Supporting Information

Palladium-Catalyzed Cascade Aryl Addition/Intramolecular Lactonization of Phthalaldehyde to Access 3-Aryl and Alkenyl Phthalides

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Table of contents

1. General experimental details

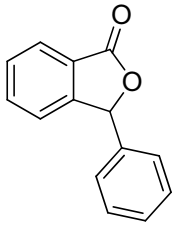
Chemicals were either purchased or purified by standard techniques without special instructions. ^{1}H NMR and ^{13}C NMR spectra were measured on a 300 or 500 MHz spectrometer (^{1}H 300 MHz, ^{13}C 75 MHz or ^{1}H 500 MHz, ^{13}C 125 MHz), using $CDCl_3$ as the solvent with tetramethylsilane (TMS) as the internal standard at room temperature. Chemical shifts are given in δ relative to TMS, the coupling constants *J* are given in Hz. All reactions were conducted under air atmosphere. Column chromatography was performed using EM Silica gel 60 (300-400 mesh).

1.1 General procedure:

Under air, a reaction tube was charged with phthalaldehyde (26.8 mg, 0.2 mmol), boronic acids (0.3 mmol), $PdCl_2$ (1.7 mg, 5 mol %), $P(1\text{-nap})_3$ (4.2 mg, 5 mol %), K_2CO_3 (82.8 mg, 3 equiv) and dry THF (3 mL). The reaction tube was kept stirring at 65 ^{o}C for 12 h. After the completion of the reaction, as monitored by TLC, the solvent was evaporated under reduced pressure and the residue was purified by flash column chromatography on a silica gel to give the product.

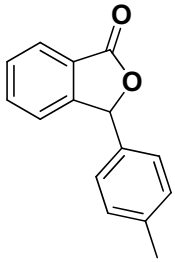
2. Experimental characterization data for compounds

3-phenylisobenzofuran-1(3*H*)-one[1] (3a)



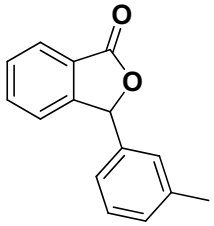
^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.97 (d, *J* = 7.6 Hz, 1H), 7.68-7.63 (m, 1H), 7.58-7.53 (m, 1H), 7.38-7.26 (m, 6H), 6.41 (s, 1H).
^{13}C NMR ($CDCl_3$, 125 MHz): δ 170.7, 149.8, 136.6, 134.5, 129.5, 129.4, 129.1 127.1, 125.8, 125.7, 123.0, 82.9.

3-*p*-tolylisobenzofuran-1(3*H*)-one[2] (3b)



^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.96 (d, *J* = 7.4 Hz, 1H), 7.67-7.62 (m, 1H), 7.57-7.52 (m, 1H), 7.32 (d, *J* = 7.6 Hz, 1H), 7.19 (d, *J* = 8.4 Hz, 2H), 7.14 (d, *J* = 8.4 Hz, 2H), 6.38 (s, 1H), 2.35 (s, 3H).
^{13}C NMR ($CDCl_3$, 125 MHz): δ 170.7, 145.0, 139.5, 134.4, 133.5, 129.8, 129.4, 127.2, 125.9, 125.8, 123.0, 82.9, 21.4.

3-*m*-tolylisobenzofuran-1(3*H*)-one[3] (3c)



^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.97 (d, *J* = 7.5 Hz, 1H), 7.65-7.60 (m, 1H), 7.58-7.53 (m, 1H), 7.35-7.19 (m, 3H), 7.10-7.07 (m, 2H), 6.37 (s, 1H), 2.33 (s, 3H).
^{13}C NMR ($CDCl_3$, 125 MHz): δ 170.8, 145.0, 139.0, 136.5, 134.4, 130.2, 129.5, 129.0, 127.6, 125.8, 125.7, 124.2, 123.0, 83.0, 21.5.

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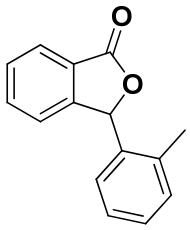
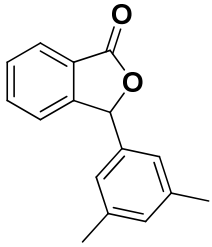
[1] Nagaki, A.; Kim, H.; Yoshida, J. *Angew. Chem., Int. Ed.* **2008**, *47*, 7833.
[2] Ye, Z.; Lv, G.; Wang, W.; Zhang, M.; Cheng, J. *Angew. Chem., Int. Ed.* **2010**, *49*, 3671.
[3] Fau, M.; Molnar, J.; Heindel, N. *Bull. Soc. Chim. Fr.* **1983**, 164

3-*o*-tolylisobenzofuran-1(3*H*)-one[4] (3d)



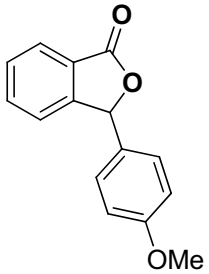
^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.89 (d, *J* = 7.5 Hz, 1H), 7.59-7.54 (m, 1H), 7.51-7.46 (m, 1H), 7.34-7.17 (m, 3H), 7.05-7.03 (m, 1H), 6.83 (d, *J* = 7.8 Hz, 1H), 6.60 (s, 1H), 2.41 (s, 3H).
^{13}C NMR ($CDCl_3$, 125 MHz): δ 170.7, 149.4, 137.3, 134.3, 134.2, 131.2, 129.5, 129.4, 127.4, 126.5, 125.8, 125.8, 123.1, 80.7, 19.4.

3-(3,5-dimethylphenyl)isobenzofuran-1(3*H*)-one[2] (3e)



^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.97 (d, *J* = 7.6 Hz, 1H), 7.65-7.60 (m, 1H), 7.58-7.54 (m, 1H), 7.34 (d, *J* = 7.6 Hz, 1H), 7.00 (s, 1H), 6.88 (s, 2H), 6.33 (s, 1H), 2.30 (s, 6H).
^{13}C NMR ($CDCl_3$, 125 MHz): δ 170.7, 145.0, 138.8, 136.4, 134.4, 131.0, 129.3, 125.7, 125.7, 124.7, 122.9, 83.0, 21.3.

3-(4-methoxyphenyl)isobenzofuran-1(3*H*)-one[5] (3f)



^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.86 (d, *J* = 7.5 Hz, 1H), 7.56-7.53 (m, 1H), 7.49-7.44 (m, 1H), 7.22 (d, *J* = 7.7 Hz, 1H), 7.08 (d, *J* = 8.6 Hz, 2H), 6.79 (d, *J* = 8.6 Hz, 2H), 6.28 (s, 1H), 3.70 (s, 3H).
^{13}C NMR ($CDCl_3$, 125 MHz): δ 170.7, 160.6, 149.9, 134.4, 129.4, 128.9, 128.4, 126.1, 125.7, 123.0, 114.5, 82.9, 55.5.

3-(4-fluorophenyl)isobenzofuran-1(3*H*)-one[2] (3g)

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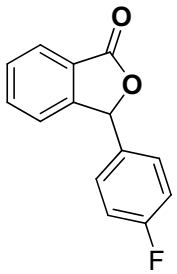
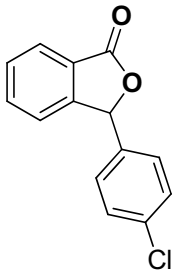
[4] Konosonoks, A.; Wright, P. J.; Tsao, M.; Pika, J.; Novak, K.; Mandel, S. M.; Krause, B.; Jeanette, A.; Bohne, C.; Gudmundsdottir, A. D. *J. Org. Chem.* **2005**, *70*, 2763.
[5] Meek, S. T.; Nesterov, E. E.; Swager, T. M. *Org. Lett.* **2008**, *10*, 2991.



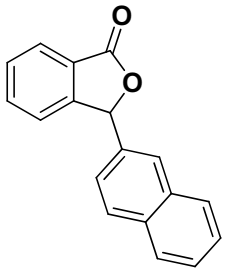
^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.88 (d, J = 7.5 Hz, 1H), 7.60-7.55 (m, 1H), 7.50-7.45 (m, 1H), 7.23-7.13 (m, 3H), 7.00-6.94 (m, 2H), 6.30 (s, 1H).
^{13}C NMR ($CDCl_3$, 125 MHz): δ 170.4, 163.3 (d, J_{C-F} = 247.5 Hz), 149.5, 134.6, 132.4, 129.7, 129.2 (d, J_{C-F} = 8.8 Hz), 125.9, 125.8, 123.0, 116.0 (d, J_{C-F} = 21.2 Hz), 82.1.

3-(4-chlorophenyl)isobenzofuran-1(3*H*)-one[6] (3h)



^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.88 (d, J = 7.7 Hz, 1H), 7.60-7.55 (m, 1H), 7.50-7.44 (m, 1H), 7.24-7.10 (m, 5H), 6.28 (s, 1H).
^{13}C NMR ($CDCl_3$, 125 MHz): δ 170.3, 149.4, 135.5, 135.1, 134.6, 129.7, 129.4, 128.5, 125.8, 126.0, 122.9, 82.0.

3-(naphthalen-2-yl)isobenzofuran-1(3*H*)-one[2] (3i)



^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.92 (d, J = 7.4 Hz, 1H), 7.76-7.73 (m, 4H), 7.56-7.41 (m, 4H), 7.29-7.14 (m, 2H), 6.48 (s, 1H).
^{13}C NMR ($CDCl_3$, 125 MHz): δ 170.8, 149.9, 134.5, 133.8, 133.8, 133.2, 129.6, 129.2, 128.2, 128.0, 127.0, 126.9, 126.8, 125.9, 125.8, 123.9, 123.0, 83.1.

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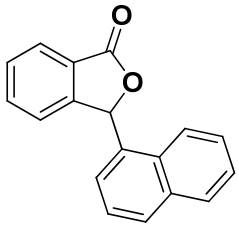
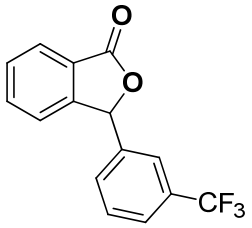
[6] Chang, H.; Jeganmohan, M.; Cheng, C. *Chem.— Eur. J.* **2007**, *13*, 4356.

