Supporting Information

Regio-selective C (sp2)-H imidation of Arenes by Redox Neutral Visible-Light Photo Catalysis

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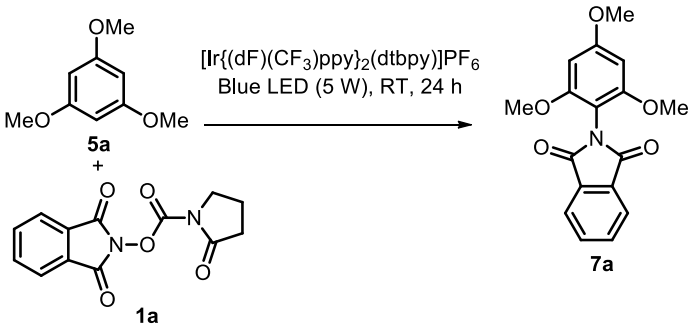
1. Optimization of Reaction Condition: Reaction are performed with different equivalent of Arnes and imides as well as different mol % of catalyst loading. (**Table-1**)



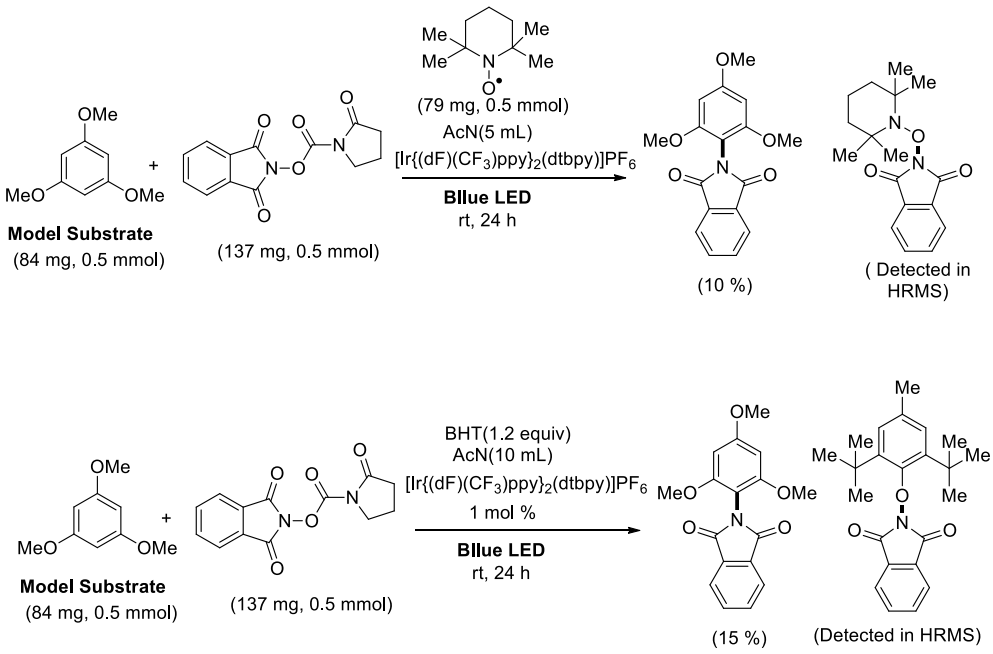
Entry	Imide (equiv)	Arene (equiv)	Catalyst (mol %)	Solvent	Yield (%)
1	1	1	1	5 mL	55
2	**1**	**1.2**	**1**	**5 mL**	**60**
3	1.2	1	1	5 mL	55
4	**1.3**	**1**	**0.5-1**	**8 mL**	**60**
5	1.5	1	1	10 mL	35
6	2	1	1	10 mL	15
7	1	2	0.5-1	5 mL	45
8	1	4	0.5-1	5 mL	50
9	1	1	5	5 mL	45

Table -1: Optimization of Reaction Condition

N.B. The limiting reactants used as 0.5 mmol (1 equivalent). All the reported yields are isolated yields after purification.

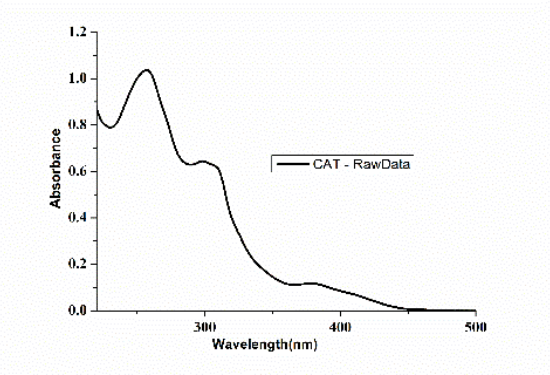
2. Controlled Experiments:

To understand the mechanism insight some controlled experiment has been done with the radical trapping experiments. The reaction in presence of TEMPO and BHT were suppressed and produce trace amount of the coupling product, although we are unable to isolate the radical-trapping adduct with TEMPO and BHT but detected by HRMS. These controlled experiments support the radical-N cross coupling reaction.

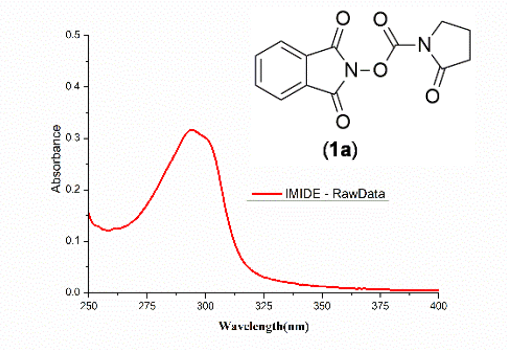


Scheme-1: Controlled radical-trapping experiment using TEMPO and BHT

3. UV-vis absorption spectrum of [Ir{(dF)(CF$_3$)ppy}$_2$(dtbpy)]PF$_6$ and imide (1a): UV-vis spectra has been recorded on a Shimadzu 1800 instrument.

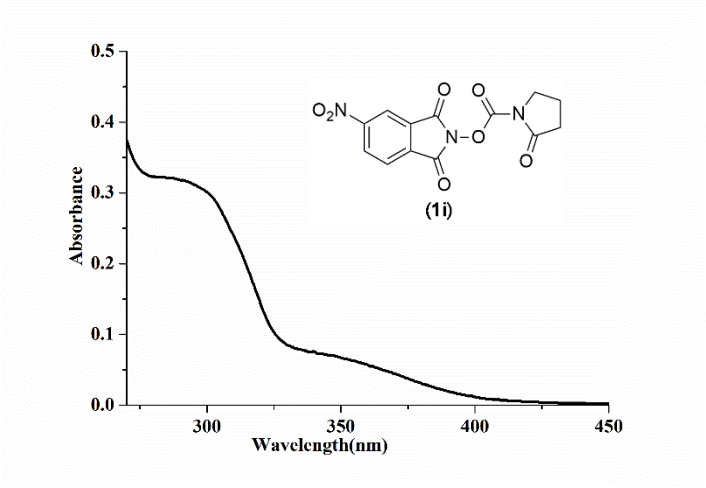


UV-vis absorption s(15 µM in acetonitrile) spectrum of [Ir{(dF)(CF$_3$)ppy}$_2$(dtbpy)]PF$_6$ (15 µM in acetonitrile)
(Figure-1)



UV-vis absorption spectrum of imide (**1a**)(100 µM in acetonitrile)
λ_{max} = 295nm
(Figure-2)

4. UV-vis absorption spectrum of imide (1i): UV-vis spectra has been recorded on a Shimadzu 1800 instrument.



UV-vis absorption spectrum of imide (**1i**)(100 µM in acetonitrile)
(Figure-3)

5. Fluorescence quenching of [Ir{(dF)(CF_3)ppy}$_2$(dtbpy)]PF_6 with varying concentration of 1,3 dioxoisoindolin-2-yl 2-oxopyrrolidine-1-carboxylate (1a): Emission spectra has been recorded on a Perkin Elmer LS55 Fluorescence instrument.

A stock solution of [Ir{(dF)(CF_3)ppy}$_2$(dtbpy)]PF_6 (0.1 mM) was prepared by dissolving 2.8 mg of [Ir{(dF)(CF_3)ppy}$_2$(dtbpy)]PF_6 in 25 mL of acetonitrile. The catalyst stock solution of the above concentration was distributed into five 10 mL standard flasks equally (1 mL in each flasks). In the same way, stock solution of imide **1a** (1 mM) was prepared by dissolving 6.8 mg of **1a** in 25 mL of acetonitrile. From the above stock solution of imide **1a,** 0 mL, 1.5 mL, 2.5 mL, 5 mL, and 8mL were pipetted out and added to the above five 10 mL slandered volumetric flasks containing 1 mL of catalyst stock solution and diluted to 10 mL with acetonitrile. The fluorescence spectrum of flask 1 containing only 0.01 mM Ir(III)-catalyst was measured by exciting at 420 nm (λ_{ex}) and emission was measured at 476 nm (λ_{em}). Similarly, fluorescence intensity of other solutions containing different concentration of imide (**1a**) was measured (**Figure-4**). The details of decrease in fluorescence intensity by increased concentration of imide (**1a**) are shown in below **Table-2**

SL No	Concentration of Imide (mM)	wavelength	I_0	I	I_0/I
Ir(III)	0	476	126.978372	126.978372	1
A1	0.15	476	126.978372	125.588657	1.01
A2	0.25	476	126.978372	126.978372	1.02
A3	0.55	476	126.978372	122.304842	1.04
A4	0.80	476	126.978372	120.129439	1.06

Table-2: Fluorescence quenching of Ir(III) with varying concentration Imide (**1a**)

Analysis of the above tabulated data using Stern-Volmer quenching kinetic equation and by plotting I_0/I on Y-axis with corresponding concentration of imide (**1a**) on X-axis resulted a straight line (**Figure-5**). The slope of the curve was 0.0747 and the intercept on Y-axis is 0.999. Incorporating the excited-state lifetime of $[Ir\{(dF)(CF_3)ppy\}_2(dtbpy)]PF_6$ (τ_0 = 2.3 μS = 2.3X 10^{-6} S)[1] in the slope, rate of quenching Kq was measured as shown below-

$I_0/I = 1 + K_q\tau\,[Q]$

Where, I_0/I = ratio of intensity of fluorescence without and with quencher concentration.

K_q = Rate constant for quenching, τ = life time of catalyst, [Q] = concentration of quencher.

Here slope = $K_q\tau_0$

So, $0.0747 = K_q\tau_0$, or $K_q = 0.0747/\tau_0$

$= 0.0747/[2.3 \times 10^{-6}]$

$= 3.2 \times 10^4\ M^{-1}\ S^{-1}$

So the measured experimental quenching rate constant (K_q) = $3.2 \times 10^4\ M^{-1}\ S^{-1}$

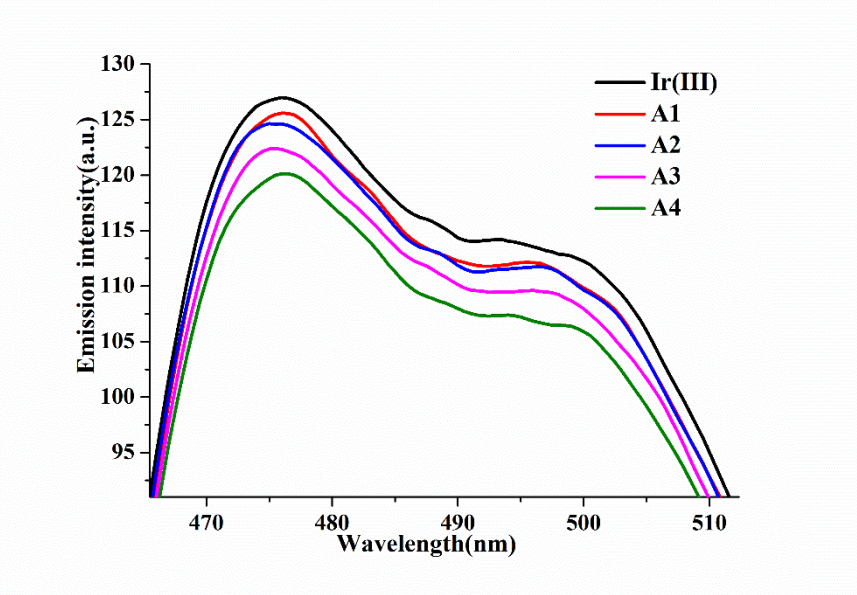


Figure-4: Fluorescence quenching spectra of [Ir{(dF)(CF_3)ppy}$_2$(dtbpy)]PF_6 (0.01 mM) with varying concentration of Imide (**1a**). All the samples excited at 420 nm (λ_{ex}) and emission was measured at 476 nm (λ_{em}). Ir (III)-only Catalyst. A1 = 0.15 mM solution of imide **1a** + catalyst Ir(III) (0.01 mM); A2 = 0.25 mM solution of imide **1a** + catalyst Ir(III) (0.01 mM); A3 = 0.5 mM solution of imide **1a** + catalyst Ir(III) (0.01 mM); A4 = 0.8 mM solution of imide **1a** + catalyst Ir(III) (0.01 mM);

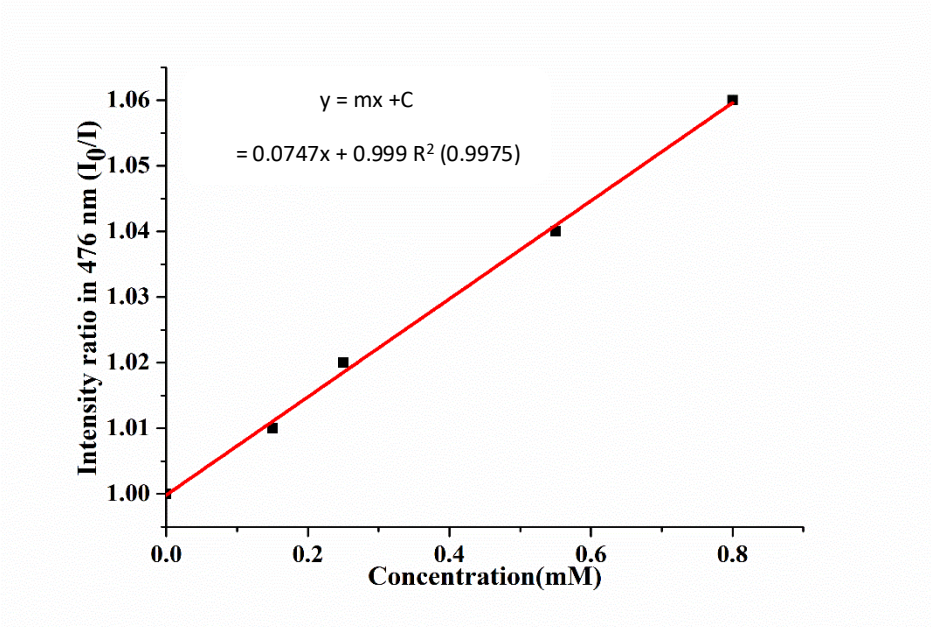
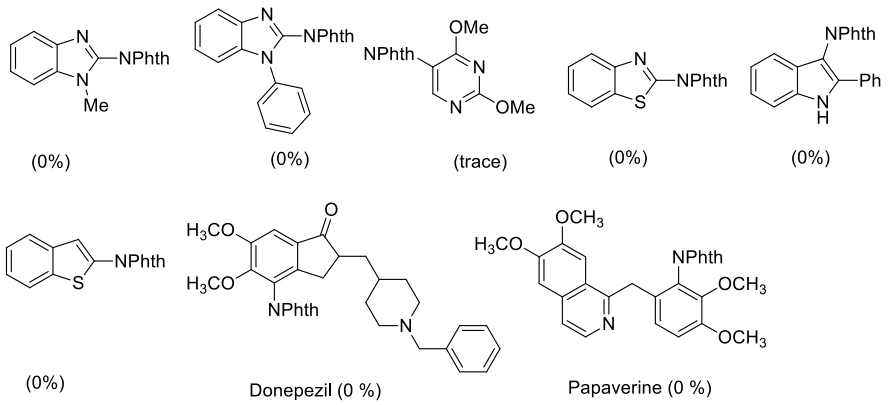


Figure-5: Stern-Volmer plot [I_0/I on Y-axis with corresponding concentration of imide (**1a**) on X-axis] Intercept = 0.999; slope = .0747; R^2 = 0.9975;

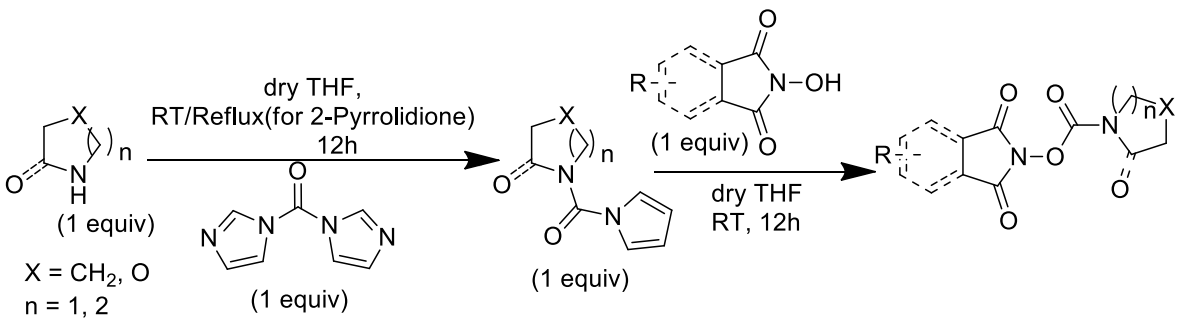
5. Unsuccessful attempts:

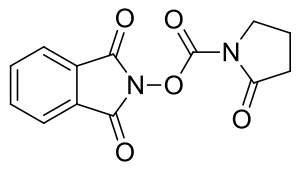


6. Reaction setup- 5W blue LED light placed 2 cm below from the reaction flask and stirred at room temperature

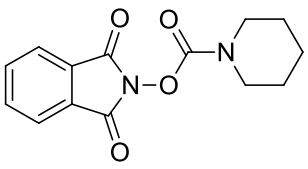
Figure-6: Photochemical reaction setup

7. Preparation of Imides: The following imides are synthesized using the general experimental procedure (A) presented in the manuscript:

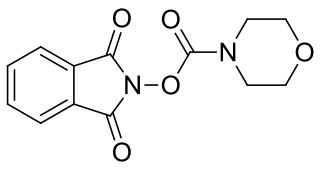




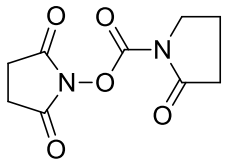
(**1a**)



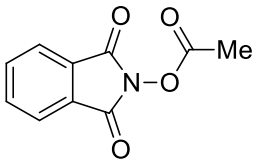
(**1b**)



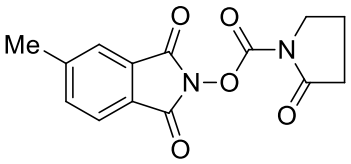
(**1c**)



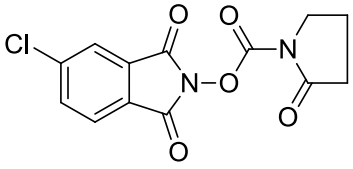
(**1d**)



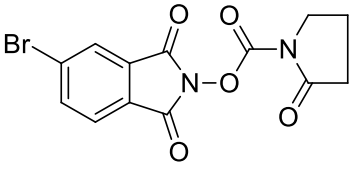
(**1e**)



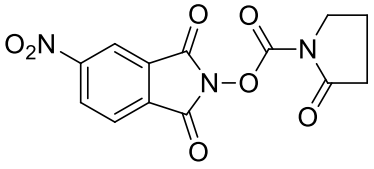
(**1f**)



(**1g**)

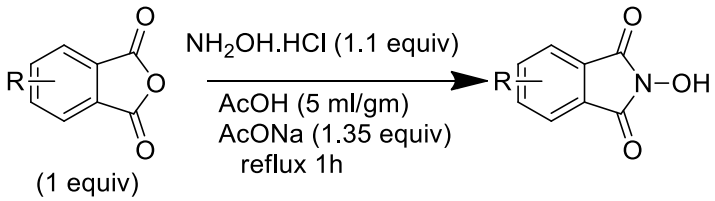


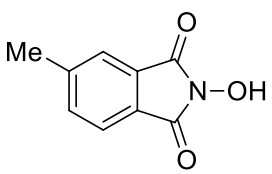
(**1h**)

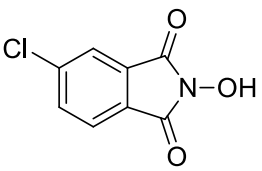


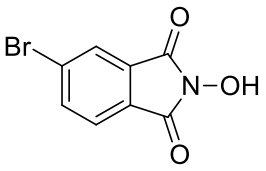
(**1i**)

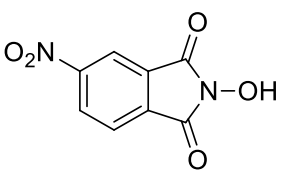
8. Preparation N-hydroxy phthalamides: The different derivative of N-hydroxy phthalamides are prepared as follows- In a round bottom flask hydroxylamine hydrochloride (1.1 equiv) and sodium acetate (1.35 equiv) was taken and then added acetic acid 1 ml/mmol of phthalic anhydride used. Then the mixture was reflux for 30 minutes and then formed NaCl was filtered and phthalic anhydride derivative (1 equiv) was dissolved in the filtrate and reflux for additional 1h. After that the reaction mixture was poured into ice cold water. Then the precipritate was collected by filtration and washed with cold water and dried in high vacuum for 1h and used without purification.



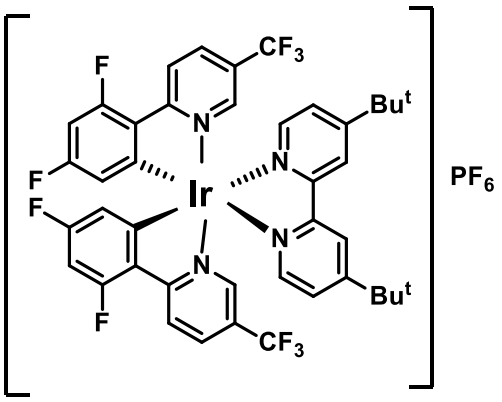






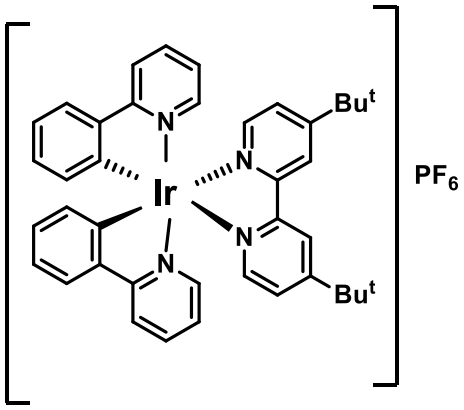


8. Details of Catalyst Used:[2]



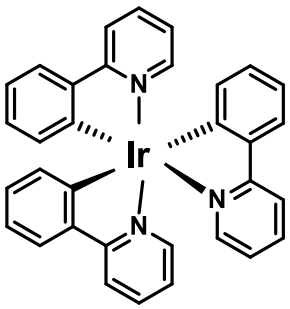
$[Ir\{(dF)(CF_3)ppy\}_2(dtbpy)]PF_6$

$E_{1/2}^{IV/III*}$ = -0.89 V vs SCE
$E_{1/2}^{III*/II}$ = +1.21 V vs SCE
λ_{max} = 380 nm
τ = 2.3 μs



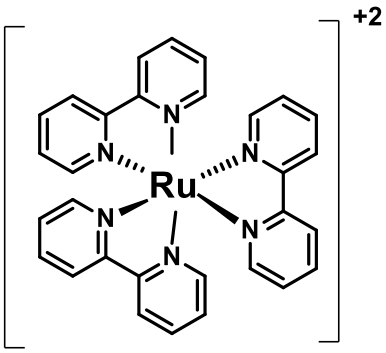
$[Ir(ppy\}_2(dtbpy)]PF_6$

$E_{1/2}^{IV/III*}$ = -0.96 V vs SCE
$E_{1/2}^{III*/II}$ = +0.66 V vs SCE
τ = 0.60 μs



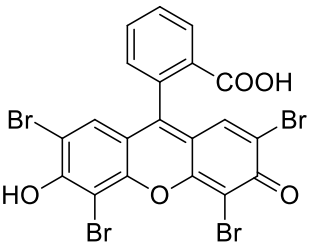
fac-Ir(ppy)$_3$

$E_{1/2}^{IV/III*}$ = -1.73 V vs SCE
$E_{1/2}^{III*/II}$ = +0.31 V vs SCE
λ_{max} =375 nm

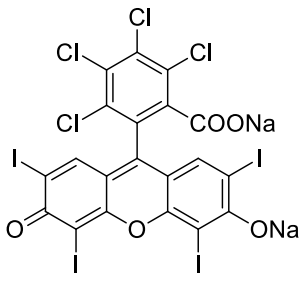


$Ru(bpy)_3^{2+}$

$E_{1/2}^{III/II*}$ = -0.81 V vs SCE
$E_{1/2}^{II*/I}$ = +0.77 V vs SCE
λ_{max} = 452 nm

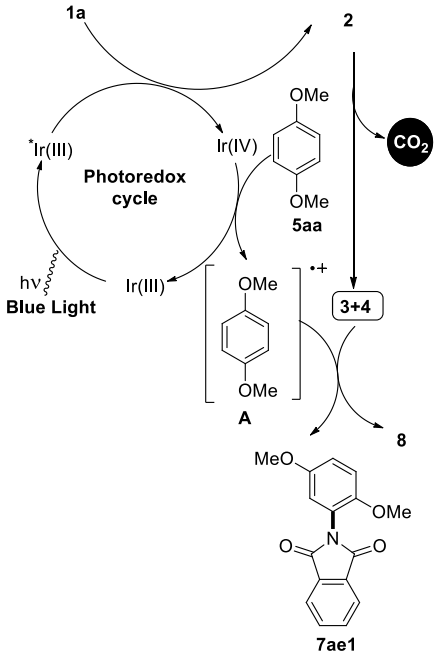


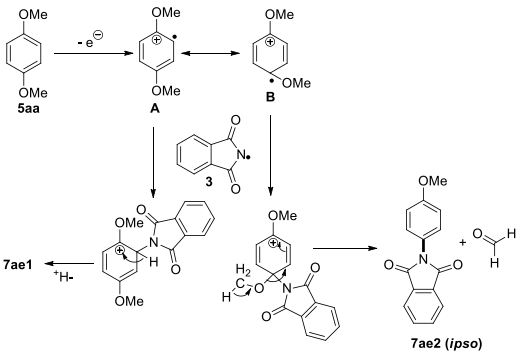
Eosin Y
$E_{1/2}(EY^{\cdot+}/{}^3EY^*)$ =-1.11 V vs SCE
$E_{1/2}({}^3EY^*/EY^{\cdot-})$ = +0.83 V vs SCE
λ_{max} = 539 nm



Rose Bengal
$E_{1/2}(RB^{\cdot+}/{}^3RB^*)$ =-0.68 V vs SCE
$E_{1/2}({}^3RB^*/RB^{\cdot-})$ = +0.83 V vs SCE
λ_{max} = 548 nm

9. **Ipso mechanism:**

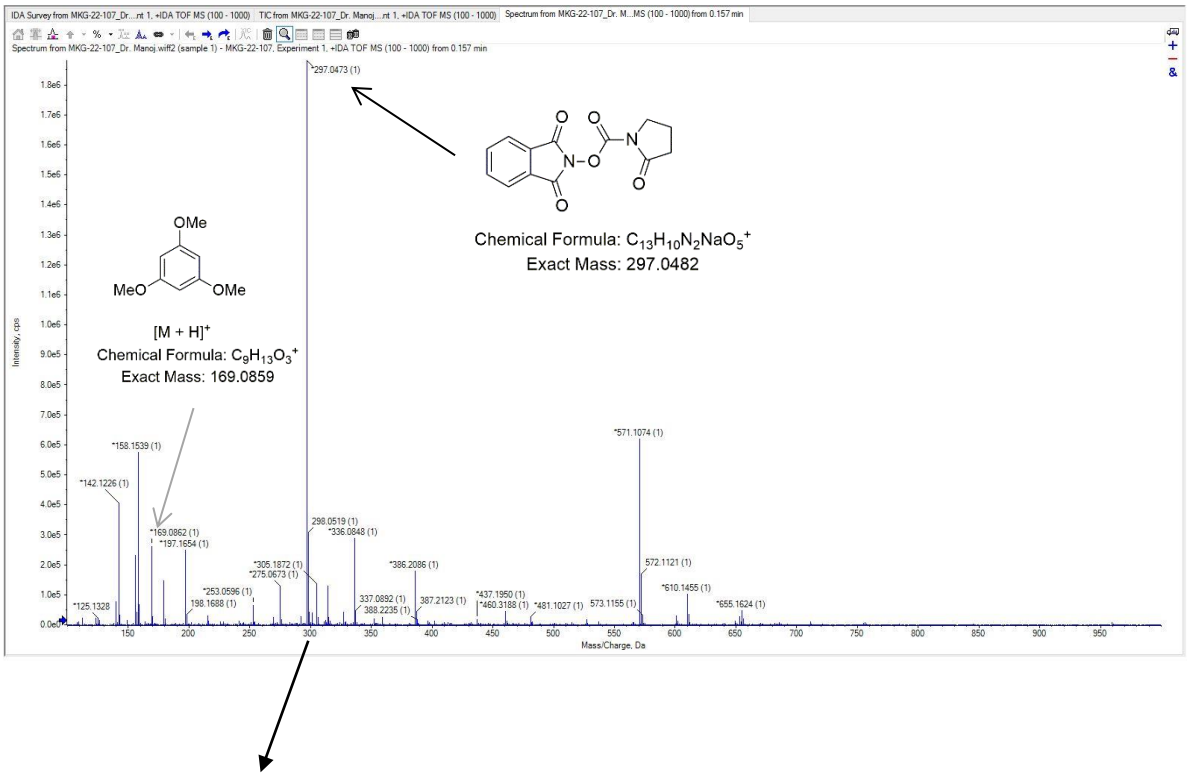


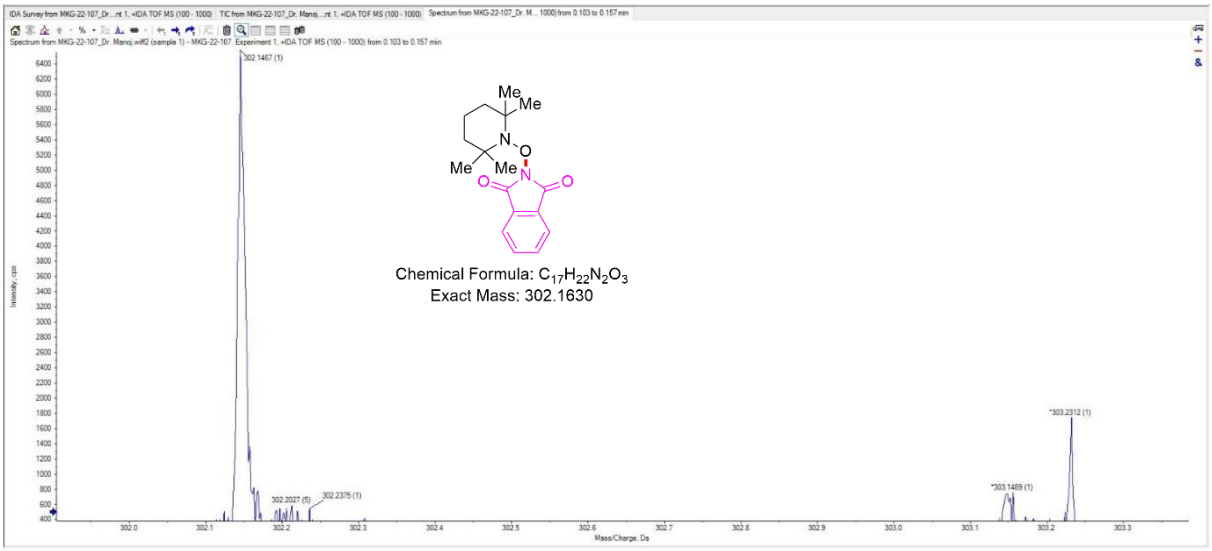


9. **References:**

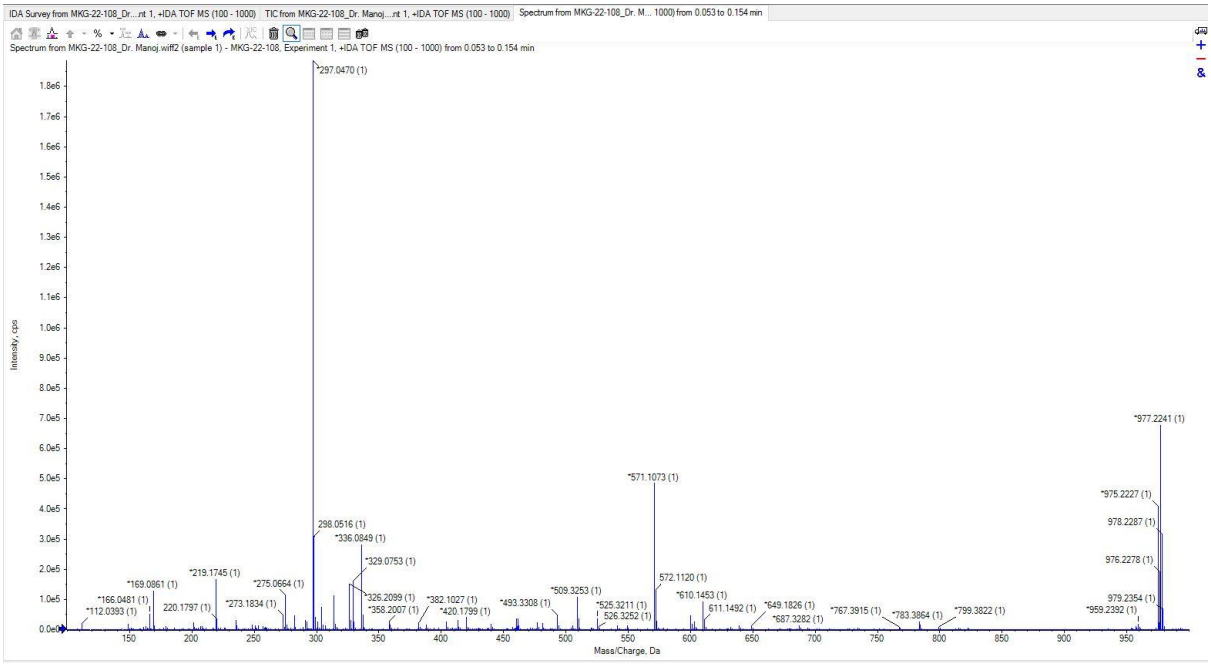
1. M.S. Lowry, J.I. Gpldsmith, J. D. Slinker, R. Rohl, R. A. Pascal, G. G. Malliaras and S. Bernhard, *Chem. Mater.*, 2005, **17**, 5712.
2. G. Duret, R. Quinlan, P. Bisseret and N. Blanchard, *Chem. Sci*., 2015, **6**, 5366.

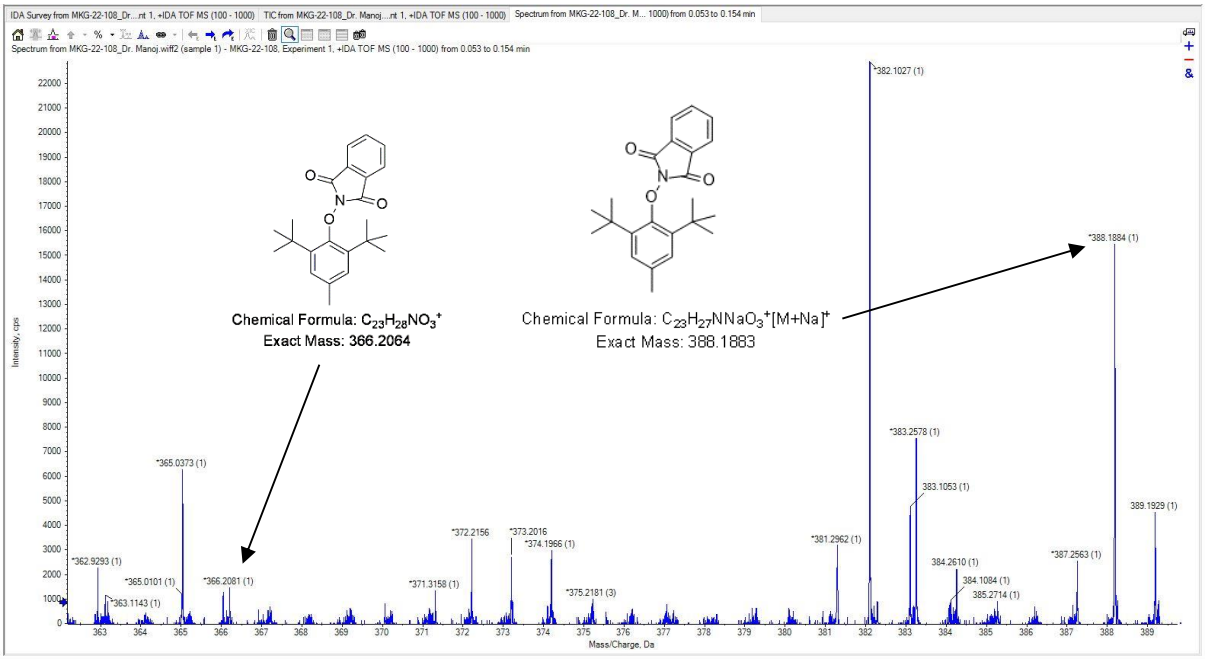
6. HRMS of TEMPO adduct (From Reaction mixture) -



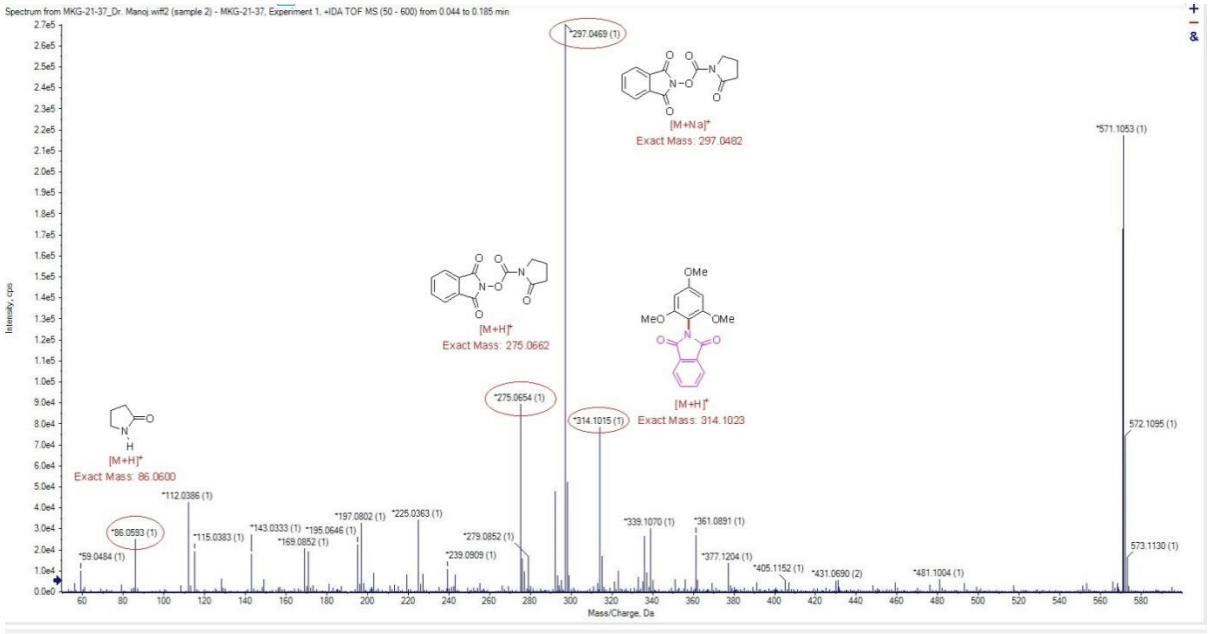


7. HRMS of BHT adduct (From reaction mixture)–

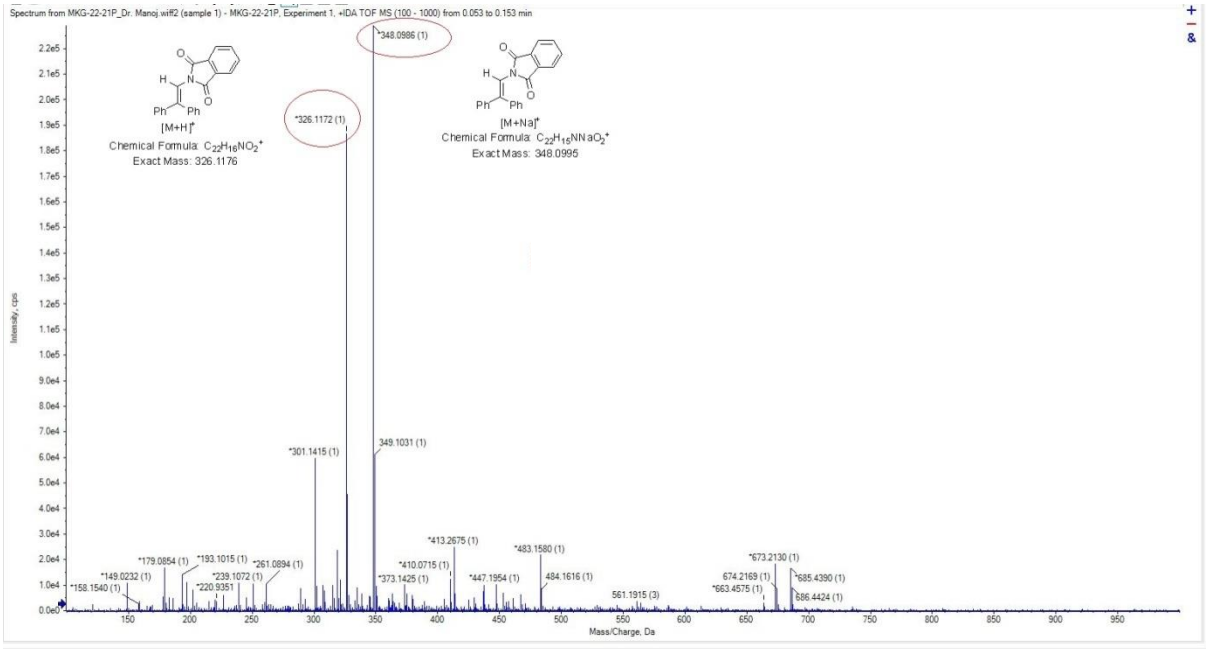


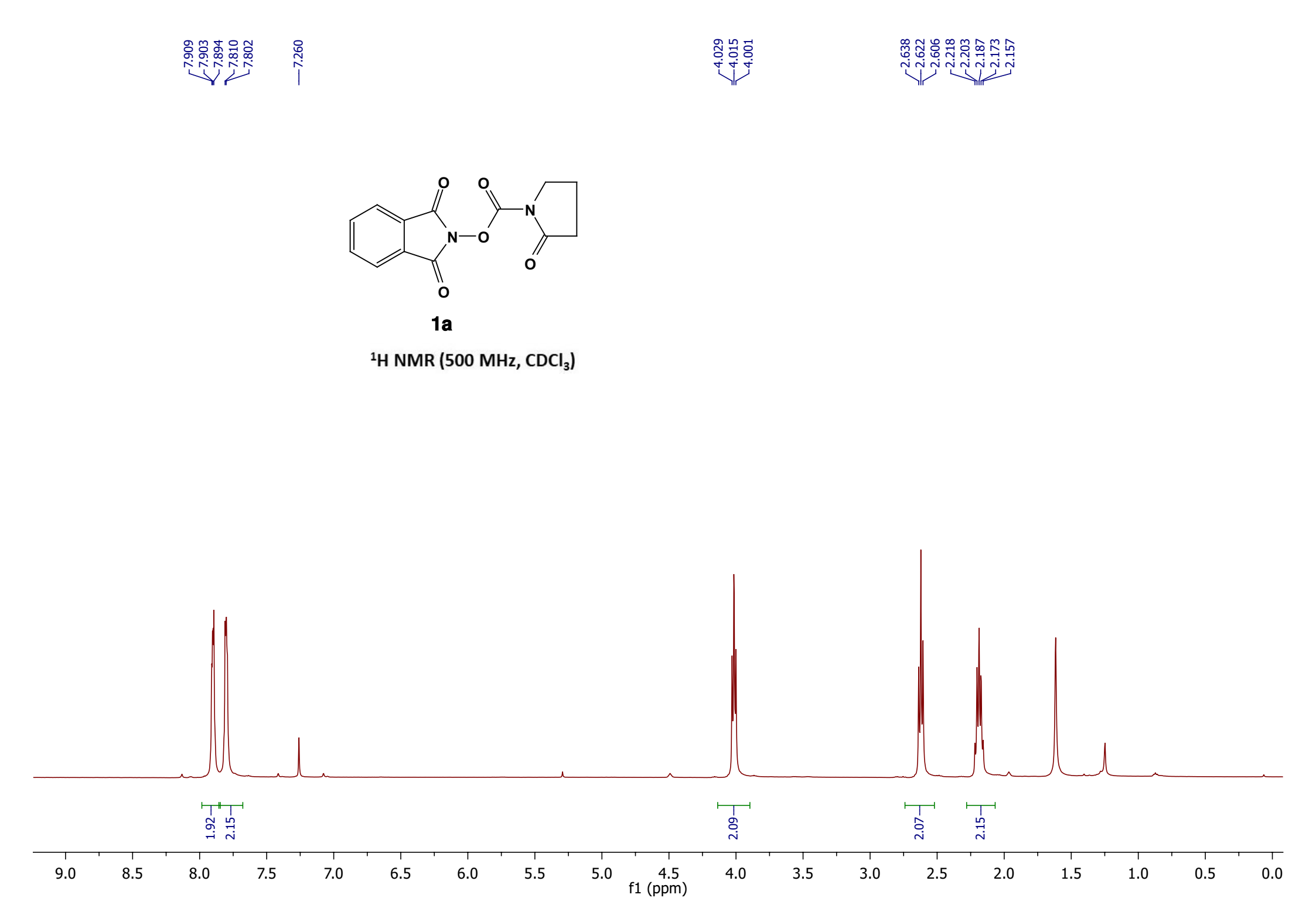


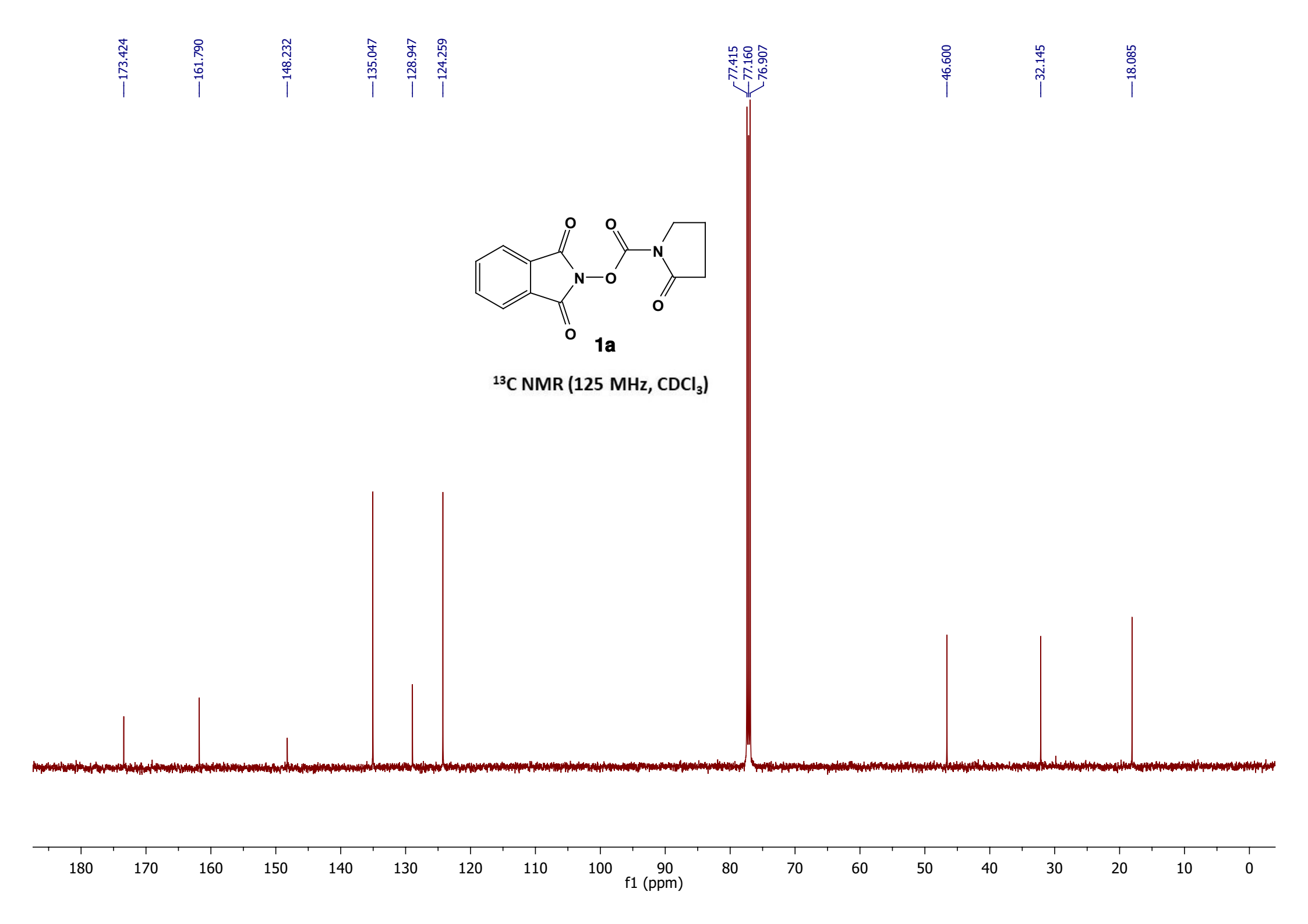
Mechanistic Support: HRMS data of the Reaction Mixture after 12h from reaction time

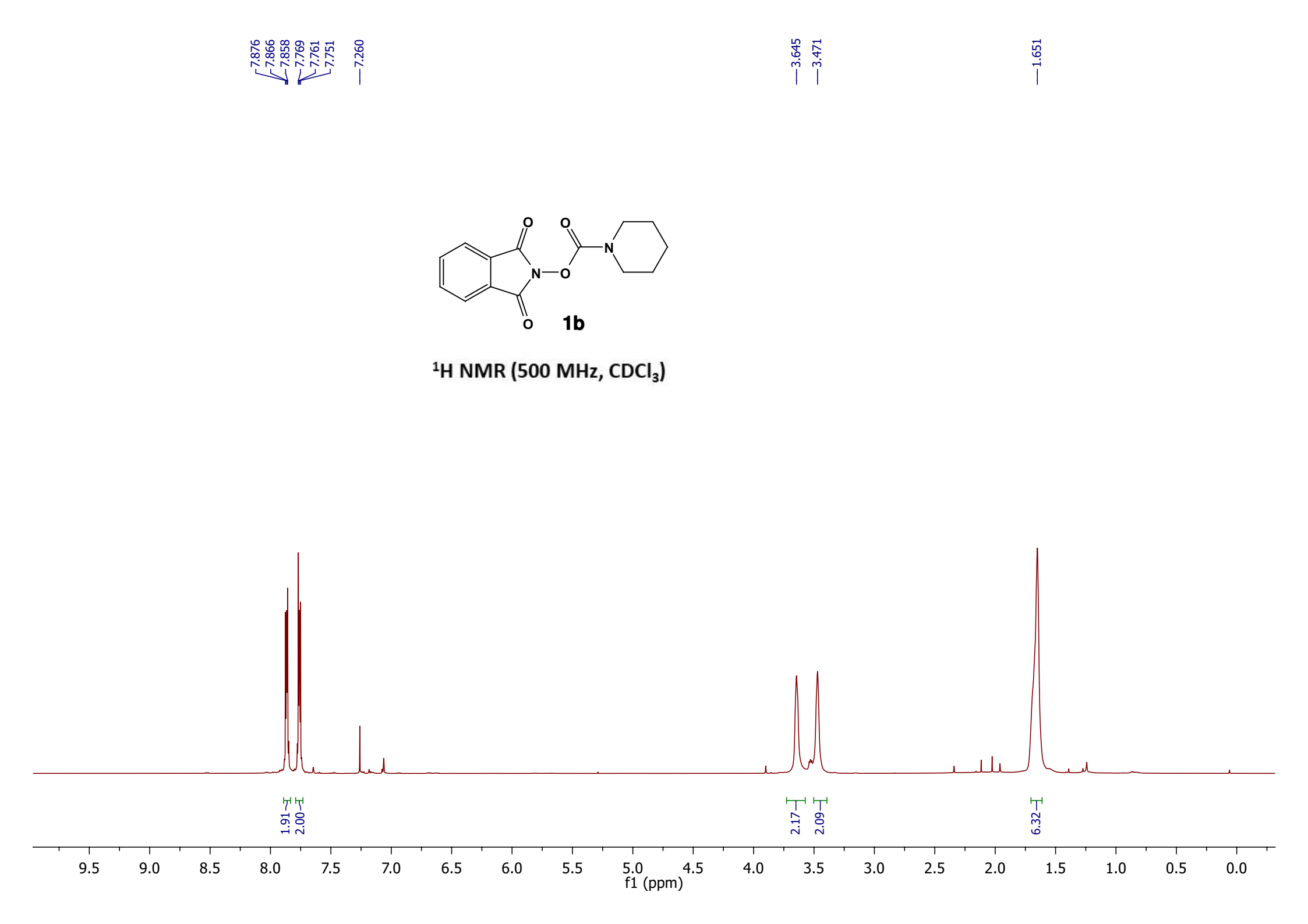


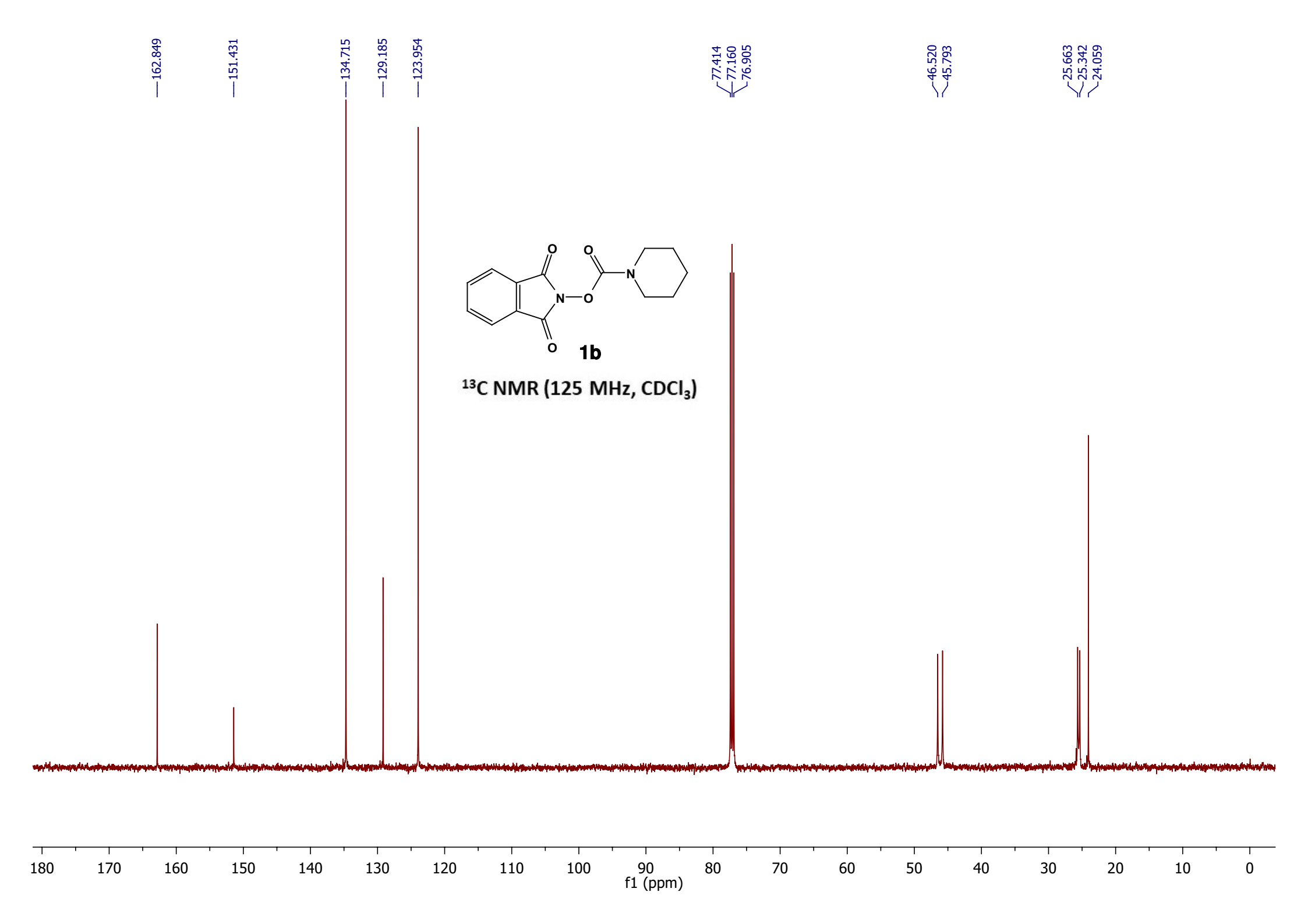
Controlled Experiment: HRMS data of the radical trapping experiment

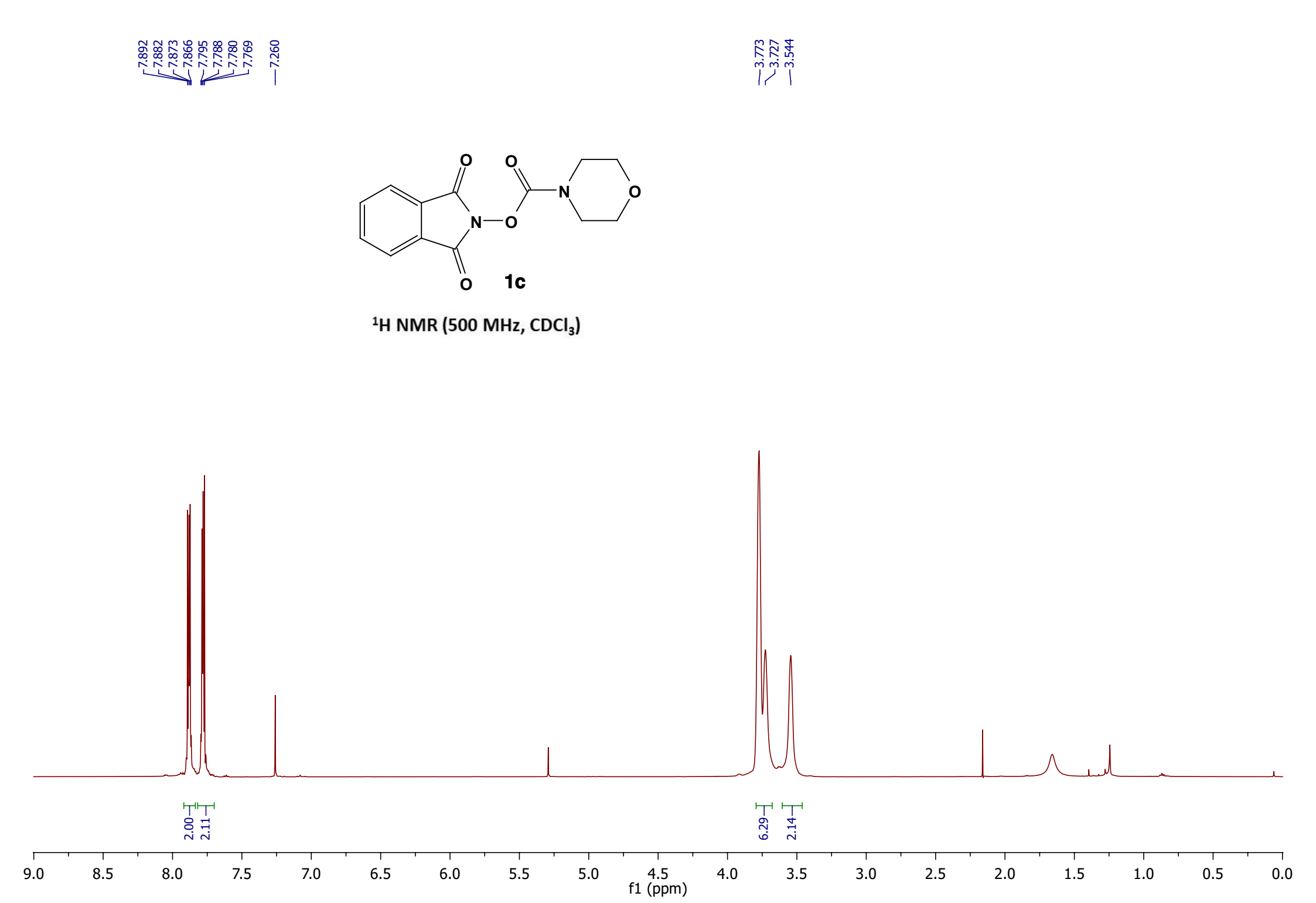


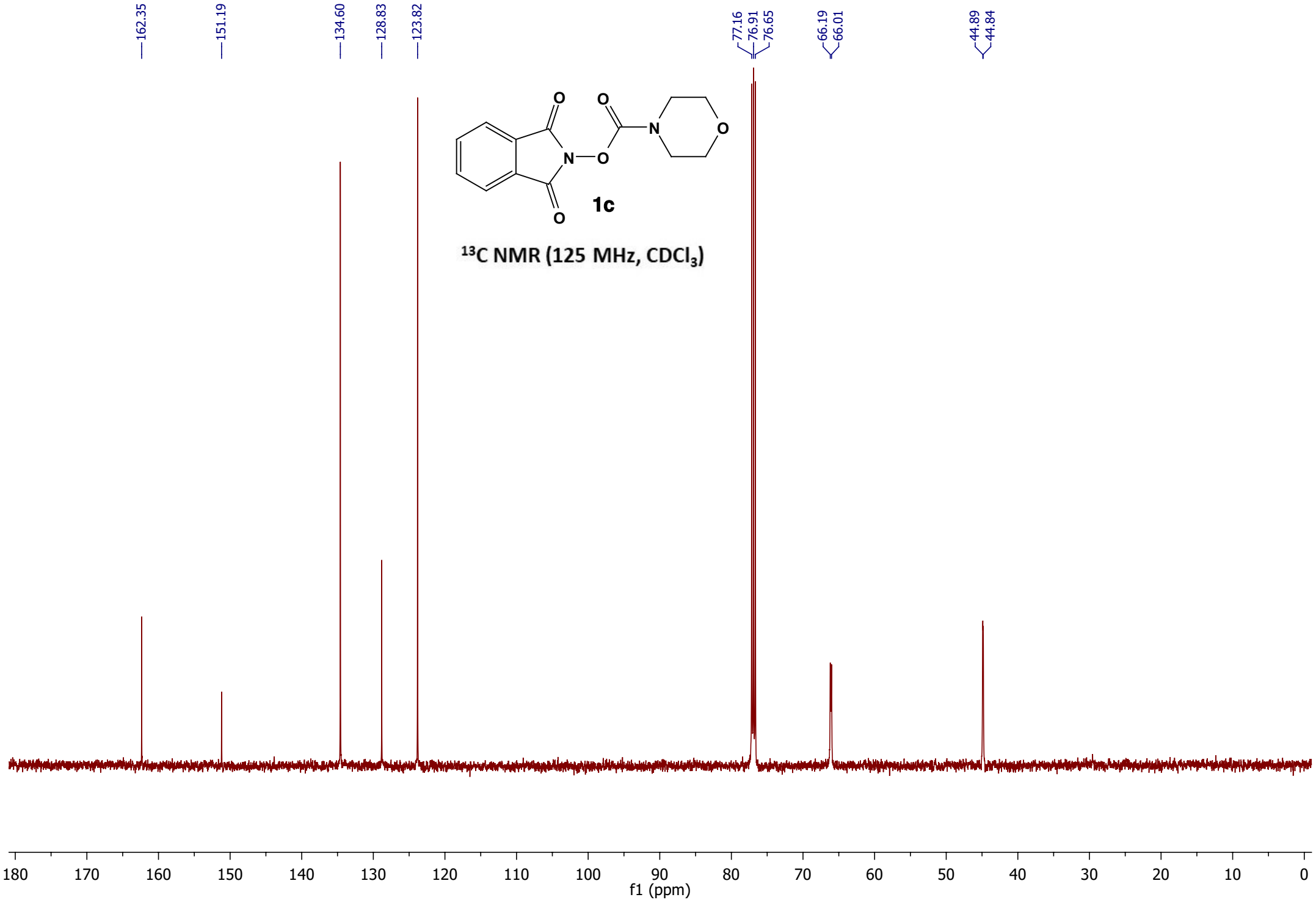

7.909
7.903
7.894
7.810
7.802
7.260
4.029
4.015
4.001
2.638
2.622
2.606
2.218
2.203
2.187
2.173
2.157
O
O
N
O
N
O
O
O
1a
1H NMR (500 MHz, CDCl3)
1.92
2.15
2.09
2.07
2.15
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

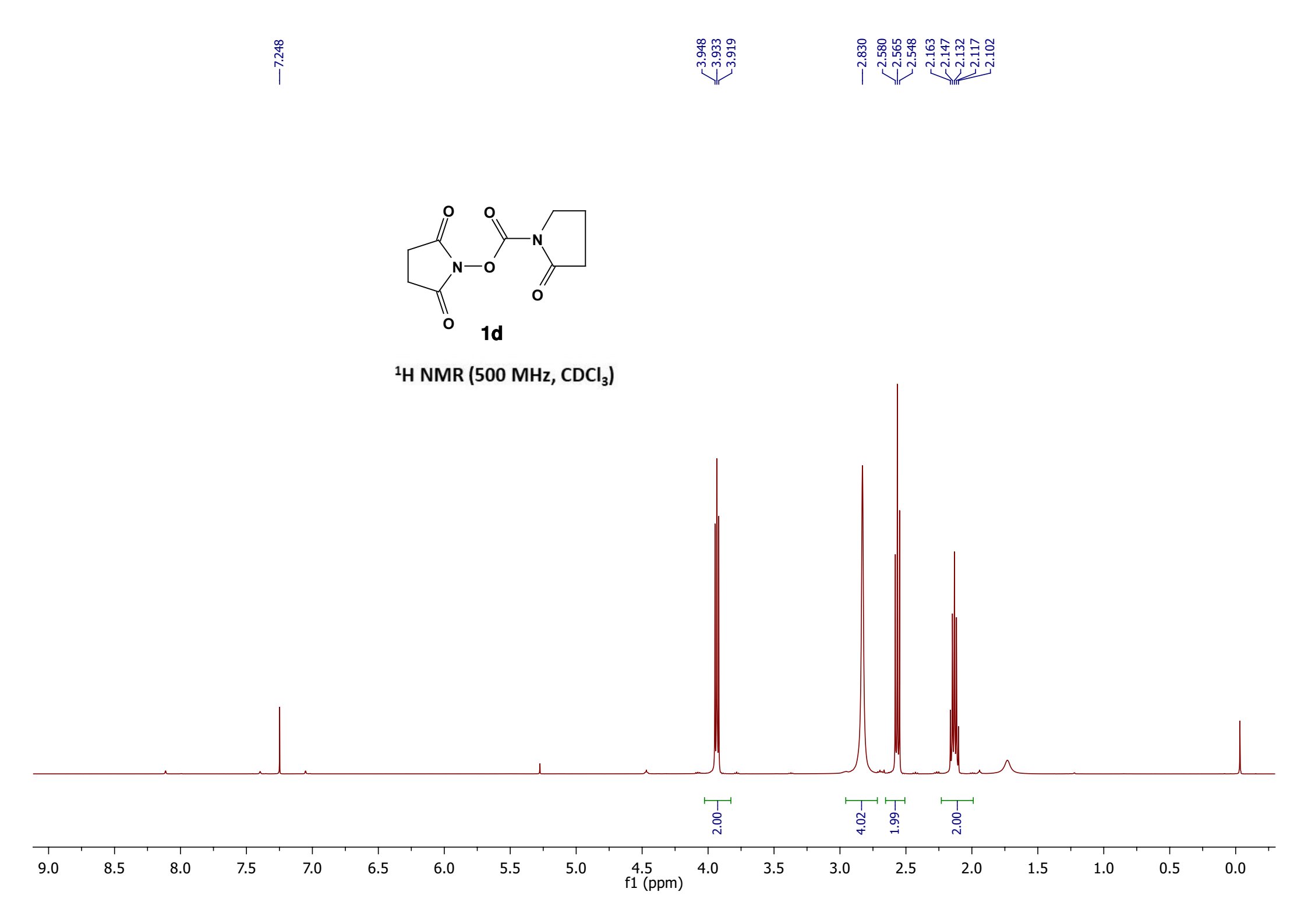

173.424
161.790
148.232
135.047
128.947
124.259
77.415
77.160
76.907
46.600
32.145
18.085
O
O
N
O
N
O
O
O
1a
^{13}C NMR (125 MHz, $CDCl_3$)
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

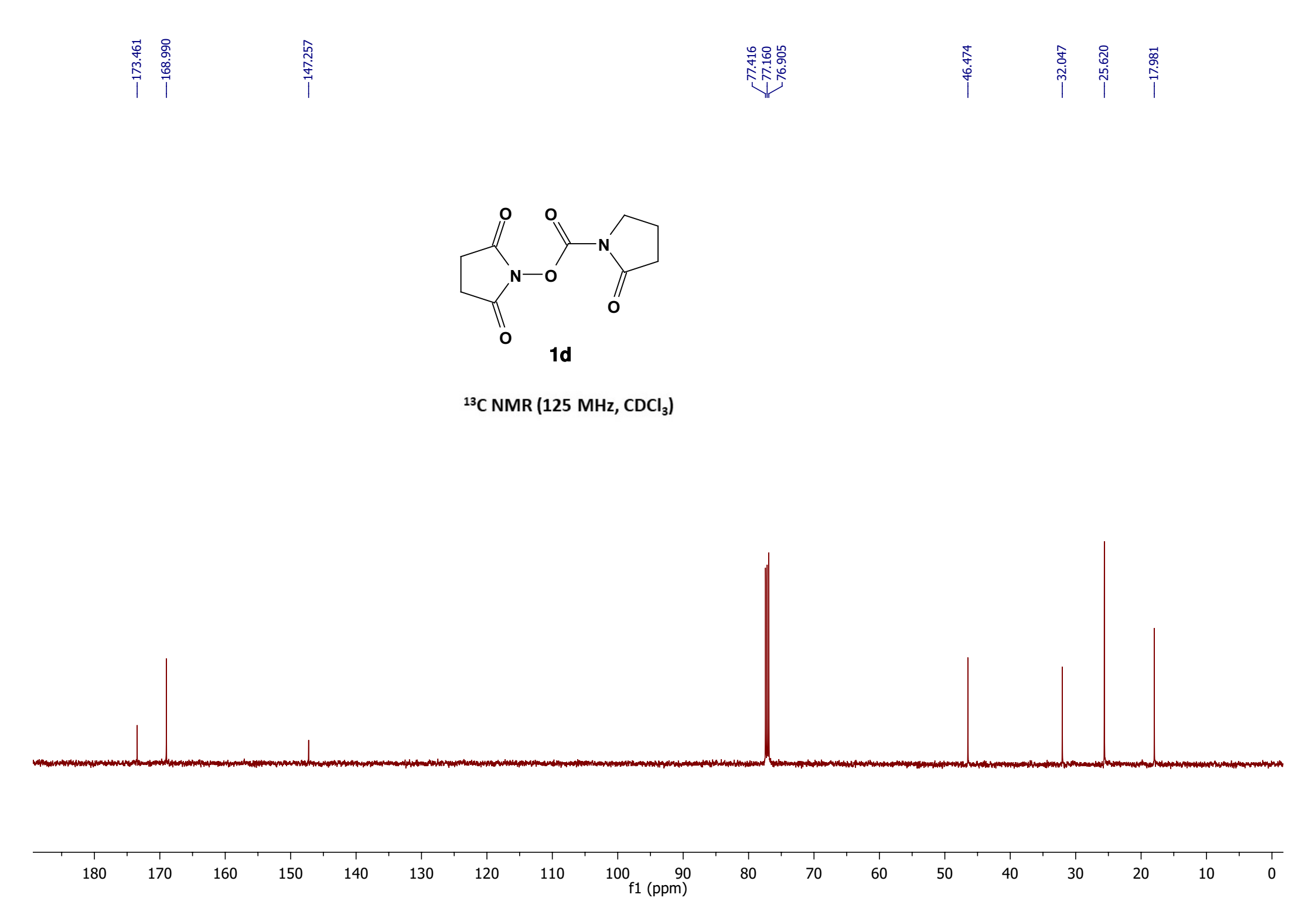

7.876
7.866
7.858
7.769
7.761
7.751
7.260
3.645
3.471
1.651
O
O
N
O
N
O
O
1b
1H NMR (500 MHz, CDCl3)
1.91
2.00
2.17
2.09
6.32
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

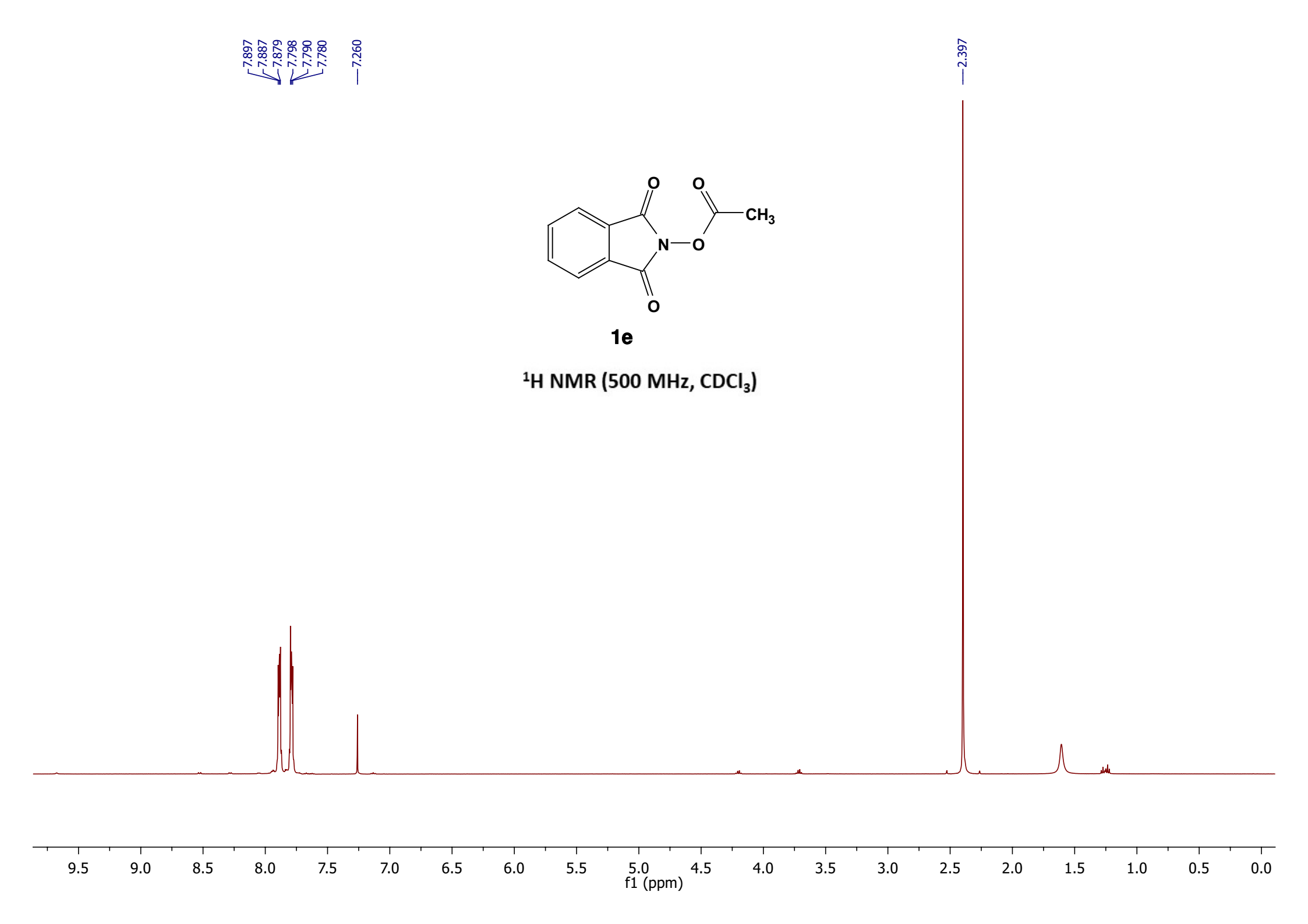

162.849
151.431
134.715
129.185
123.954
77.414
77.160
76.905
46.520
45.793
25.663
25.342
24.059
O
O
N
O
N
O
O
1b
13C NMR (125 MHz, CDCl3)
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

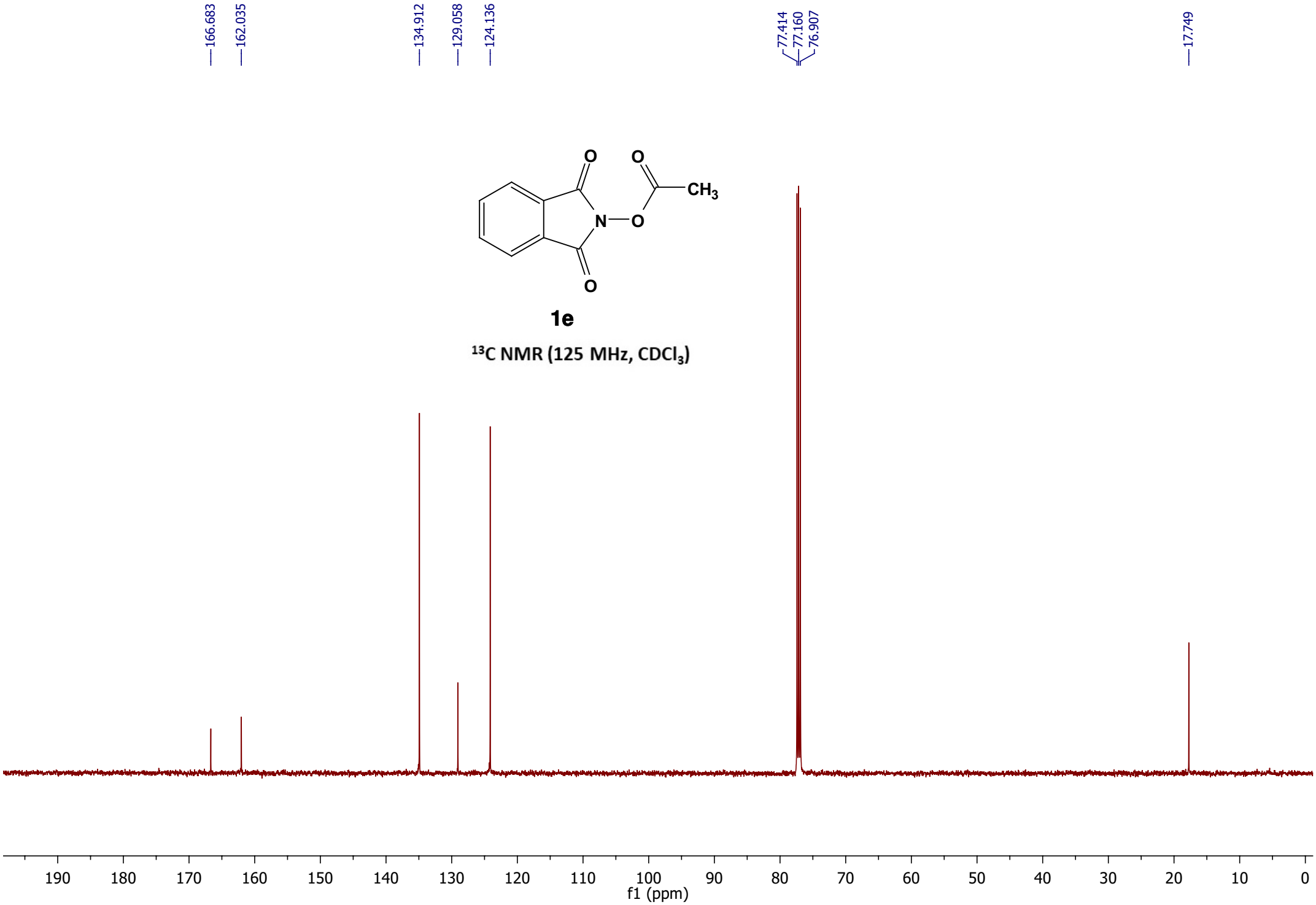

7.892
7.882
7.873
7.866
7.795
7.788
7.780
7.769
7.260
3.773
3.727
3.544
O
O
N
O
N
O
O
O
1c
^{1}H NMR (500 MHz, $CDCl_3$)
2.00
2.11
6.29
2.14
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

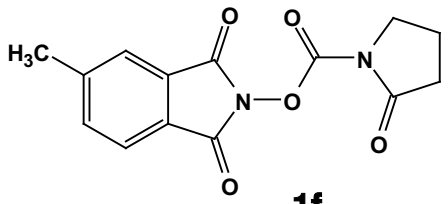

162.35
151.19
134.60
128.83
123.82
77.16
76.91
76.65
66.19
66.01
44.89
44.84
O
O
N
O
N
O
O
1c
13C NMR (125 MHz, CDCl3)
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.248
3.948
3.933
3.919
2.830
2.580
2.565
2.548
2.163
2.147
2.132
2.117
2.102
O
O
N
O
N
O
O
1d
¹H NMR (500 MHz, CDCl₃)
2.00
4.02
1.99
2.00
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
1.0
0.5
0.0
f1 (ppm)


173.461
168.990
147.257
77.416
77.160
76.905
46.474
32.047
25.620
17.981
O
O
N
O
N
O
O
O
1d
^{13}C NMR (125 MHz, $CDCl_3$)
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

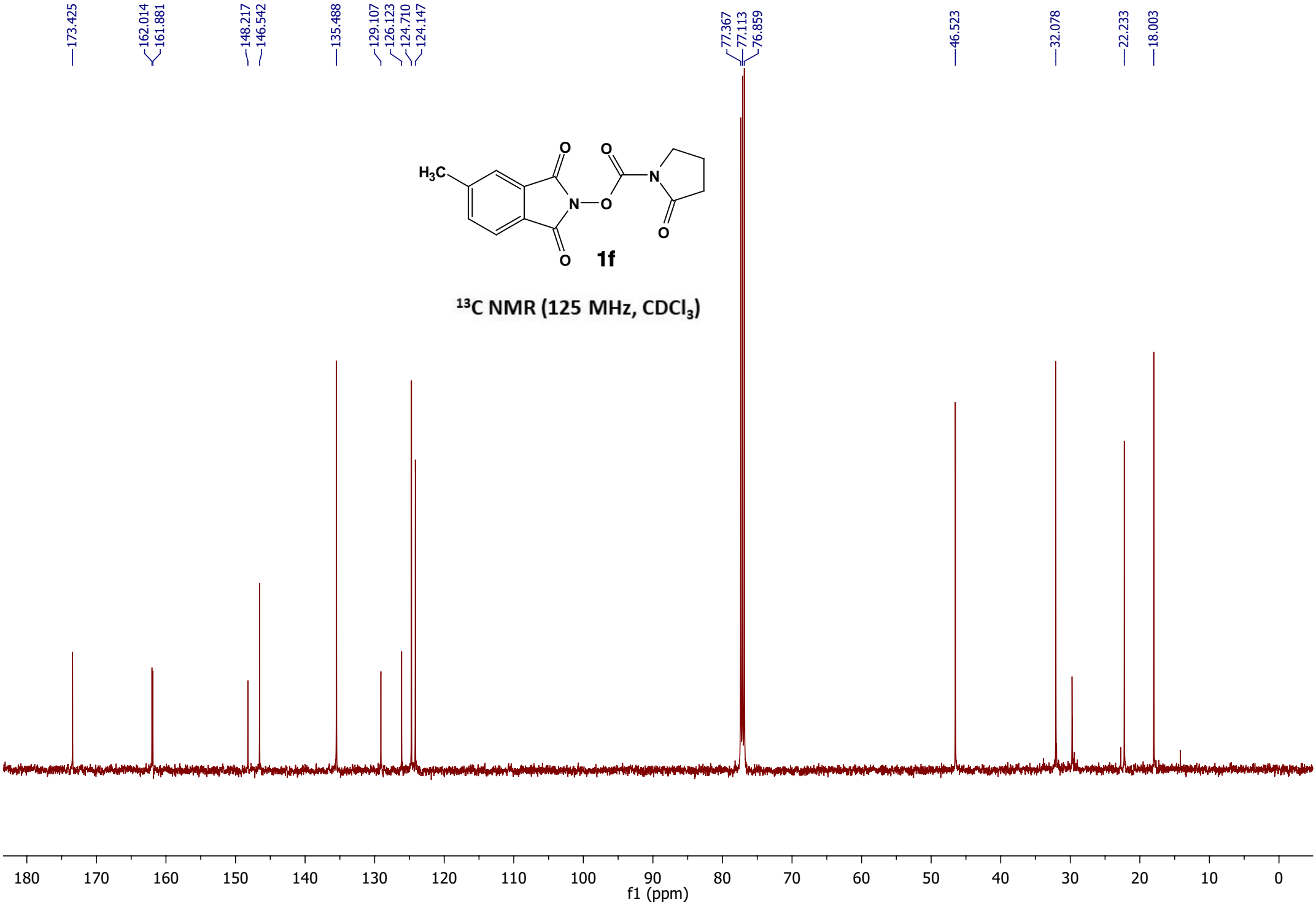

7.897
7.887
7.879
7.798
7.790
7.780
7.260
2.397
O
O
CH3
N
O
O
1e
1H NMR (500 MHz, CDCl3)
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

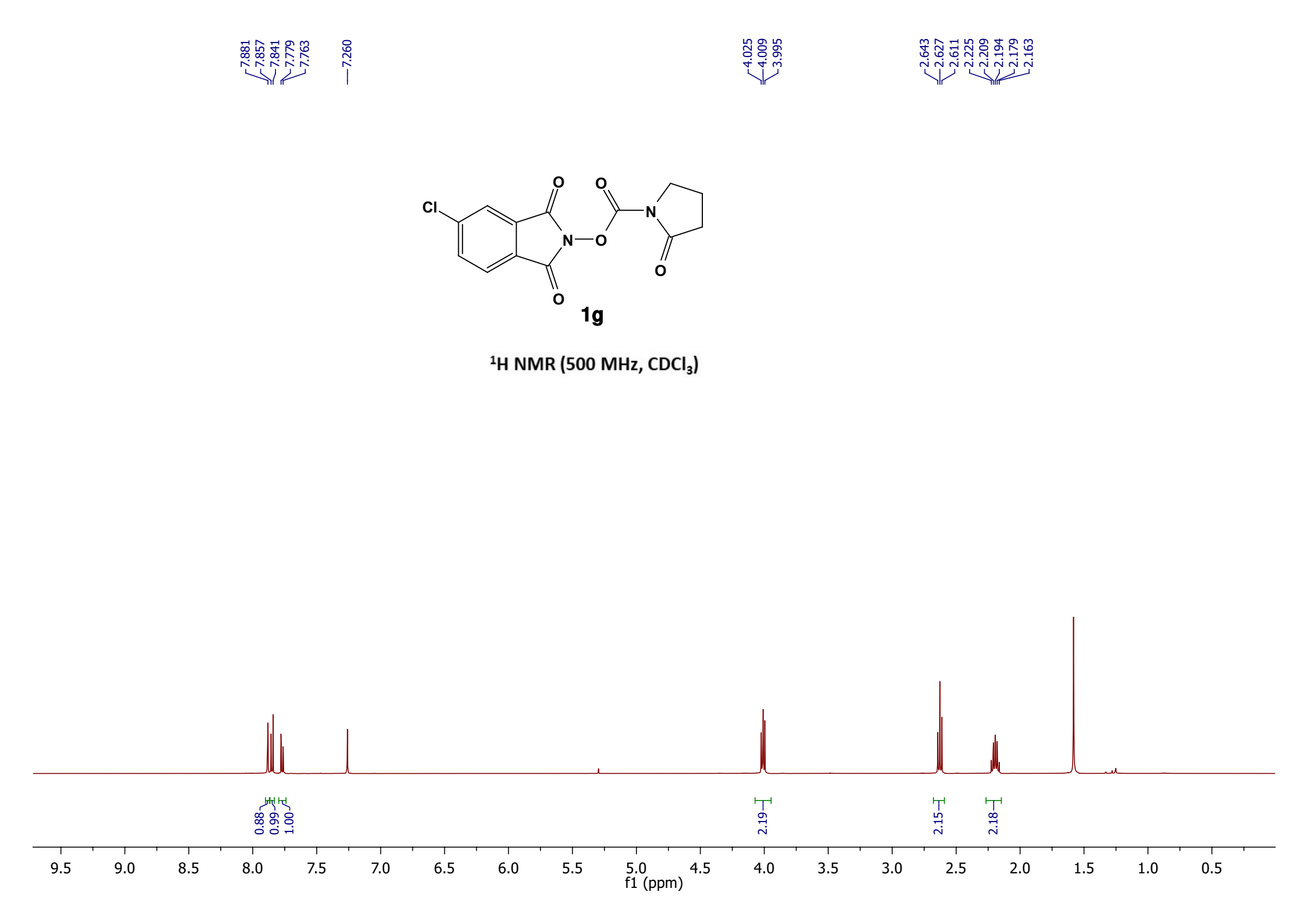

166.683
162.035
134.912
129.058
124.136
77.414
77.160
76.907
17.749
O
O
CH3
N
O
O
1e
13C NMR (125 MHz, CDCl3)
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

