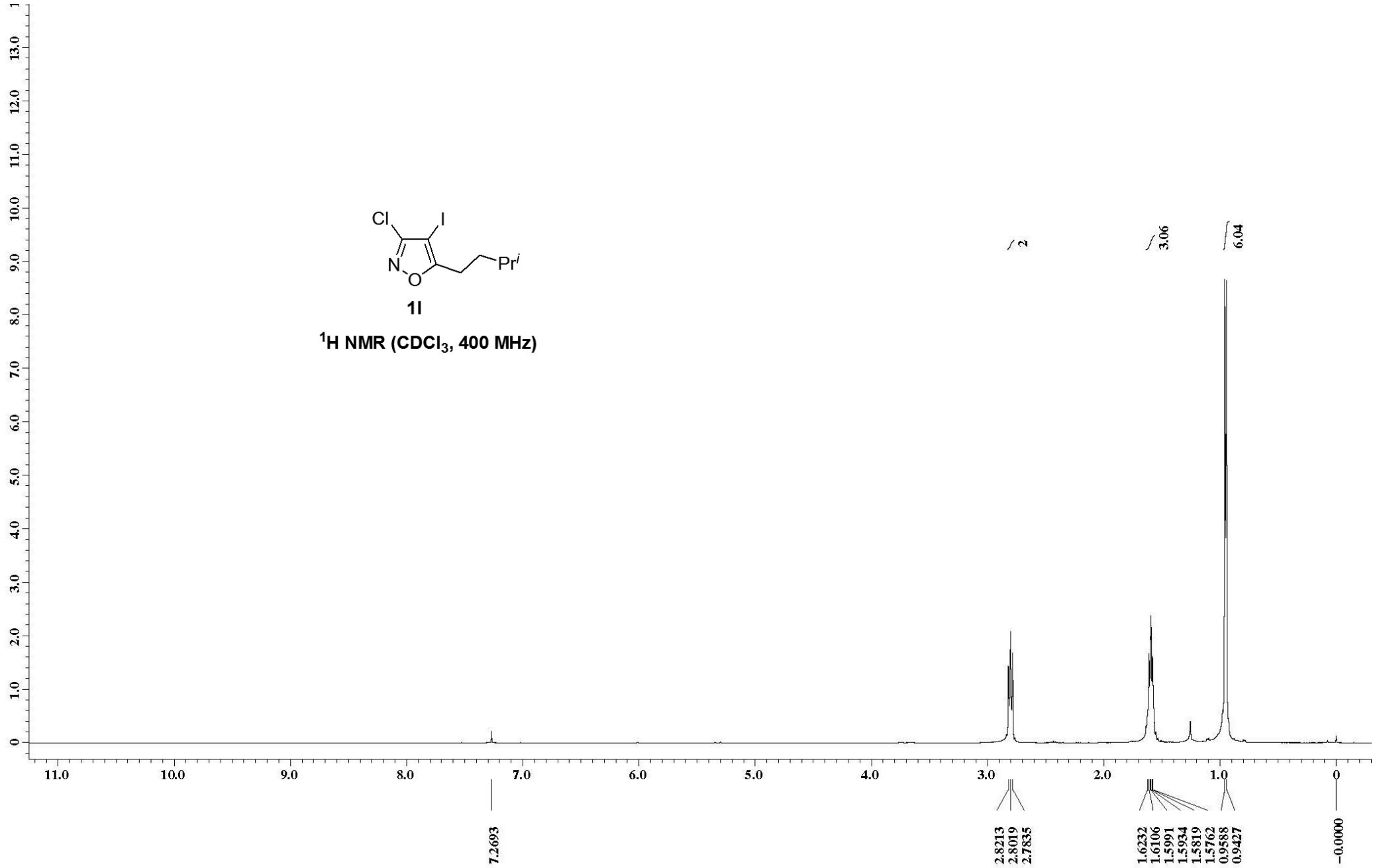

Cl
I
N
O
Ph-4-NO2
1i
13C NMR (CDCl3, 100 MHz)
167.3020
159.1499
148.9765
131.8048
128.0100
124.1580
77.3242
77.0000
76.6854
60.8103

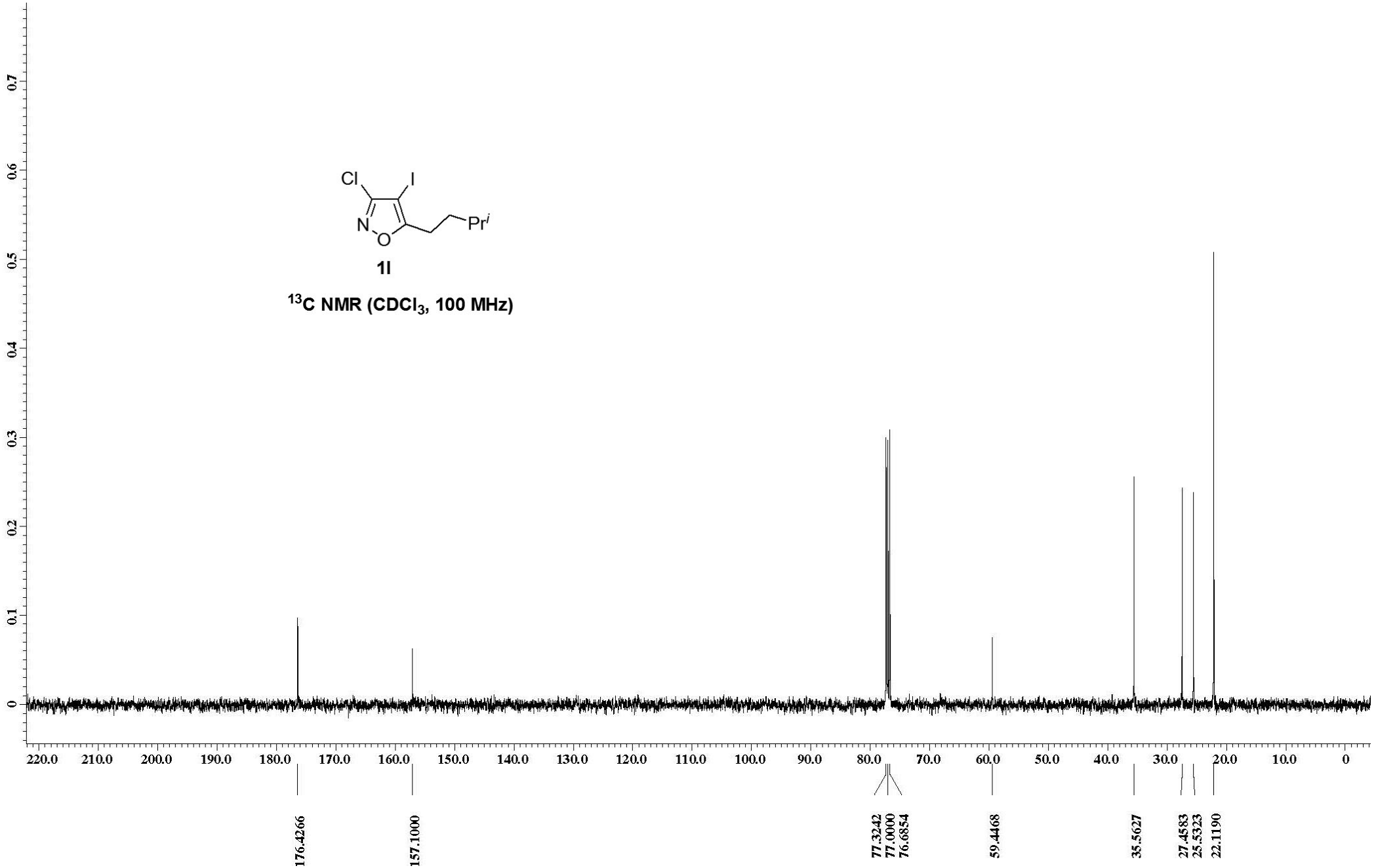

Cl
I
N
O
Ph-4-Cl
1j
^{1}H NMR ($CDCl_3$, 400 MHz)
2
2.01
7.9887
7.9680
7.4961
7.4743
7.2613
-0.0000

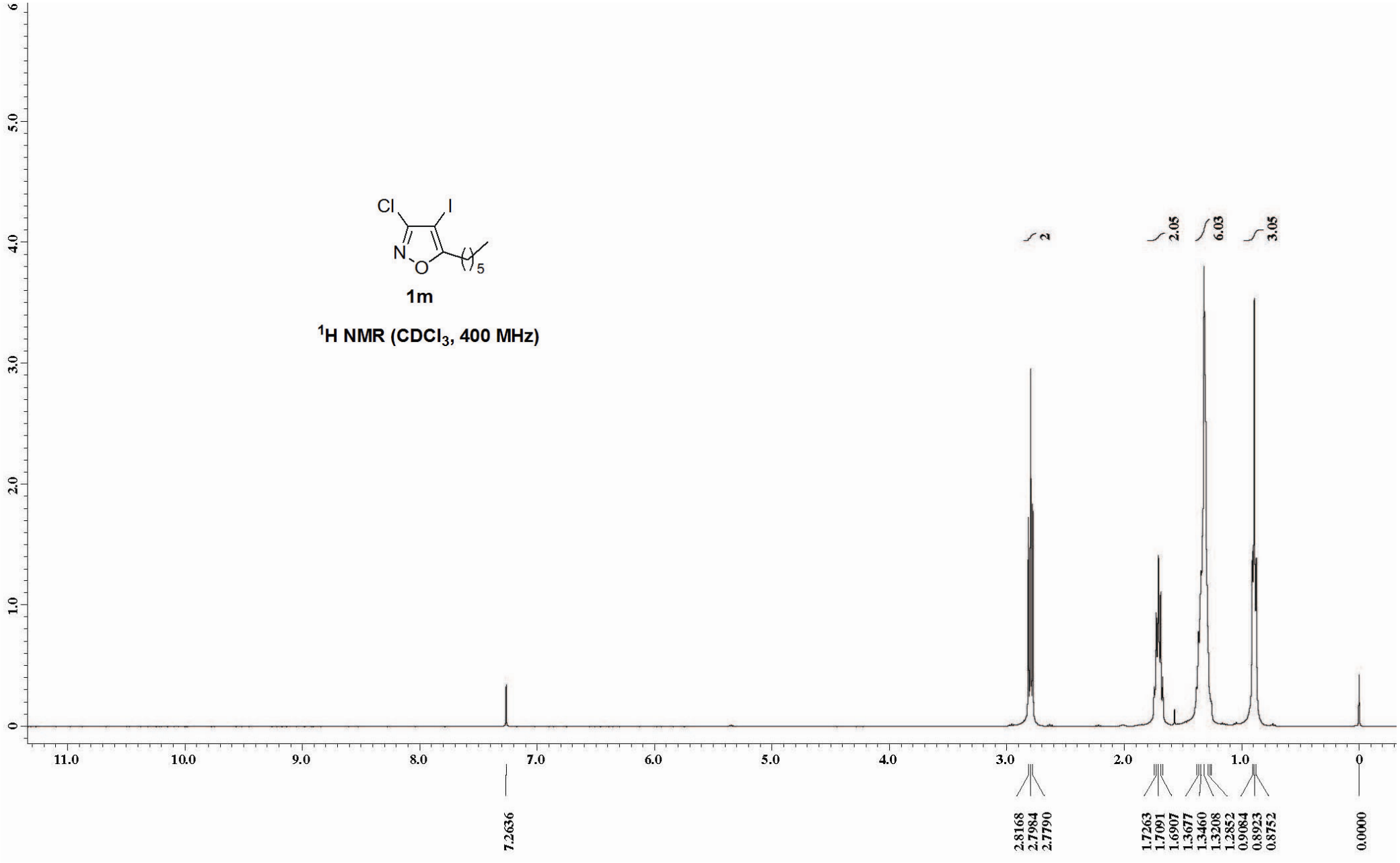

Cl
I
N
O
Ph-4-Cl
1j
13C NMR (CDCl3, 75 MHz)
168.6196
158.6096
137.5188
129.2106
128.3215
124.7363
77.4207
77.0000
76.5698
58.1369