3-(naphthalen-1-yl)isobenzofuran-1(3*H*)-one[6] (3j)



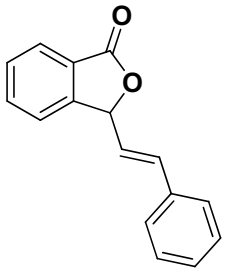
^{1}H NMR ($CDCl_3$, 300 MHz): δ 8.16 (d, J = 8.1 Hz, 1H), 7.93 (d, J = 7.2 Hz, 1H), 7.86-7.78 (m, 2H), 7.58-7.50 (m, 4H), 7.36-7.28 (m, 2H), 7.19-7.17 (m, 2H).
^{13}C NMR ($CDCl_3$, 125 MHz): δ 170.7, 149.4, 134.3, 134.1, 132.1, 131.5, 130.1, 129.6, 129.2, 127.2, 126.4, 126.3, 126.2, 125.4, 124.7, 123.4, 123.1, 79.8.

3-(3-(trifluoromethyl)phenyl)isobenzofuran-1(3*H*)-one[7] (3k)



^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.92 (d, J = 7.6 Hz, 1H), 7.62-7.42 (m, 6H), 7.26 (d, J = 7.6 Hz, 1H), 6.39 (s, 1H).
^{13}C NMR ($CDCl_3$, 125 MHz): δ 170.2, 149.0, 137.8, 134.8, 131.7 (q, $J_{C\text{-}F}$ = 33.8 Hz), 130.2, 126.3 (q, $J_{C\text{-}F}$ = 3.8 Hz), 125.6, 124.9, 123.9 (q, $J_{C\text{-}F}$ = 270.2 Hz), 123.6 (q, $J_{C\text{-}F}$ = 3.6 Hz), 81.8.

(*E*)-3-styrylisobenzofuran-1(3*H*)-one[6] (3l)



^{1}H NMR ($CDCl_3$, 300 MHz): δ 7.90 (d, J = 7.6 Hz, 1H), 7.65-7.61 (m, 1H), 7.53-7.51 (m, 1H), 7.43-7.29 (m, 6H), 6.86 (d, J = 15.5 Hz, 1H), 6.07 (dd, J_1 = 15.5 Hz, J_2 = 7.9 Hz, 1H), 5.97 (d, J_1 = 7.9 Hz, 1H).
^{13}C NMR ($CDCl_3$, 125 MHz): δ 170.4, 148.9, 135.6, 135.3, 134.3, 129.6, 128.9, 128.8, 127.0, 126.0, 125.9, 124.0, 122.8, 82.2.

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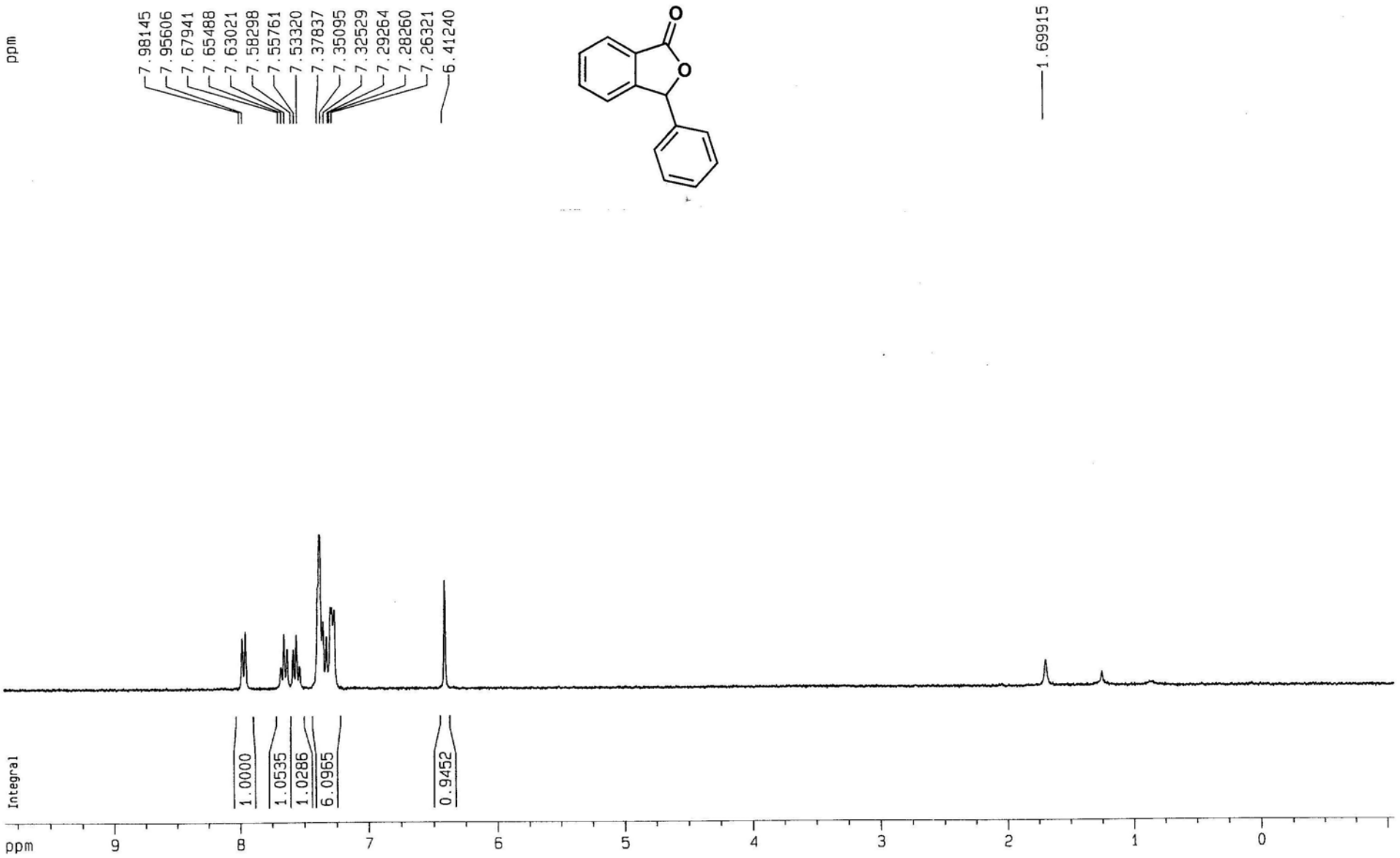
[7] Font-Sanchis, E.; Aliaga, C.; Cornejo, R.; Scaiano, J. C. *Org. Lett.* **2003**, *5*, 1515.

3a ^{1}H NMR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4
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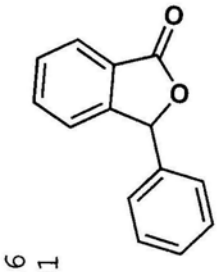
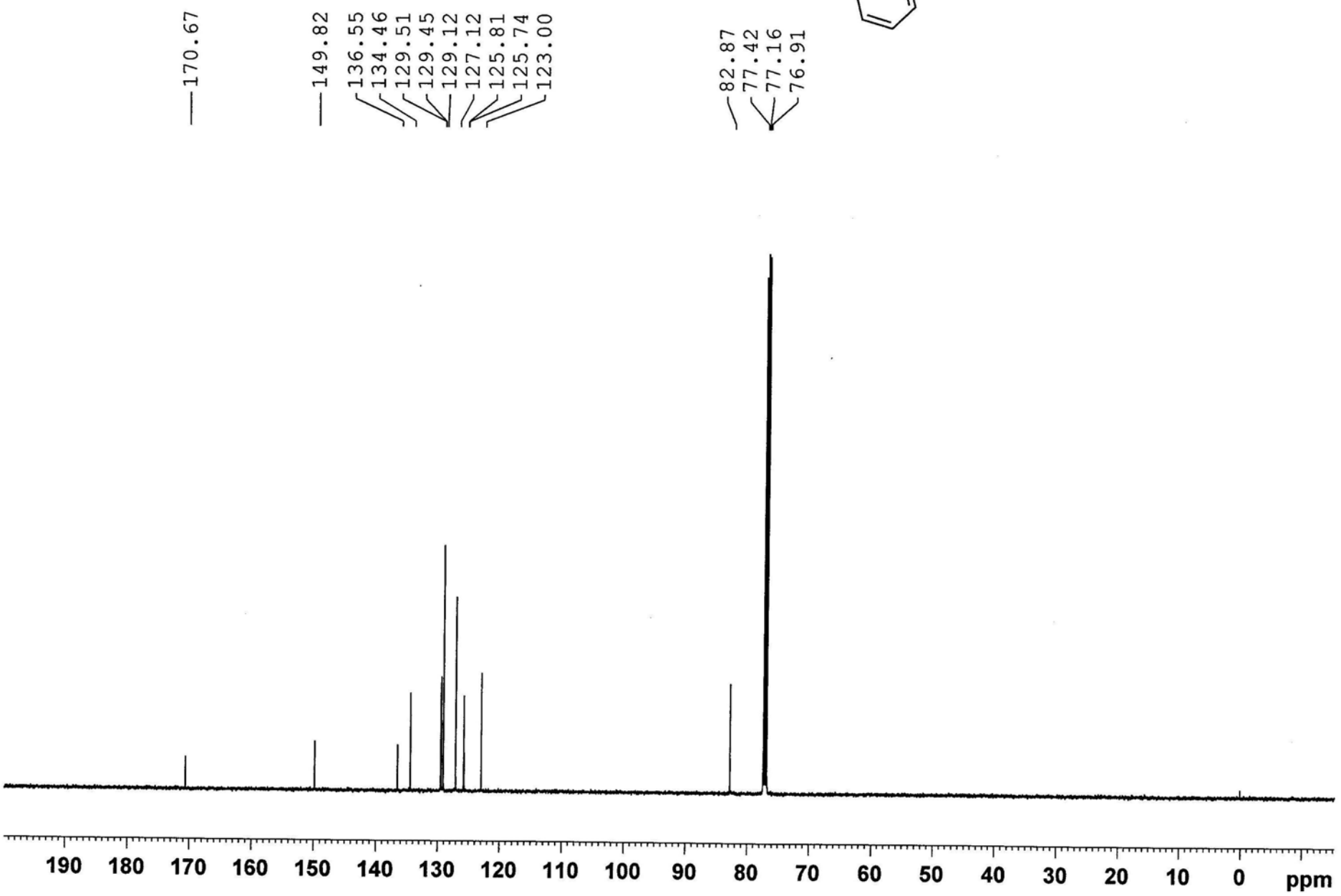
3a ^{13}C NMR