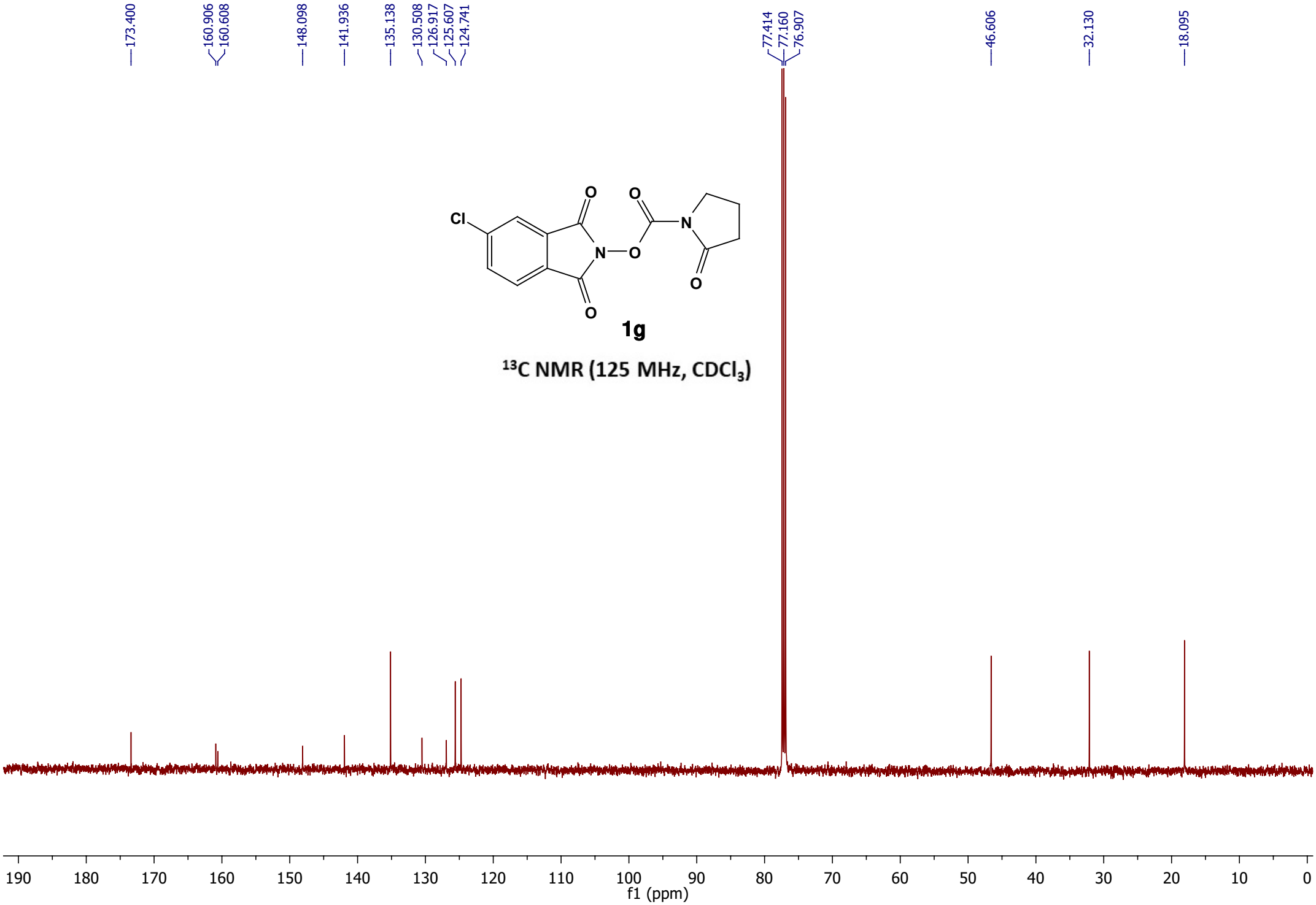


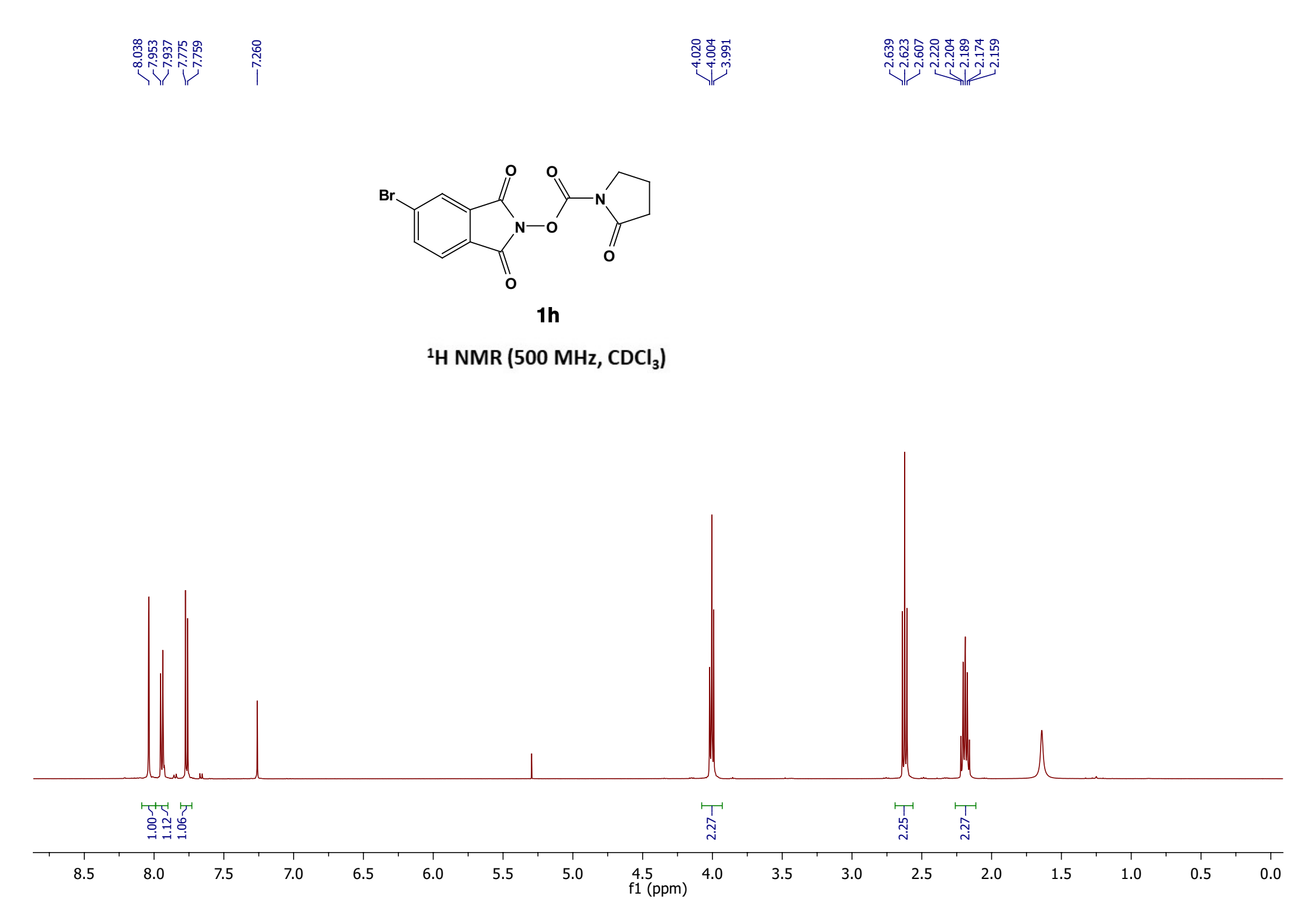
1f

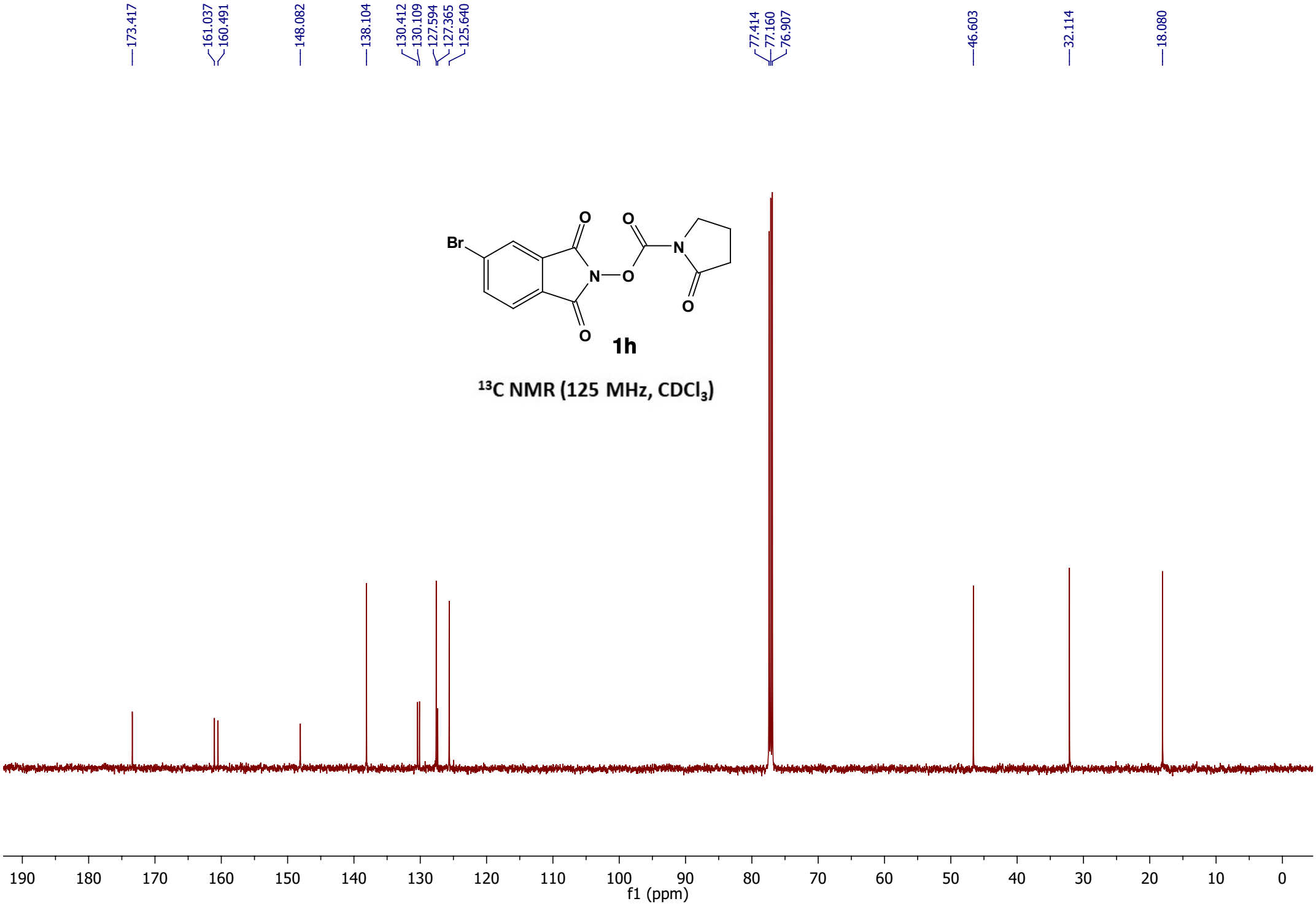
^{1}H NMR (500 MHz, $CDCl_3$)

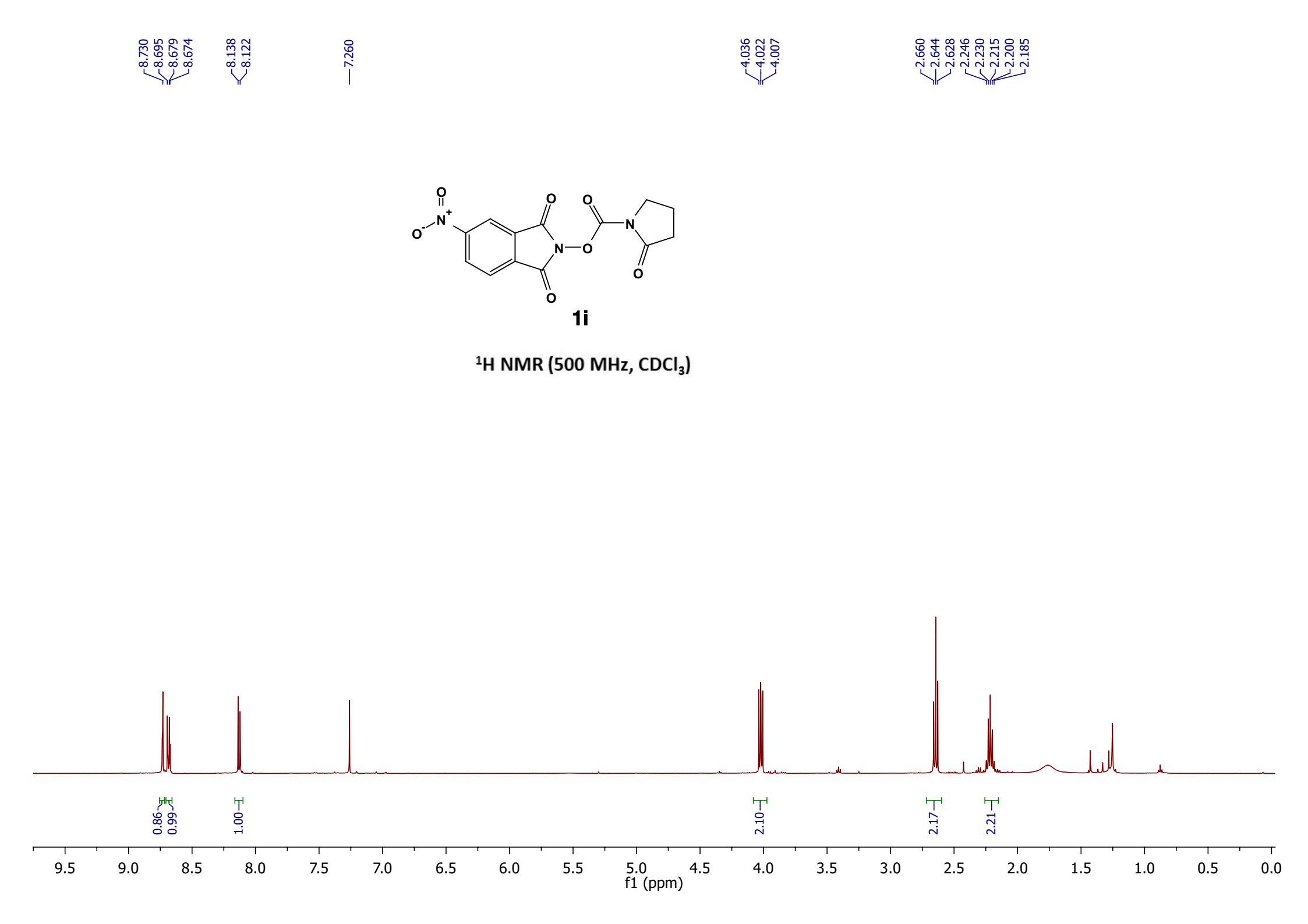

173.425
162.014
161.881
148.217
146.542
135.488
129.107
126.123
124.710
124.147
77.367
77.113
76.859
46.523
32.078
22.233
18.003
H3C
O
O
N
O
N
O
O
O
1f
13C NMR (125 MHz, CDCl3)
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.881
7.857
7.841
7.779
7.763
7.260
4.025
4.009
3.995
2.643
2.627
2.611
2.225
2.209
2.194
2.179
2.163
Cl
O
O
N
O
N
O
O
O
1g
^{1}H NMR (500 MHz, $CDCl_3$)
0.88
0.99
1.00
2.19
2.15
2.18
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)

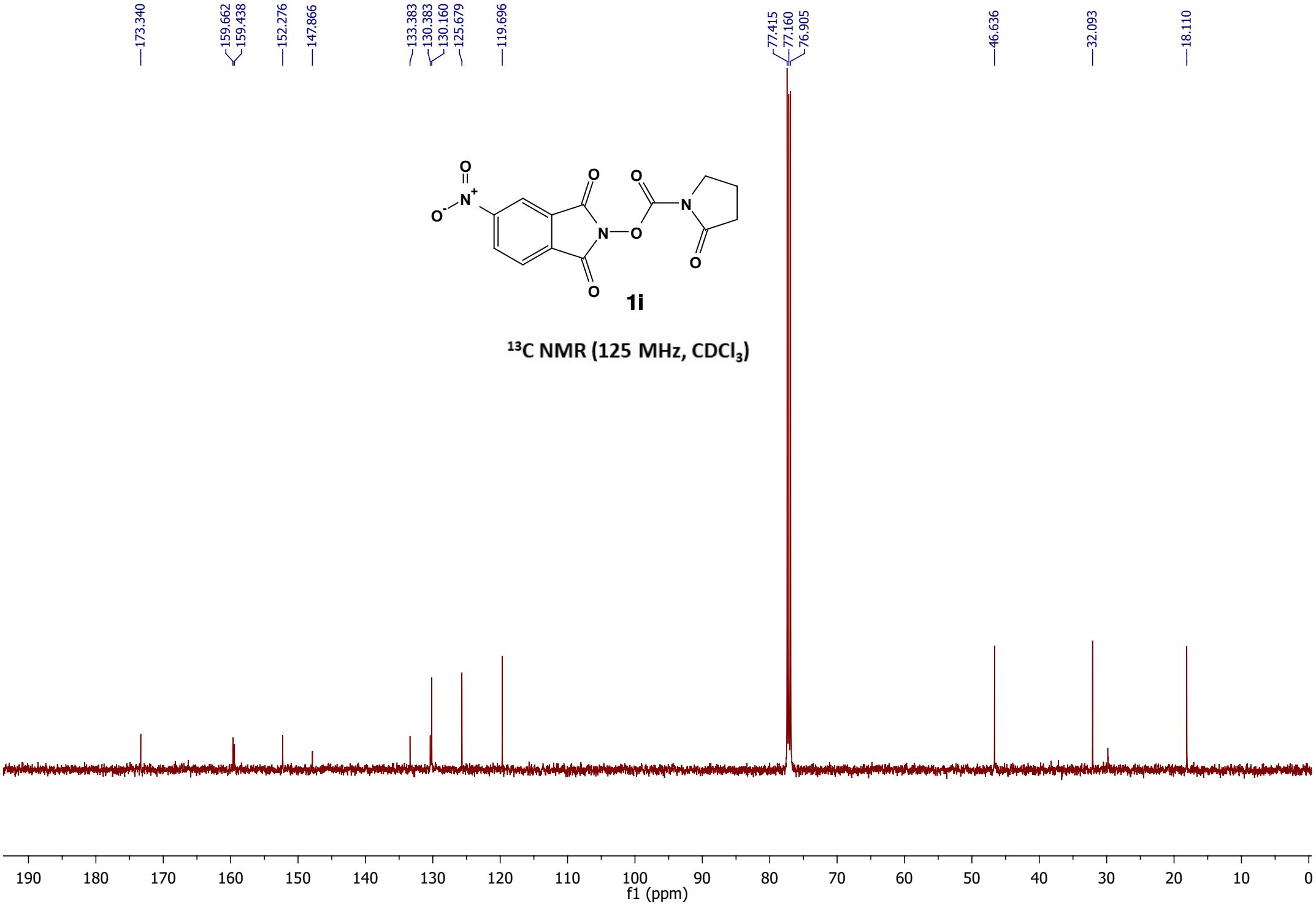

173.400
160.906
160.608
148.098
141.936
135.138
130.508
126.917
125.607
124.741
77.414
77.160
76.907
46.606
32.130
18.095
Cl
O
O
N
O
N
O
O
O
1g
13C NMR (125 MHz, CDCl3)
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


8.038
7.953
7.937
7.775
7.759
7.260
4.020
4.004
3.991
2.639
2.623
2.607
2.220
2.204
2.189
2.174
2.159
Br
O
O
N
O
N
O
O
O
1h
1H NMR (500 MHz, CDCl3)
1.00
1.12
1.06
2.27
2.25
2.27
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)

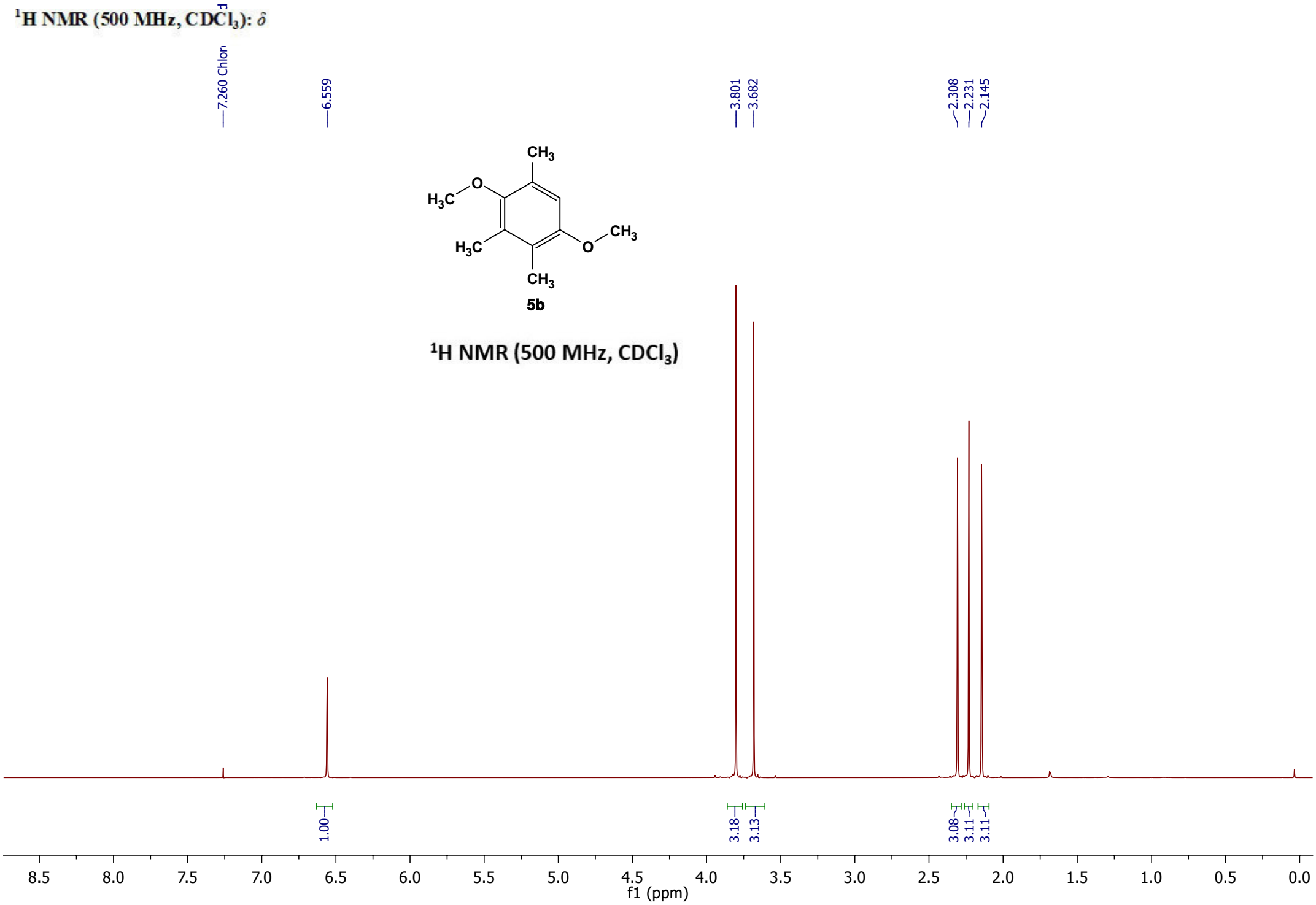

173.417
161.037
160.491
148.082
138.104
130.412
130.109
127.594
127.365
125.640
77.414
77.160
76.907
46.603
32.114
18.080
Br
O
O
N
O
N
O
O
O
1h
13C NMR (125 MHz, CDCl3)
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)



^{1}H NMR (500 MHz, $CDCl_3$)

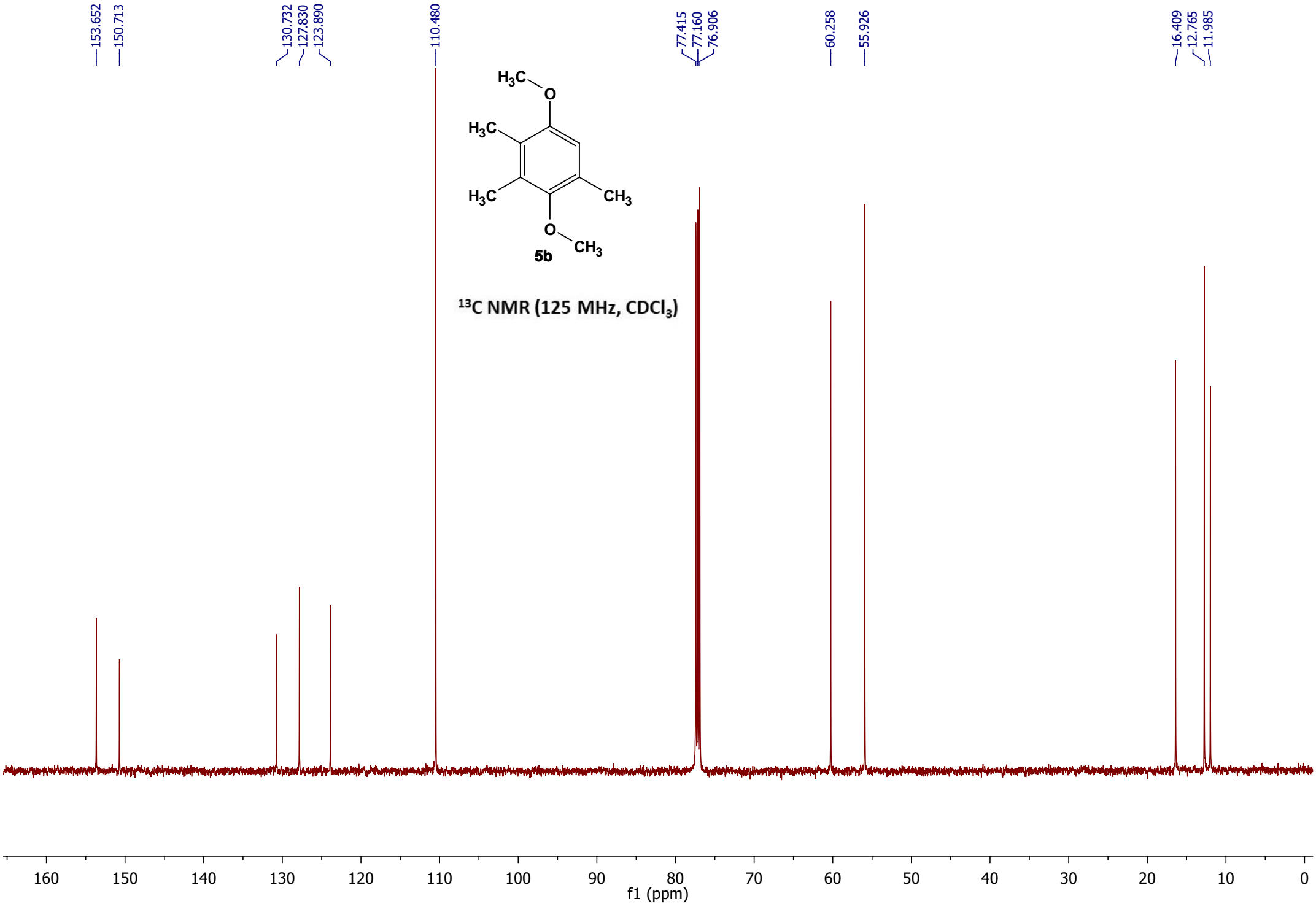

173.340
159.662
159.438
152.276
147.866
133.383
130.383
130.160
125.679
119.696
77.415
77.160
76.905
46.636
32.093
18.110
O
N+
O-
O
N
O
O
O
N
O
O
1i
13C NMR (125 MHz, CDCl3)
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

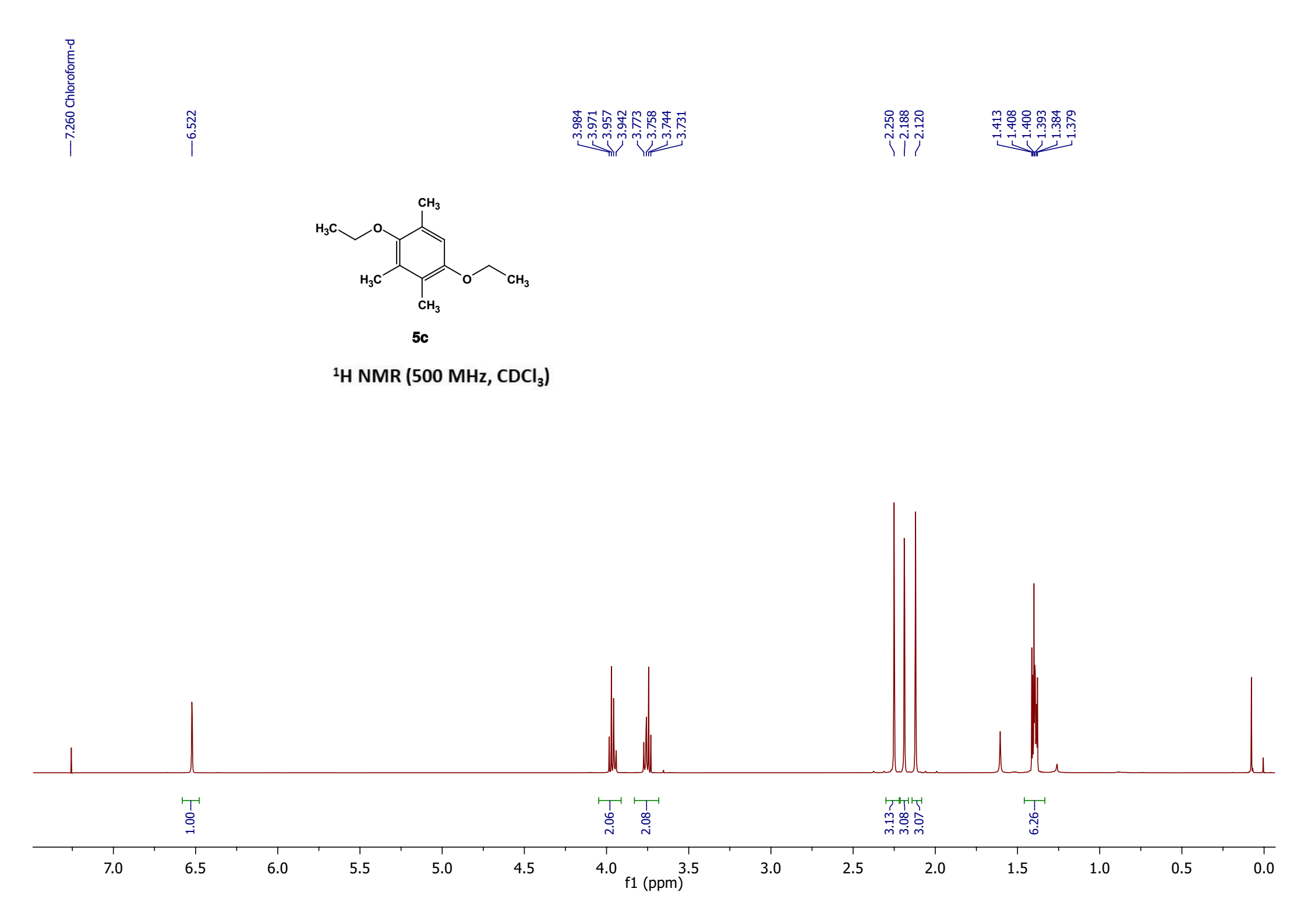
^{1}H NMR (500 MHz, $CDCl_3$): δ

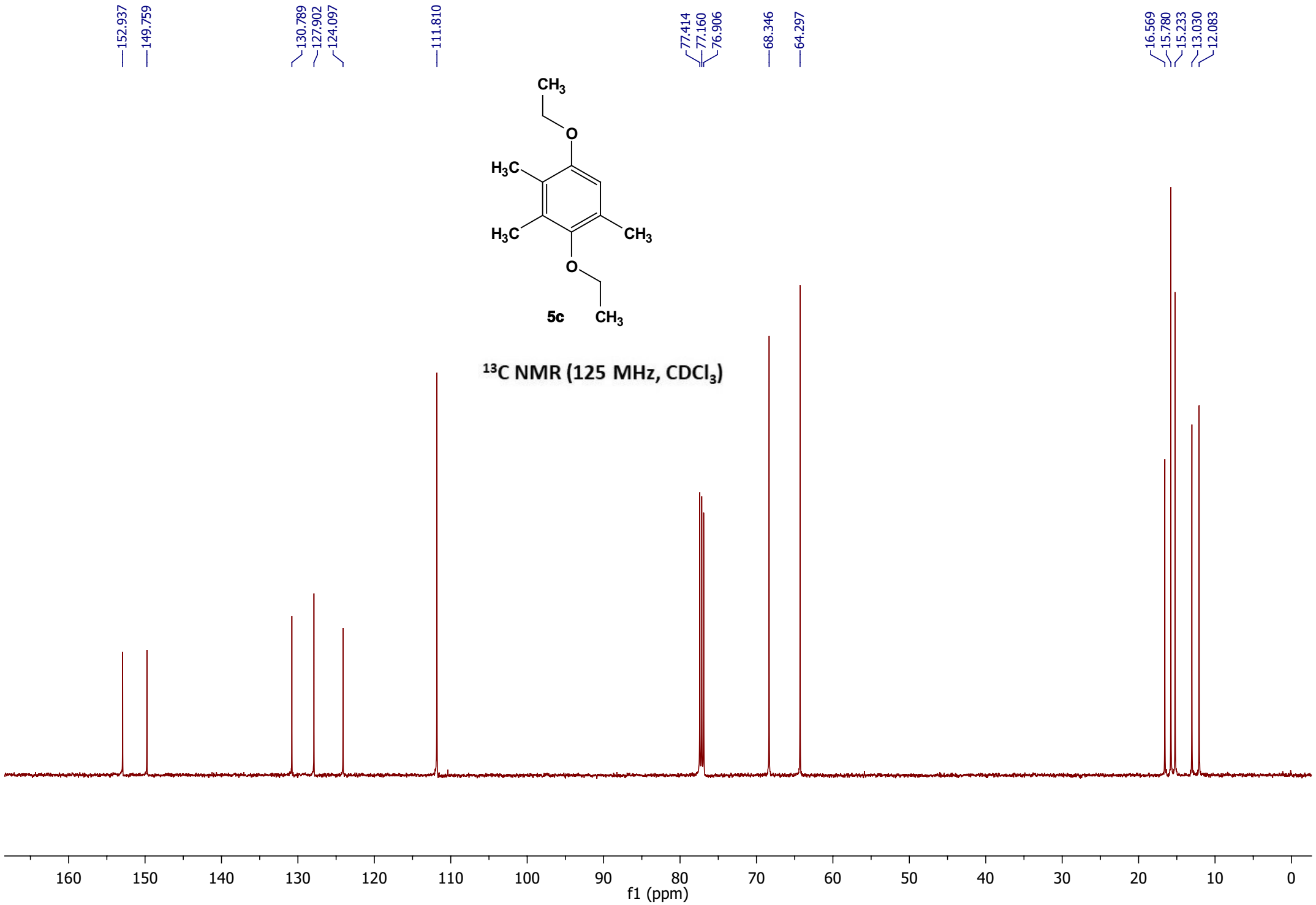


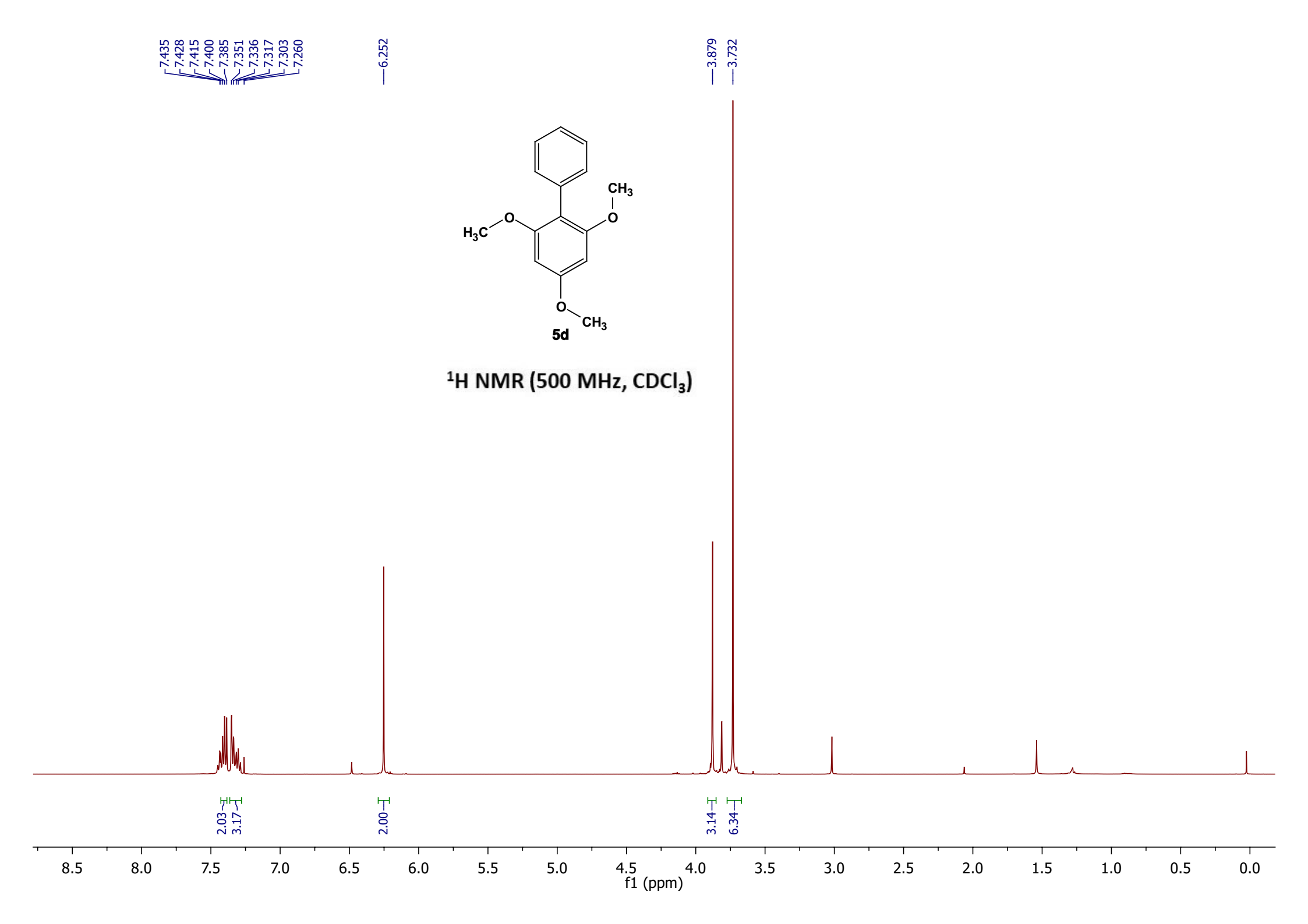
5b

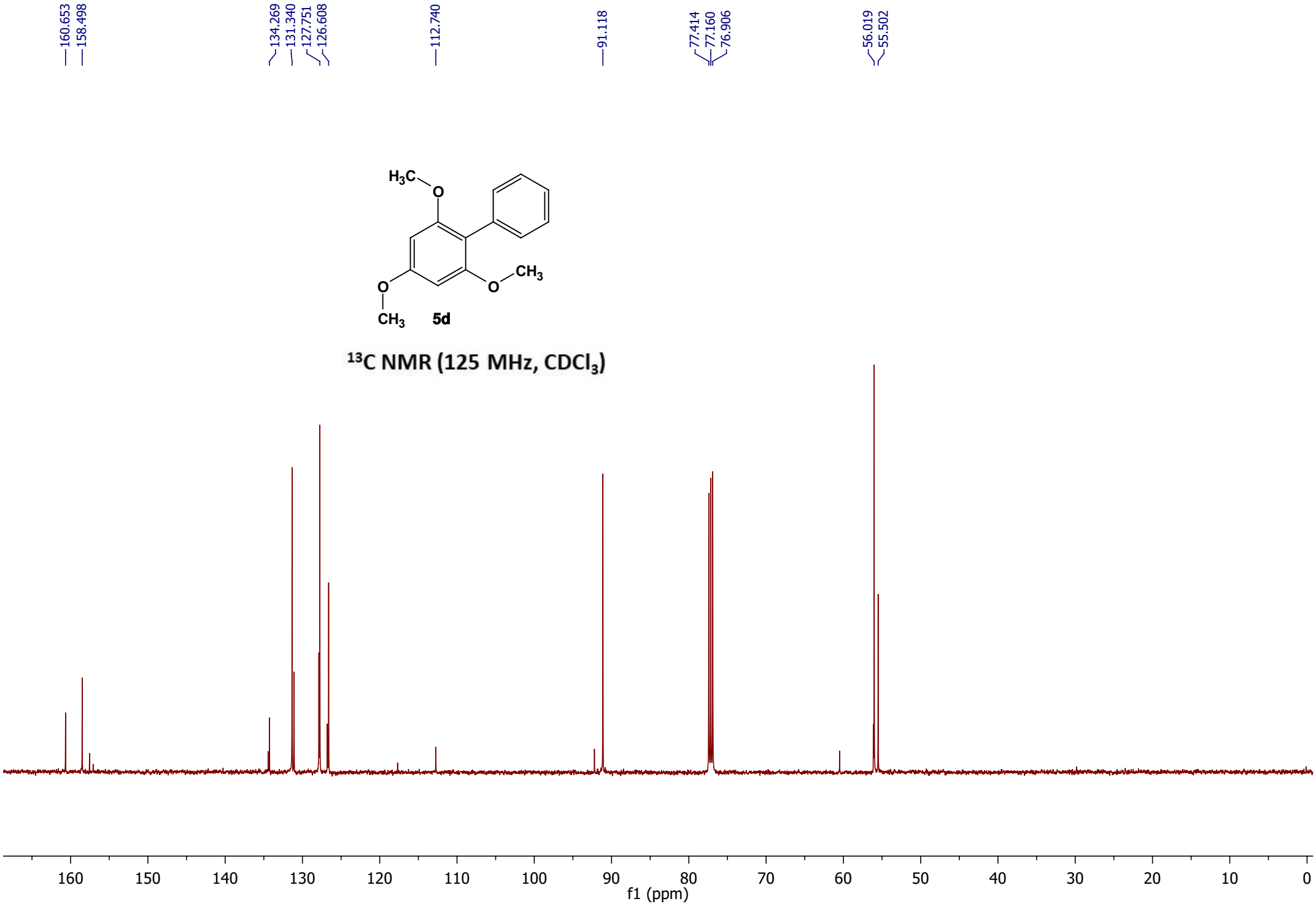
^{1}H NMR (500 MHz, $CDCl_3$)

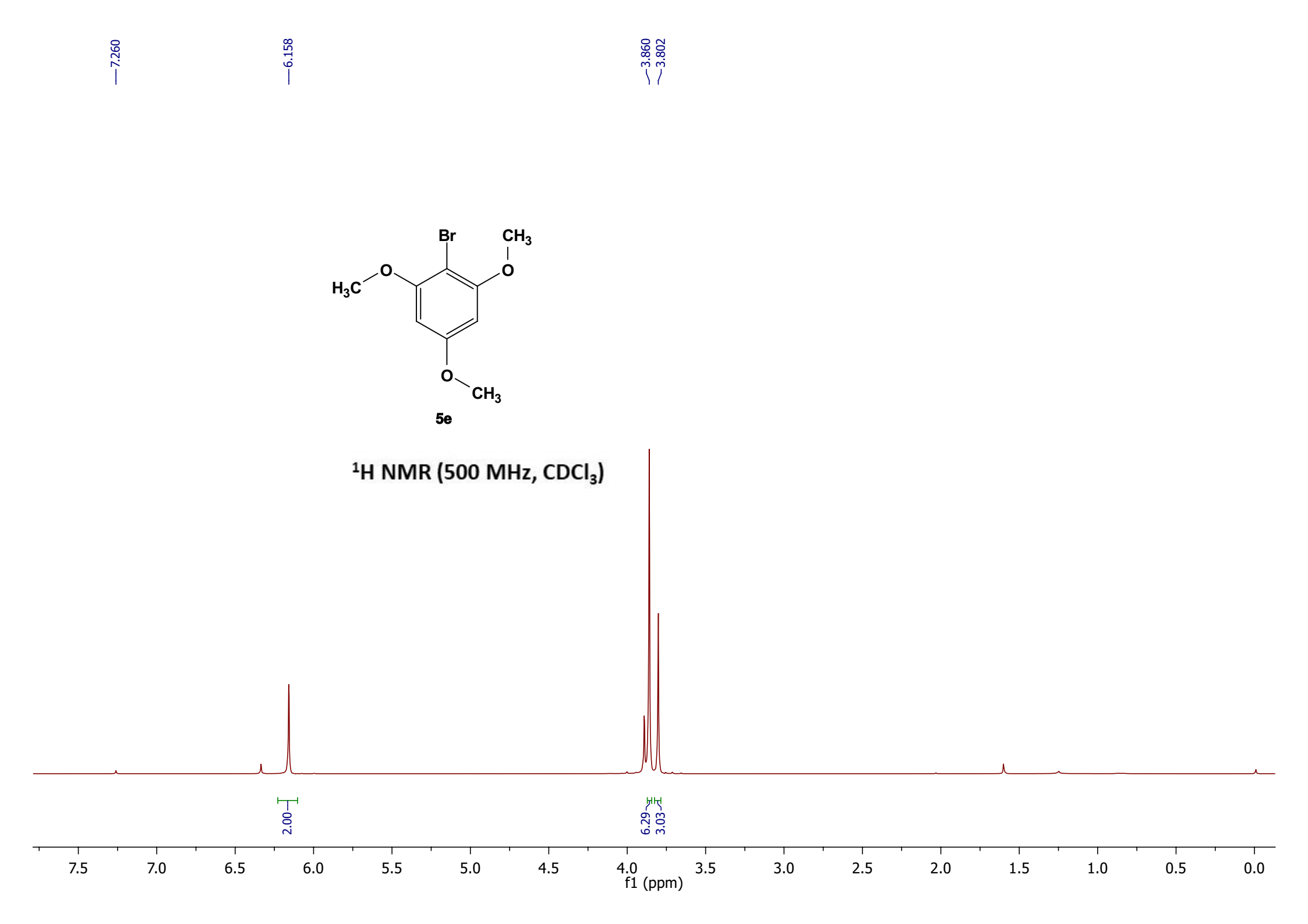

153.652
150.713
130.732
127.830
123.890
110.480
77.415
77.160
76.906
60.258
55.926
16.409
12.765
11.985
H3C
O
H3C
H3C
CH3
O
CH3
5b
13C NMR (125 MHz, CDCl3)
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

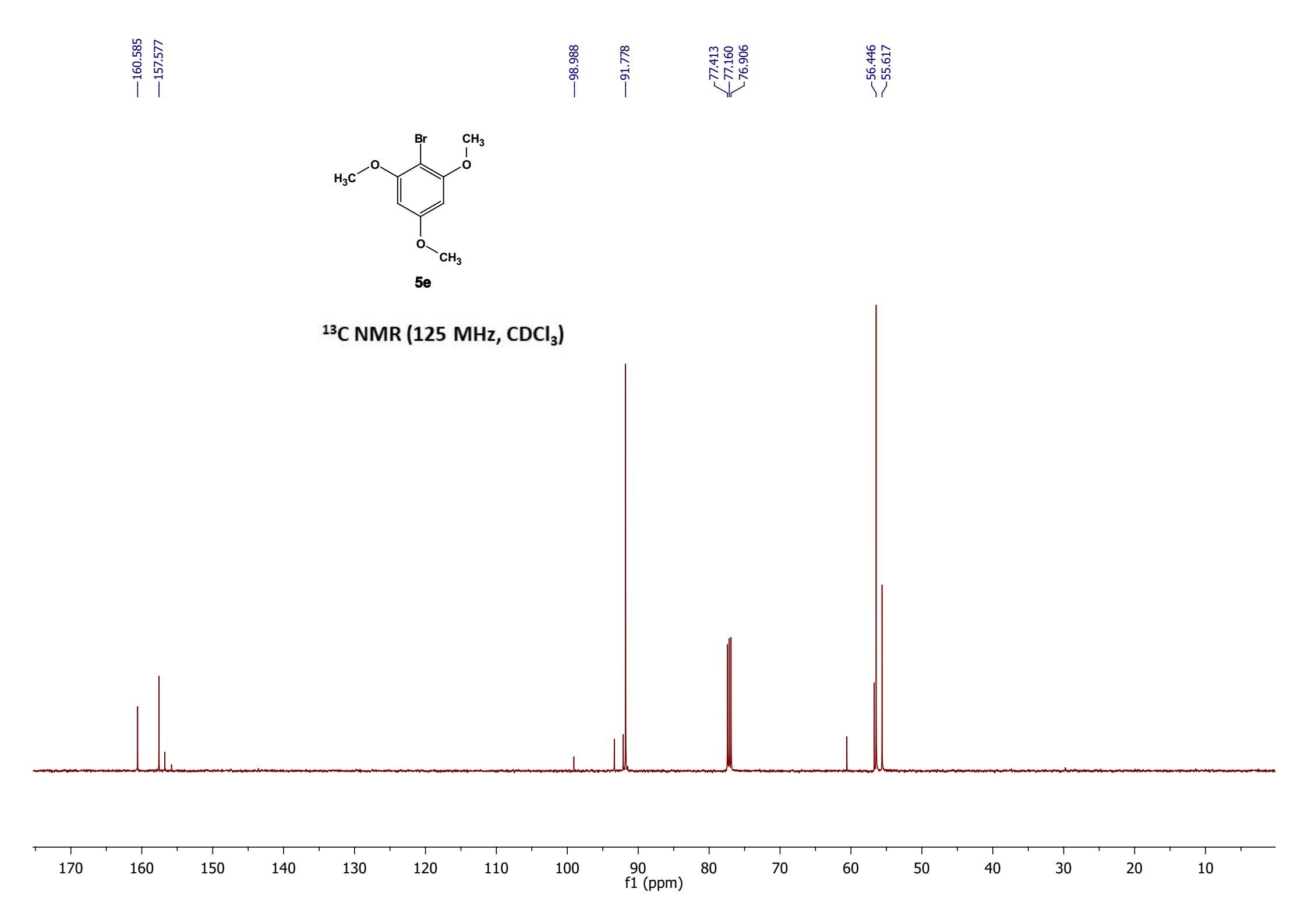

7.260 Chloroform-d
6.522
3.984
3.971
3.957
3.942
3.773
3.758
3.744
3.731
2.250
2.188
2.120
1.413
1.408
1.400
1.393
1.384
1.379
CH3
H3C
O
H3C
O
CH3
CH3
5c
1H NMR (500 MHz, CDCl3)
1.00
2.06
2.08
3.13
3.08
3.07
6.26
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

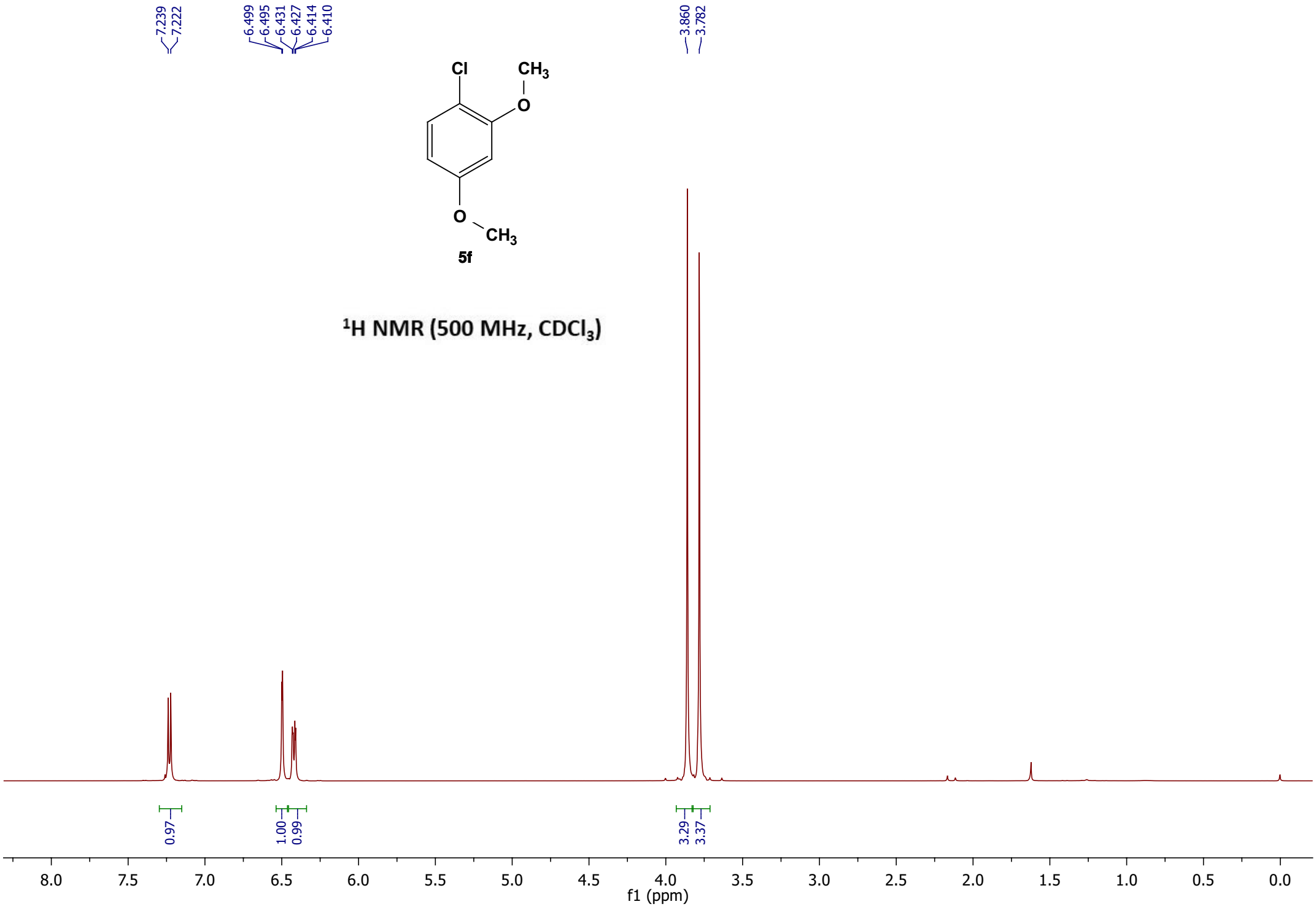

152.937
149.759
130.789
127.902
124.097
111.810
77.414
77.160
76.906
68.346
64.297
16.569
15.780
15.233
13.030
12.083
CH3
O
H3C
H3C
CH3
O
CH3
5c
13C NMR (125 MHz, CDCl3)
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

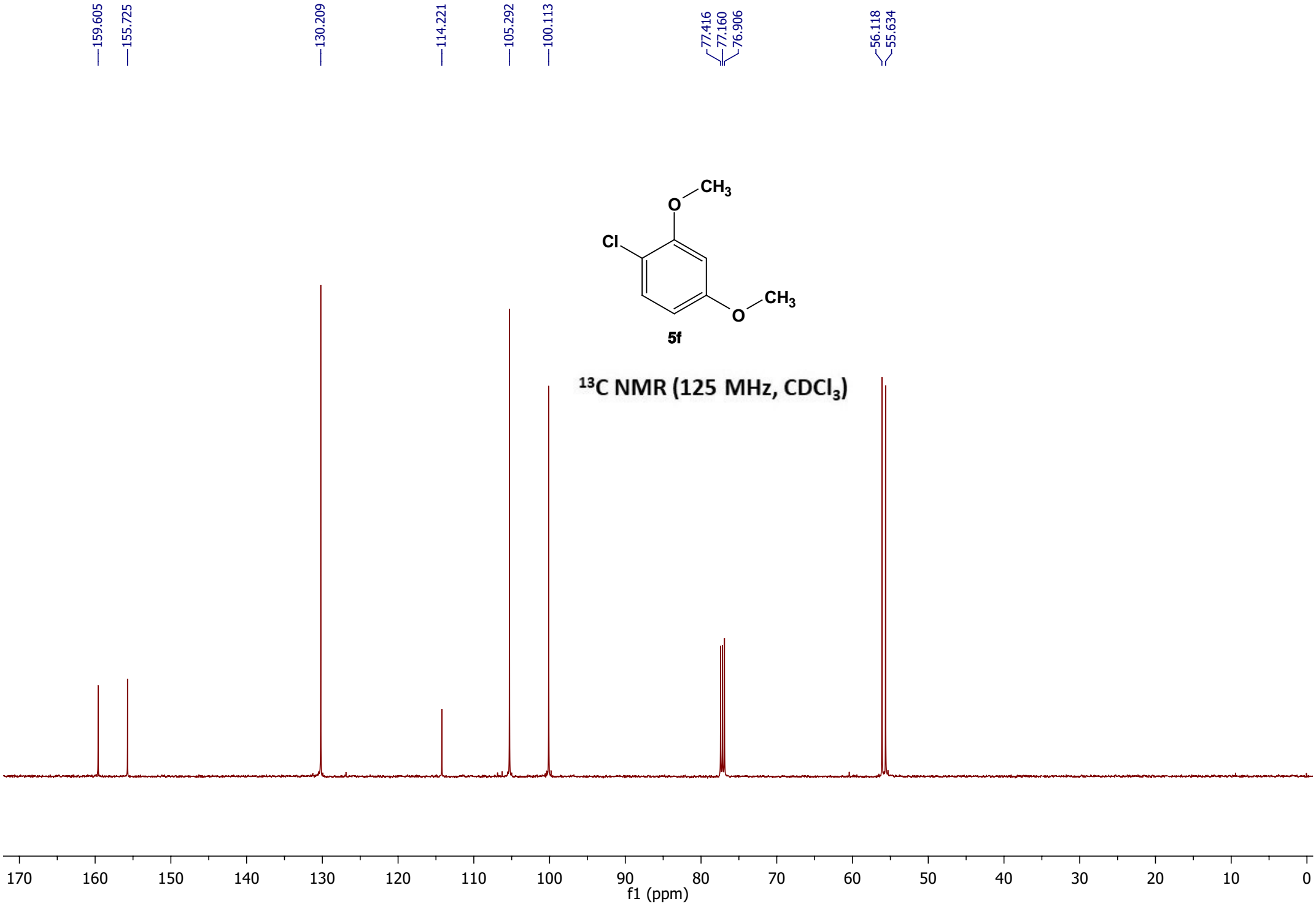

7.435
7.428
7.415
7.400
7.385
7.351
7.336
7.317
7.303
7.260
6.252
3.879
3.732
CH3
O
H3C
O
O
CH3
5d
1H NMR (500 MHz, CDCl3)
2.03
3.17
2.00
3.14
6.34
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

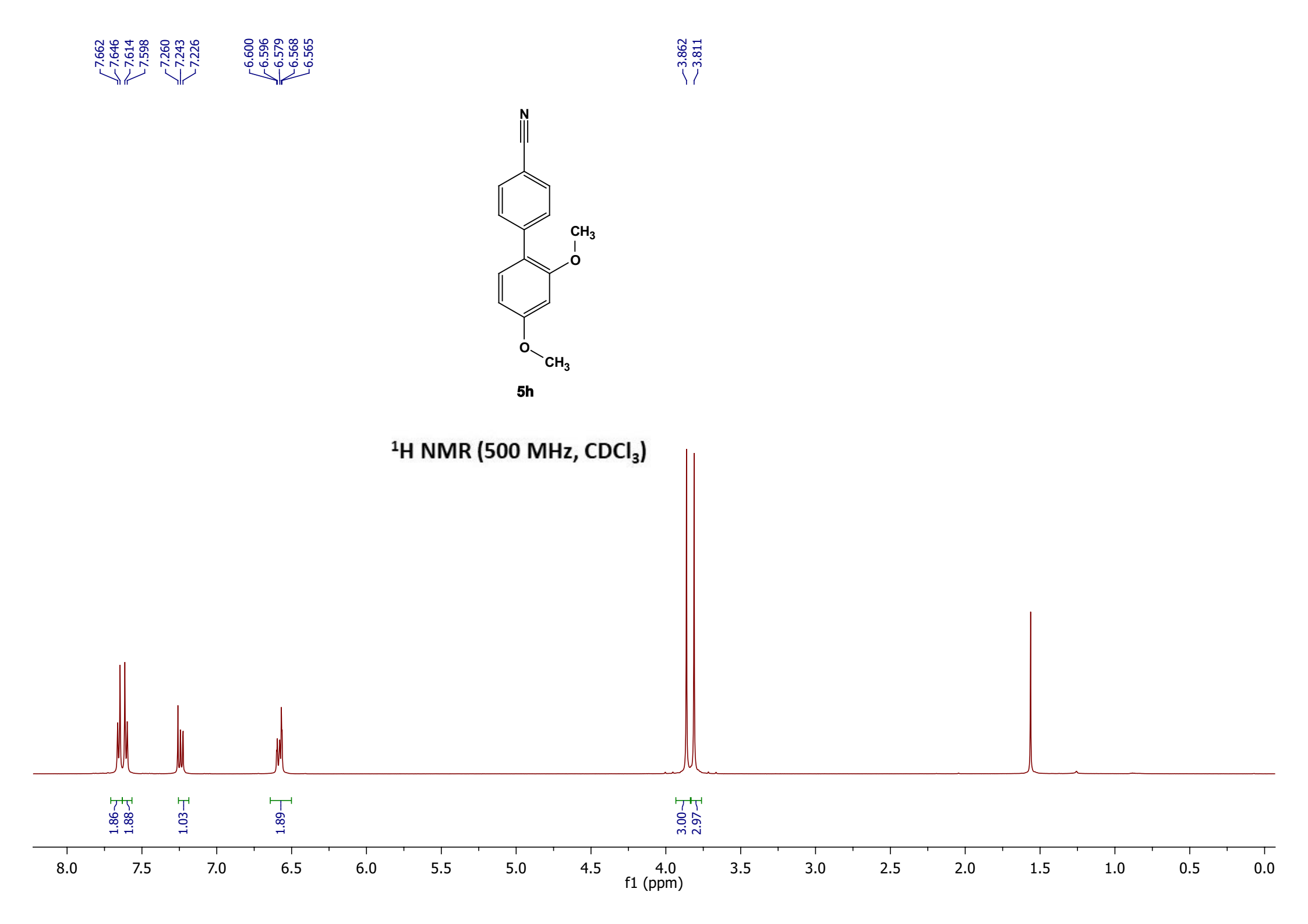

160.653
158.498
134.269
131.340
127.751
126.608
112.740
91.118
77.414
77.160
76.906
56.019
55.502
H3C
O
CH3
O
O
CH3
5d
13C NMR (125 MHz, CDCl3)
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

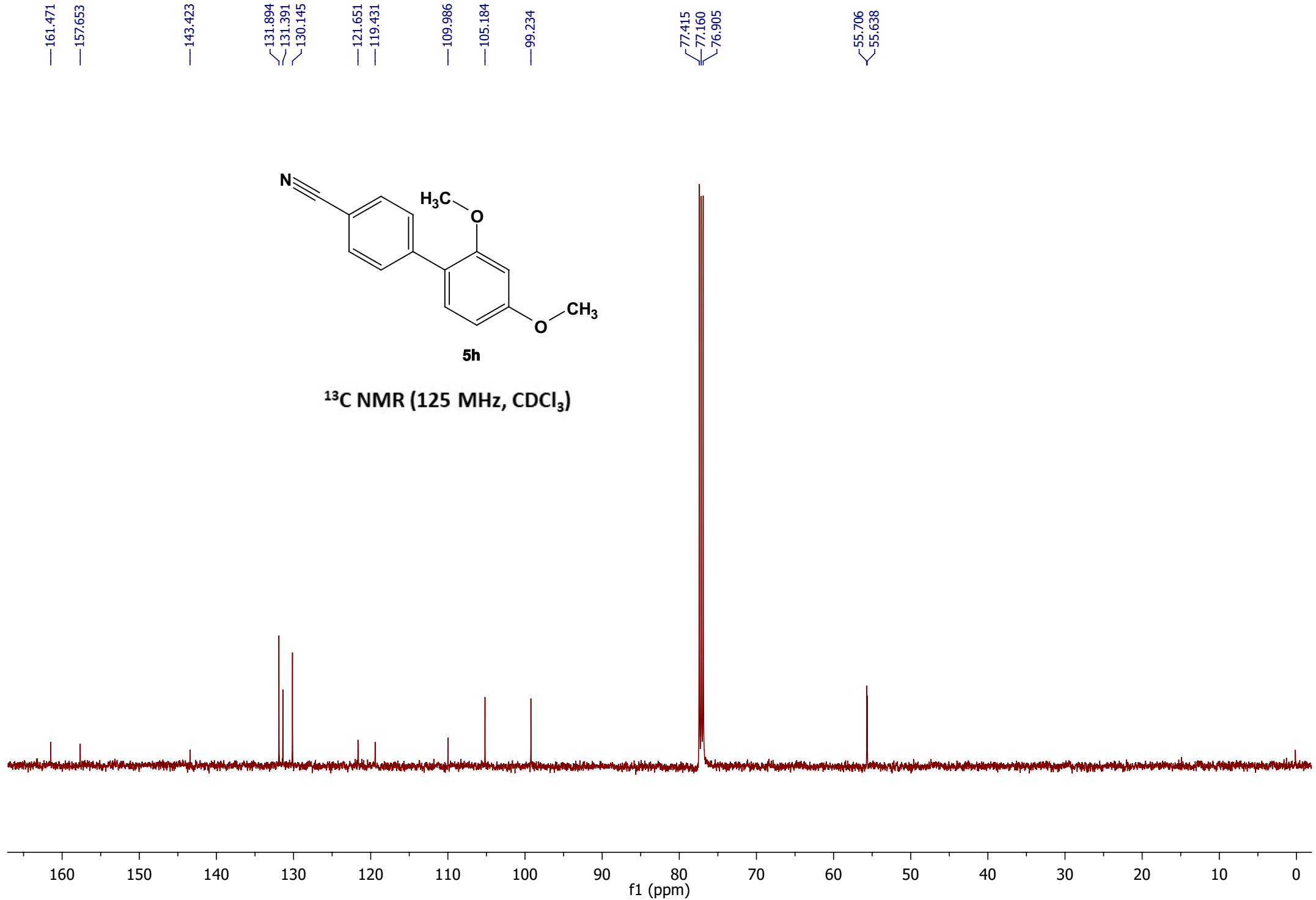

7.260
6.158
3.860
3.802
Br
CH3
O
H3C
O
O
CH3
5e
1H NMR (500 MHz, CDCl3)
2.00
6.29
3.03
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

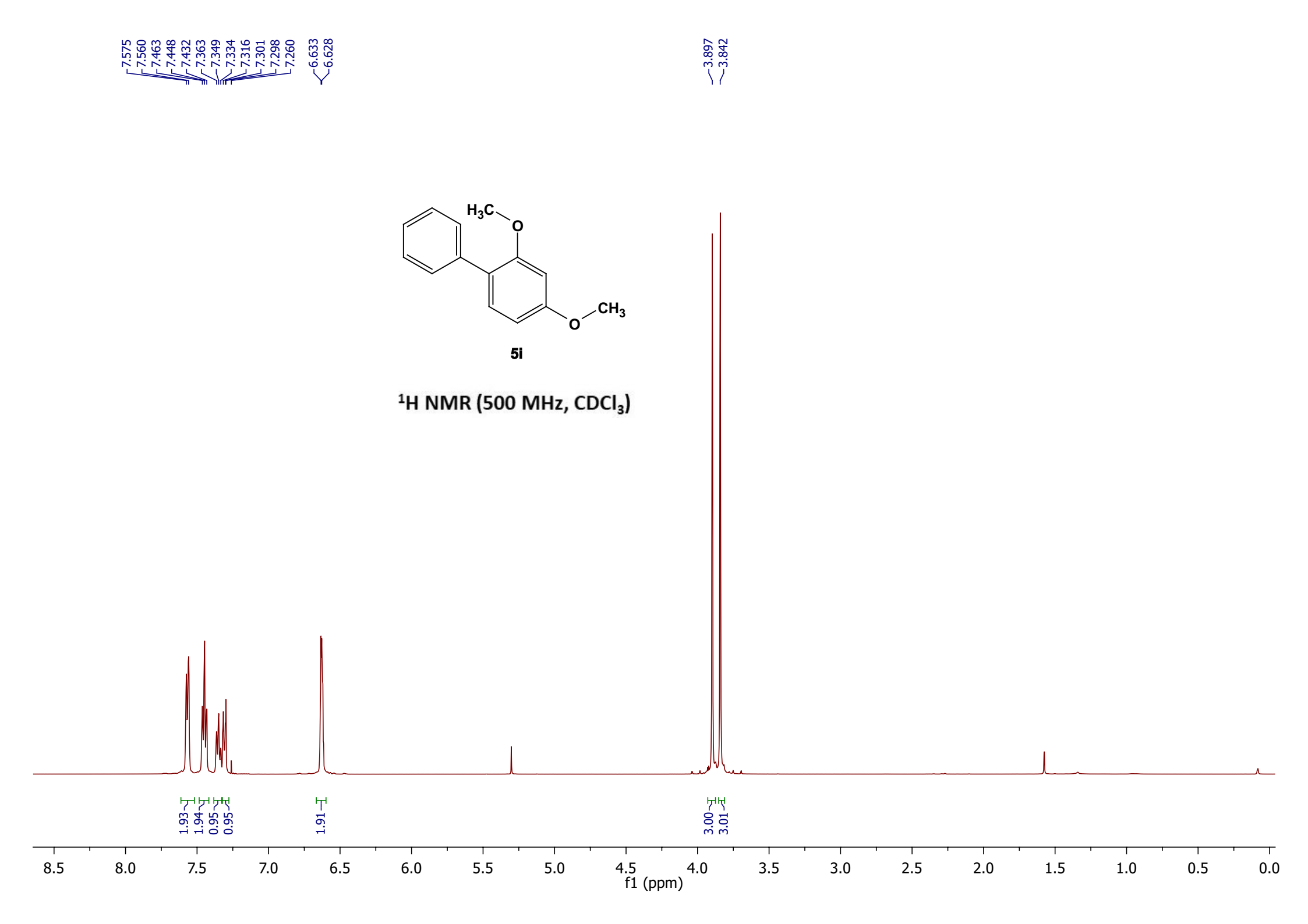

160.585
157.577
98.988
91.778
77.413
77.160
76.906
56.446
55.617
Br
CH3
O
H3C
O
O
CH3
5e
13C NMR (125 MHz, CDCl3)
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

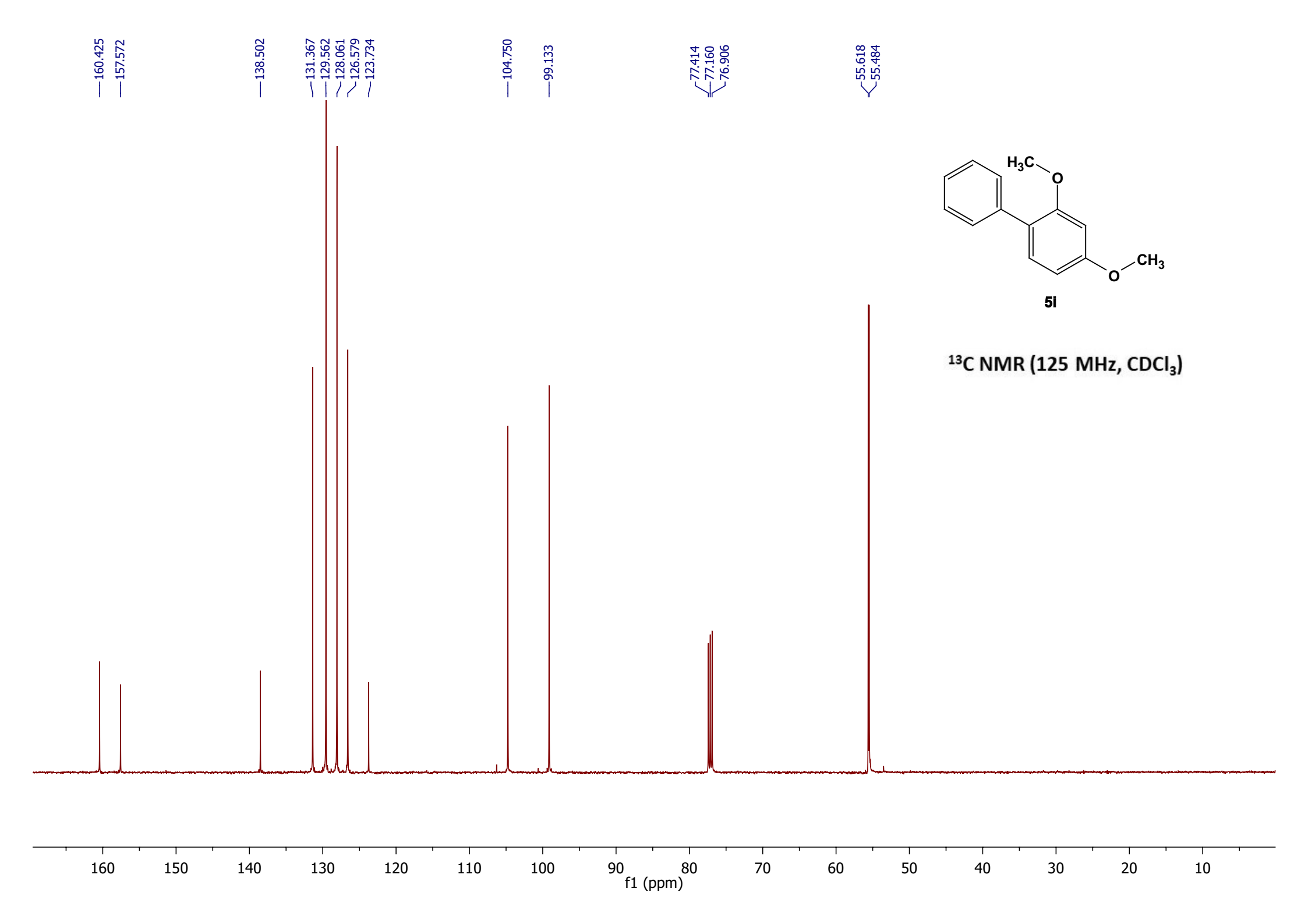

7.239
7.222
6.499
6.495
6.431
6.427
6.414
6.410
3.860
3.782
Cl
CH3
O
O
CH3
5f
1H NMR (500 MHz, CDCl3)
0.97
1.00
0.99
3.29
3.37
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

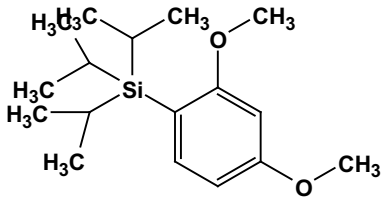

159.605
155.725
130.209
114.221
105.292
100.113
77.416
77.160
76.906
56.118
55.634
CH3
O
Cl
CH3
O
5f
13C NMR (125 MHz, CDCl3)
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.662
7.646
7.614
7.598
7.260
7.243
7.226
6.600
6.596
6.579
6.568
6.565
3.862
3.811
N
CH3
O
O
CH3
5h
1H NMR (500 MHz, CDCl3)
1.86
1.88
1.03
1.89
3.00
2.97
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)


161.471
157.653
143.423
131.894
131.391
130.145
121.651
119.431
109.986
105.184
99.234
77.415
77.160
76.905
55.706
55.638
N
H3C
O
O
CH3
5h
13C NMR (125 MHz, CDCl3)
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

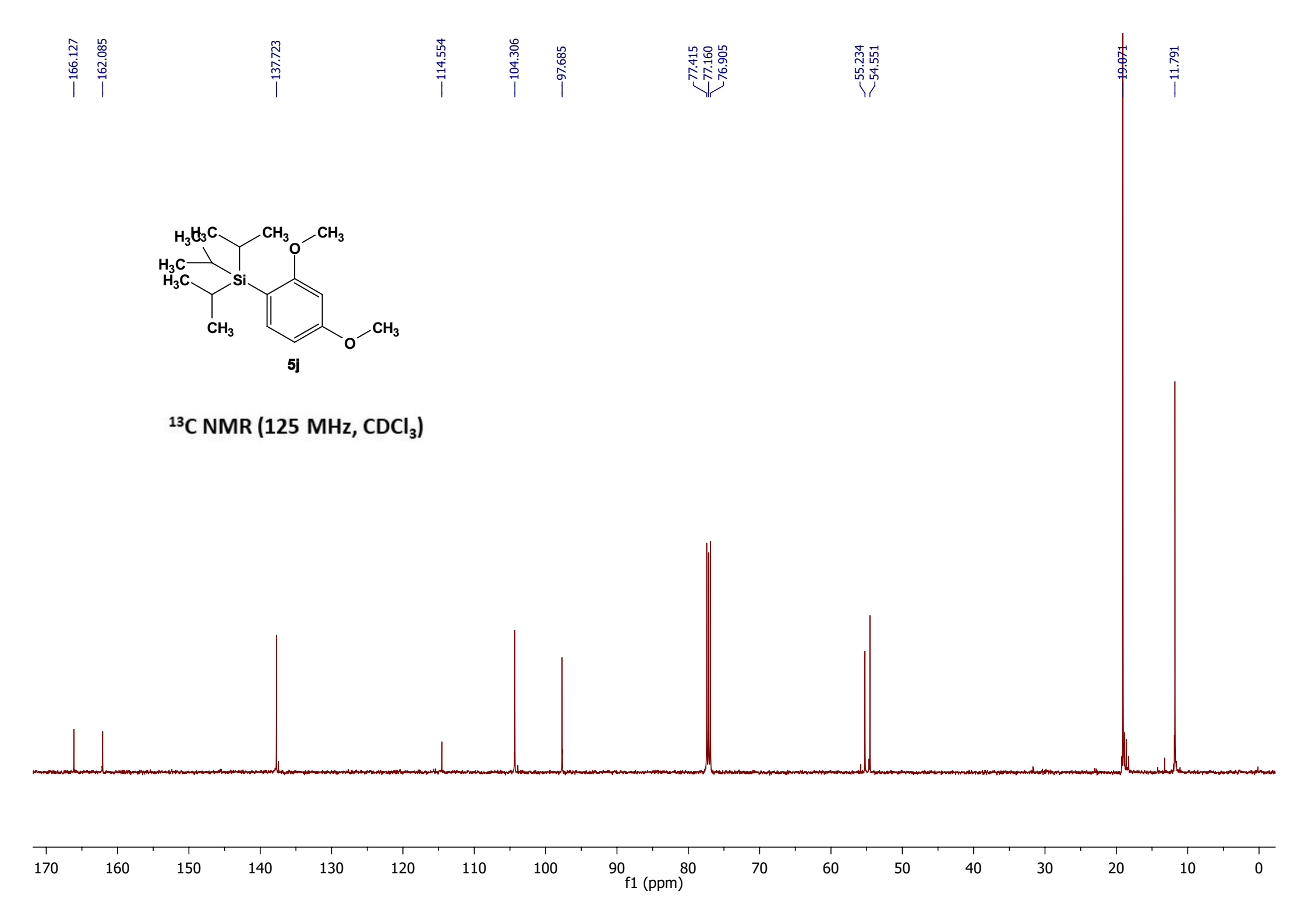

7.575
7.560
7.463
7.448
7.432
7.363
7.349
7.334
7.316
7.301
7.298
7.260
6.633
6.628
3.897
3.842
H3C
O
CH3
O
5i
1H NMR (500 MHz, CDCl3)
1.93
1.94
0.95
0.95
1.91
3.00
3.01
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

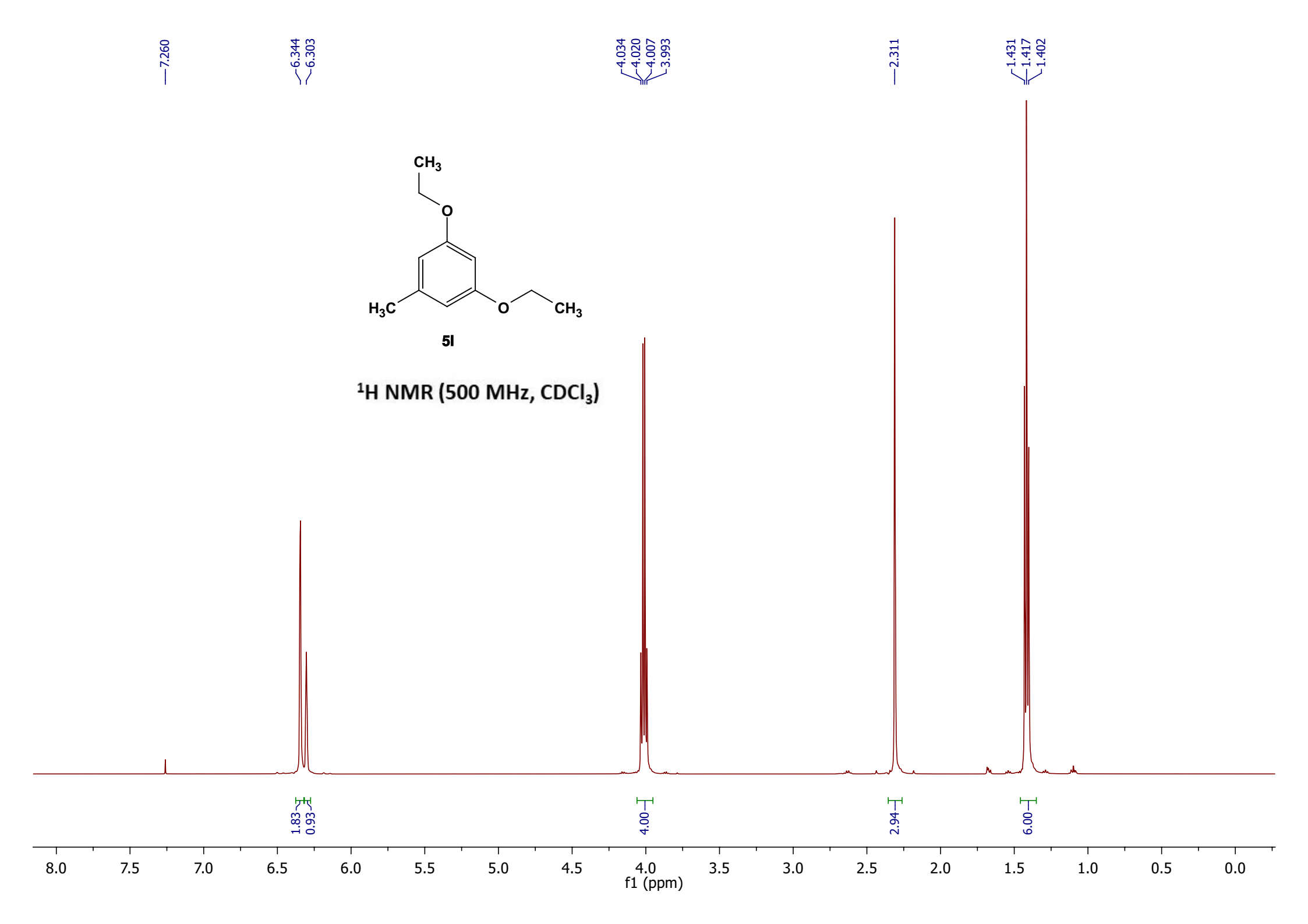

160.425
157.572
138.502
131.367
129.562
128.061
126.579
123.734
104.750
99.133
77.414
77.160
76.906
55.618
55.484
H3C
O
O
CH3
5l
13C NMR (125 MHz, CDCl3)
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