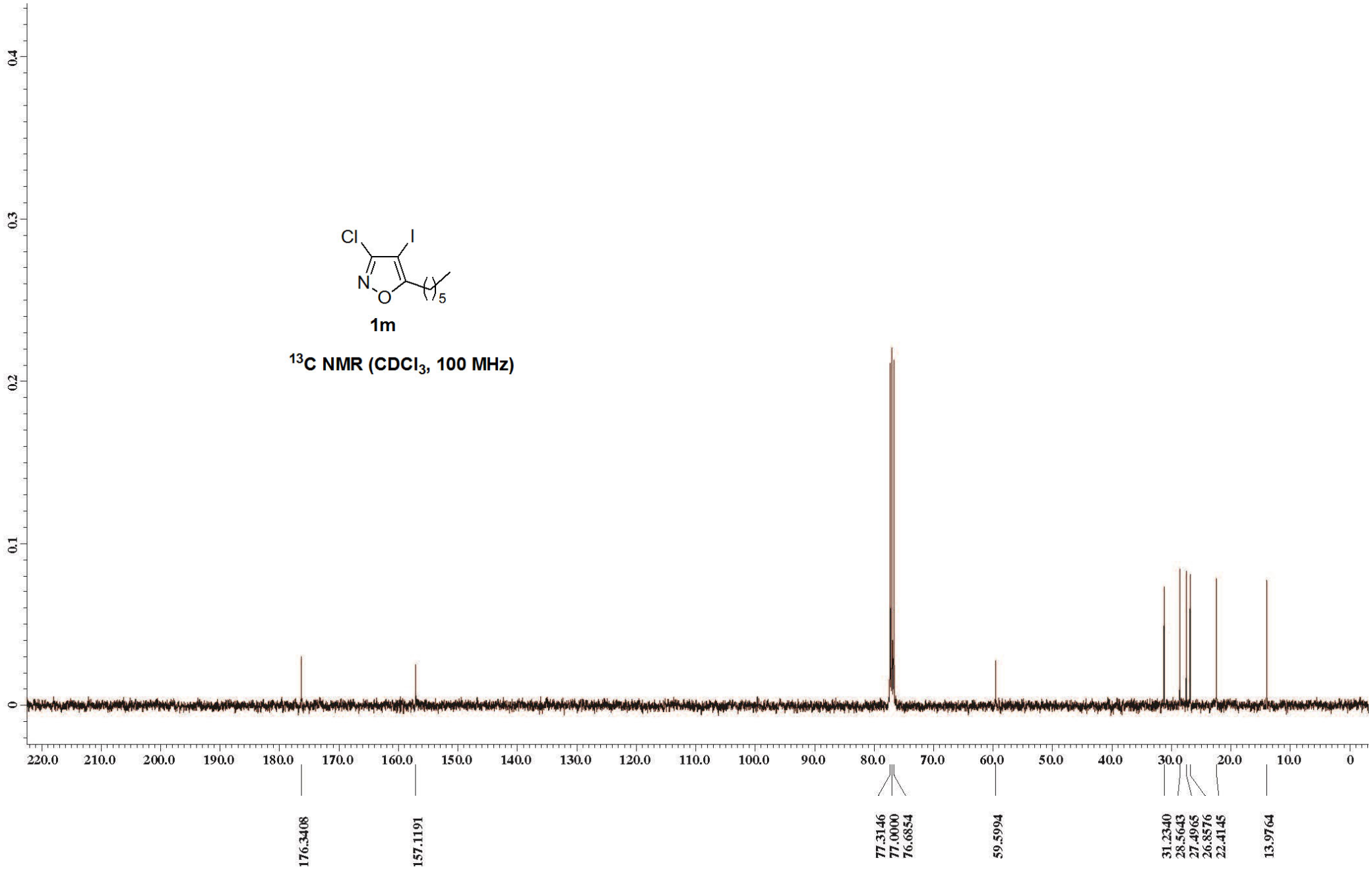

Cl
I
N
O
Ph-4-Br
1k
^{1}H NMR ($CDCl_3$, 400 MHz)
2
2.08
7.9337
7.9131
7.6782
7.6565
7.2601
-0.0000

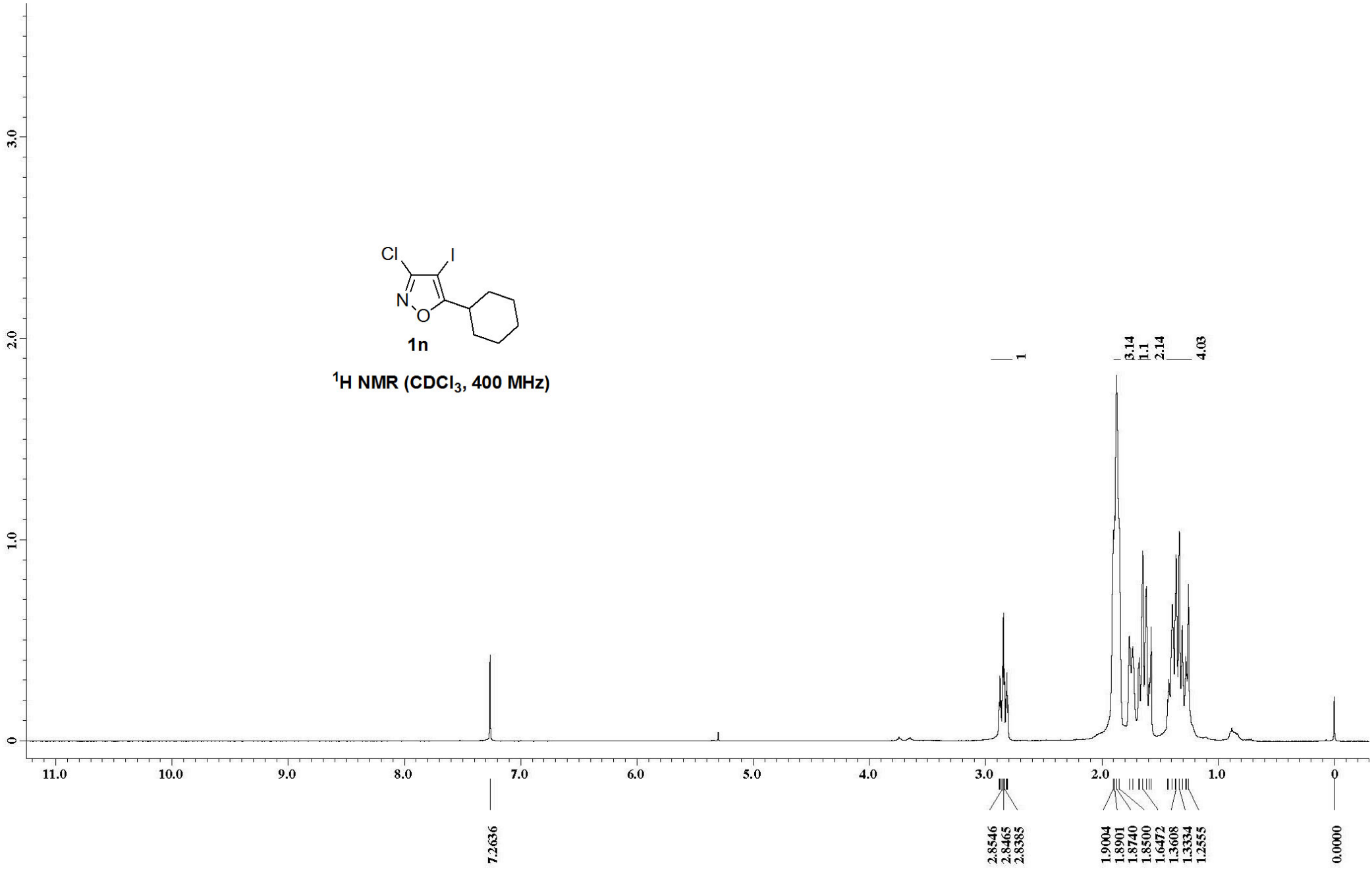

Cl
I
N
O
Ph-4-Br
1k
13C NMR (CDCl3, 100 MHz)
0.2
0.1
0
220.0
210.0
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
168.8275
158.7209
132.2529
128.5535
126.0173
125.2736
77.3146
77.0000
76.6854
58.2264

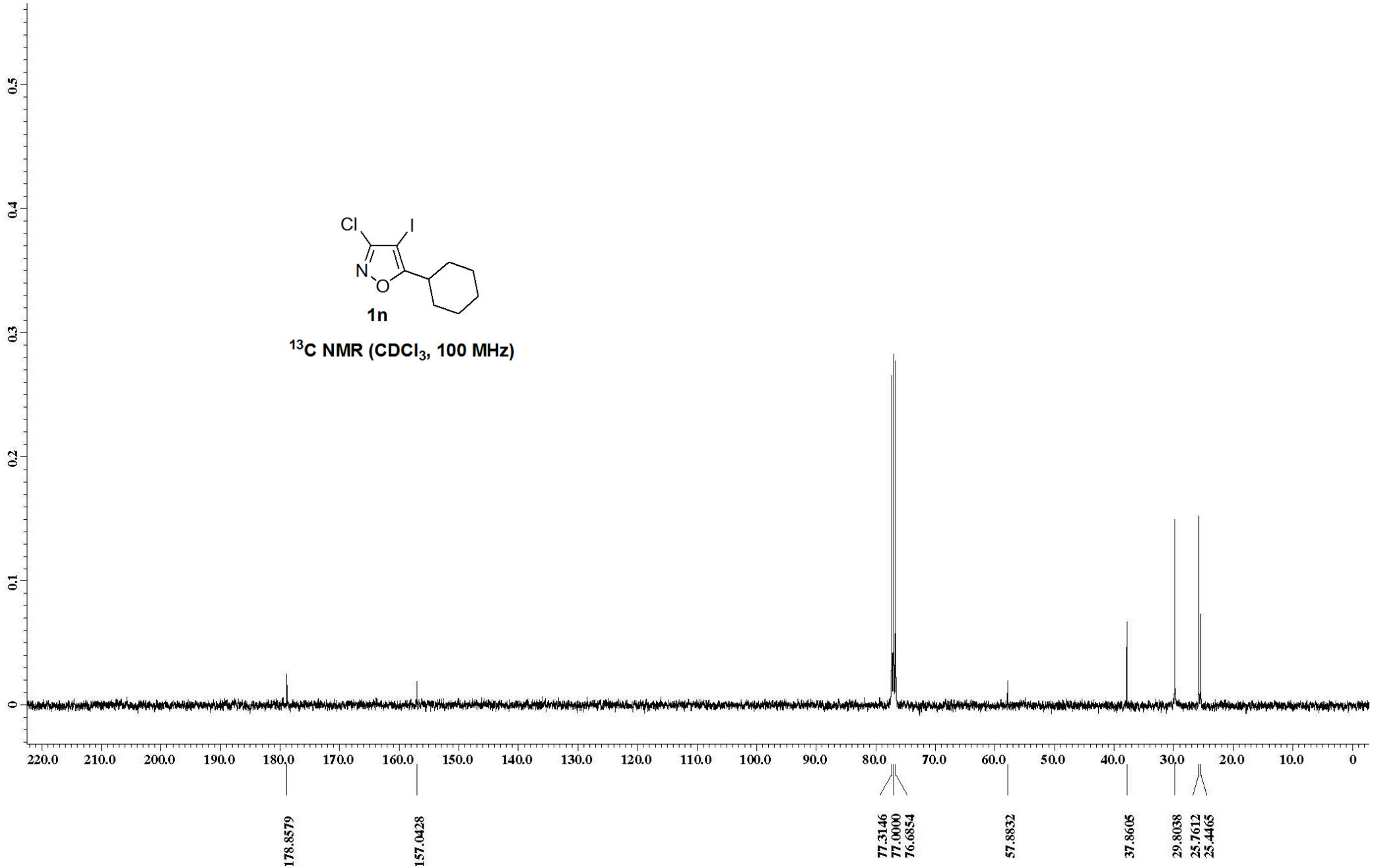

Cl
I
N
O
Pr^i
1l
^{1}H NMR (CDCl$_3$, 400 MHz)
2
3.06
6.04
7.2693
2.8213
2.8019
2.7835
1.6232
1.6106
1.5991
1.5934
1.5819
1.5762
0.9588
0.9427
−0.0000