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76.91
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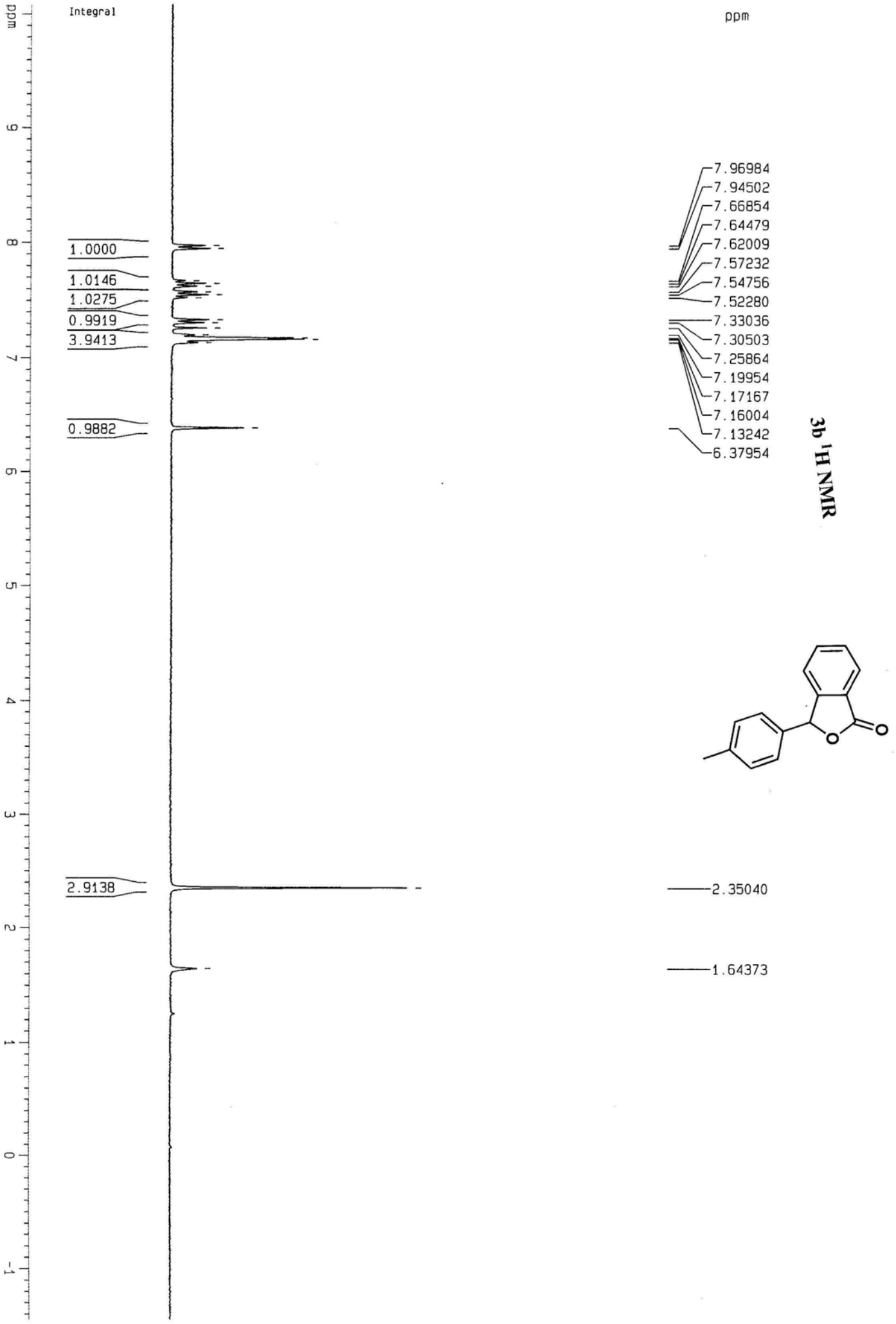
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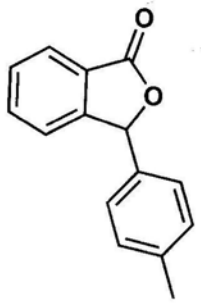
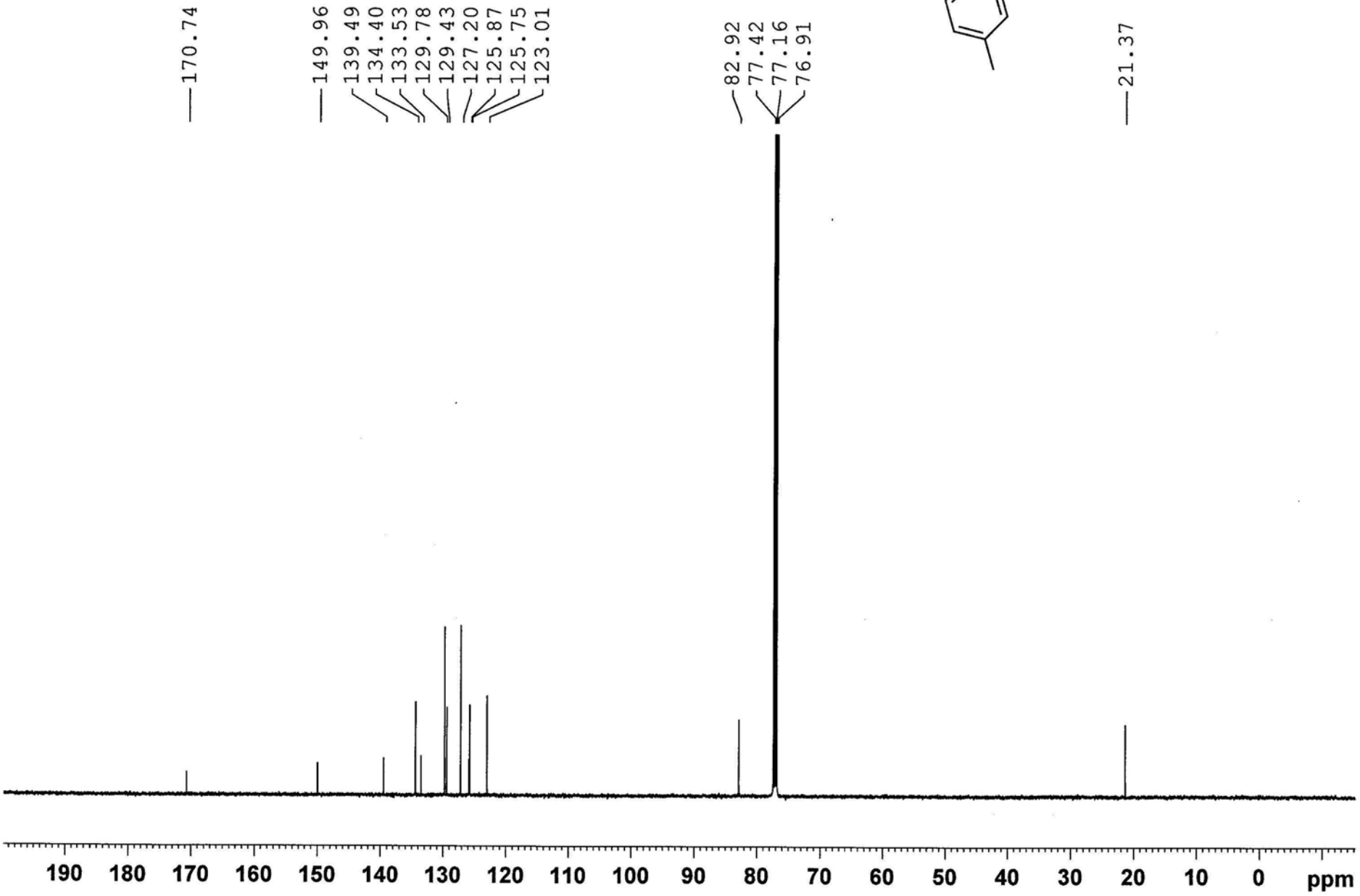
3b ^{1}H NMR