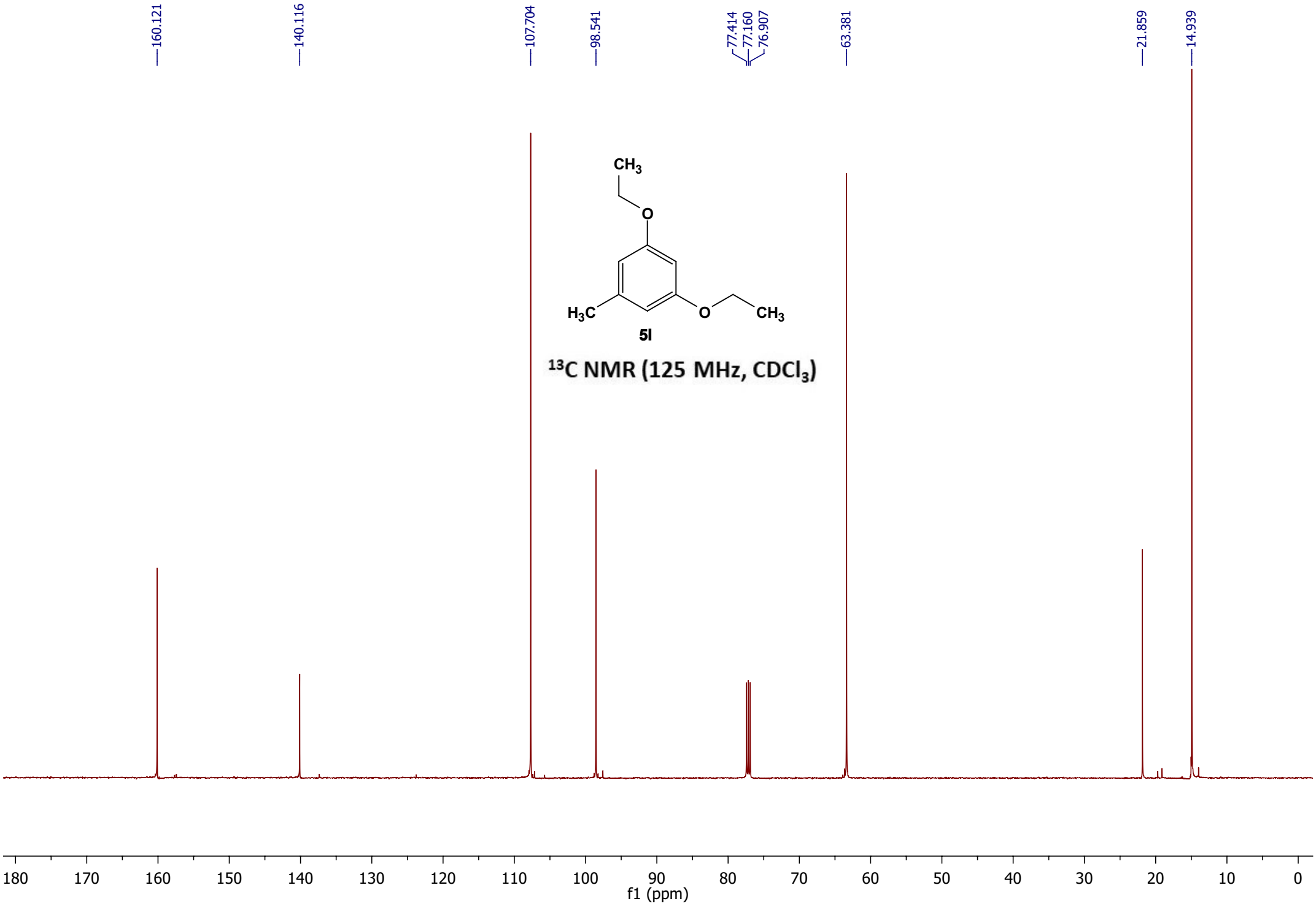


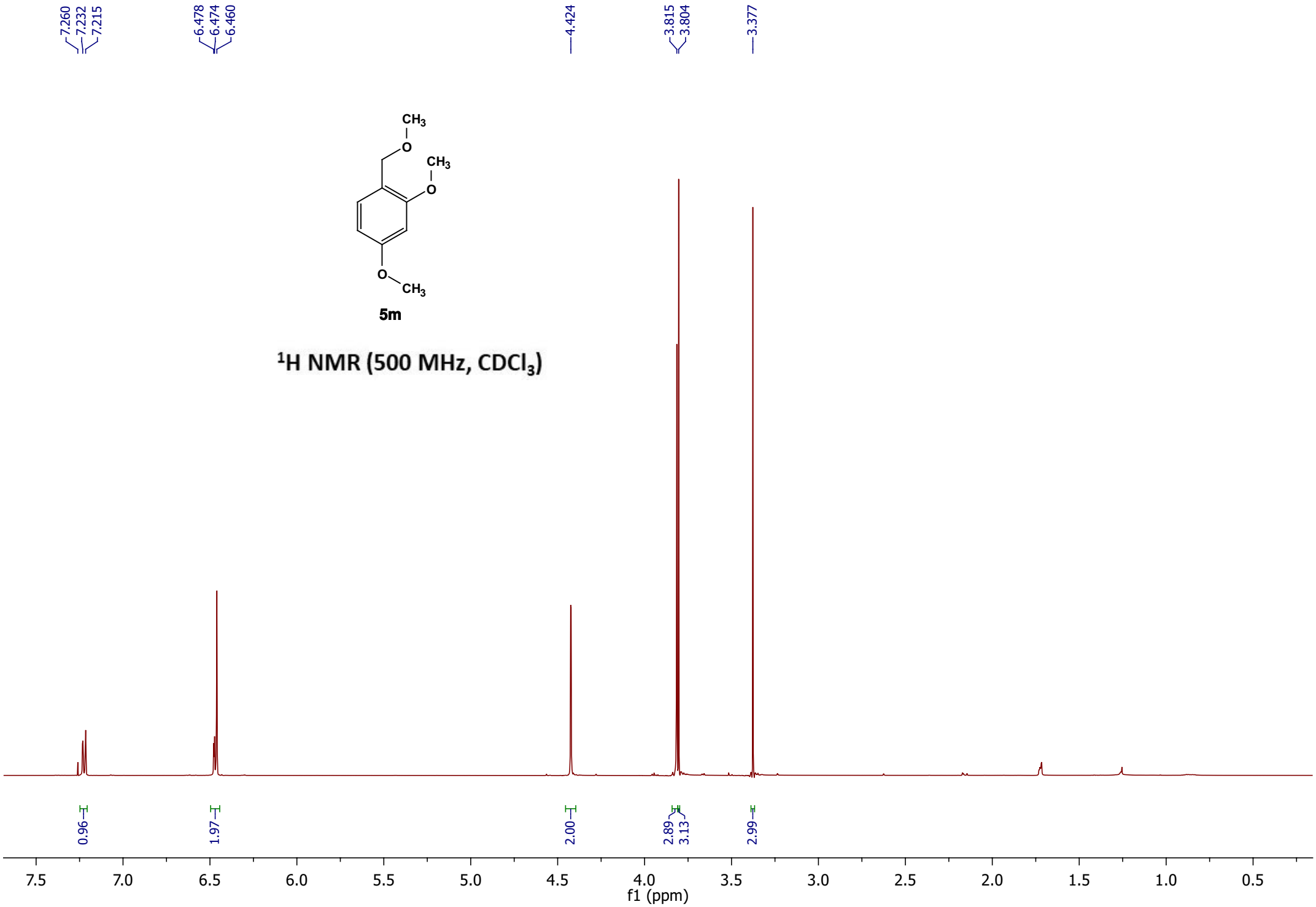
5j

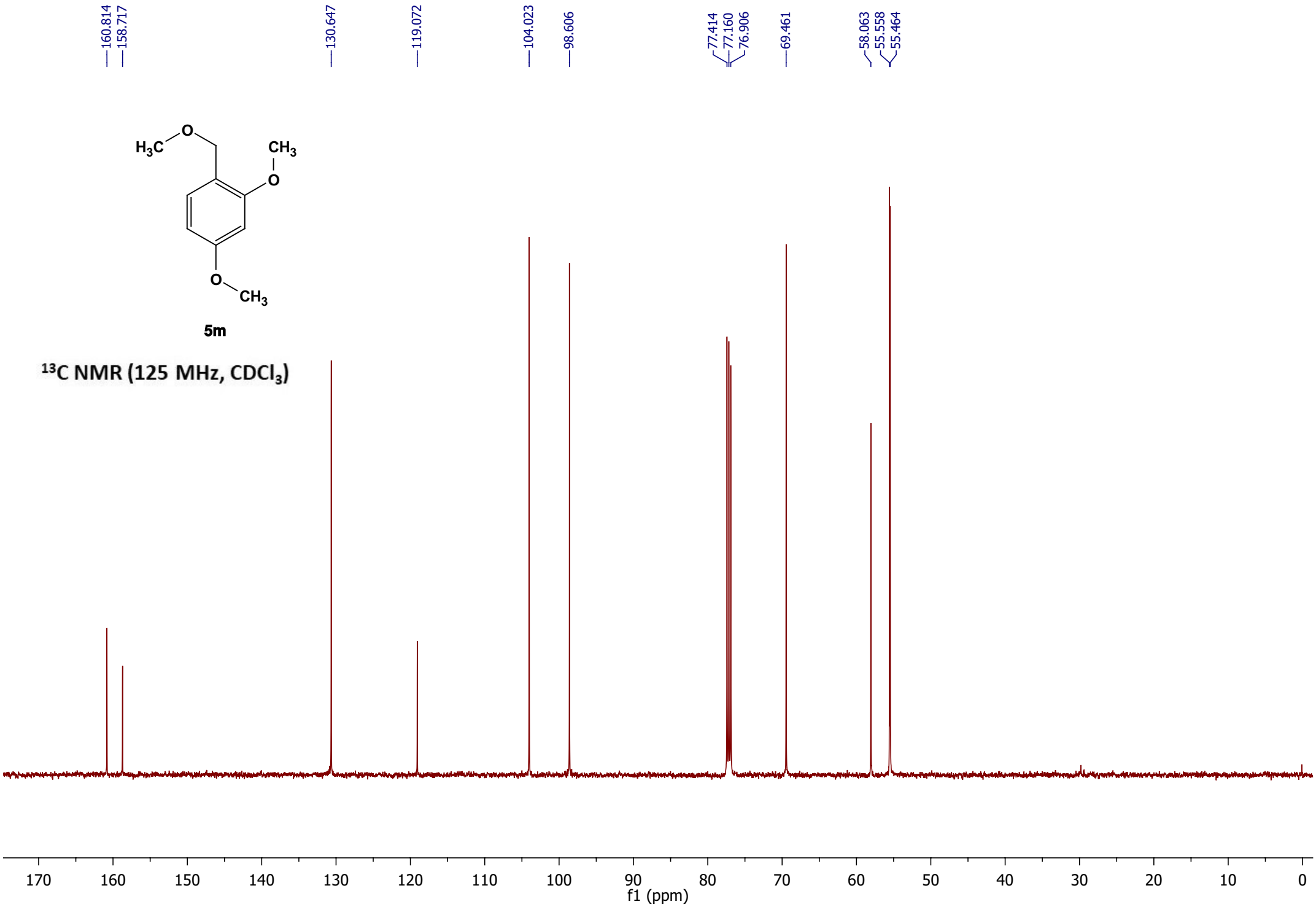
^{1}H NMR (500 MHz, $CDCl_3$)

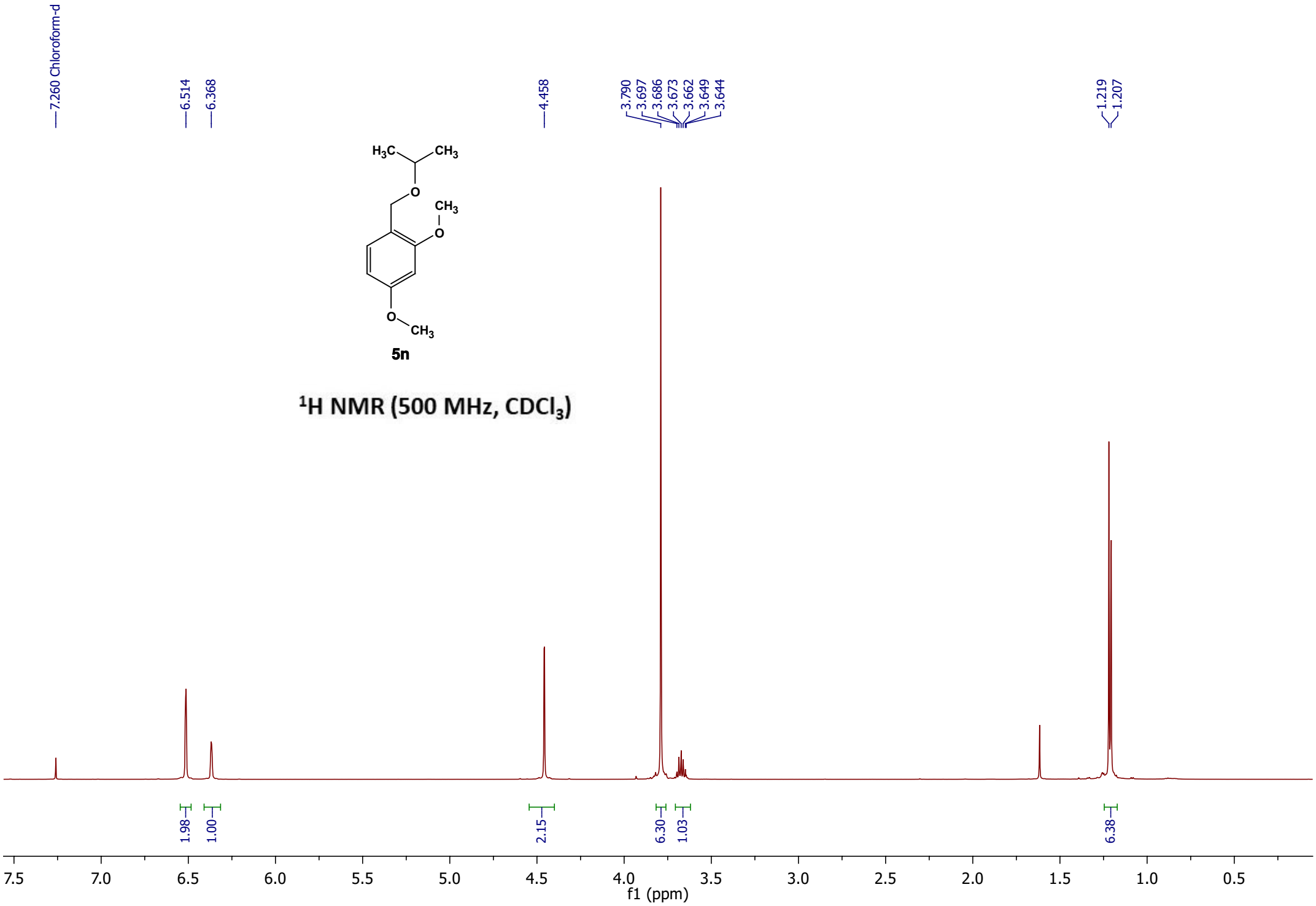

166.127
162.085
137.723
114.554
104.306
97.685
77.415
77.160
76.905
55.234
54.551
19.071
11.791
5j
13C NMR (125 MHz, CDCl3)
f1 (ppm)

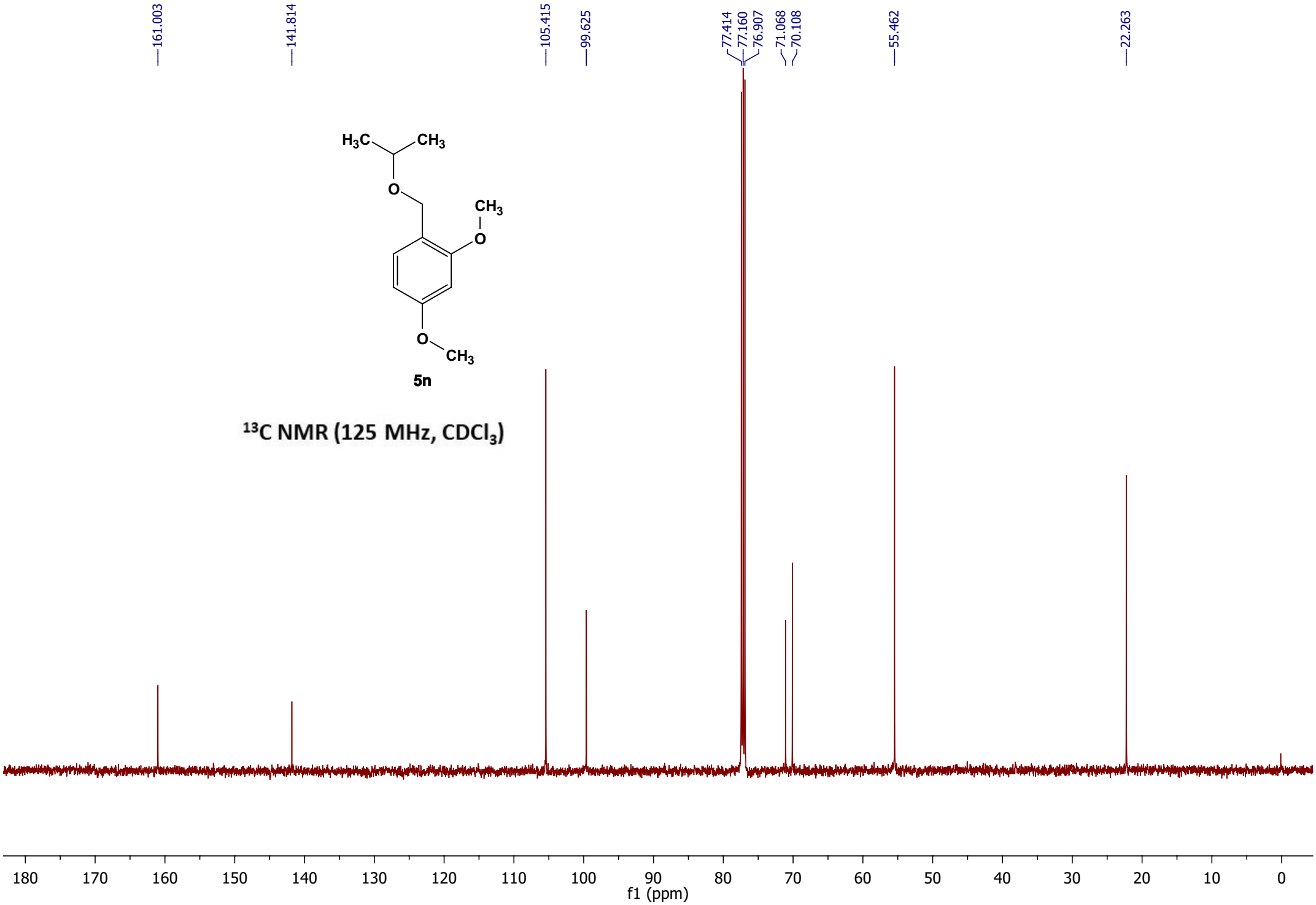

7.260
6.344
6.303
4.034
4.020
4.007
3.993
2.311
1.431
1.417
1.402
CH3
O
H3C
O
CH3
5l
1H NMR (500 MHz, CDCl3)
1.83
0.93
4.00
2.94
6.00
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

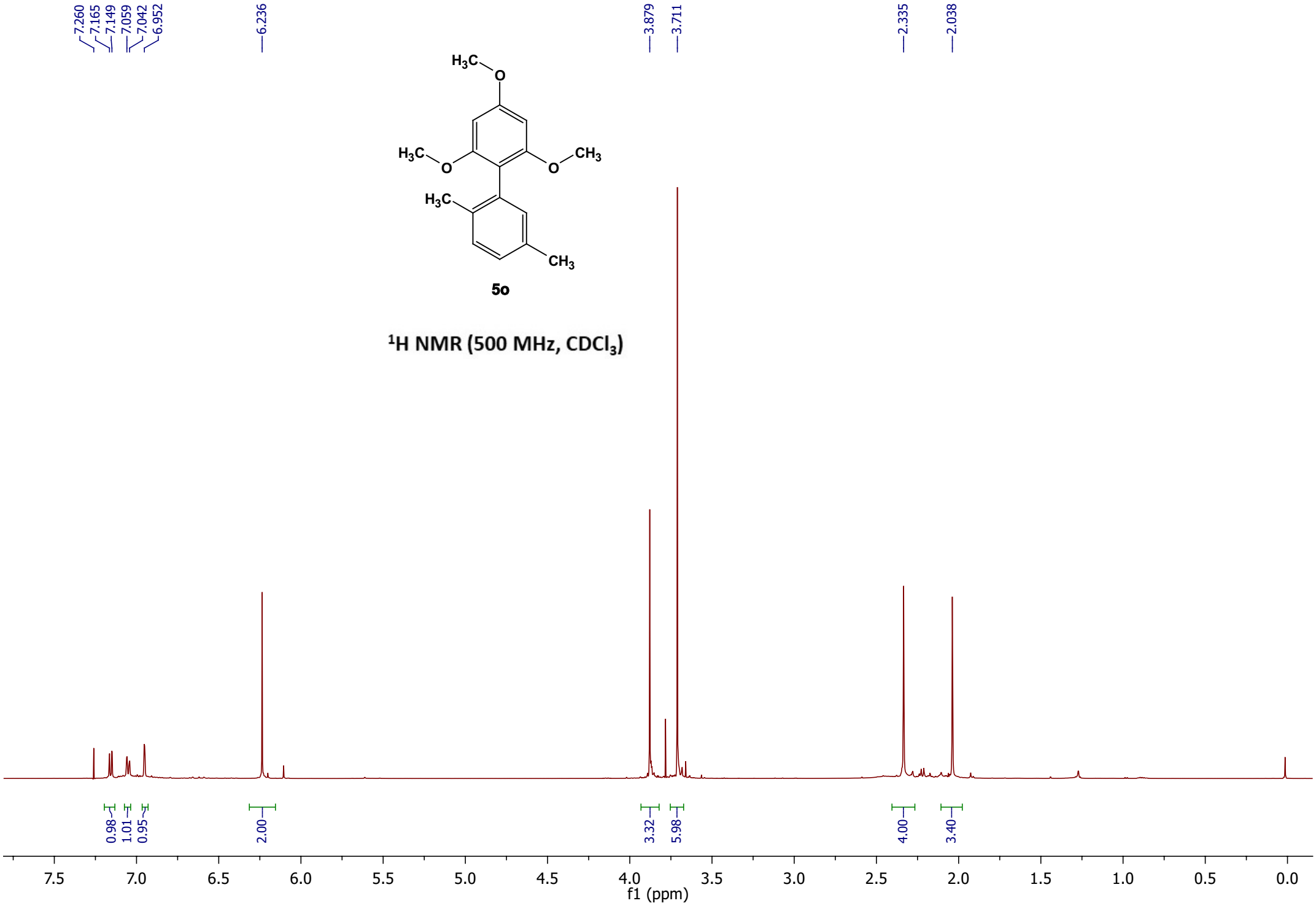

160.121
140.116
107.704
98.541
77.414
77.160
76.907
63.381
21.859
14.939
CH3
O
H3C
O
CH3
5l
13C NMR (125 MHz, CDCl3)
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

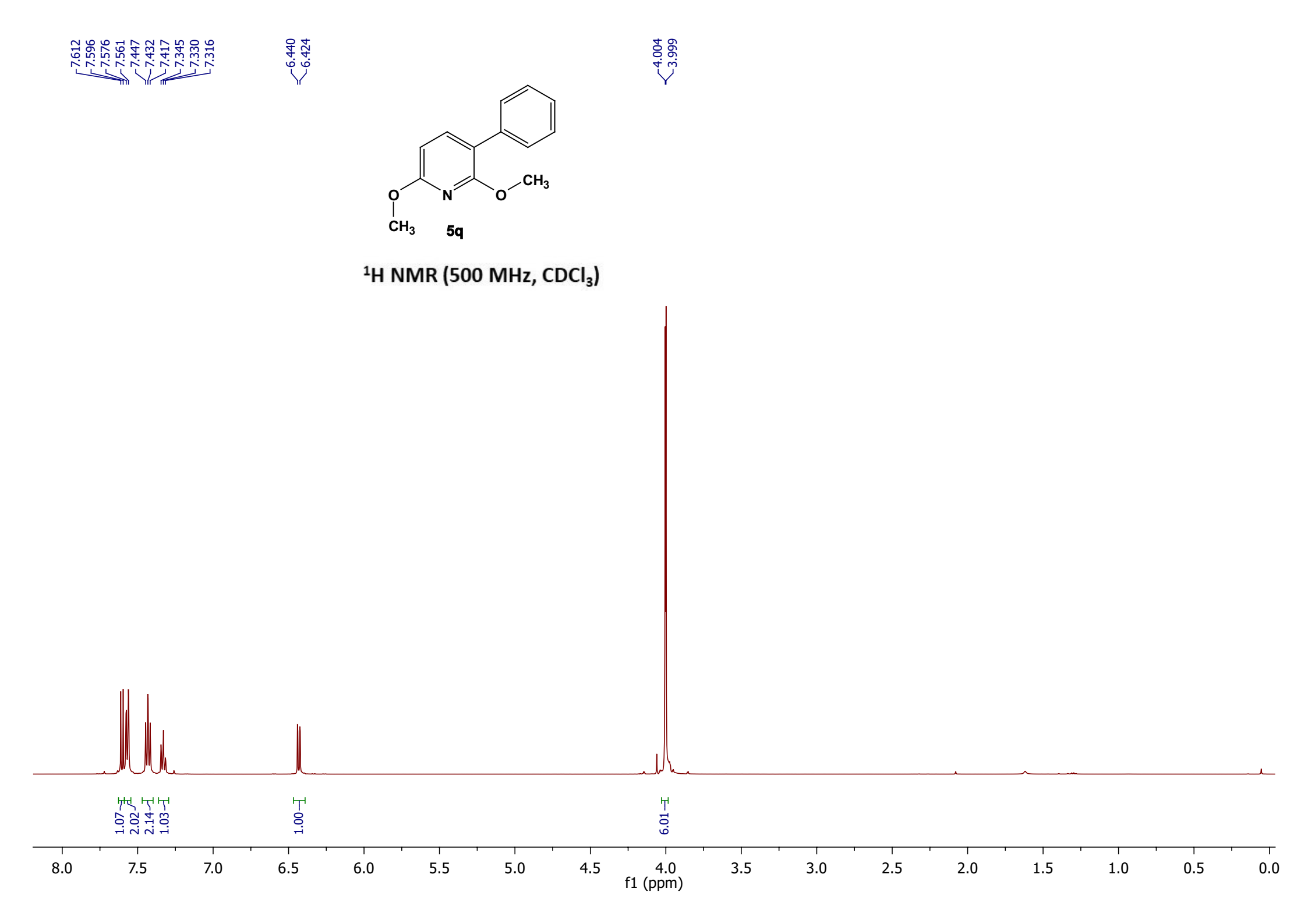

7.260
7.232
7.215
6.478
6.474
6.460
4.424
3.815
3.804
3.377
CH3
O
CH3
O
O
CH3
5m
1H NMR (500 MHz, CDCl3)
0.96
1.97
2.00
2.89
3.13
2.99
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

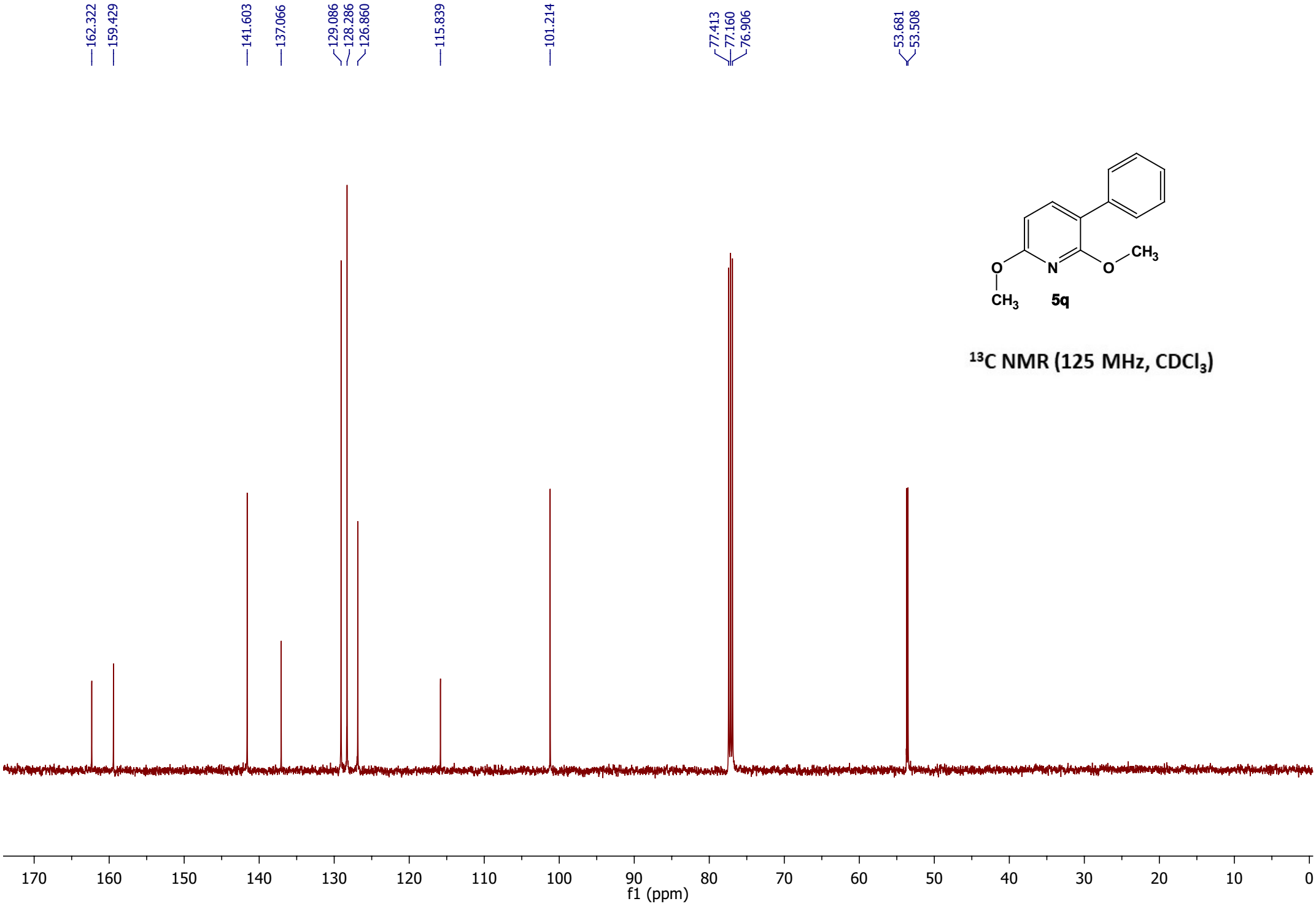

160.814
158.717
130.647
119.072
104.023
98.606
77.414
77.160
76.906
69.461
58.063
55.558
55.464
H3C
O
CH3
O
O
CH3
5m
13C NMR (125 MHz, CDCl3)
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

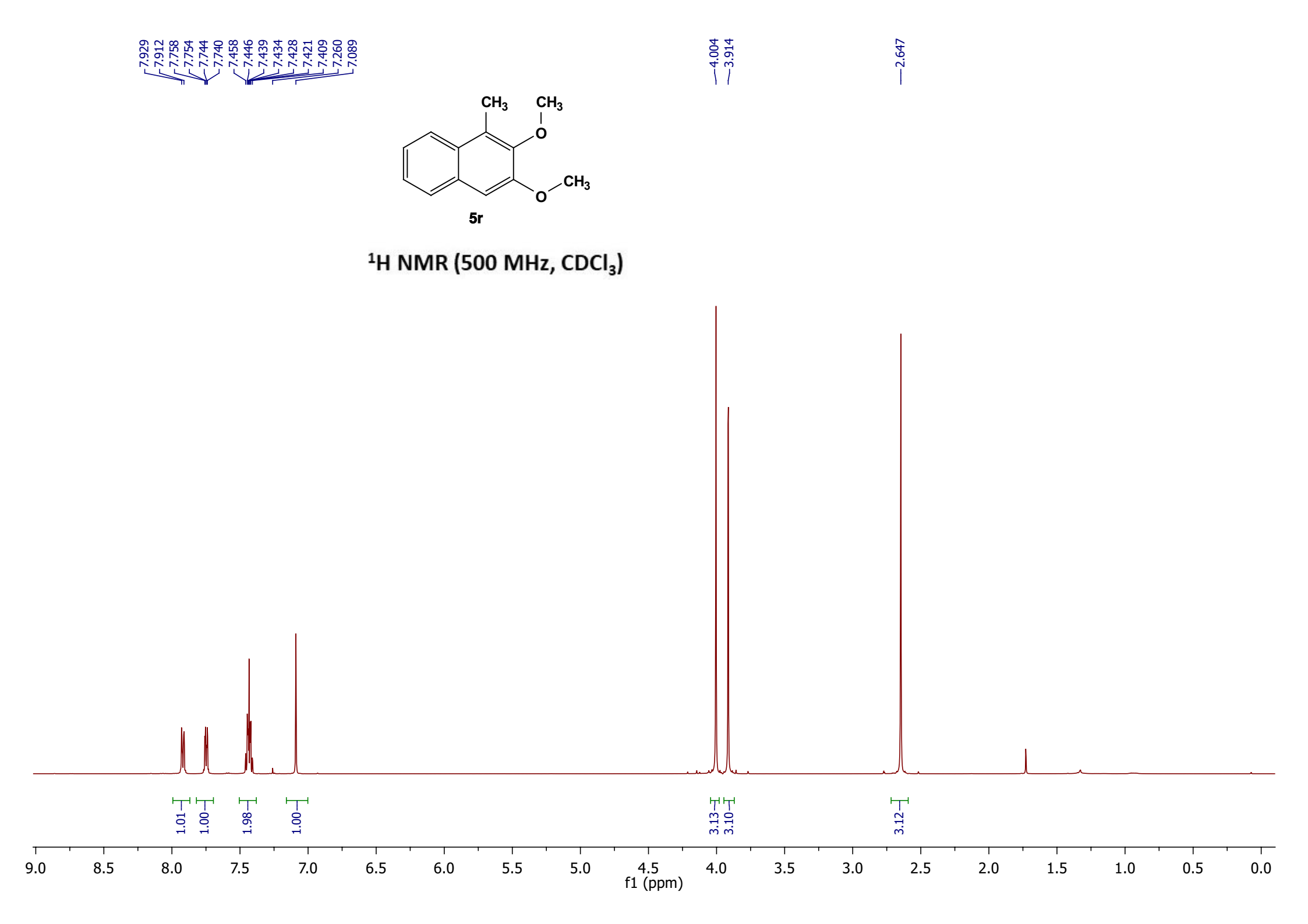

7.260 Chloroform-d
6.514
6.368
4.458
3.790
3.697
3.686
3.673
3.662
3.649
3.644
1.219
1.207
H3C
CH3
O
CH3
O
O
CH3
5n
1H NMR (500 MHz, CDCl3)
1.98
1.00
2.15
6.30
1.03
6.38
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

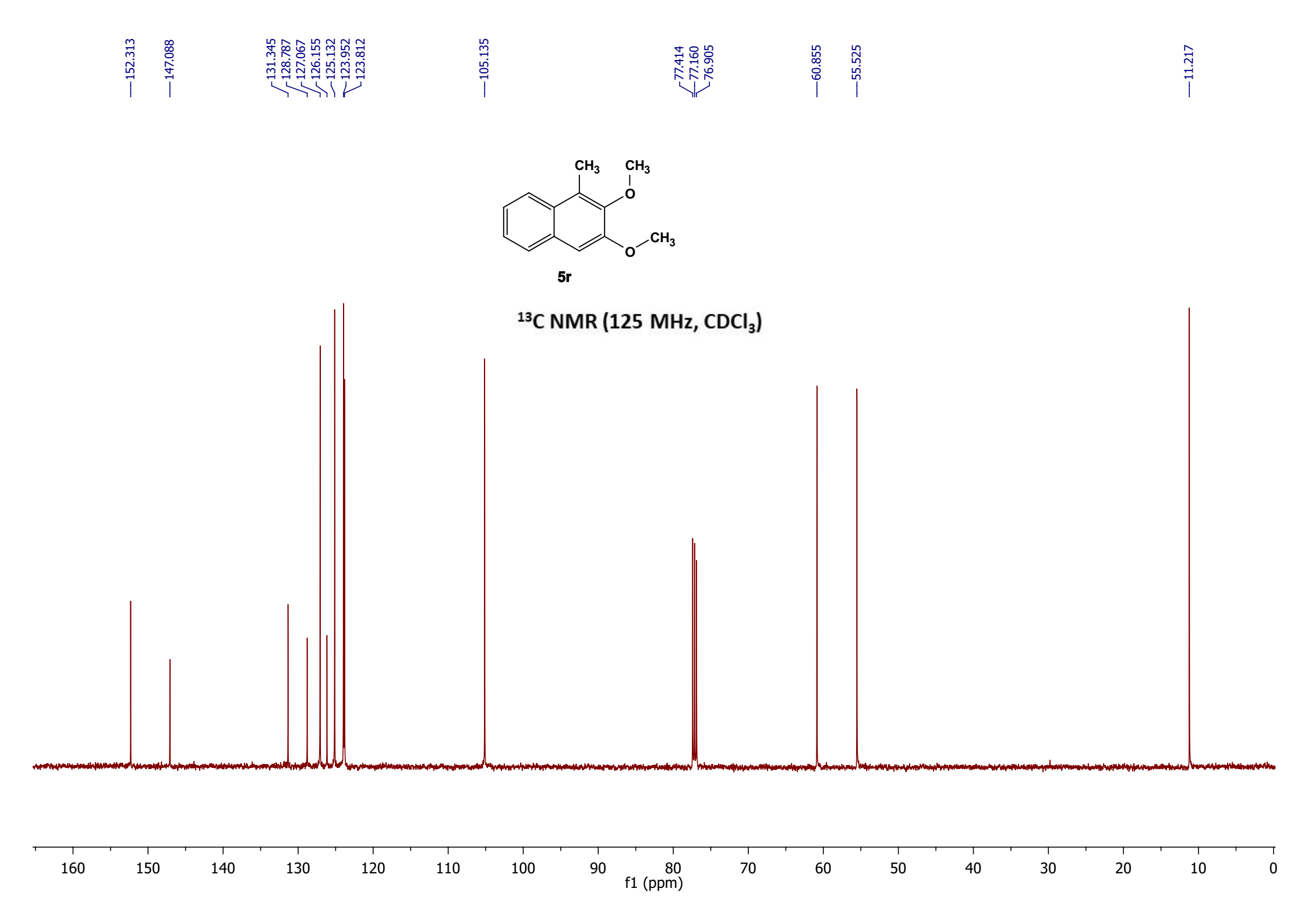

161.003
141.814
105.415
99.625
77.414
77.160
76.907
71.068
70.108
55.462
22.263
H3C
CH3
O
CH3
O
O
CH3
5n
13C NMR (125 MHz, CDCl3)
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

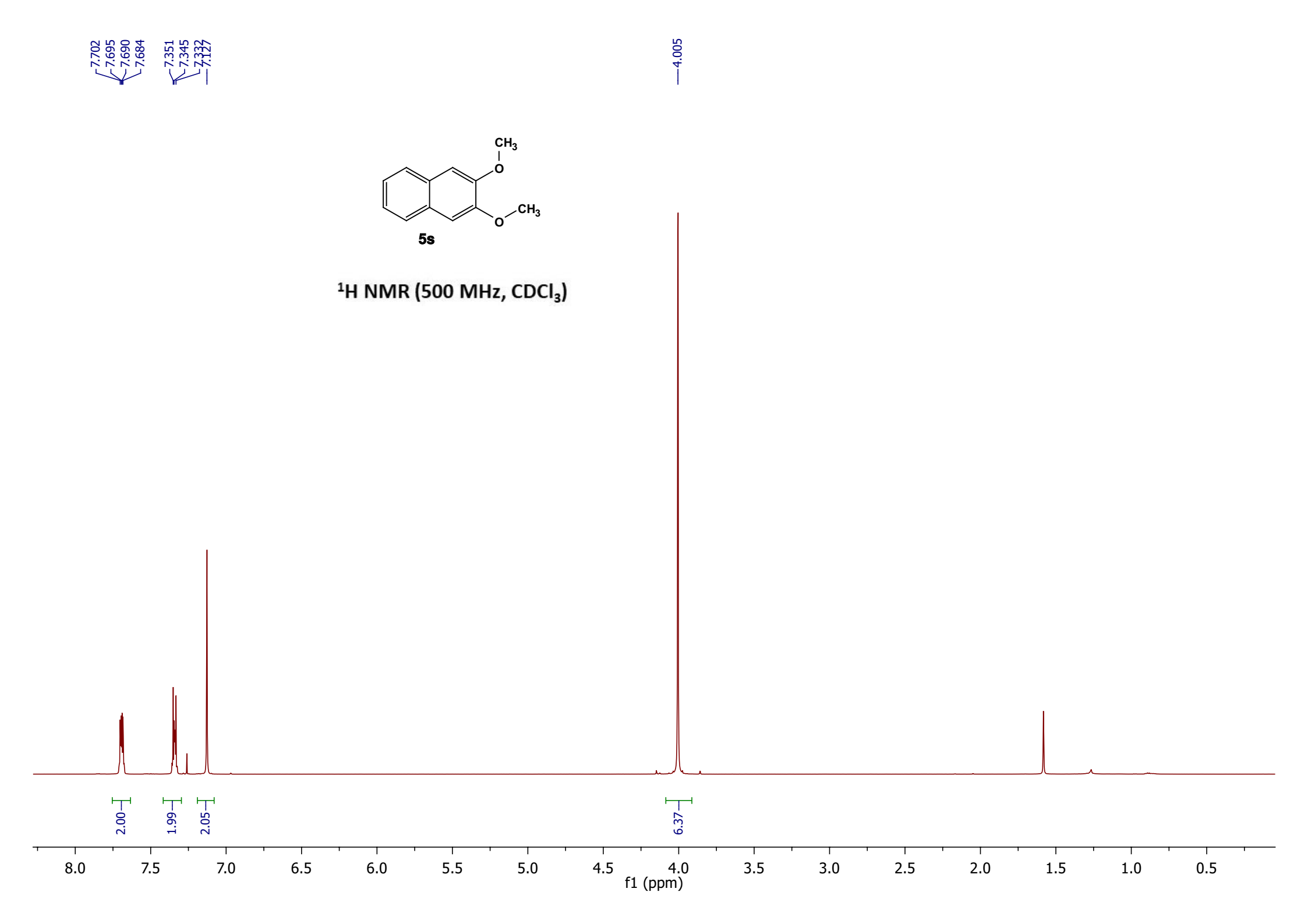

7.260
7.165
7.149
7.059
7.042
6.952
6.236
3.879
3.711
2.335
2.038
H3C
O
H3C
O
O
CH3
H3C
CH3
5o
1H NMR (500 MHz, CDCl3)
0.98
1.01
0.95
2.00
3.32
5.98
4.00
3.40
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

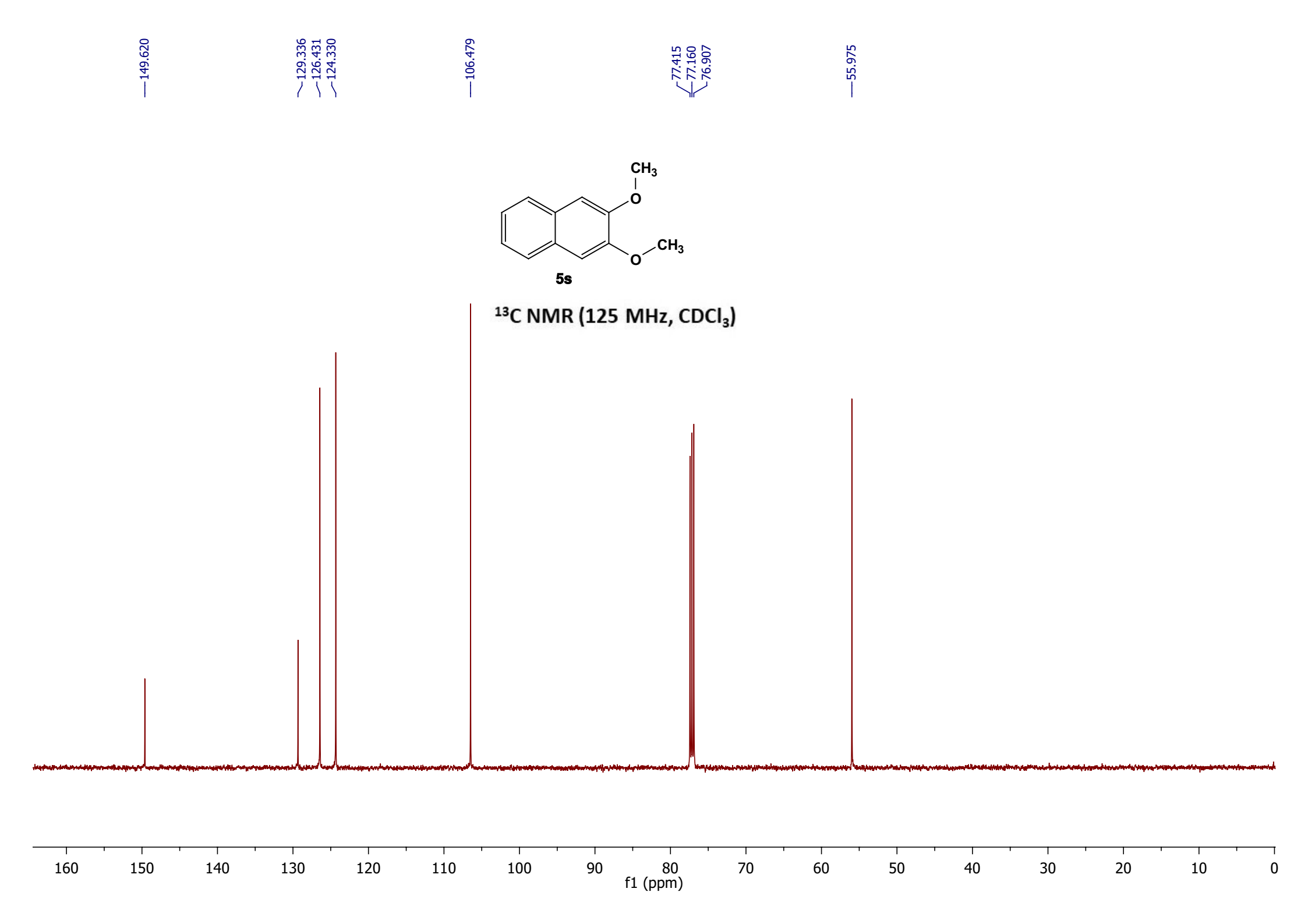

7.612
7.596
7.576
7.561
7.447
7.432
7.417
7.345
7.330
7.316
6.440
6.424
4.004
3.999
O
N
O
CH3
CH3
5q
1H NMR (500 MHz, CDCl3)
1.07
2.02
2.14
1.03
1.00
6.01
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

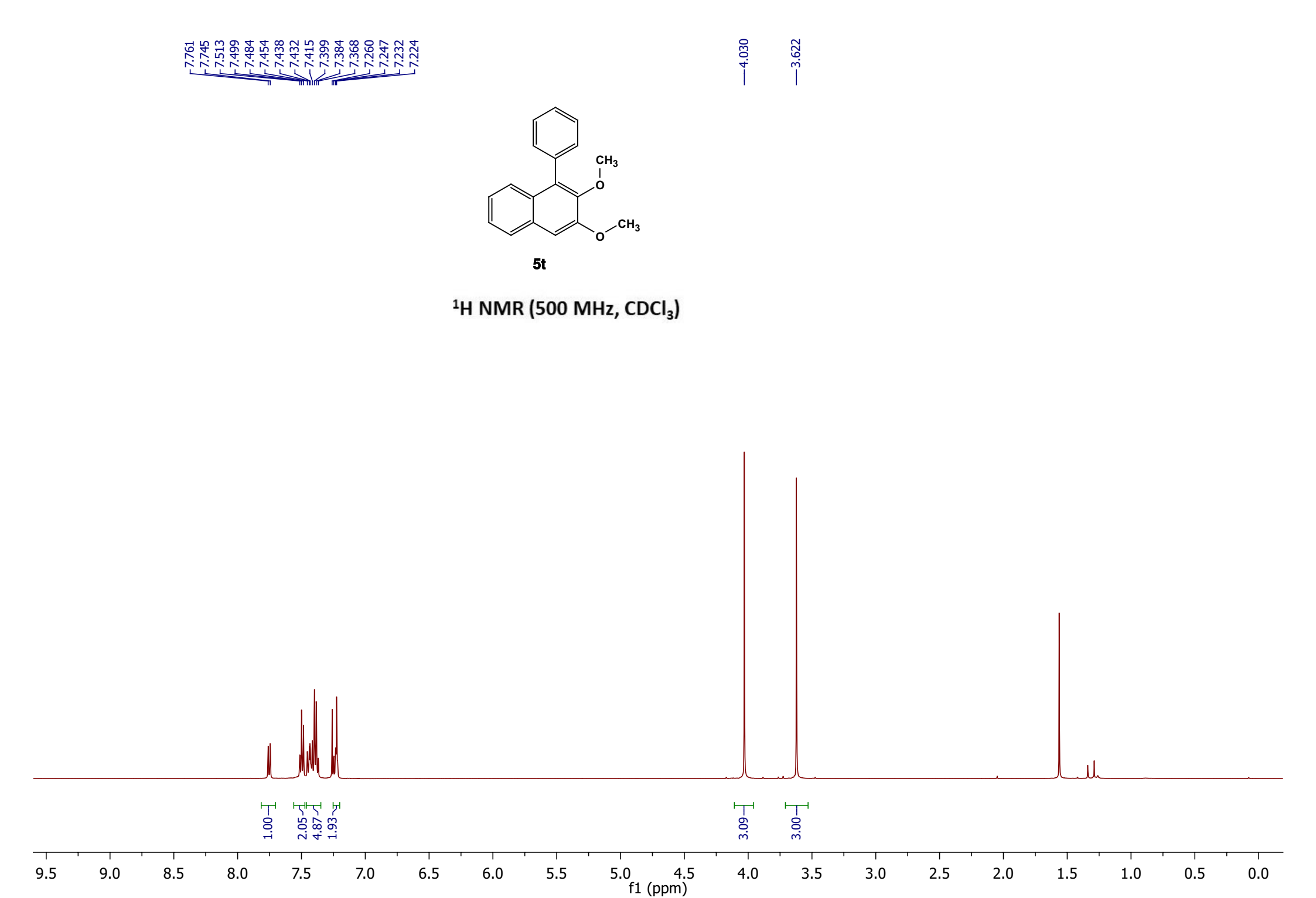

162.322
159.429
141.603
137.066
129.086
128.286
126.860
115.839
101.214
77.413
77.160
76.906
53.681
53.508
O
N
O
CH3
CH3
5q
13C NMR (125 MHz, CDCl3)
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.929
7.912
7.758
7.754
7.744
7.740
7.458
7.446
7.439
7.434
7.428
7.421
7.409
7.260
7.089
4.004
3.914
2.647
CH3
CH3
O
O
CH3
5r
1H NMR (500 MHz, CDCl3)
1.01
1.00
1.98
1.00
3.13
3.10
3.12
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)


152.313
147.088
131.345
128.787
127.067
126.155
125.132
123.952
123.812
105.135
77.414
77.160
76.905
60.855
55.525
11.217
CH3
CH3
O
O
CH3
5r
13C NMR (125 MHz, CDCl3)
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

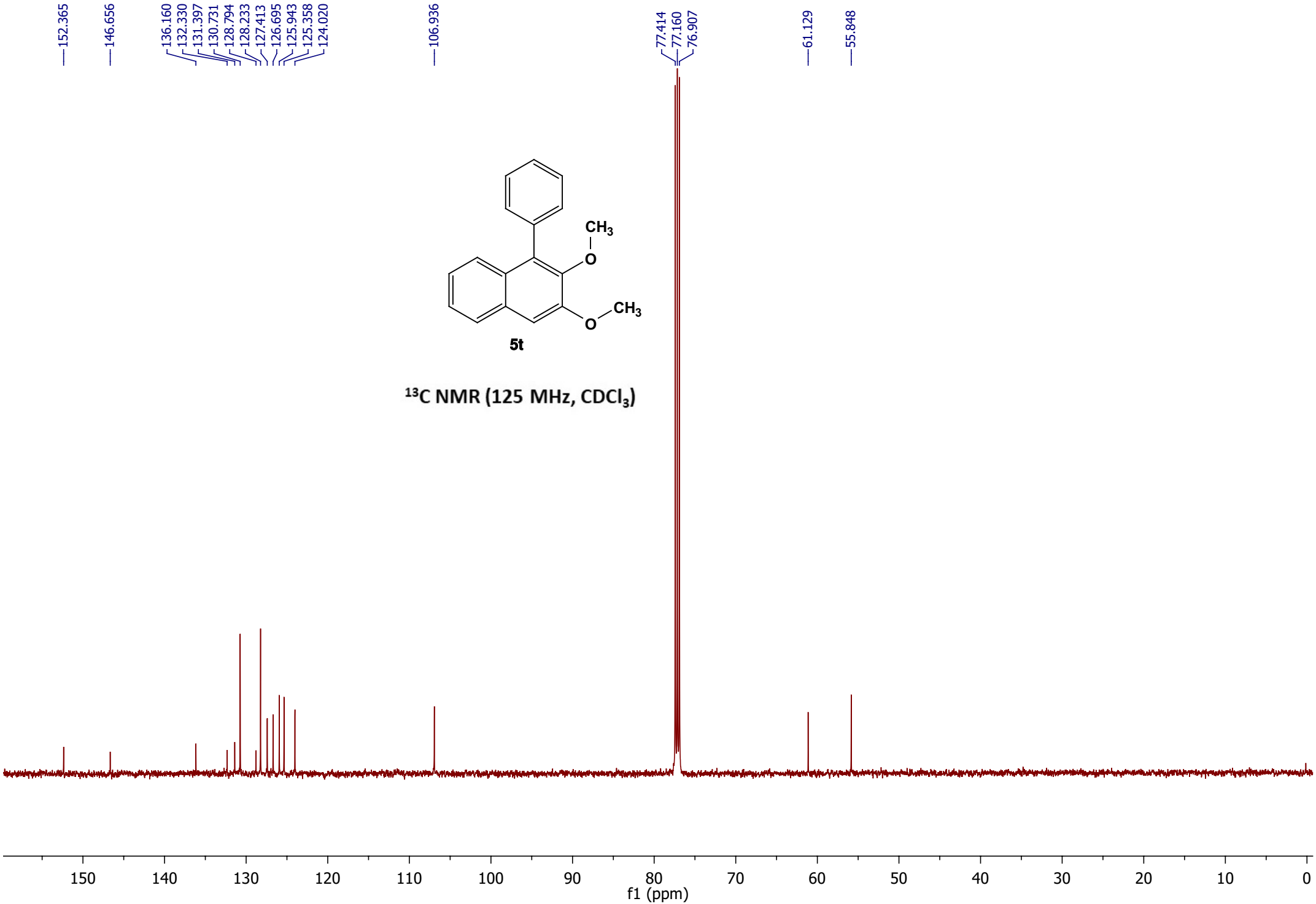

7.702
7.695
7.690
7.684
7.351
7.345
7.332
7.127
4.005
CH3
O
O
CH3
5s
1H NMR (500 MHz, CDCl3)
2.00
1.99
2.05
6.37
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

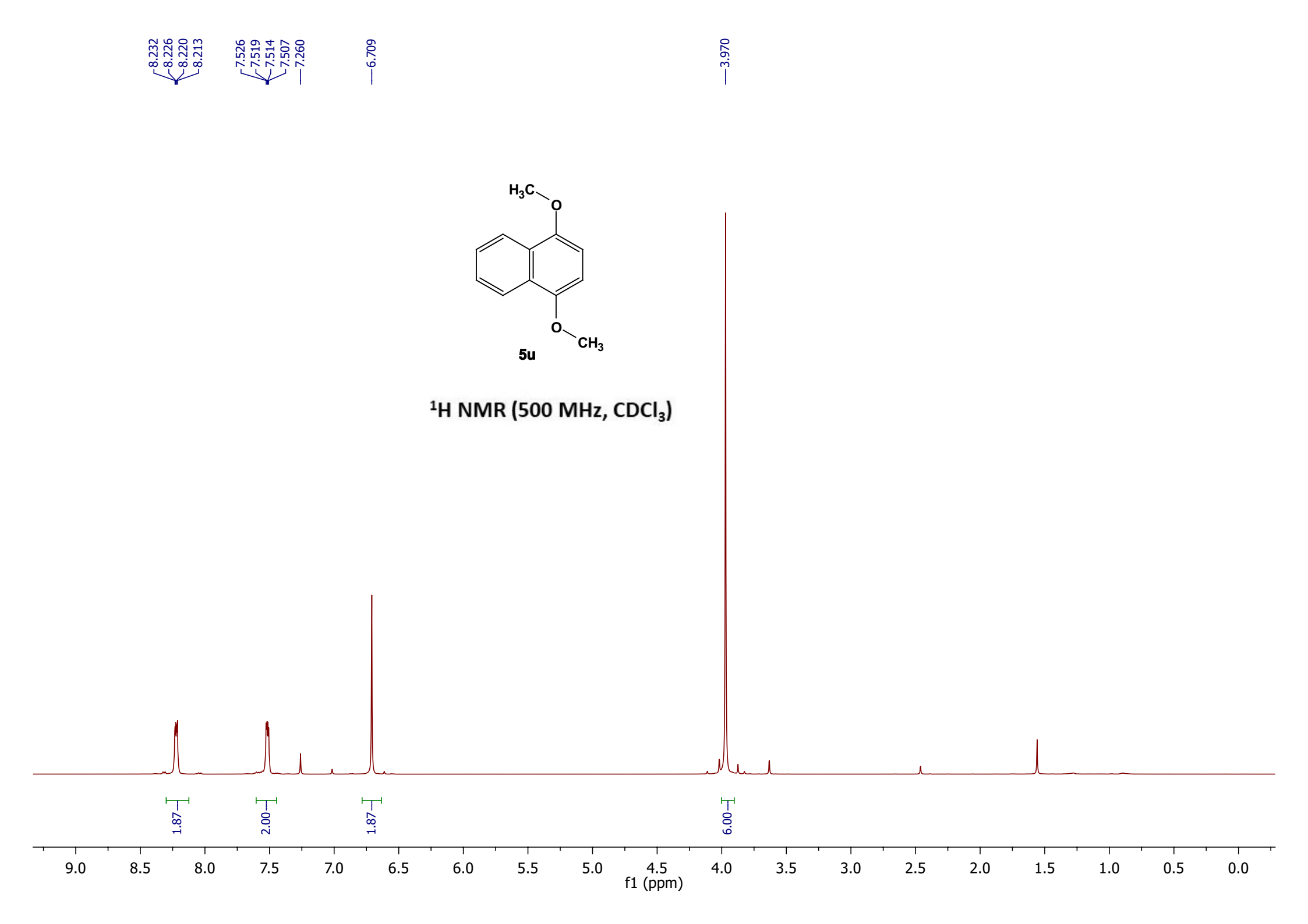

149.620
129.336
126.431
124.330
106.479
77.415
77.160
76.907
55.975
CH3
O
CH3
O
5s
13C NMR (125 MHz, CDCl3)
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

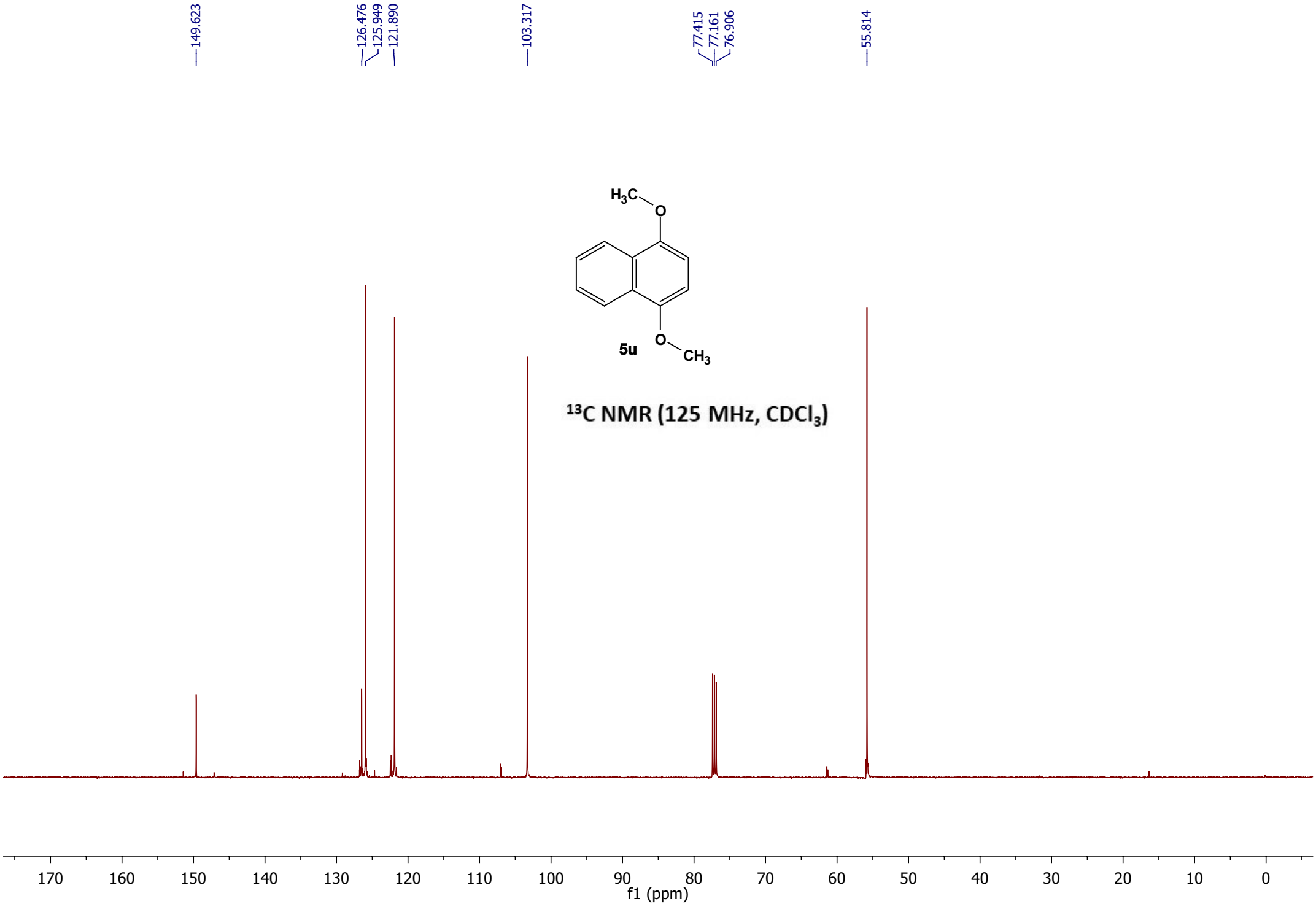


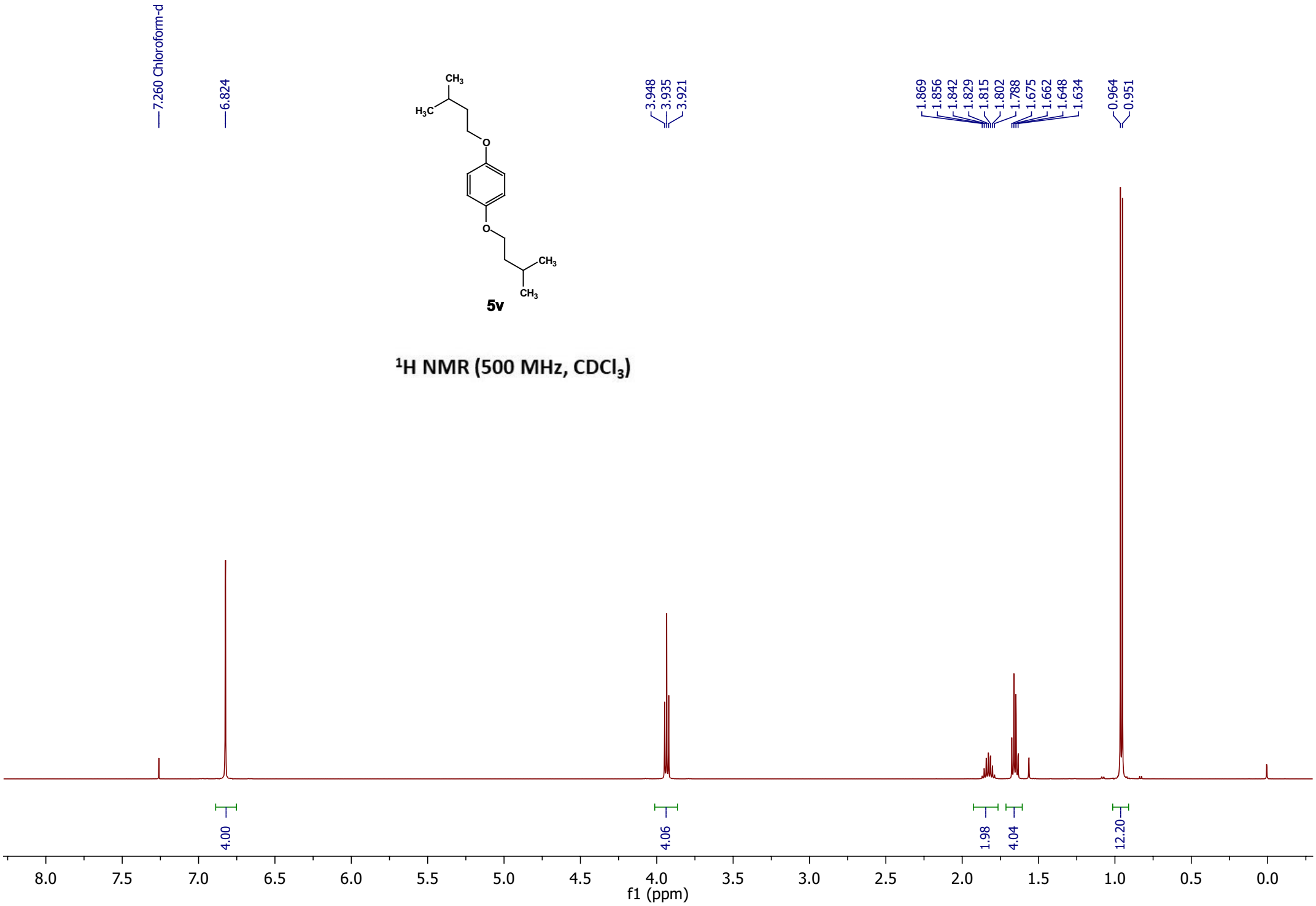
5t

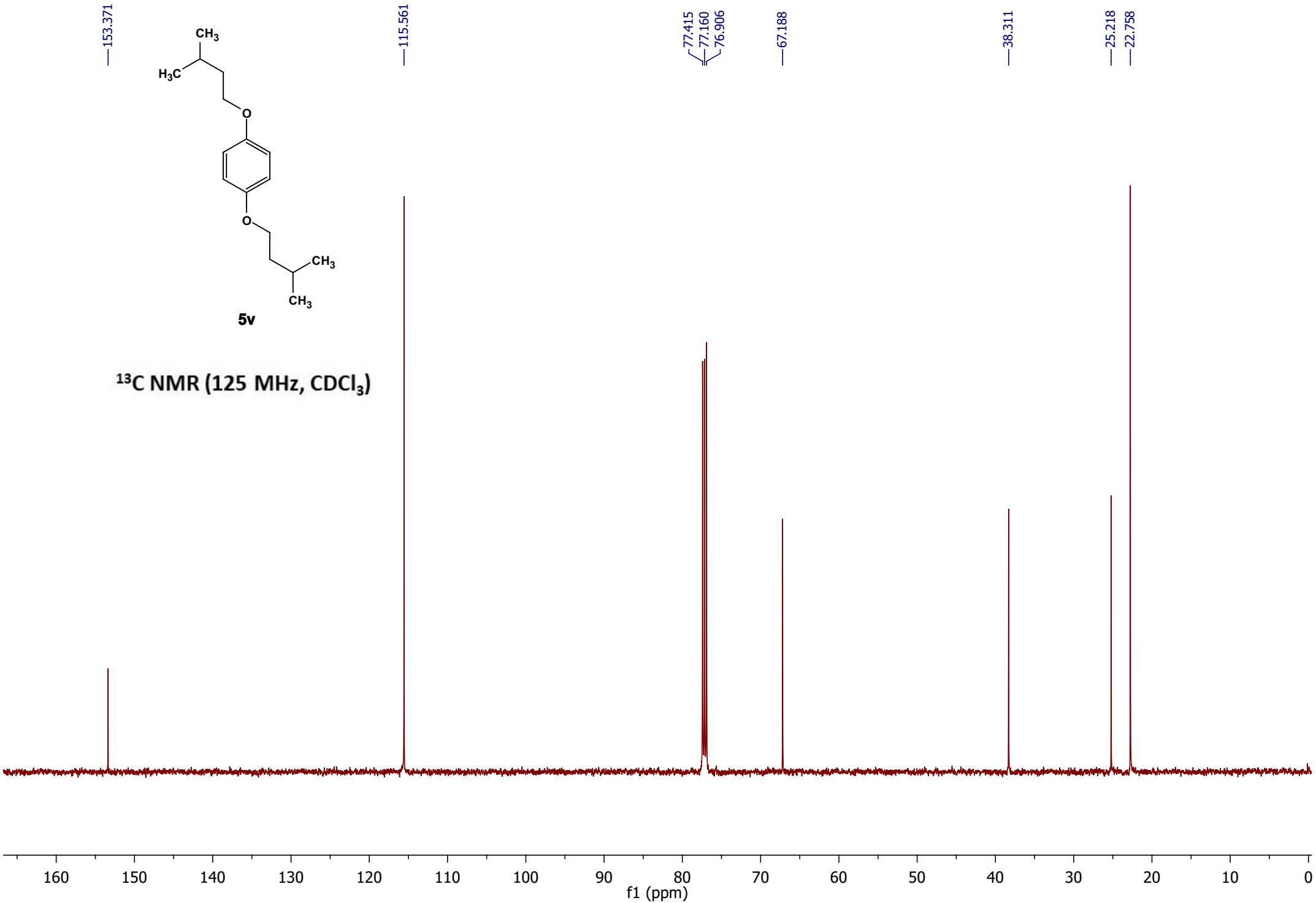
^{1}H NMR (500 MHz, $CDCl_3$)

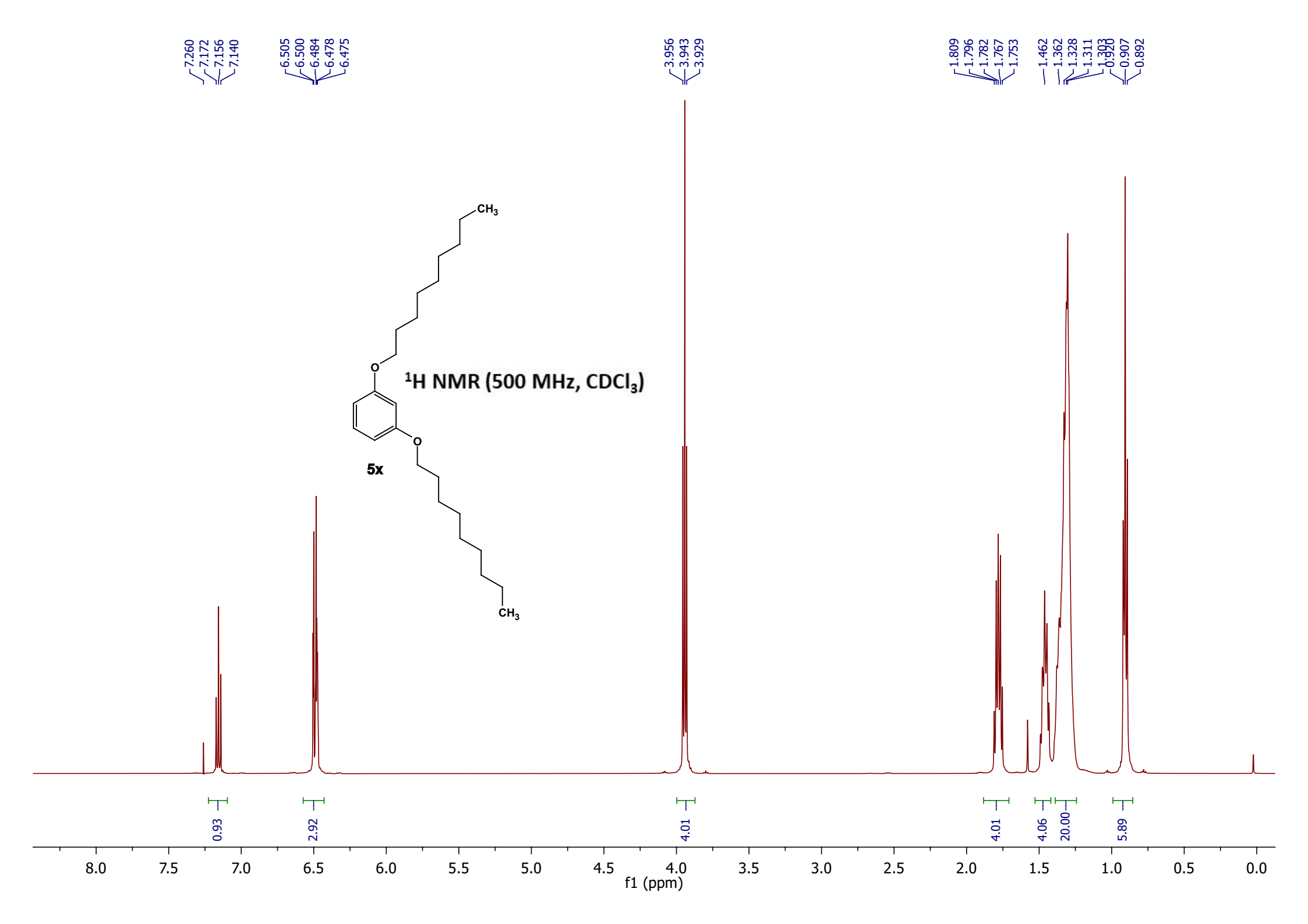

152.365
146.656
136.160
132.330
131.397
130.731
128.794
128.233
127.413
126.695
125.943
125.358
124.020
106.936
77.414
77.160
76.907
61.129
55.848
CH3
O
O
CH3
5t
13C NMR (125 MHz, CDCl3)
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

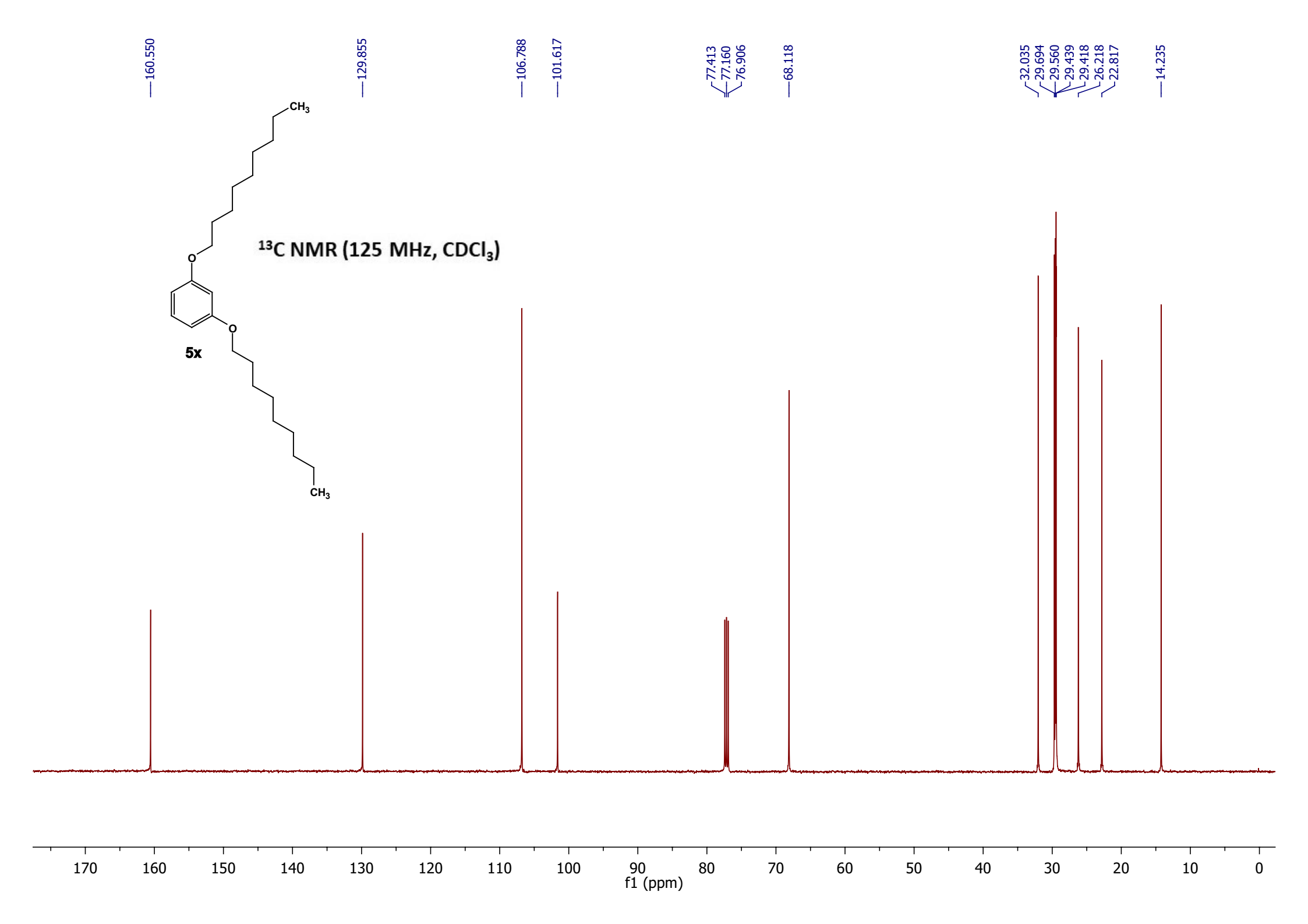

8.232
8.226
8.220
8.213
7.526
7.519
7.514
7.507
7.260
6.709
3.970
H3C
O
O
CH3
5u
1H NMR (500 MHz, CDCl3)
1.87
2.00
1.87
6.00
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

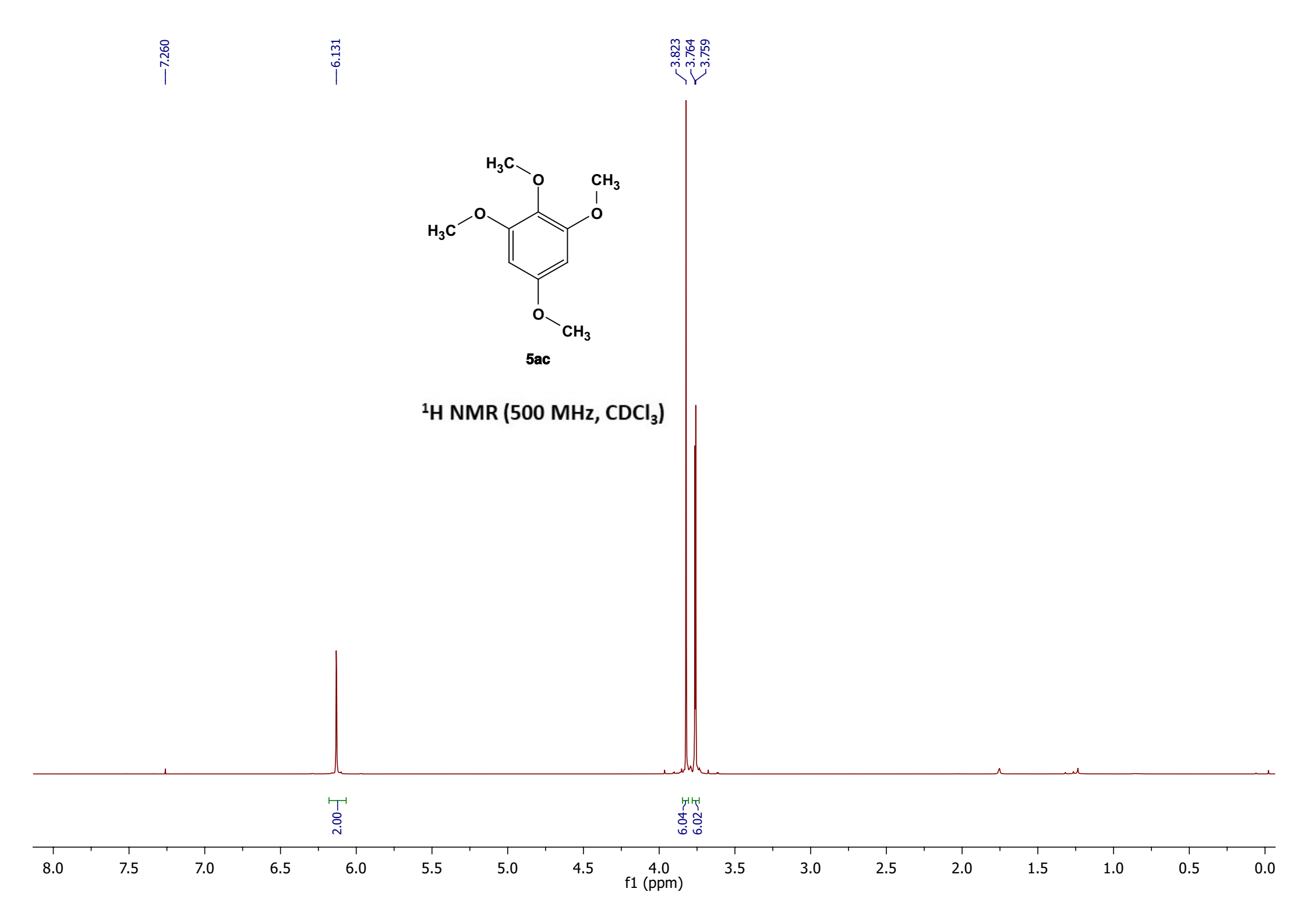

149.623
126.476
125.949
121.890
103.317
77.415
77.161
76.906
55.814
H3C
O
O
CH3
5u
13C NMR (125 MHz, CDCl3)
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

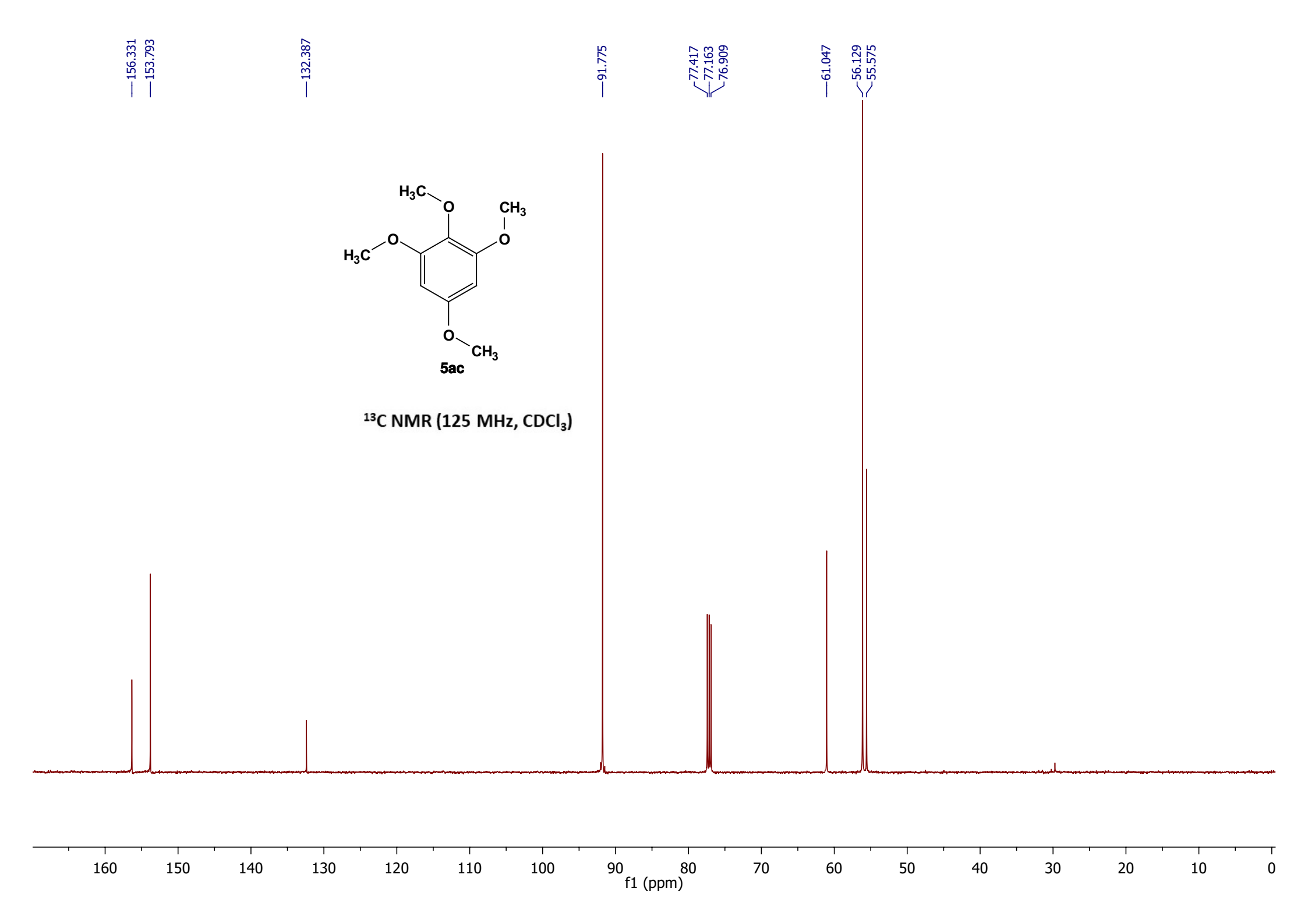

7.260 Chloroform-d
6.824
3.948
3.935
3.921
1.869
1.856
1.842
1.829
1.815
1.802
1.788
1.675
1.662
1.648
1.634
0.964
0.951
CH3
H3C
O
O
CH3
CH3
5v
1H NMR (500 MHz, CDCl3)
4.00
4.06
1.98
4.04
12.20
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

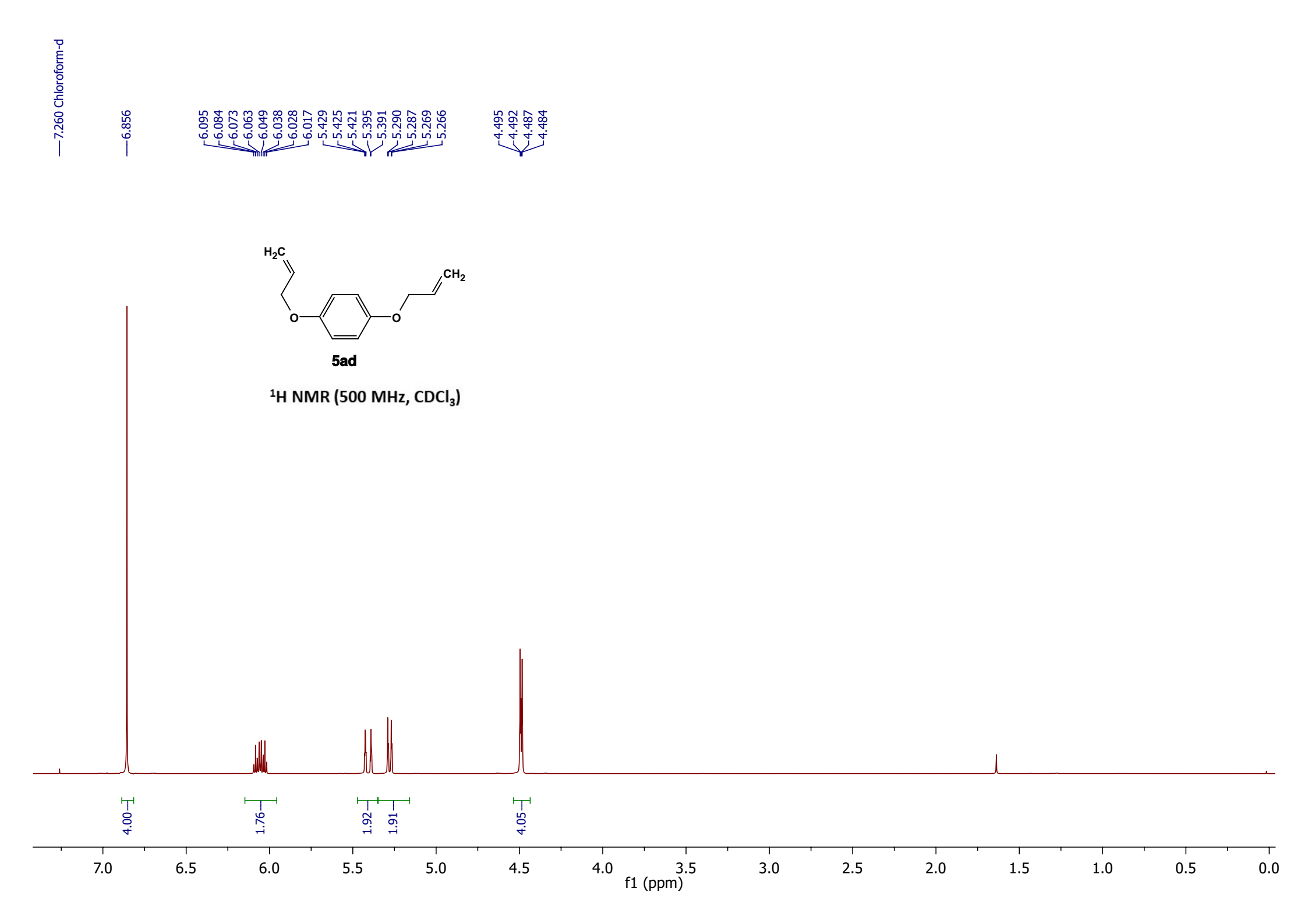

153.371
115.561
77.415
77.160
76.906
67.188
38.311
25.218
22.758
CH3
H3C
O
O
CH3
CH3
5v
13C NMR (125 MHz, CDCl3)
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