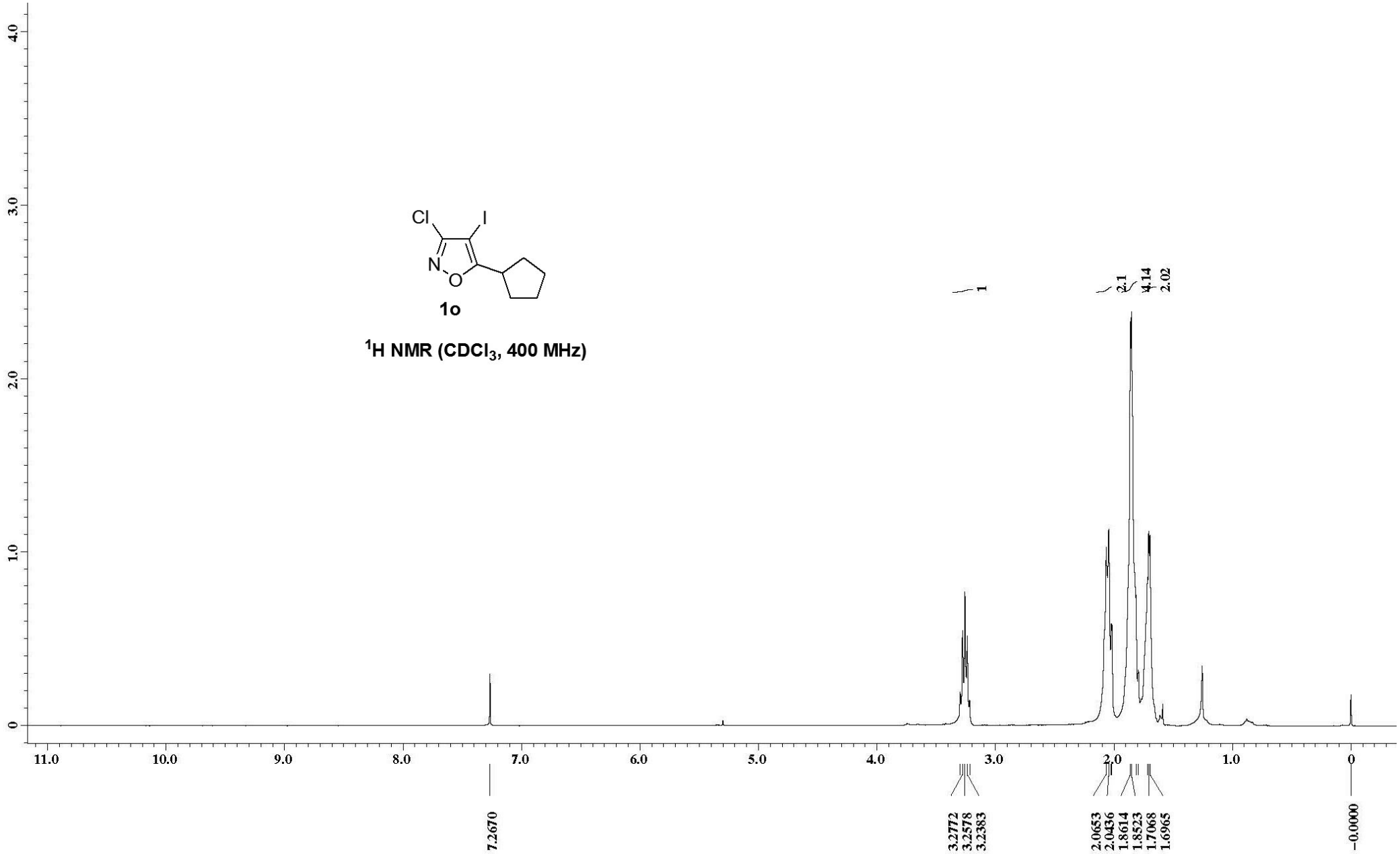

Cl
I
N
O
Pr^i
1l
^{13}C NMR (CDCl$_3$, 100 MHz)
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
220.0
210.0
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
176.4266
157.1000
77.3242
77.0000
76.6854
59.4468
35.5627
27.4583
25.5323
22.1190

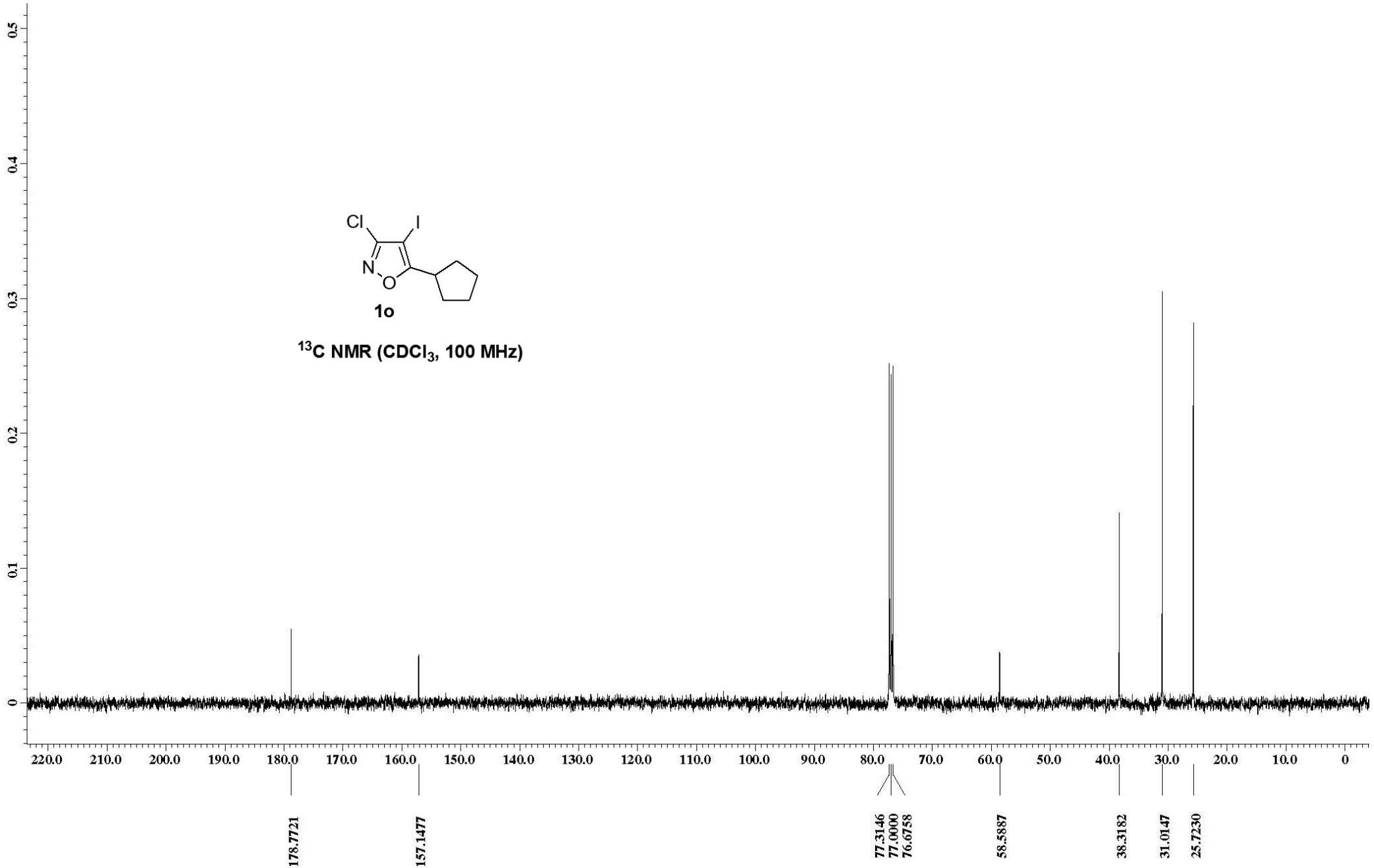

Cl
I
N
O
5
1m
1H NMR (CDCl3, 400 MHz)
2
2.05
6.03
3.05
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.2636
2.8168
2.7984
2.7790
1.7263
1.7091
1.6907
1.3677
1.3460
1.3208
1.2852
0.9084
0.8923
0.8752
0.0000

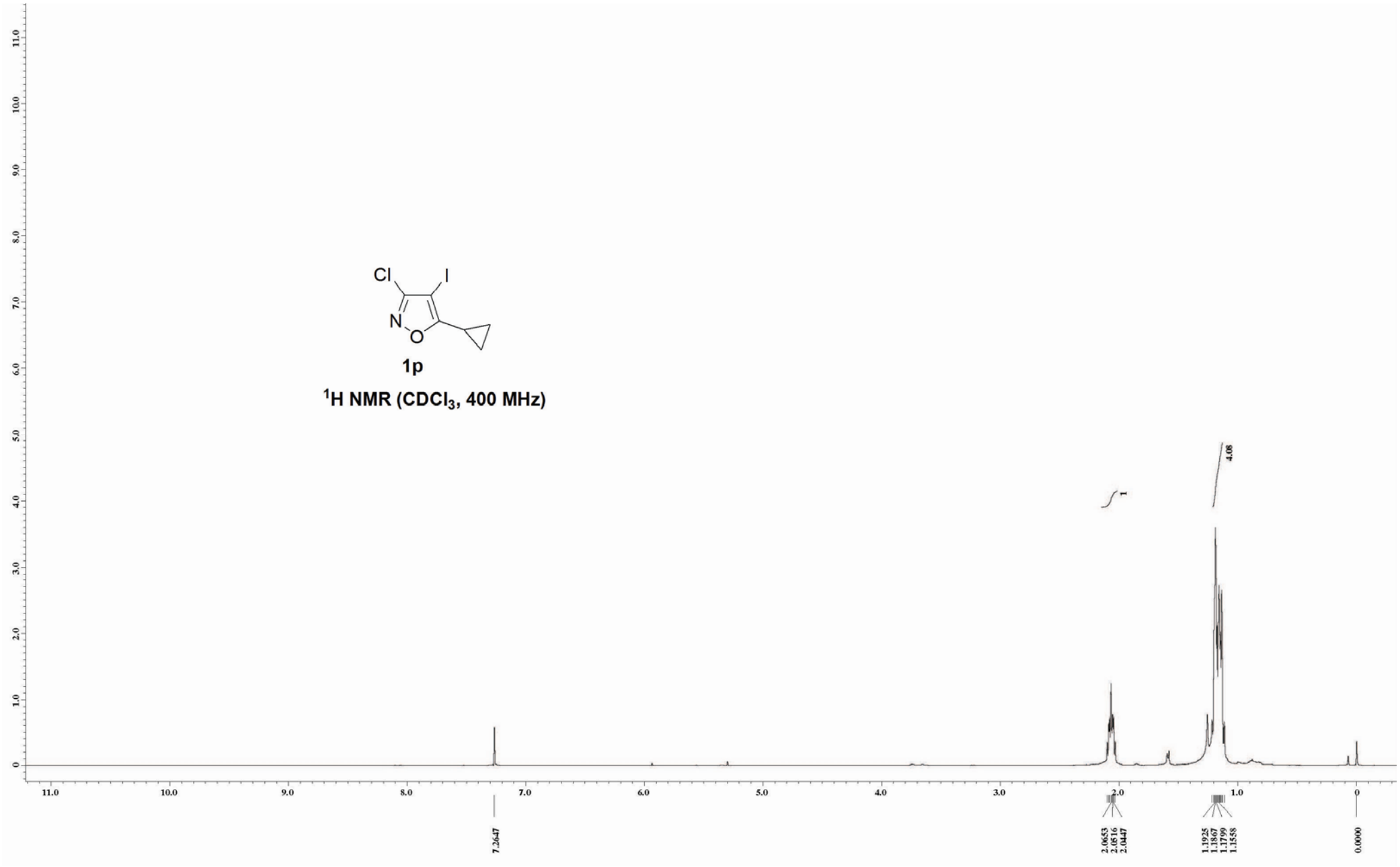

Cl
I
N
O
5
1m
^{13}C NMR (CDCl$_3$, 100 MHz)
0.4
0.3
0.2
0.1
0
220.0
210.0
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
176.3408
157.1191
77.3146
77.0000
76.6854
59.5994
31.2340
28.5643
27.4965
26.8576
22.4145
13.9764