3b ^{13}C NMR

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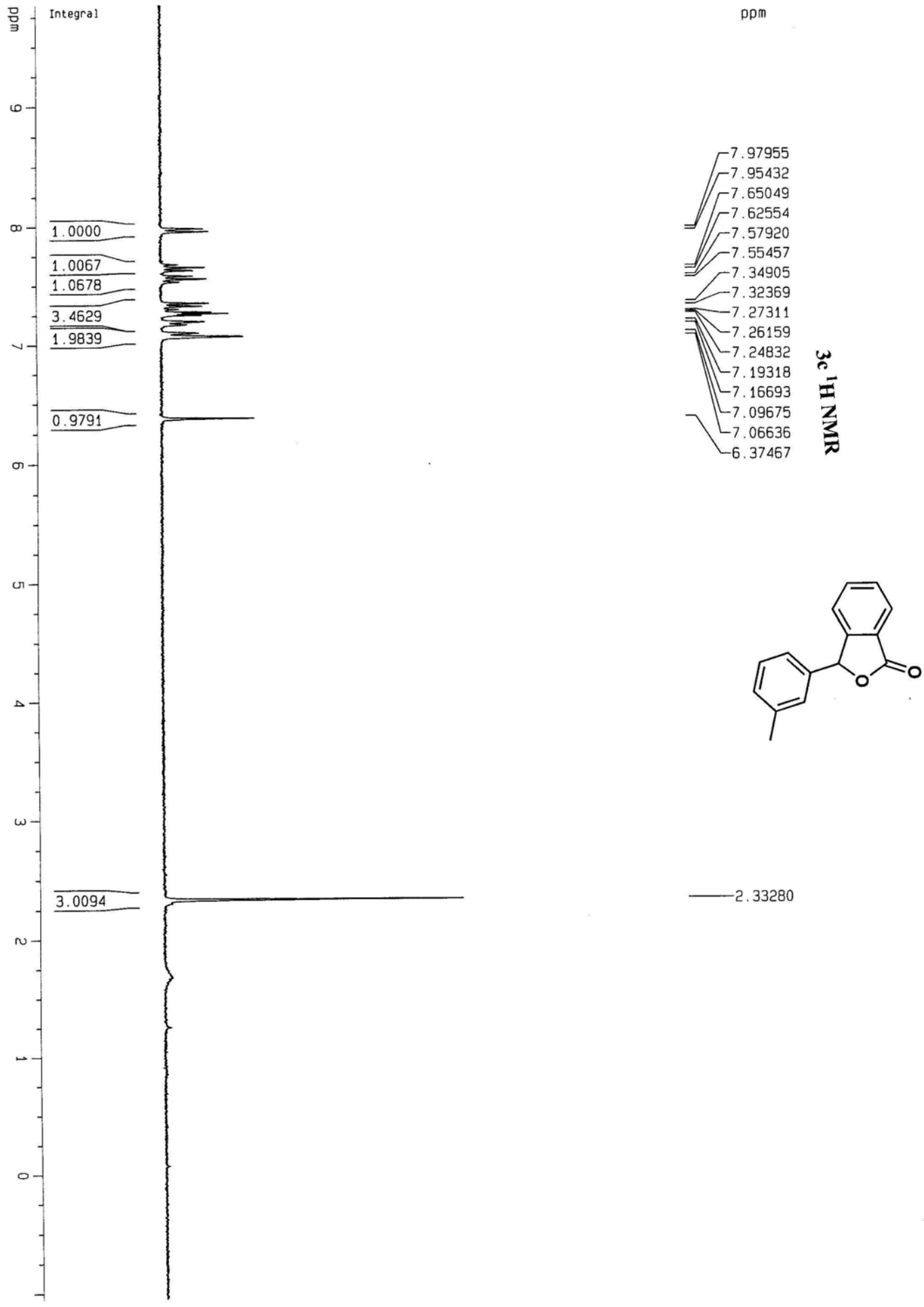
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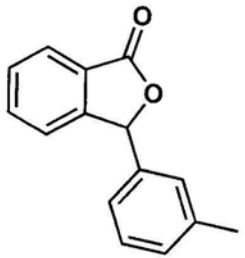
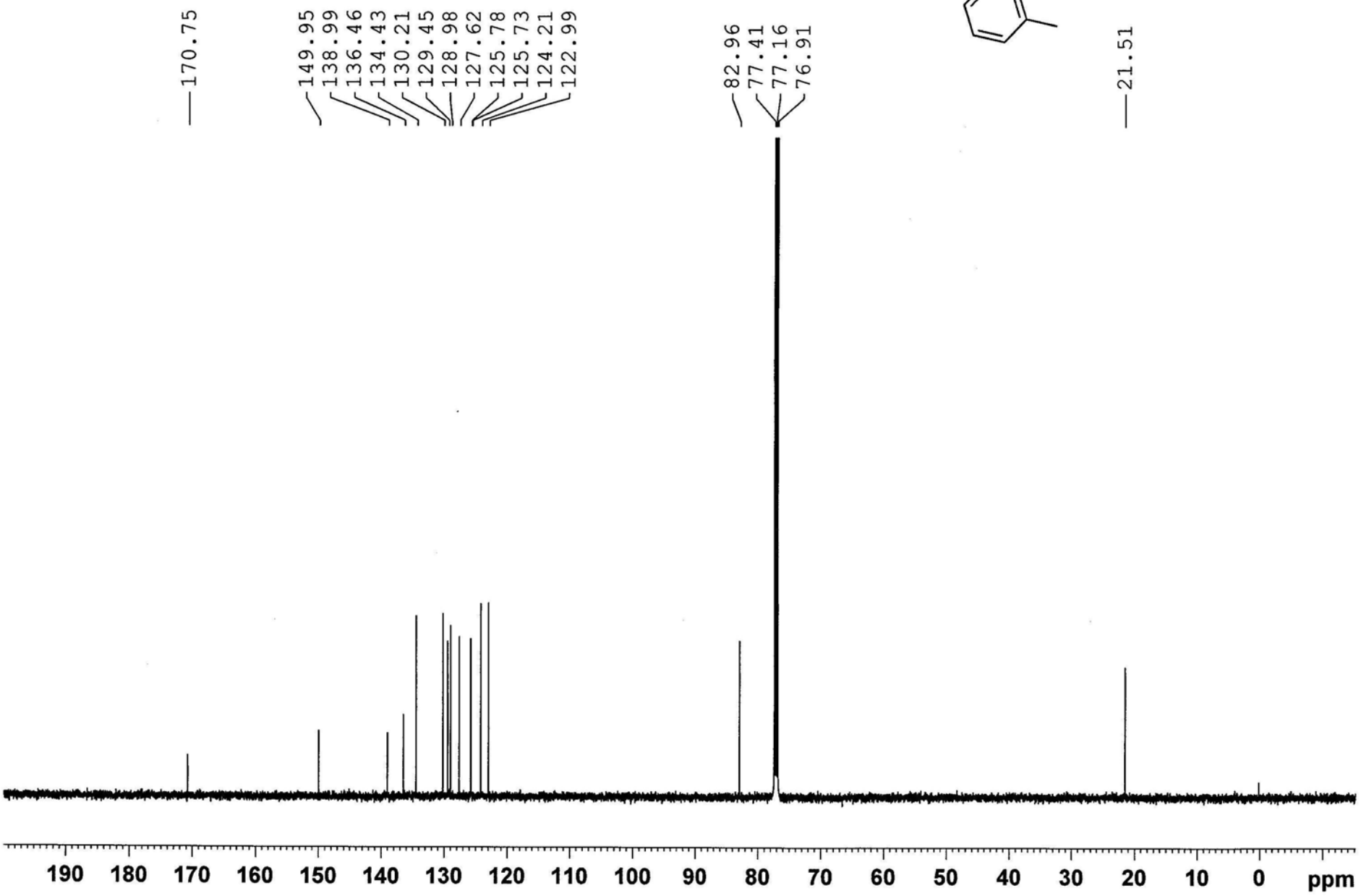
3c ^{1}H NMR

3c ^{13}C NMR

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134.43
130.21
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128.98
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125.78
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122.99
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21.51
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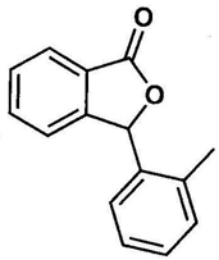
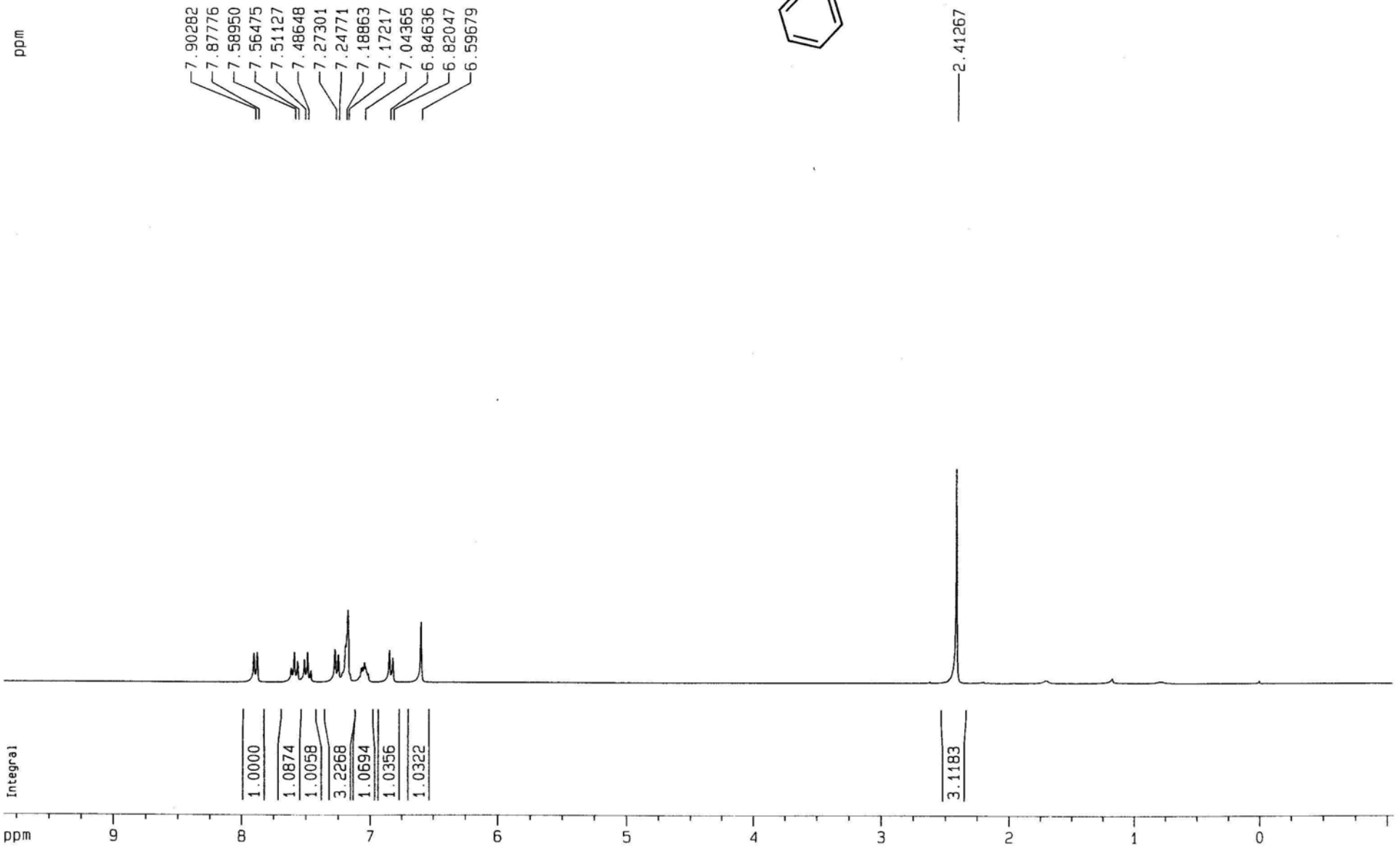
3d ^{1}H NMR