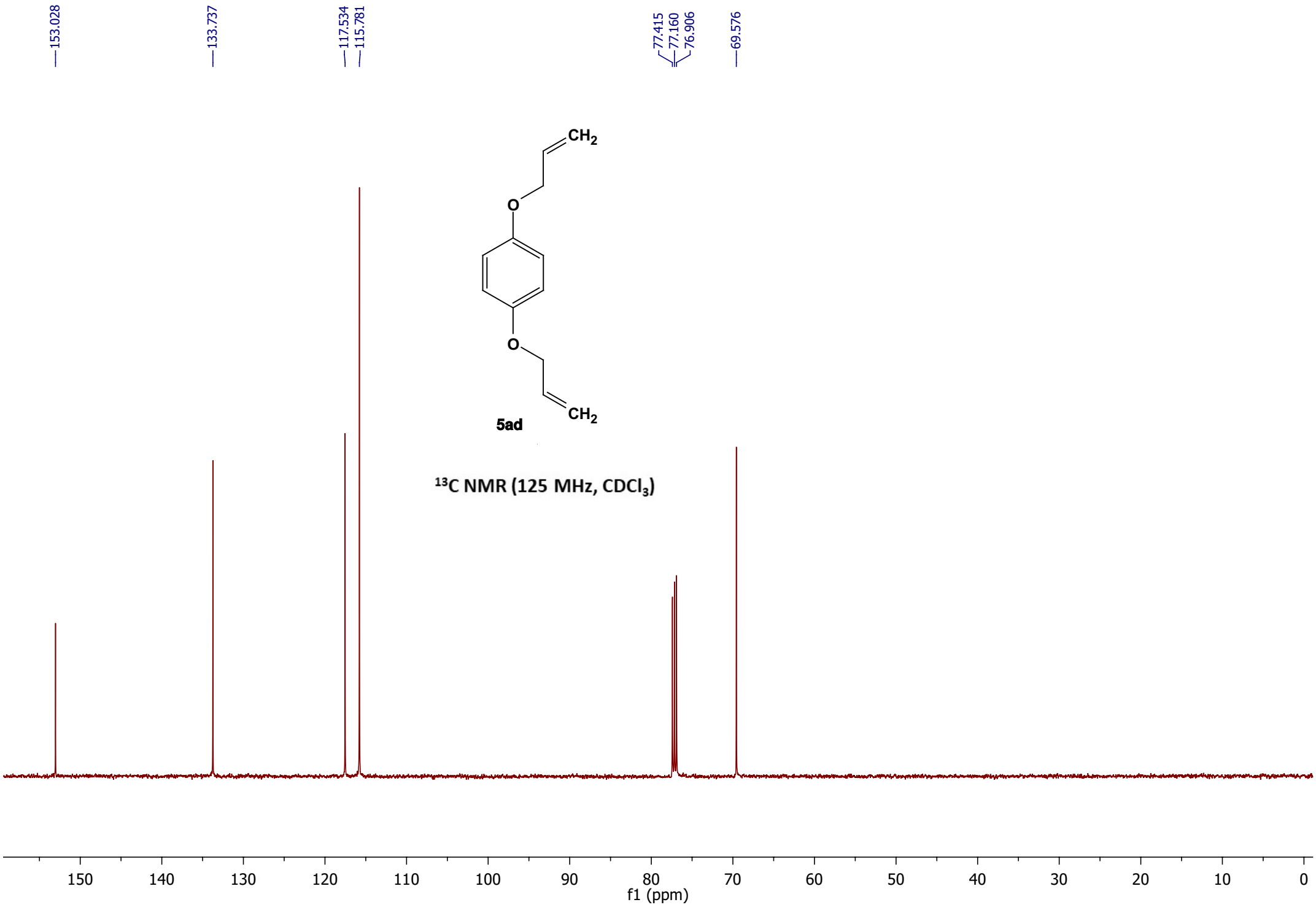

7.260
7.172
7.156
7.140
6.505
6.500
6.484
6.478
6.475
3.956
3.943
3.929
1.809
1.796
1.782
1.767
1.753
1.462
1.362
1.328
1.311
0.920
0.907
0.892
CH3
O
O
CH3
5x
1H NMR (500 MHz, CDCl3)
0.93
2.92
4.01
4.01
4.06
20.00
5.89
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

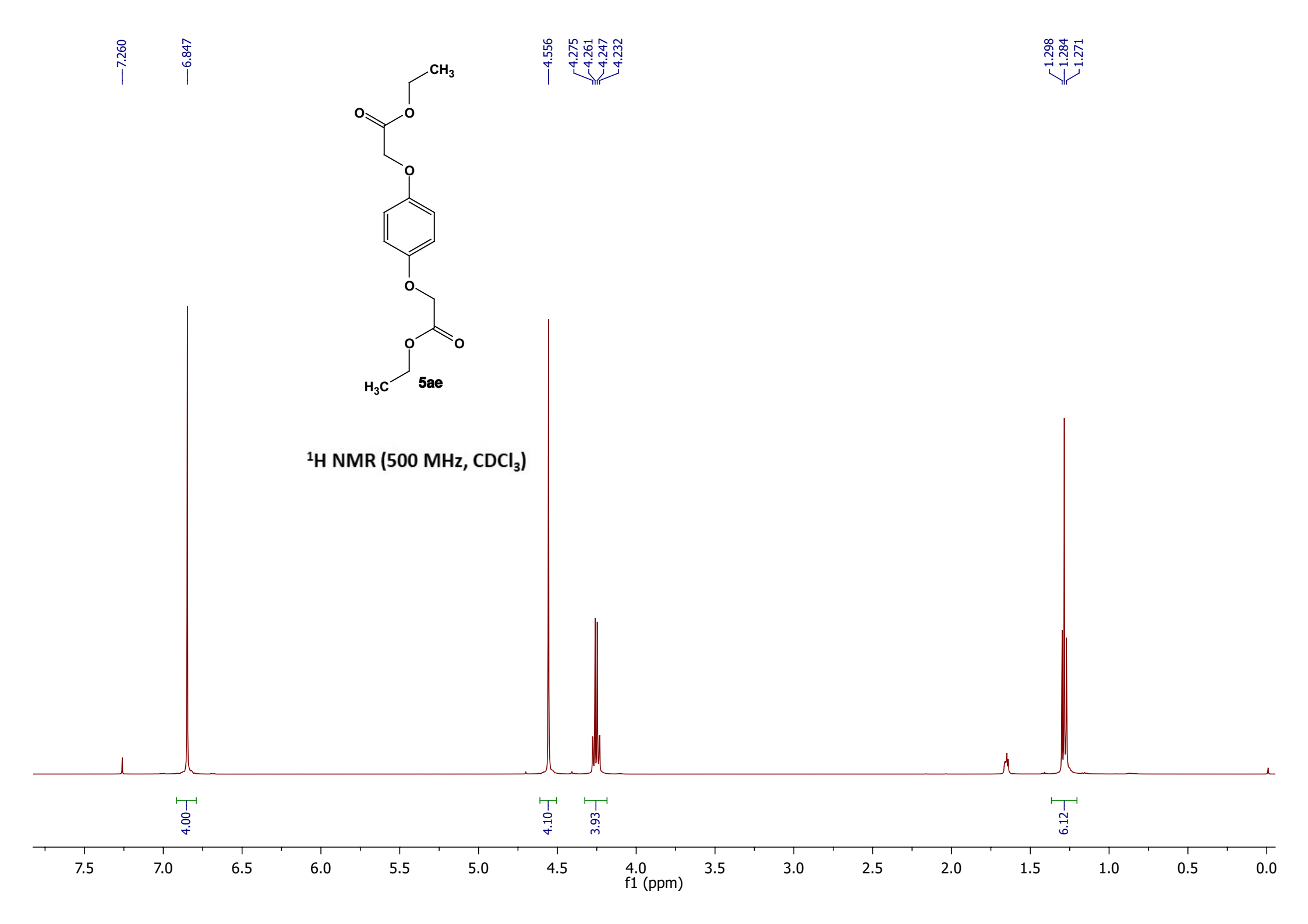

160.550
129.855
106.788
101.617
77.413
77.160
76.906
68.118
32.035
29.694
29.560
29.439
29.418
26.218
22.817
14.235
^{13}C NMR (125 MHz, $CDCl_3$)
5x
CH3
O
O
CH3
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

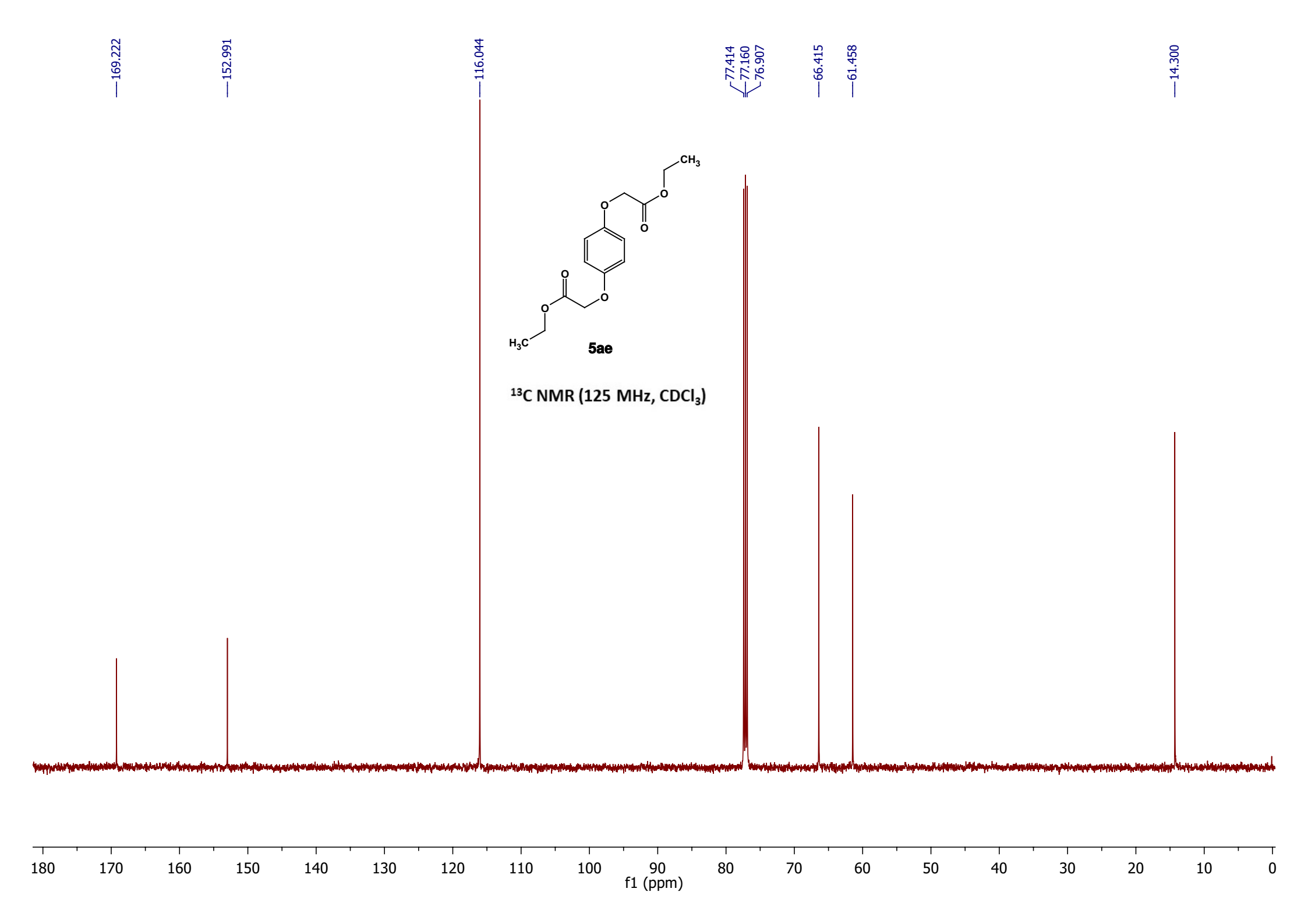

7.260
6.131
3.823
3.764
3.759
H3C
O
CH3
O
H3C
O
O
CH3
5ac
1H NMR (500 MHz, CDCl3)
2.00
6.04
6.02
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

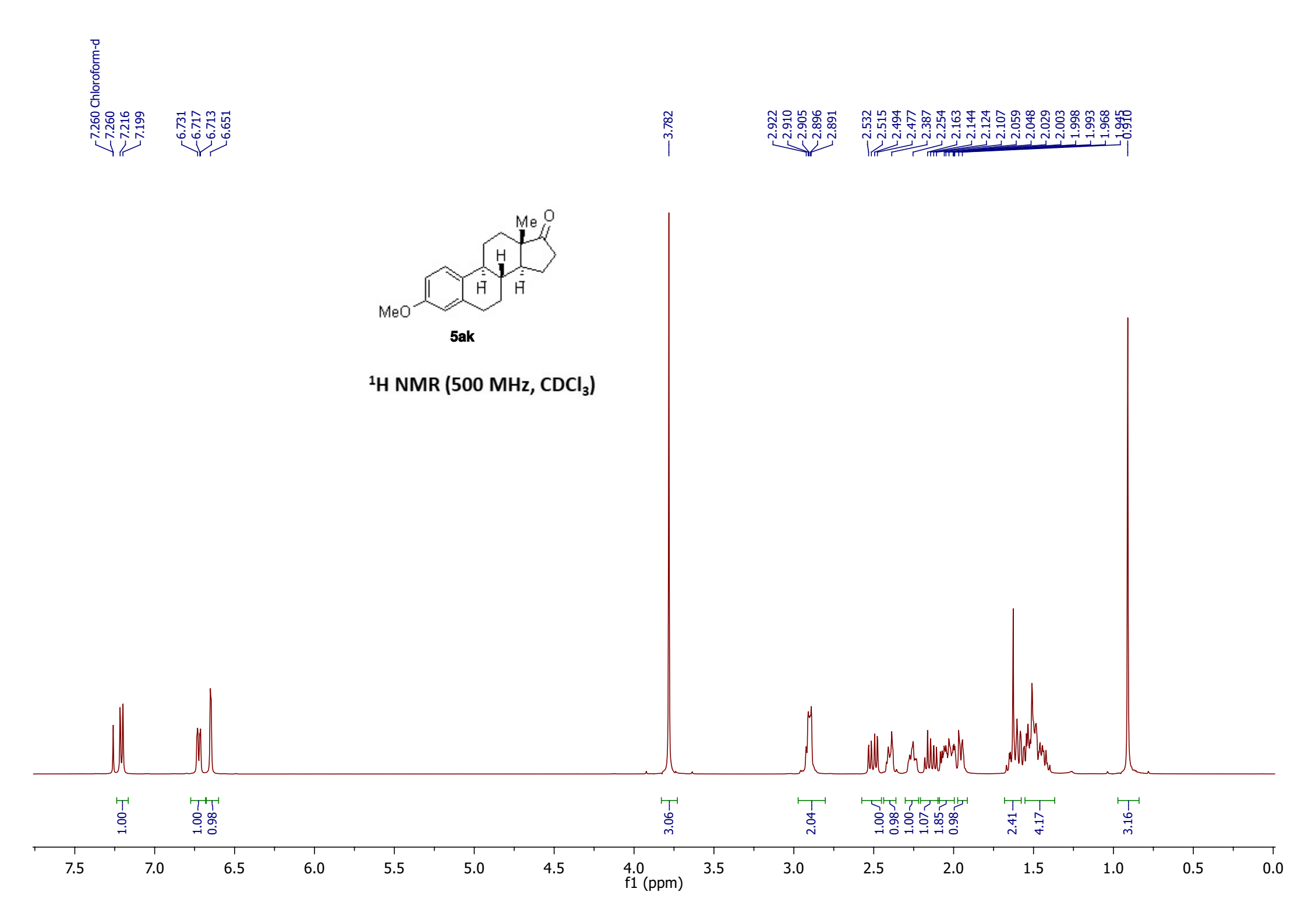

156.331
153.793
132.387
91.775
77.417
77.163
76.909
61.047
56.129
55.575
H3C
O
CH3
O
H3C
O
O
CH3
5ac
13C NMR (125 MHz, CDCl3)
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

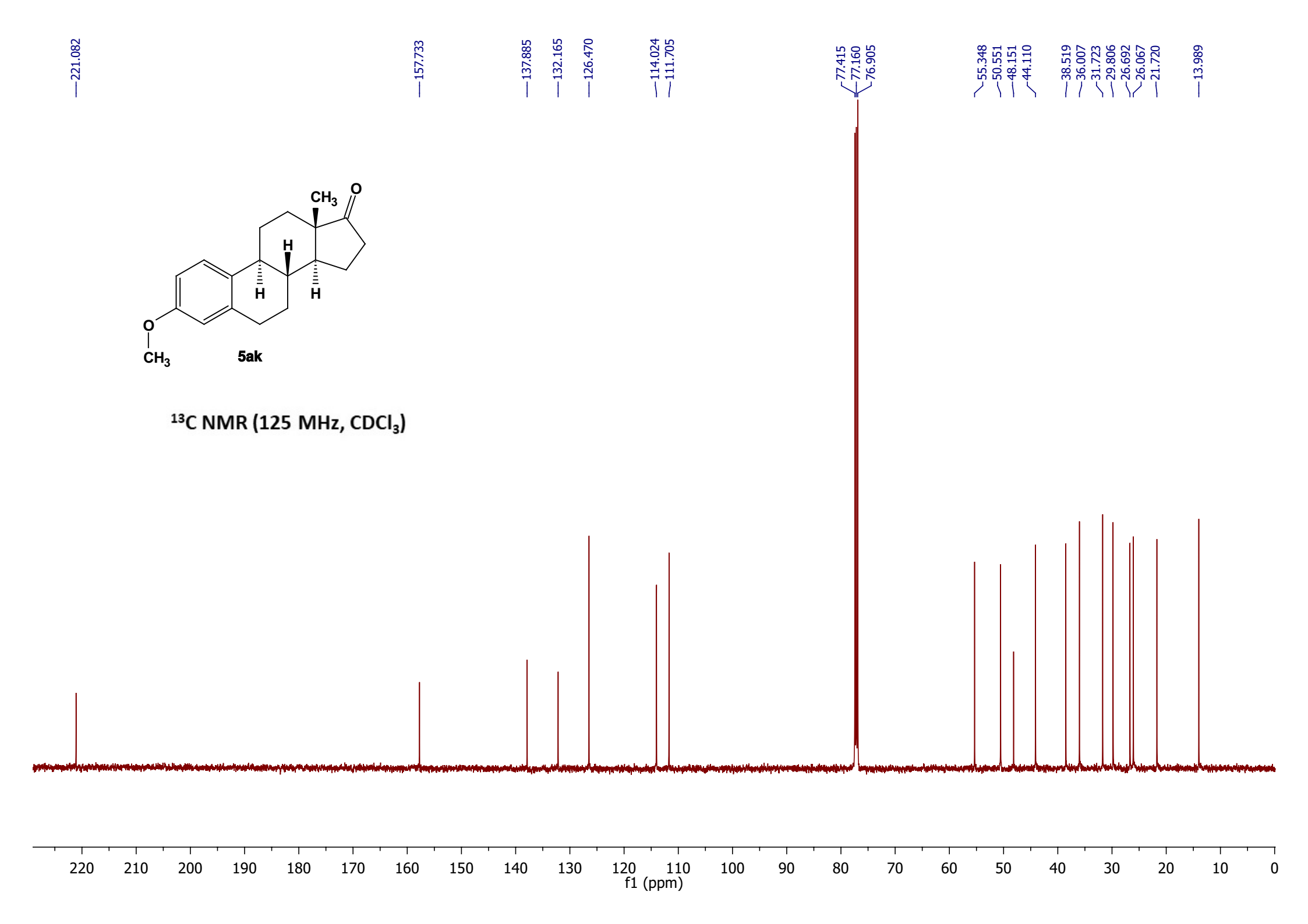

7.260 Chloroform-d
6.856
6.095
6.084
6.073
6.063
6.049
6.038
6.028
6.017
5.429
5.425
5.421
5.395
5.391
5.290
5.287
5.269
5.266
4.495
4.492
4.487
4.484
H2C
CH2
O
O
5ad
1H NMR (500 MHz, CDCl3)
4.00
1.76
1.92
1.91
4.05
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

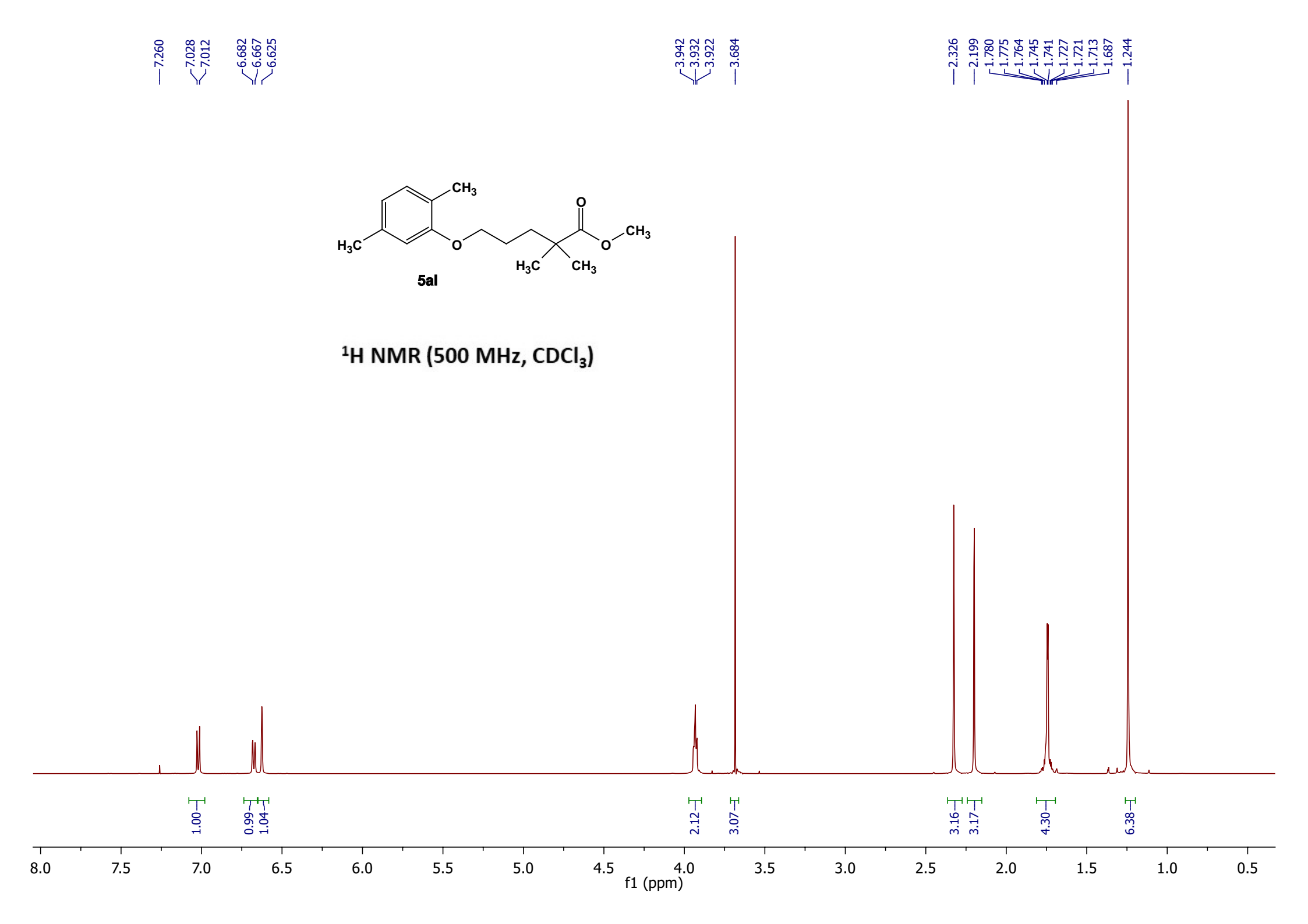

153.028
133.737
117.534
115.781
77.415
77.160
76.906
69.576
CH2
O
O
CH2
5ad
13C NMR (125 MHz, CDCl3)
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

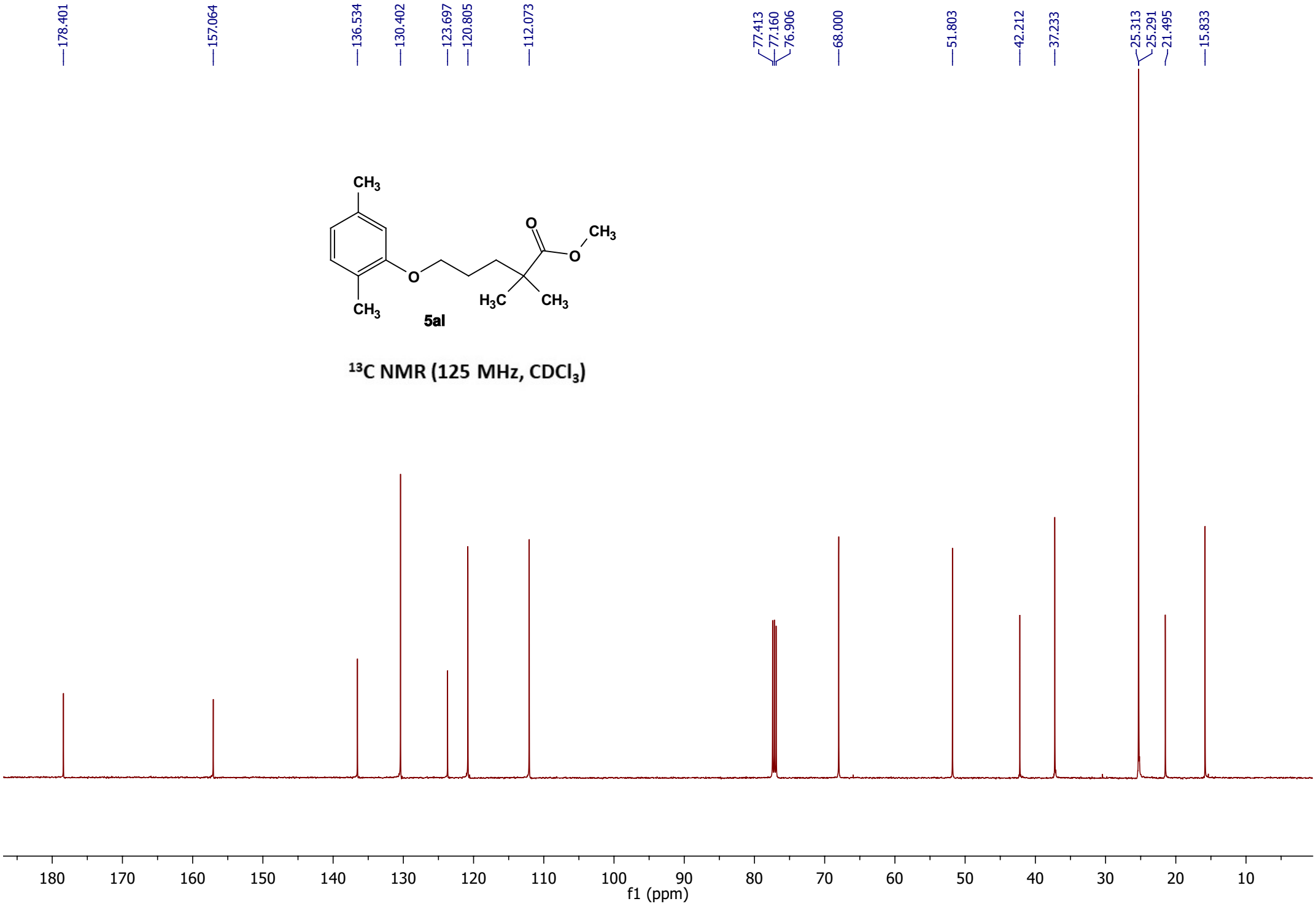

7.260
6.847
4.556
4.275
4.261
4.247
4.232
1.298
1.284
1.271
CH3
O
O
O
O
O
O
H3C
5ae
1H NMR (500 MHz, CDCl3)
4.00
4.10
3.93
6.12
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

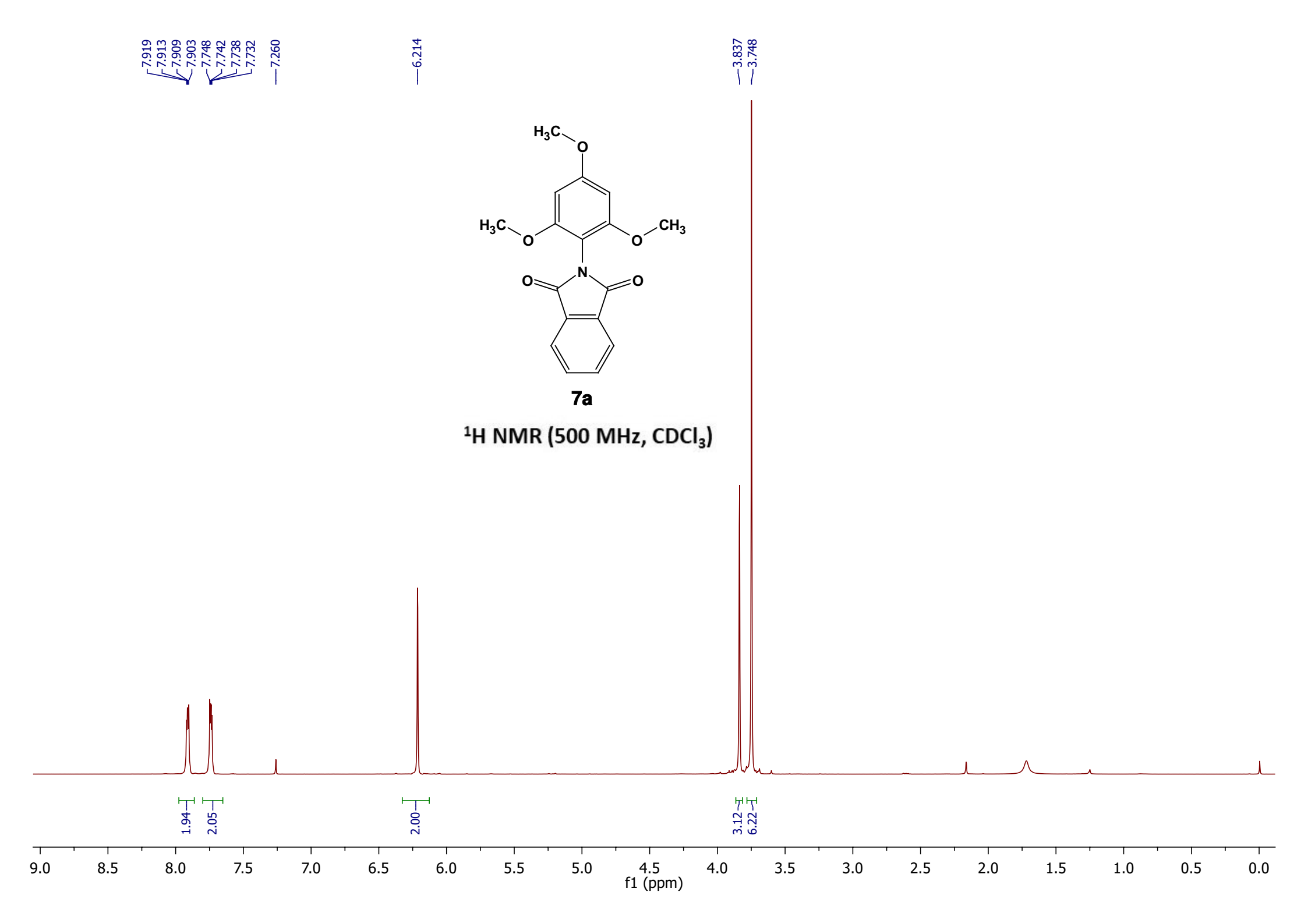

169.222
152.991
116.044
77.414
77.160
76.907
66.415
61.458
14.300
CH3
O
O
O
O
O
O
H3C
5ae
13C NMR (125 MHz, CDCl3)
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

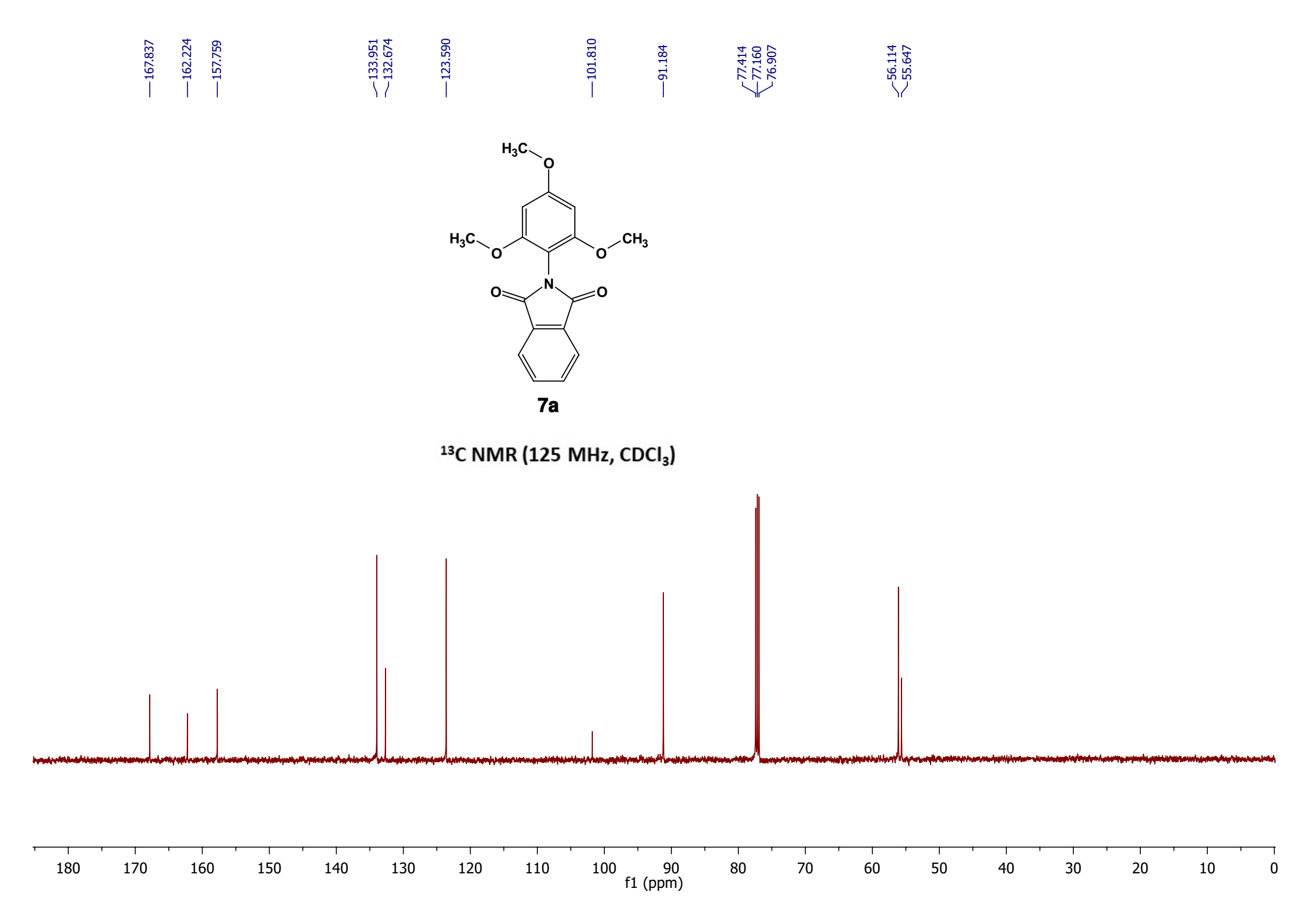

7.260 Chloroform-d
7.260
7.216
7.199
6.731
6.717
6.713
6.651
3.782
2.922
2.910
2.905
2.896
2.891
2.532
2.515
2.494
2.477
2.387
2.254
2.163
2.144
2.124
2.107
2.059
2.048
2.029
2.003
1.998
1.993
1.968
1.945
0.910
Me
O
H
H
H
MeO
5ak
¹H NMR (500 MHz, CDCl₃)
1.00
1.00
0.98
3.06
2.04
1.00
0.98
1.00
1.07
1.85
0.98
2.41
4.17
3.16
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)

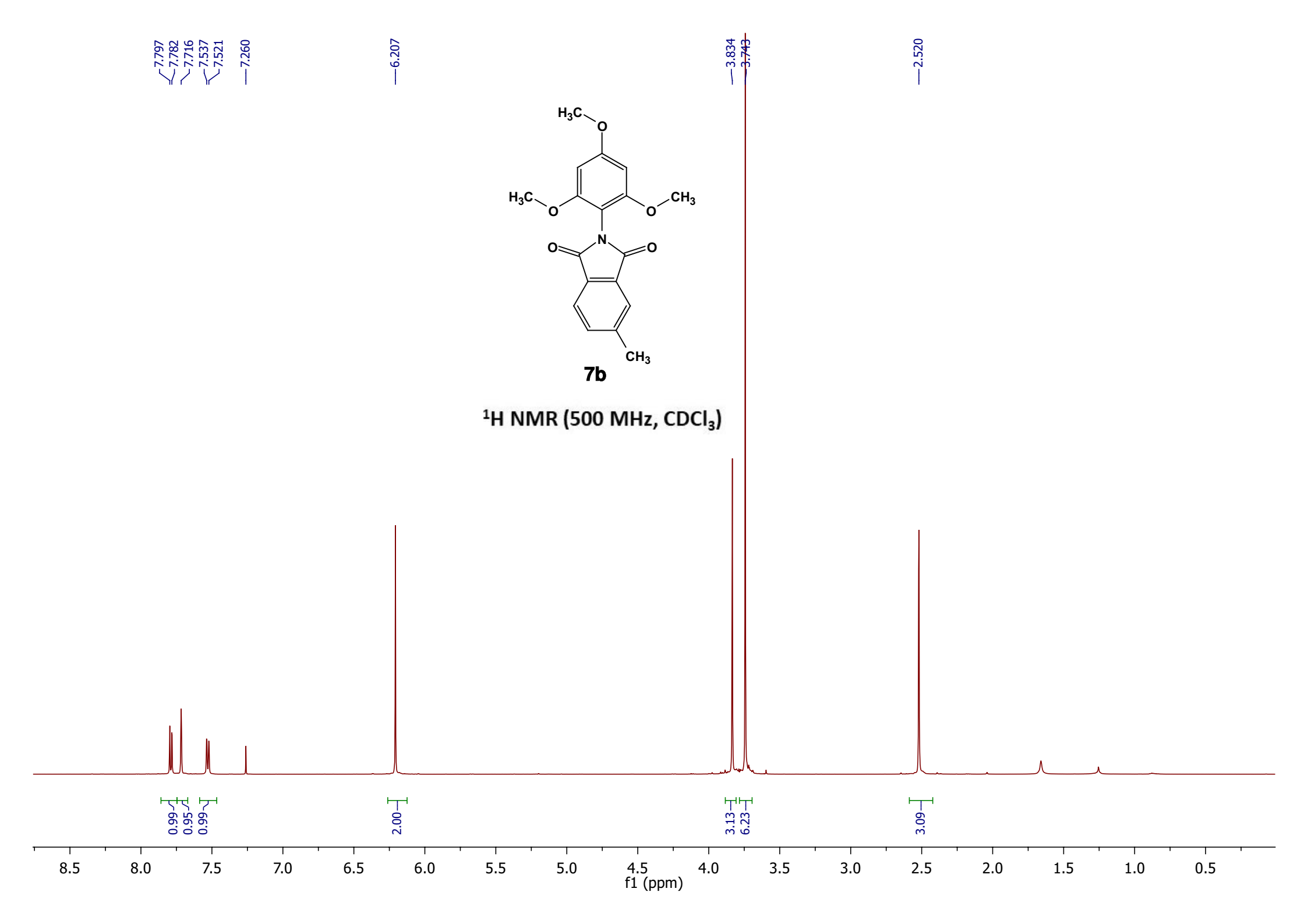

221.082
157.733
137.885
132.165
126.470
114.024
111.705
77.415
77.160
76.905
55.348
50.551
48.151
44.110
38.519
36.007
31.723
29.806
26.692
26.067
21.720
13.989
CH3
O
H
H
H
O
CH3
5ak
13C NMR (125 MHz, CDCl3)
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

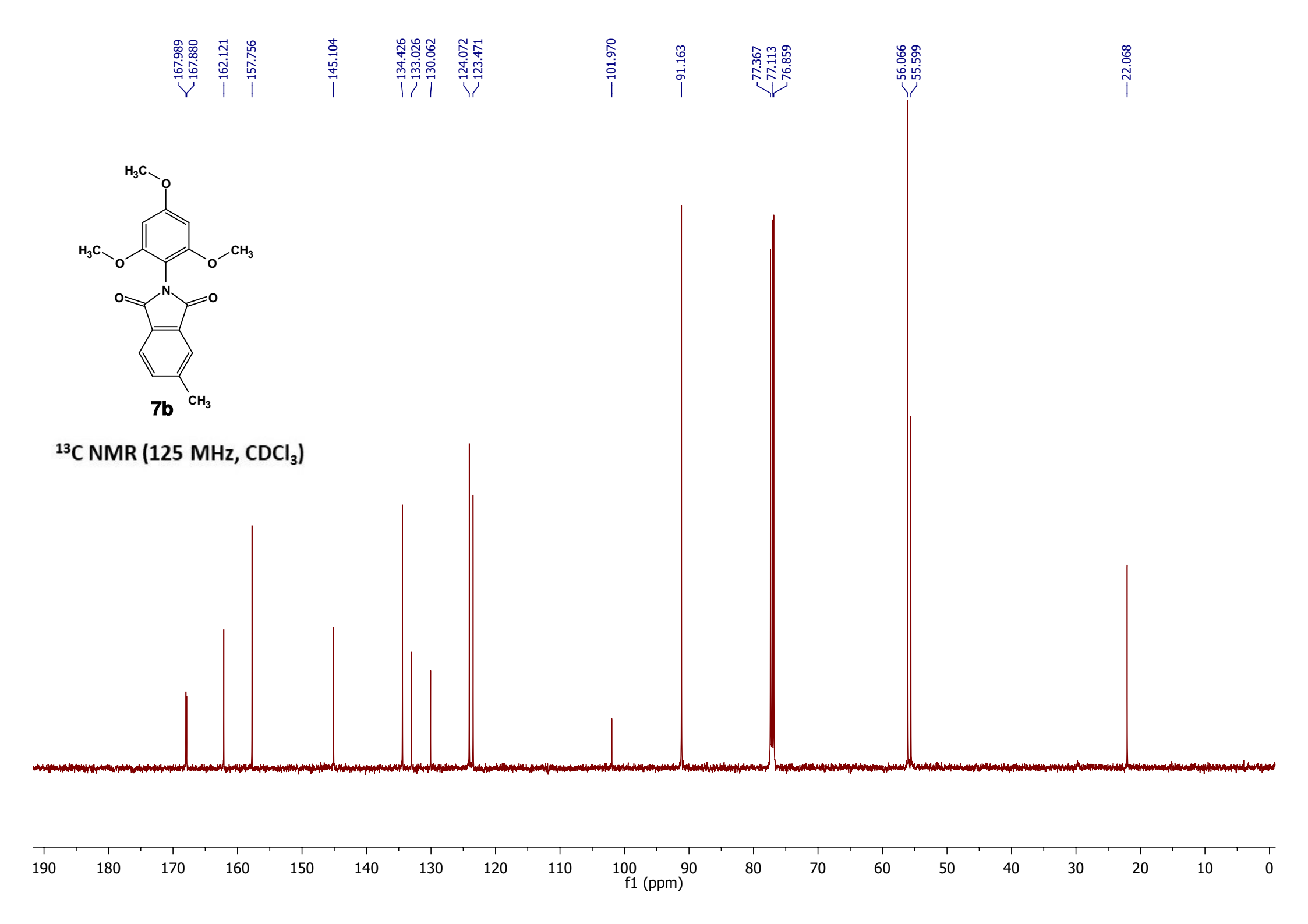

7.260
7.028
7.012
6.682
6.667
6.625
3.942
3.932
3.922
3.684
2.326
2.199
1.780
1.775
1.764
1.745
1.741
1.727
1.721
1.713
1.687
1.244
CH3
O
H3C
O
O
CH3
H3C
CH3
5al
1H NMR (500 MHz, CDCl3)
1.00
0.99
1.04
2.12
3.07
3.16
3.17
4.30
6.38
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

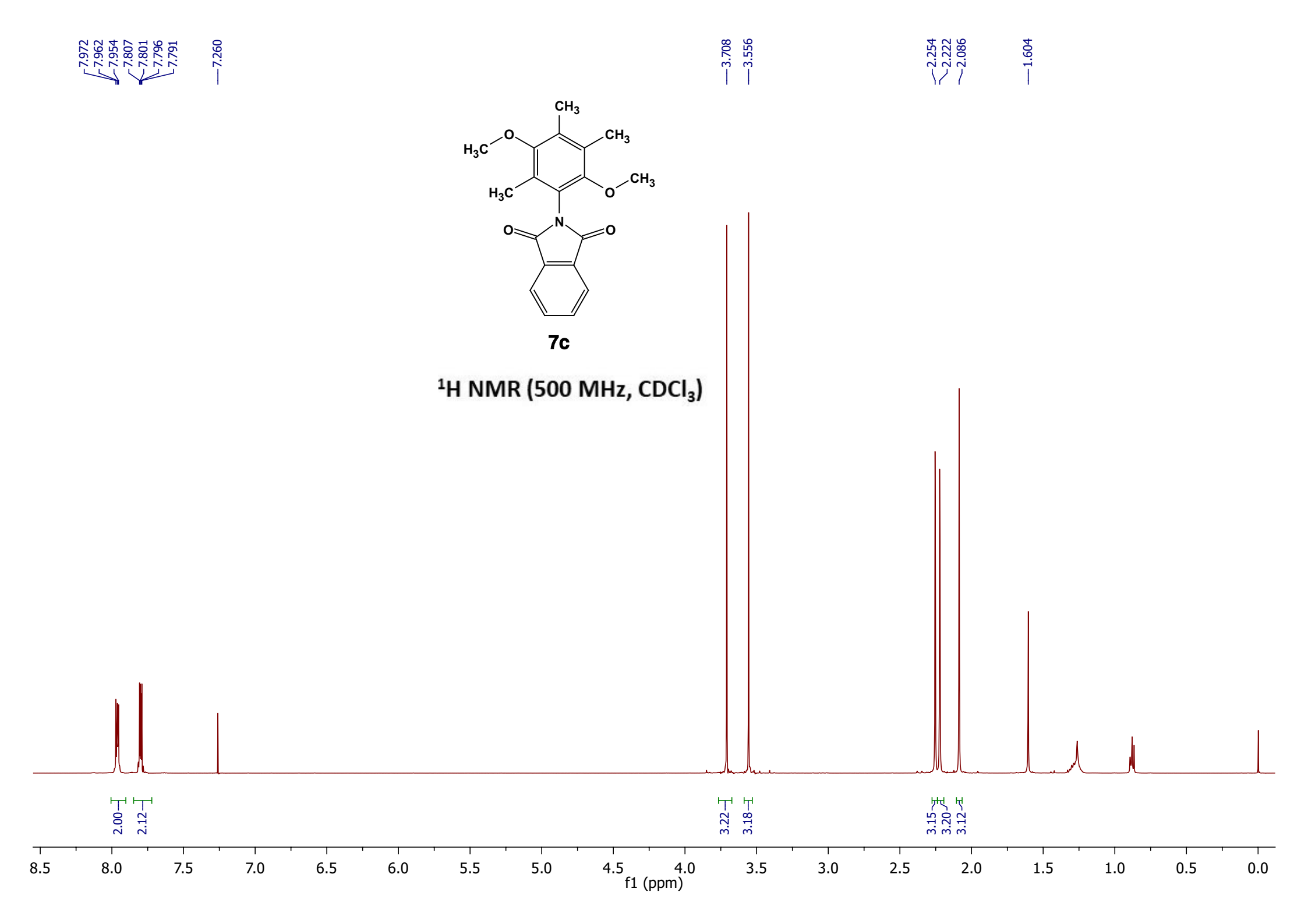

178.401
157.064
136.534
130.402
123.697
120.805
112.073
77.413
77.160
76.906
68.000
51.803
42.212
37.233
25.313
25.291
21.495
15.833
CH3
O
CH3
O
O
CH3
H3C
CH3
5al
13C NMR (125 MHz, CDCl3)
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

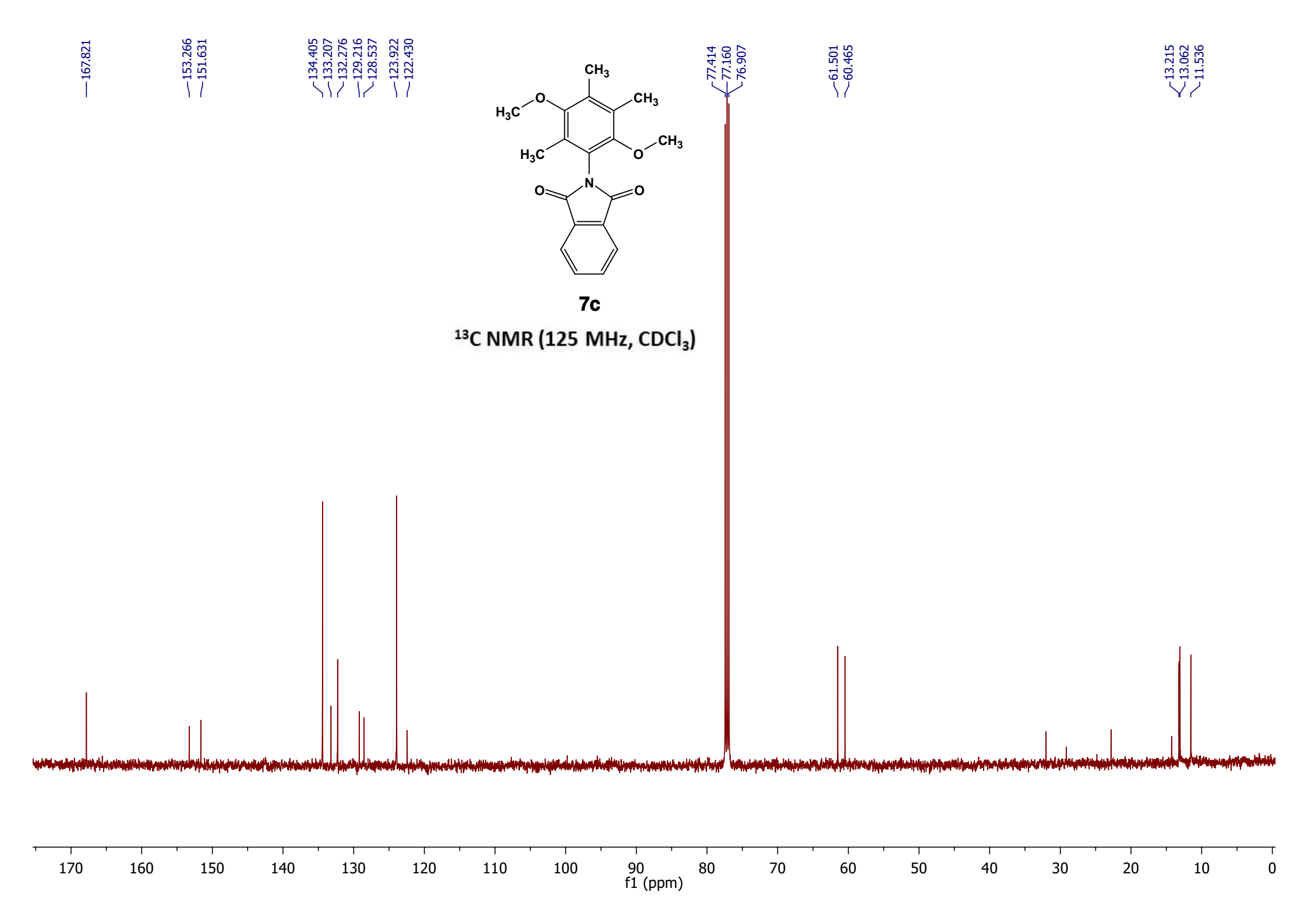

7.919
7.913
7.909
7.903
7.748
7.742
7.738
7.732
7.260
6.214
3.837
3.748
H3C
O
H3C
O
O
CH3
N
O
O
7a
1H NMR (500 MHz, CDCl3)
1.94
2.05
2.00
3.12
6.22
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

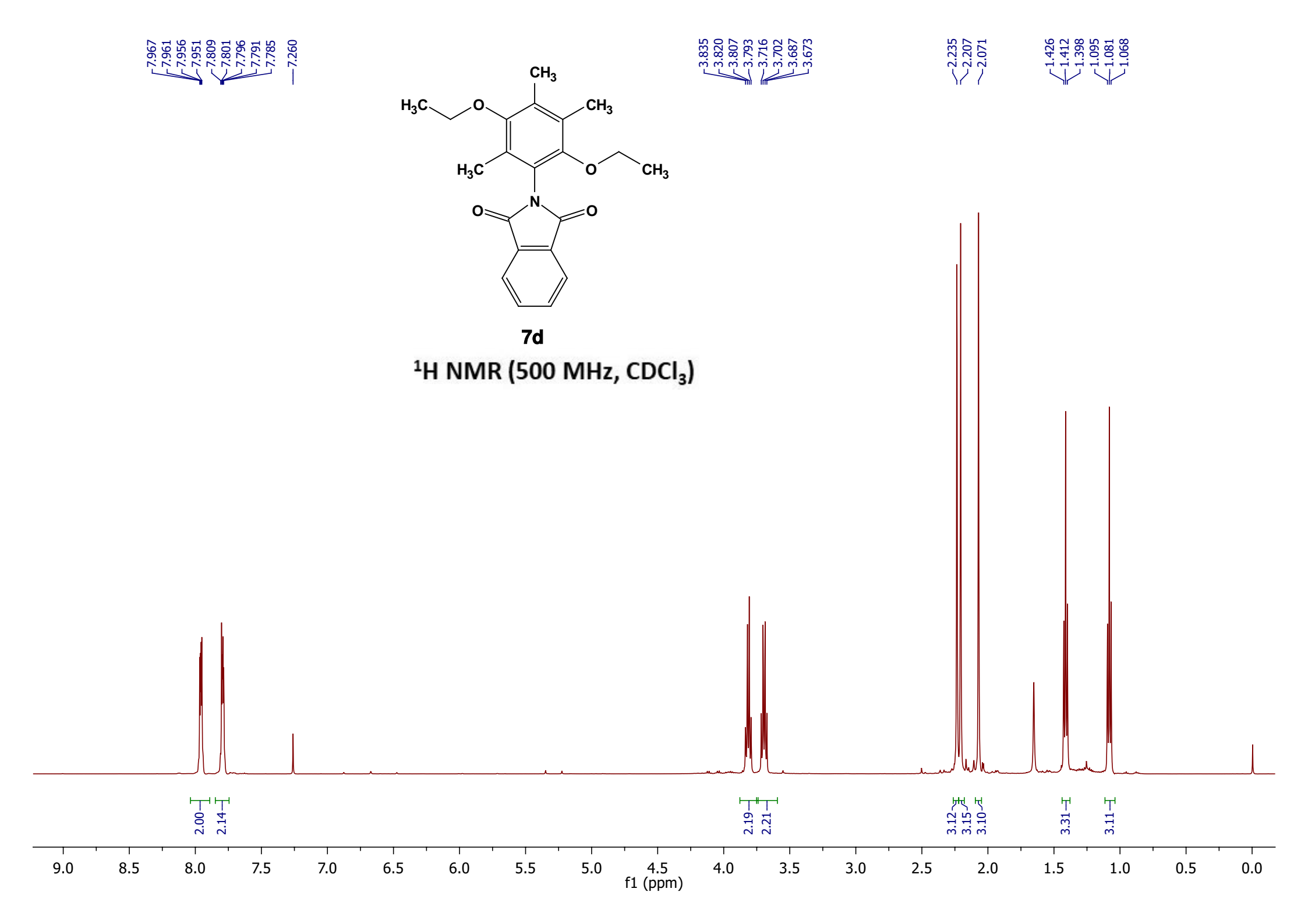

167.837
162.224
157.759
133.951
132.674
123.590
101.810
91.184
77.414
77.160
76.907
56.114
55.647
H3C
O
H3C
O
O
CH3
N
O
O
7a
13C NMR (125 MHz, CDCl3)
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

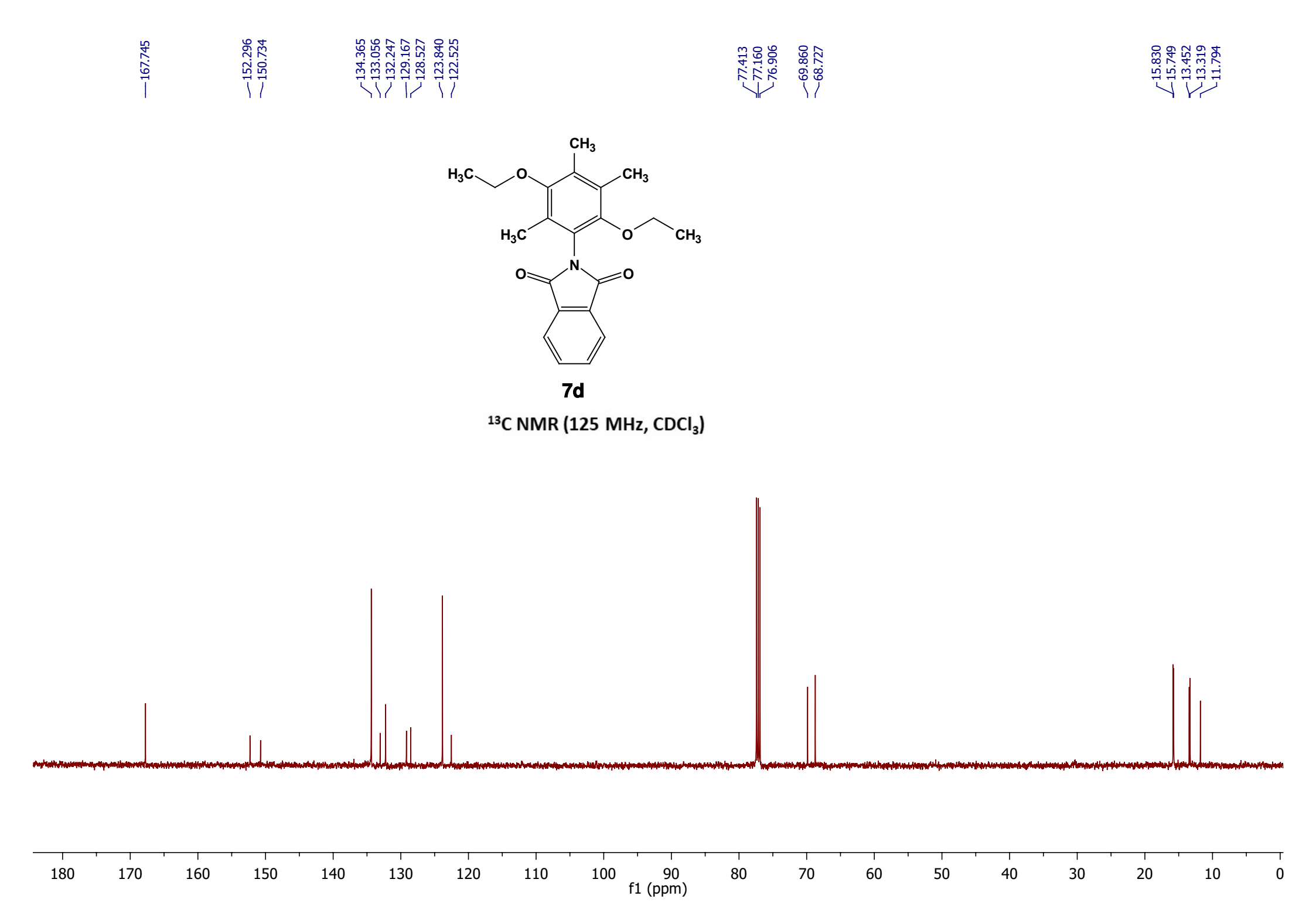

7.797
7.782
7.716
7.537
7.521
7.260
6.207
3.834
3.743
2.520
H3C
O
H3C
O
O
CH3
N
O
O
CH3
7b
1H NMR (500 MHz, CDCl3)
0.99
0.95
0.99
2.00
3.13
6.23
3.09
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

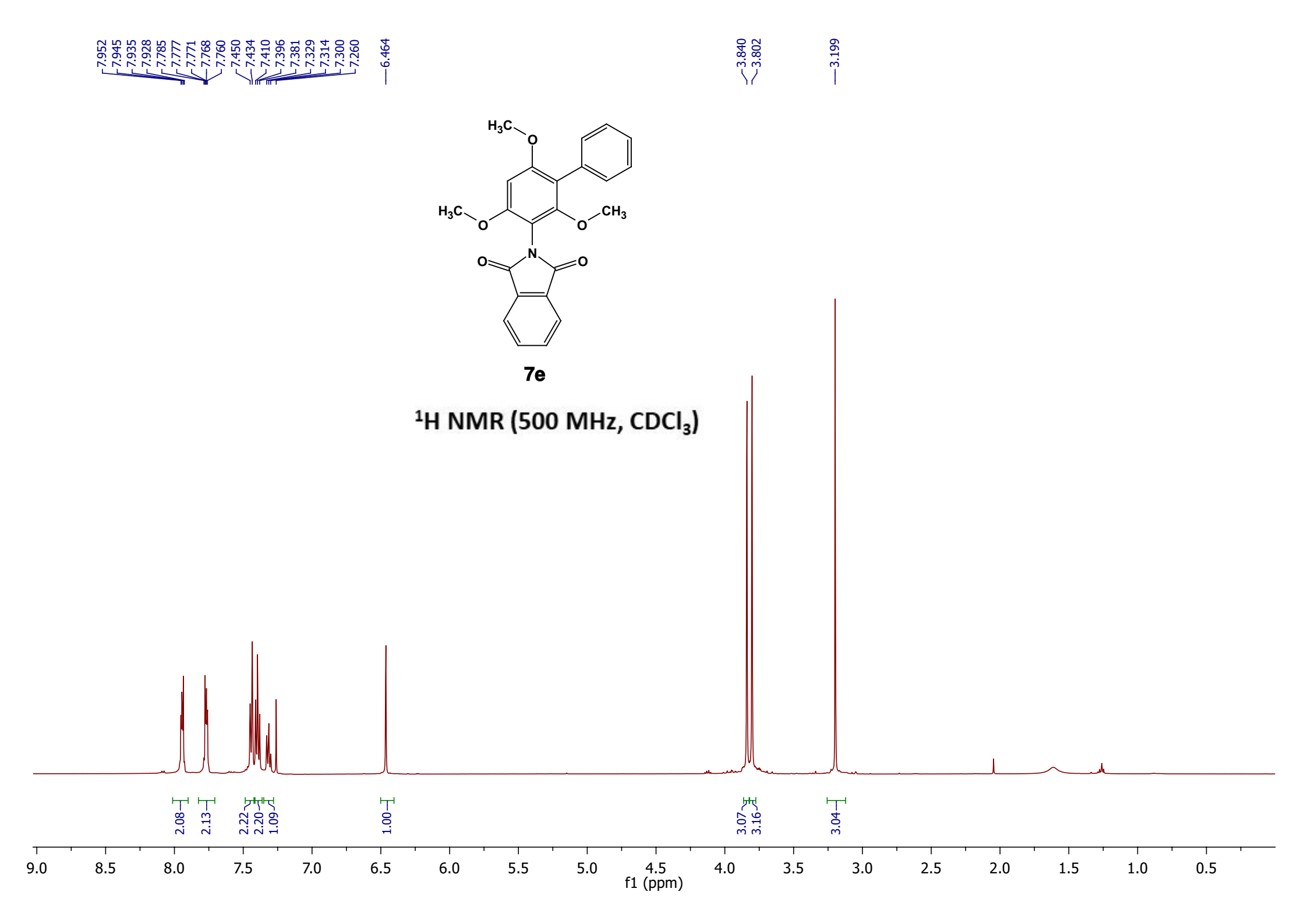

167.989
167.880
162.121
157.756
145.104
134.426
133.026
130.062
124.072
123.471
101.970
91.163
77.367
77.113
76.859
56.066
55.599
22.068
H3C
O
H3C
O
O
CH3
N
O
O
CH3
7b
13C NMR (125 MHz, CDCl3)
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

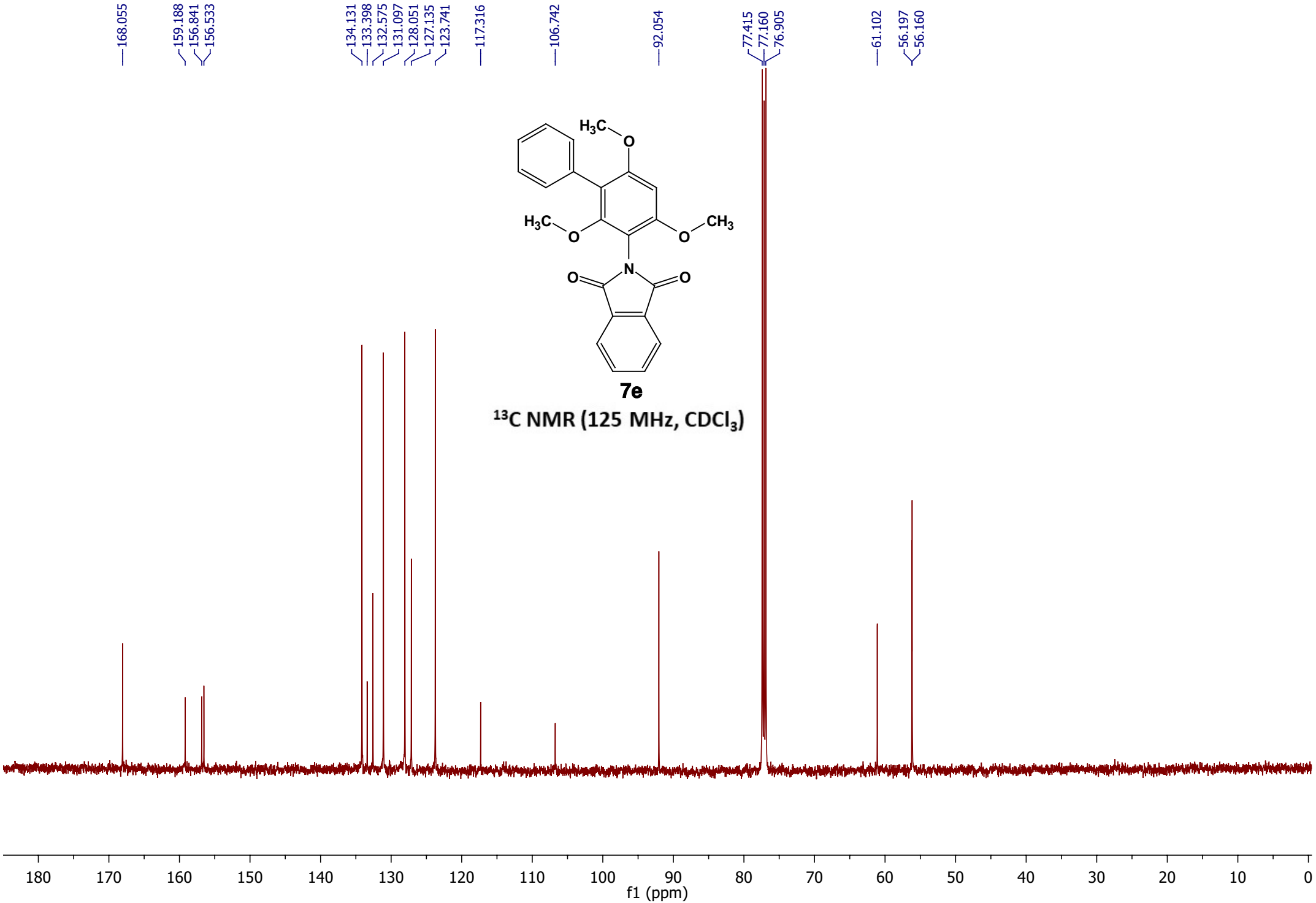

7.972
7.962
7.954
7.807
7.801
7.796
7.791
7.260
3.708
3.556
2.254
2.222
2.086
1.604
CH3
H3C
O
CH3
H3C
O
CH3
N
O
O
7c
1H NMR (500 MHz, CDCl3)
2.00
2.12
3.22
3.18
3.15
3.20
3.12
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

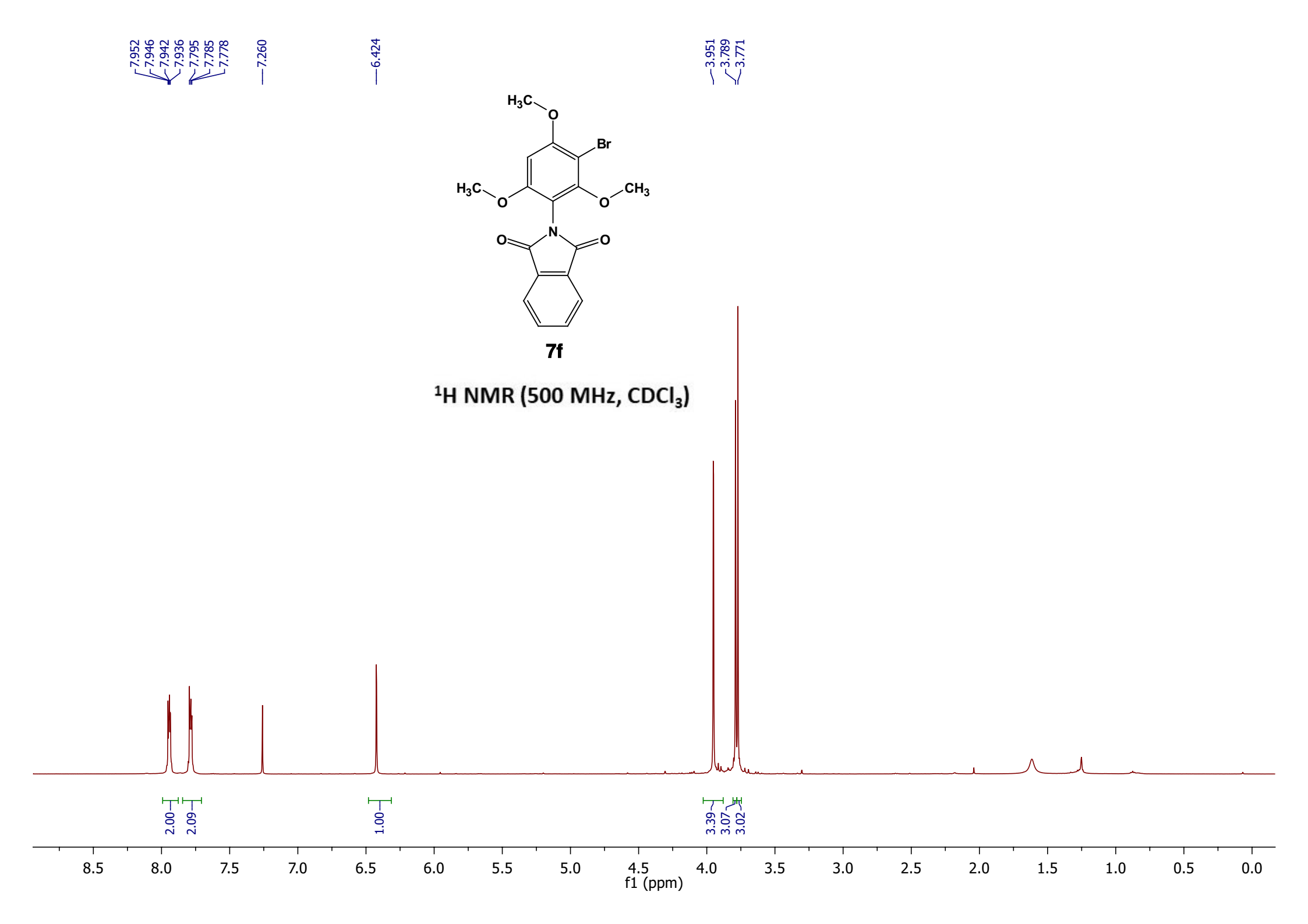

167.821
153.266
151.631
134.405
133.207
132.276
129.216
128.537
123.922
122.430
77.414
77.160
76.907
61.501
60.465
13.215
13.062
11.536
CH3
H3C
O
CH3
H3C
O
CH3
N
O
O
7c
13C NMR (125 MHz, CDCl3)
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

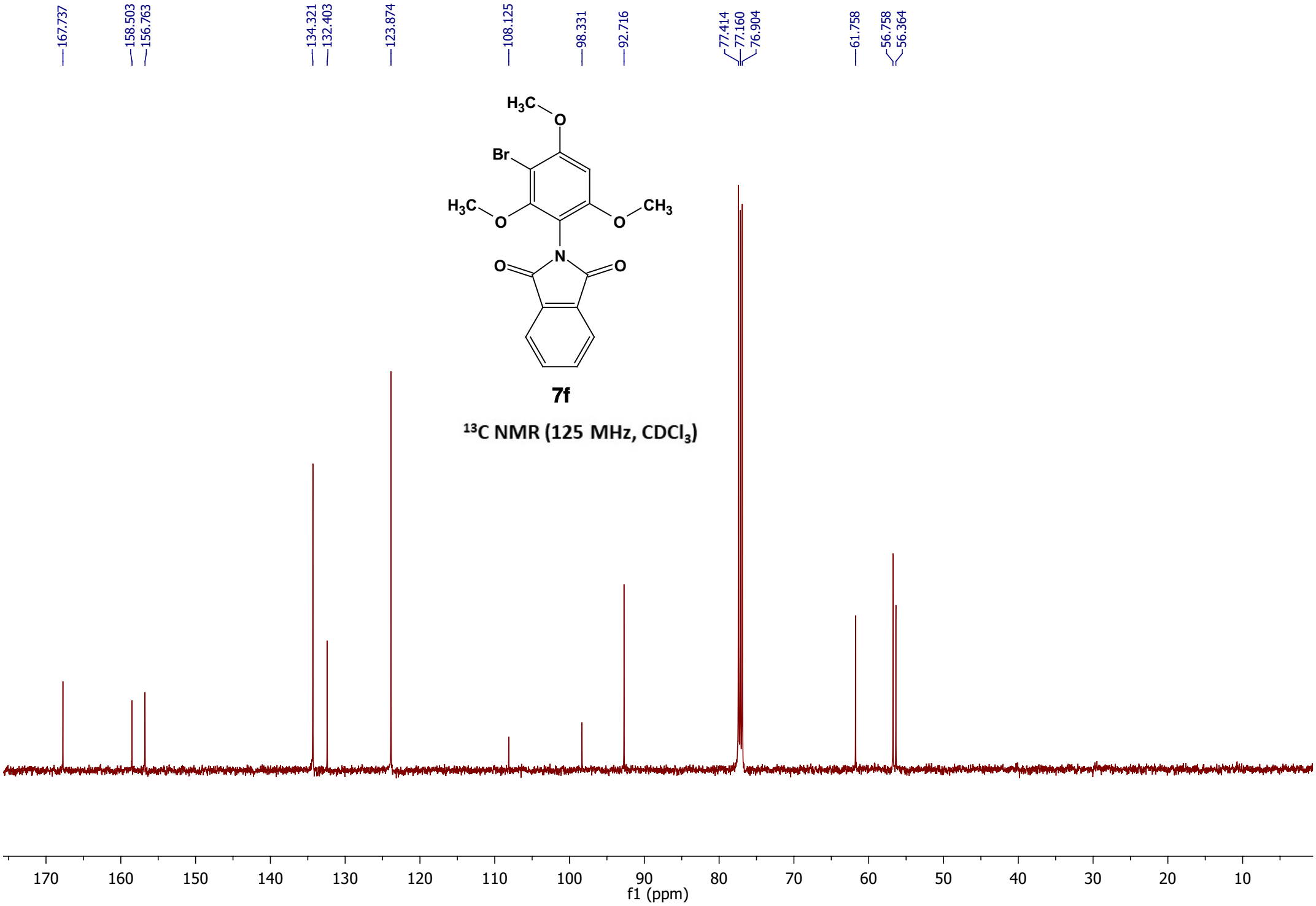

7.967
7.961
7.956
7.951
7.809
7.801
7.796
7.791
7.785
7.260
3.835
3.820
3.807
3.793
3.716
3.702
3.687
3.673
2.235
2.207
2.071
1.426
1.412
1.398
1.095
1.081
1.068
CH3
H3C
O
CH3
H3C
O
CH3
N
O
O
7d
1H NMR (500 MHz, CDCl3)
2.00
2.14
2.19
2.21
3.12
3.15
3.10
3.31
3.11
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

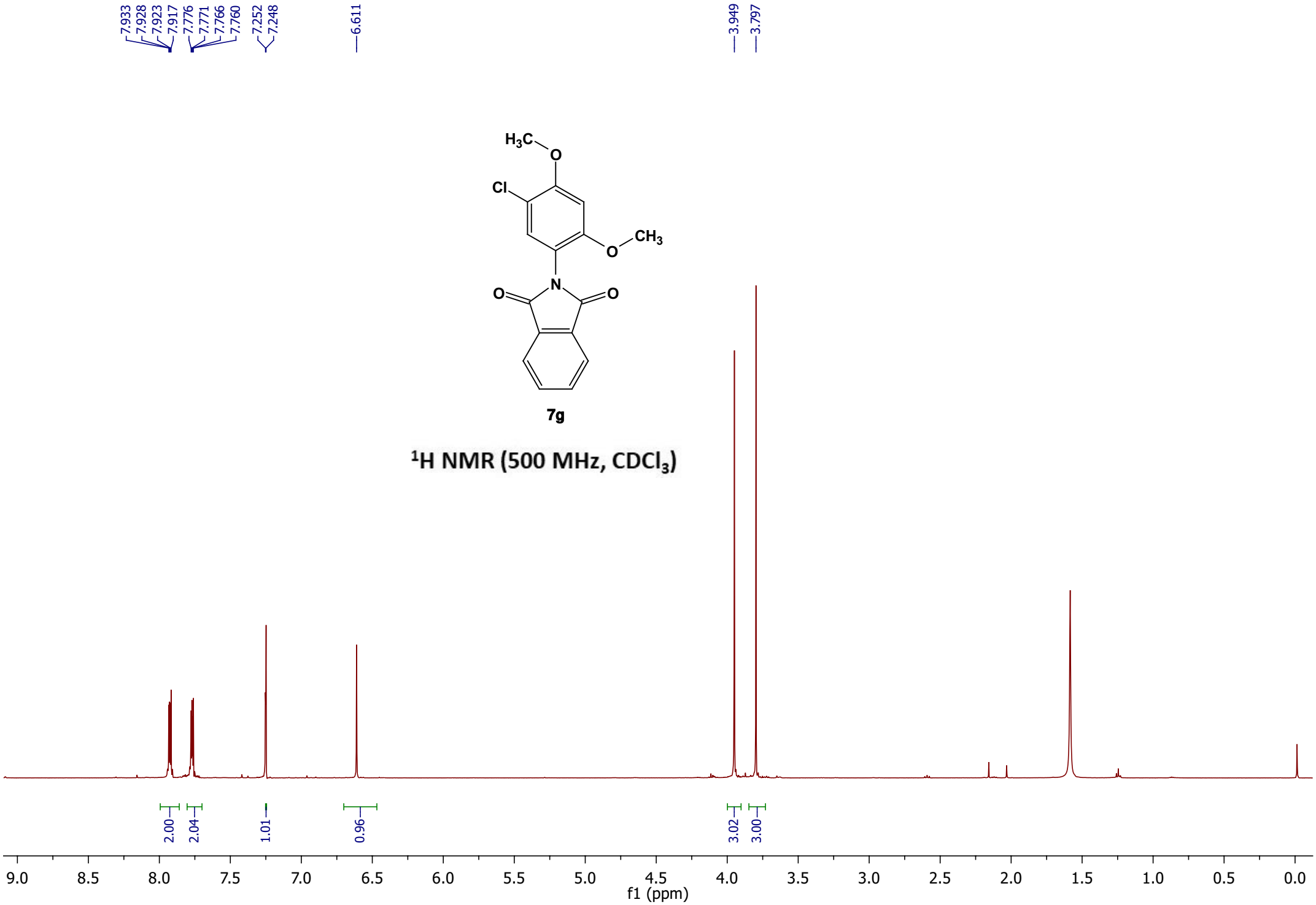

167.745
152.296
150.734
134.365
133.056
132.247
129.167
128.527
123.840
122.525
77.413
77.160
76.906
69.860
68.727
15.830
15.749
13.452
13.319
11.794
CH3
H3C
O
CH3
H3C
O
CH3
N
O
O
7d
13C NMR (125 MHz, CDCl3)
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

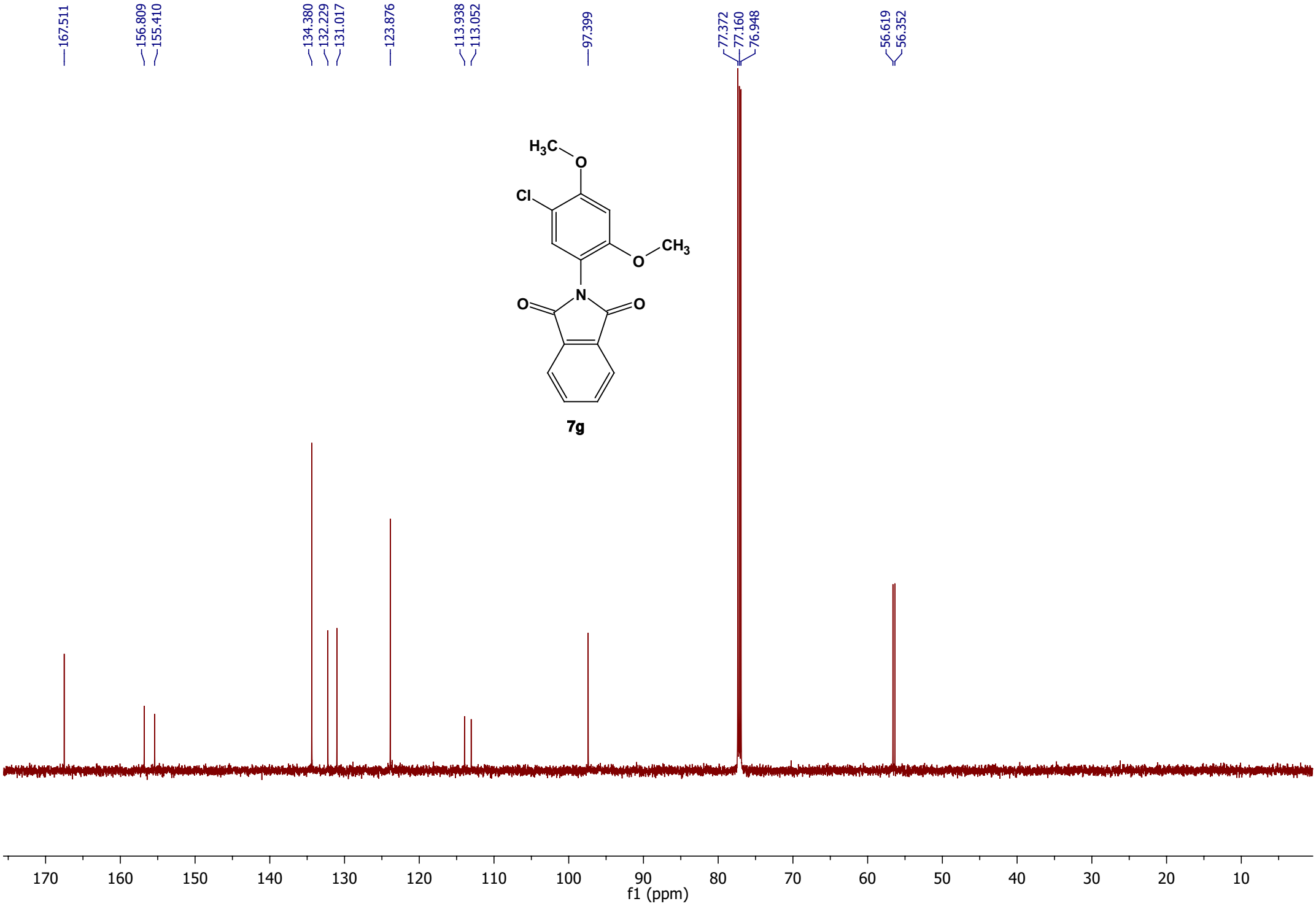

7.952
7.945
7.935
7.928
7.785
7.777
7.771
7.768
7.760
7.450
7.434
7.410
7.396
7.381
7.329
7.314
7.300
7.260
6.464
3.840
3.802
3.199
H3C
O
H3C
O
O
CH3
N
O
O
7e
1H NMR (500 MHz, CDCl3)
2.08
2.13
2.22
2.20
1.09
1.00
3.07
3.16
3.04
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

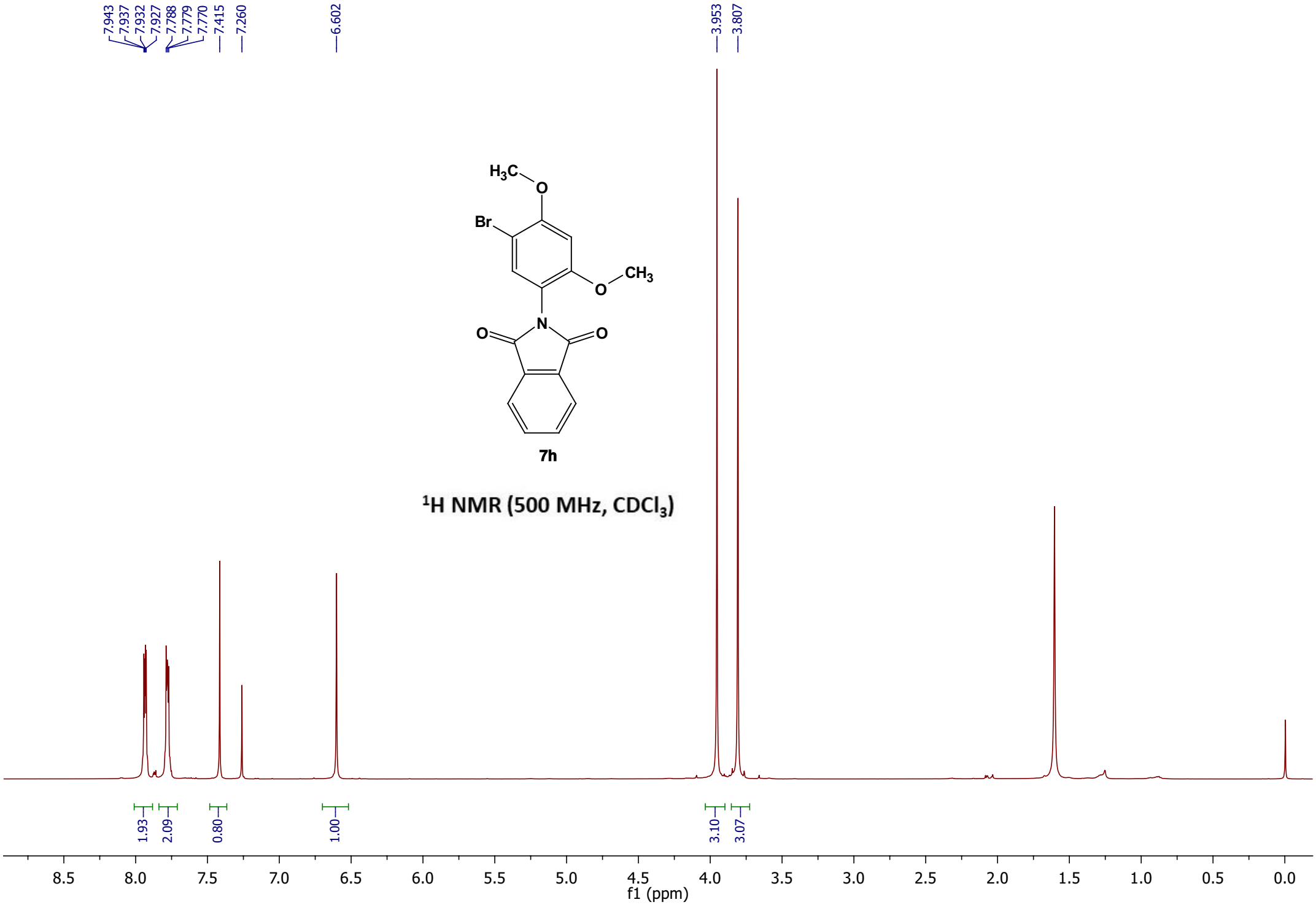

168.055
159.188
156.841
156.533
134.131
133.398
132.575
131.097
128.051
127.135
123.741
117.316
106.742
92.054
77.415
77.160
76.905
61.102
56.197
56.160
H3C
O
H3C
O
O
CH3
N
O
O
7e
13C NMR (125 MHz, CDCl3)
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