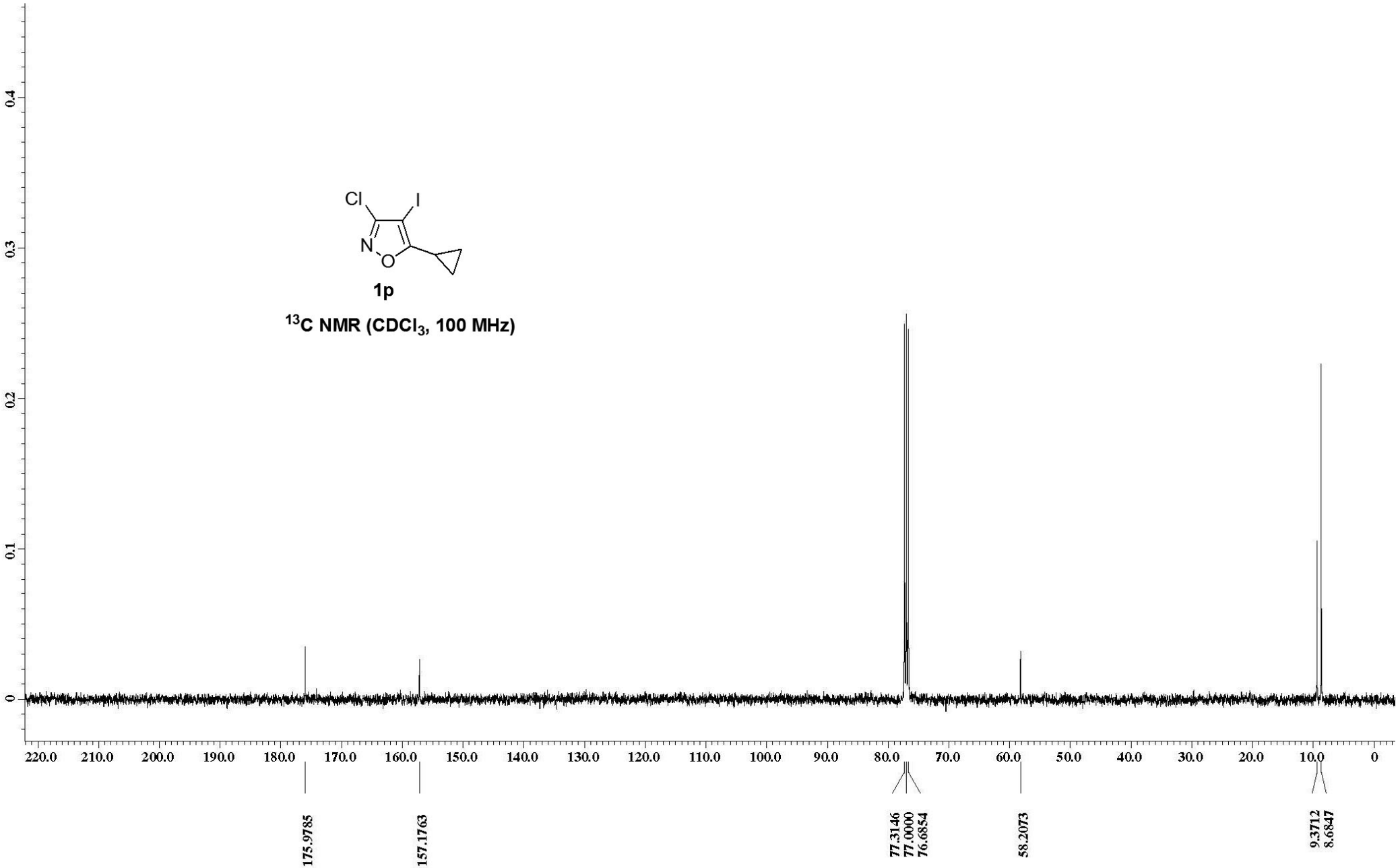

Cl
I
N
O
1n
1H NMR (CDCl3, 400 MHz)
1
3.14
1.1
2.14
4.03
3.0
2.0
1.0
0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.2636
2.8546
2.8465
2.8385
1.9004
1.8901
1.8740
1.8500
1.6472
1.3608
1.3334
1.2555
0.0000

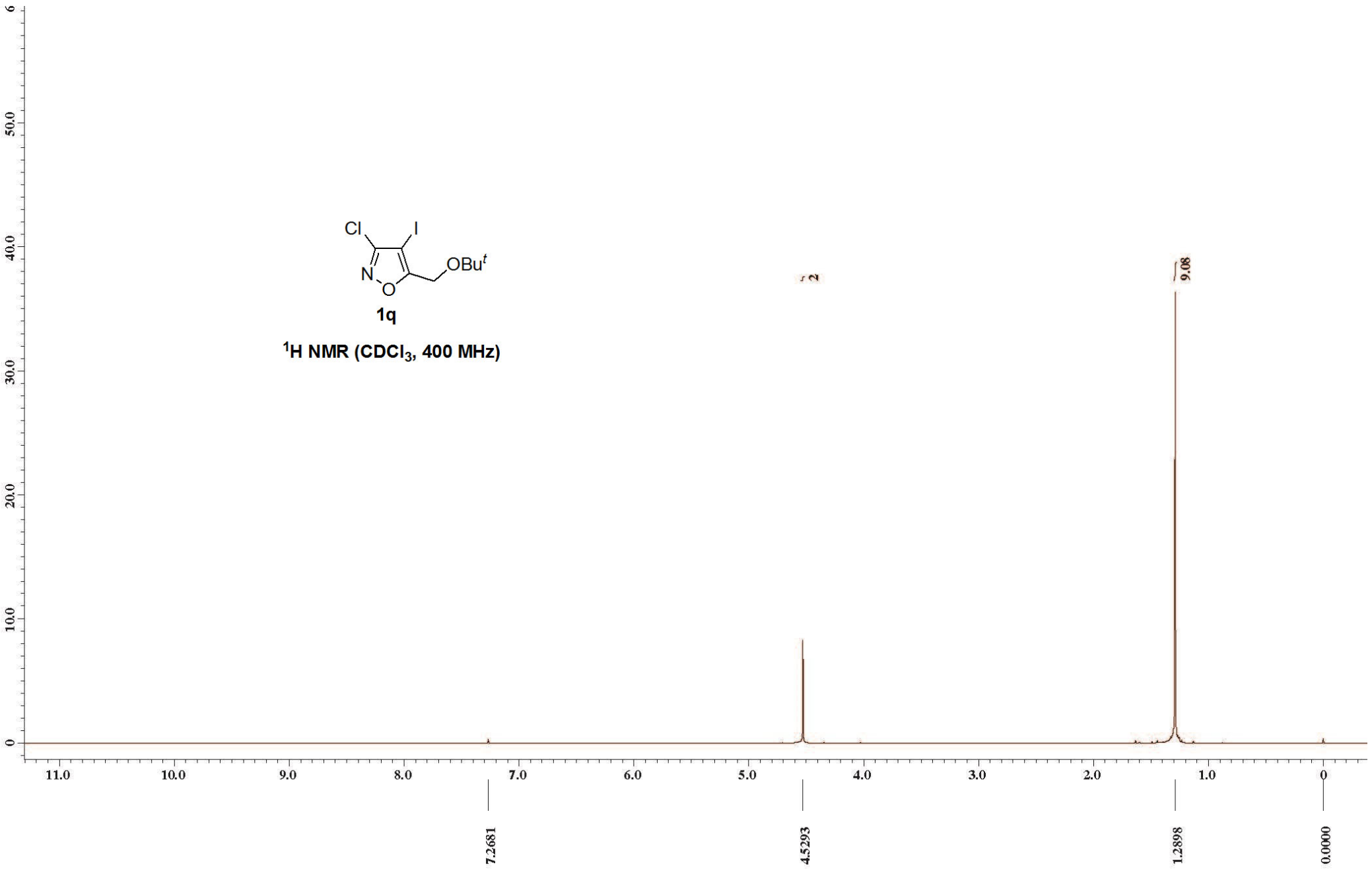

Cl
I
N
O
1n
^{13}C NMR ($CDCl_3$, 100 MHz)
0.5
0.4
0.3
0.2
0.1
0
220.0
210.0
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
178.8579
157.0428
77.3146
77.0000
76.6854
57.8832
37.8605
29.8038
25.7612
25.4465

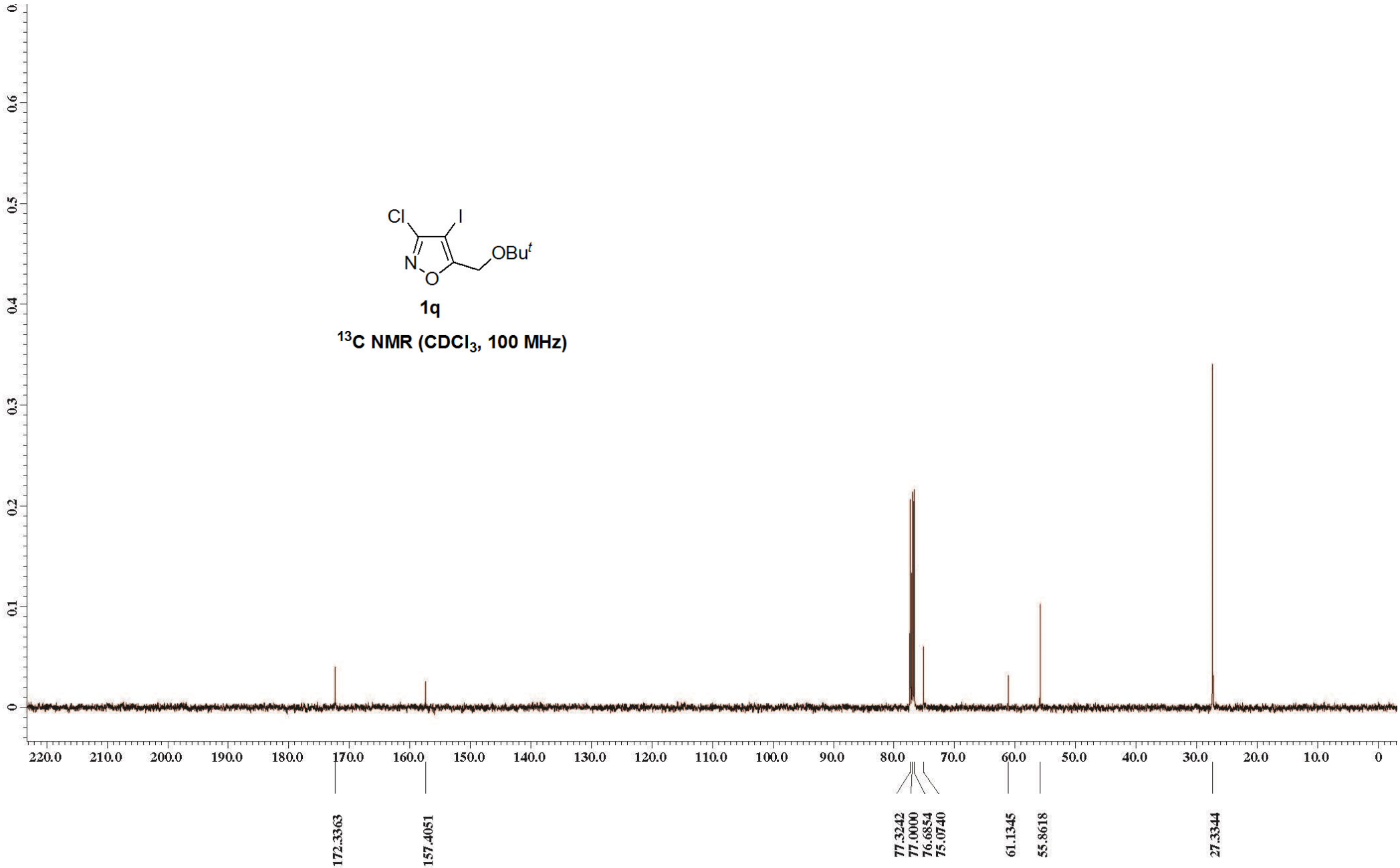

Cl
I
N
O
1o
^{1}H NMR ($CDCl_3$, 400 MHz)
1
2.1
4.14
2.02
4.0
3.0
2.0
1.0
0
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.2670
3.2772
3.2578
3.2383
2.0653
2.0436
1.8614
1.8523
1.7068
1.6965
−0.0000