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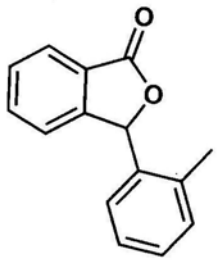
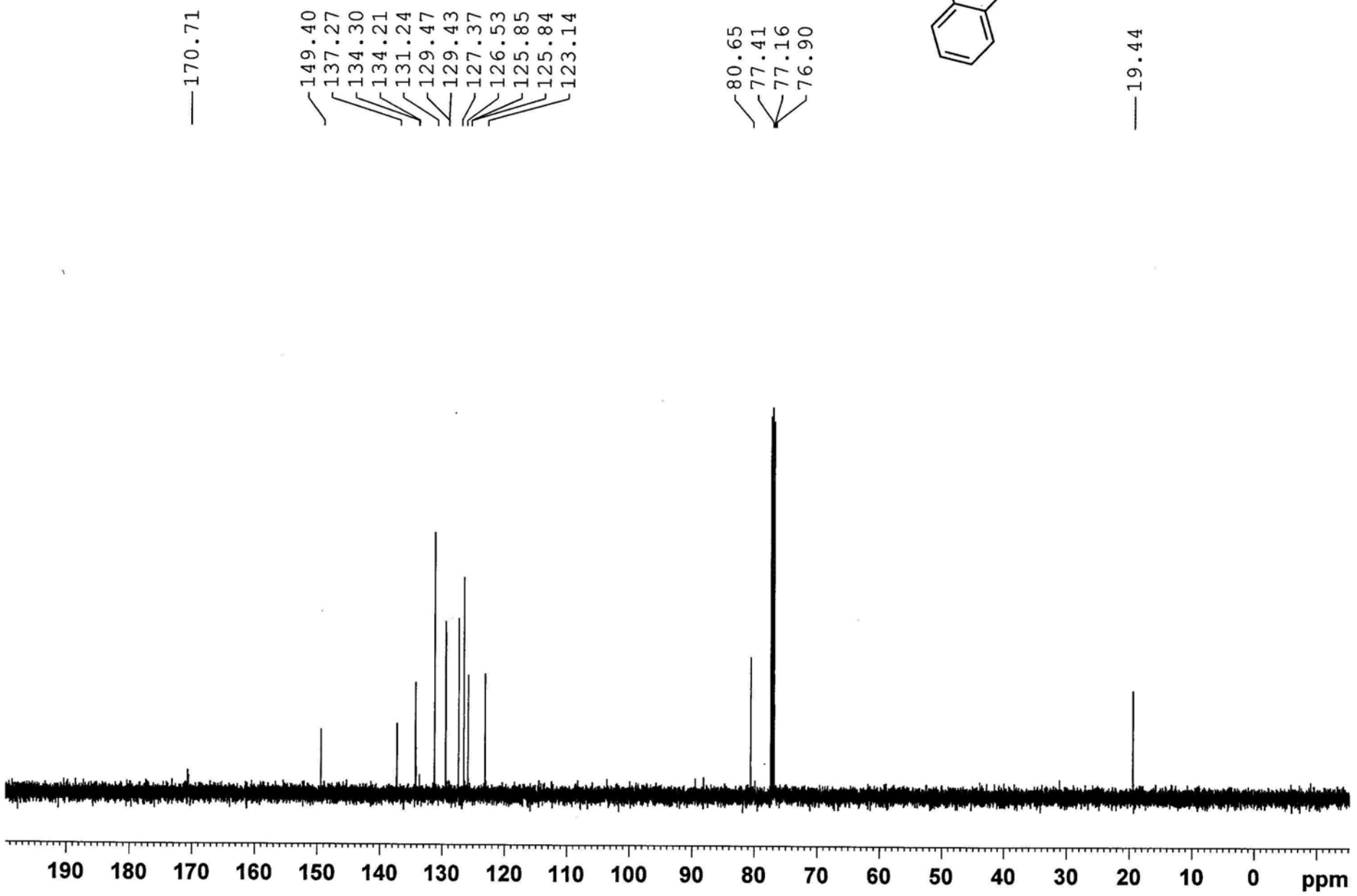
3d ^{13}C NMR

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134.30
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125.84
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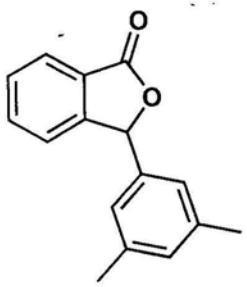
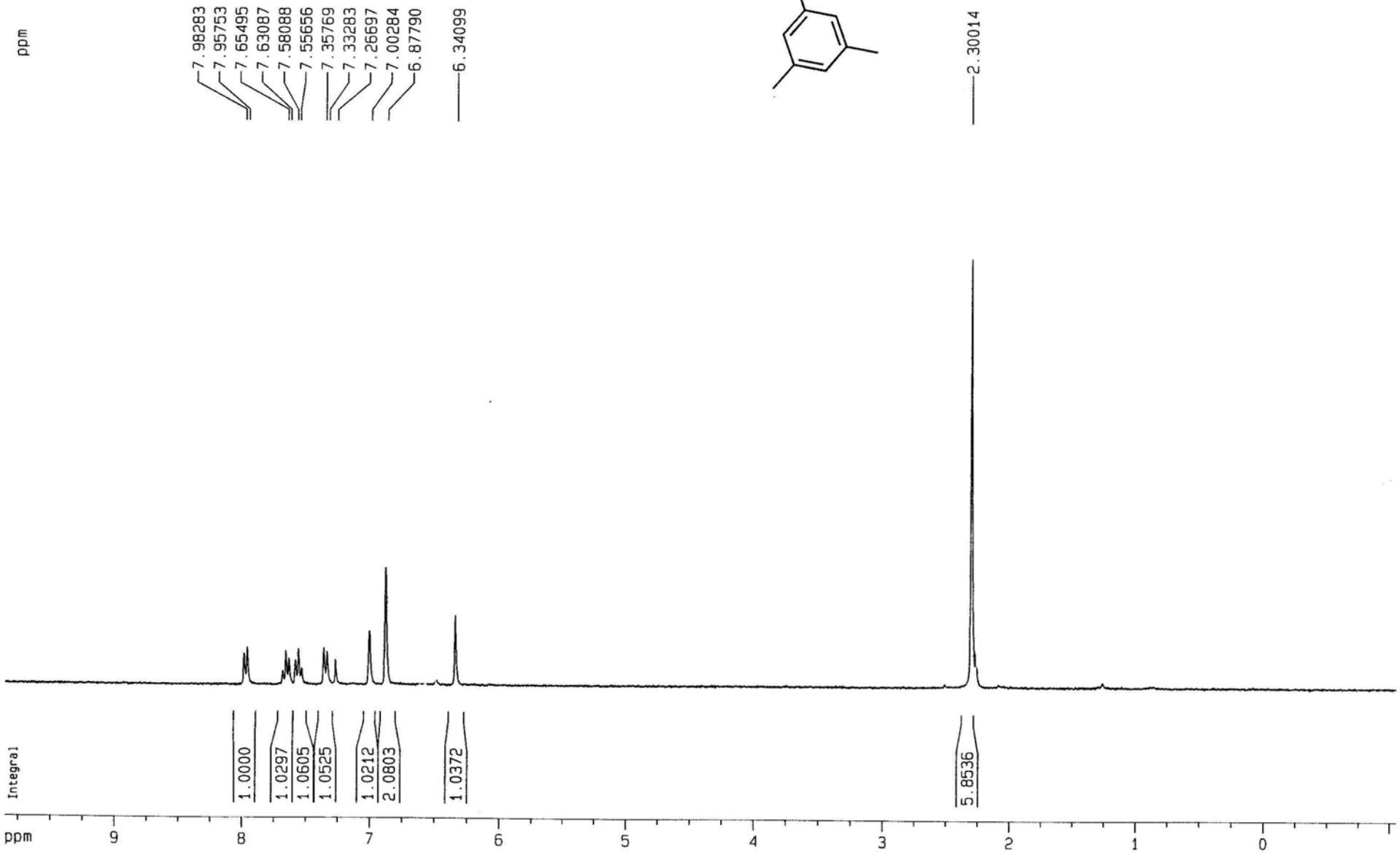
76.90
19.44
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

3e ^{1}H NMR

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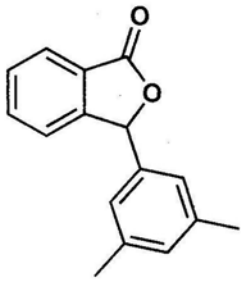
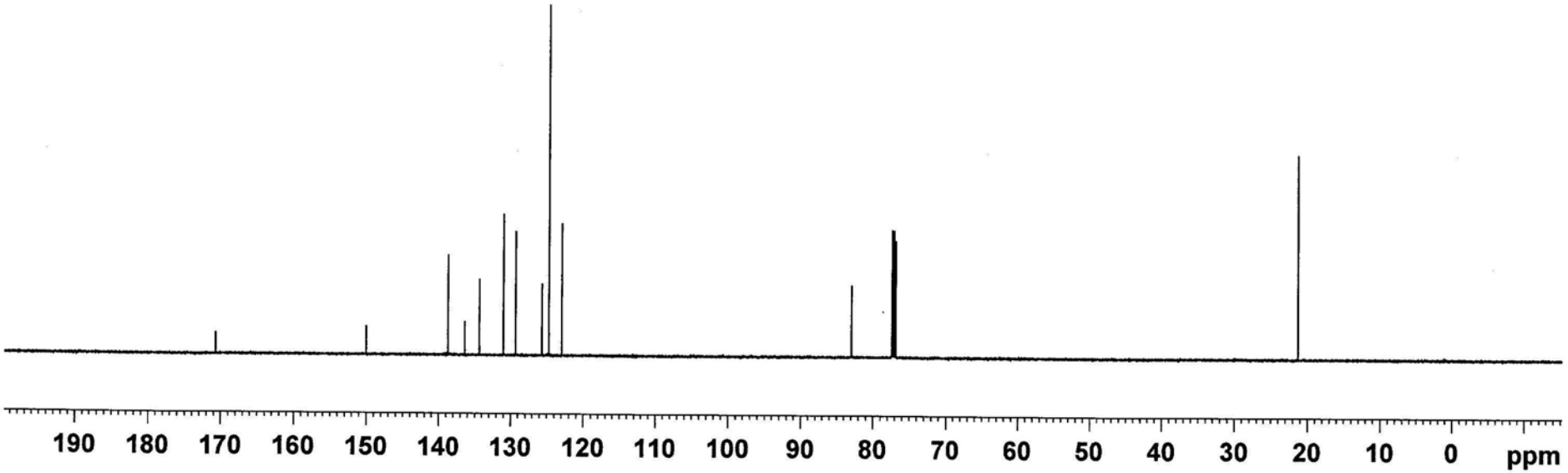
ppm
9
8
7
6
5
4
3
2
1
0