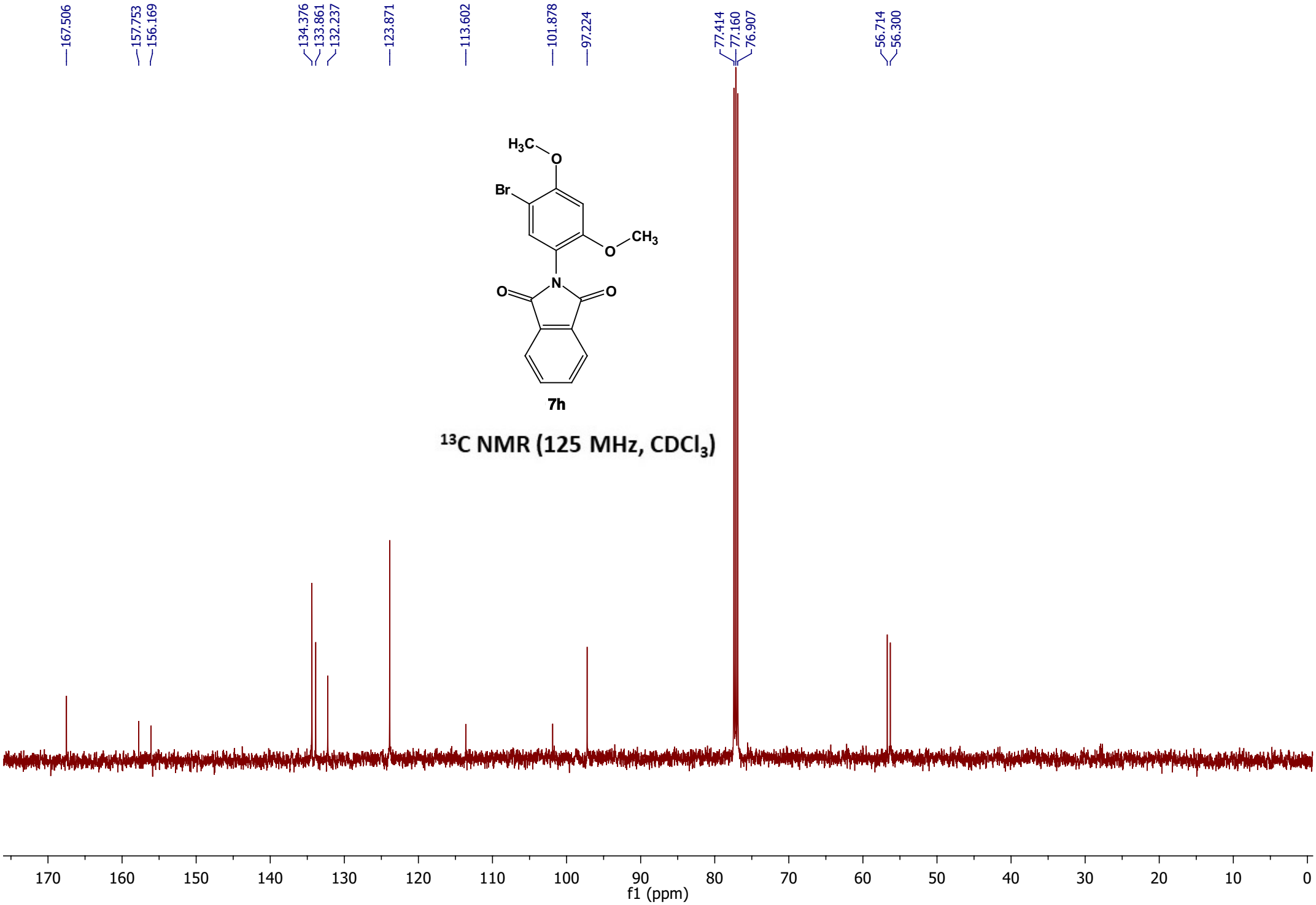

7.952
7.946
7.942
7.936
7.795
7.785
7.778
7.260
6.424
3.951
3.789
3.771
H3C
O
Br
H3C
O
O
CH3
N
O
O
7f
1H NMR (500 MHz, CDCl3)
2.00
2.09
1.00
3.39
3.07
3.02
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

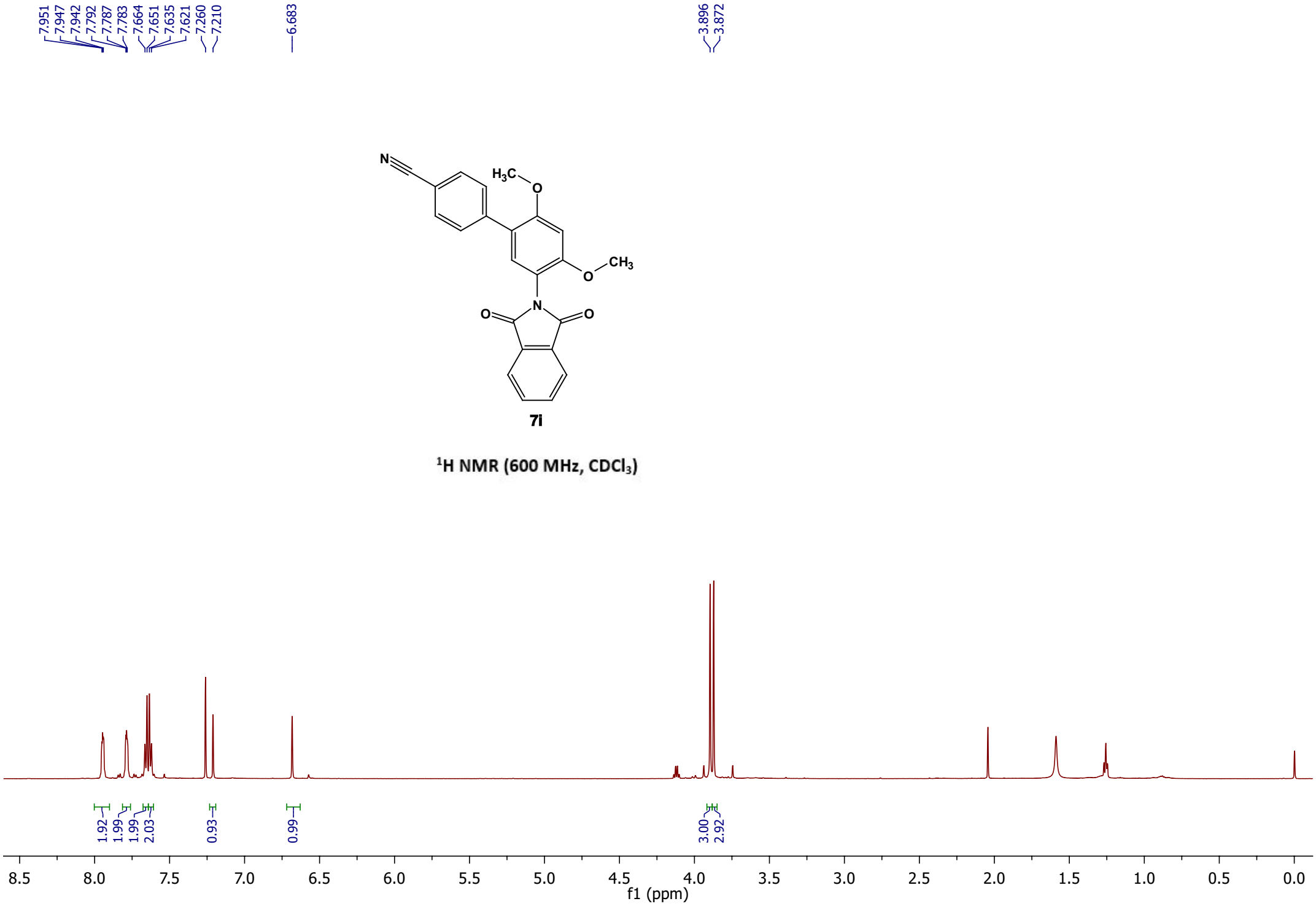

167.737
158.503
156.763
134.321
132.403
123.874
108.125
98.331
92.716
77.414
77.160
76.904
61.758
56.758
56.364
H3C
O
Br
H3C
O
O
CH3
N
O
O
7f
13C NMR (125 MHz, CDCl3)
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)


7.933
7.928
7.923
7.917
7.776
7.771
7.766
7.760
7.252
7.248
6.611
3.949
3.797
H3C
O
Cl
O
CH3
N
O
O
7g
1H NMR (500 MHz, CDCl3)
2.00
2.04
1.01
0.96
3.02
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
1.0
0.5
0.0
f1 (ppm)


167.511
156.809
155.410
134.380
132.229
131.017
123.876
113.938
113.052
97.399
77.372
77.160
76.948
56.619
56.352
H3C
O
Cl
O
CH3
N
O
O
7g
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

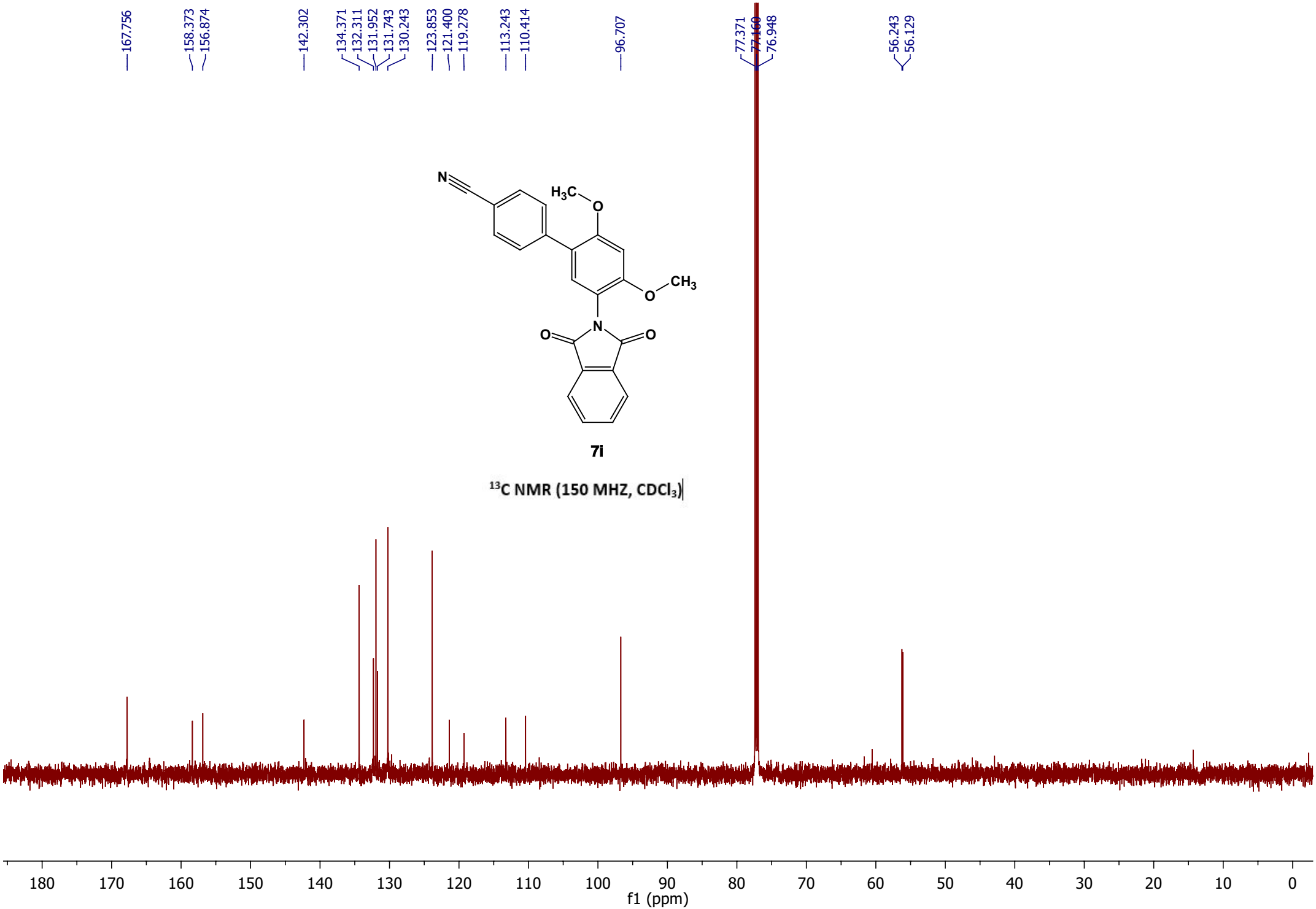

7.943
7.937
7.932
7.927
7.788
7.779
7.770
7.415
7.260
6.602
3.953
3.807
H3C
O
Br
CH3
O
N
O
O
7h
1H NMR (500 MHz, CDCl3)
1.93
2.09
0.80
1.00
3.10
3.07
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

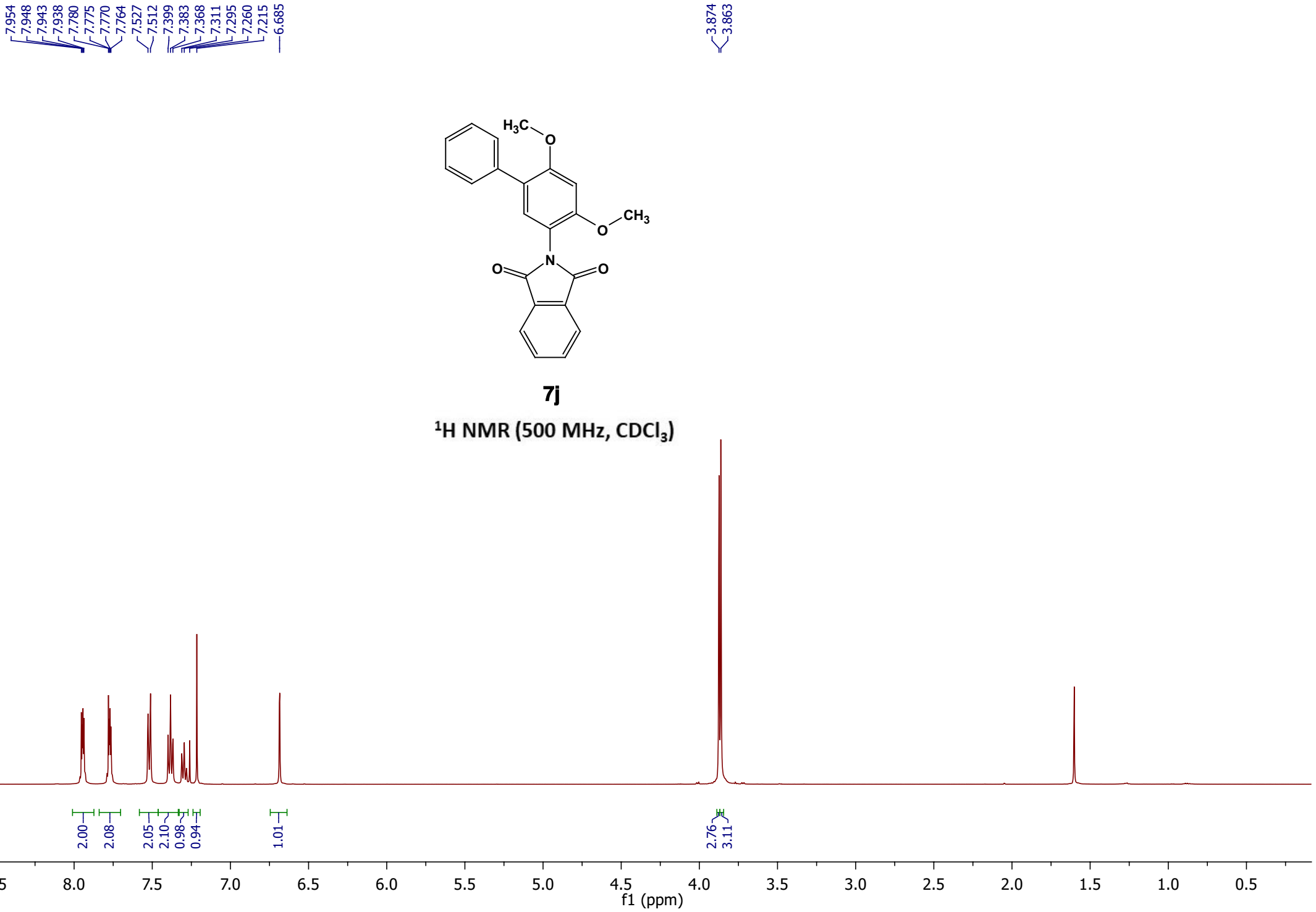

167.506
157.753
156.169
134.376
133.861
132.237
123.871
113.602
101.878
97.224
77.414
77.160
76.907
56.714
56.300
H3C
O
Br
CH3
O
N
O
O
7h
13C NMR (125 MHz, CDCl3)
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

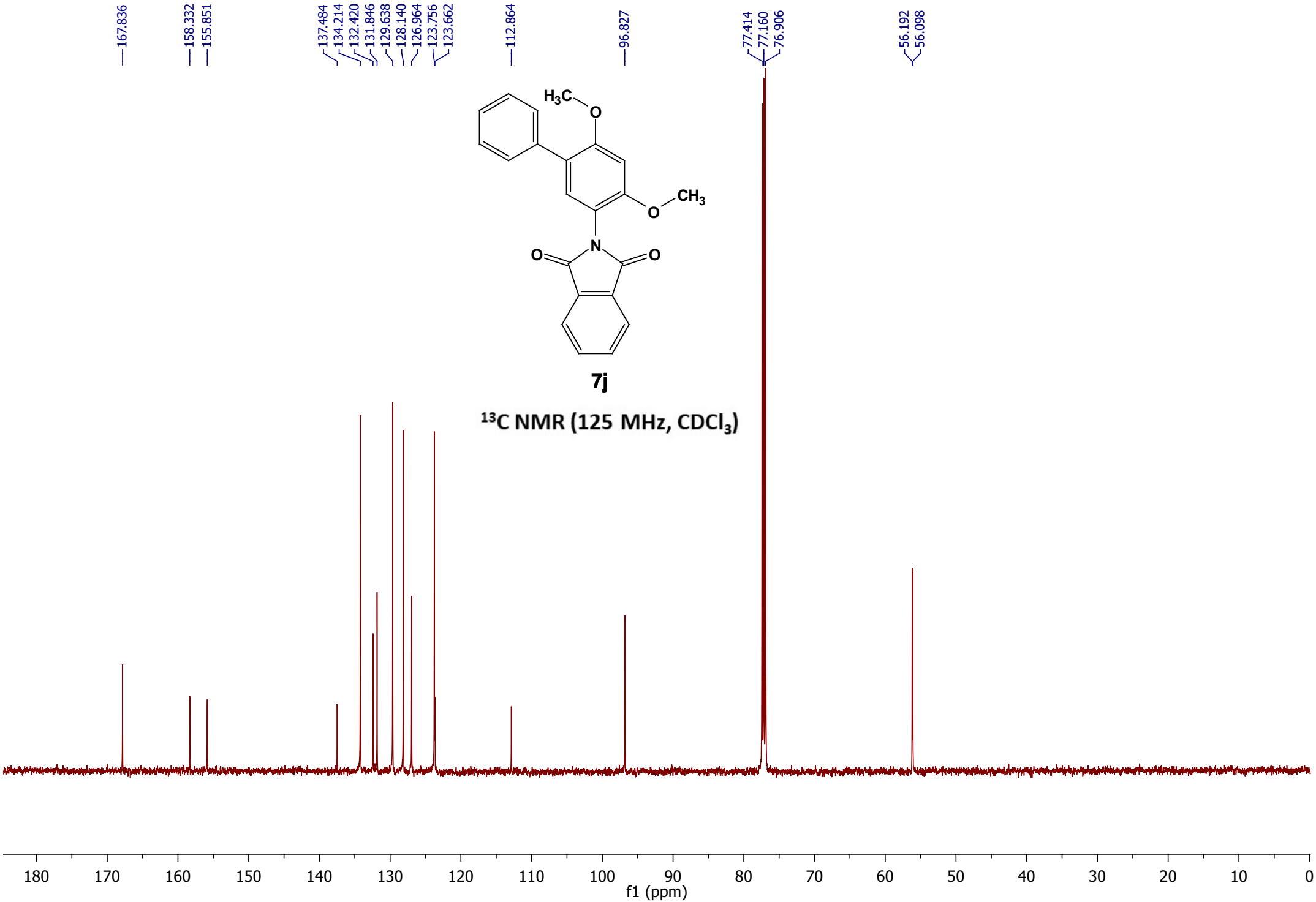


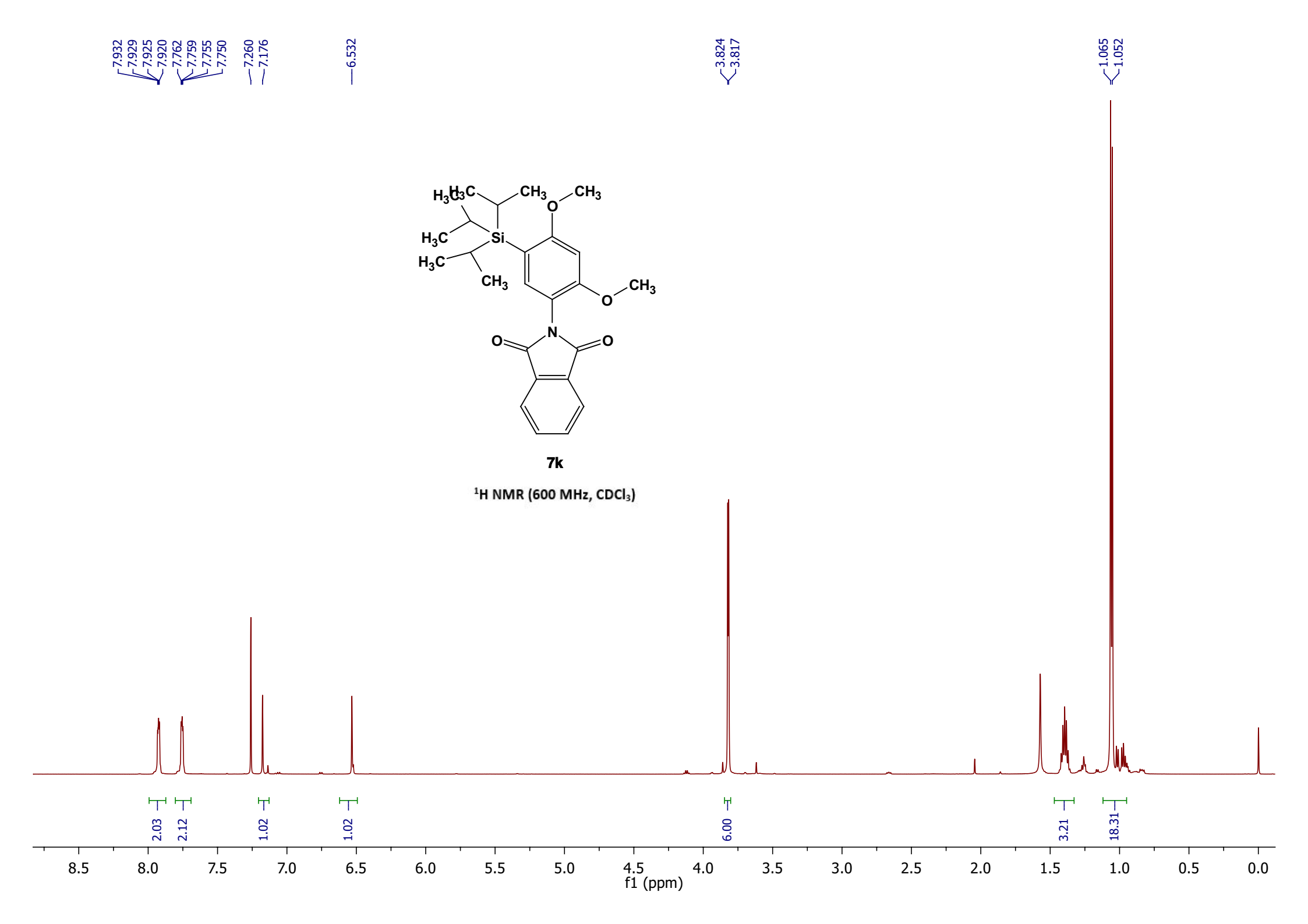
7i

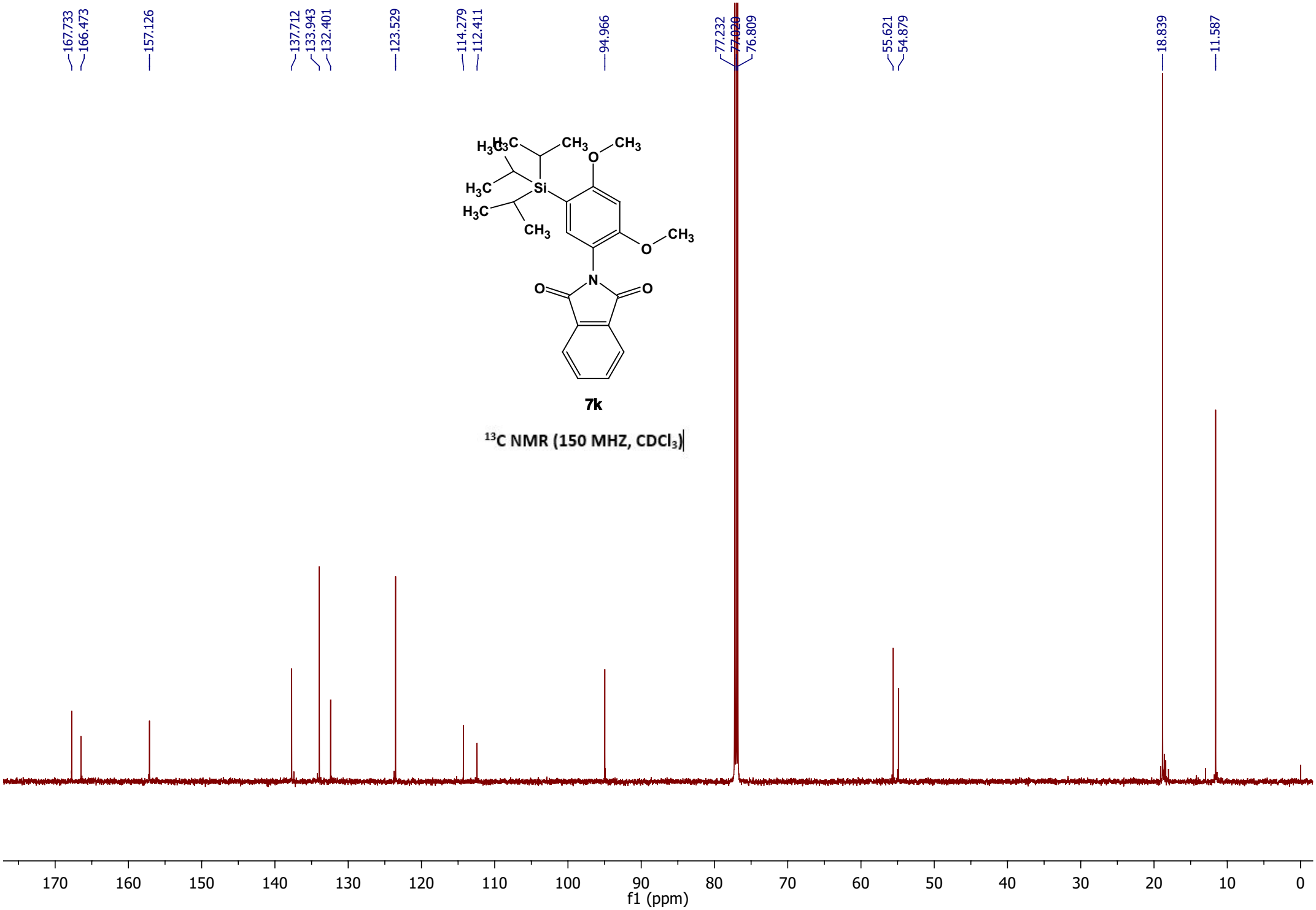
^{1}H NMR (600 MHz, $CDCl_3$)

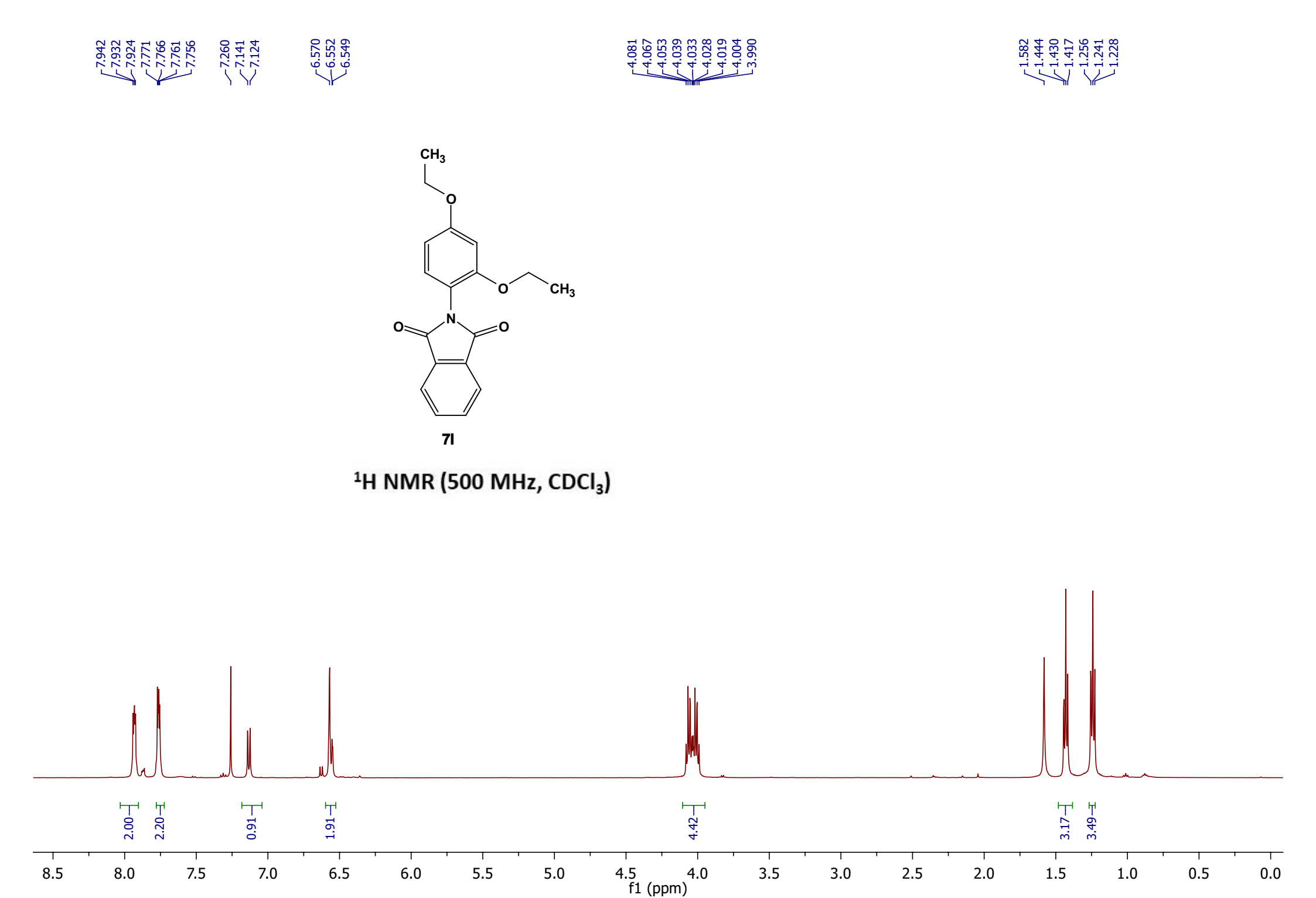

167.756
158.373
156.874
142.302
134.371
132.311
131.952
131.743
130.243
123.853
121.400
119.278
113.243
110.414
96.707
77.371
77.160
76.948
56.243
56.129
N
H3C
O
O
CH3
N
O
O
7i
13C NMR (150 MHZ, CDCl3)
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

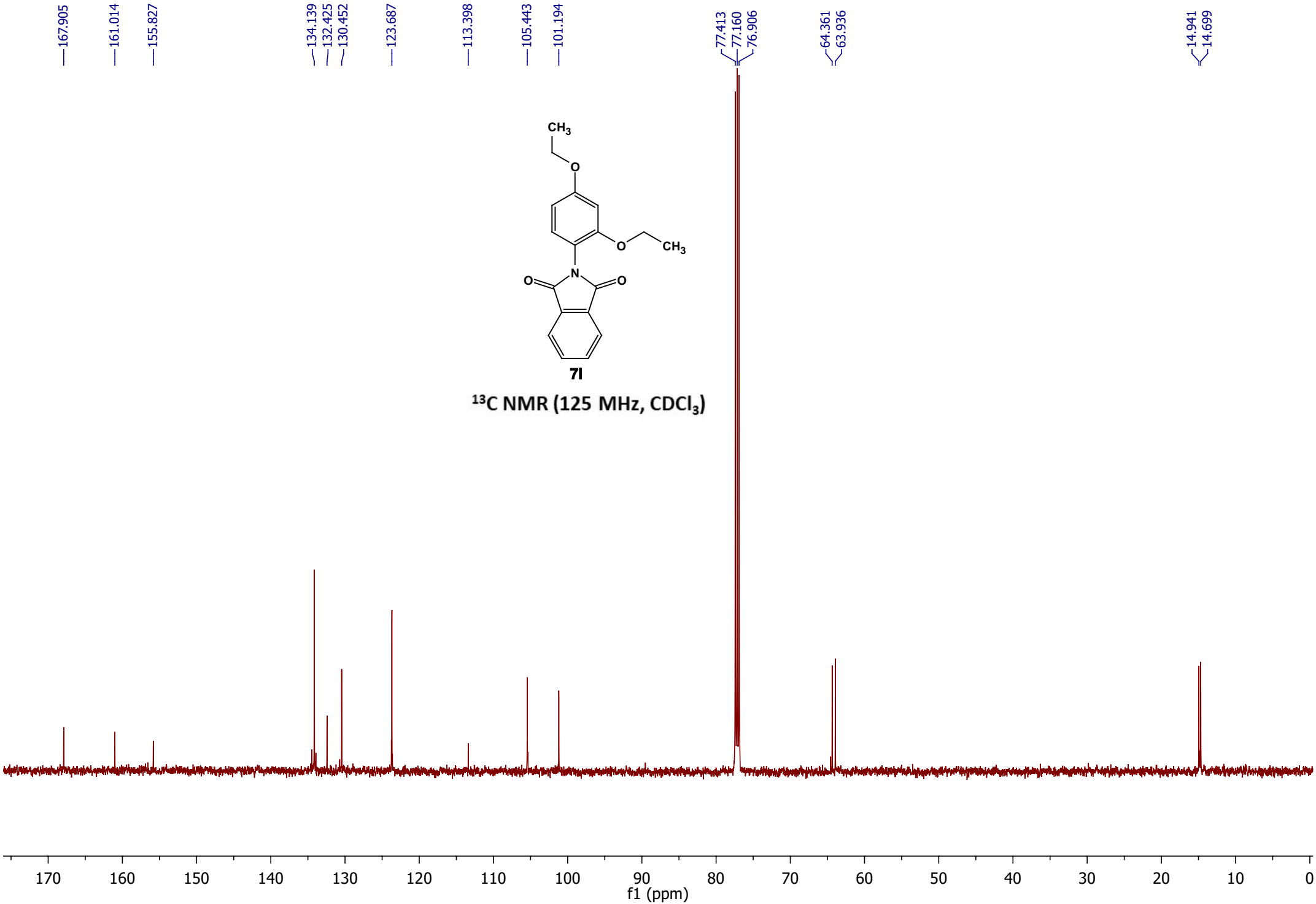

7.954
7.948
7.943
7.938
7.780
7.775
7.770
7.764
7.527
7.512
7.399
7.383
7.368
7.311
7.295
7.260
7.215
6.685
3.874
3.863
H3C
O
CH3
O
N
O
O
7j
1H NMR (500 MHz, CDCl3)
2.00
2.08
2.05
2.10
0.98
0.94
1.01
2.76
3.11
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

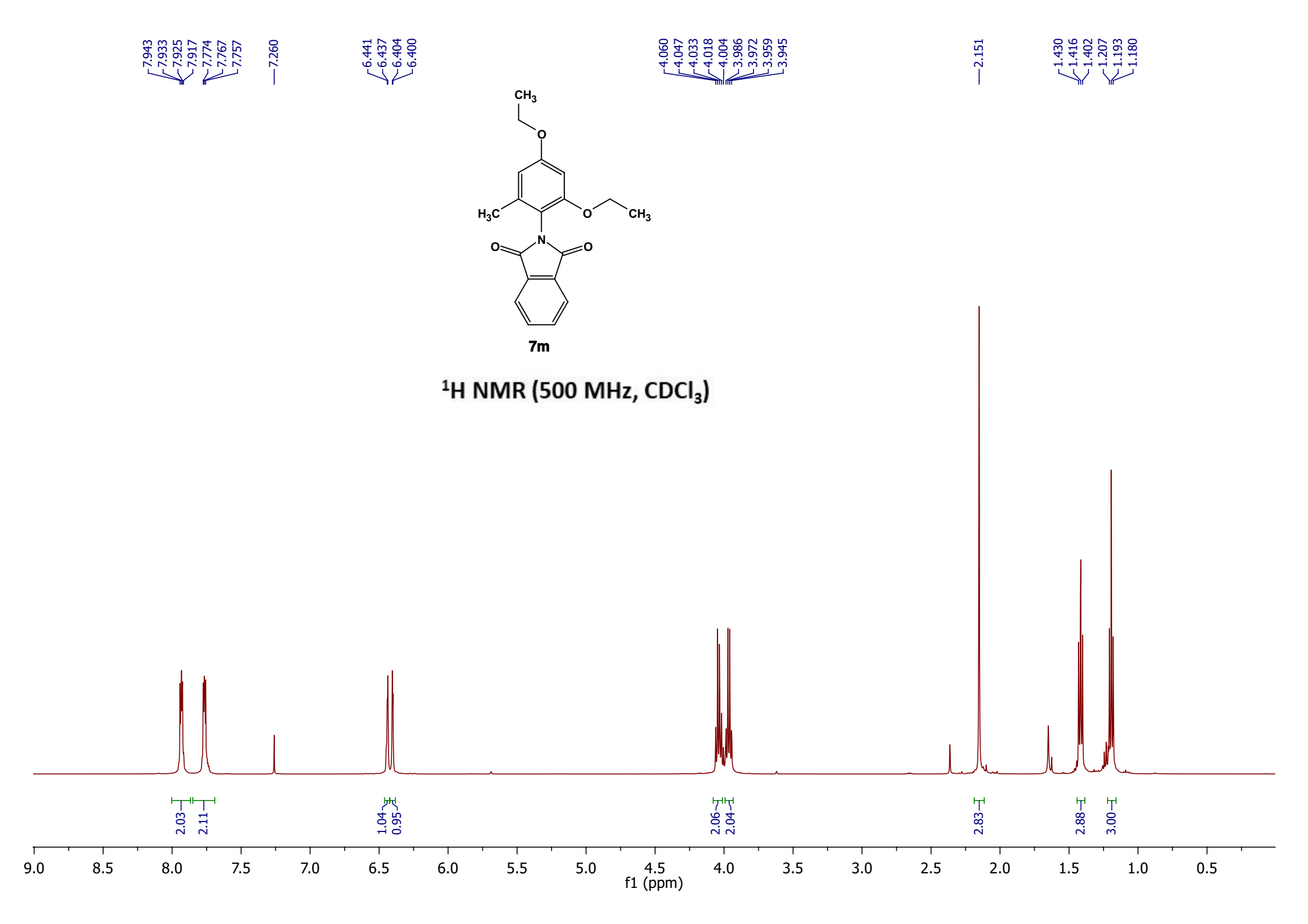

167.836
158.332
155.851
137.484
134.214
132.420
131.846
129.638
128.140
126.964
123.756
123.662
112.864
96.827
77.414
77.160
76.906
56.192
56.098
H3C
O
CH3
O
N
O
O
7j
13C NMR (125 MHz, CDCl3)
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

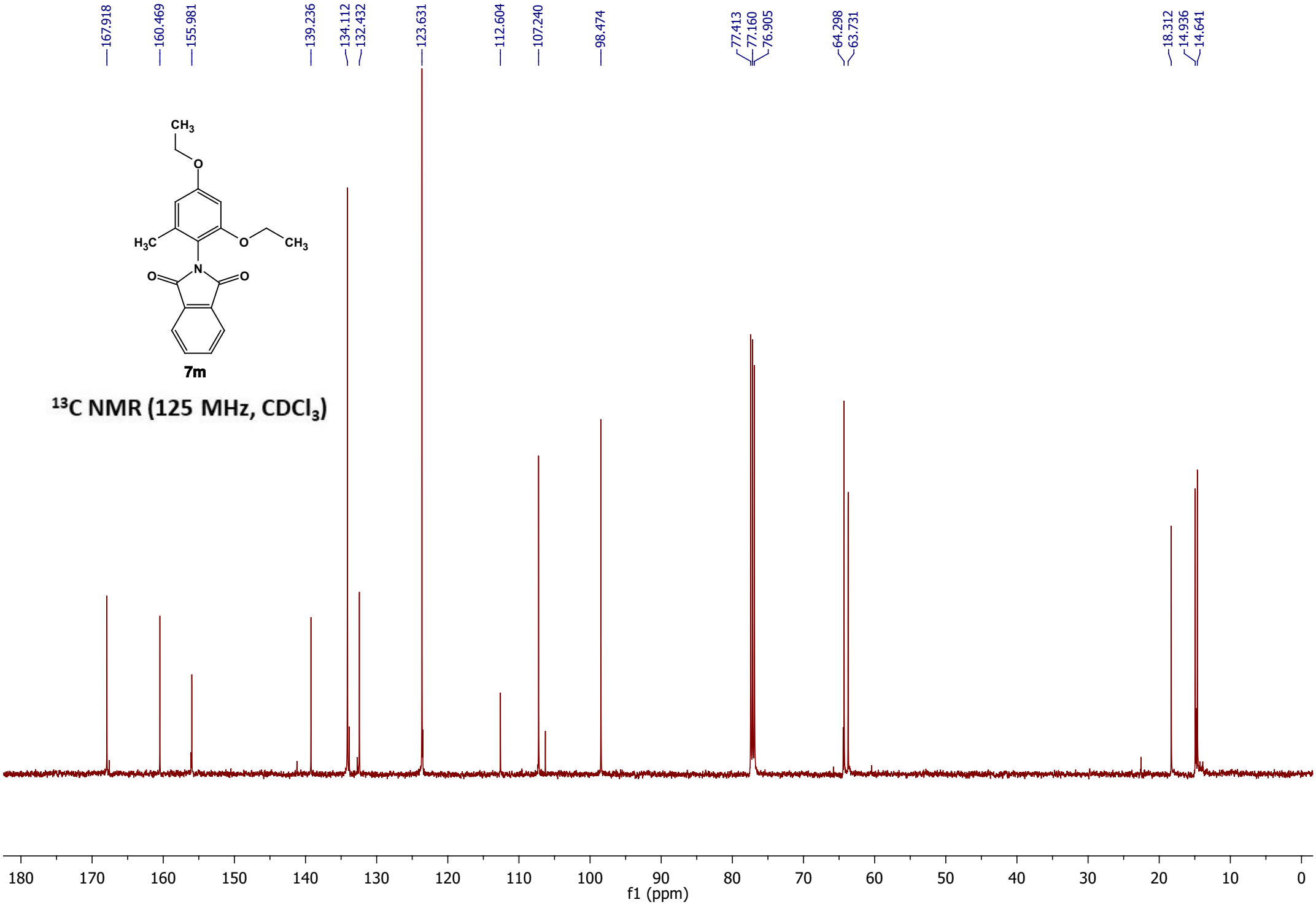

7.932
7.929
7.925
7.920
7.762
7.759
7.755
7.750
7.260
7.176
6.532
3.824
3.817
1.065
1.052
7k
1H NMR (600 MHz, CDCl3)
2.03
2.12
1.02
1.02
6.00
3.21
18.31
f1 (ppm)

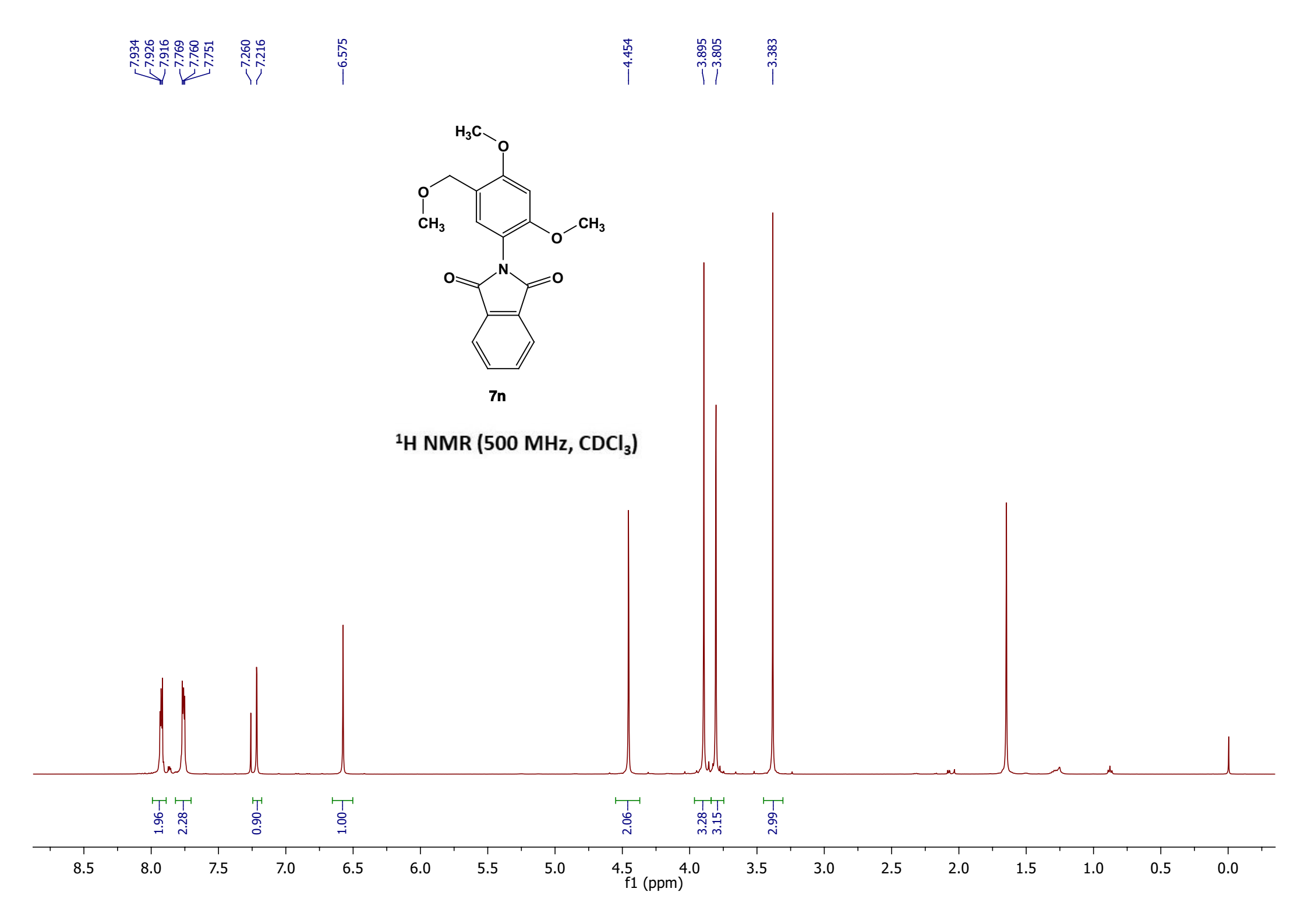

167.733
166.473
157.126
137.712
133.943
132.401
123.529
114.279
112.411
94.966
77.232
77.030
76.809
55.621
54.879
18.839
11.587
7k
13C NMR (150 MHZ, CDCl3)
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

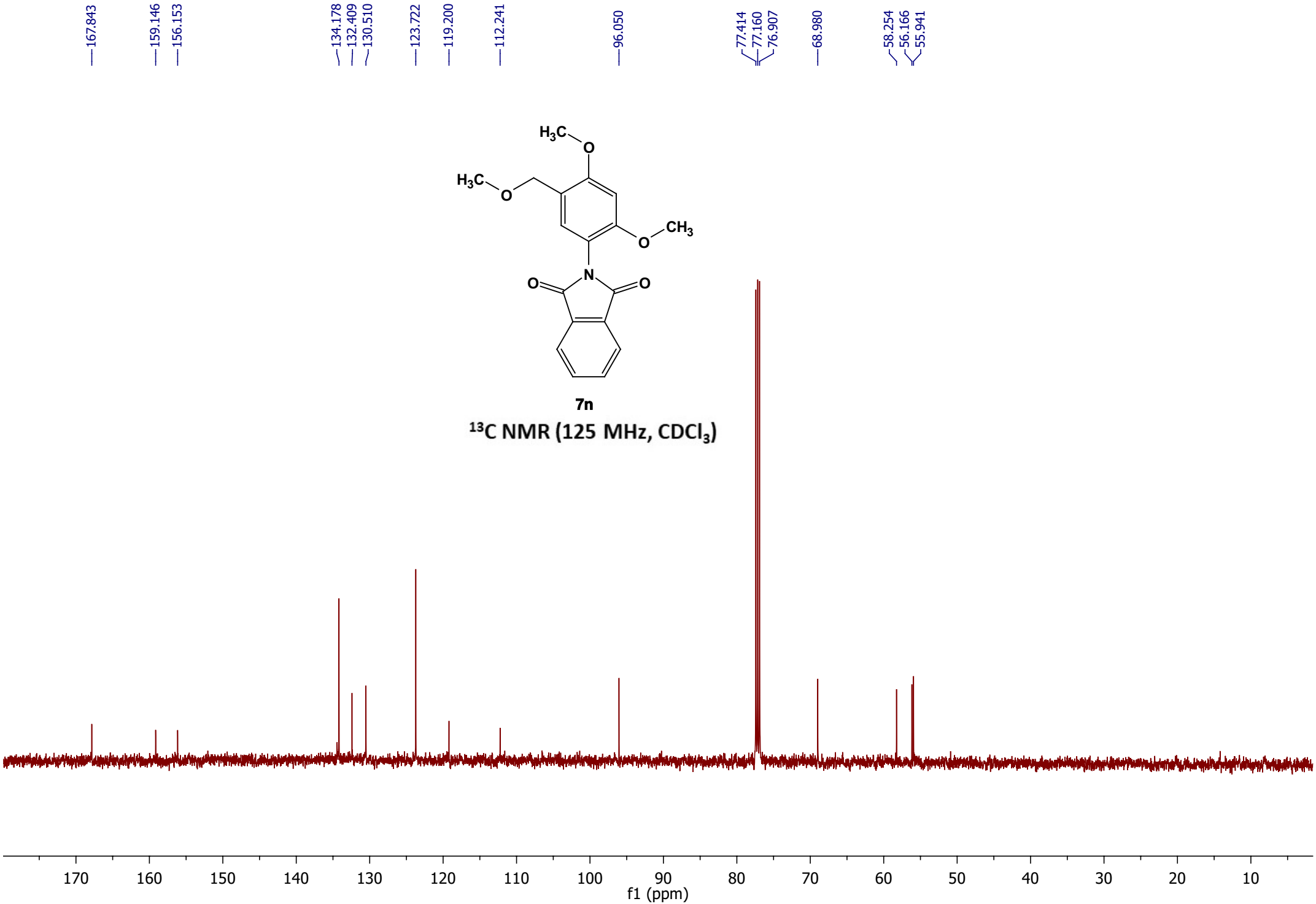

7.942
7.932
7.924
7.771
7.766
7.761
7.756
7.260
7.141
7.124
6.570
6.552
6.549
4.081
4.067
4.053
4.039
4.033
4.028
4.019
4.004
3.990
1.582
1.444
1.430
1.417
1.256
1.241
1.228
CH3
O
O
CH3
N
O
O
7l
1H NMR (500 MHz, CDCl3)
2.00
2.20
0.91
1.91
4.42
3.17
3.49
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

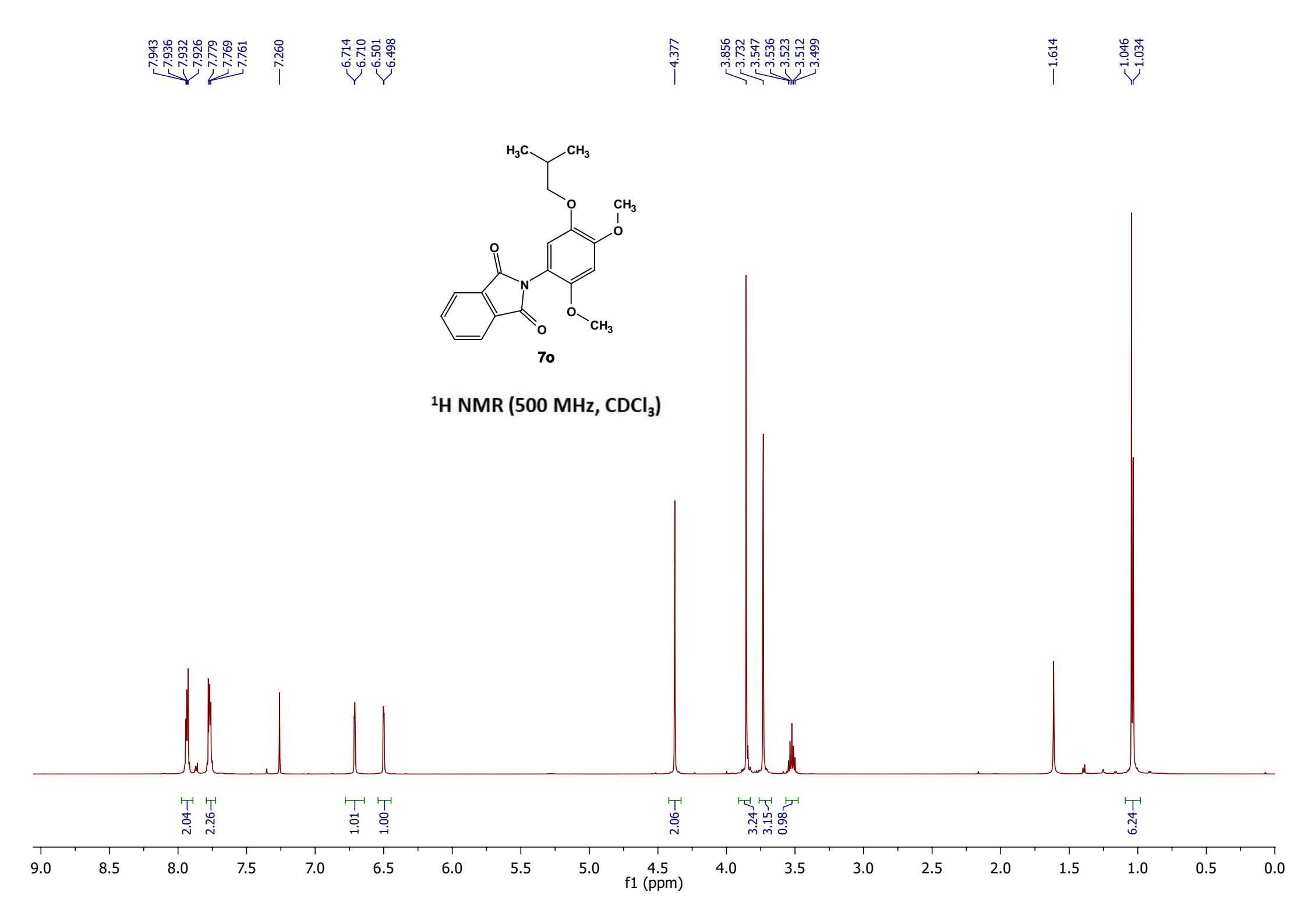

167.905
161.014
155.827
134.139
132.425
130.452
123.687
113.398
105.443
101.194
77.413
77.160
76.906
64.361
63.936
14.941
14.699
CH3
O
O
CH3
N
O
O
7l
13C NMR (125 MHz, CDCl3)
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

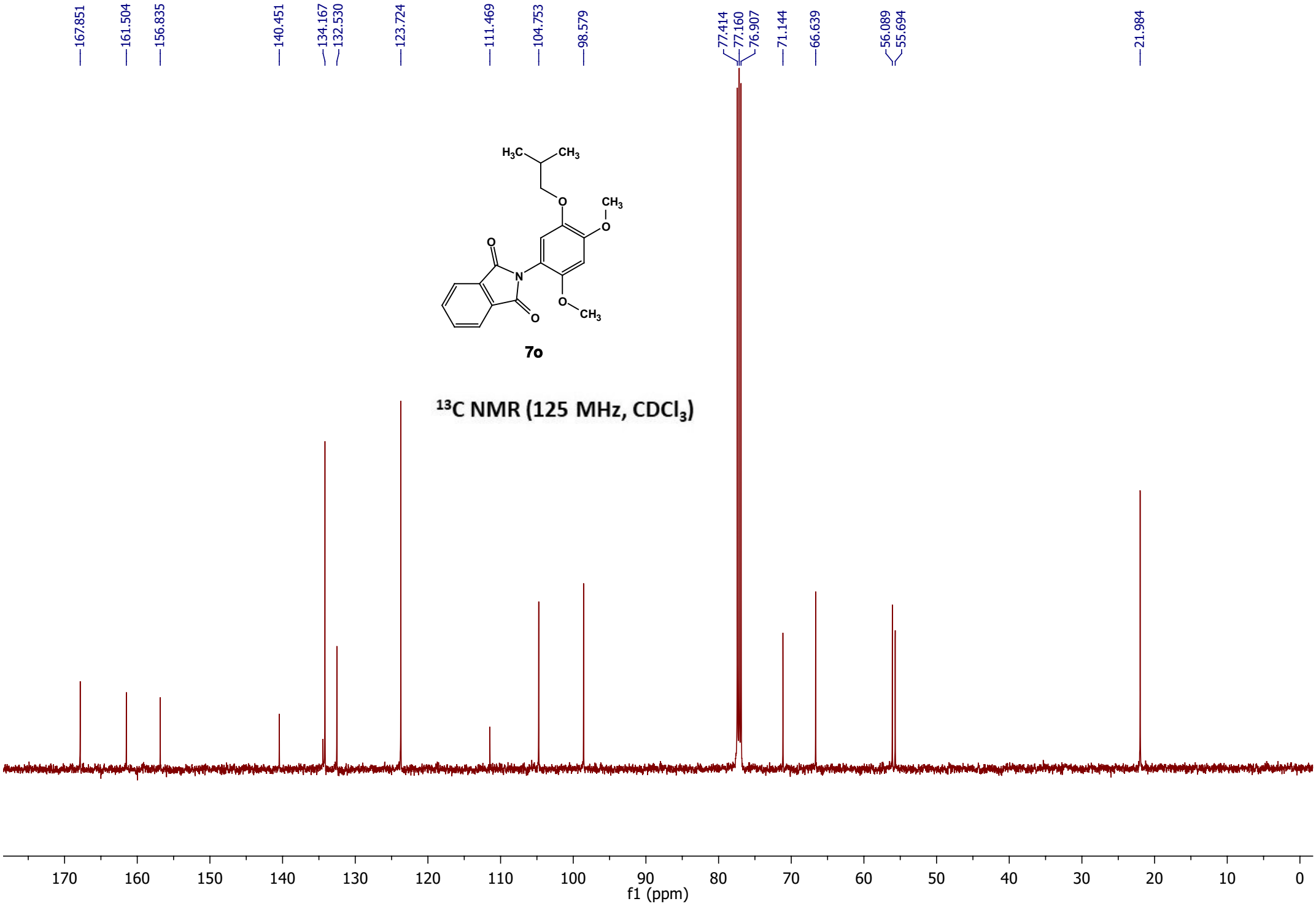

7.943
7.933
7.925
7.917
7.774
7.767
7.757
7.260
6.441
6.437
6.404
6.400
4.060
4.047
4.033
4.018
4.004
3.986
3.972
3.959
3.945
2.151
1.430
1.416
1.402
1.207
1.193
1.180
CH3
O
H3C
O
CH3
N
O
O
7m
1H NMR (500 MHz, CDCl3)
2.03
2.11
1.04
0.95
2.06
2.04
2.83
2.88
3.00
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

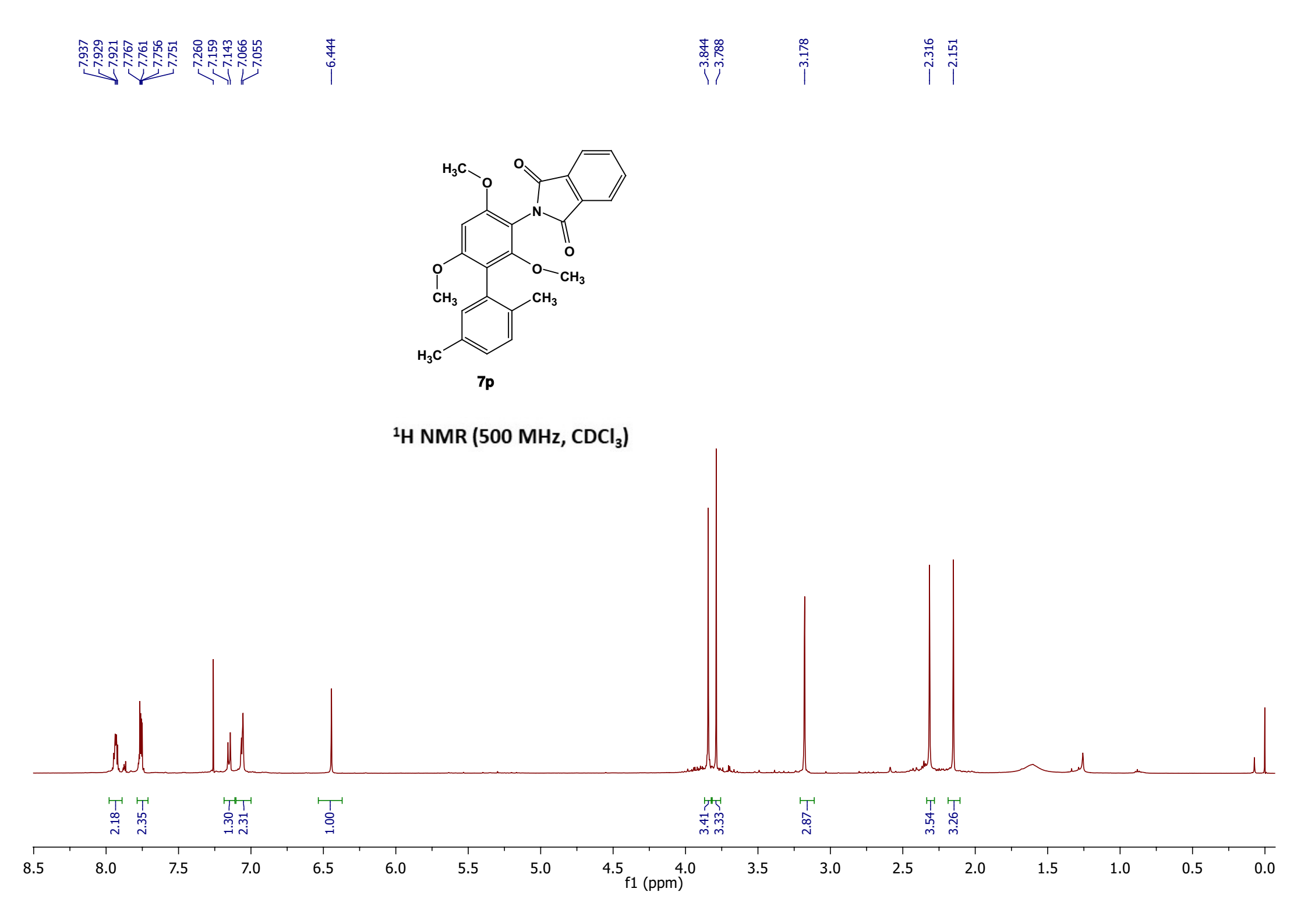

167.918
160.469
155.981
139.236
134.112
132.432
123.631
112.604
107.240
98.474
77.413
77.160
76.905
64.298
63.731
18.312
14.936
14.641
CH3
O
H3C
O
CH3
N
O
O
7m
13C NMR (125 MHz, CDCl3)
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

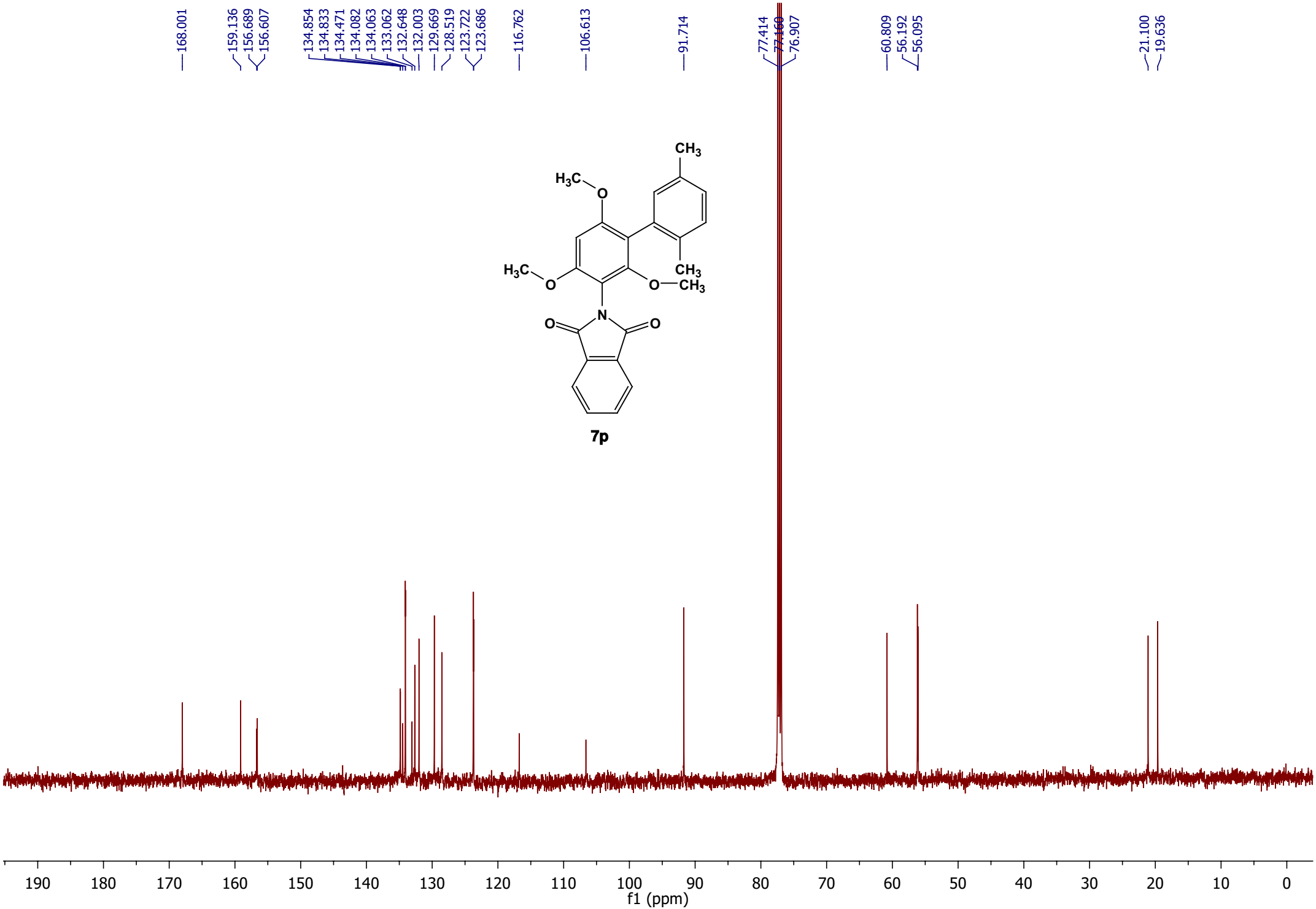

7.934
7.926
7.916
7.769
7.760
7.751
7.260
7.216
6.575
4.454
3.895
3.805
3.383
H3C
O
O
CH3
O
CH3
N
O
O
7n
1H NMR (500 MHz, CDCl3)
1.96
2.28
0.90
1.00
2.06
3.28
3.15
2.99
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

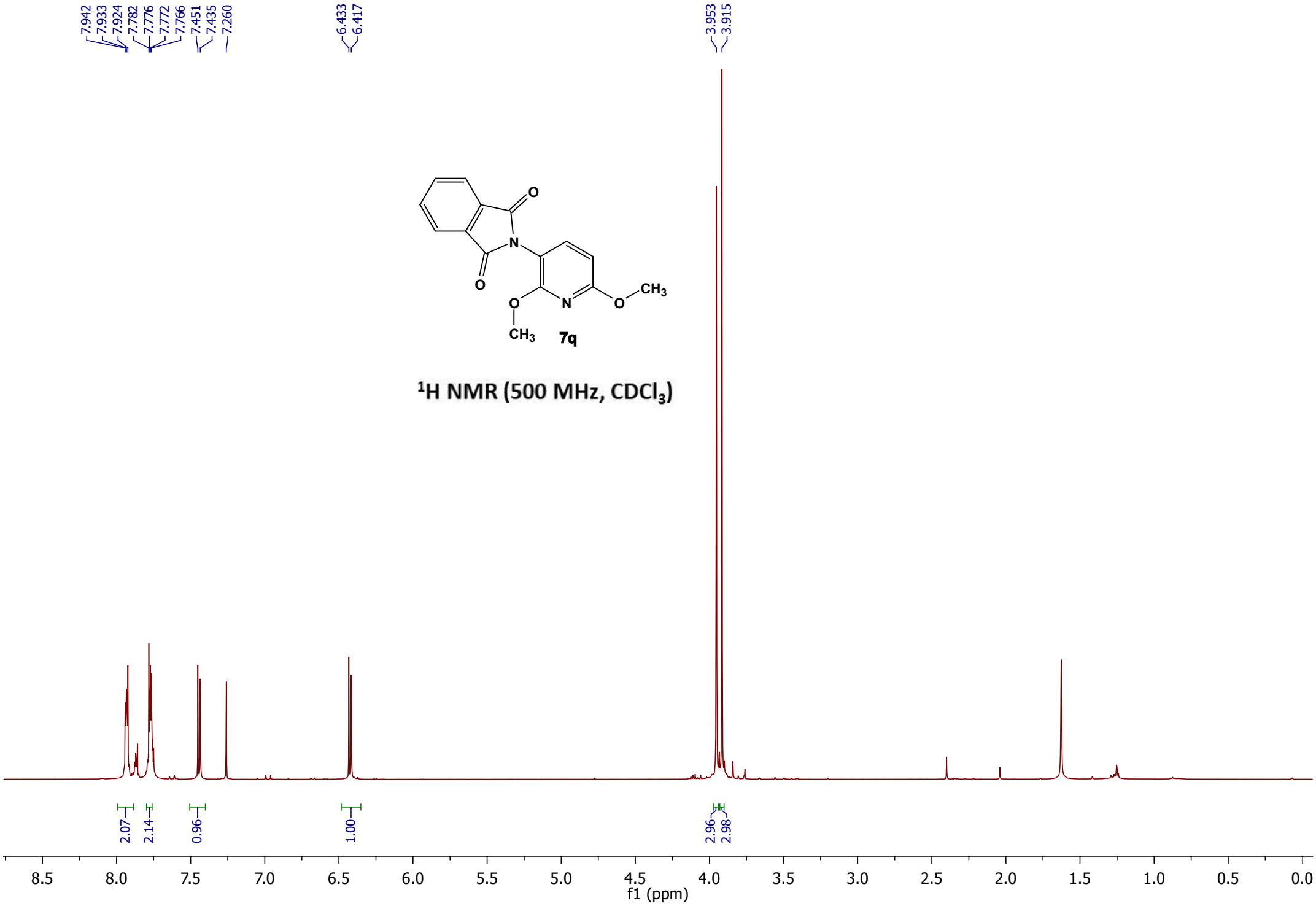

167.843
159.146
156.153
134.178
132.409
130.510
123.722
119.200
112.241
96.050
77.414
77.160
76.907
68.980
58.254
56.166
55.941
H3C
O
H3C
O
O
CH3
N
O
O
7n
13C NMR (125 MHz, CDCl3)
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

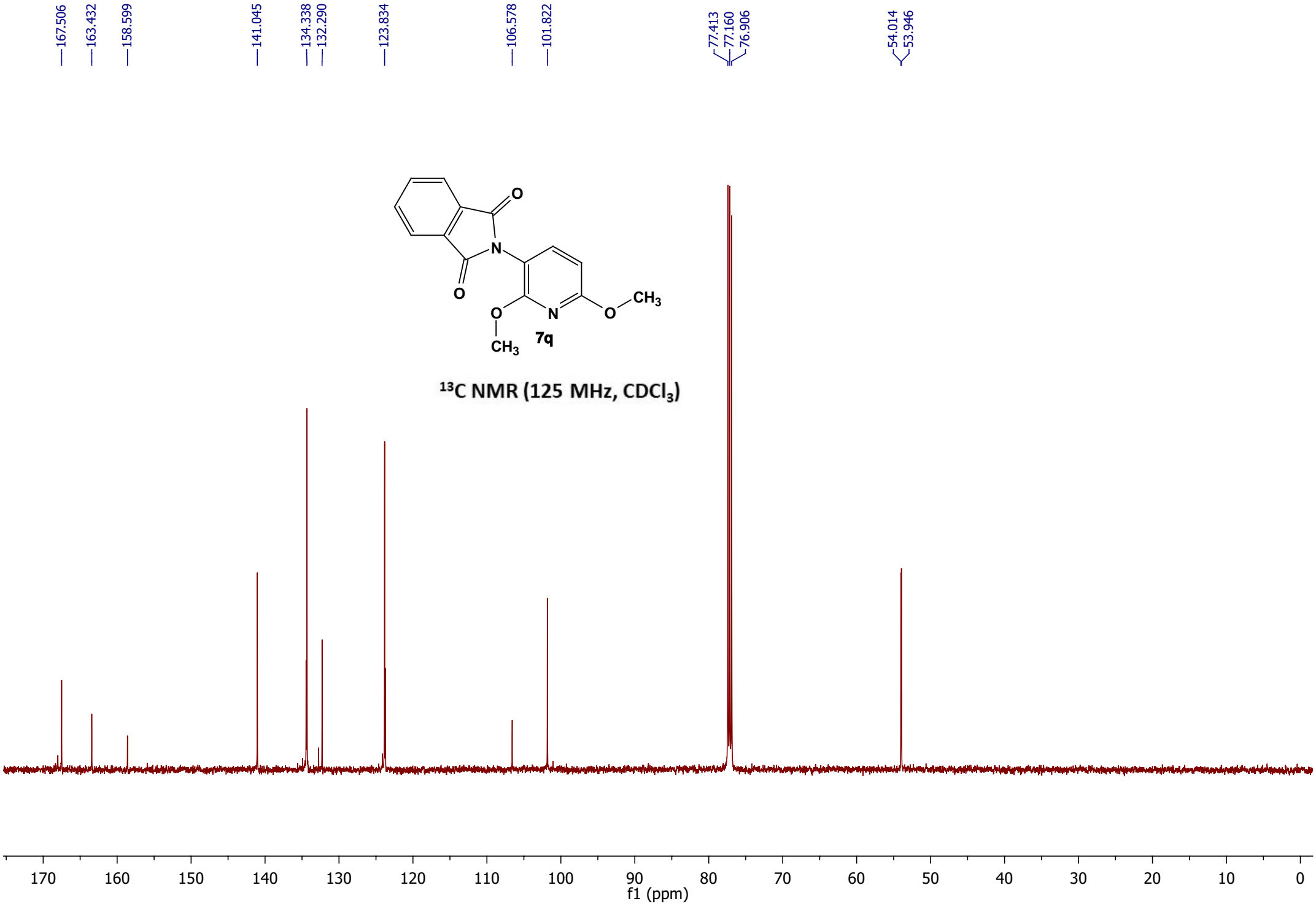

7.943
7.936
7.932
7.926
7.779
7.769
7.761
7.260
6.714
6.710
6.501
6.498
4.377
3.856
3.732
3.547
3.536
3.523
3.512
3.499
1.614
1.046
1.034
H3C
CH3
O
CH3
O
O
N
O
O
CH3
7o
1H NMR (500 MHz, CDCl3)
2.04
2.26
1.01
1.00
2.06
3.24
3.15
0.98
6.24
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

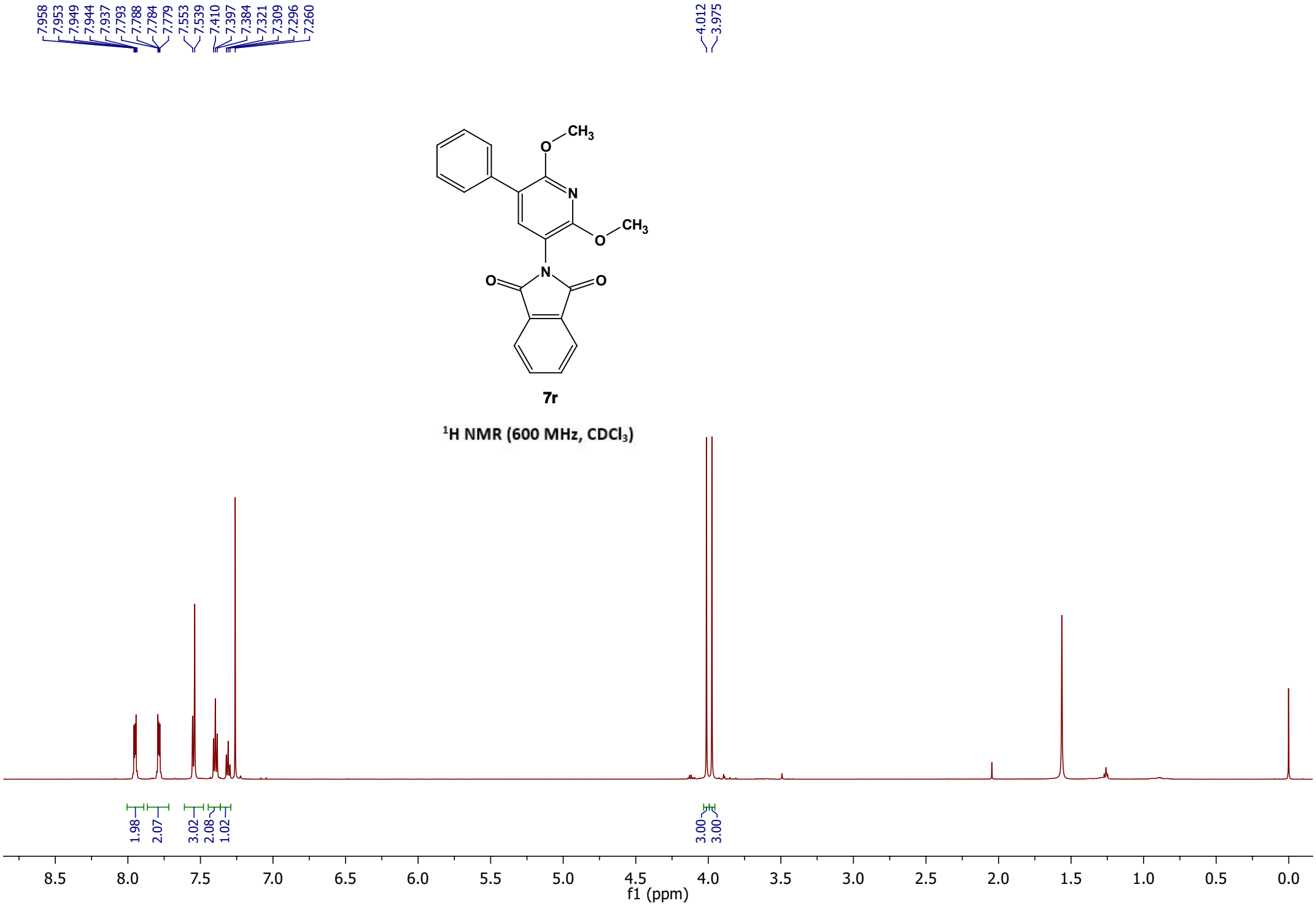

167.851
161.504
156.835
140.451
134.167
132.530
123.724
111.469
104.753
98.579
77.414
77.160
76.907
71.144
66.639
56.089
55.694
21.984
H3C
CH3
O
CH3
O
O
N
O
O
CH3
7o
13C NMR (125 MHz, CDCl3)
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

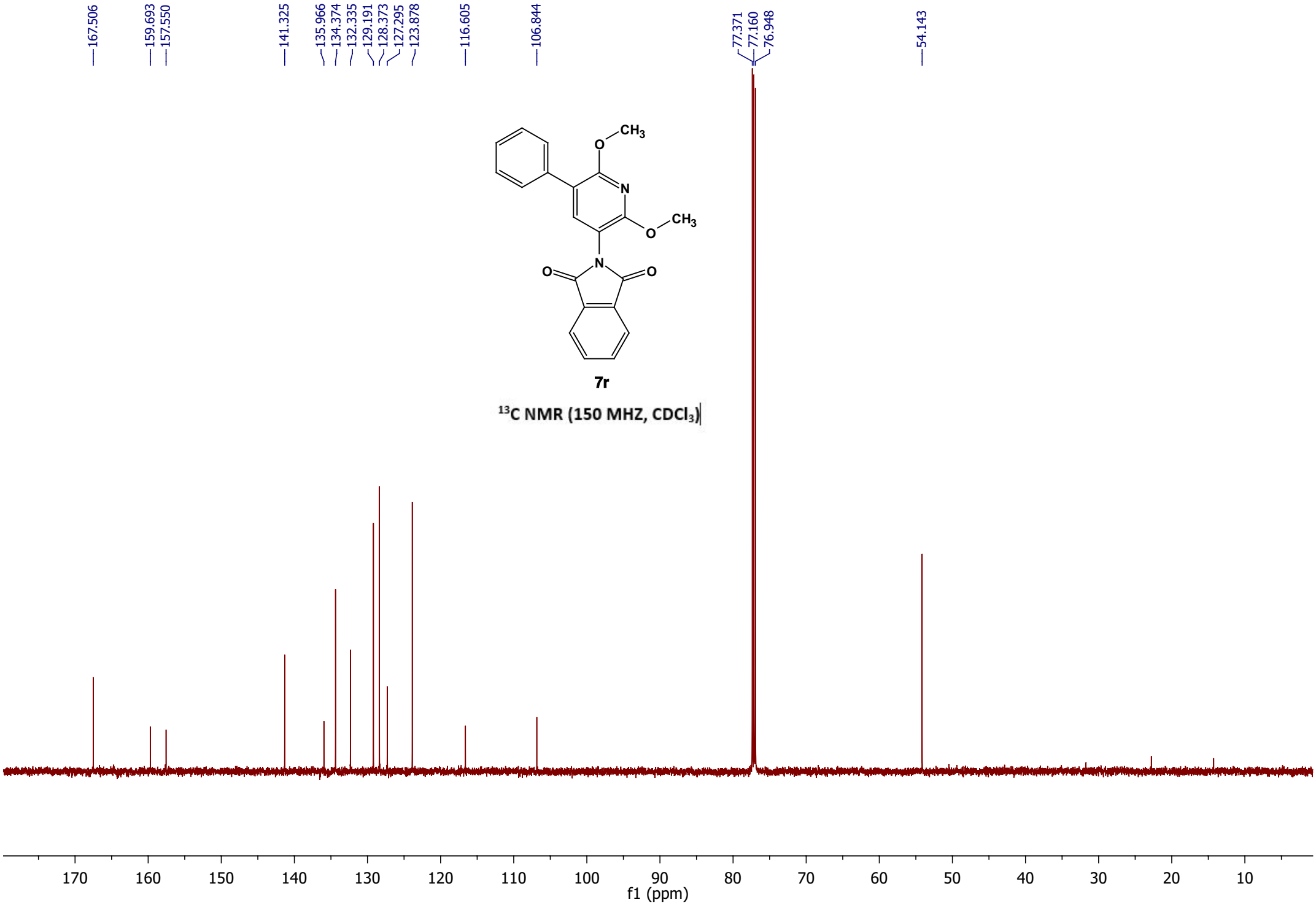

7.937
7.929
7.921
7.767
7.761
7.756
7.751
7.260
7.159
7.143
7.066
7.055
6.444
3.844
3.788
3.178
2.316
2.151
H3C
O
O
N
O
O
O
CH3
O
CH3
CH3
H3C
7p
1H NMR (500 MHz, CDCl3)
2.18
2.35
1.30
2.31
1.00
3.41
3.33
2.87
3.54
3.26
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


168.001
159.136
156.689
156.607
134.854
134.833
134.471
134.082
134.063
133.062
132.648
132.003
129.669
128.519
123.722
123.686
116.762
106.613
91.714
77.414
77.160
76.907
60.809
56.192
56.095
21.100
19.636
CH3
H3C
O
H3C
O
CH3
O
O—CH3
N
O
O
7p
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.942
7.933
7.924
7.782
7.776
7.772
7.766
7.451
7.435
7.260
6.433
6.417
3.953
3.915
O
N
O
O
N
O
CH3
CH3
7q
1H NMR (500 MHz, CDCl3)
2.07
2.14
0.96
1.00
2.96
2.98
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)

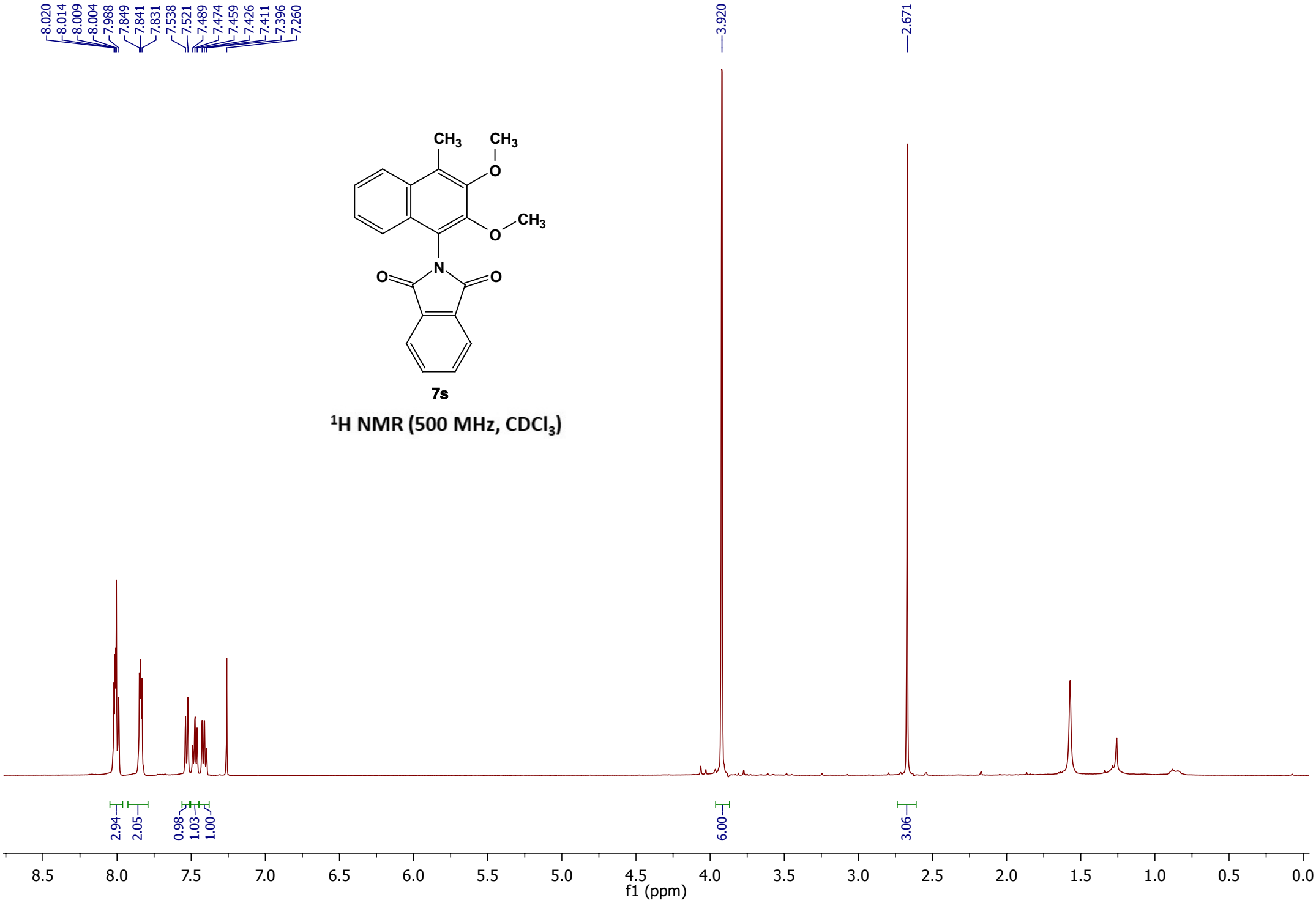

167.506
163.432
158.599
141.045
134.338
132.290
123.834
106.578
101.822
77.413
77.160
76.906
54.014
53.946
O
N
O
O
N
O
CH3
O
CH3
7q
13C NMR (125 MHz, CDCl3)
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.958
7.953
7.949
7.944
7.937
7.793
7.788
7.784
7.779
7.553
7.539
7.410
7.397
7.384
7.321
7.309
7.296
7.260
4.012
3.975
O
CH3
N
O
CH3
N
O
O
7r
1H NMR (600 MHz, CDCl3)
1.98
2.07
3.02
2.08
1.02
3.00
3.00
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)