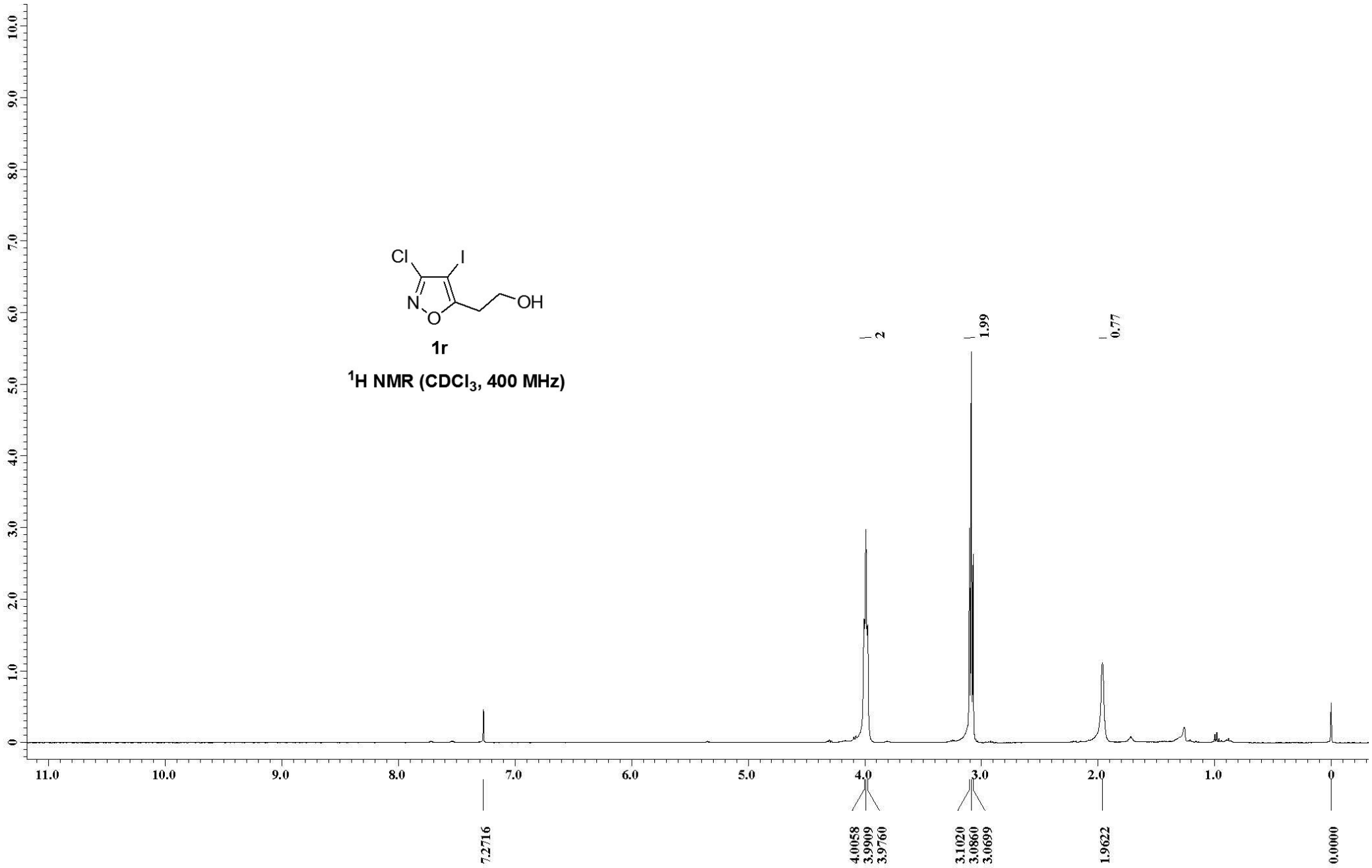

Cl
I
N
O
1o
^{13}C NMR ($CDCl_3$, 100 MHz)
0.5
0.4
0.3
0.2
0.1
0
220.0
210.0
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
178.7721
157.1477
77.3146
77.0000
76.6758
58.5887
38.3182
31.0147
25.7230

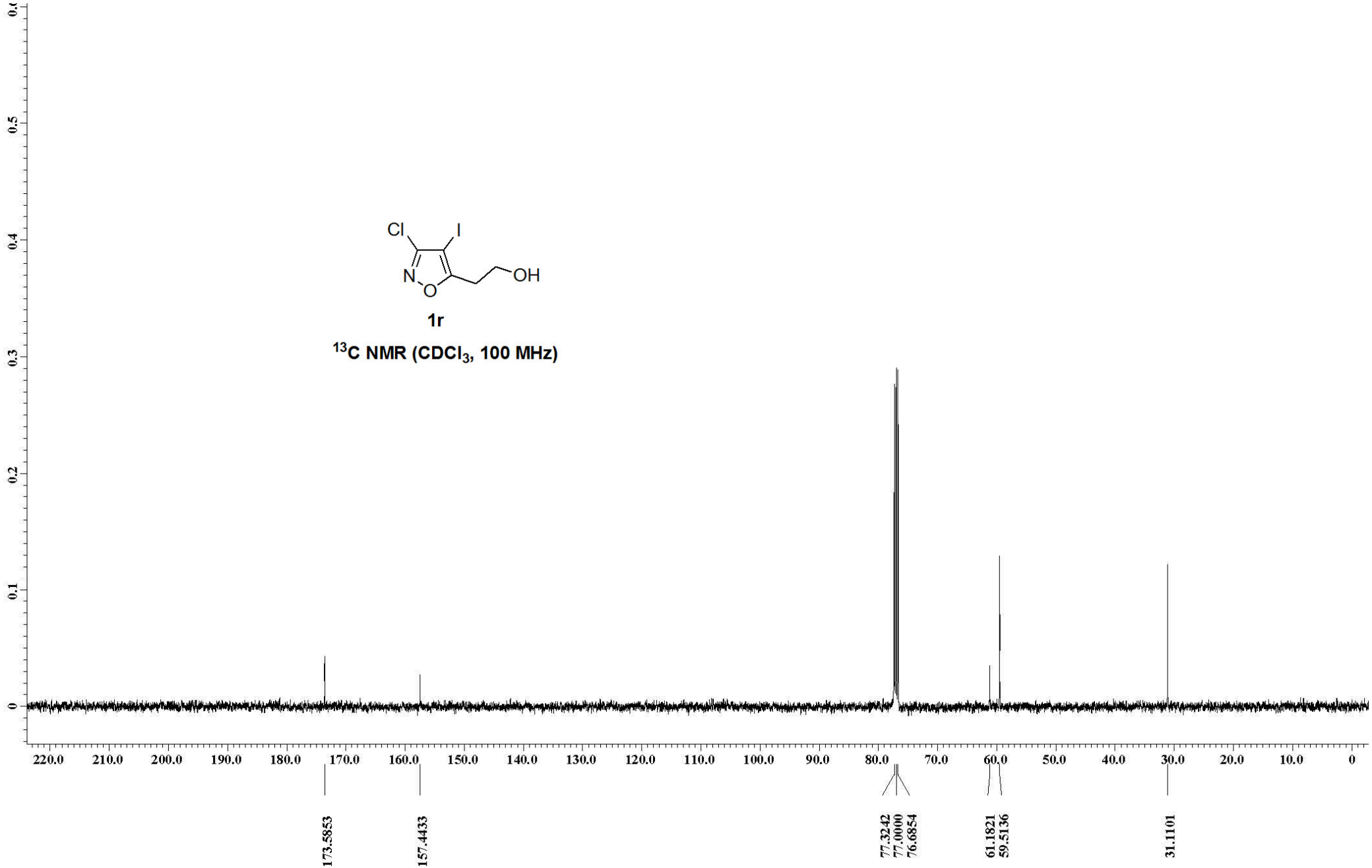

Cl
I
N
O
1p
1H NMR (CDCl3, 400 MHz)
1
4.08
11.0
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0
7.2647
2.0653
2.0516
2.0447
1.1925
1.1867
1.1799
1.1558
0.0000

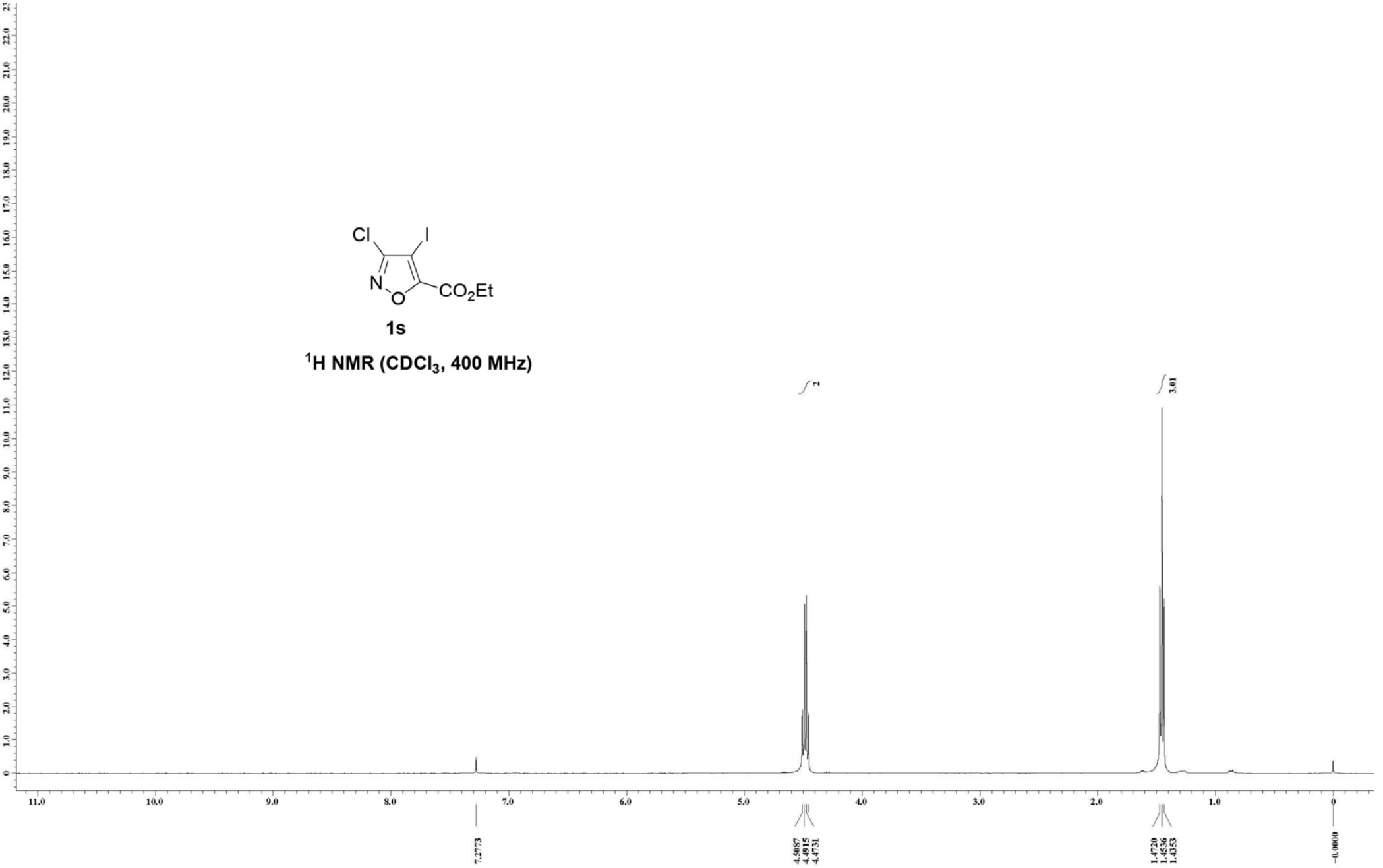

Cl
I
N
O
1p
13C NMR (CDCl3, 100 MHz)
0.4
0.3
0.2
0.1
0
220.0
210.0
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
175.9785
157.1763
77.3146
77.0000
76.6854
58.2073
9.3712
8.6847