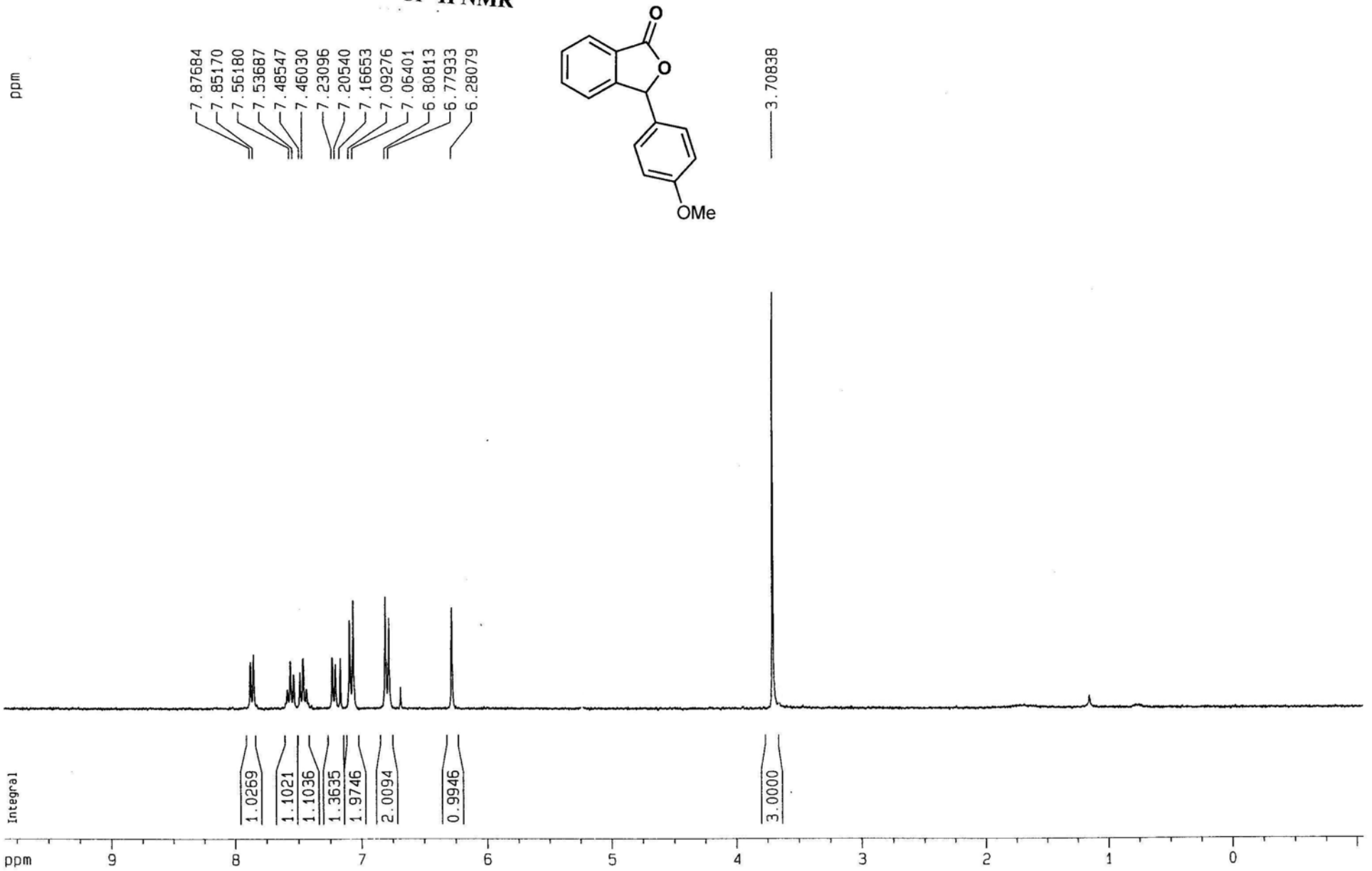
3e ^{13}C NMR



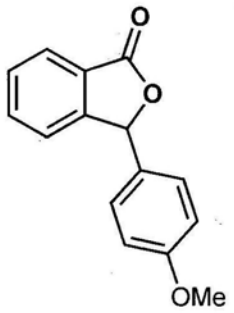
170.73
150.02
138.75
136.39
134.36
131.02
129.33
125.69
125.66
124.72
122.96
82.99
77.41
77.16
76.90
21.32