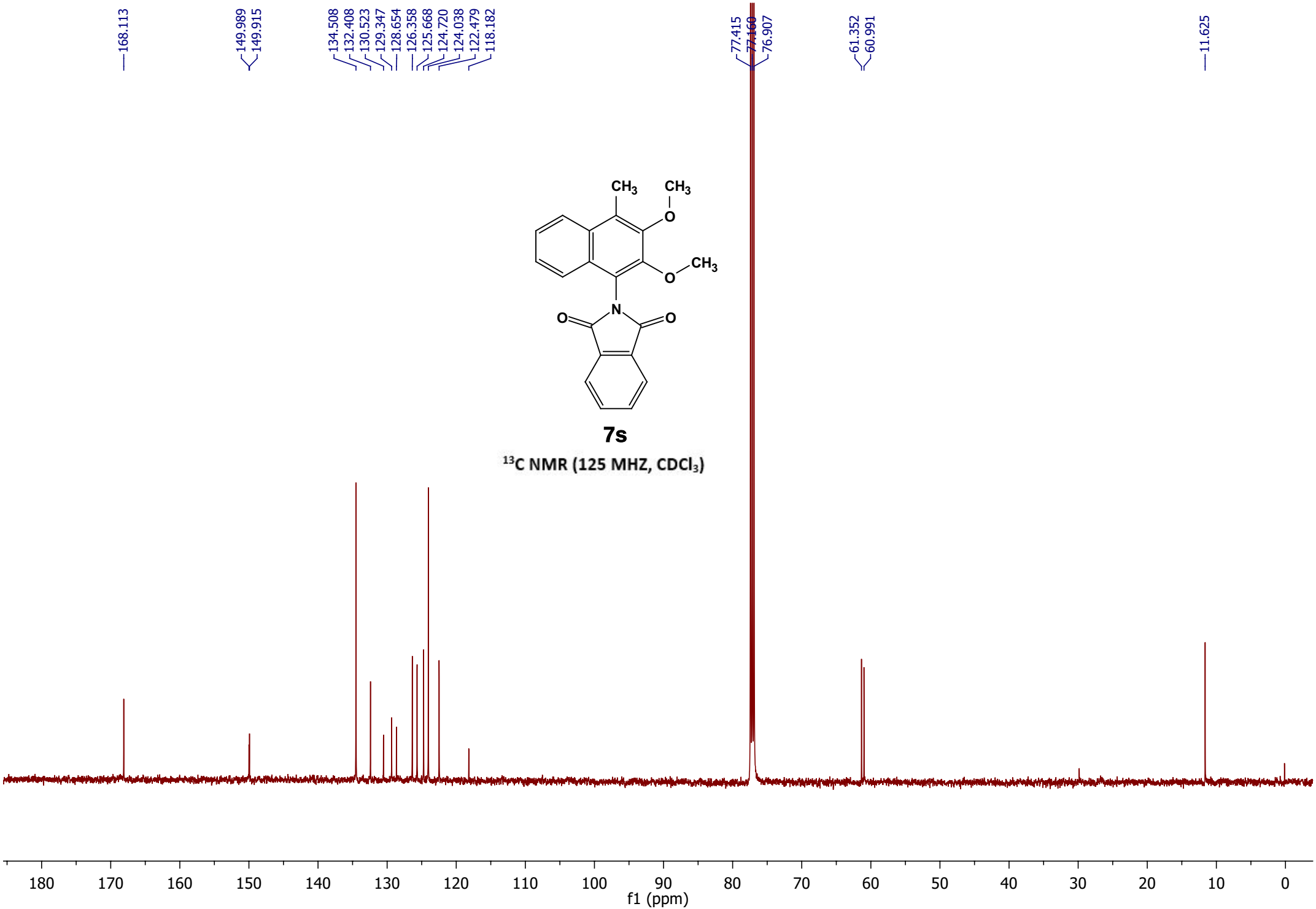
7r

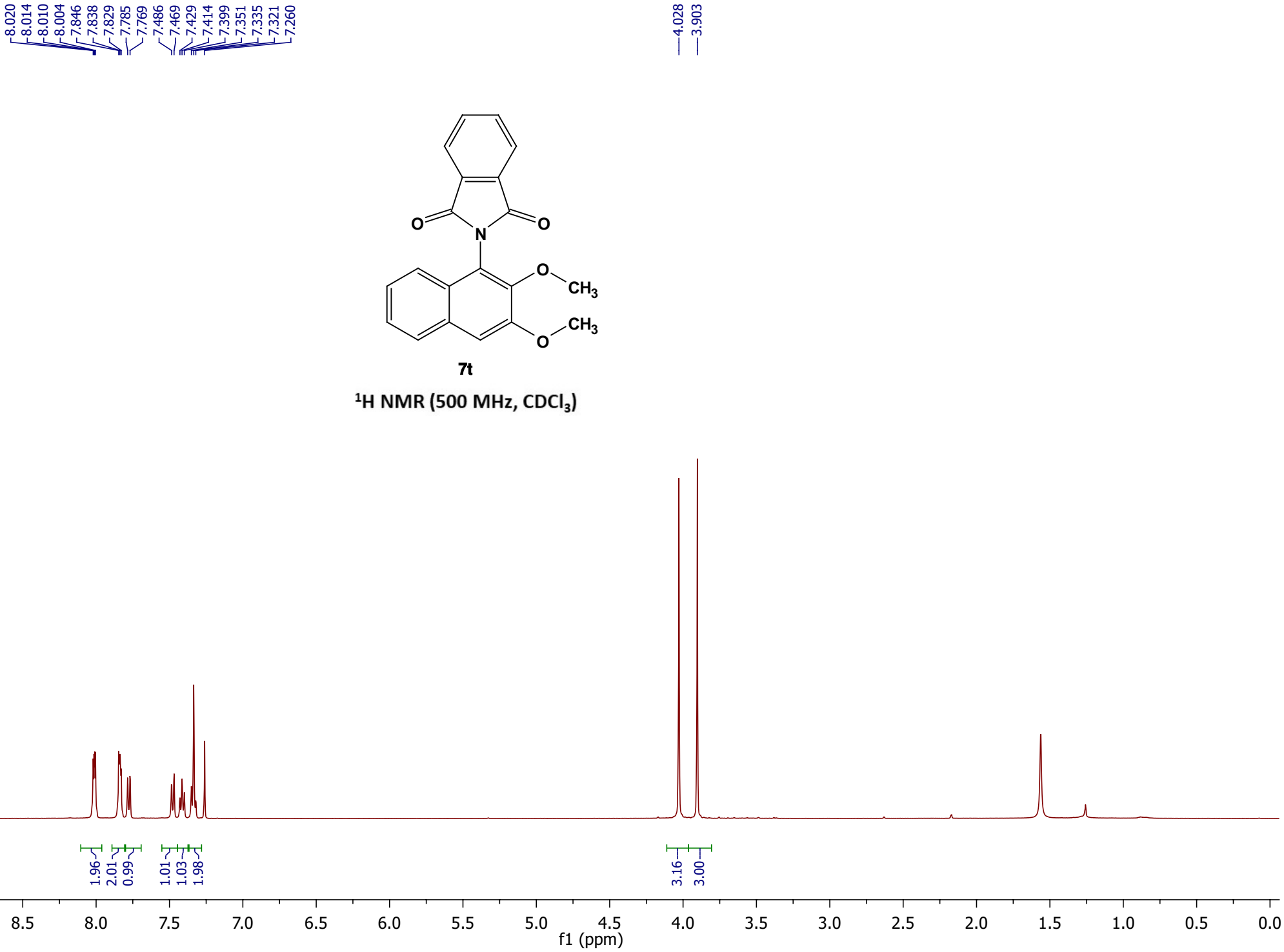
^{13}C NMR (150 MHZ, $CDCl_3$)



7s

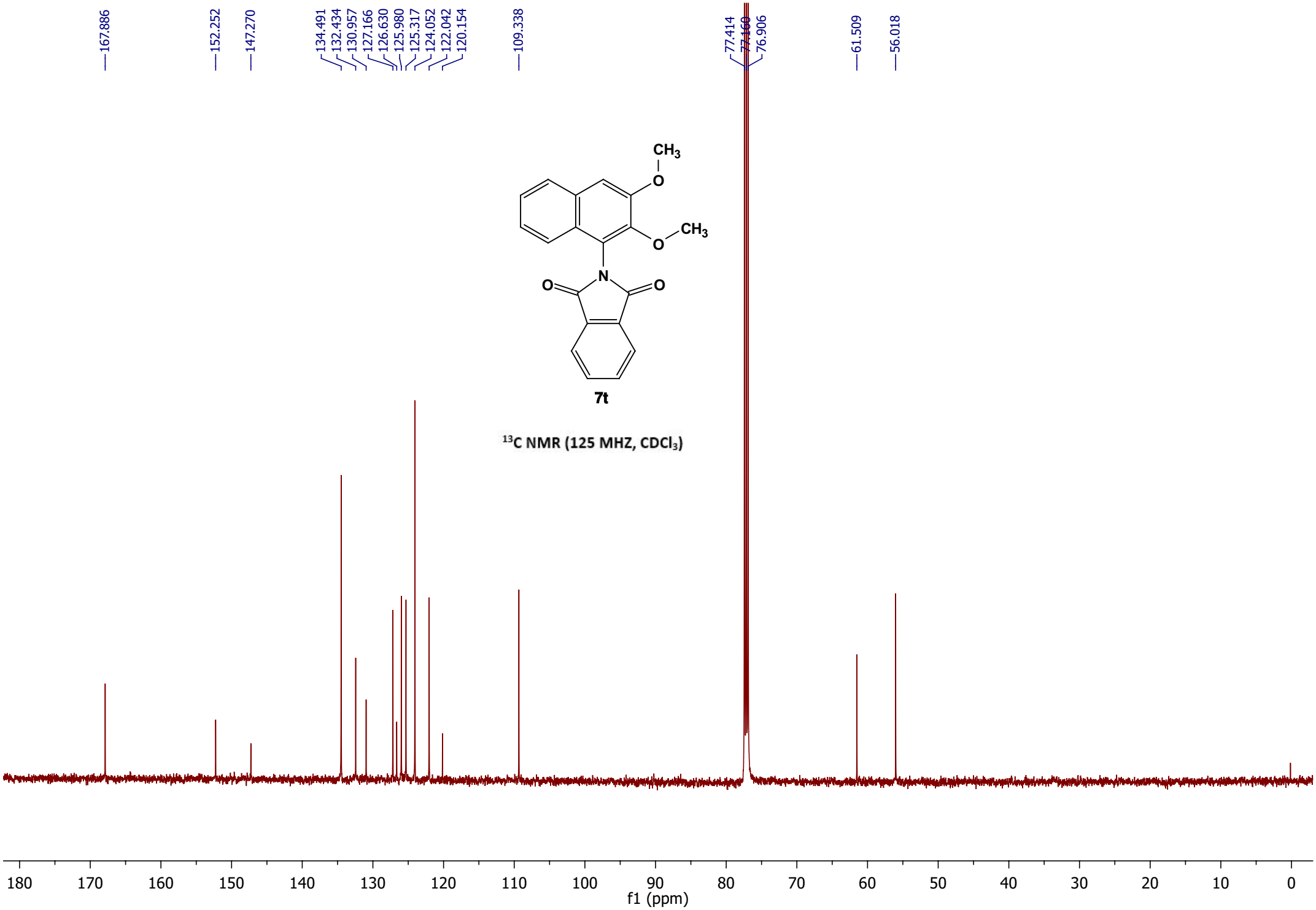
^{1}H NMR (500 MHz, $CDCl_3$)

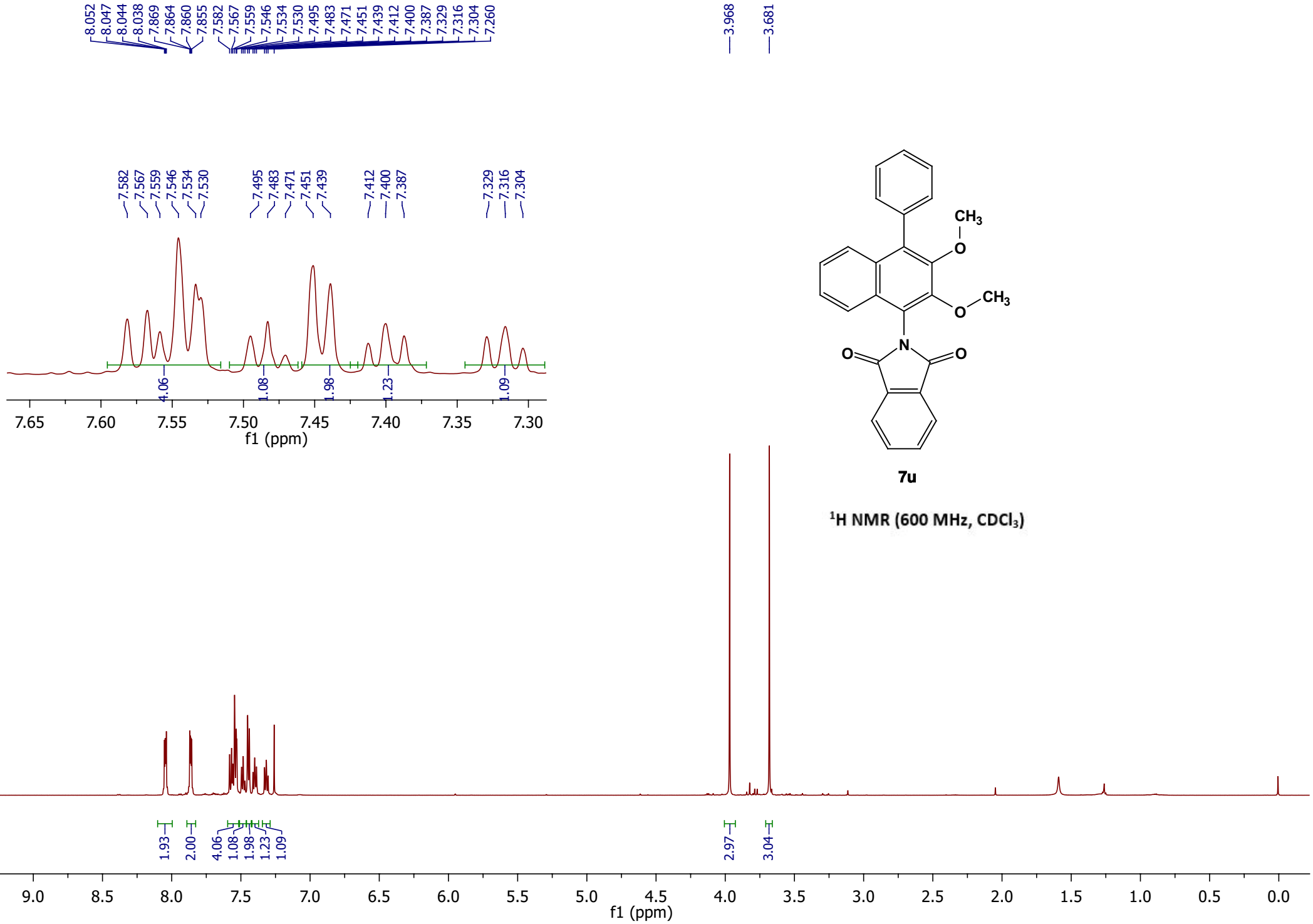

168.113
149.989
149.915
134.508
132.408
130.523
129.347
128.654
126.358
125.668
124.720
124.038
122.479
118.182
77.415
77.160
76.907
61.352
60.991
11.625
CH3
CH3
O
O
CH3
N
O
O
7s
13C NMR (125 MHZ, CDCl3)
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

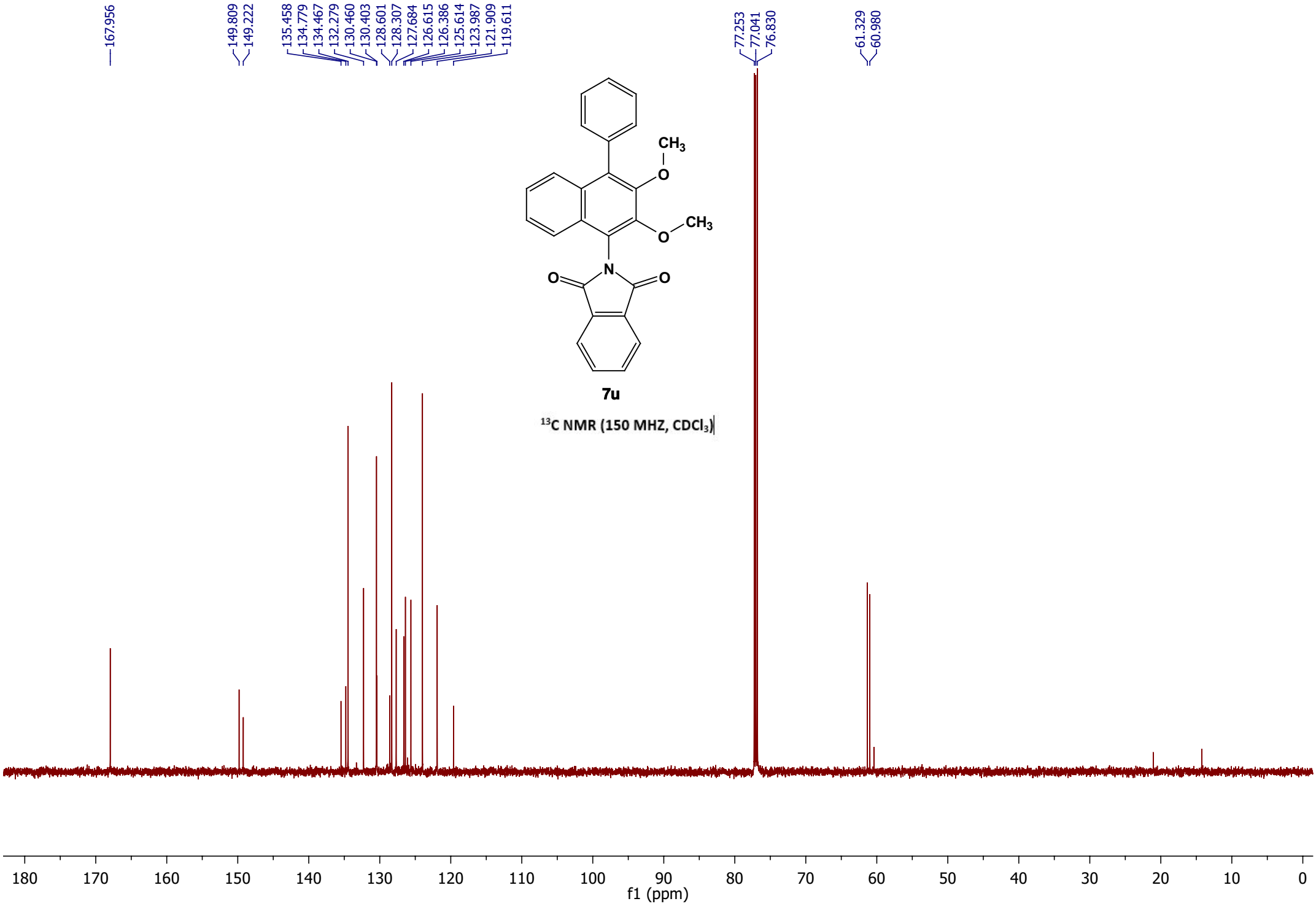


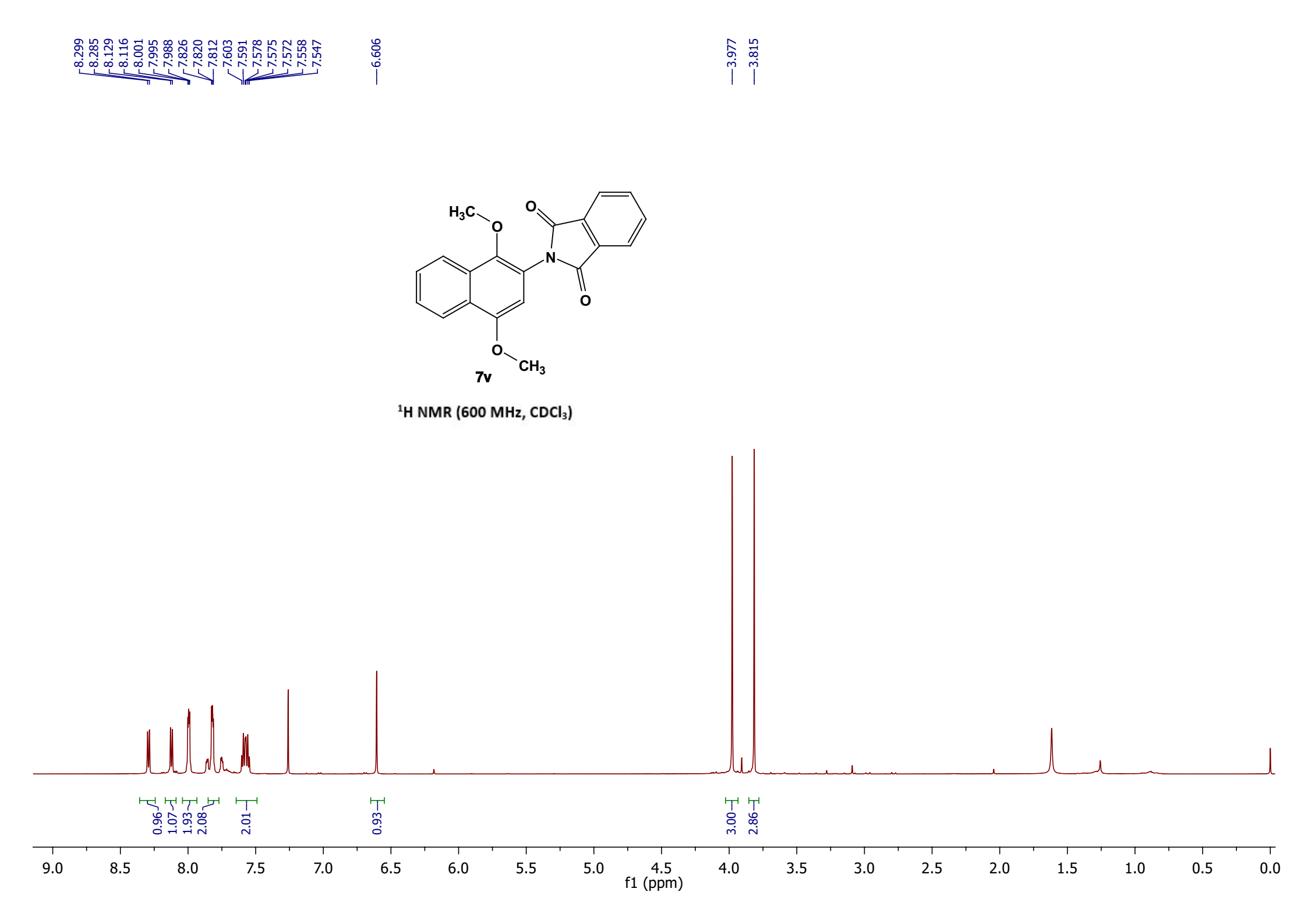
7t

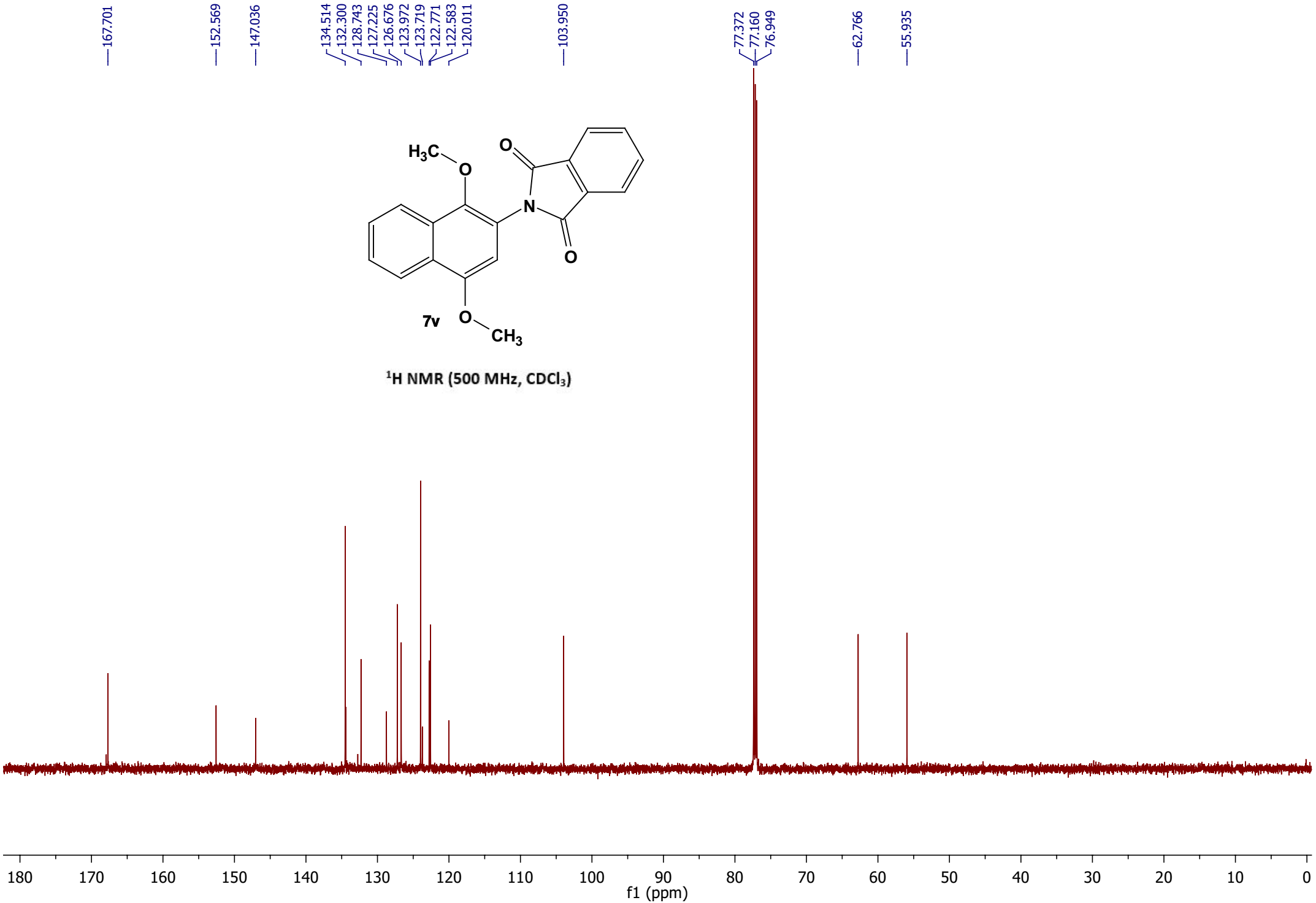
^{1}H NMR (500 MHz, $CDCl_3$)

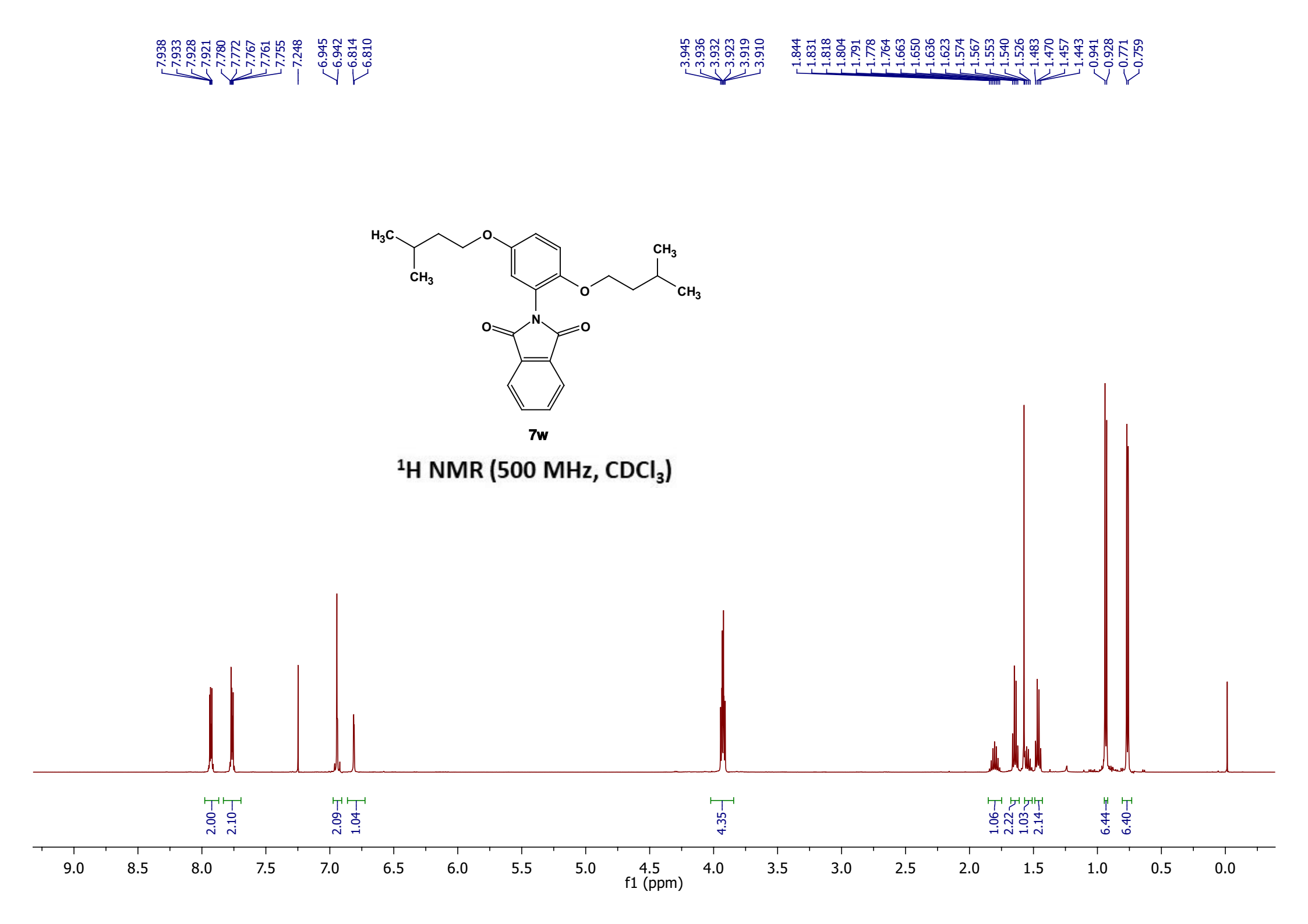

167.886
152.252
147.270
134.491
132.434
130.957
127.166
126.630
125.980
125.317
124.052
122.042
120.154
109.338
77.414
77.160
76.906
61.509
56.018
CH3
O
CH3
O
N
O
O
7t
13C NMR (125 MHZ, CDCl3)
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

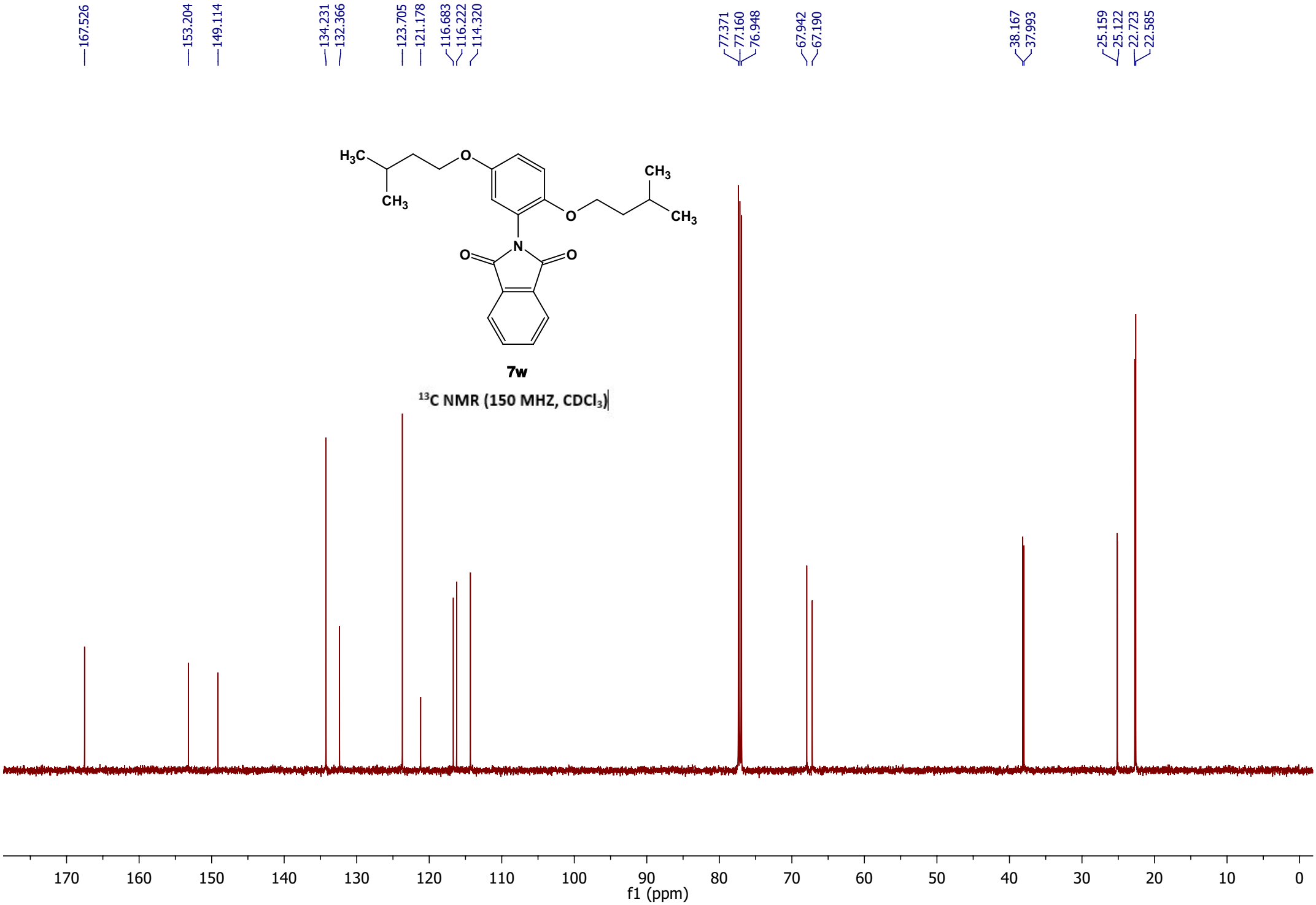

8.052
8.047
8.044
8.038
7.869
7.864
7.860
7.855
7.582
7.567
7.559
7.546
7.534
7.530
7.495
7.483
7.471
7.451
7.439
7.412
7.400
7.387
7.329
7.316
7.304
7.260
3.968
3.681
4.06
1.08
1.98
1.23
1.09
f1 (ppm)
CH3
O
CH3
O
N
O
O
7u
1H NMR (600 MHz, CDCl3)
1.93
2.00
4.06
1.08
1.98
1.23
1.09
2.97
3.04
f1 (ppm)

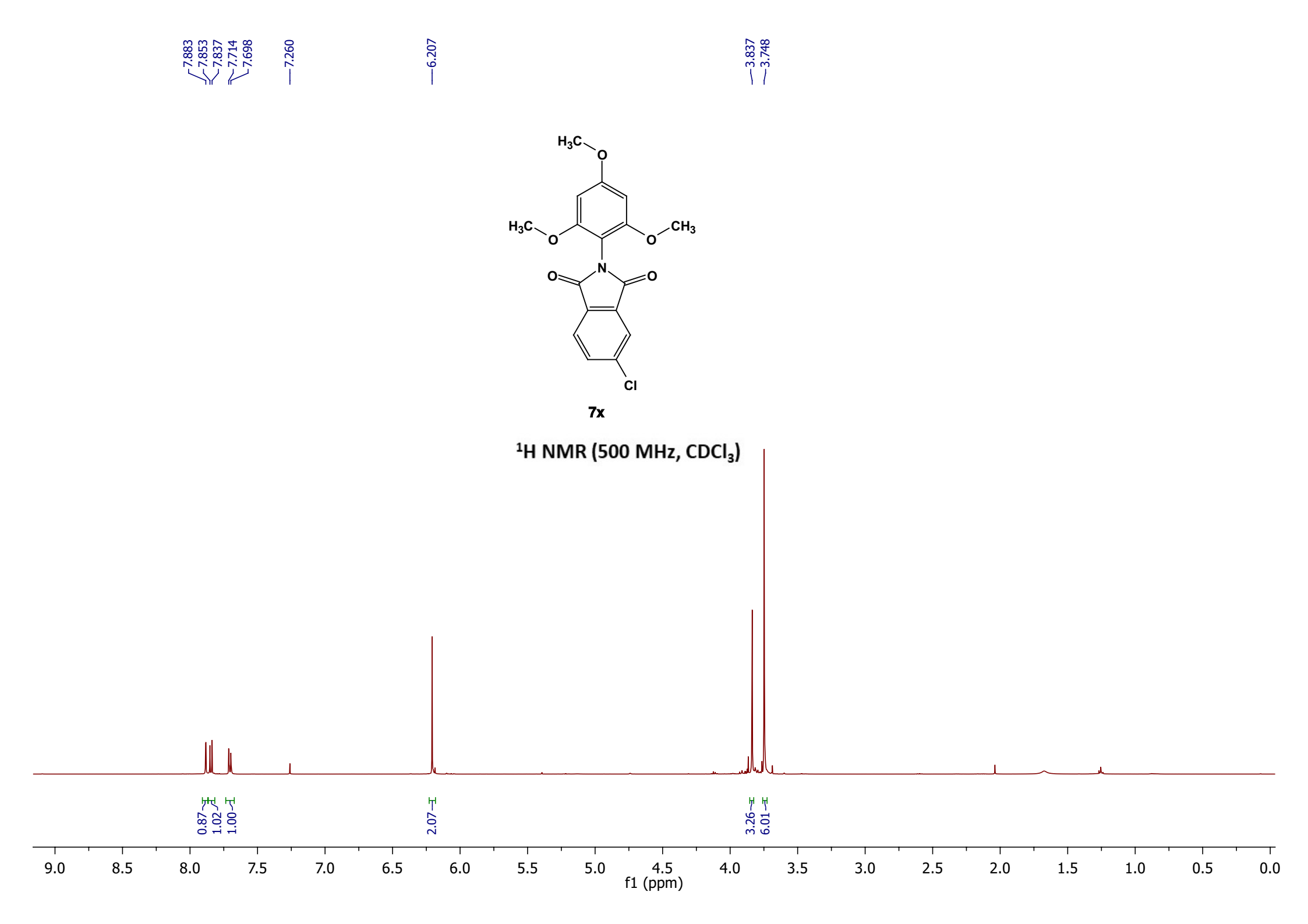

167.956
149.809
149.222
135.458
134.779
134.467
132.279
130.460
130.403
128.601
128.307
127.684
126.615
126.386
125.614
123.987
121.909
119.611
77.253
77.041
76.830
61.329
60.980
CH3
O
O
CH3
N
O
O
7u
13C NMR (150 MHZ, CDCl3)
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

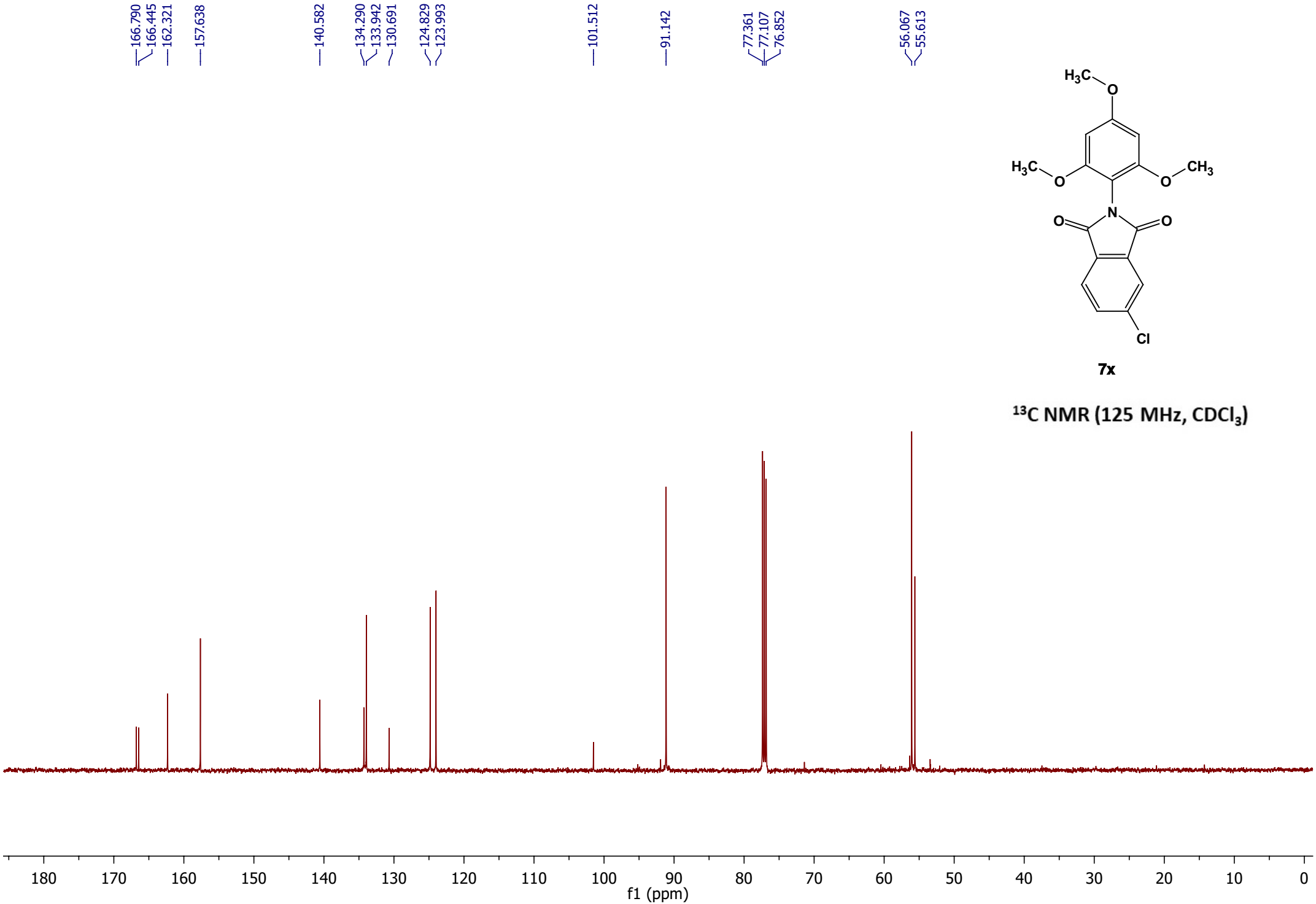

8.299
8.285
8.129
8.116
8.001
7.995
7.988
7.826
7.820
7.812
7.603
7.591
7.578
7.575
7.572
7.558
7.547
6.606
3.977
3.815
H3C
O
O
N
O
O
O
CH3
7v
1H NMR (600 MHz, CDCl3)
0.96
1.07
1.93
2.08
2.01
0.93
3.00
2.86
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

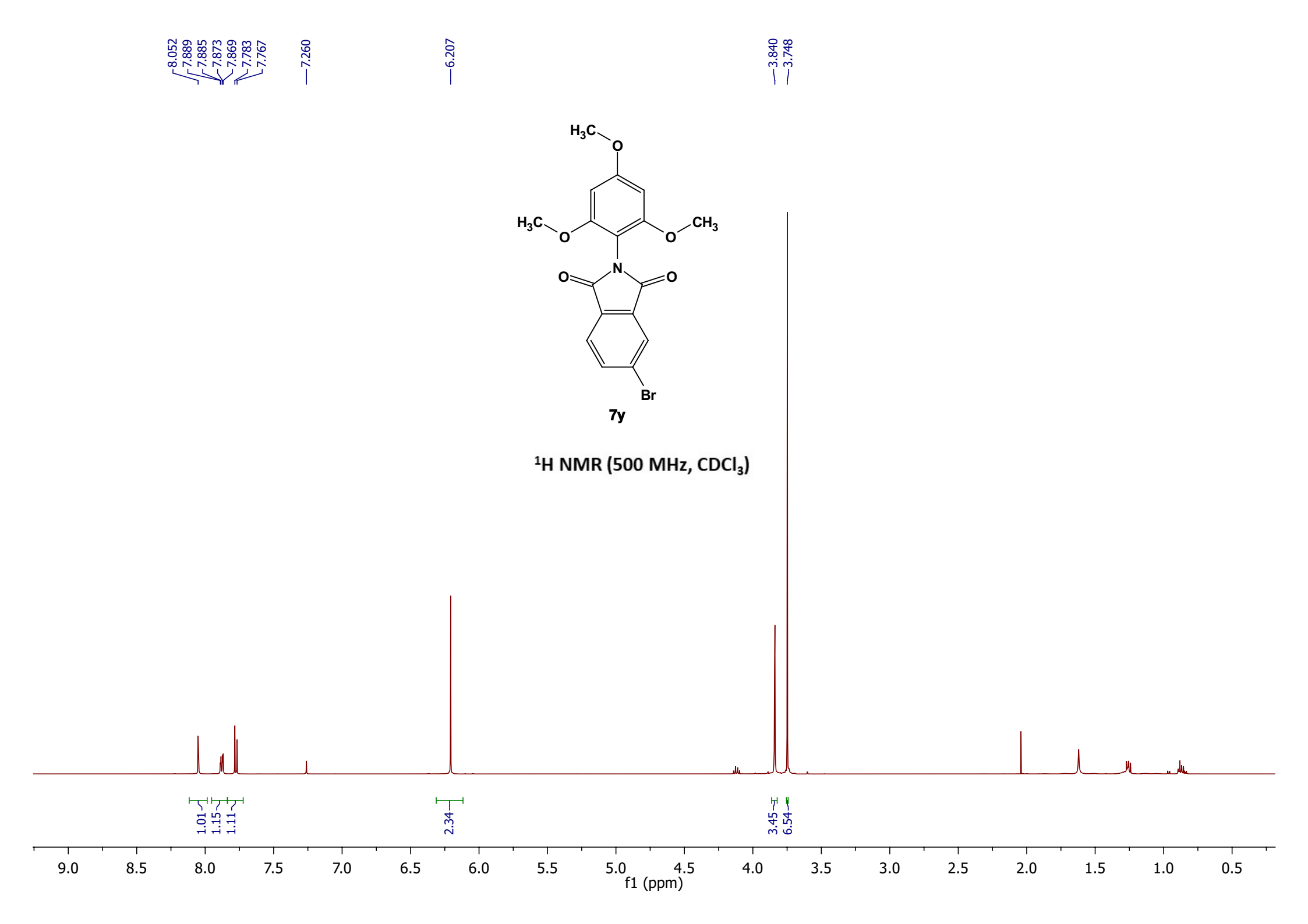

167.701
152.569
147.036
134.514
132.300
128.743
127.225
126.676
123.972
123.719
122.771
122.583
120.011
103.950
77.372
77.160
76.949
62.766
55.935
H3C
O
O
N
O
O
7v
CH3
1H NMR (500 MHz, CDCl3)
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

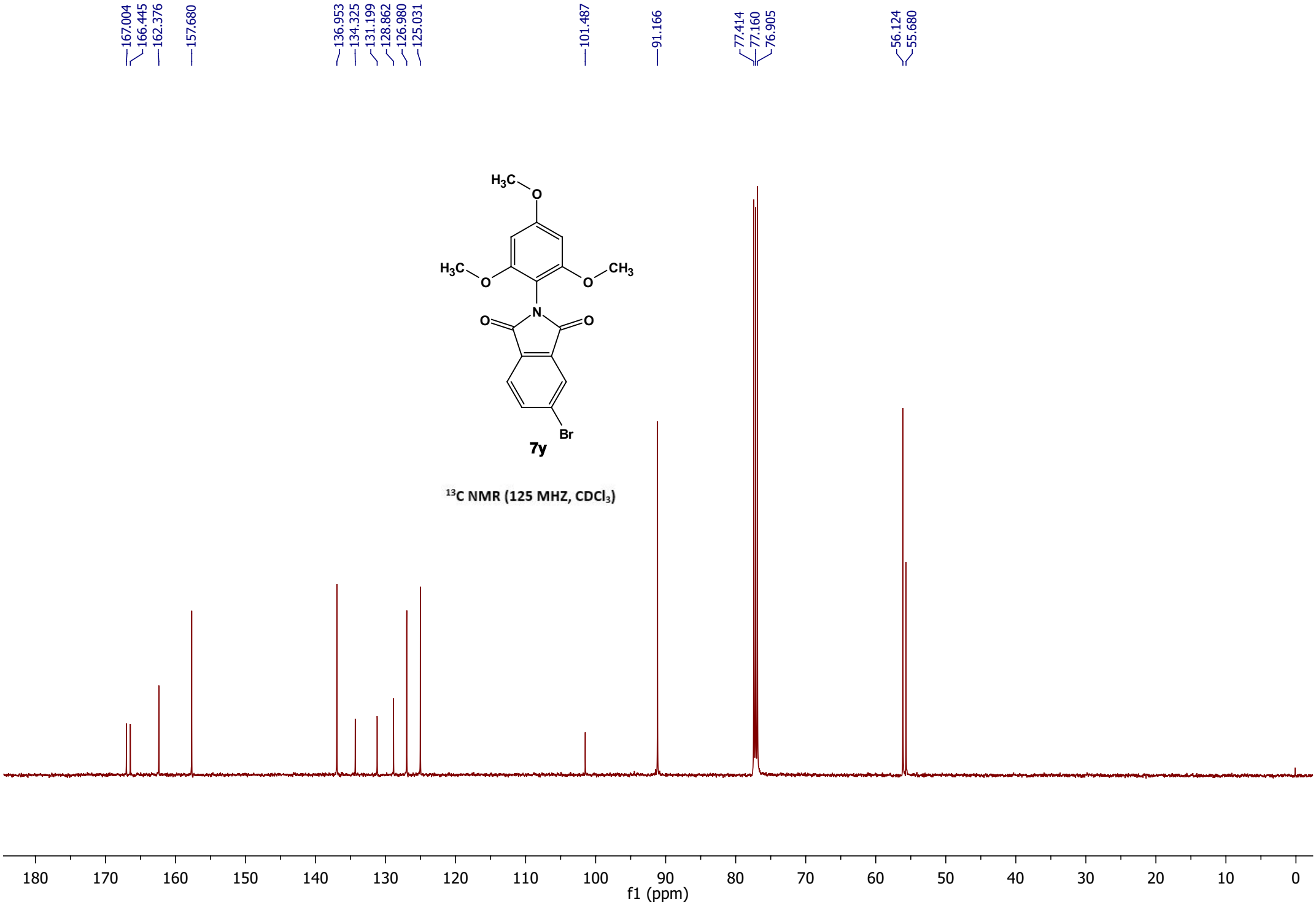

7.938
7.933
7.928
7.921
7.780
7.772
7.767
7.761
7.755
7.248
6.945
6.942
6.814
6.810
3.945
3.936
3.932
3.923
3.919
3.910
1.844
1.831
1.818
1.804
1.791
1.778
1.764
1.663
1.650
1.636
1.623
1.574
1.567
1.553
1.540
1.526
1.483
1.470
1.457
1.443
0.941
0.928
0.771
0.759
H3C
CH3
O
CH3
CH3
O
N
O
O
7w
1H NMR (500 MHz, CDCl3)
2.00
2.10
2.09
1.04
4.35
1.06
2.22
1.03
2.14
6.44
6.40
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)

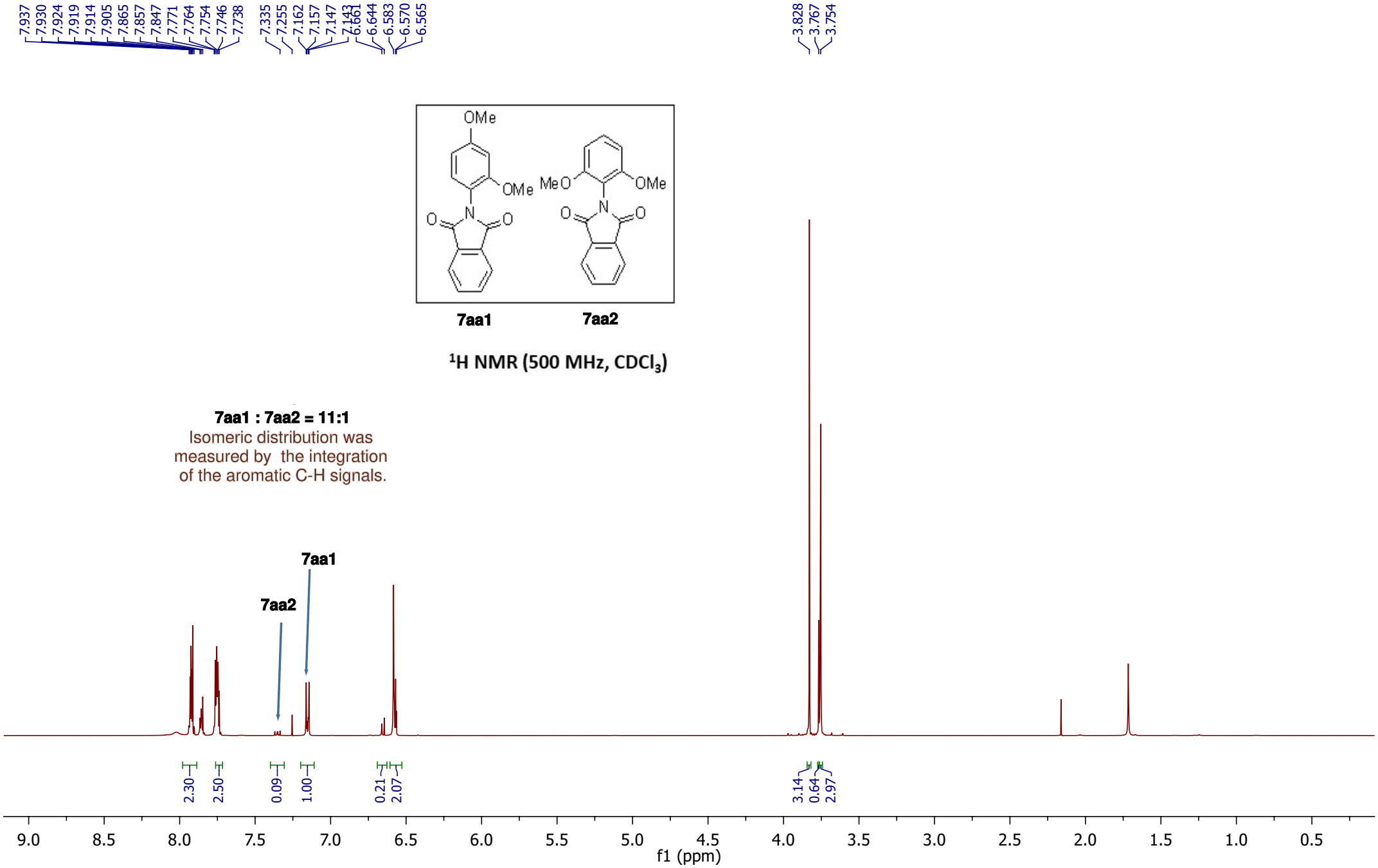

167.526
153.204
149.114
134.231
132.366
123.705
121.178
116.683
116.222
114.320
77.371
77.160
76.948
67.942
67.190
38.167
37.993
25.159
25.122
22.723
22.585
H3C
CH3
O
CH3
O
CH3
N
O
O
7w
13C NMR (150 MHZ, CDCl3)
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

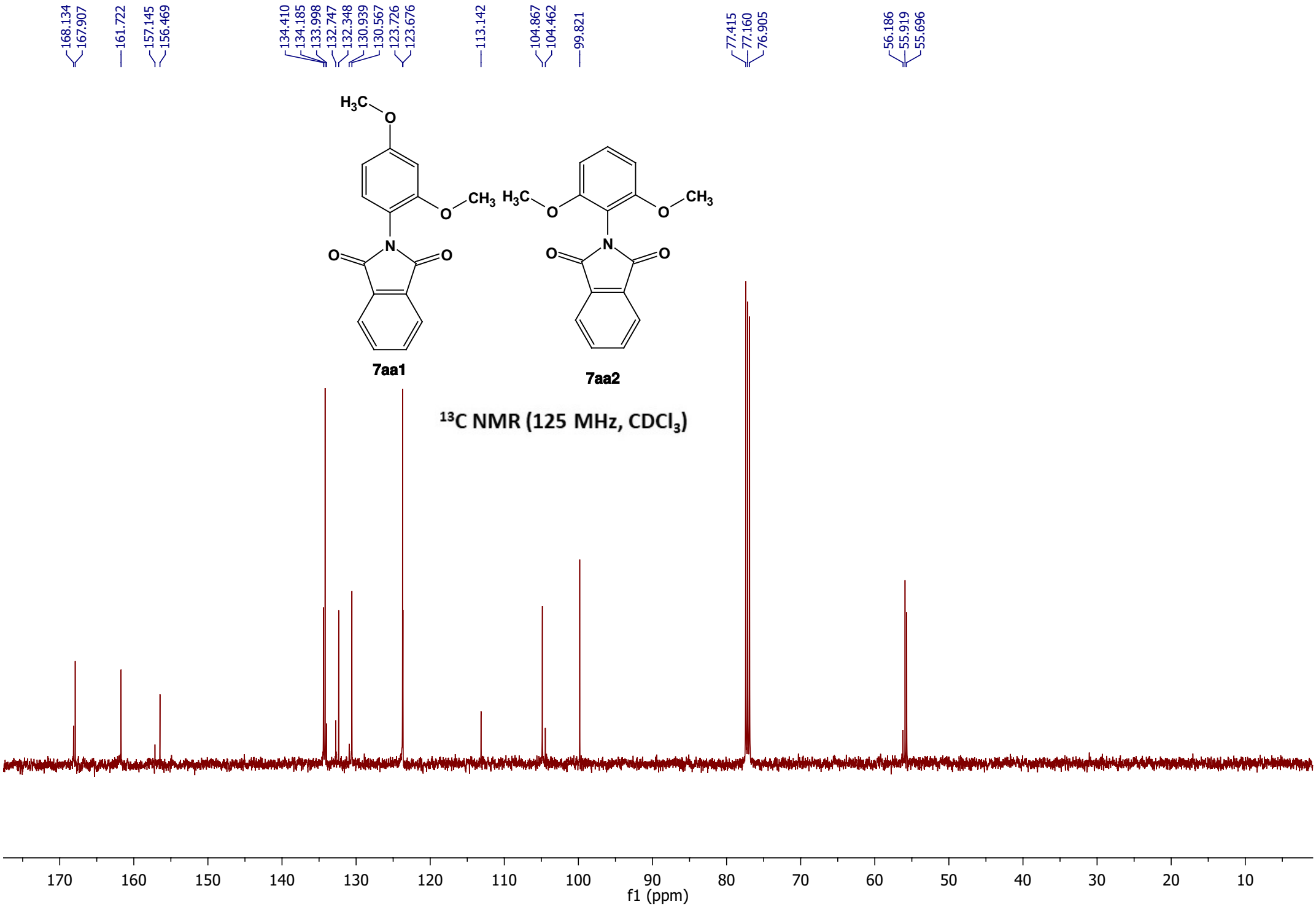

7.883
7.853
7.837
7.714
7.698
7.260
6.207
3.837
3.748
H3C
O
H3C
O
O
CH3
N
O
O
Cl
7x
1H NMR (500 MHz, CDCl3)
0.87
1.02
1.00
2.07
3.26
6.01
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

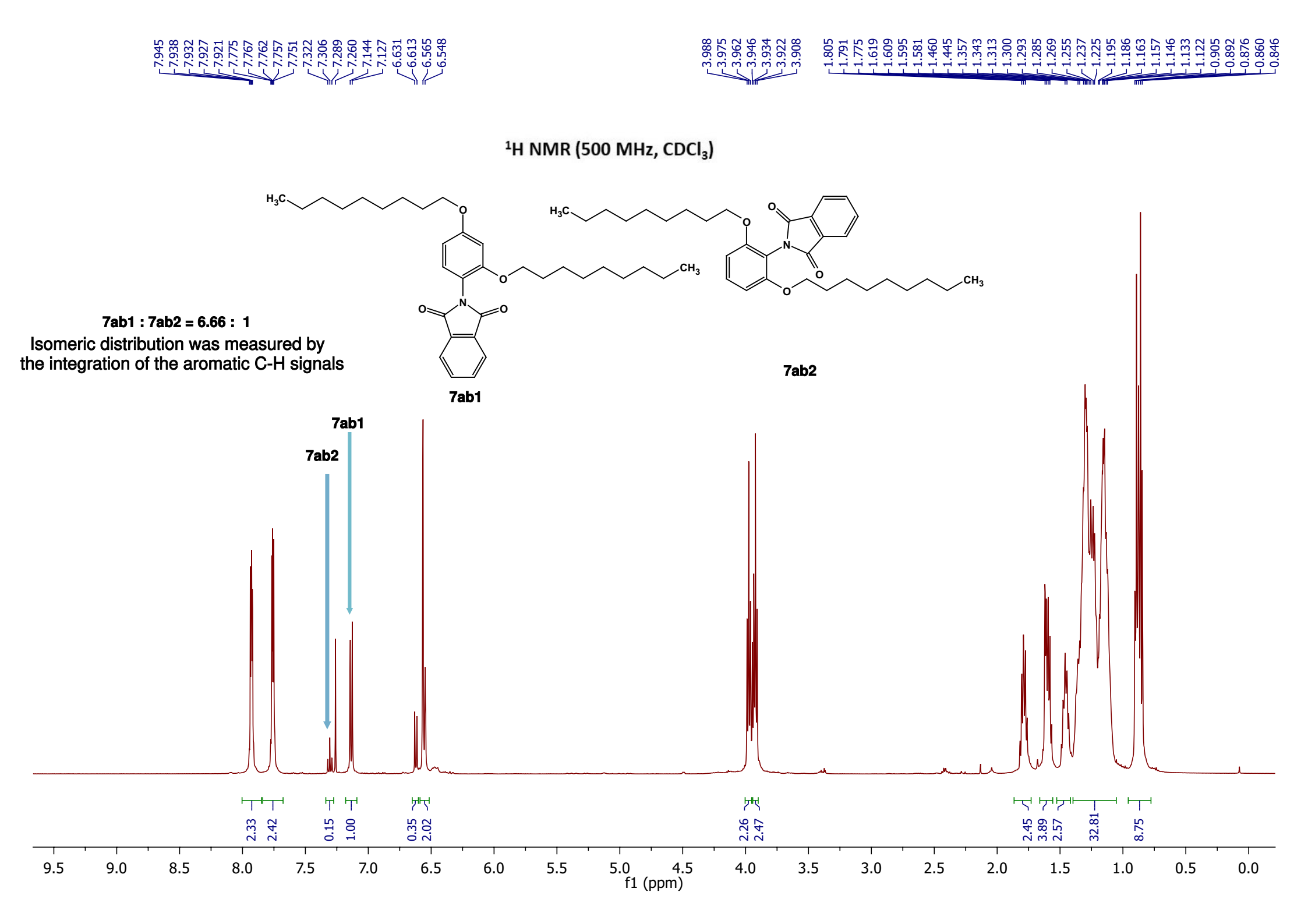

166.790
166.445
162.321
157.638
140.582
134.290
133.942
130.691
124.829
123.993
101.512
91.142
77.361
77.107
76.852
56.067
55.613
H3C
O
H3C
O
O
CH3
N
O
O
Cl
7x
13C NMR (125 MHz, CDCl3)
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

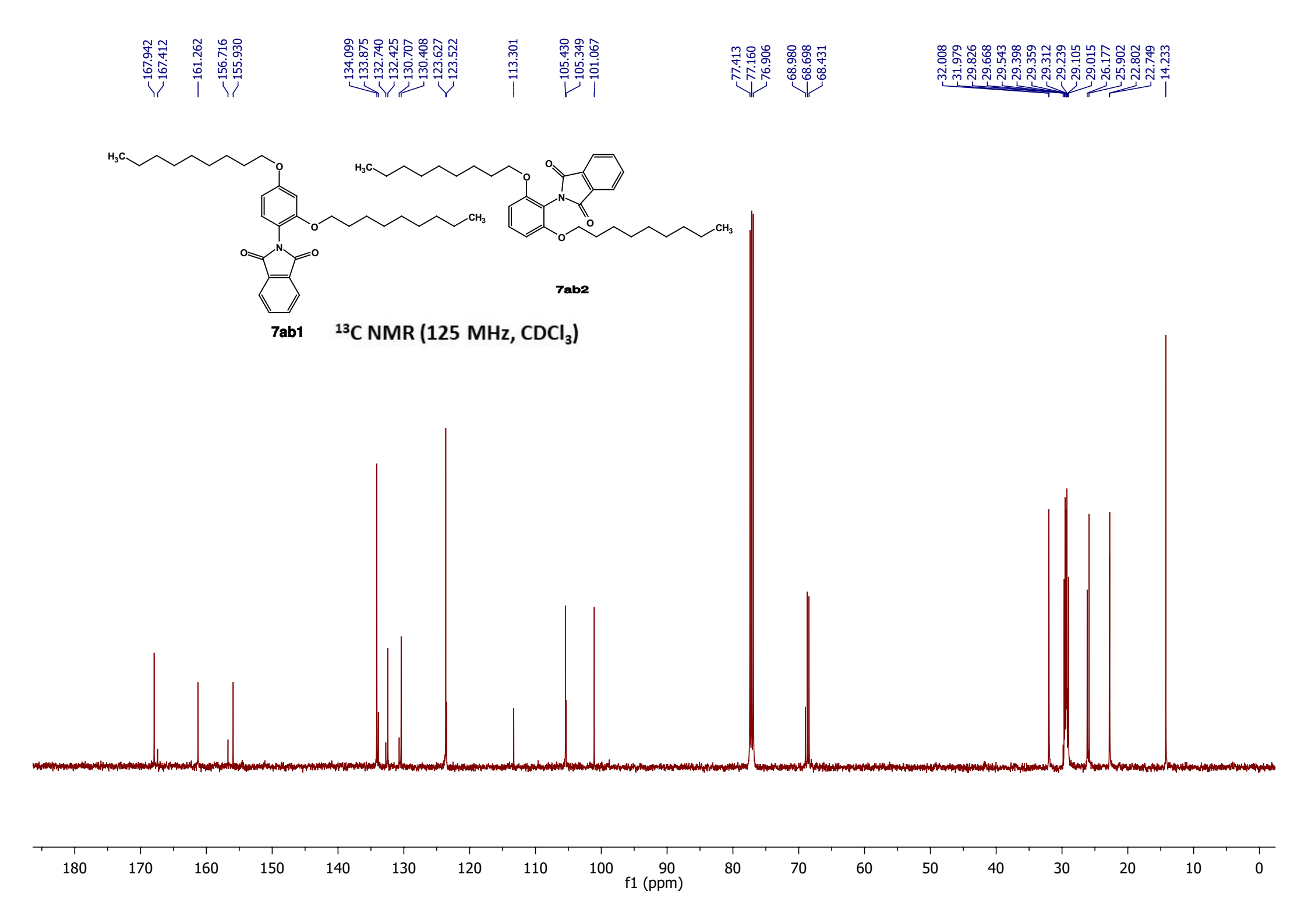

8.052
7.889
7.885
7.873
7.869
7.783
7.767
7.260
6.207
3.840
3.748
H3C
O
H3C
O
O
CH3
N
O
O
Br
7y
1H NMR (500 MHz, CDCl3)
1.01
1.15
1.11
2.34
3.45
6.54
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

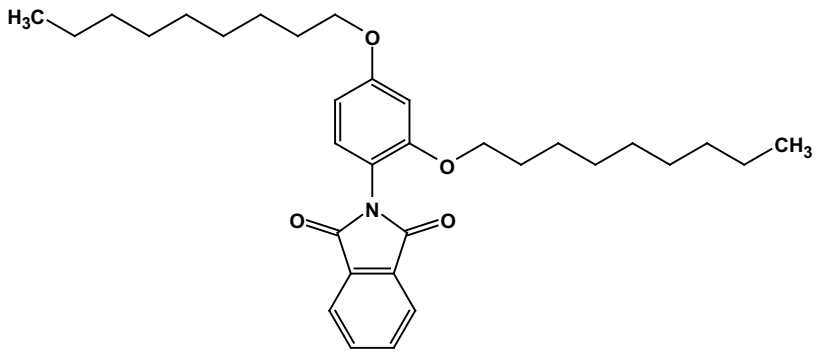

167.004
166.445
162.376
157.680
136.953
134.325
131.199
128.862
126.980
125.031
101.487
91.166
77.414
77.160
76.905
56.124
55.680
H3C
O
H3C
O
O
CH3
N
O
O
Br
7y
13C NMR (125 MHZ, CDCl3)
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.937
7.930
7.924
7.919
7.914
7.905
7.865
7.857
7.847
7.771
7.764
7.754
7.746
7.738
7.335
7.255
7.162
7.157
7.147
7.143
6.661
6.644
6.583
6.570
6.565
3.828
3.767
3.754
OMe
OMe
MeO
OMe
N
O
O
7aa1
7aa2
1H NMR (500 MHz, CDCl3)
7aa1 : 7aa2 = 11:1
Isomeric distribution was measured by the integration of the aromatic C-H signals.
7aa1
7aa2
2.30
2.50
0.09
1.00
0.21
2.07
3.14
0.64
2.97
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)


168.134
167.907
161.722
157.145
156.469
134.410
134.185
133.998
132.747
132.348
130.939
130.567
123.726
123.676
113.142
104.867
104.462
99.821
77.415
77.160
76.905
56.186
55.919
55.696
7aa1
7aa2
13C NMR (125 MHz, CDCl3)
f1 (ppm)

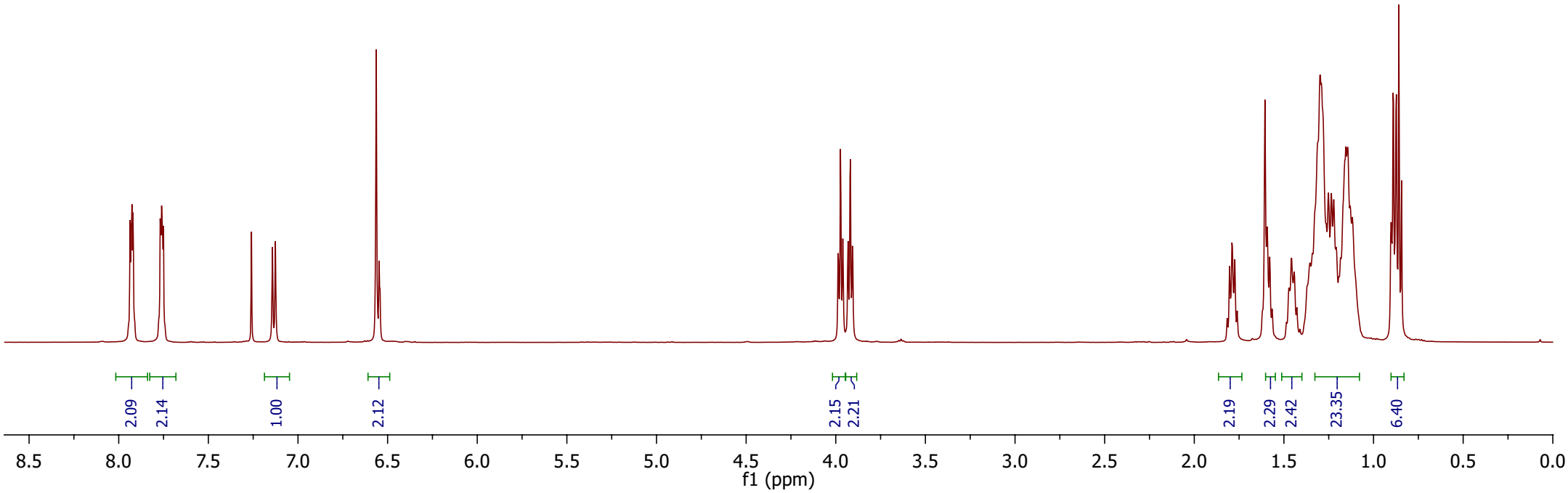

7.945 7.938 7.932 7.927 7.921 7.775 7.767 7.762 7.757 7.751 7.322 7.306 7.289 7.260 7.144 7.127 6.631 6.613 6.565 6.548
3.988 3.975 3.962 3.946 3.934 3.922 3.908
1.805 1.791 1.775 1.619 1.609 1.595 1.581 1.460 1.445 1.357 1.343 1.313 1.300 1.293 1.285 1.269 1.255 1.237 1.225 1.195 1.186 1.163 1.157 1.146 1.133 1.122 0.905 0.892 0.876 0.860 0.846
¹H NMR (500 MHz, CDCl3)
7ab1 : 7ab2 = 6.66 : 1
Isomeric distribution was measured by the integration of the aromatic C-H signals
7ab1
7ab2
7ab1
7ab2
2.33 2.42 0.15 1.00 0.35 2.02 2.26 2.47 2.45 3.89 2.57 32.81 8.75
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)

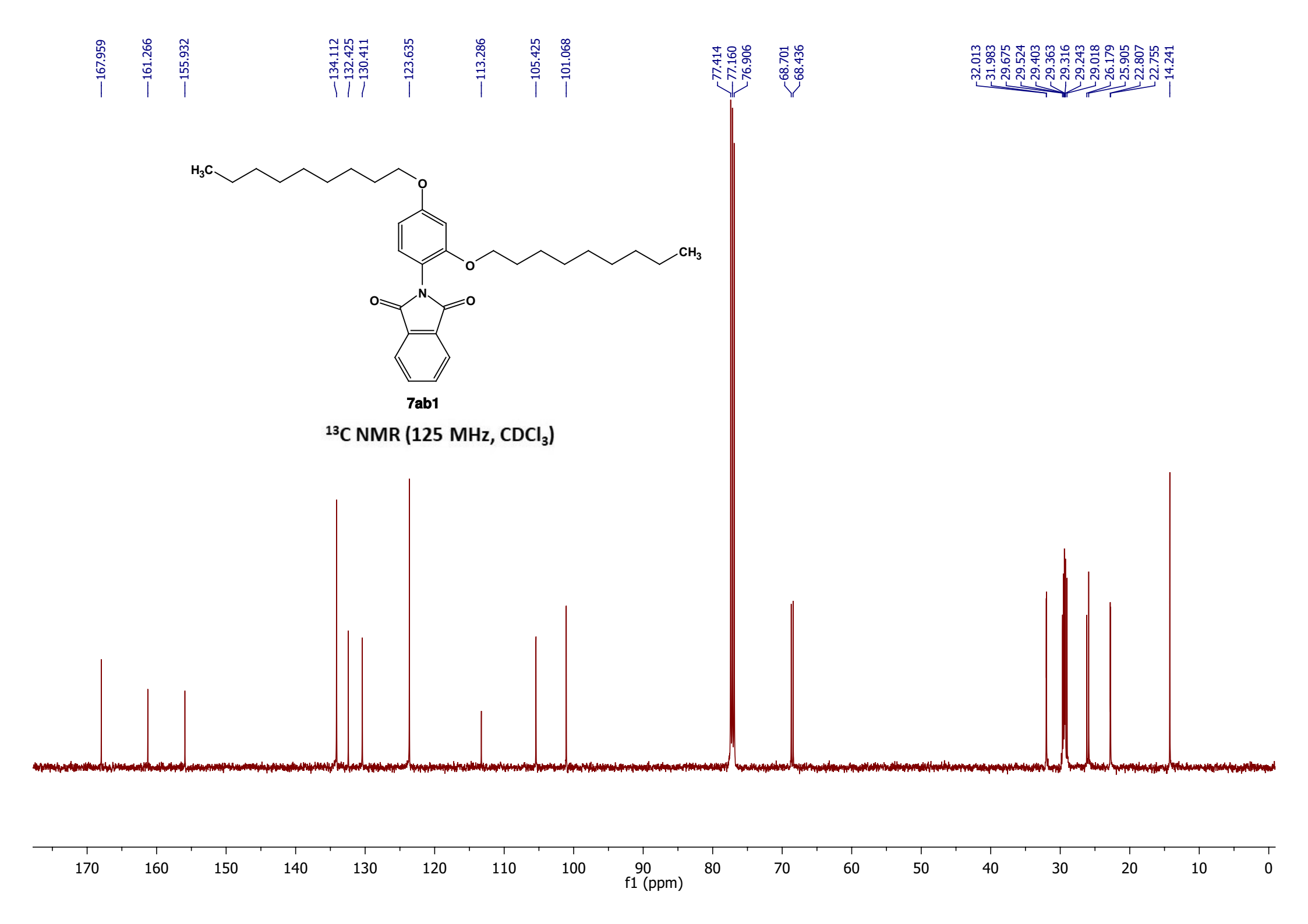

167.942
167.412
161.262
156.716
155.930
134.099
133.875
132.740
132.425
130.707
130.408
123.627
123.522
113.301
105.430
105.349
101.067
77.413
77.160
76.906
68.980
68.698
68.431
32.008
31.979
29.826
29.668
29.543
29.398
29.359
29.312
29.239
29.105
29.015
26.177
25.902
22.802
22.749
14.233
7ab1
7ab2
13C NMR (125 MHz, CDCl3)
f1 (ppm)

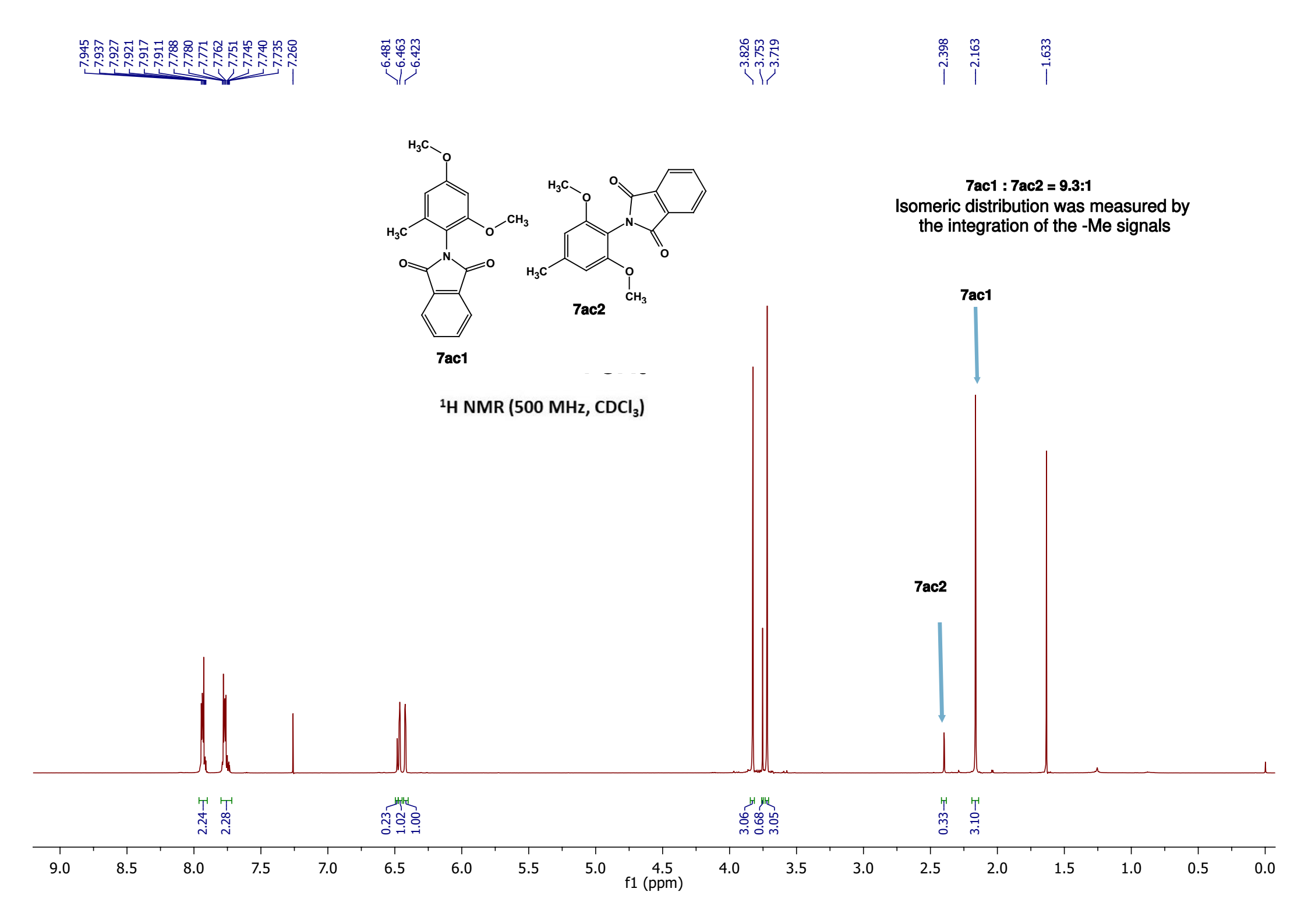


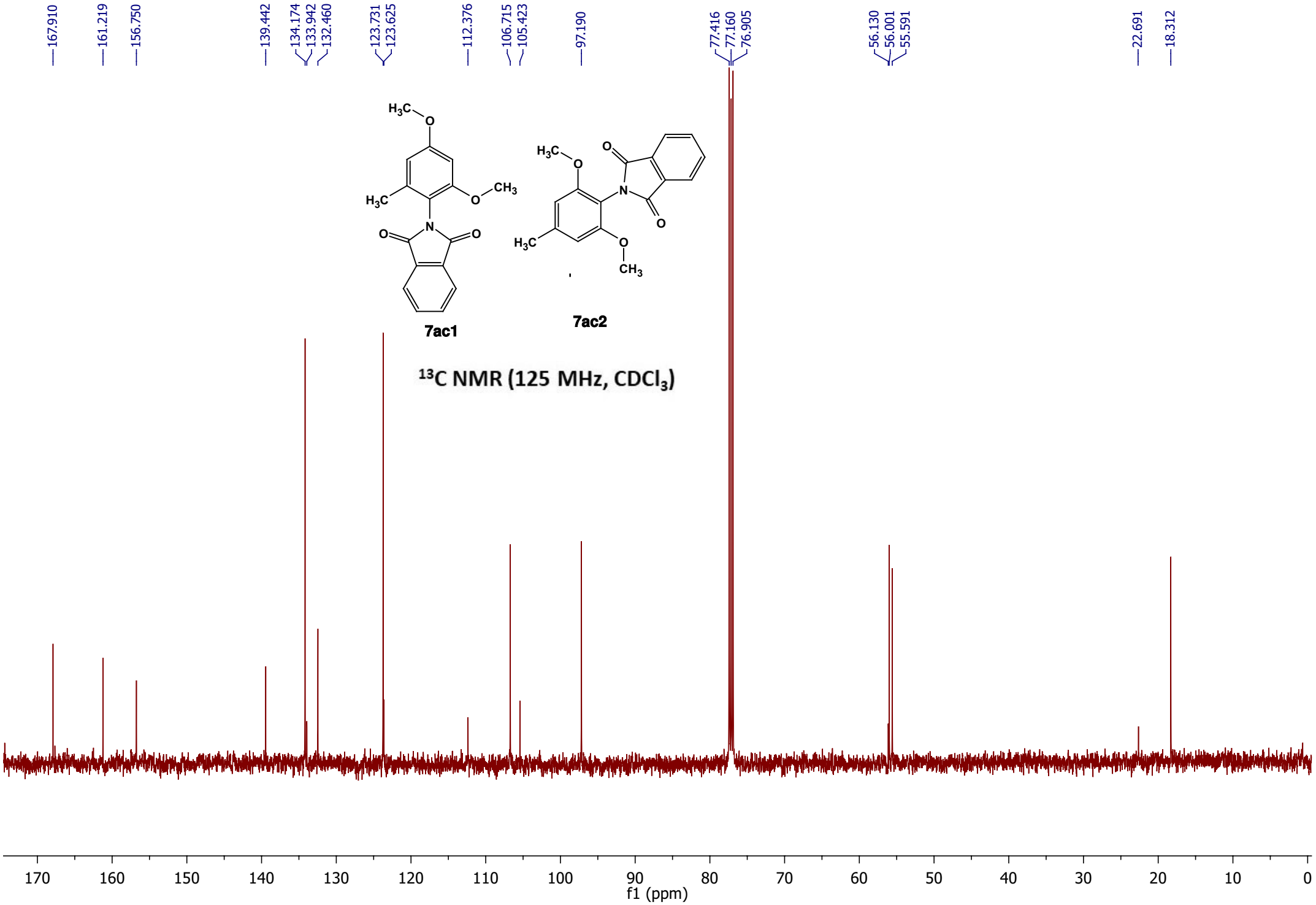
7ab1

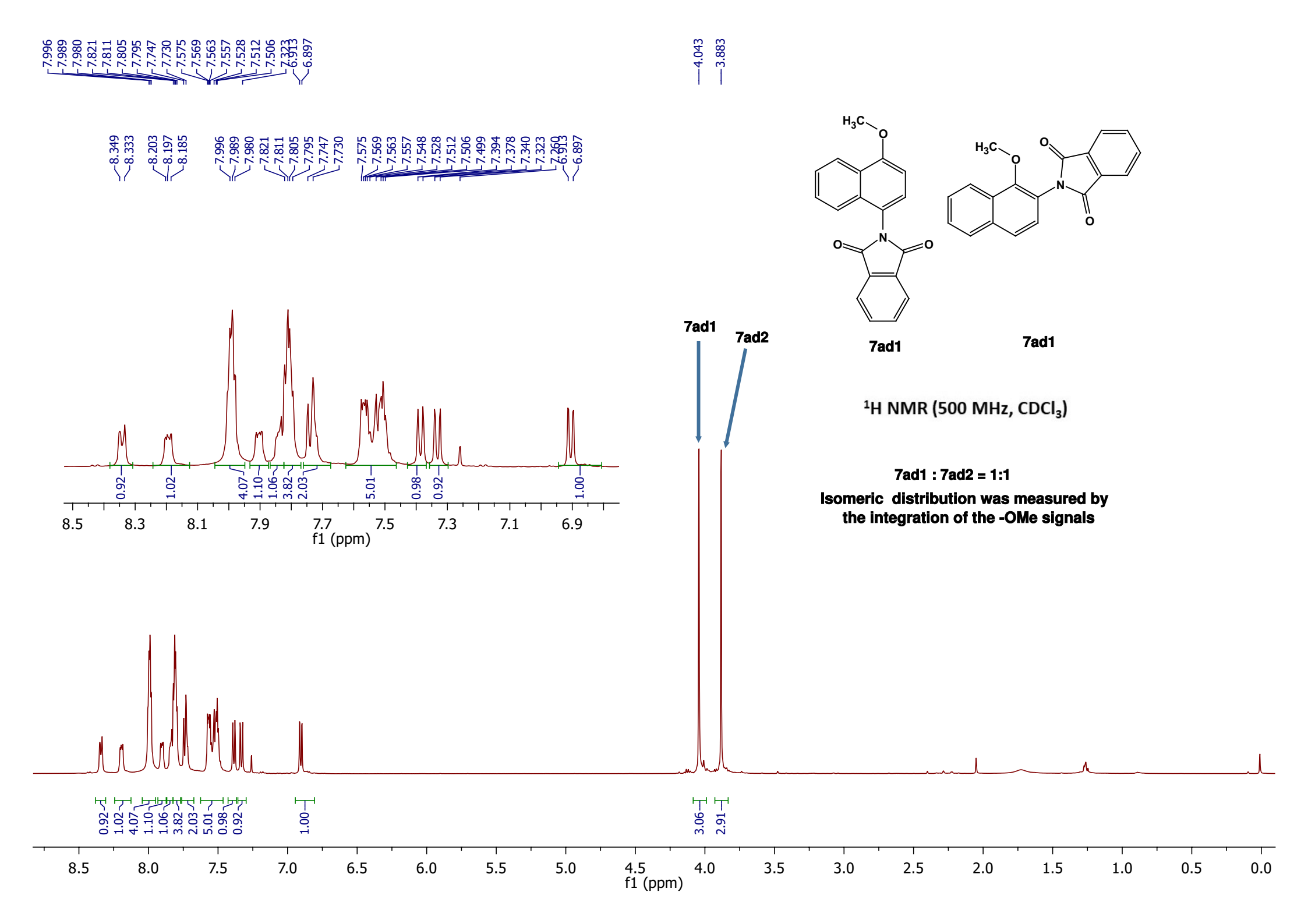
^{1}H NMR (500 MHz, $CDCl_3$)