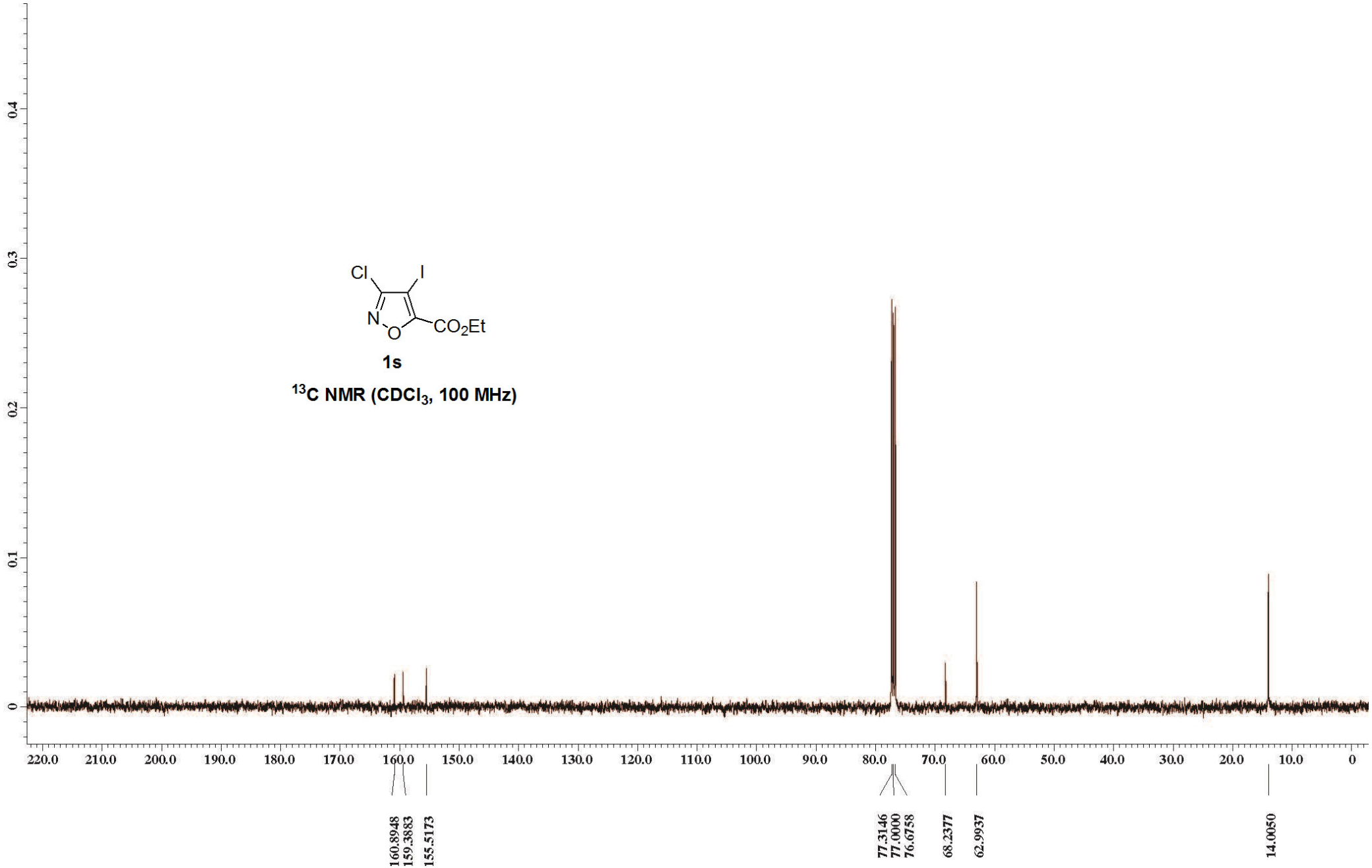

Cl
I
N
O
OBu^t
1q
^{1}H NMR (CDCl$_3$, 400 MHz)
9.08
7.2681
4.5293
1.2898
0.0000

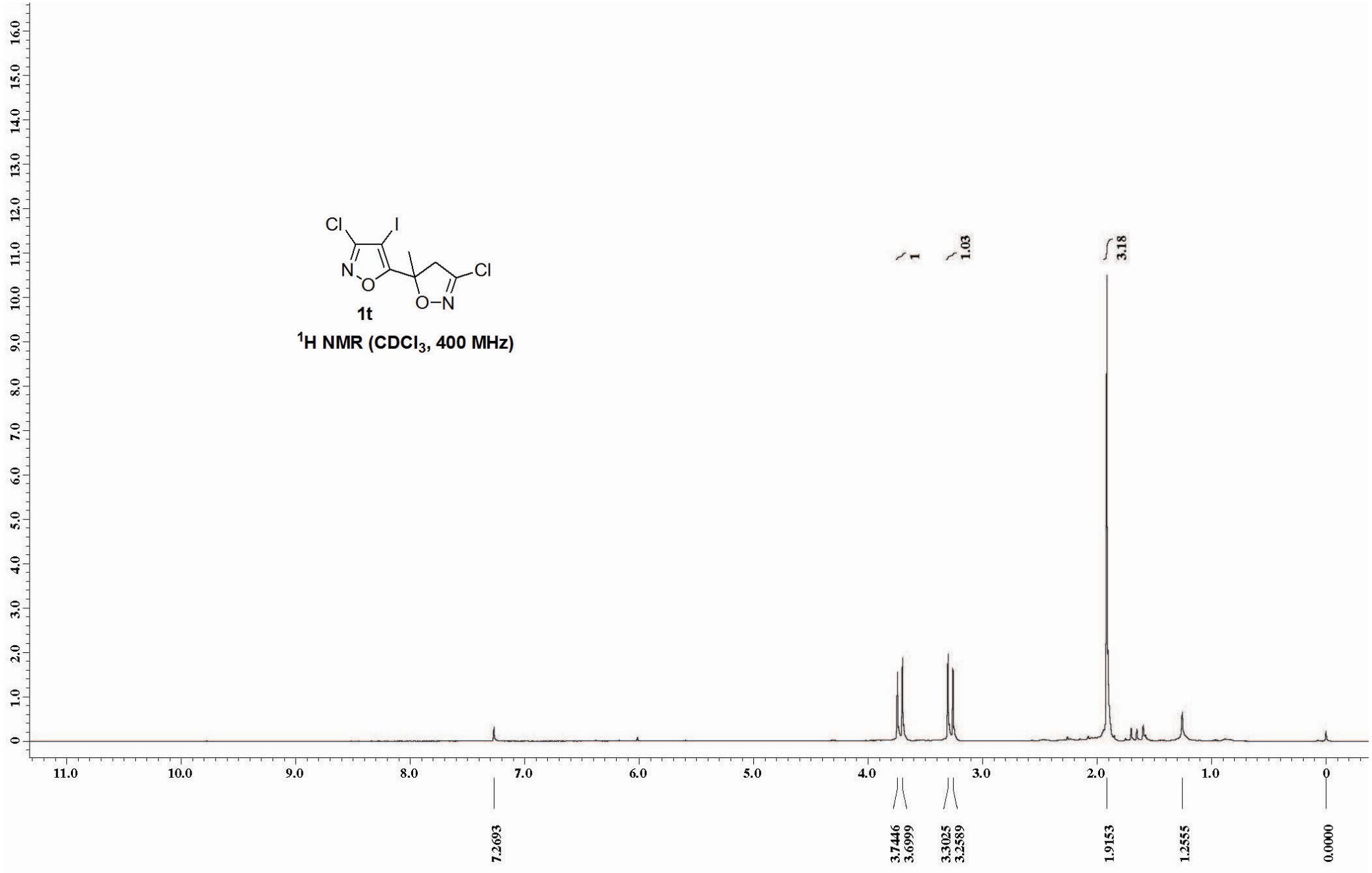

Cl
I
N
O
OBu^t
1q
^{13}C NMR ($CDCl_3$, 100 MHz)
0
0.1
0.2
0.3
0.4
0.5
0.6
220.0
210.0
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
172.3363
157.4051
77.3242
77.0000
76.6854
75.0740
61.1345
55.8618
27.3344

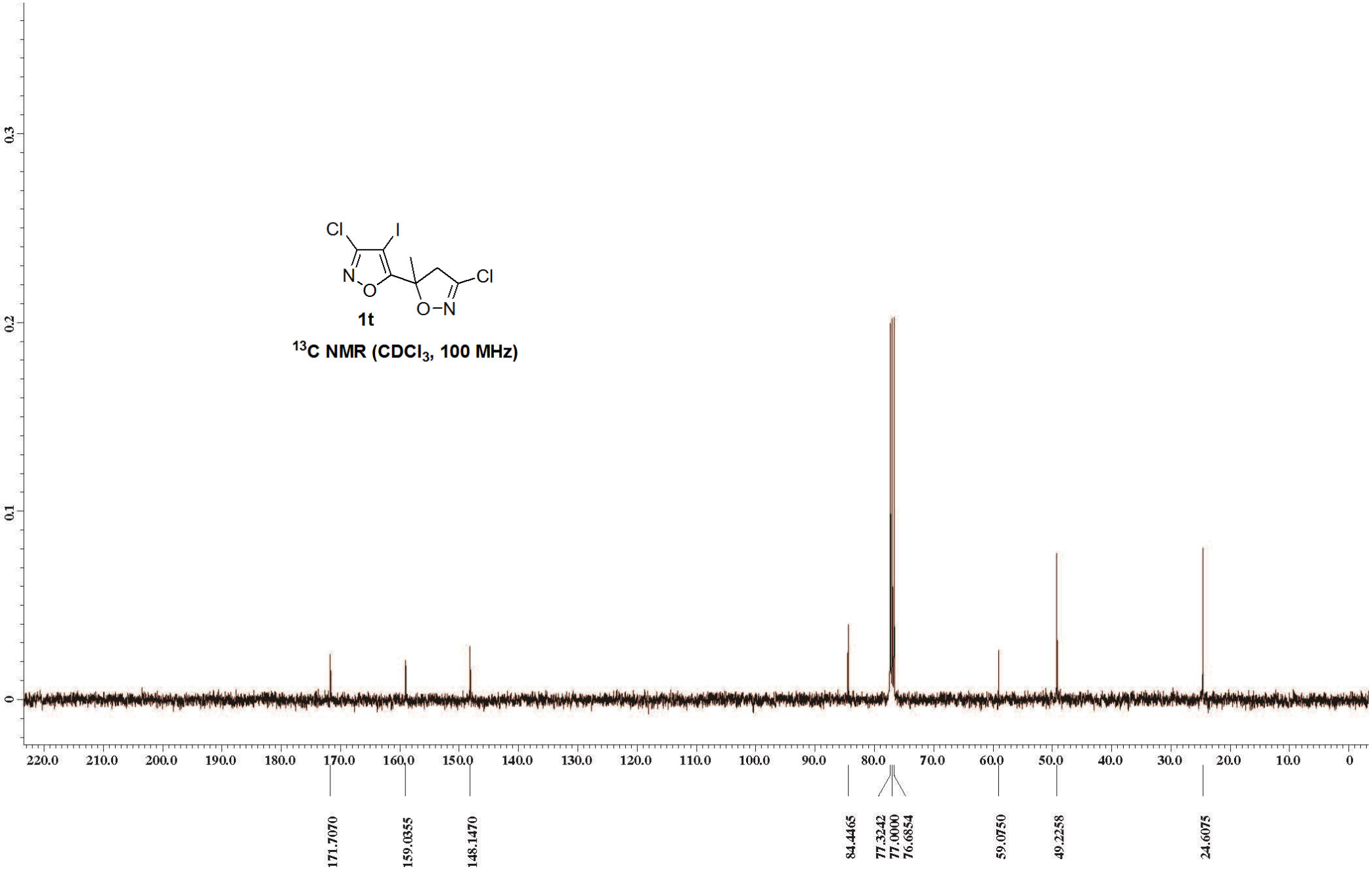

Cl
I
N
O
OH
1r
1H NMR (CDCl3, 400 MHz)
2
1.99
0.77
7.2716
4.0058
3.9909
3.9760
3.1020
3.0860
3.0699
1.9622
0.0000