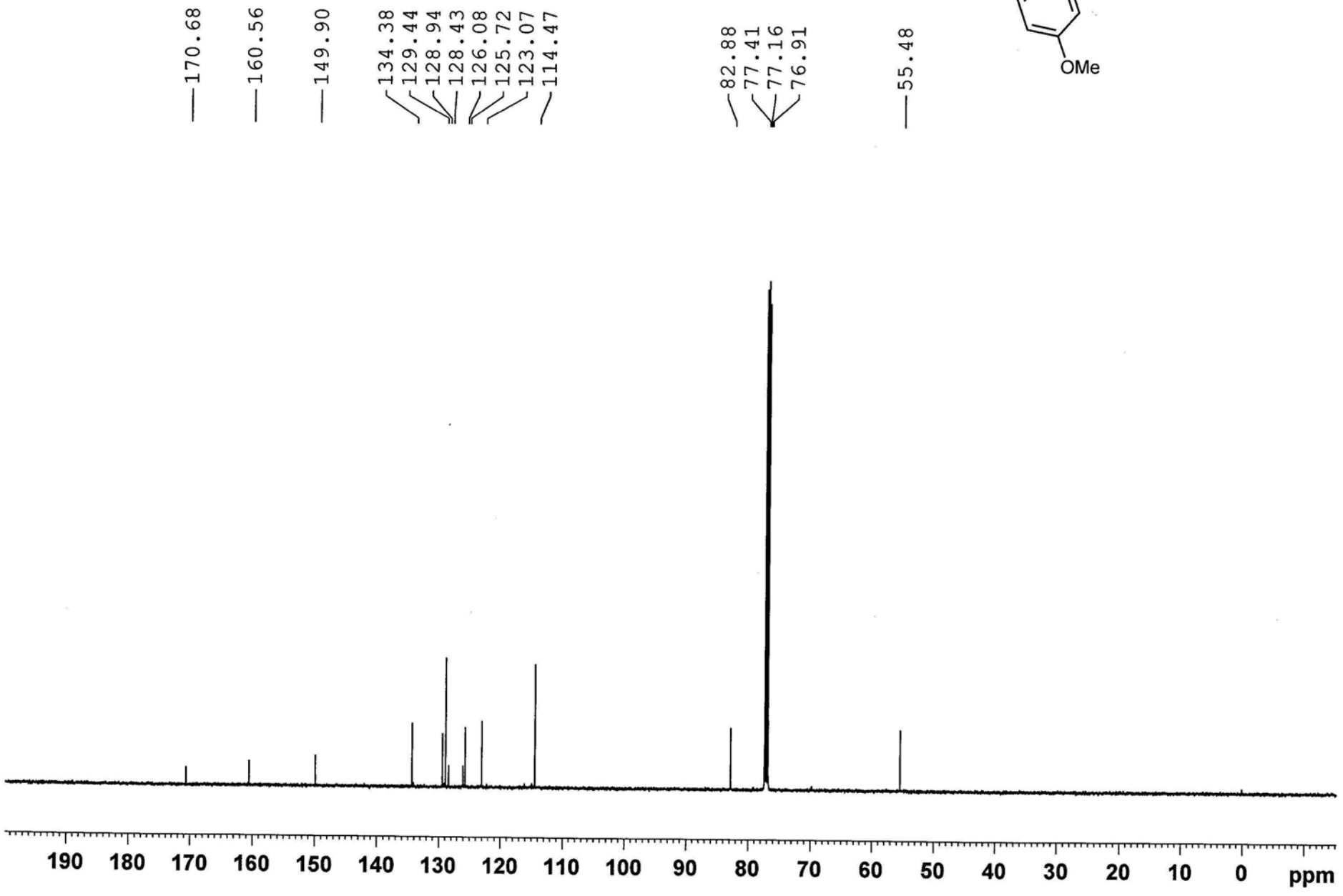


3f ^{1}H NMR

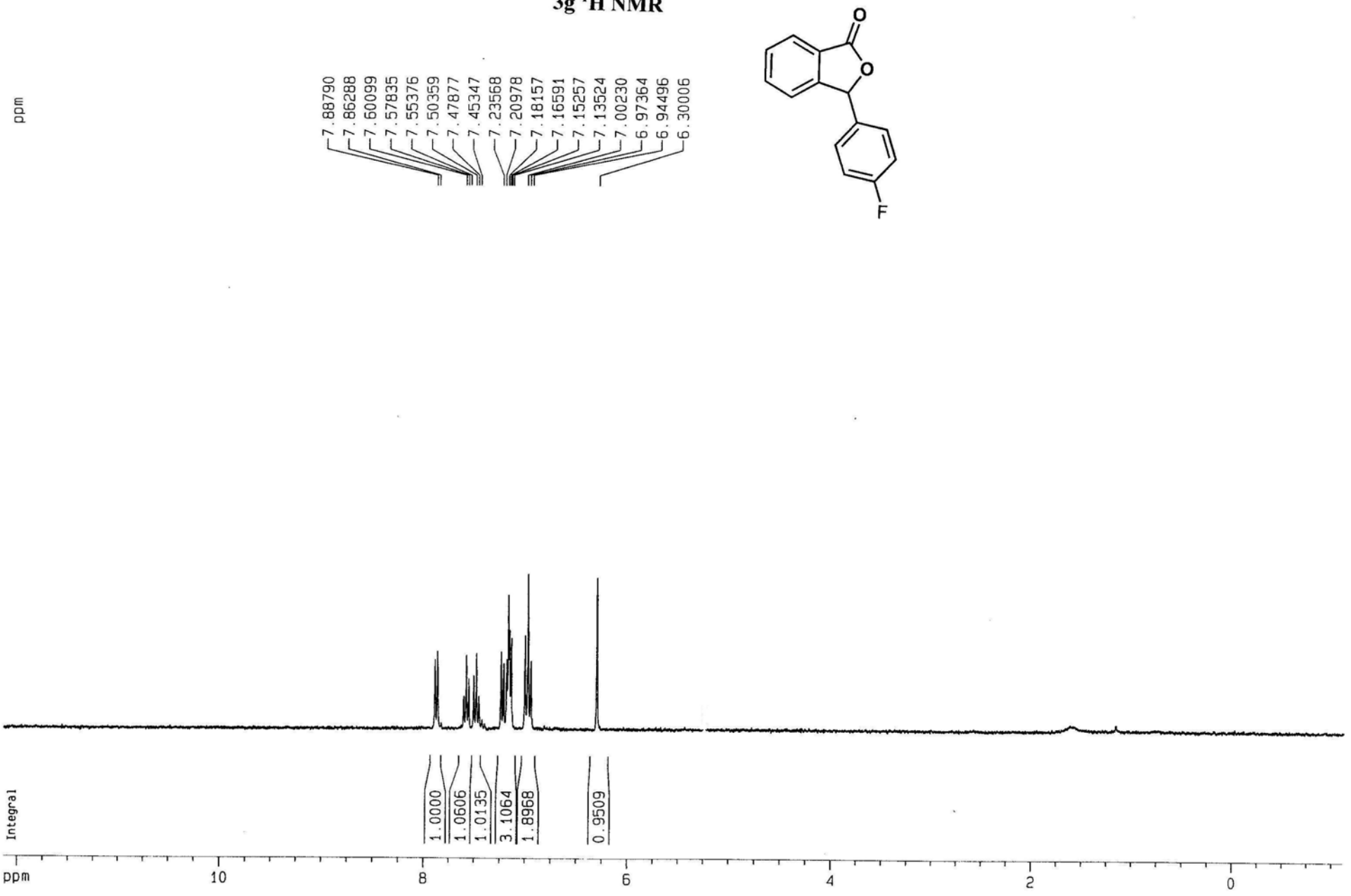

ppm
7.87684
7.85170
7.56180
7.53687
7.48547
7.46030
7.23096
7.20540
7.16653
7.09276
7.06401
6.80813
6.77933
6.28079
3.70838
O
O
OMe
Integral
1.0269
1.1021
1.1036
1.3635
1.9746
2.0094
0.9946
3.0000
ppm
9
8
7
6
5
4
3
2
1
0

3f ^{13}C NMR

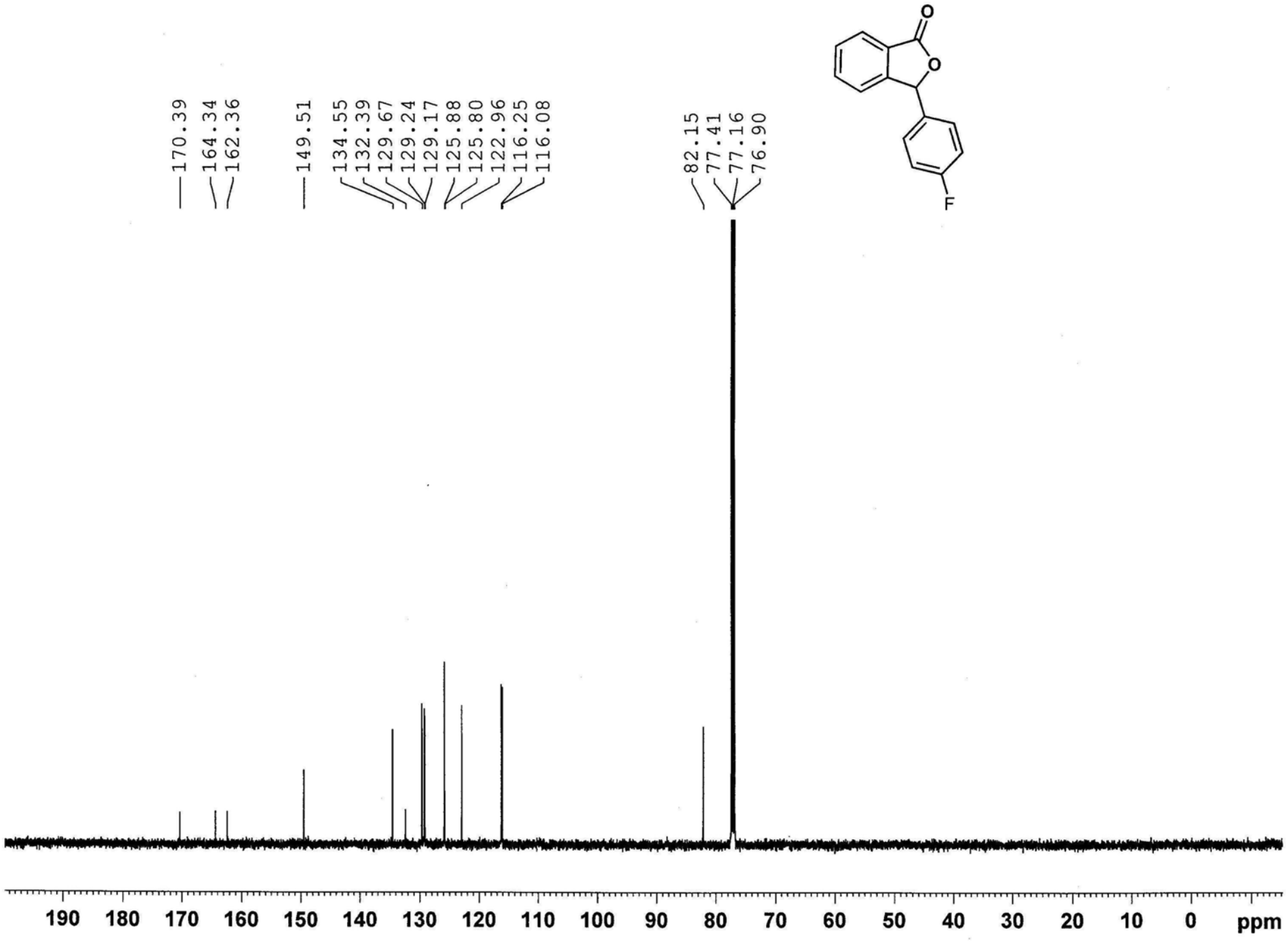

O
O
OMe


170.68
160.56
149.90
134.38
129.44
128.94
128.43
126.08
125.72
123.07
114.47
82.88
77.41
77.16
76.91
55.48
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