167.959
161.266
155.932
134.112
132.425
130.411
123.635
113.286
105.425
101.068
77.414
77.160
76.906
68.701
68.436
32.013
31.983
29.675
29.524
29.403
29.363
29.316
29.243
29.018
26.179
25.905
22.807
22.755
14.241
H3C
O
O
N
O
O
CH3
7ab1
13C NMR (125 MHz, CDCl3)
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.945
7.937
7.927
7.921
7.917
7.911
7.788
7.780
7.771
7.762
7.751
7.745
7.740
7.735
7.260
6.481
6.463
6.423
3.826
3.753
3.719
2.398
2.163
1.633
7ac1
7ac2
7ac1 : 7ac2 = 9.3:1
Isomeric distribution was measured by
the integration of the -Me signals
7ac1
7ac2
¹H NMR (500 MHz, CDCl₃)
2.24
2.28
0.23
1.02
1.00
3.06
0.68
3.05
0.33
3.10
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)


167.910
161.219
156.750
139.442
134.174
133.942
132.460
123.731
123.625
112.376
106.715
105.423
97.190
77.416
77.160
76.905
56.130
56.001
55.591
22.691
18.312
7ac1
7ac2
13C NMR (125 MHz, CDCl3)
f1 (ppm)

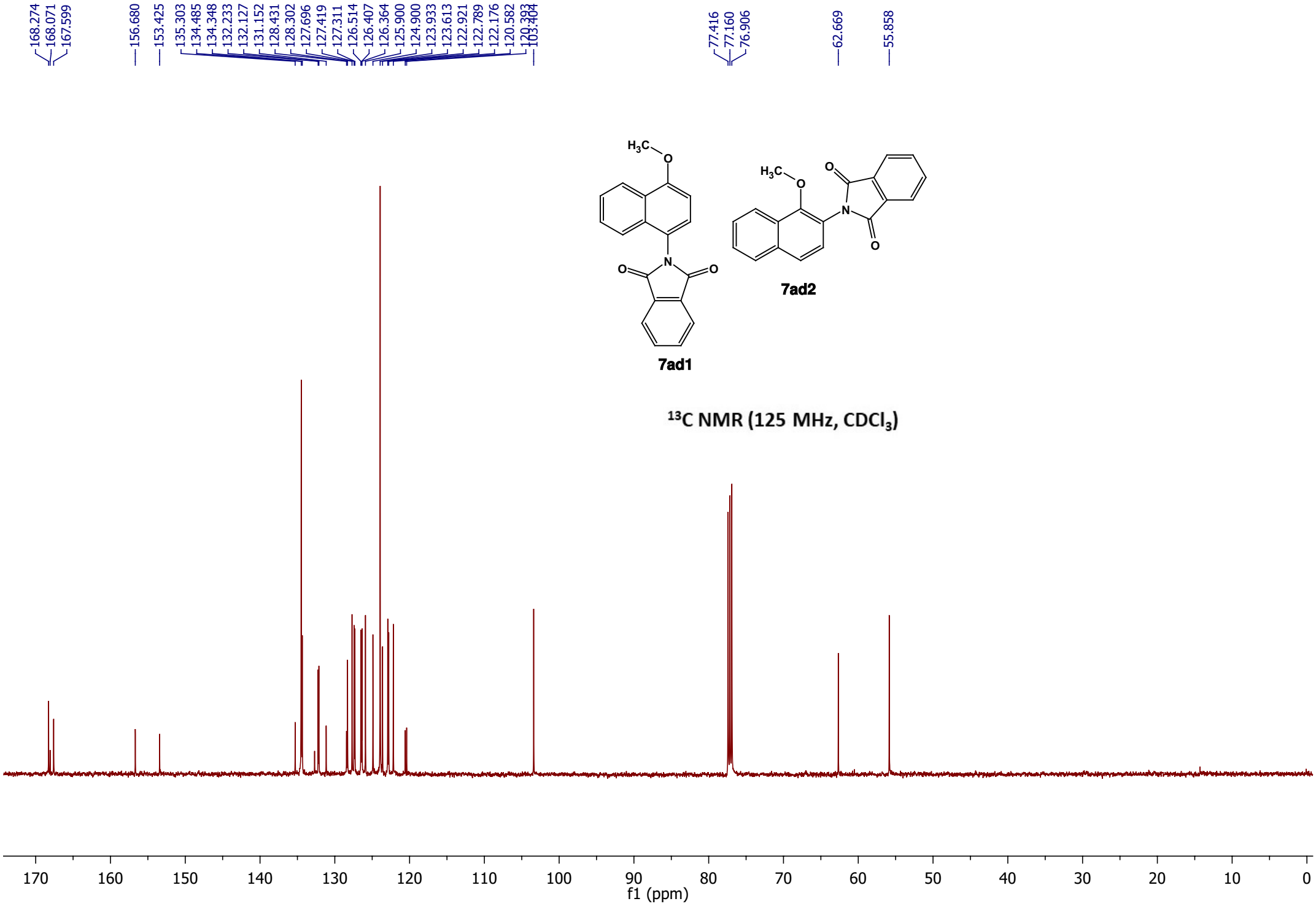


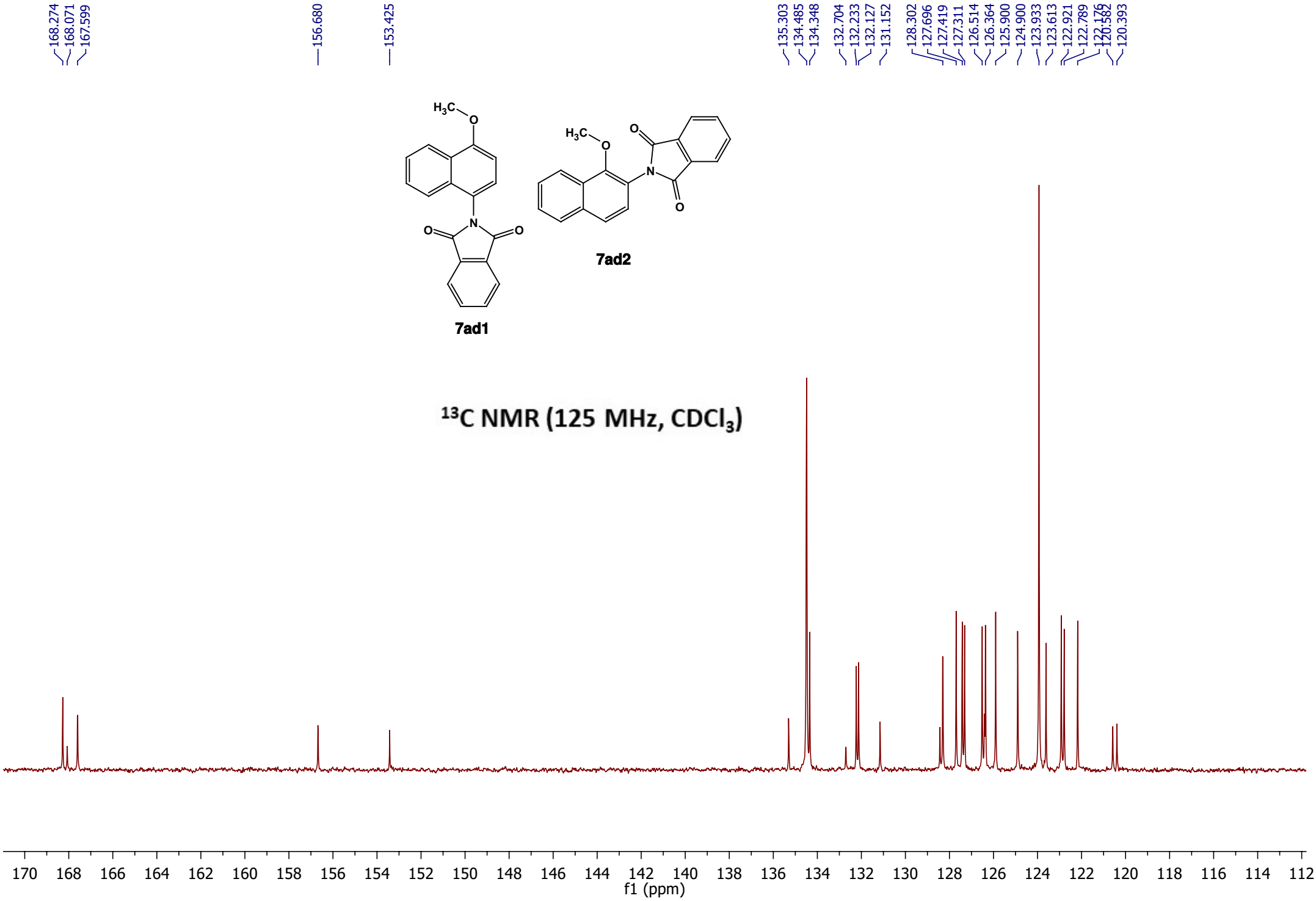
7ad1 **7ad1**

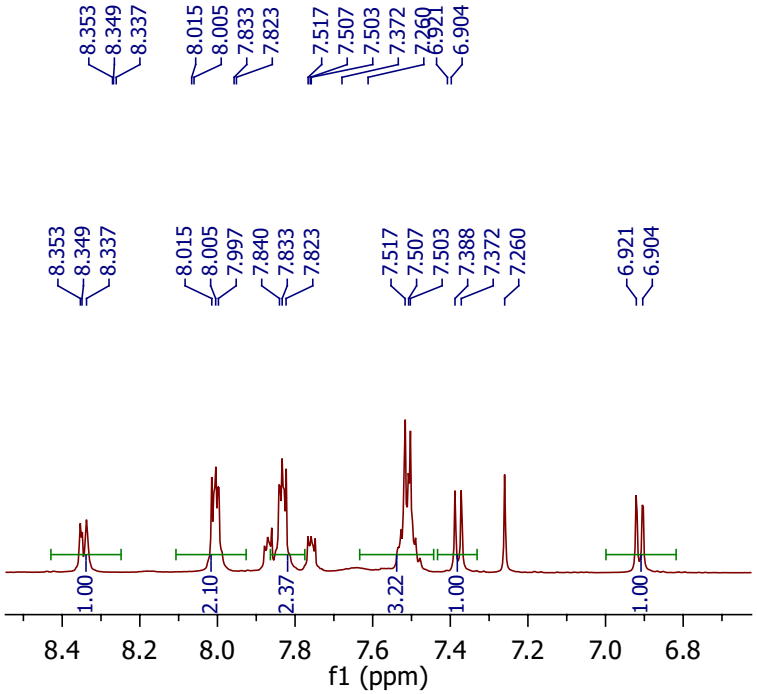
^{1}H NMR (500 MHz, $CDCl_3$)

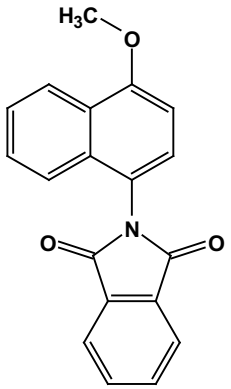
7ad1 : 7ad2 = 1:1

Isomeric distribution was measured by the integration of the -OMe signals


168.274
168.071
167.599
156.680
153.425
135.303
134.485
134.348
132.233
132.127
131.152
128.431
128.302
127.696
127.419
127.311
126.514
126.407
126.364
125.900
124.900
123.933
123.613
122.921
122.789
122.176
120.582
120.393
103.404
77.416
77.160
76.906
62.669
55.858
7ad1
7ad2
13C NMR (125 MHz, CDCl3)
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

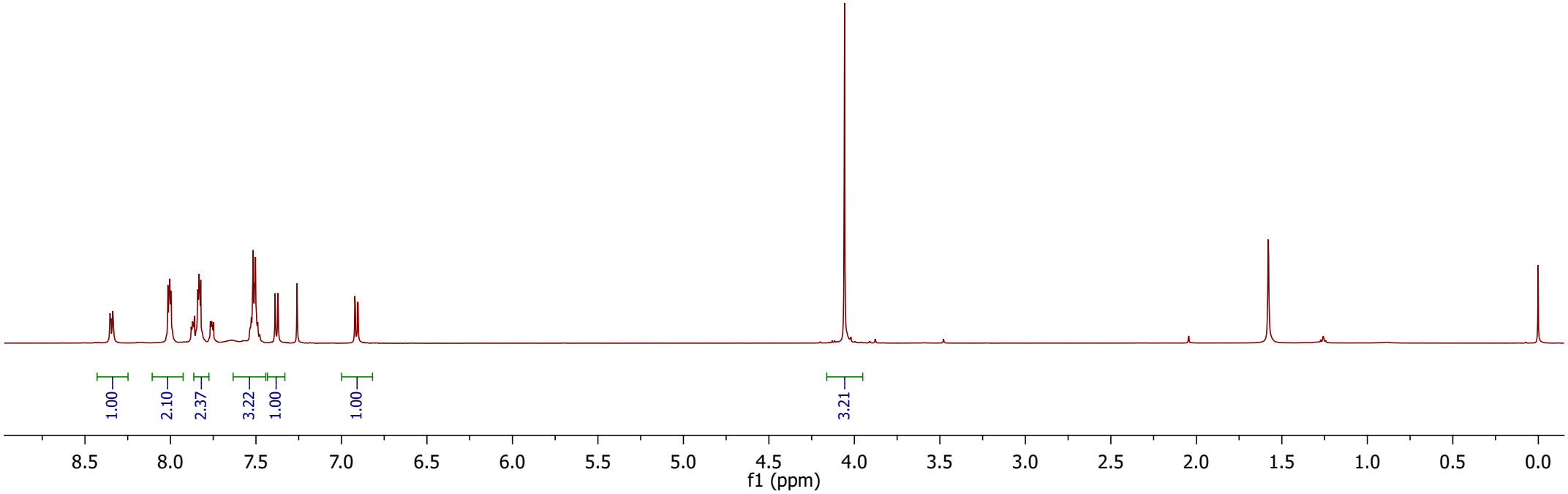

168.274
168.071
167.599
156.680
153.425
135.303
134.485
134.348
132.704
132.233
132.127
131.152
128.302
127.696
127.419
127.311
126.514
126.364
125.900
124.900
123.933
123.613
122.921
122.789
120.582
120.393
7ad1
7ad2
13C NMR (125 MHz, CDCl3)
170 168 166 164 162 160 158 156 154 152 150 148 146 144 142 140 138 136 134 132 130 128 126 124 122 120 118 116 114 112
f1 (ppm)

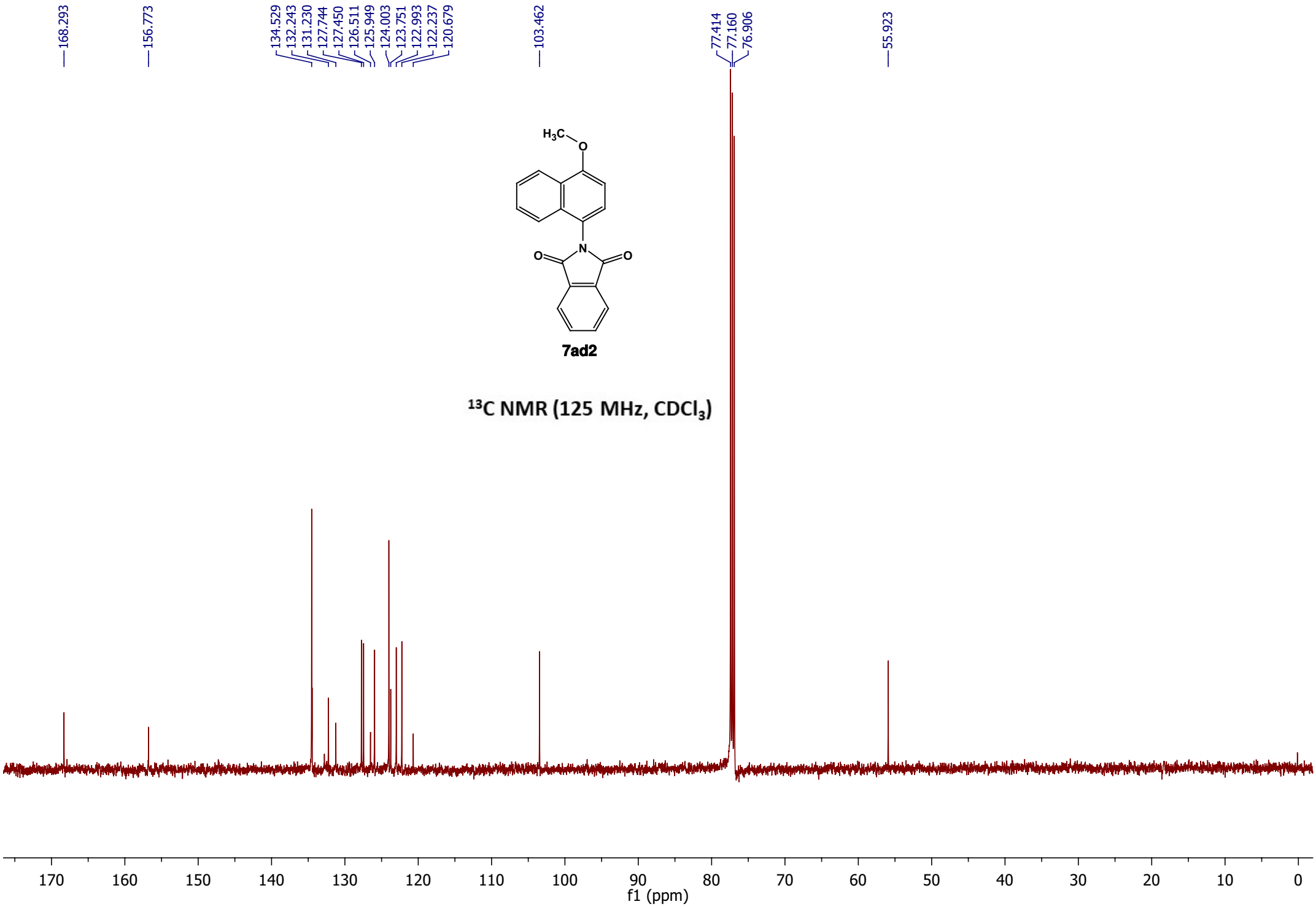


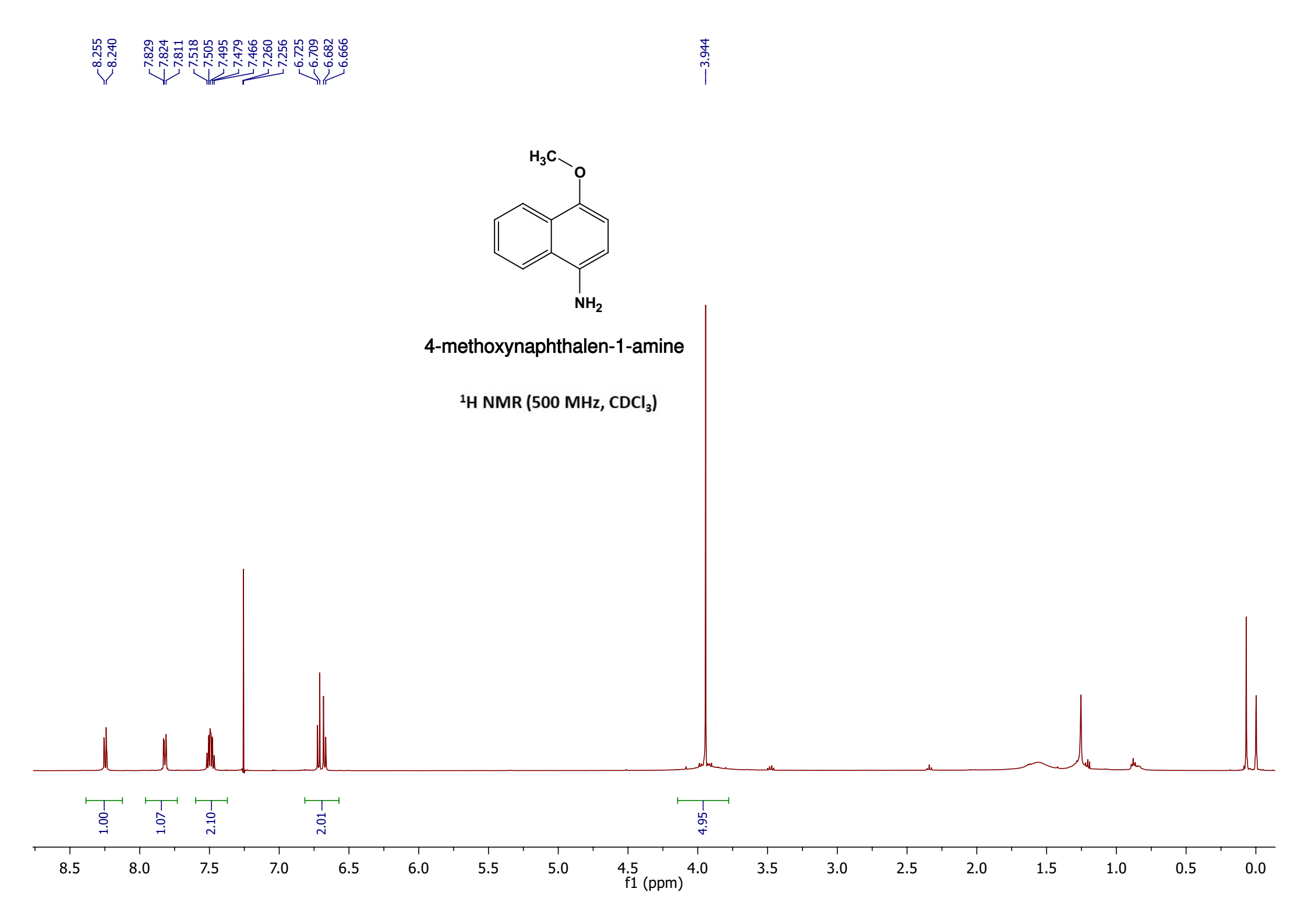


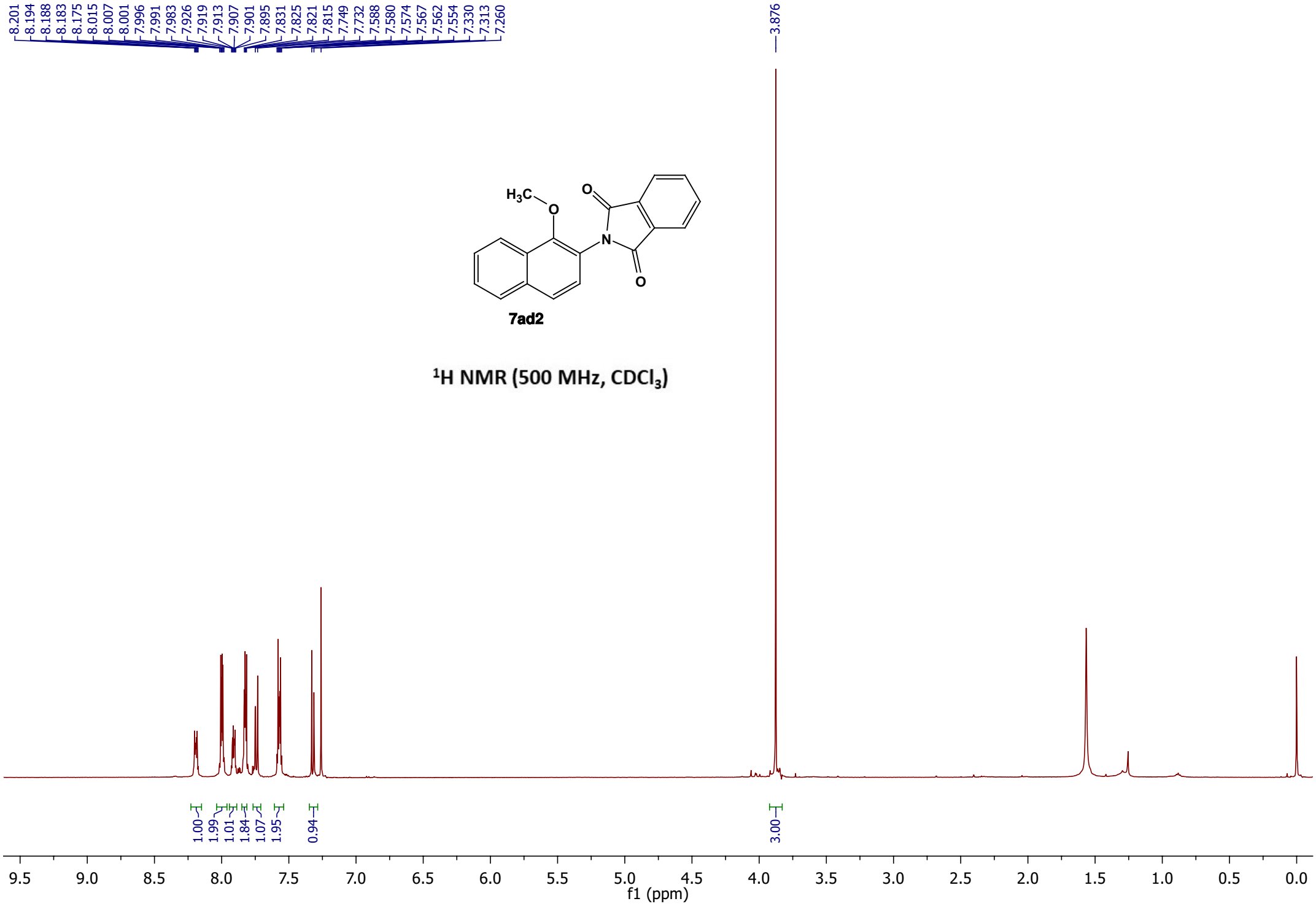
7ad1

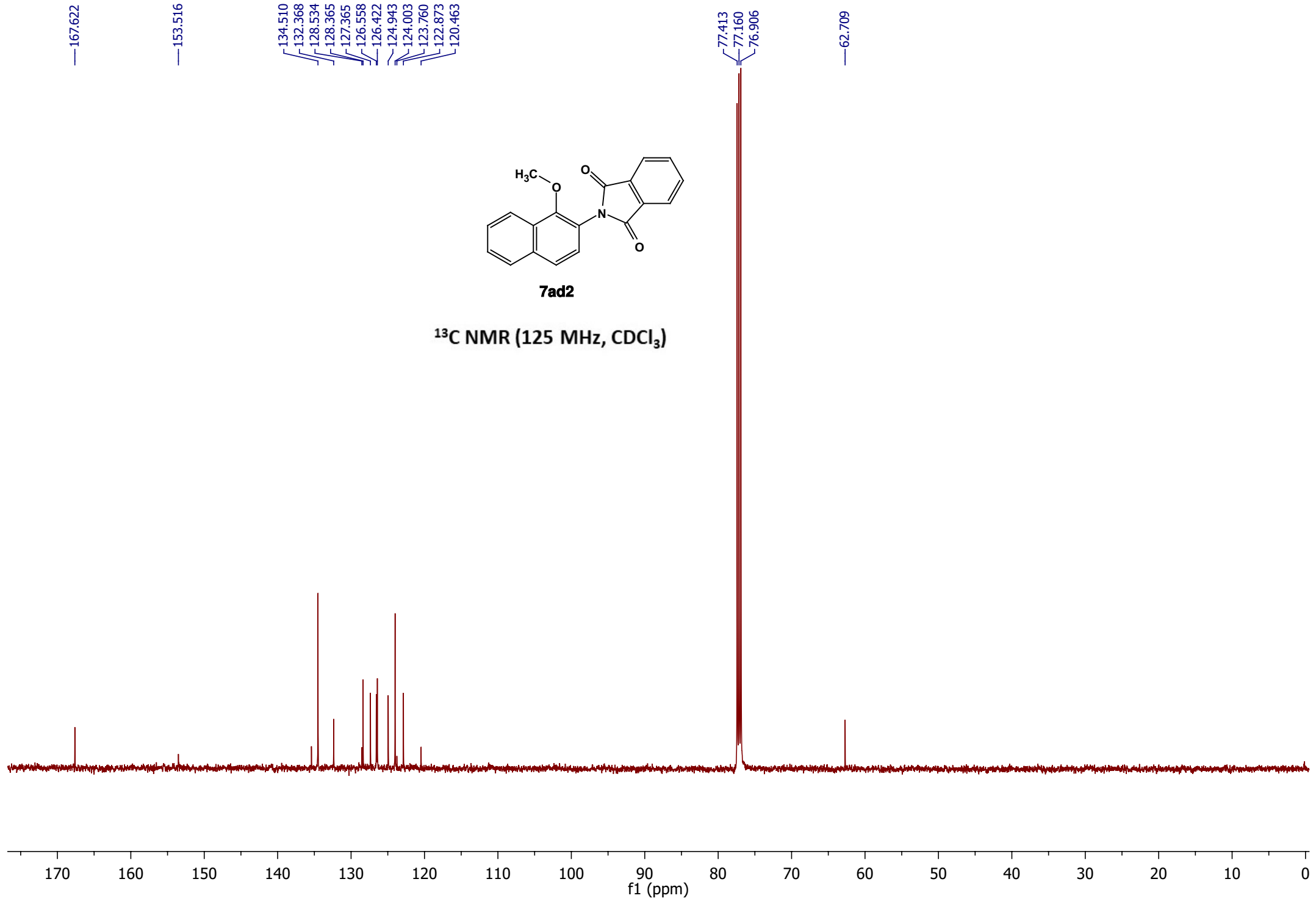
^{1}H NMR (500 MHz, $CDCl_3$)

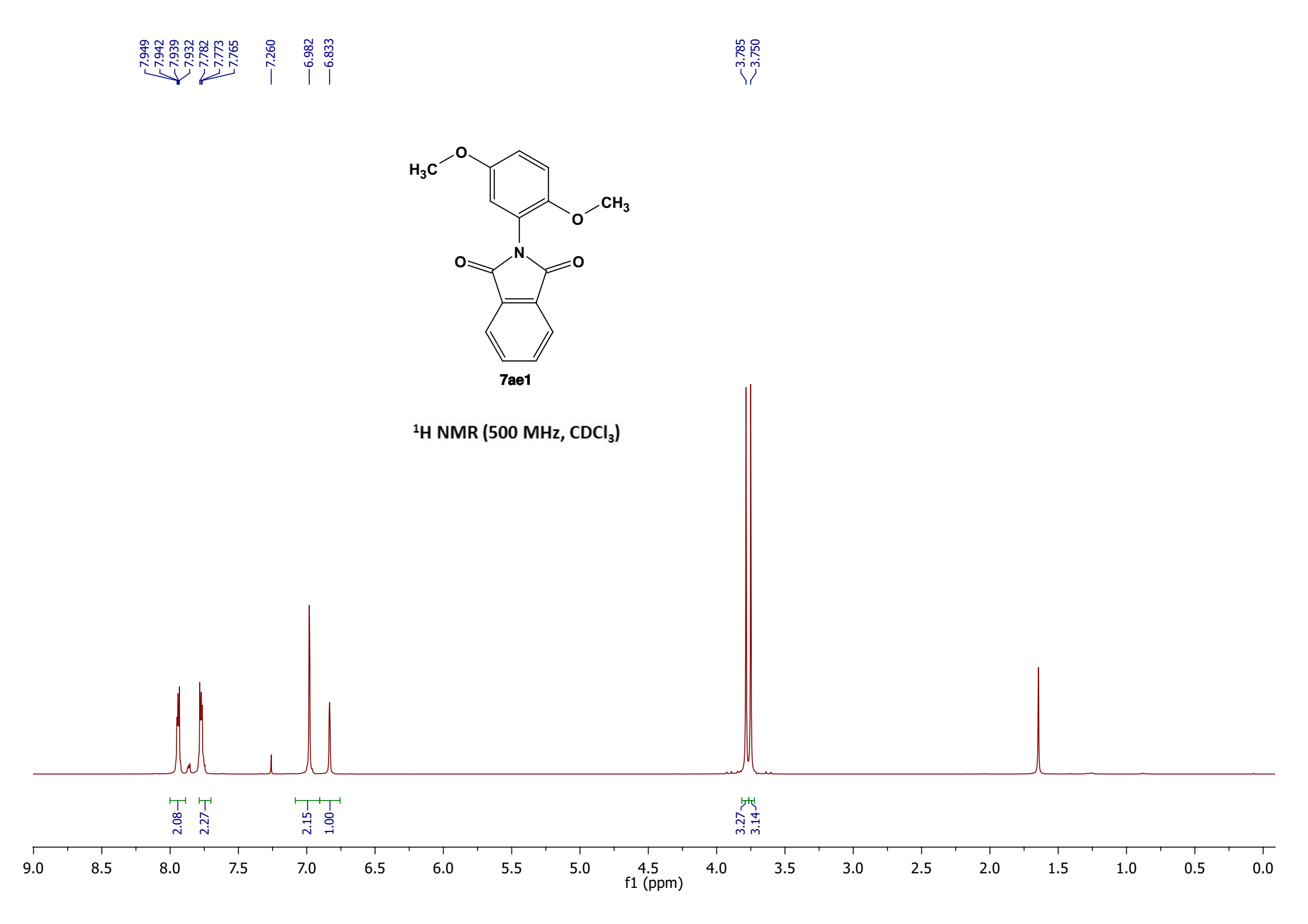


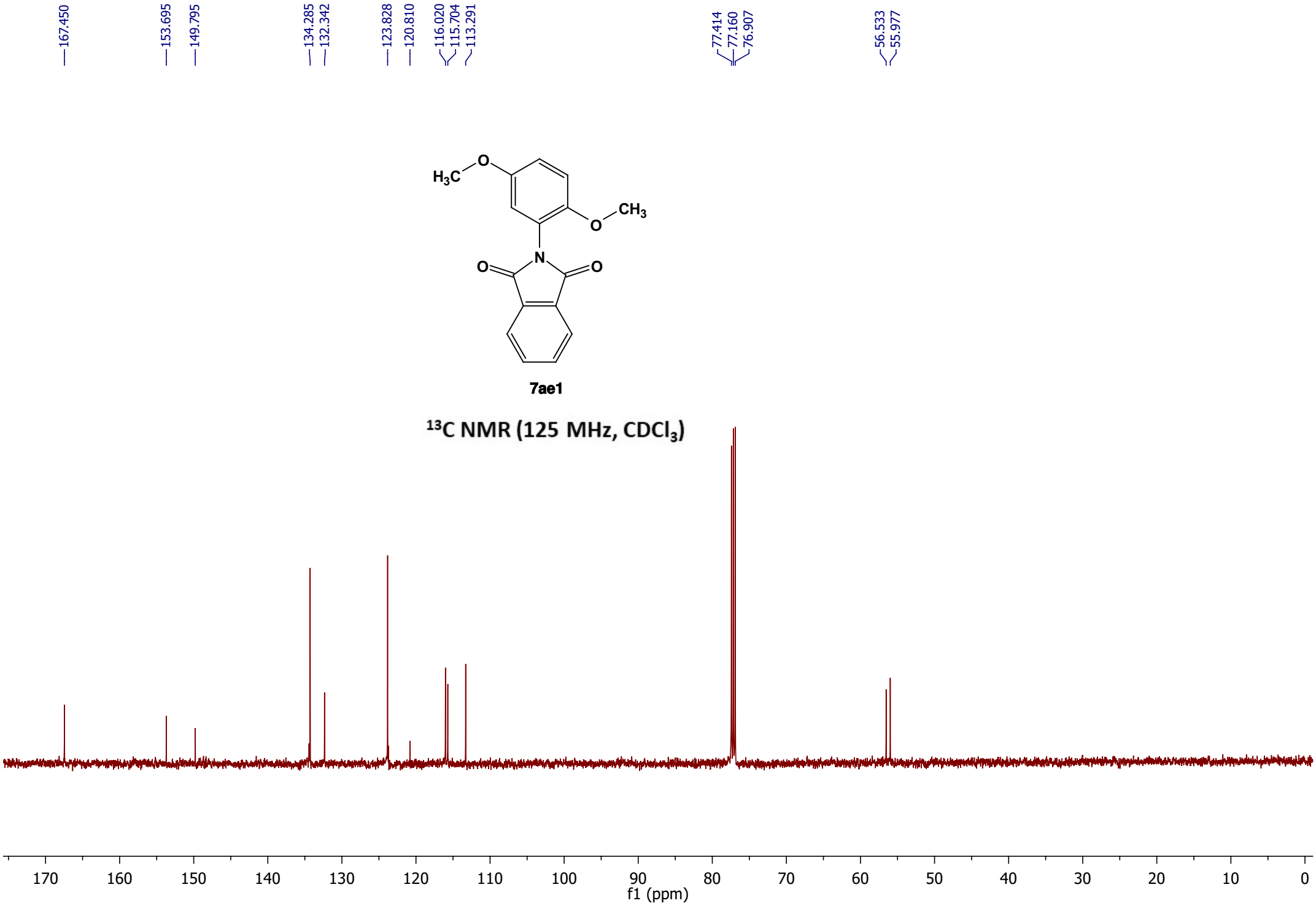

168.293
156.773
134.529
132.243
131.230
127.744
127.450
126.511
125.949
124.003
123.751
122.993
122.237
120.679
103.462
77.414
77.160
76.906
55.923
H3C
O
N
O
O
7ad2
13C NMR (125 MHz, CDCl3)
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

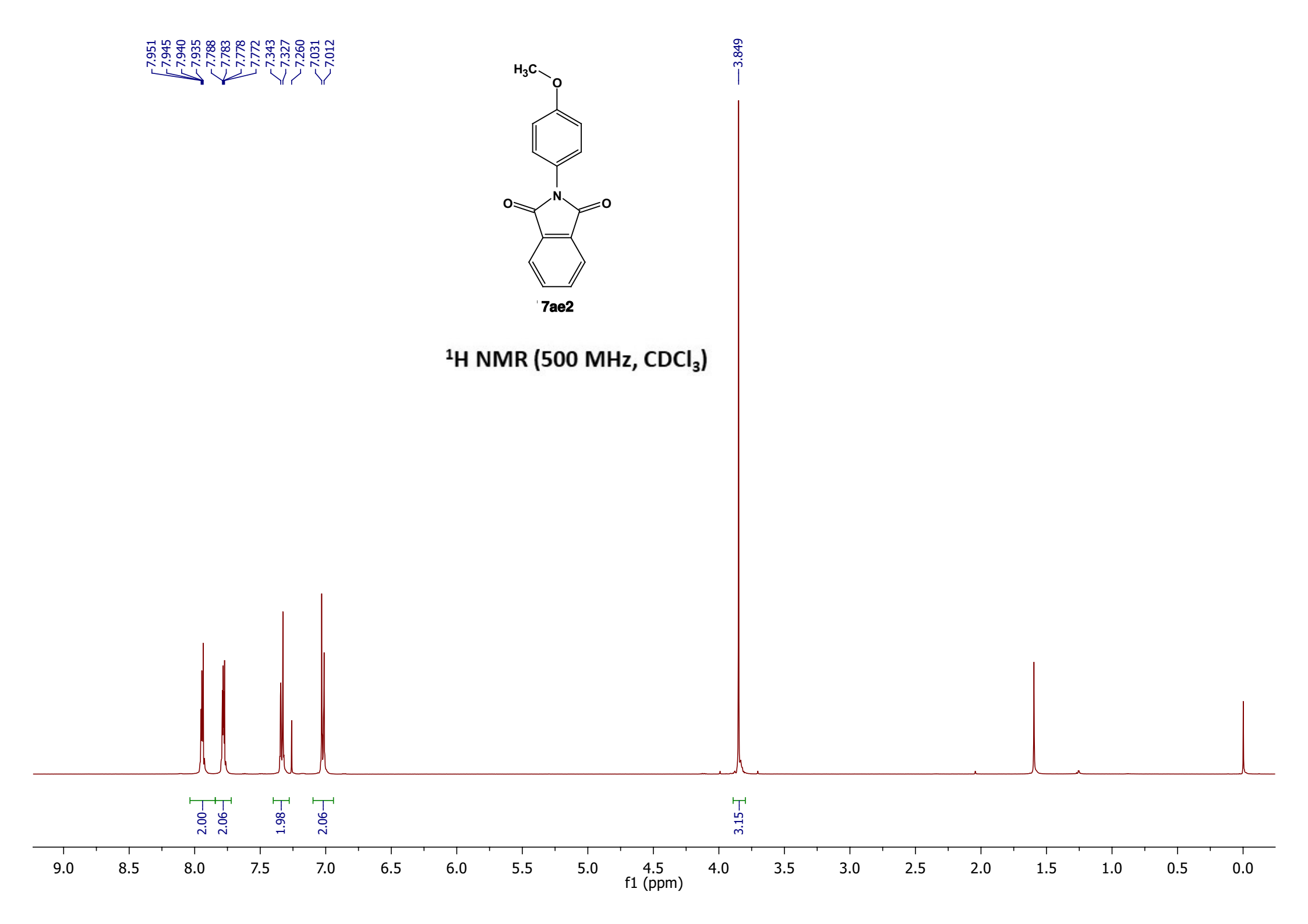

8.255
8.240
7.829
7.824
7.811
7.518
7.505
7.495
7.479
7.466
7.260
7.256
6.725
6.709
6.682
6.666
3.944
H3C
O
NH2
4-methoxynaphthalen-1-amine
1H NMR (500 MHz, CDCl3)
1.00
1.07
2.10
2.01
4.95
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

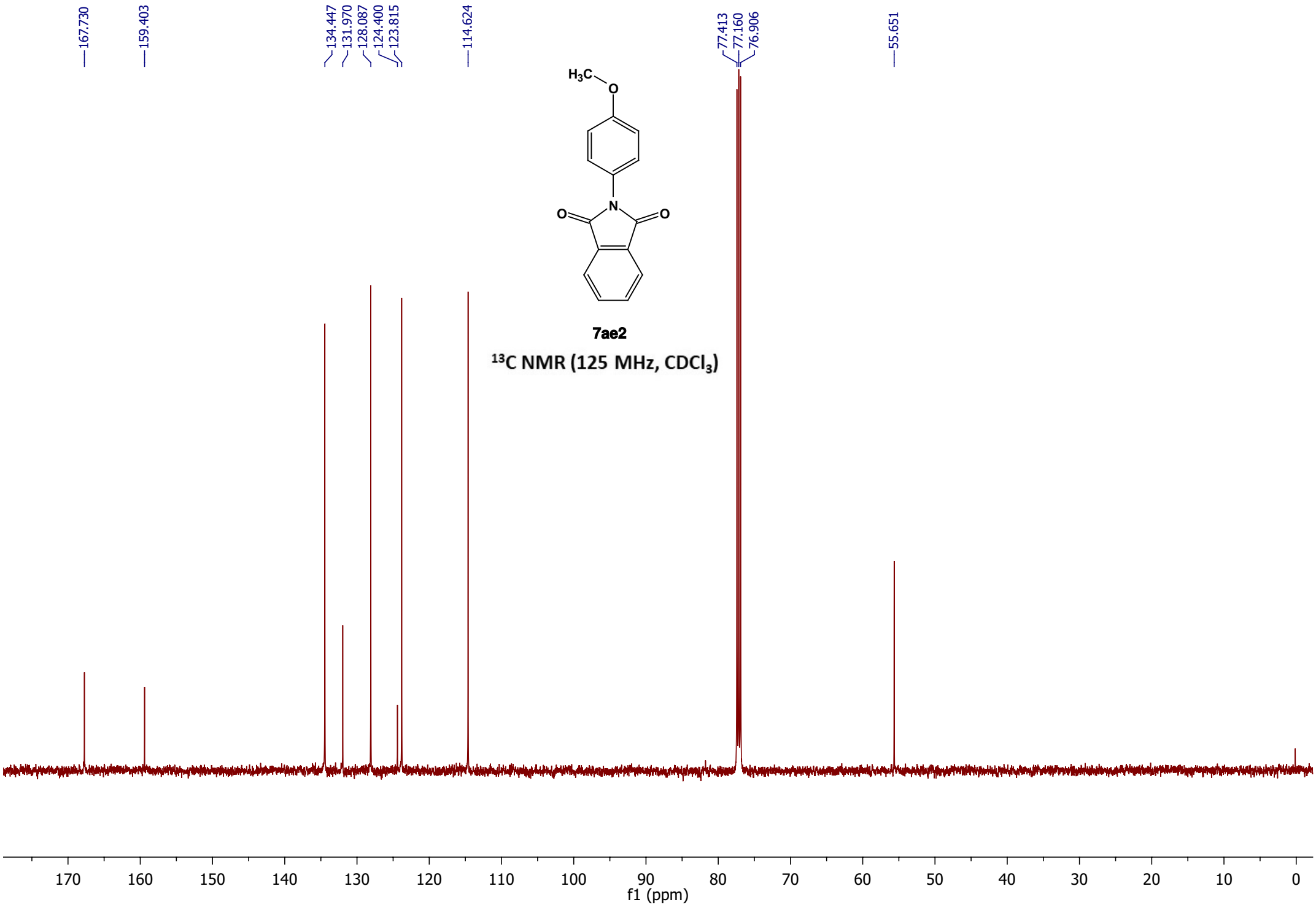

8.201
8.194
8.188
8.183
8.175
8.015
8.007
8.001
7.996
7.991
7.983
7.926
7.919
7.913
7.907
7.901
7.895
7.831
7.825
7.821
7.815
7.749
7.732
7.588
7.580
7.574
7.567
7.562
7.554
7.330
7.313
7.260
3.876
H3C
O
O
N
O
O
7ad2
1H NMR (500 MHz, CDCl3)
1.00
1.99
1.01
1.84
1.07
1.95
0.94
3.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

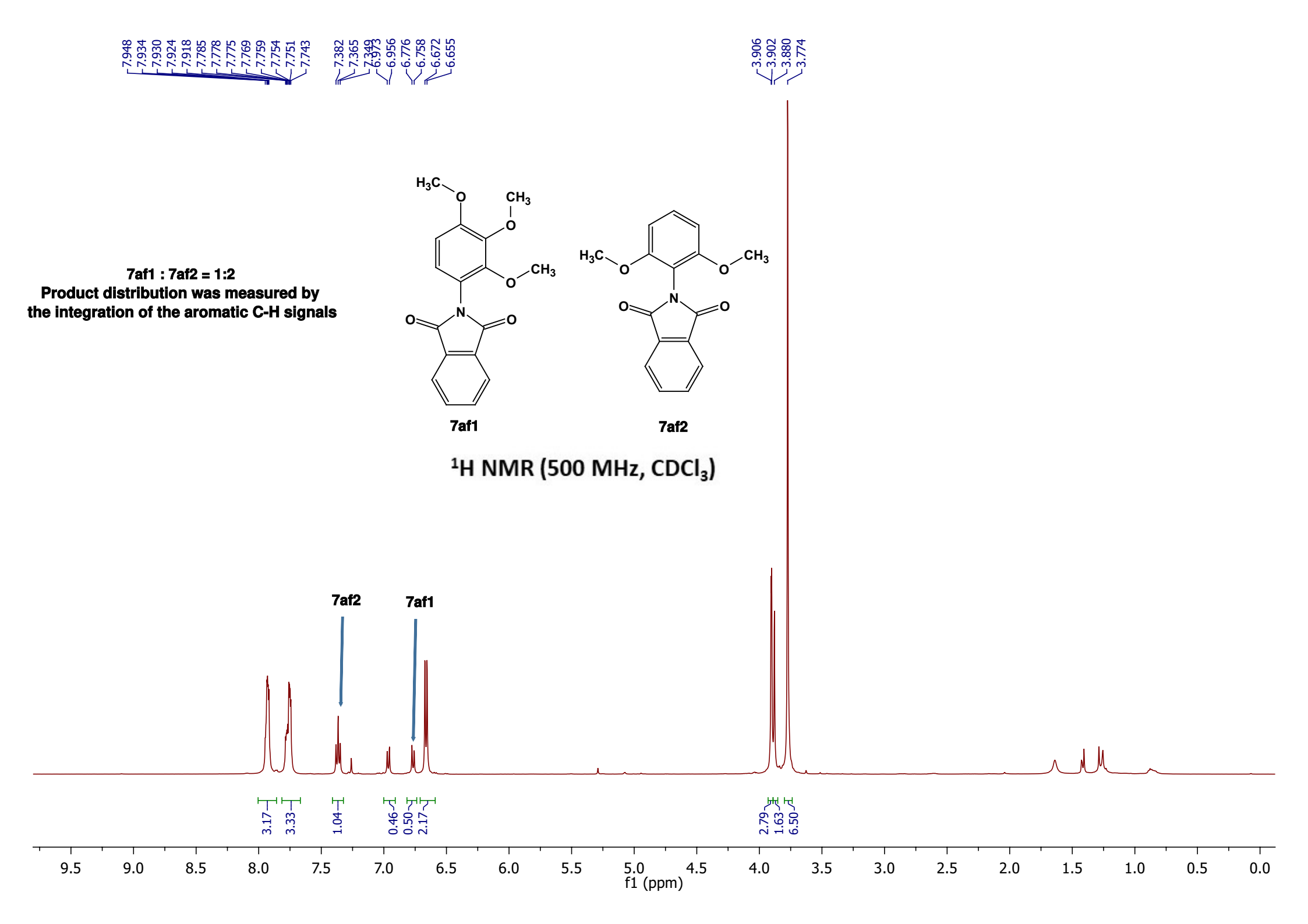

167.622
153.516
134.510
132.368
128.534
128.365
127.365
126.558
126.422
124.943
124.003
123.760
122.873
120.463
77.413
77.160
76.906
62.709
H3C
O
O
N
O
7ad2
13C NMR (125 MHz, CDCl3)
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

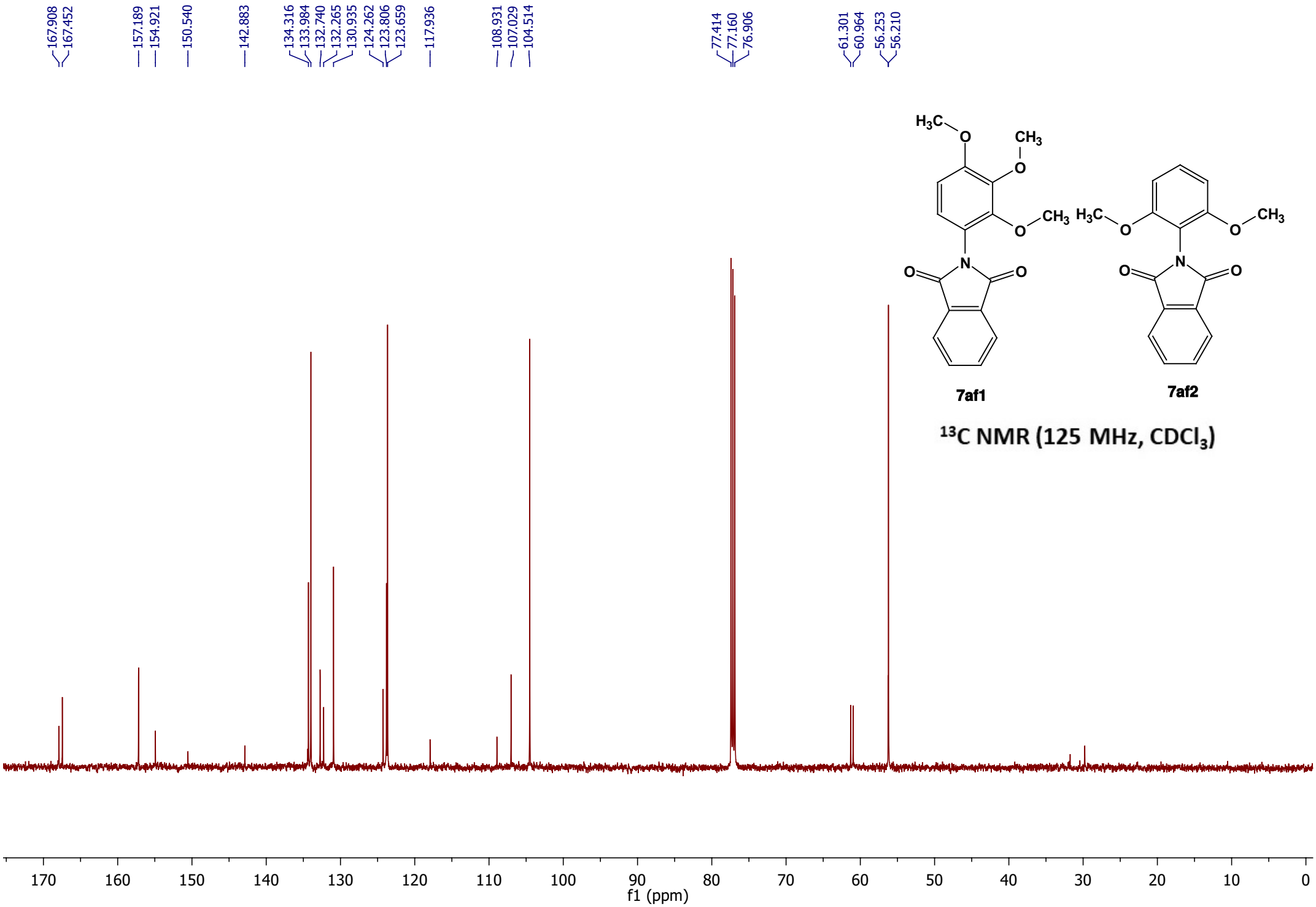

7.949
7.942
7.939
7.932
7.782
7.773
7.765
7.260
6.982
6.833
3.785
3.750
H3C
O
O
CH3
N
O
O
7ae1
1H NMR (500 MHz, CDCl3)
2.08
2.27
2.15
1.00
3.27
3.14
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

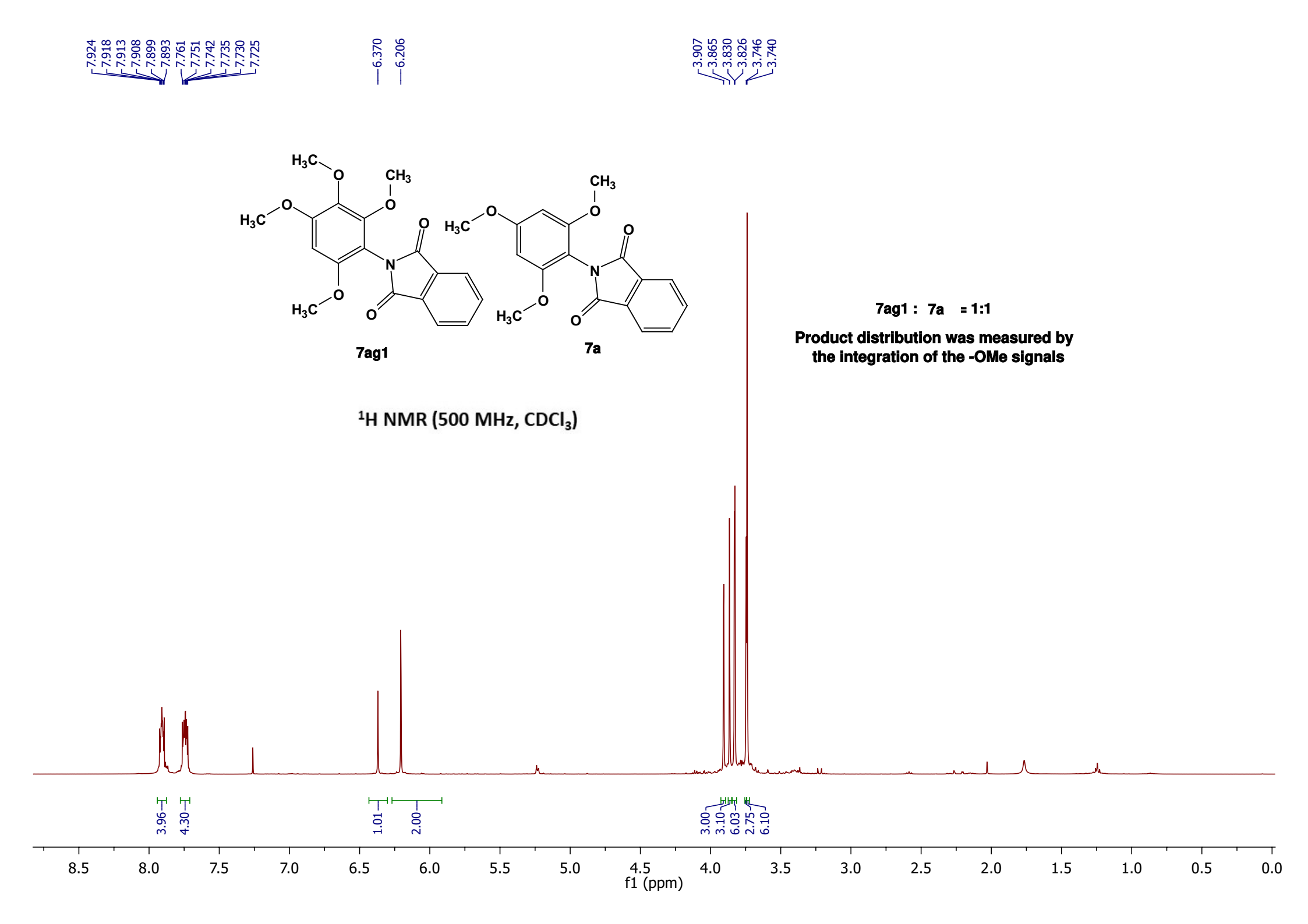

167.450
153.695
149.795
134.285
132.342
123.828
120.810
116.020
115.704
113.291
77.414
77.160
76.907
56.533
55.977
O
H3C
O
CH3
N
O
O
7ae1
13C NMR (125 MHz, CDCl3)
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

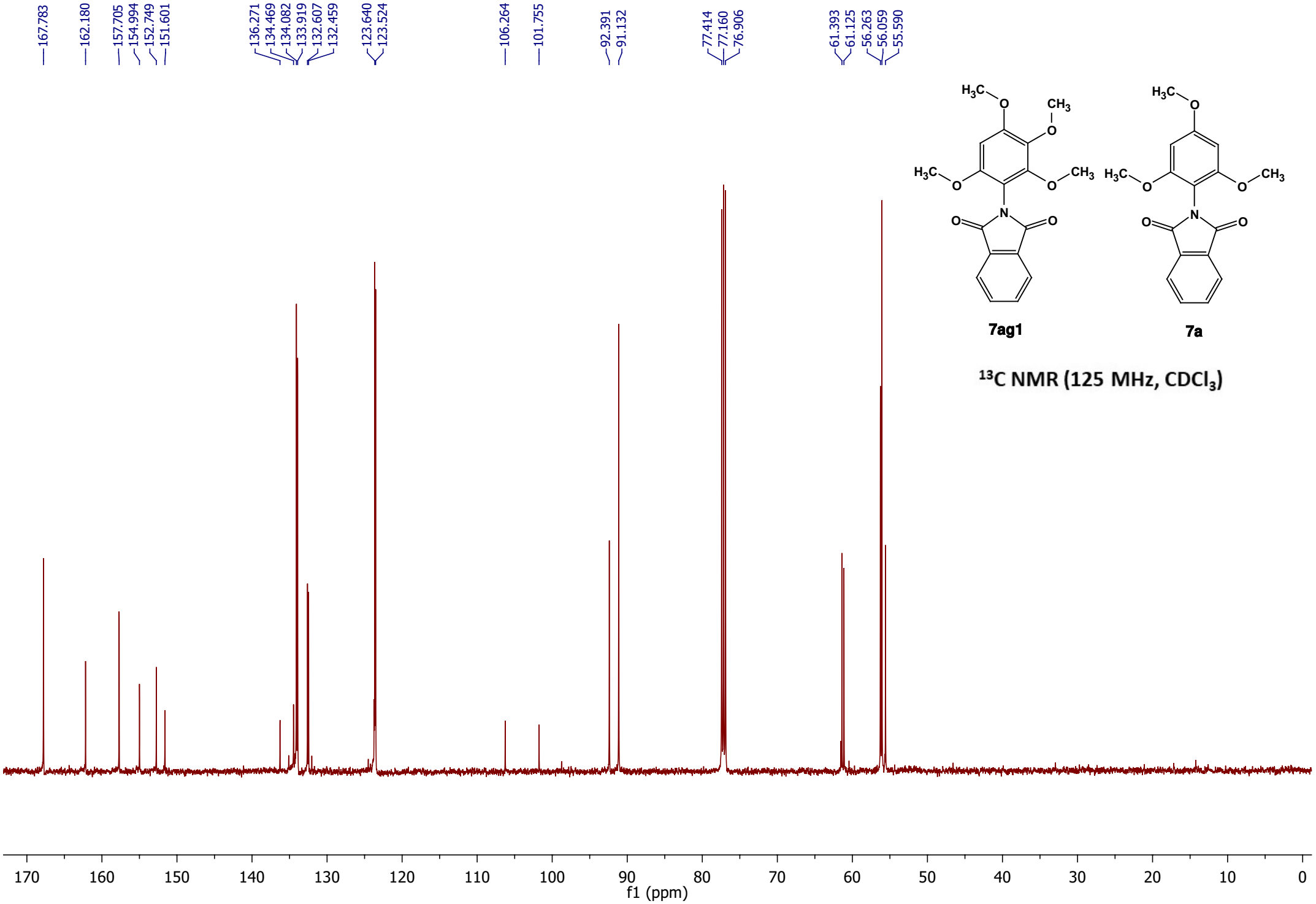

7.951
7.945
7.940
7.935
7.788
7.783
7.778
7.772
7.343
7.327
7.260
7.031
7.012
3.849
H3C
O
N
O
O
7ae2
1H NMR (500 MHz, CDCl3)
2.00
2.06
1.98
2.06
3.15
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

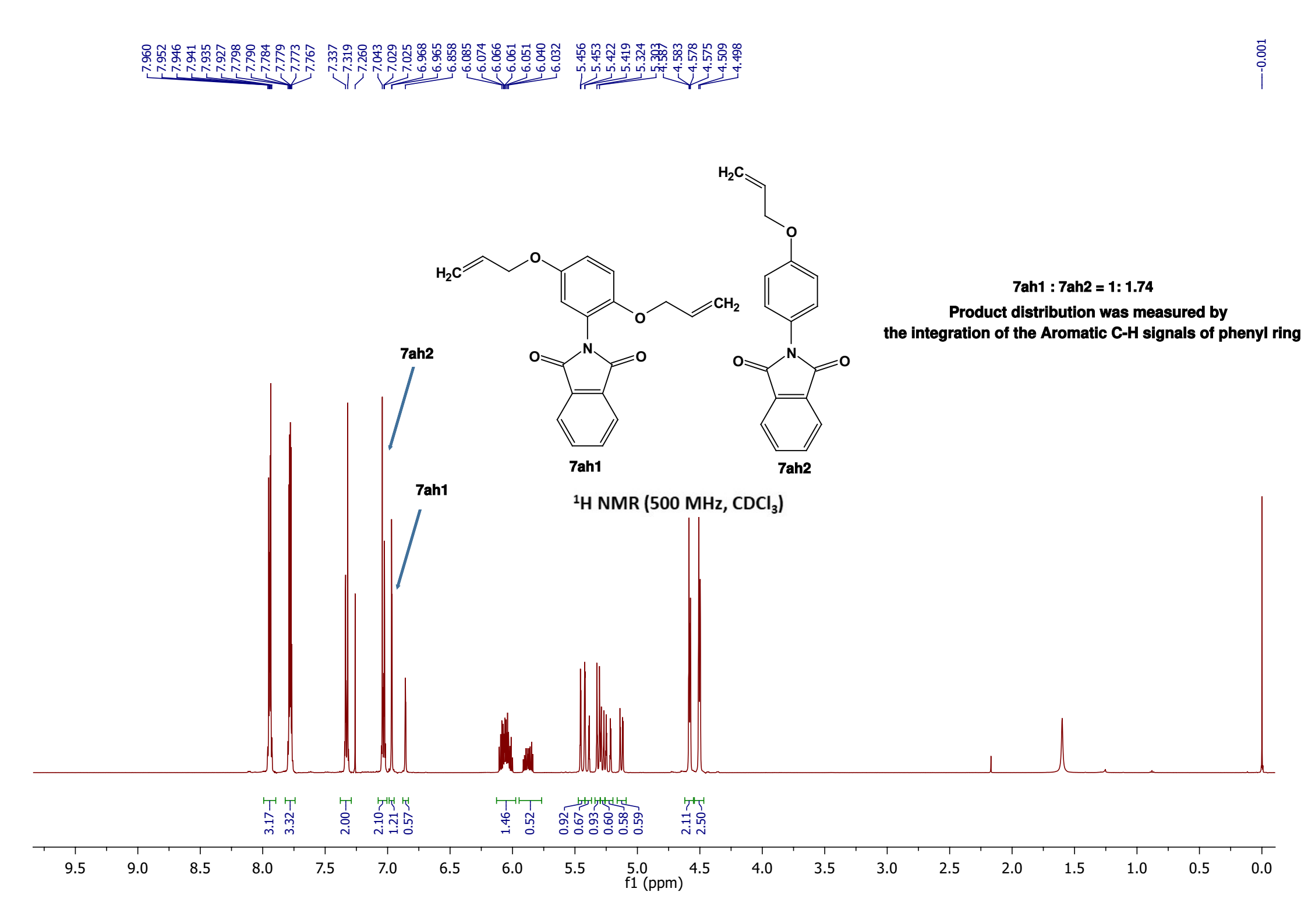

167.730
159.403
134.447
131.970
128.087
124.400
123.815
114.624
77.413
77.160
76.906
55.651
H3C
O
N
O
O
7ae2
13C NMR (125 MHz, CDCl3)
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

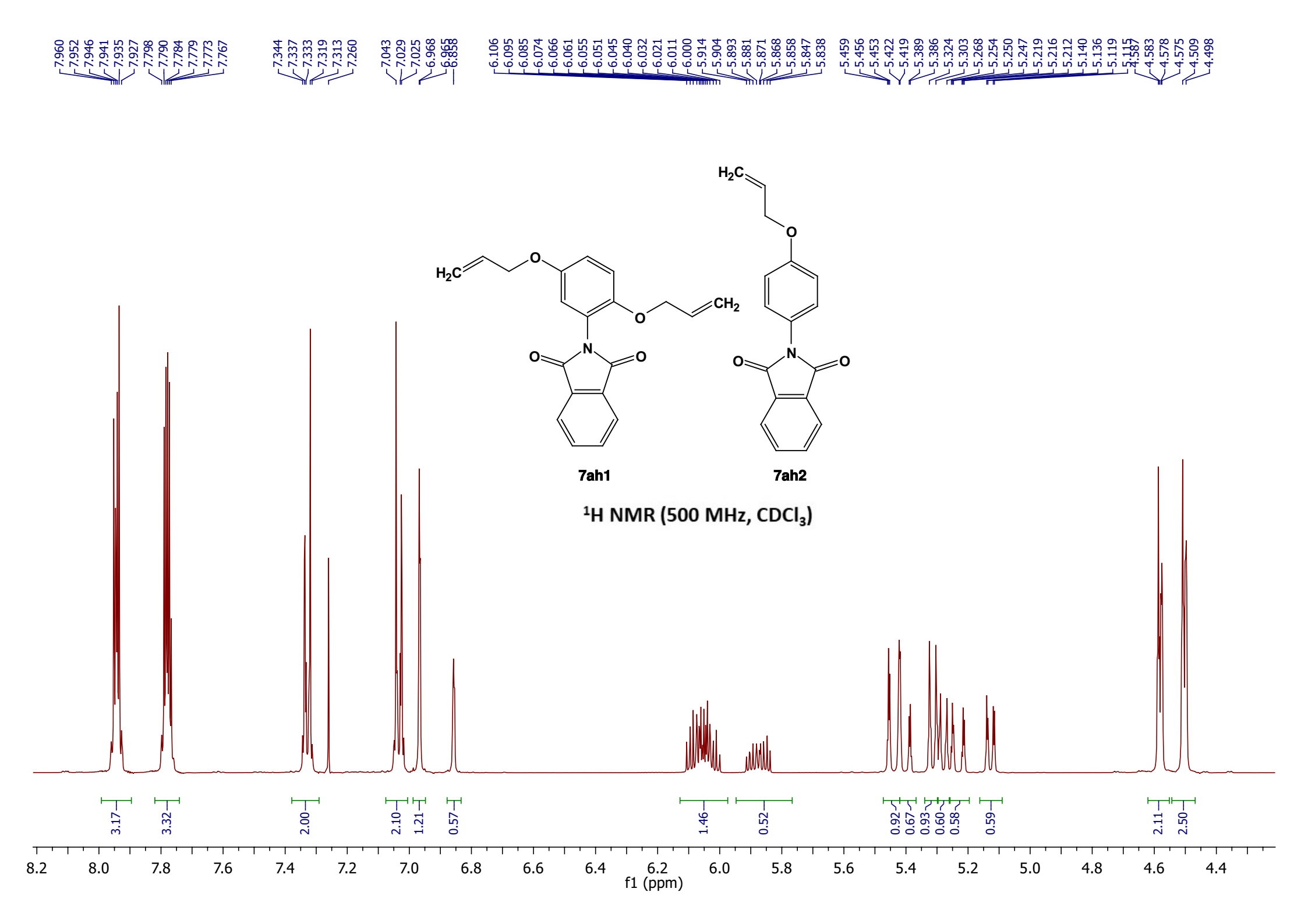

7.948
7.934
7.930
7.924
7.918
7.785
7.778
7.775
7.769
7.759
7.754
7.751
7.743
7.382
7.365
7.349
6.973
6.956
6.776
6.758
6.672
6.655
3.906
3.902
3.880
3.774
7af1 : 7af2 = 1:2
Product distribution was measured by
the integration of the aromatic C-H signals
7af1
7af2
1H NMR (500 MHz, CDCl3)
7af2
7af1
3.17
3.33
1.04
0.46
0.50
2.17
2.79
1.63
6.50
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)

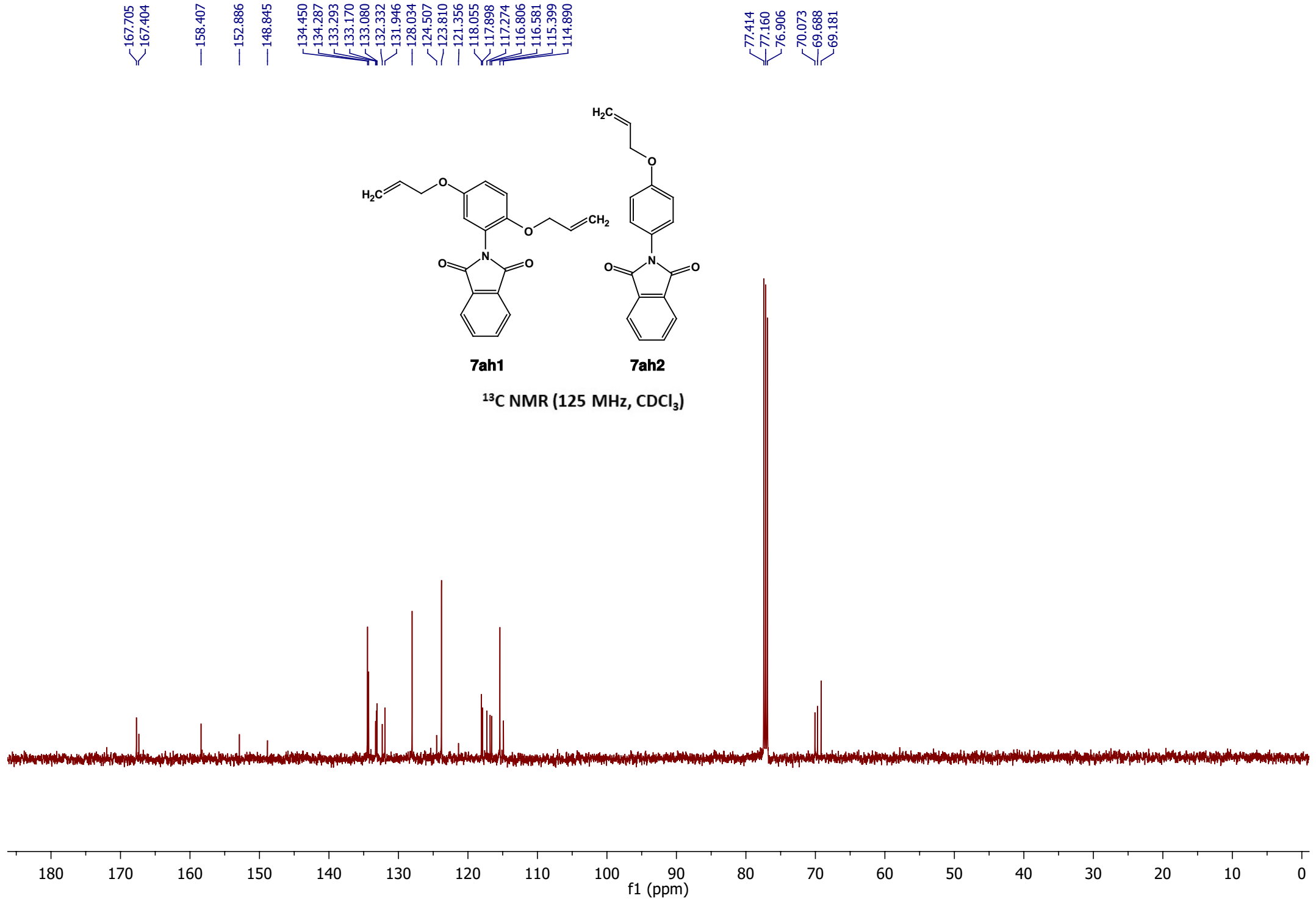

167.908
167.452
157.189
154.921
150.540
142.883
134.316
133.984
132.740
132.265
130.935
124.262
123.806
123.659
117.936
108.931
107.029
104.514
77.414
77.160
76.906
61.301
60.964
56.253
56.210
7af1
7af2
13C NMR (125 MHz, CDCl3)
f1 (ppm)

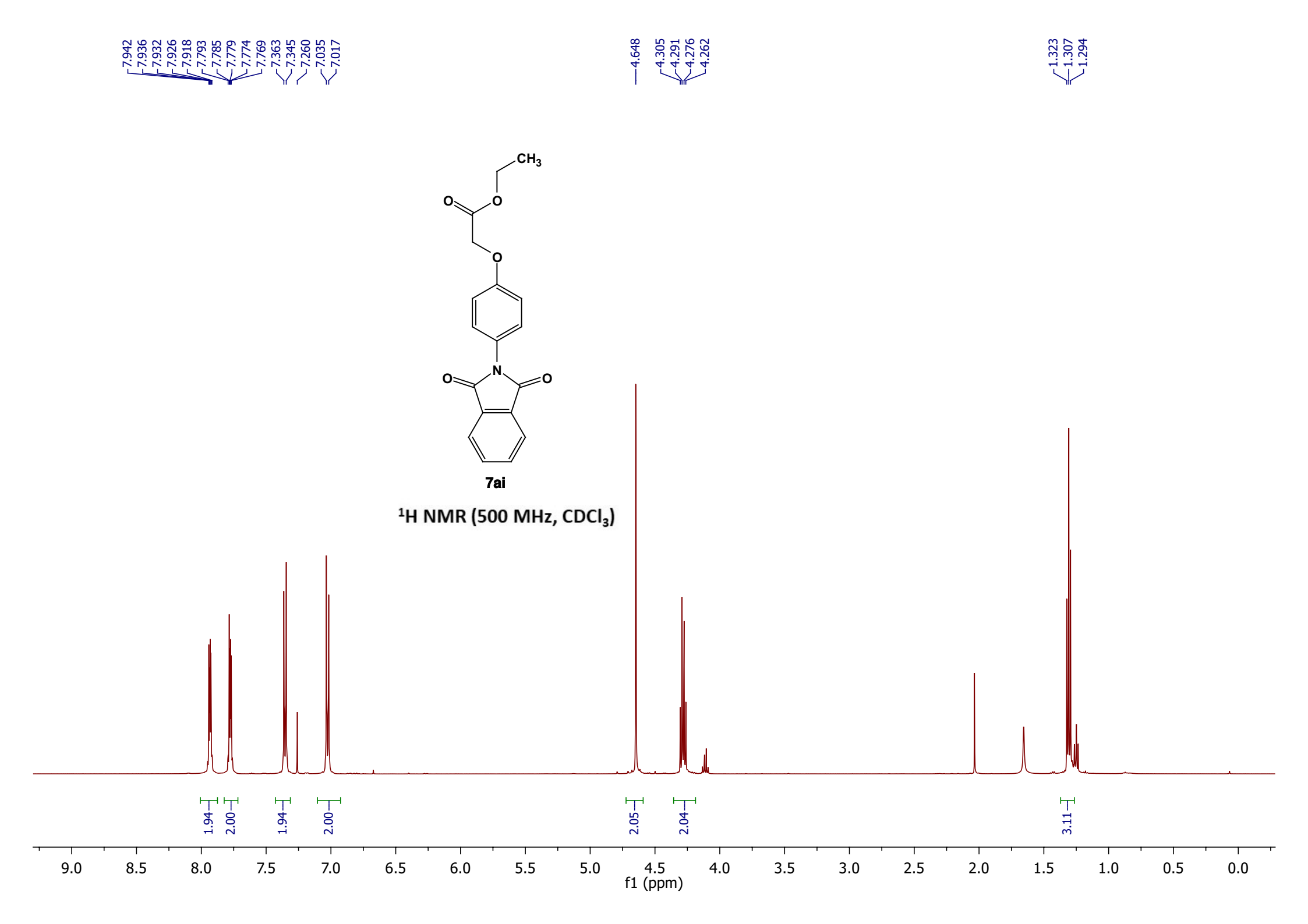

7.924
7.918
7.913
7.908
7.899
7.893
7.761
7.751
7.742
7.735
7.730
7.725
6.370
6.206
3.907
3.865
3.830
3.826
3.746
3.740
7ag1
7a
7ag1 : 7a = 1:1
Product distribution was measured by the integration of the -OMe signals
1H NMR (500 MHz, CDCl3)
3.96
4.30
1.01
2.00
3.00
3.10
6.03
2.75
6.10
f1 (ppm)

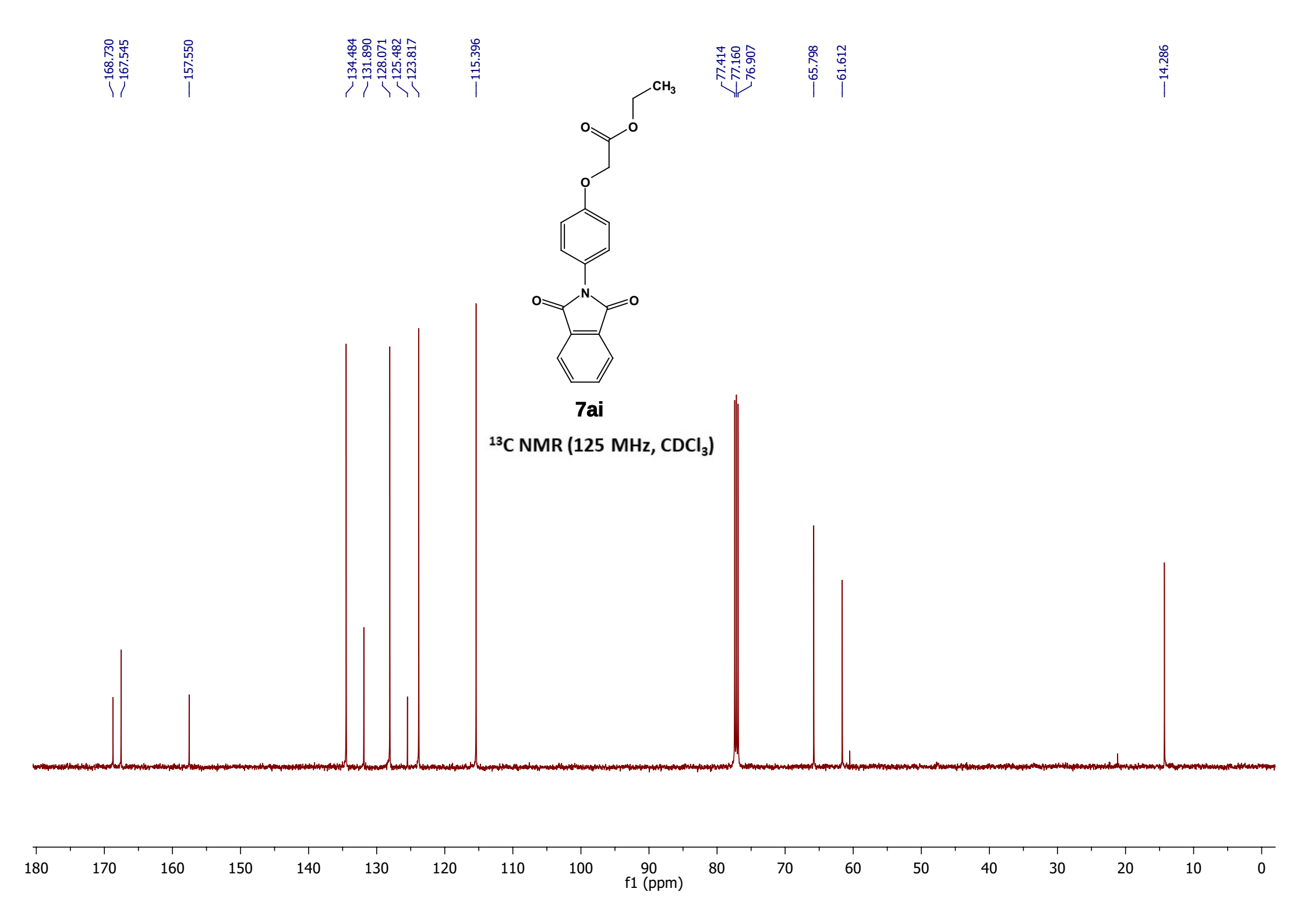

167.783
162.180
157.705
154.994
152.749
151.601
136.271
134.469
134.082
133.919
132.607
132.459
123.640
123.524
106.264
101.755
92.391
91.132
77.414
77.160
76.906
61.393
61.125
56.263
56.059
55.590
7ag1
7a
13C NMR (125 MHz, CDCl3)
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

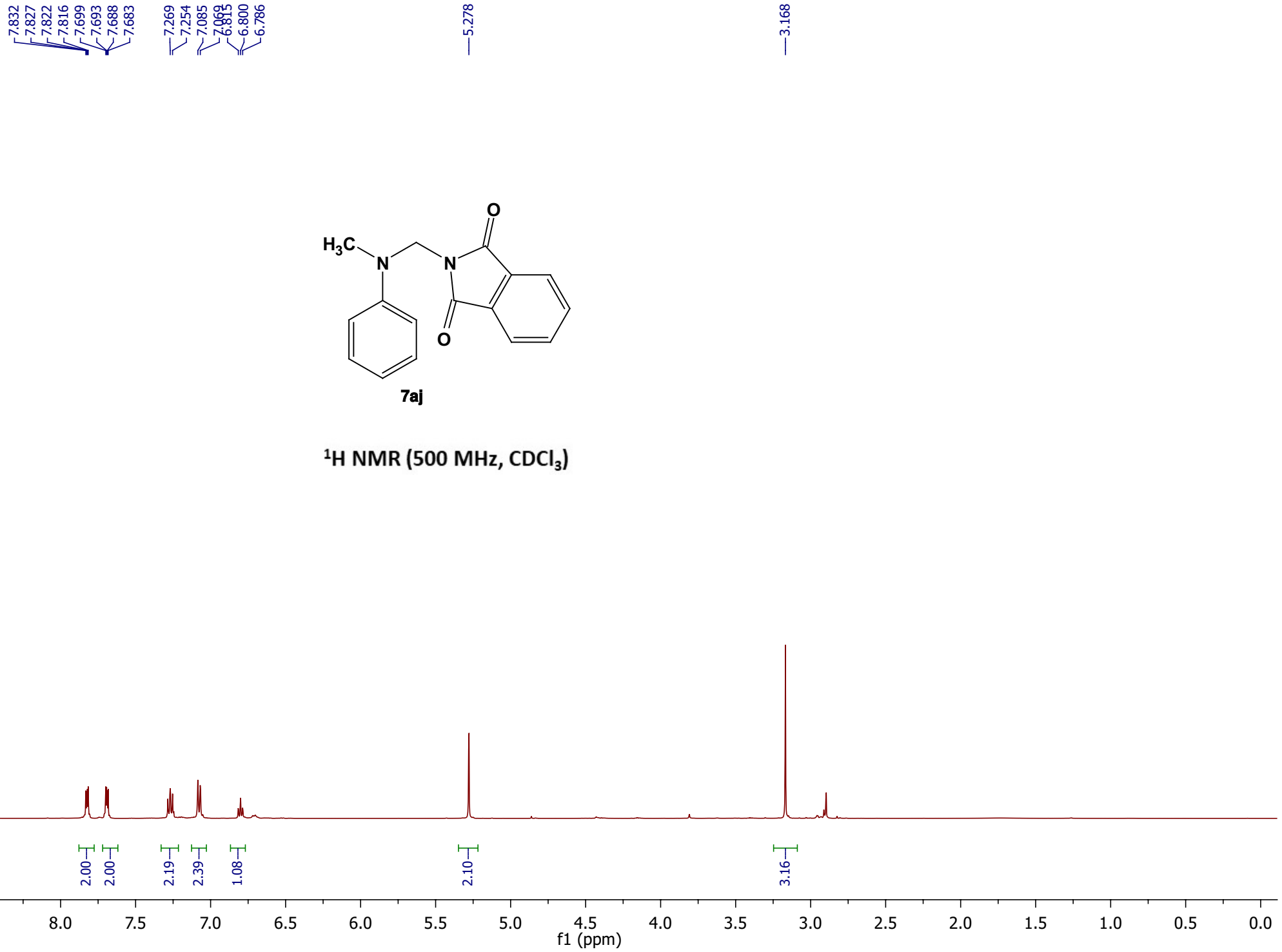

7.960
7.952
7.946
7.941
7.935
7.927
7.798
7.790
7.784
7.779
7.773
7.767
7.337
7.319
7.260
7.043
7.029
7.025
6.968
6.965
6.858
6.085
6.074
6.066
6.061
6.051
6.040
6.032
5.456
5.453
5.422
5.419
5.324
4.583
4.578
4.575
4.509
4.498
-0.001
7ah2
7ah1
7ah1
7ah2
7ah1 : 7ah2 = 1: 1.74
Product distribution was measured by
the integration of the Aromatic C-H signals of phenyl ring
1H NMR (500 MHz, CDCl3)
3.17
3.32
2.00
2.10
1.21
0.57
1.46
0.52
0.92
0.67
0.93
0.60
0.58
0.59
2.11
2.50
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)


7.960
7.952
7.946
7.941
7.935
7.927
7.798
7.790
7.784
7.779
7.773
7.767
7.344
7.337
7.333
7.319
7.313
7.260
7.043
7.029
7.025
6.968
6.965
6.858
6.106
6.095
6.085
6.074
6.066
6.061
6.055
6.051
6.045
6.040
6.032
6.021
6.011
6.000
5.914
5.904
5.893
5.881
5.871
5.868
5.858
5.847
5.838
5.459
5.456
5.453
5.422
5.419
5.389
5.386
5.324
5.303
5.268
5.254
5.250
5.247
5.219
5.216
5.212
5.140
5.136
5.119
5.115
4.587
4.583
4.578
4.575
4.509
4.498
H2C
O
CH2
N
O
O
7ah1
7ah2
1H NMR (500 MHz, CDCl3)
3.17
3.32
2.00
2.10
1.21
0.57
1.46
0.52
0.92
0.67
0.93
0.60
0.58
0.59
2.11
2.50
8.2
8.0
7.8
7.6
7.4
7.2
7.0
6.8
6.6
6.4
6.2
6.0
5.8
5.6
5.4
5.2
5.0
4.8
4.6
4.4
f1 (ppm)


167.705
167.404
158.407
152.886
148.845
134.450
134.287
133.293
133.170
133.080
132.332
131.946
128.034
124.507
123.810
121.356
118.055
117.898
117.274
116.806
116.581
115.399
114.890
77.414
77.160
76.906
70.073
69.688
69.181
H2C
O
CH2
N
7ah1
7ah2
13C NMR (125 MHz, CDCl3)
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