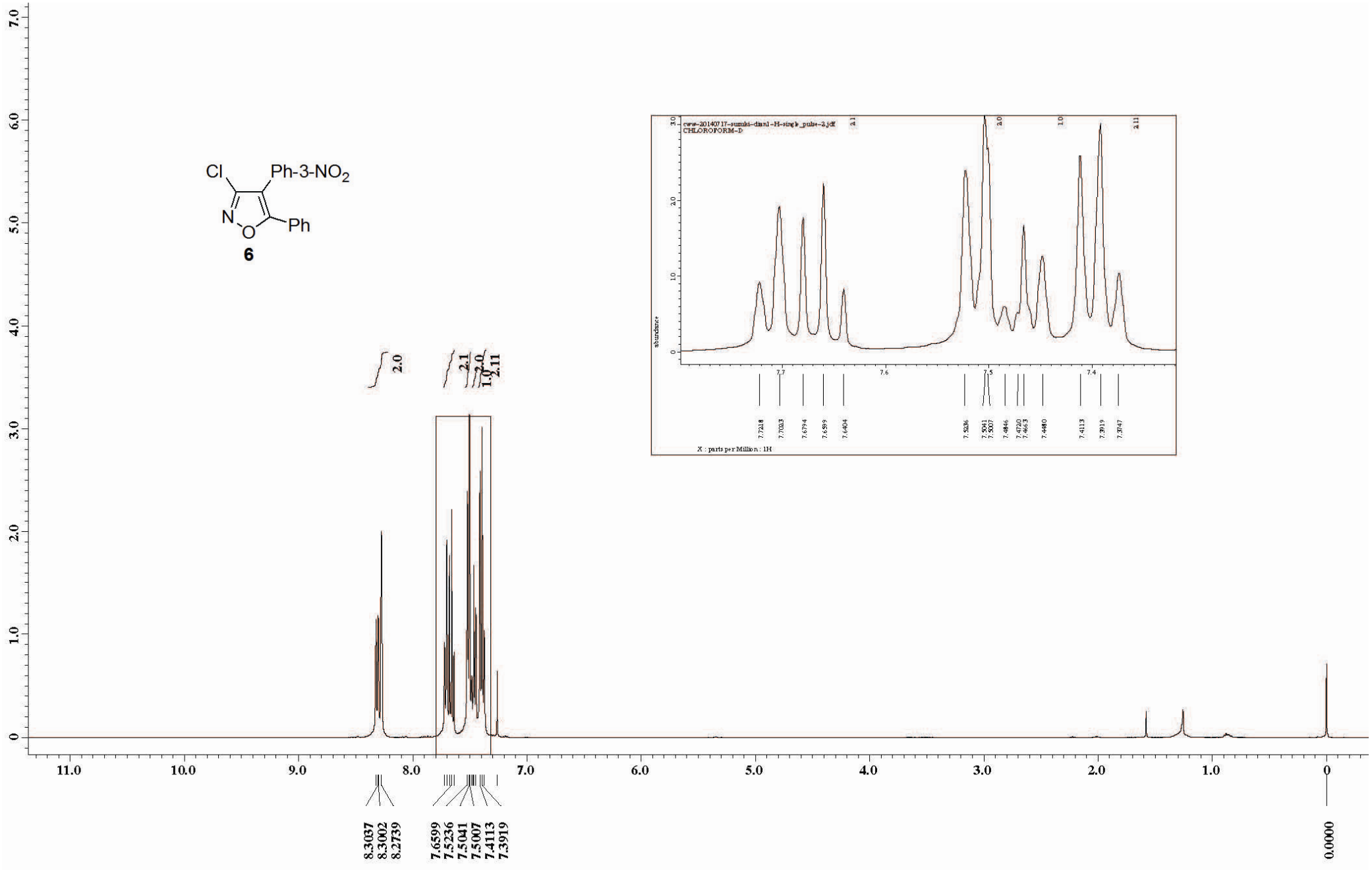

Cl
I
N
O
OH
1r
13C NMR (CDCl3, 100 MHz)
0.5
0.4
0.3
0.2
0.1
0
220.0
210.0
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
173.5853
157.4433
77.3242
77.0000
76.6854
61.1821
59.5136
31.1101

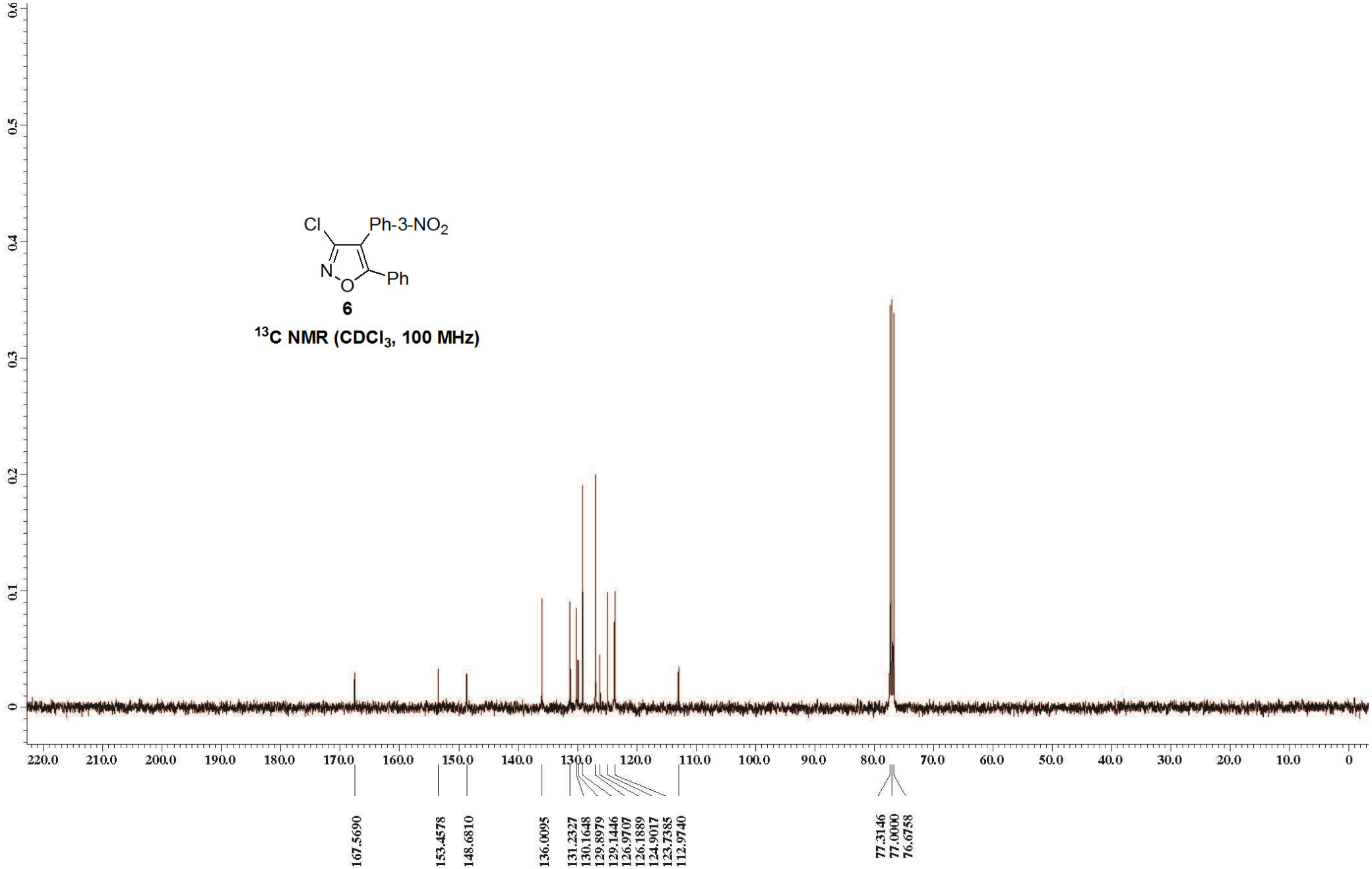

Cl
I
N
O
CO2Et
1s
1H NMR (CDCl3, 400 MHz)
2
3.01
7.2773
4.5087
4.4915
4.4731
1.4720
1.4536
1.4353
-0.0000

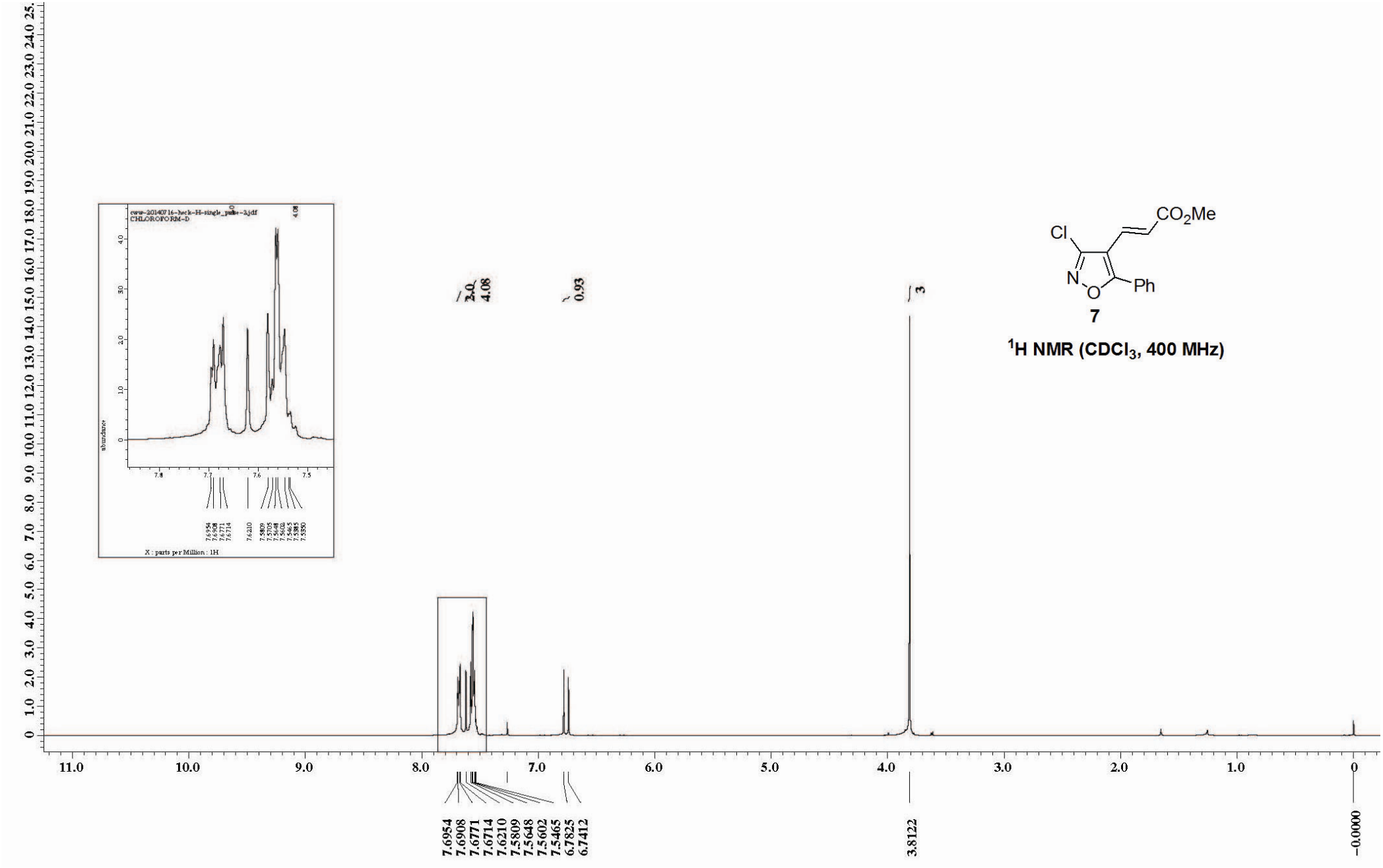

Cl
I
N
O
CO2Et
1s
13C NMR (CDCl3, 100 MHz)
0.4
0.3
0.2
0.1
0
220.0
210.0
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
160.8948
159.3883
155.5173
77.3146
77.0000
76.6758
68.2377
62.9937
14.0050

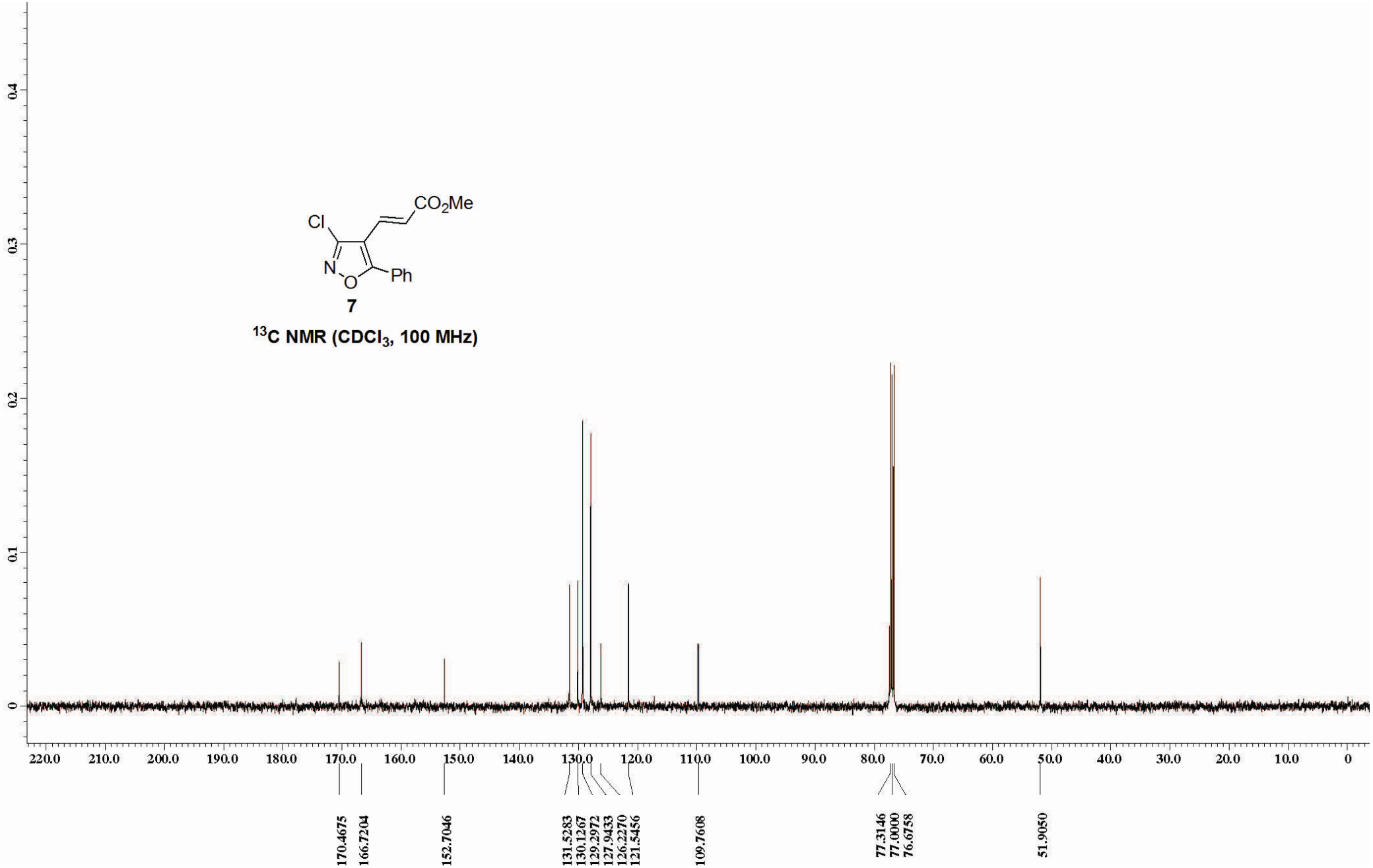

Cl
I
N
O
Cl
O–N
1t
^{1}H NMR (CDCl$_3$, 400 MHz)
1
1.03
3.18
7.2693
3.7446
3.6999
3.3025
3.2589
1.9153
1.2555
0.0000

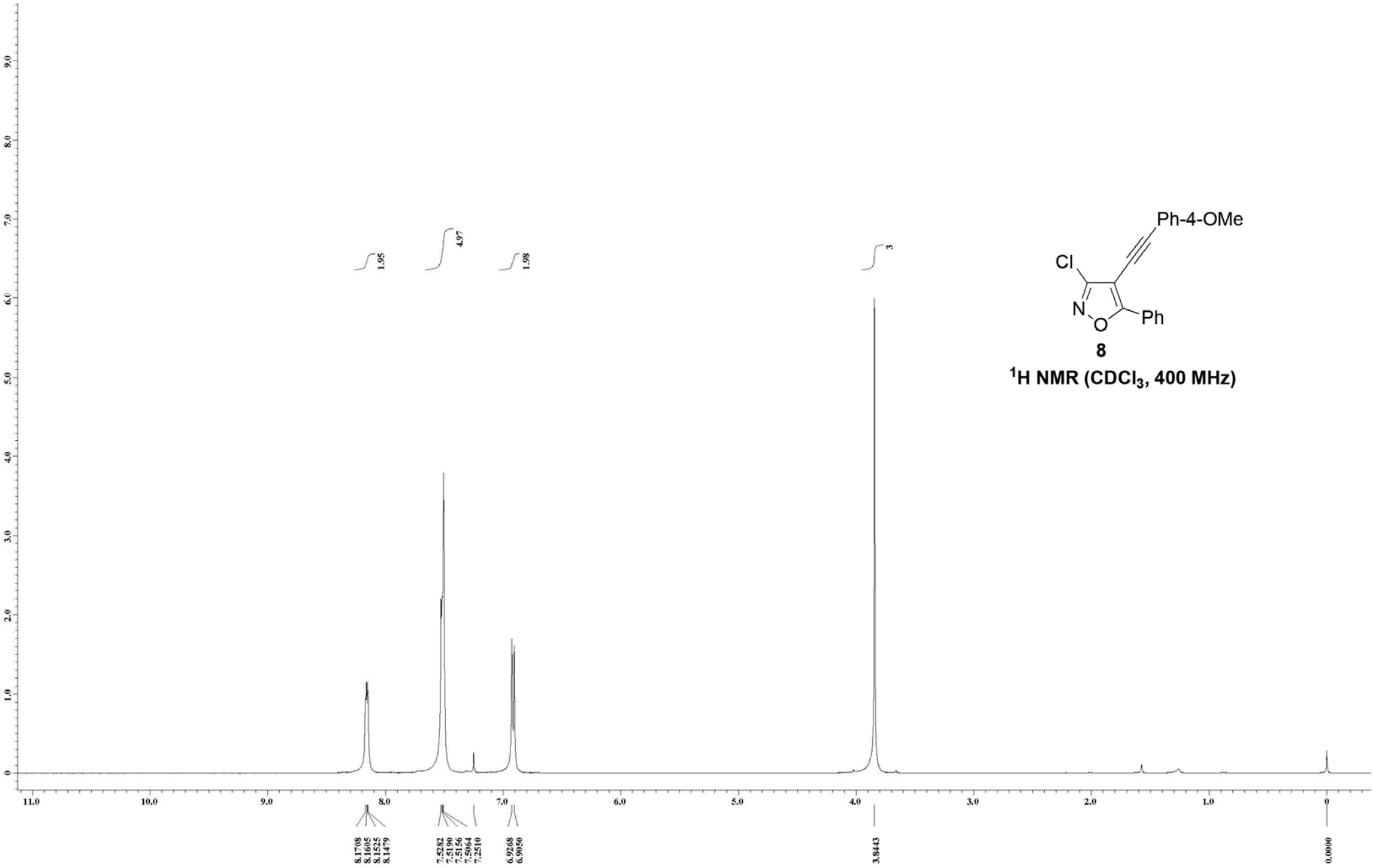

Cl
I
N
O
Cl
O–N
1t
^{13}C NMR (CDCl$_3$, 100 MHz)
0.3
0.2
0.1
0
220.0
210.0
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
171.7070
159.0355
148.1470
84.4465
77.3242
77.0000
76.6854
59.0750
49.2258
24.6075

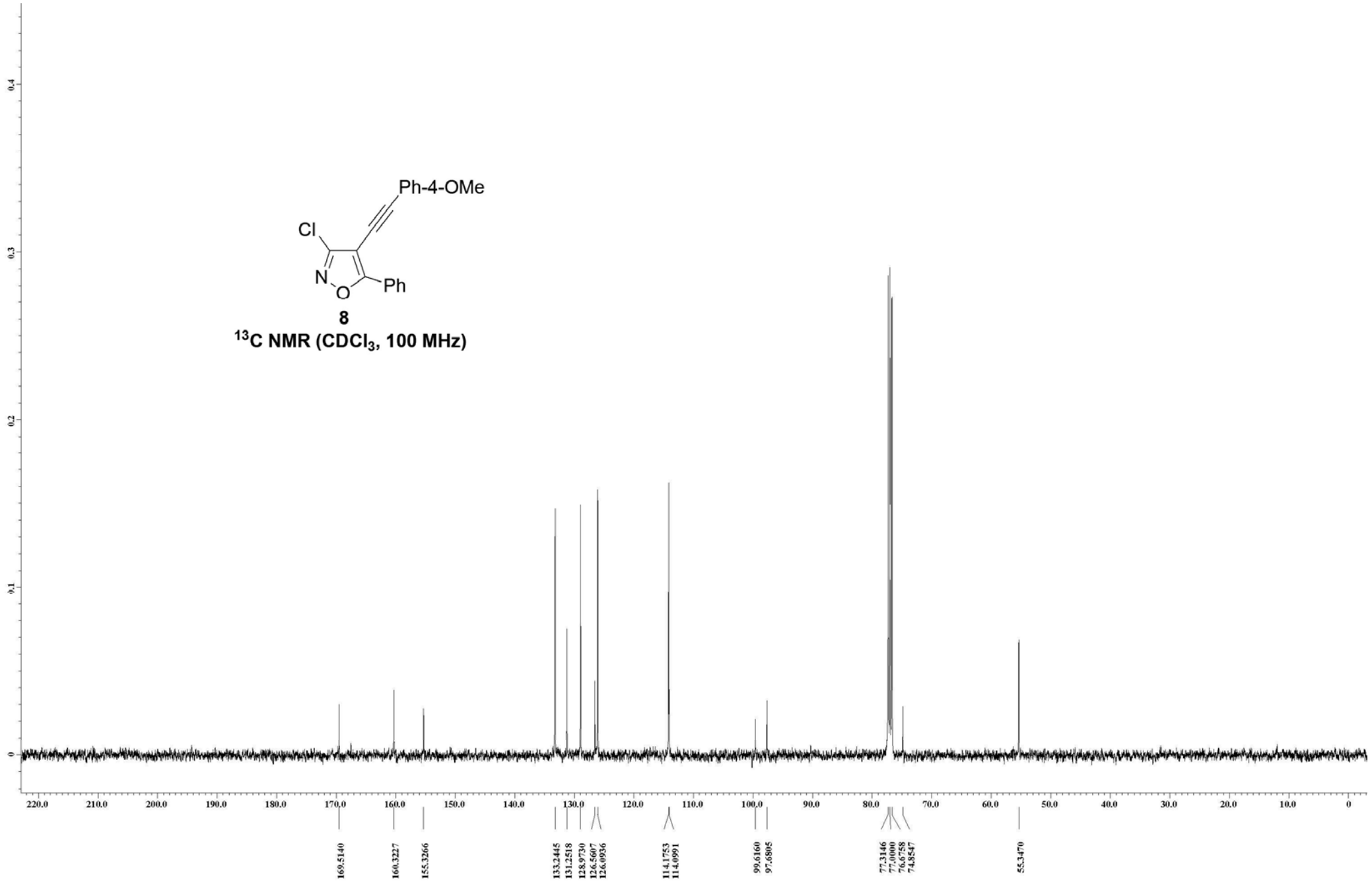

Cl
Ph-3-NO2
N
O
Ph
6
2.0
2.1
2.0
1.0
2.11
8.3037
8.3002
8.2739
7.6599
7.5236
7.5041
7.5007
7.4113
7.3919
0.0000
cwe-20140717-suzuki-diaryl-H-single_pulse-2.jdf
CHLOROFORM-D
X : parts per Million : 1H


Cl
Ph-3-NO2
N
O
Ph
6
13C NMR (CDCl3, 100 MHz)
0.6
0.5
0.4
0.3
0.2
0.1
0
220.0
210.0
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
167.5690
153.4578
148.6810
136.0095
131.2327
130.1648
129.8979
129.1446
126.9707
126.1889
124.9017
123.7385
112.9740
77.3146
77.0000
76.6758


Cl
N
O
Ph
CO2Me
7
1H NMR (CDCl3, 400 MHz)
2.0
4.08
0.93
3
7.6954
7.6908
7.6771
7.6714
7.6210
7.5809
7.5648
7.5602
7.5465
6.7825
6.7412
3.8122
−0.0000


CO2Me
Cl
N
O
Ph
7
13C NMR (CDCl3, 100 MHz)
0.4
0.3
0.2
0.1
0
220.0
210.0
200.0
190.0
180.0
170.0
160.0
150.0
140.0
130.0
120.0
110.0
100.0
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
170.4675
166.7204
152.7046
131.5283
130.1267
129.2972
127.9433
126.2270
121.5456
109.7608
77.3146
77.0000
76.6758
51.9050


1.95
4.97
1.98
3
Ph-4-OMe
Cl
N
O
Ph
8
^{1}H NMR ($CDCl_3$, 400 MHz)
8.1708
8.1605
8.1525
8.1479
7.5282
7.5190
7.5156
7.5064
7.2510
6.9268
6.9050
3.8443
0.0000


Ph-4-OMe
Cl
N
O
Ph
8
13C NMR (CDCl3, 100 MHz)
169.5140
160.3227
155.3266
133.2445
131.2518
128.9730
126.5607
126.0936
114.1753
114.0991
99.6160
97.6805
77.3146
77.0000
76.6758
74.8547
55.3470