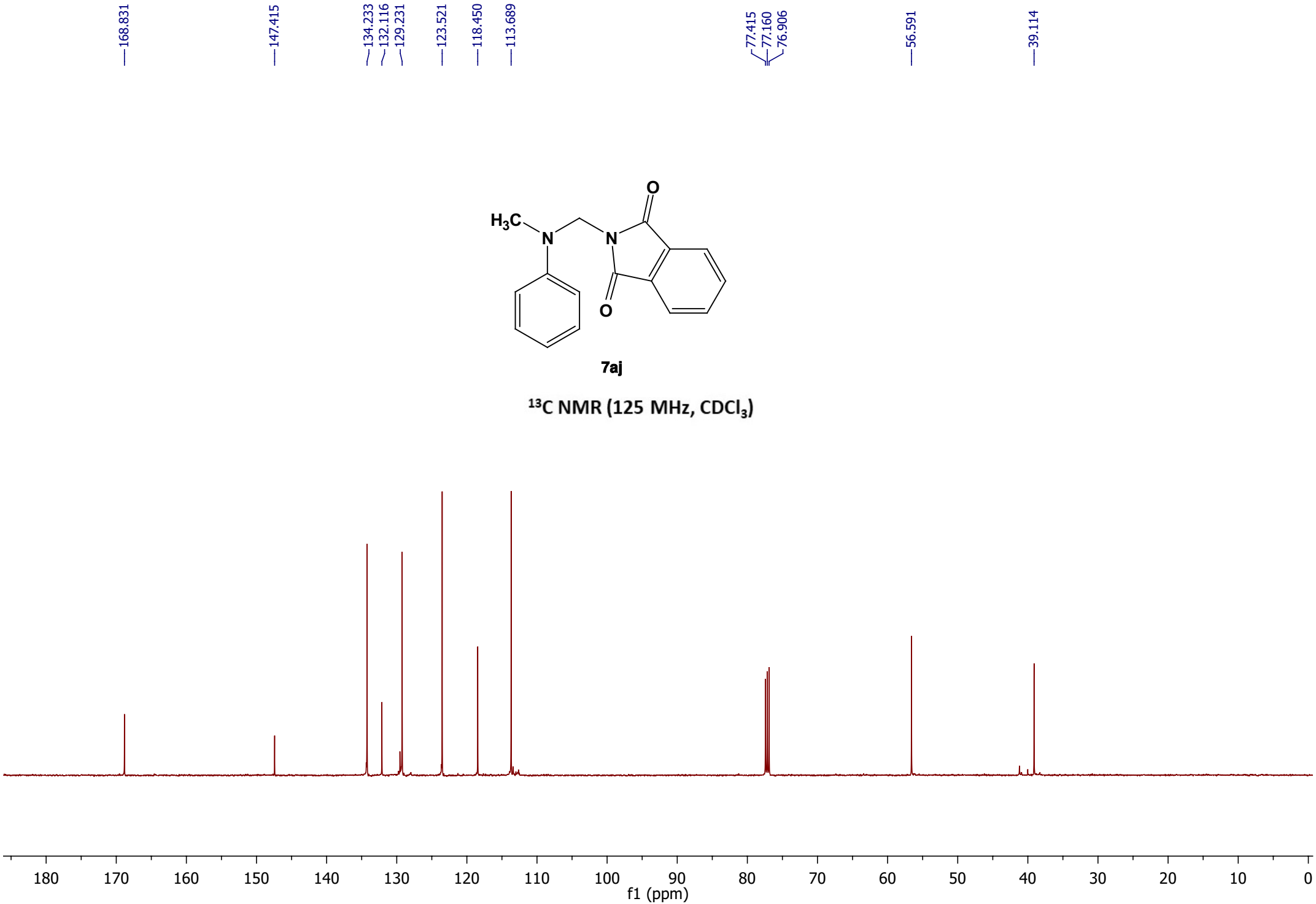

7.942
7.936
7.932
7.926
7.918
7.793
7.785
7.779
7.774
7.769
7.363
7.345
7.260
7.035
7.017
4.648
4.305
4.291
4.276
4.262
1.323
1.307
1.294
CH3
O
O
O
N
O
O
7ai
1H NMR (500 MHz, CDCl3)
1.94
2.00
1.94
2.00
2.05
2.04
3.11
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

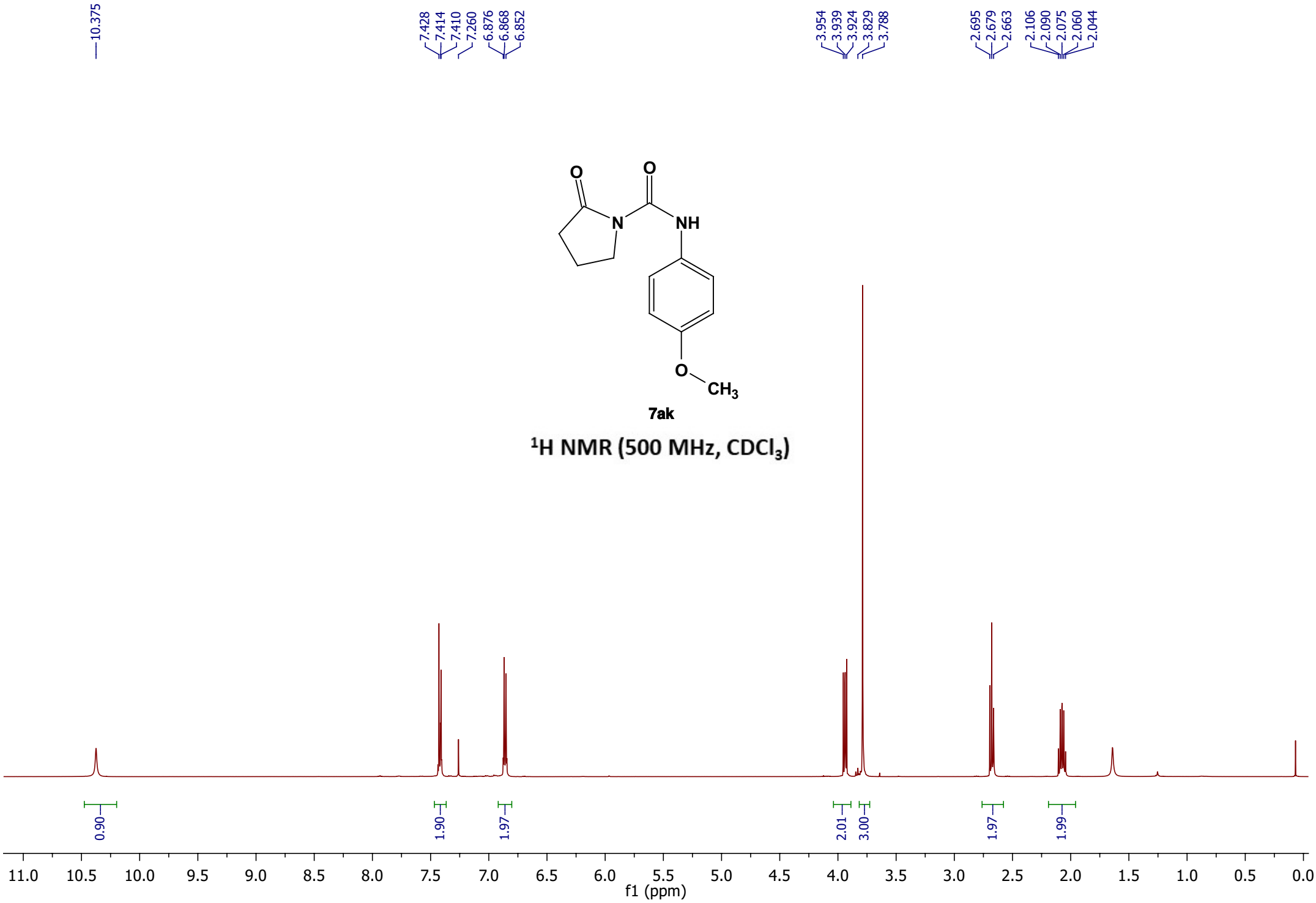

168.730
167.545
157.550
134.484
131.890
128.071
125.482
123.817
115.396
77.414
77.160
76.907
65.798
61.612
14.286
CH3
O
O
O
N
O
O
7ai
13C NMR (125 MHz, CDCl3)
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

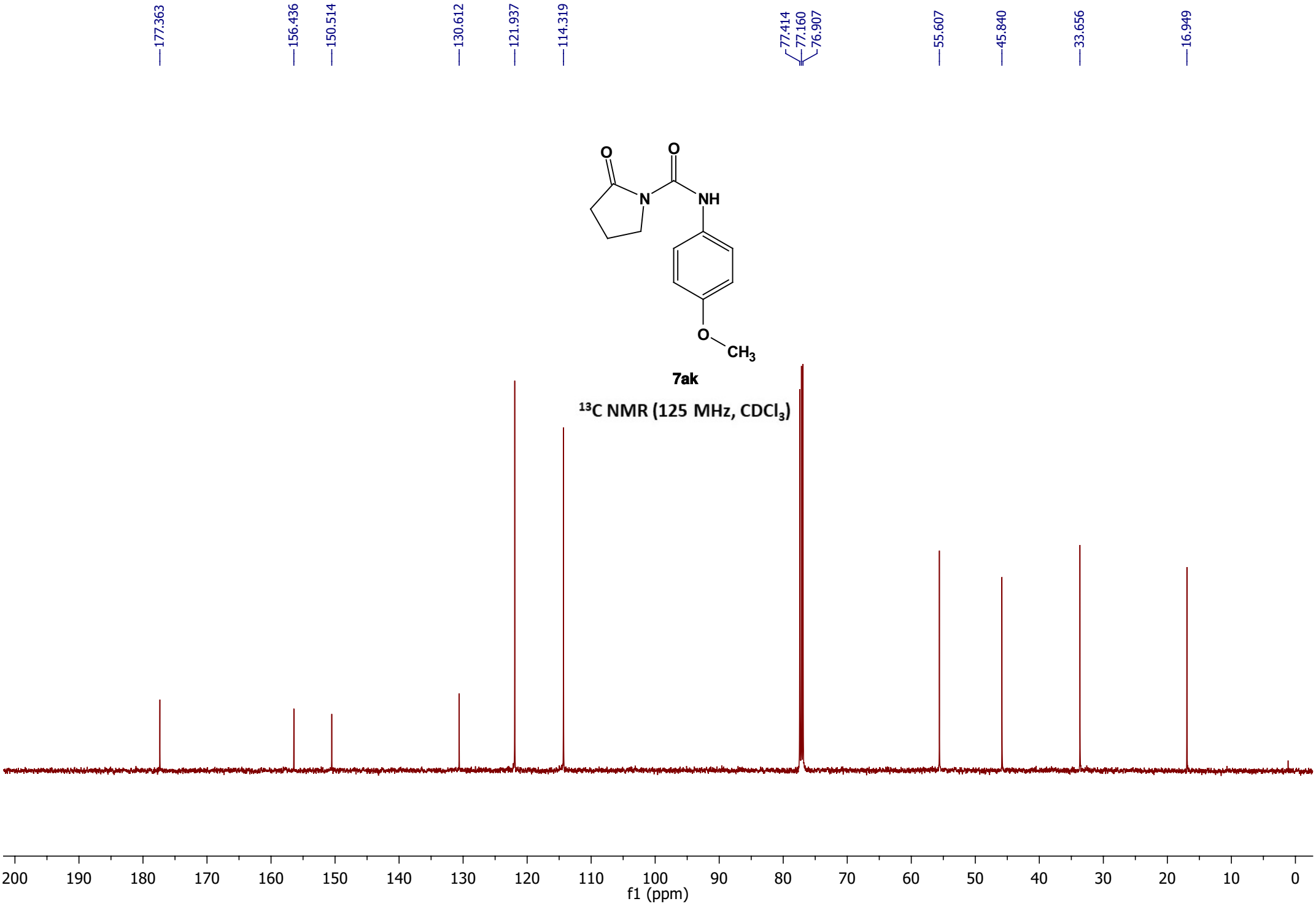


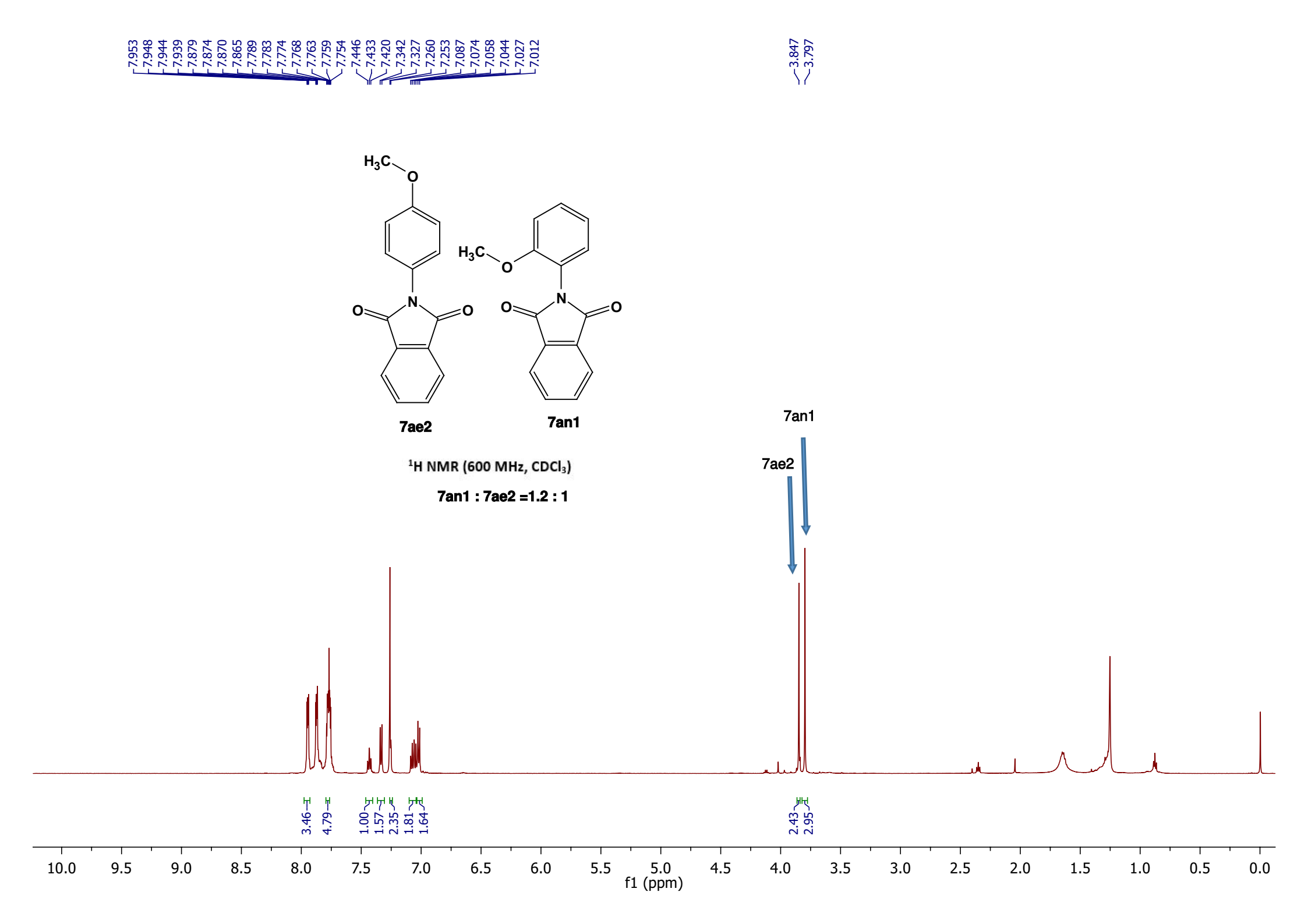
7aj

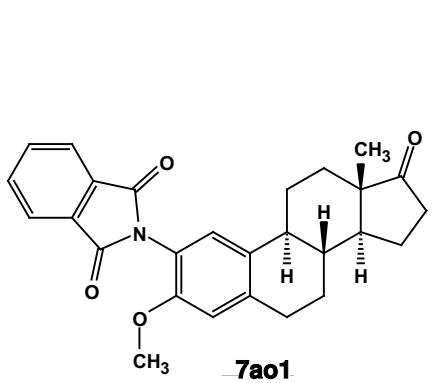
^{1}H NMR (500 MHz, $CDCl_3$)

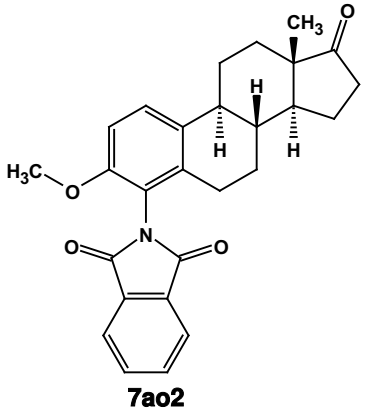

168.831
147.415
134.233
132.116
129.231
123.521
118.450
113.689
77.415
77.160
76.906
56.591
39.114
H3C
N
N
O
O
7aj
13C NMR (125 MHz, CDCl3)
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


10.375
7.428
7.414
7.410
7.260
6.876
6.868
6.852
3.954
3.939
3.924
3.829
3.788
2.695
2.679
2.663
2.106
2.090
2.075
2.060
2.044
O
O
N
NH
O
CH3
7ak
1H NMR (500 MHz, CDCl3)
0.90
1.90
1.97
2.01
3.00
1.97
1.99
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)


177.363
156.436
150.514
130.612
121.937
114.319
77.414
77.160
76.907
55.607
45.840
33.656
16.949
O
O
N
NH
O
CH3
7ak
13C NMR (125 MHz, CDCl3)
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


7.953
7.948
7.944
7.939
7.879
7.874
7.870
7.865
7.789
7.783
7.774
7.768
7.763
7.759
7.754
7.446
7.433
7.420
7.342
7.327
7.260
7.253
7.087
7.074
7.058
7.044
7.027
7.012
3.847
3.797
H3C
O
N
O
O
7ae2
7an1
1H NMR (600 MHz, CDCl3)
7an1 : 7ae2 =1.2 : 1
7an1
7ae2
3.46
4.79
1.00
1.57
2.35
1.81
1.64
2.43
2.95
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)

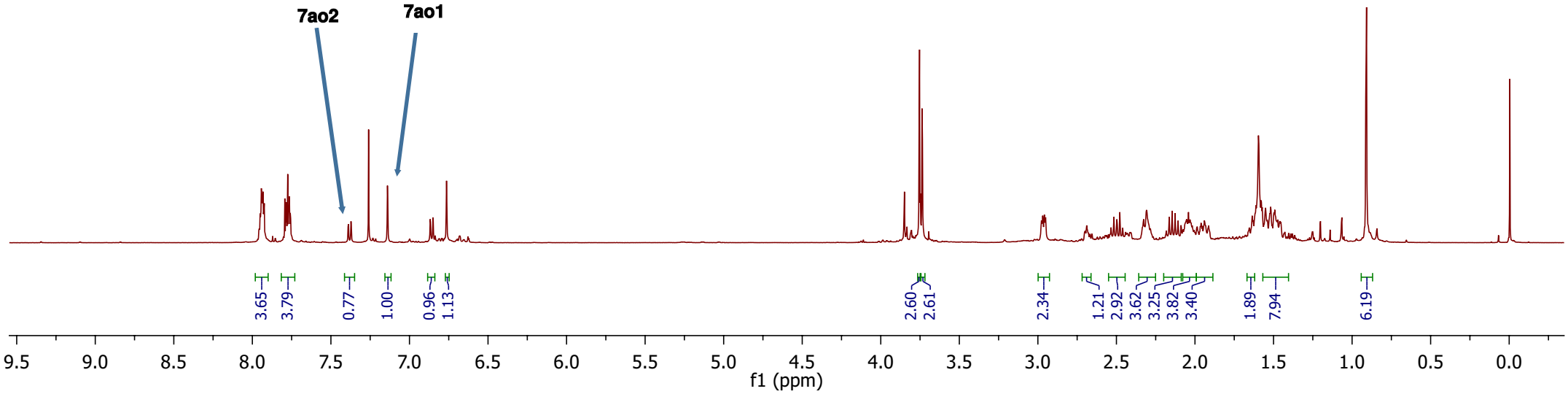


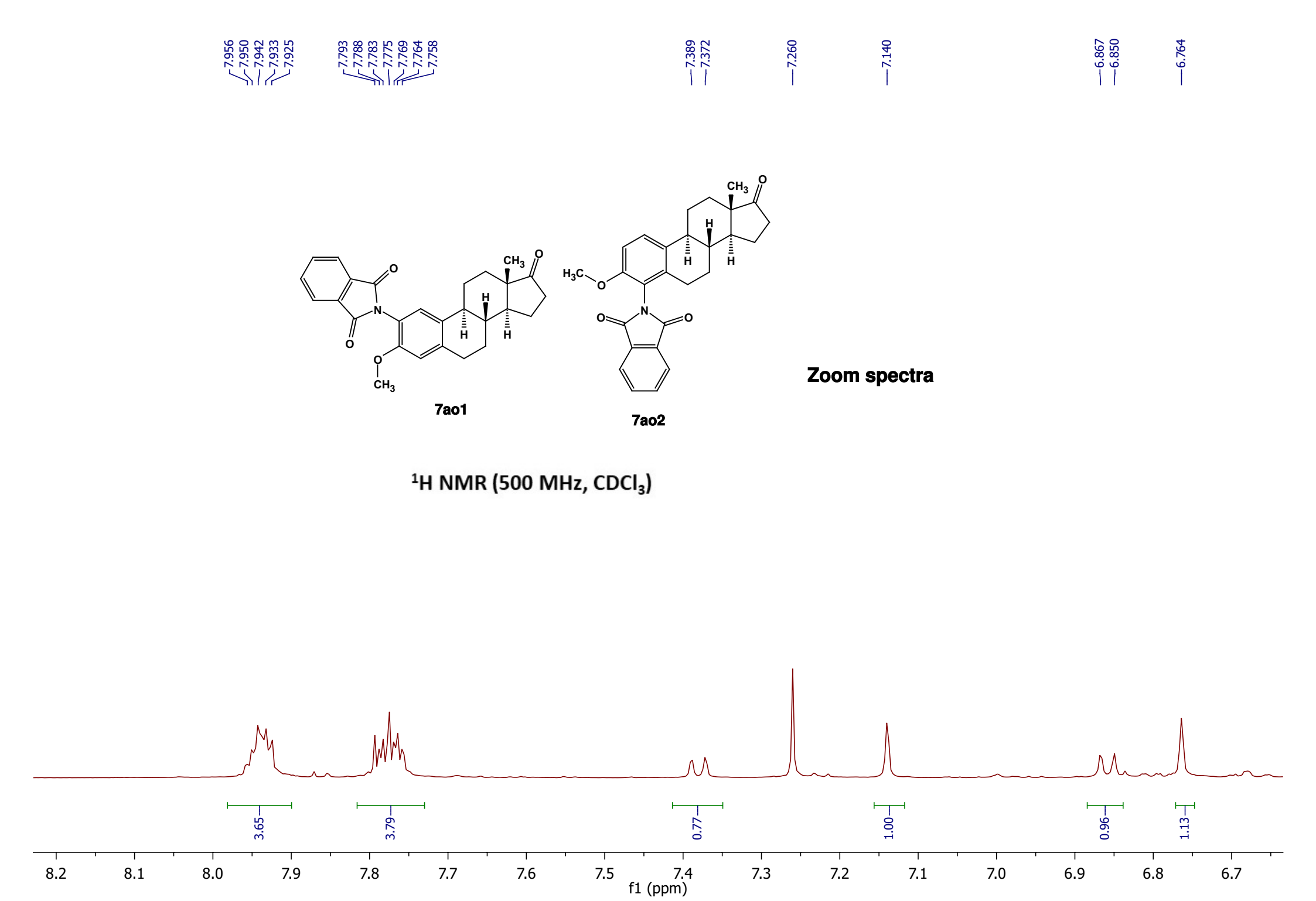


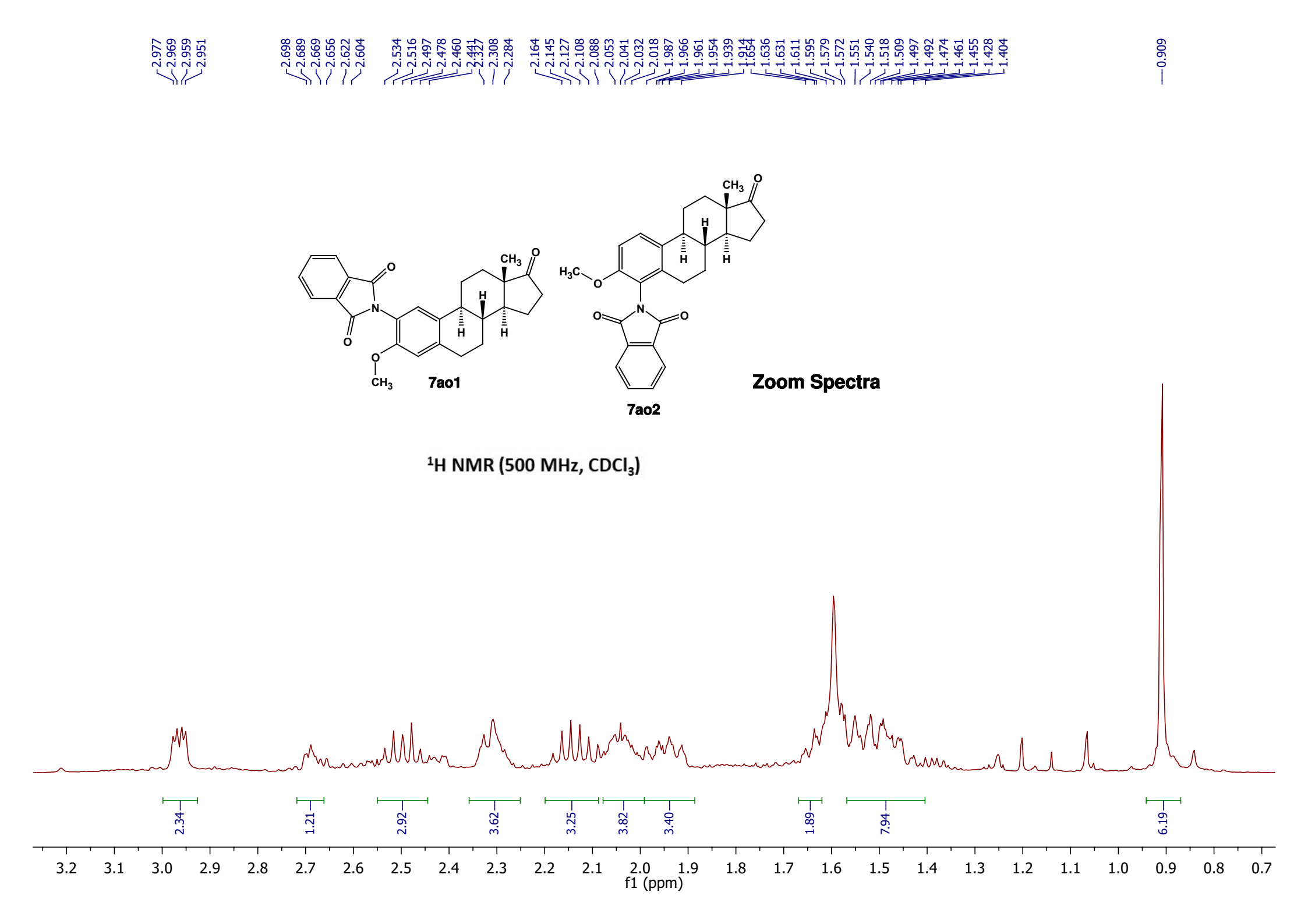
7ao1 : 7ao2 = 1.3 :1

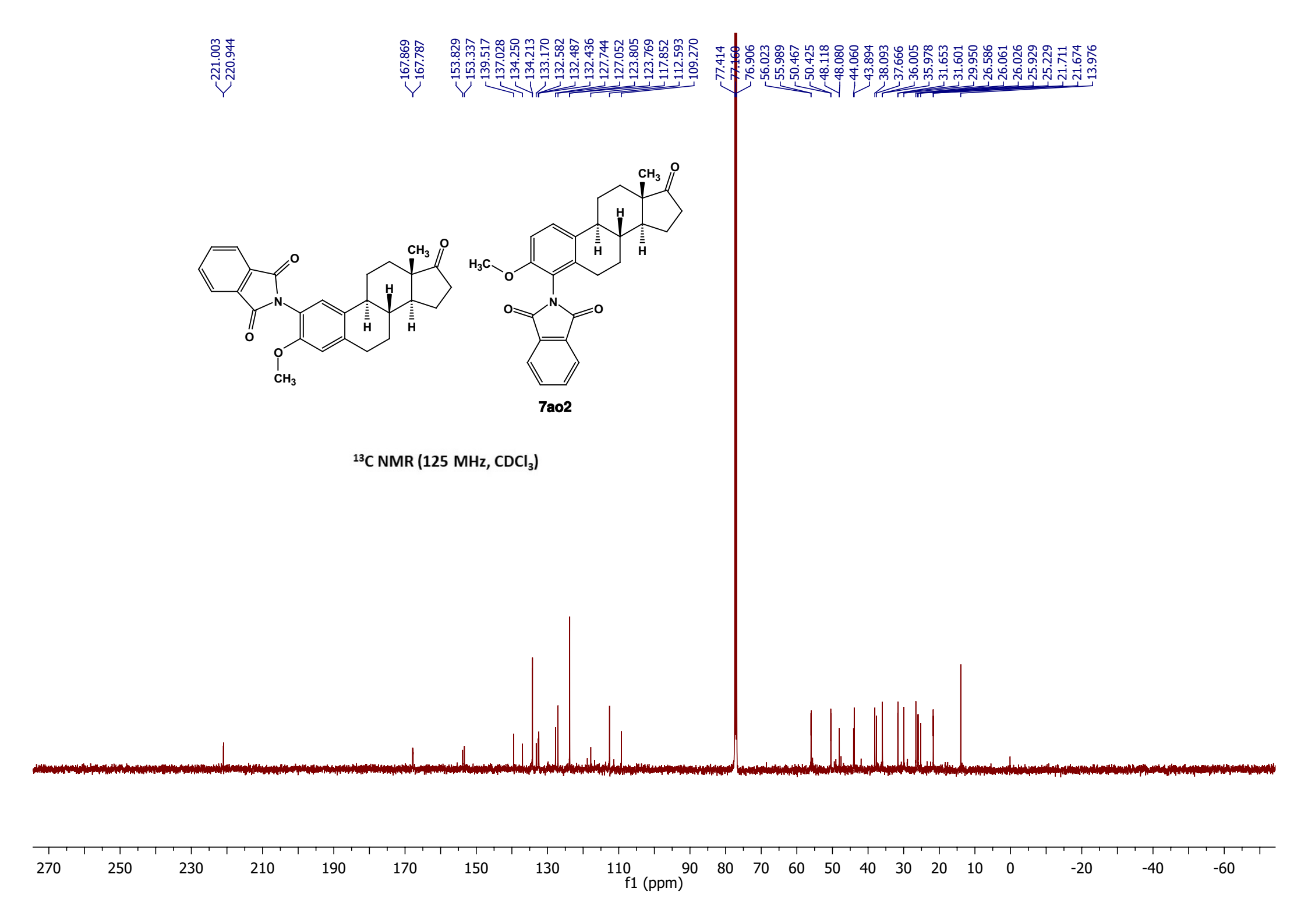
Isomeric distribution was measured by the integration of the aromatic C-H signals.

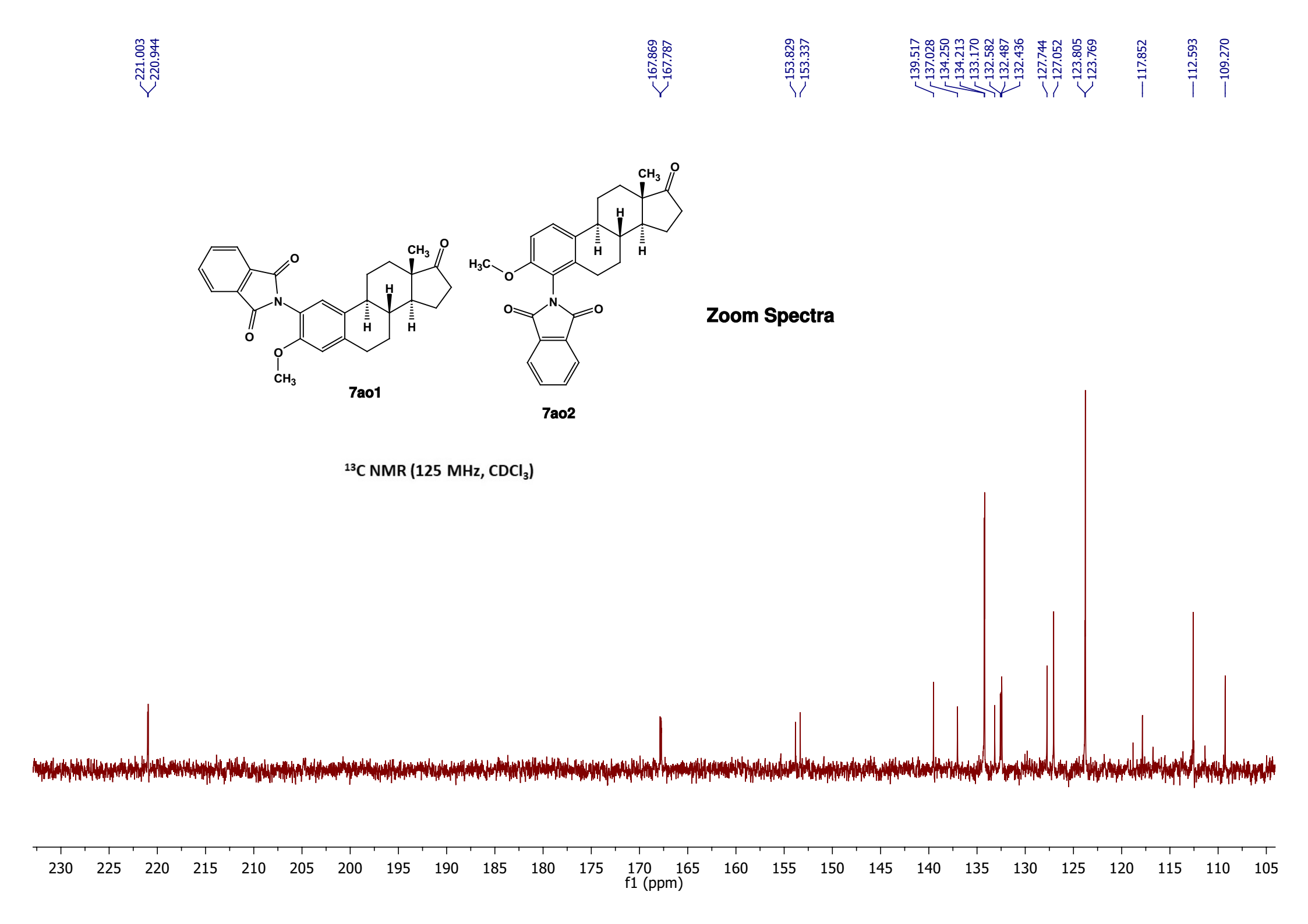
^{1}H NMR (500 MHz, $CDCl_3$)



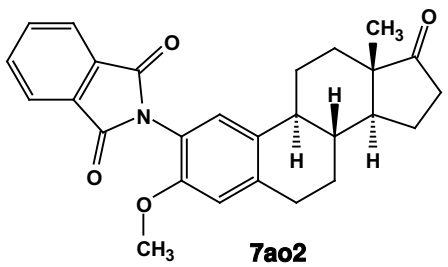

7.956
7.950
7.942
7.933
7.925
7.793
7.788
7.783
7.775
7.769
7.764
7.758
7.389
7.372
7.260
7.140
6.867
6.850
6.764
7ao1
7ao2
Zoom spectra
1H NMR (500 MHz, CDCl3)
3.65
3.79
0.77
1.00
0.96
1.13
8.2
8.1
8.0
7.9
7.8
7.7
7.6
7.5
7.4
7.3
7.2
7.1
7.0
6.9
6.8
6.7
f1 (ppm)


2.977
2.969
2.959
2.951
2.698
2.689
2.669
2.656
2.622
2.604
2.534
2.516
2.497
2.478
2.460
2.441
2.327
2.308
2.284
2.164
2.145
2.127
2.108
2.088
2.053
2.041
2.032
2.018
1.987
1.966
1.961
1.954
1.939
1.914
1.654
1.636
1.631
1.611
1.595
1.579
1.572
1.551
1.540
1.518
1.509
1.497
1.492
1.474
1.461
1.455
1.428
1.404
0.909
7ao1
7ao2
Zoom Spectra
1H NMR (500 MHz, CDCl3)
2.34
1.21
2.92
3.62
3.25
3.82
3.40
1.89
7.94
6.19
3.2 3.1 3.0 2.9 2.8 2.7 2.6 2.5 2.4 2.3 2.2 2.1 2.0 1.9 1.8 1.7 1.6 1.5 1.4 1.3 1.2 1.1 1.0 0.9 0.8 0.7
f1 (ppm)

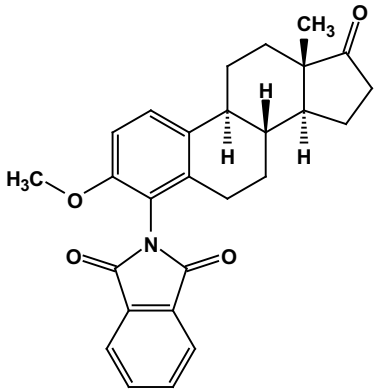

221.003
220.944
167.869
167.787
153.829
153.337
139.517
137.028
134.250
134.213
133.170
132.582
132.487
132.436
127.744
127.052
123.805
123.769
117.852
112.593
109.270
77.414
77.160
76.906
56.023
55.989
50.467
50.425
48.118
48.080
44.060
43.894
38.093
37.666
36.005
35.978
31.653
31.601
29.950
26.586
26.061
26.026
25.929
25.229
21.711
21.674
13.976
7ao2
13C NMR (125 MHz, CDCl3)
270
250
230
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
-20
-40
-60
f1 (ppm)


221.003
220.944
167.869
167.787
153.829
153.337
139.517
137.028
134.250
134.213
133.170
132.582
132.487
132.436
127.744
127.052
123.805
123.769
117.852
112.593
109.270
7ao1
7ao2
Zoom Spectra
13C NMR (125 MHz, CDCl3)
230
225
220
215
210
205
200
195
190
185
180
175
170
165
160
155
150
145
140
135
130
125
120
115
110
105
f1 (ppm)

56.023
55.989
50.467
50.425
48.118
48.080
44.060
43.894
38.093
37.666
36.005
35.978
31.653
31.601
29.950
26.586
26.061
26.026
25.929
25.229
21.711
21.674
13.976



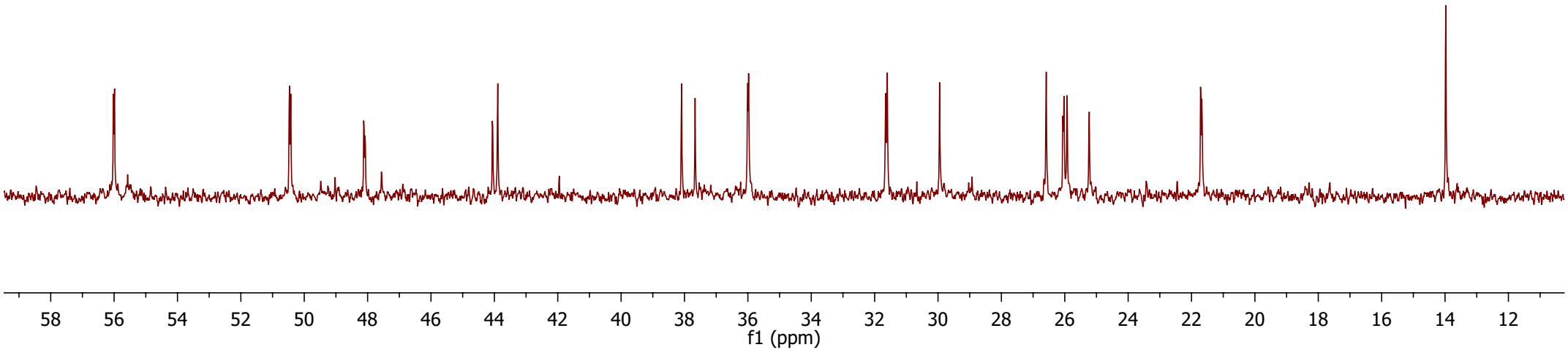
7ao2

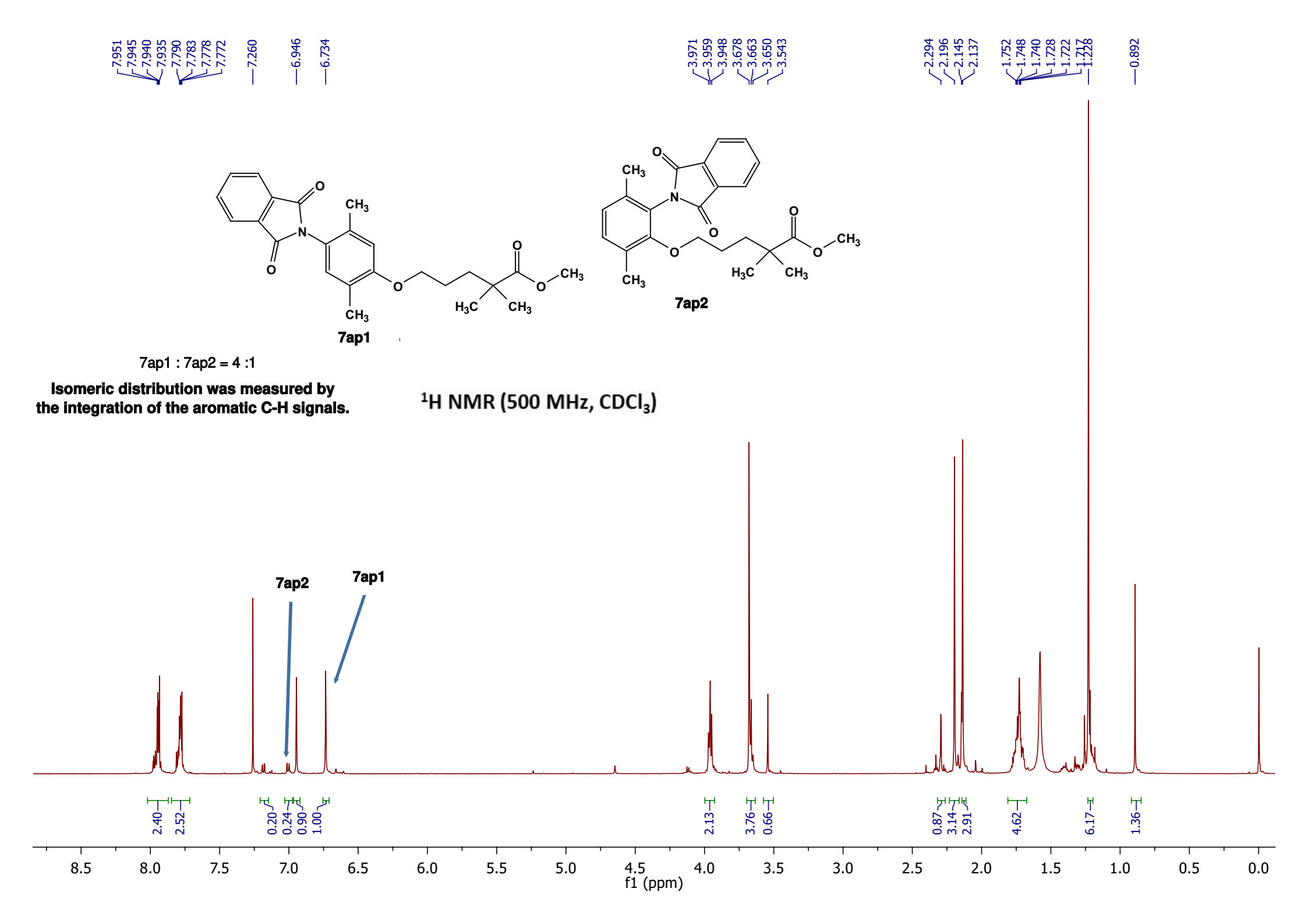


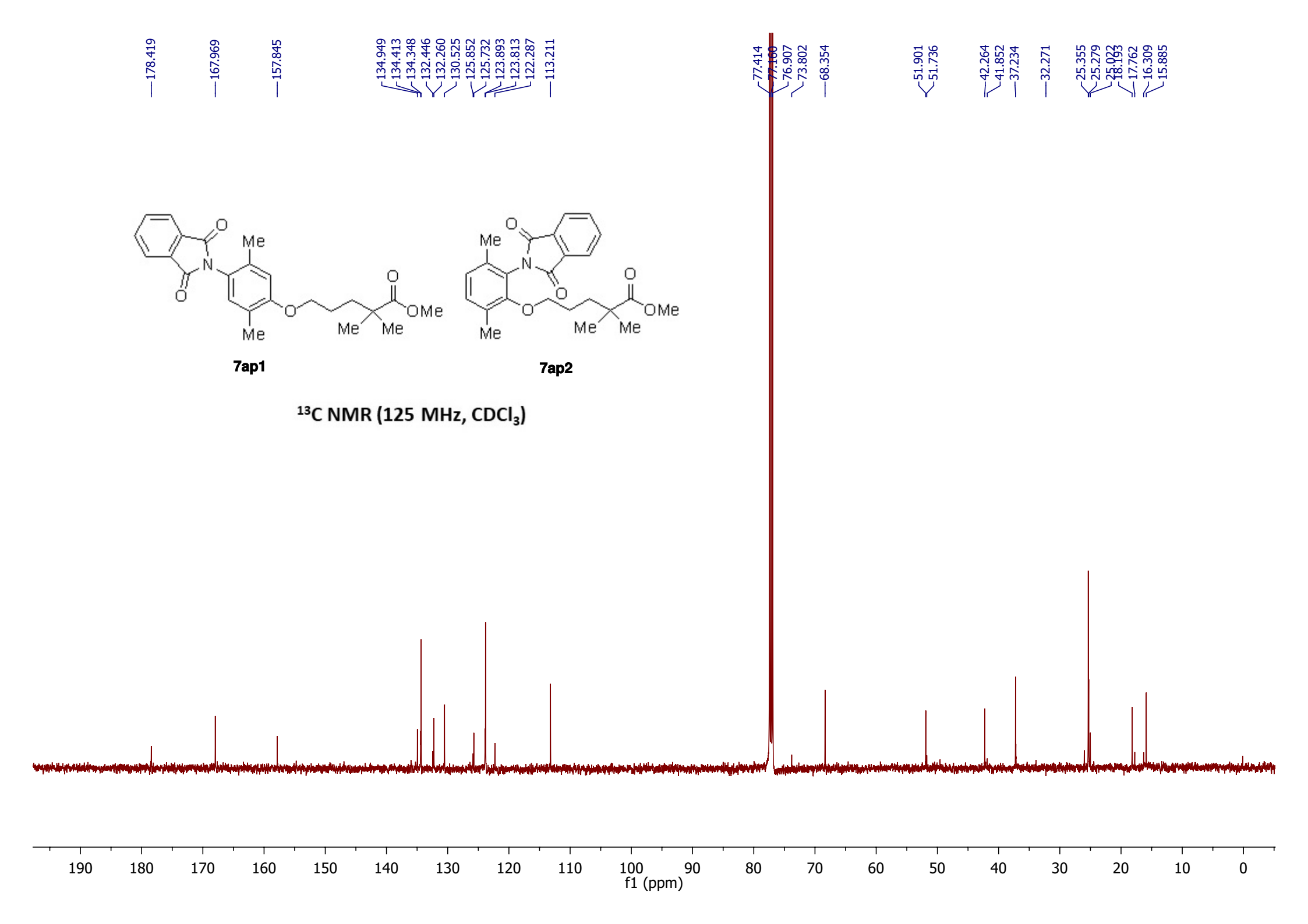
7ao2

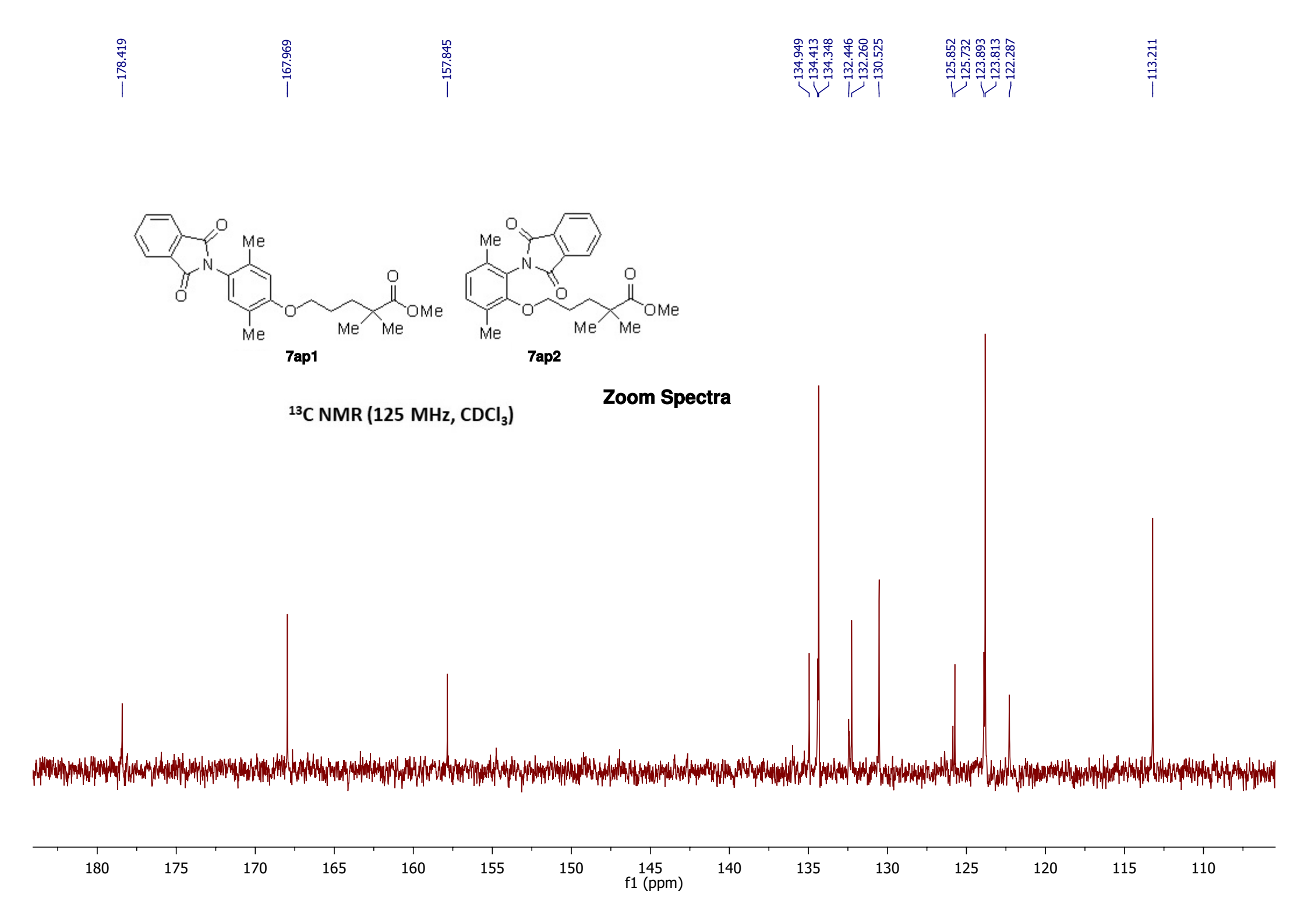
zoom Spectra

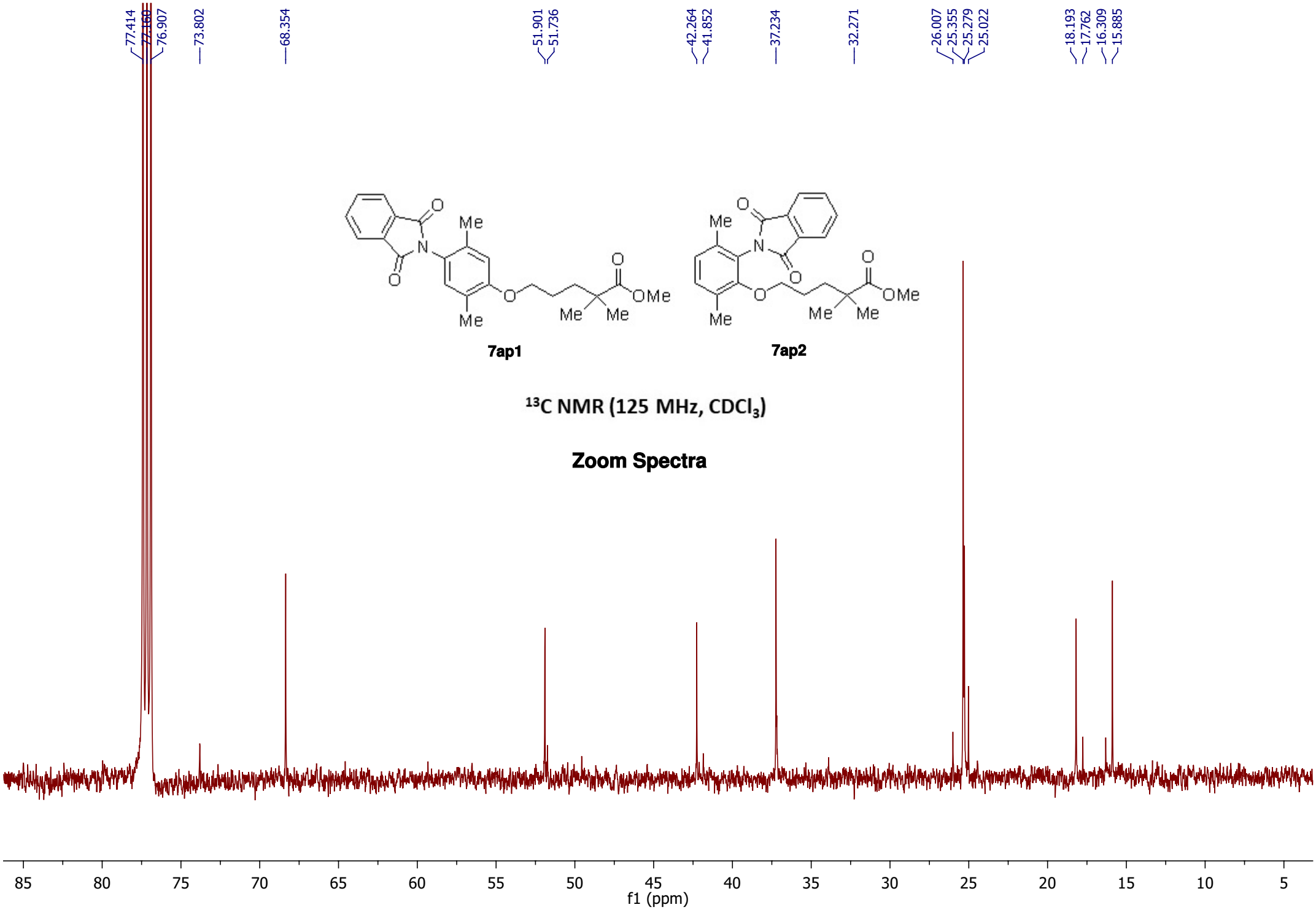
^{13}C NMR (125 MHz, $CDCl_3$)




7.951
7.945
7.940
7.935
7.790
7.783
7.778
7.772
7.260
6.946
6.734
3.971
3.959
3.948
3.678
3.663
3.650
3.543
2.294
2.196
2.145
2.137
1.752
1.748
1.740
1.728
1.722
1.717
1.228
0.892
7ap1
7ap2
7ap1 : 7ap2 = 4 :1
Isomeric distribution was measured by the integration of the aromatic C-H signals.
1H NMR (500 MHz, CDCl3)
7ap2
7ap1
2.40
2.52
0.20
0.24
0.90
1.00
2.13
3.76
0.66
0.87
3.14
2.91
4.62
6.17
1.36
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)


178.419
167.969
157.845
134.949
134.413
134.348
132.446
132.260
130.525
125.852
125.732
123.893
123.813
122.287
113.211
77.414
77.160
76.907
73.802
68.354
51.901
51.736
42.264
41.852
37.234
32.271
25.355
25.279
25.022
18.193
17.762
16.309
15.885
Me
O
N
O
O
Me
Me
Me
O
OMe
Me
Me
7ap1
7ap2
13C NMR (125 MHz, CDCl3)
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

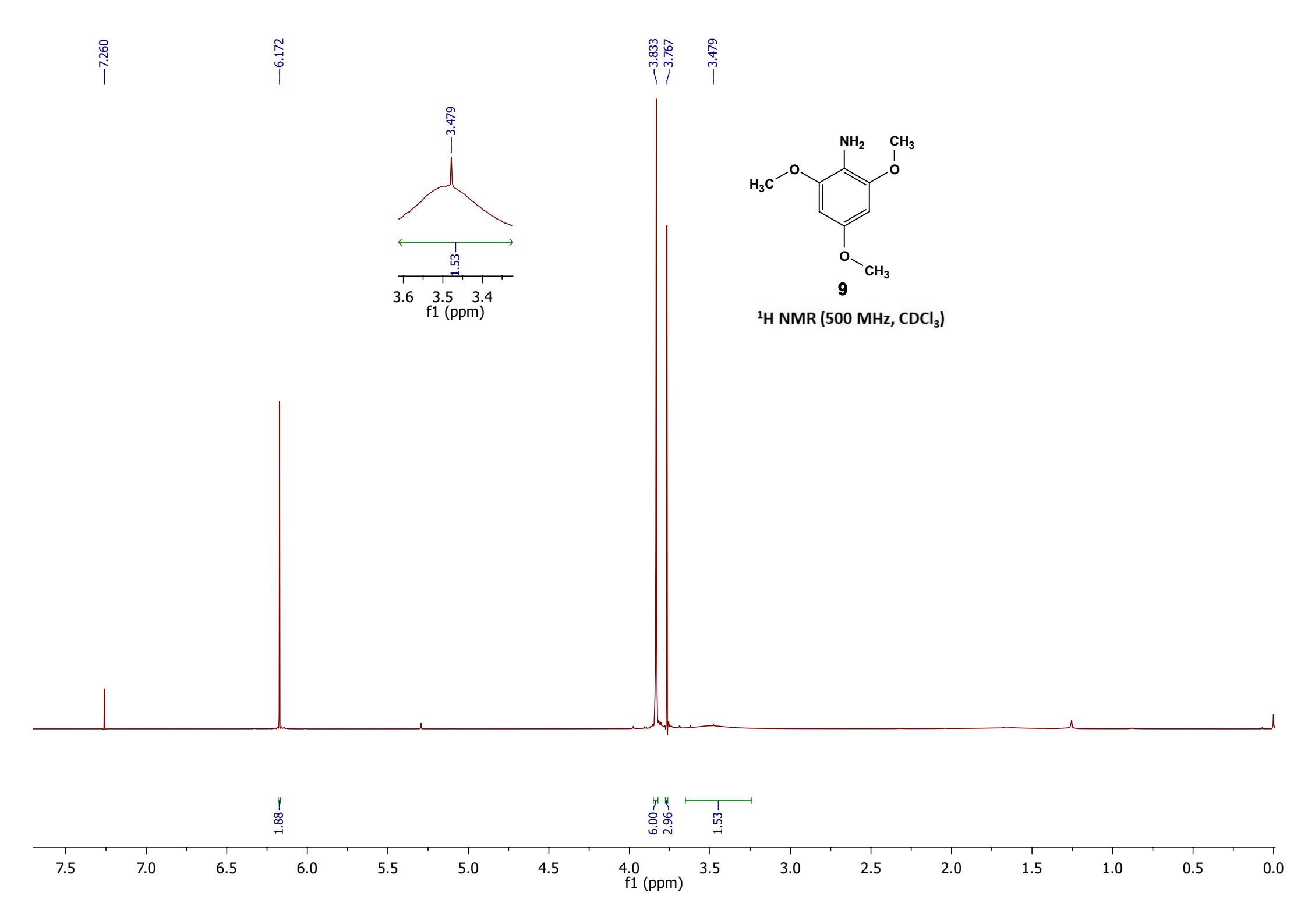

178.419
167.969
157.845
134.949
134.413
134.348
132.446
132.260
130.525
125.852
125.732
123.893
123.813
122.287
113.211
7ap1
7ap2
Zoom Spectra
13C NMR (125 MHz, CDCl3)
180
175
170
165
160
155
150
145
140
135
130
125
120
115
110
f1 (ppm)

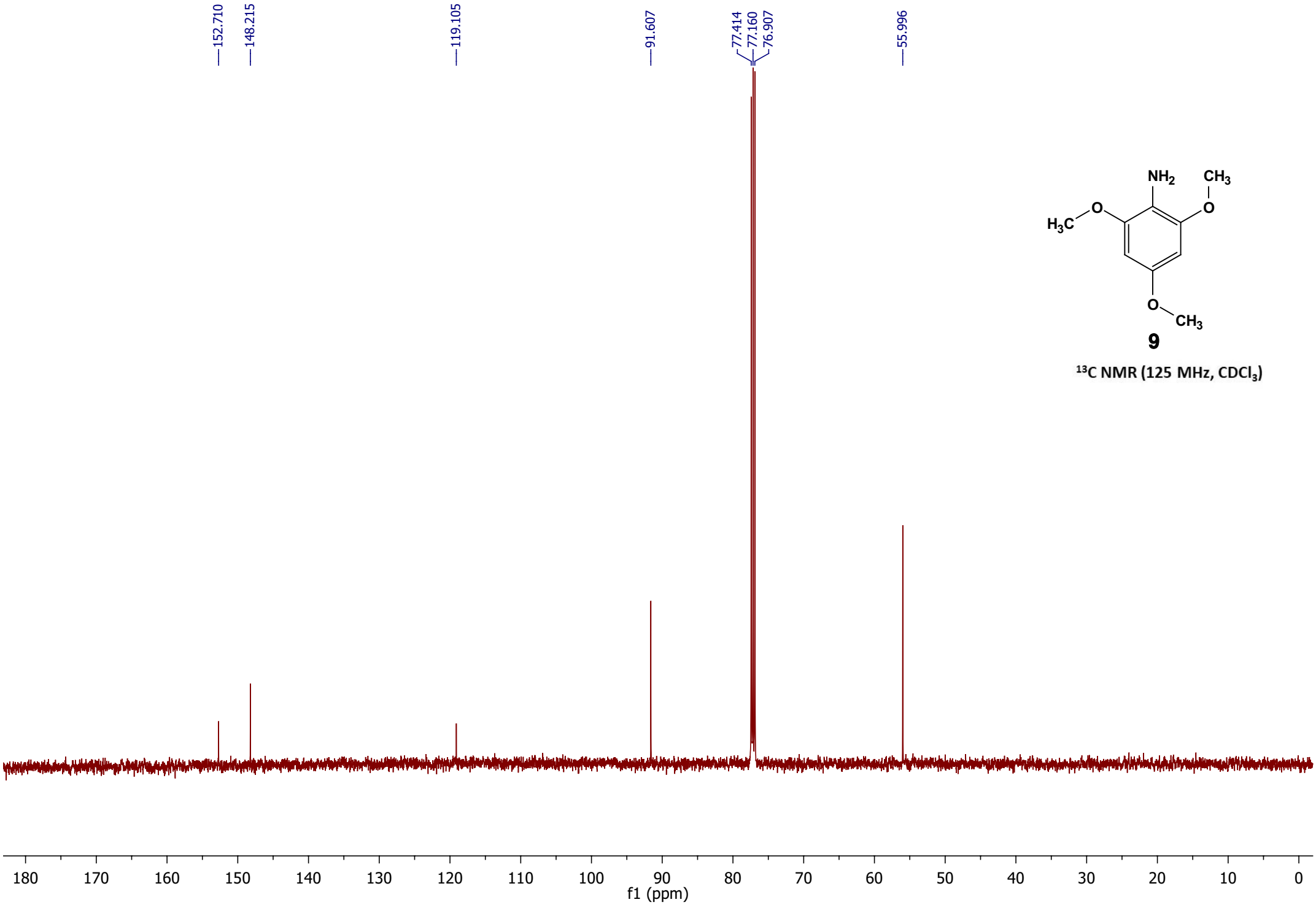


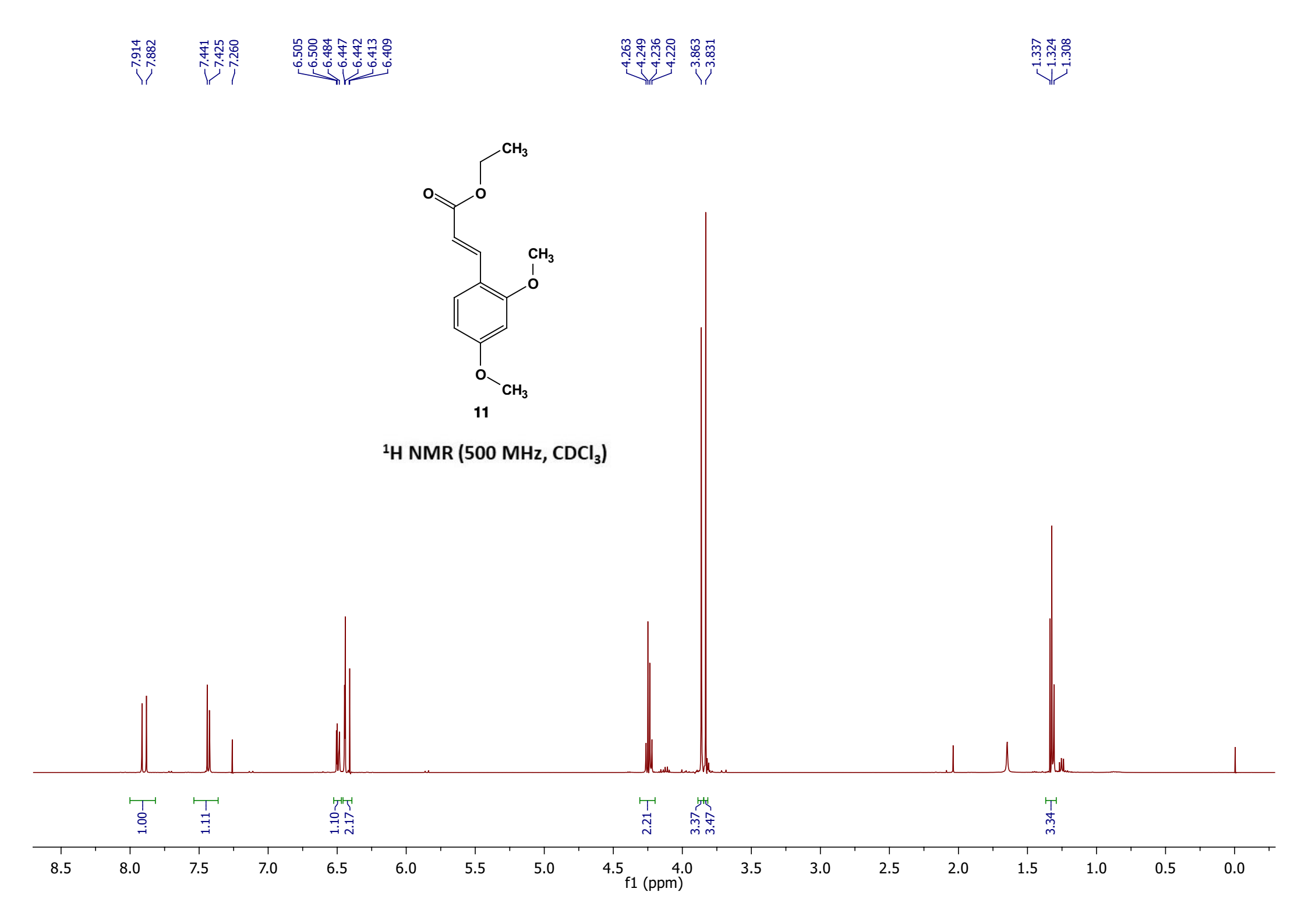
7ap1 **7ap2**

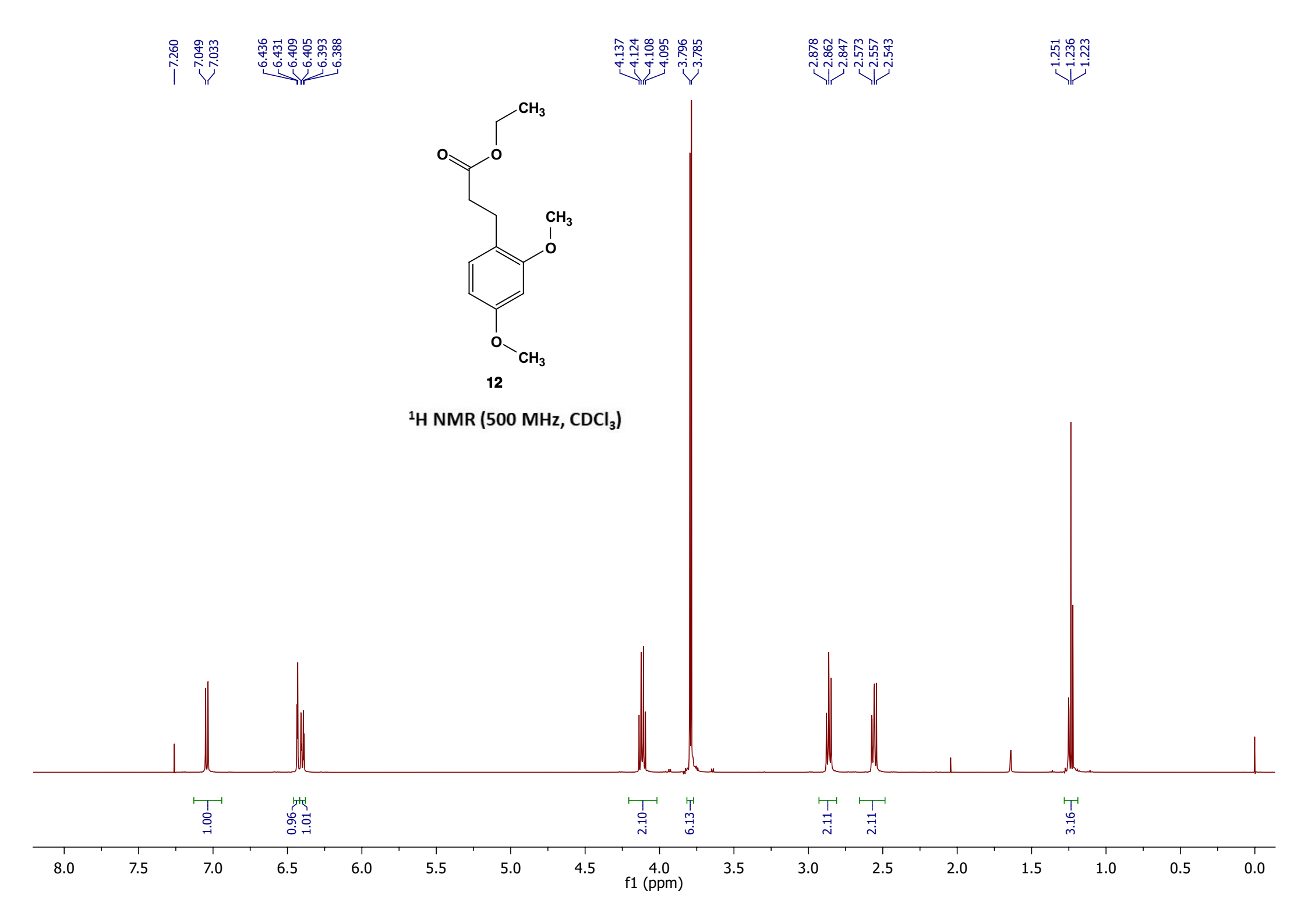
^{13}C NMR (125 MHz, $CDCl_3$)

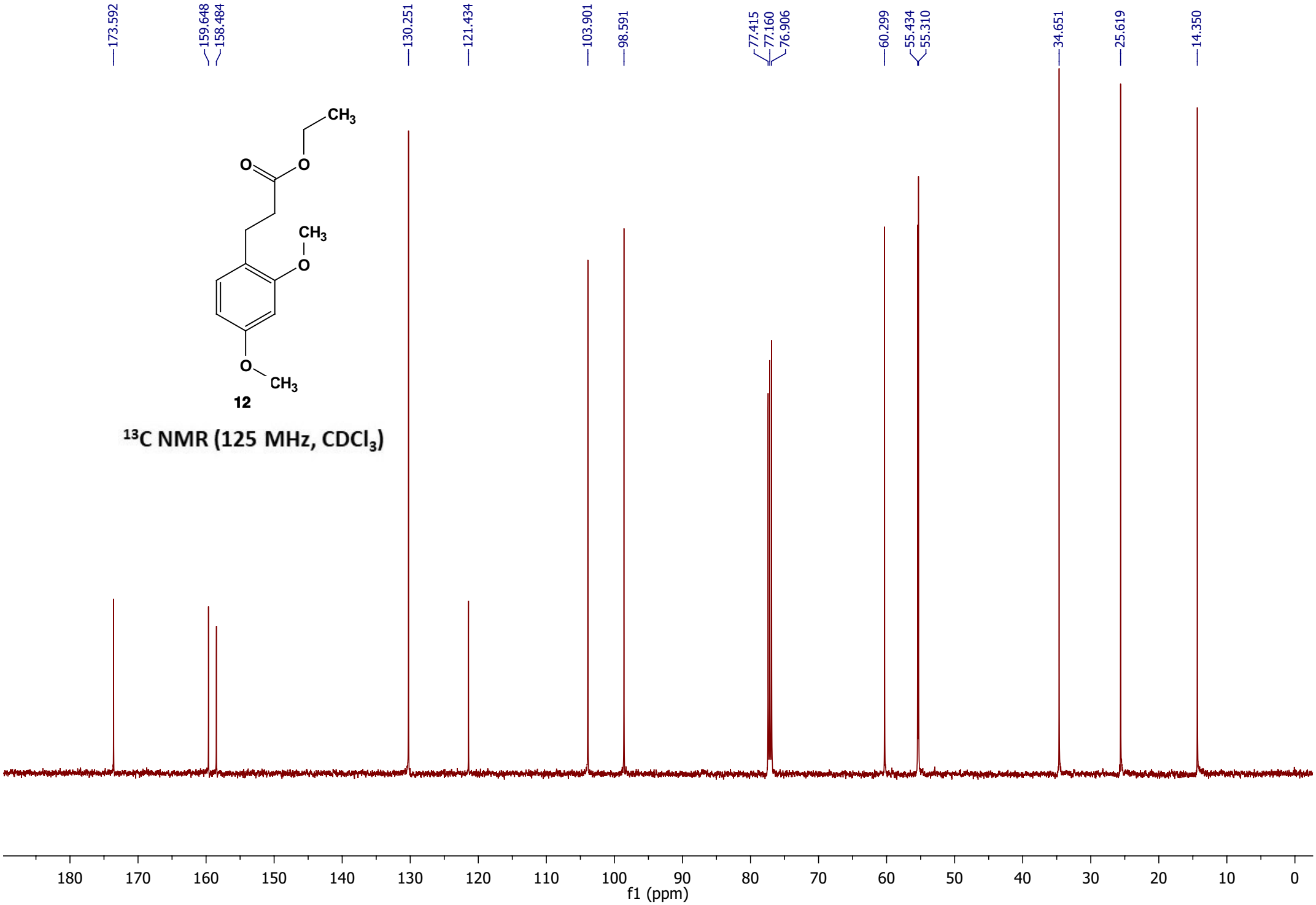
Zoom Spectra

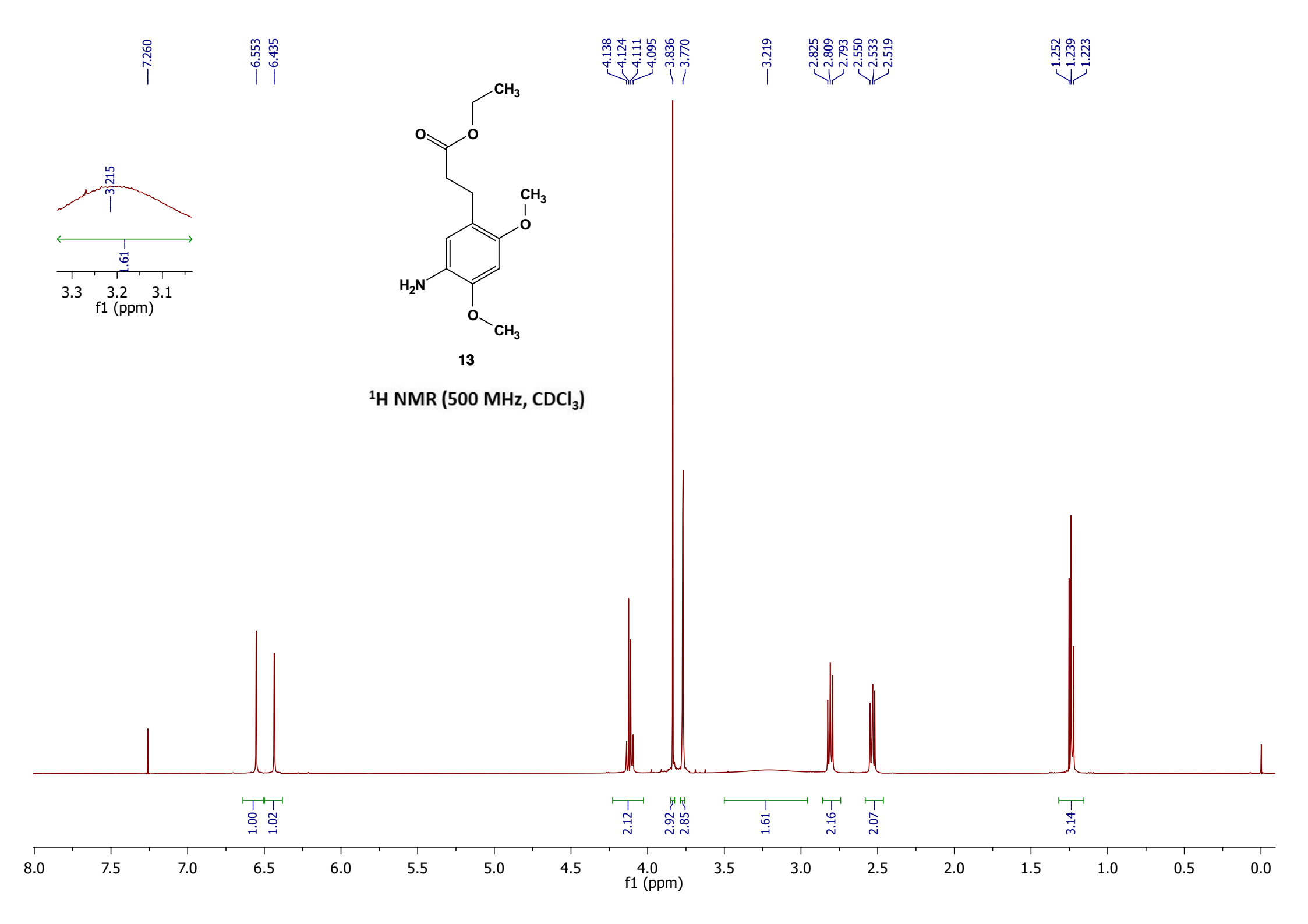

7.260
6.172
3.833
3.767
3.479
3.479
1.53
3.6
3.5
3.4
f1 (ppm)
NH2
CH3
O
H3C
O
O
CH3
9
1H NMR (500 MHz, CDCl3)
1.88
6.00
2.96
1.53
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

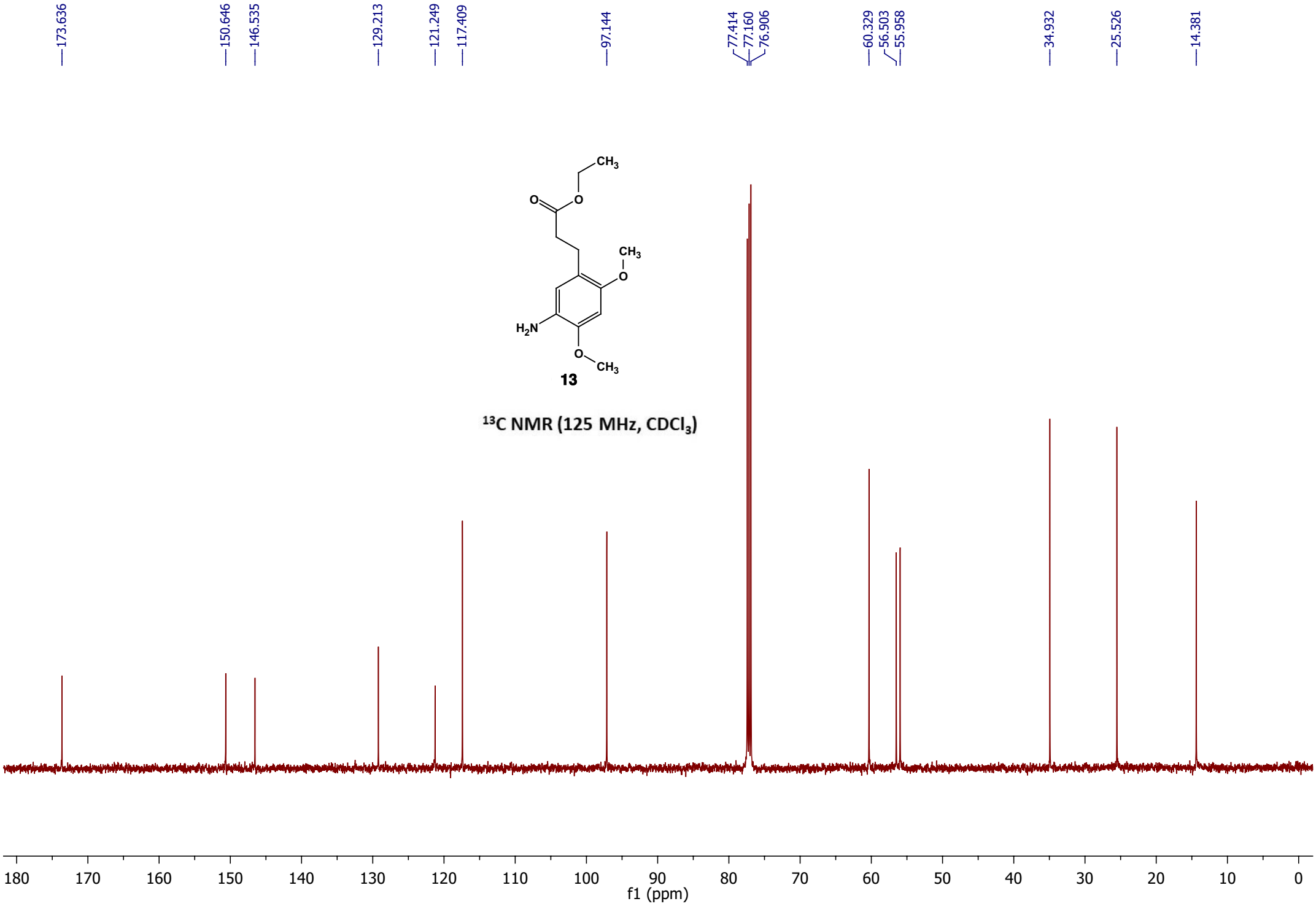

152.710
148.215
119.105
91.607
77.414
77.160
76.907
55.996
NH2
CH3
O
H3C
O
O
CH3
9
13C NMR (125 MHz, CDCl3)
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

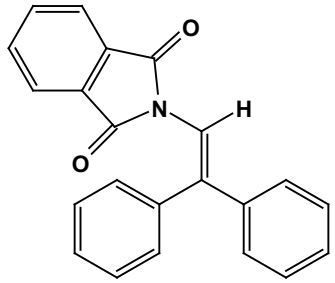

7.914
7.882
7.441
7.425
7.260
6.505
6.500
6.484
6.447
6.442
6.413
6.409
4.263
4.249
4.236
4.220
3.863
3.831
1.337
1.324
1.308
CH3
O
O
CH3
O
O
CH3
11
1H NMR (500 MHz, CDCl3)
1.00
1.11
1.10
2.17
2.21
3.37
3.47
3.34
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


7.260
7.049
7.033
6.436
6.431
6.409
6.405
6.393
6.388
4.137
4.124
4.108
4.095
3.796
3.785
2.878
2.862
2.847
2.573
2.557
2.543
1.251
1.236
1.223
CH3
O
O
CH3
O
O
CH3
12
1H NMR (500 MHz, CDCl3)
1.00
0.96
1.01
2.10
6.13
2.11
2.11
3.16
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)


173.592
159.648
158.484
130.251
121.434
103.901
98.591
77.415
77.160
76.906
60.299
55.434
55.310
34.651
25.619
14.350
CH3
O
O
CH3
O
O
CH3
12
13C NMR (125 MHz, CDCl3)
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

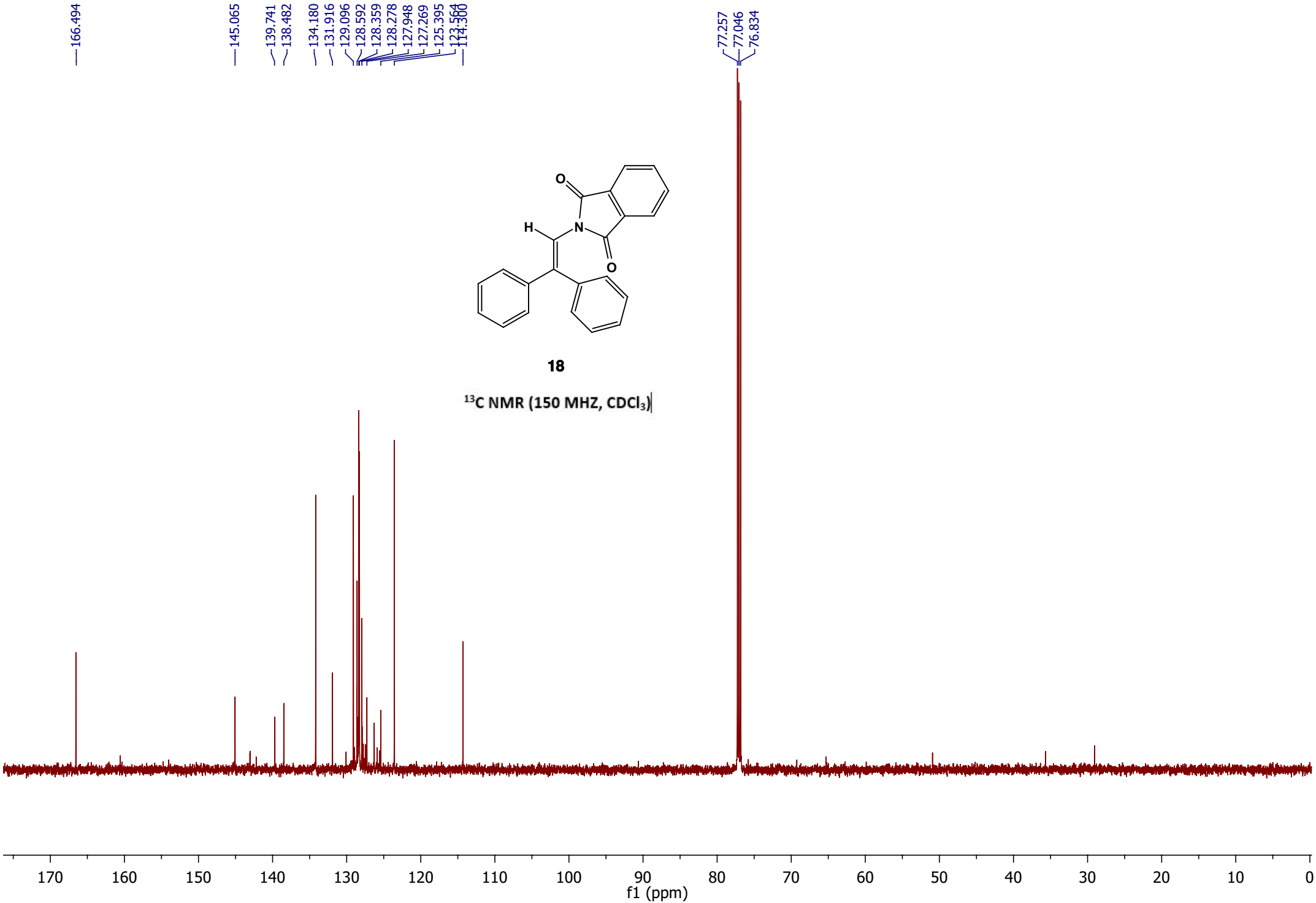

7.260
6.553
6.435
4.138
4.124
4.111
4.095
3.836
3.770
3.219
2.825
2.809
2.793
2.550
2.533
2.519
1.252
1.239
1.223
3.215
1.61
3.3
3.2
3.1
f1 (ppm)
CH3
O
O
CH3
O
H2N
O
CH3
13
1H NMR (500 MHz, CDCl3)
1.00
1.02
2.12
2.92
2.85
1.61
2.16
2.07
3.14
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


173.636
150.646
146.535
129.213
121.249
117.409
97.144
77.414
77.160
76.906
60.329
56.503
55.958
34.932
25.526
14.381
CH3
O
O
CH3
O
H2N
O
CH3
13
13C NMR (125 MHz, CDCl3)
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)



18

^{1}H NMR (500 MHz, $CDCl_3$)


166.494
145.065
139.741
138.482
134.180
131.916
129.096
128.592
128.359
128.278
127.948
127.269
125.395
123.564
114.300
77.257
77.046
76.834
O
H
N
O
18
^{13}C NMR (150 MHZ, $CDCl_3$)
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)