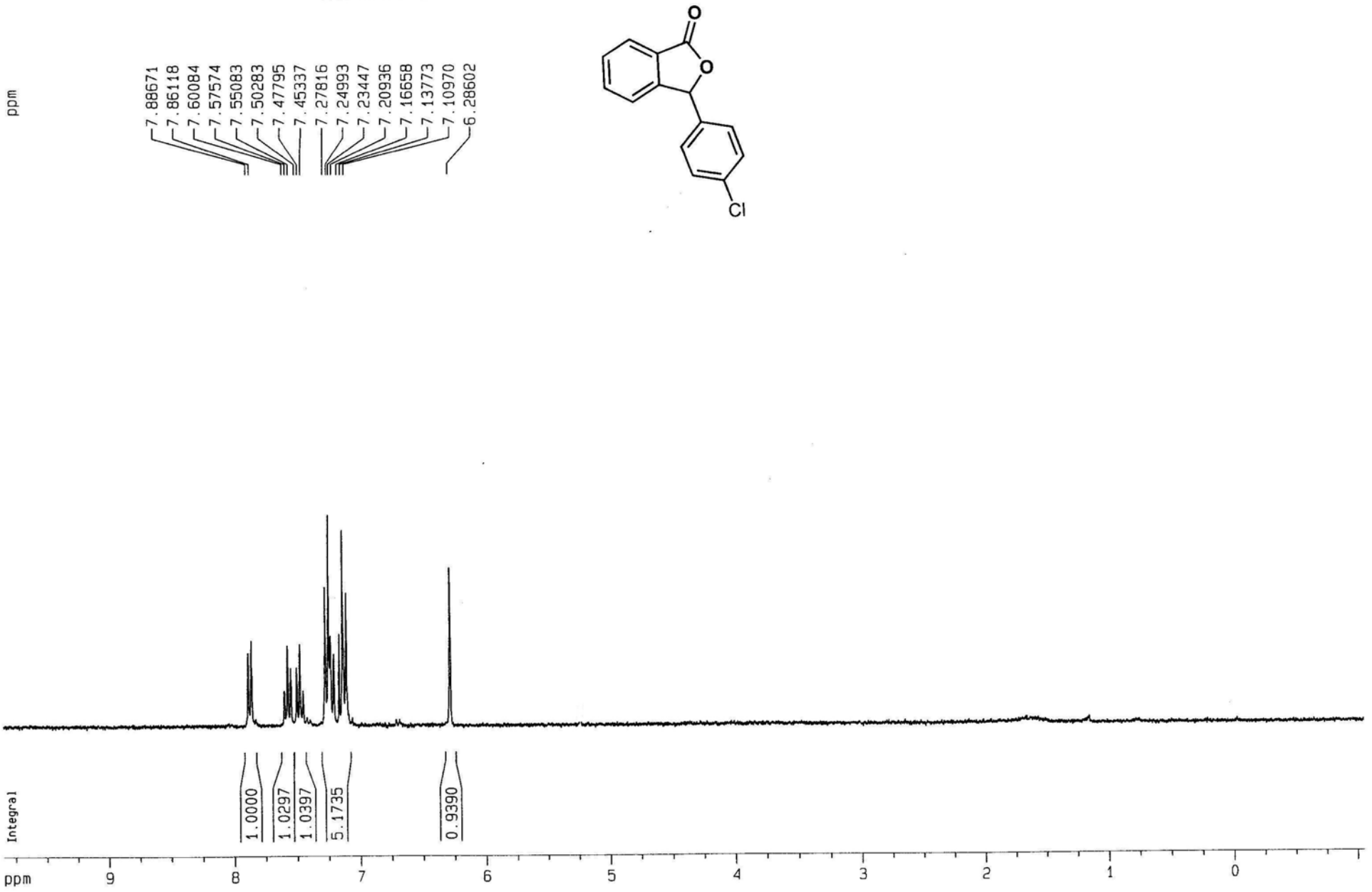
3g ^{1}H NMR


ppm
7.88790
7.86288
7.60099
7.57835
7.55376
7.50359
7.47877
7.45347
7.23568
7.20978
7.18157
7.16591
7.15257
7.13524
7.00230
6.97364
6.94496
6.30006
O
O
F
Integral
1.0000
1.0606
1.0135
3.1064
1.8968
0.9509
ppm
10
8
6
4
2
0

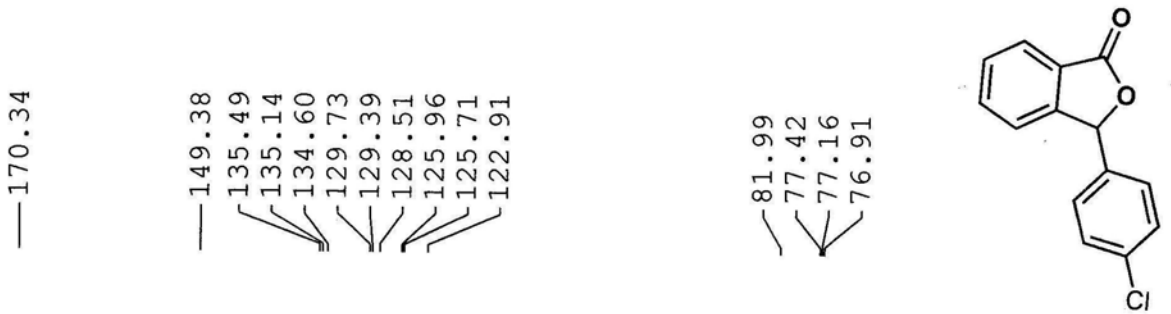
3g ^{13}C NMR

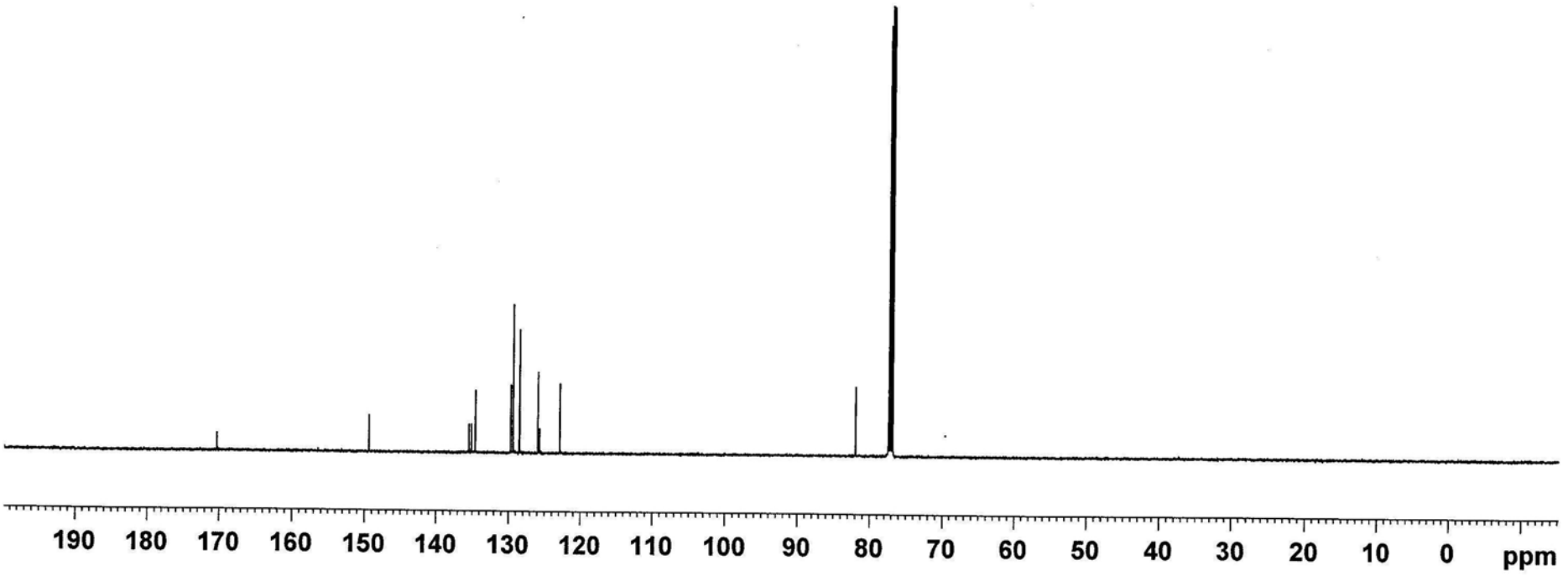

170.39
164.34
162.36
149.51
134.55
132.39
129.67
129.24
129.17
125.88
125.80
122.96
116.25
116.08
82.15
77.41
77.16
76.90
O
O
F
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

3h ^{1}H NMR

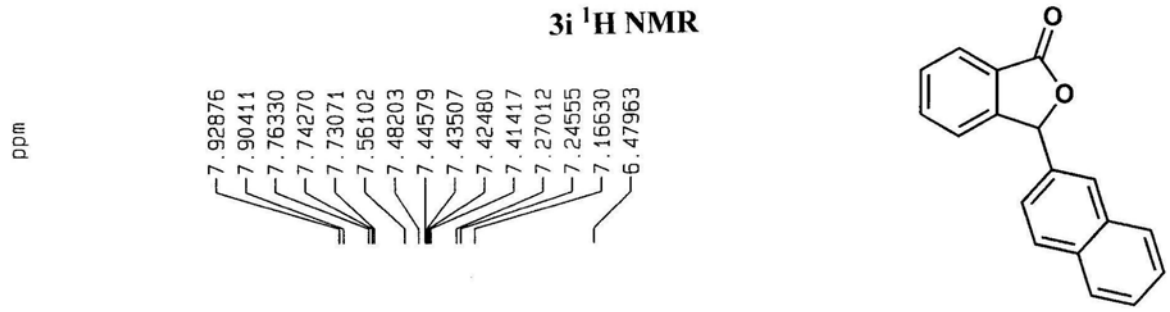

ppm
7.88671
7.86118
7.60084
7.57574
7.55083
7.50283
7.47795
7.45337
7.27816
7.24993
7.23447
7.20936
7.16658
7.13773
7.10970
6.28602
O
O
Cl
Integral
1.0000
1.0297
1.0397
5.1735
0.9390
ppm
9
8
7
6
5
4
3
2
1
0

3h ^{13}C NMR

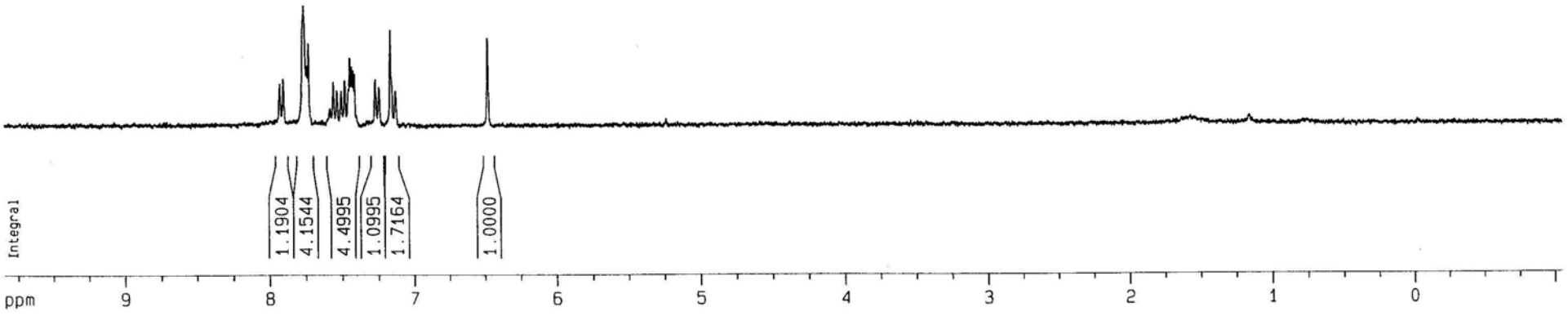

170.34
149.38
135.49
135.14
134.60
129.73
129.39
128.51
125.96
125.71
122.91
81.99
77.42
77.16
76.91
O
O
Cl


190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

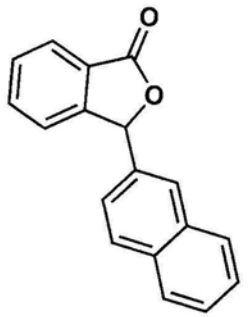
3i ^{1}H NMR

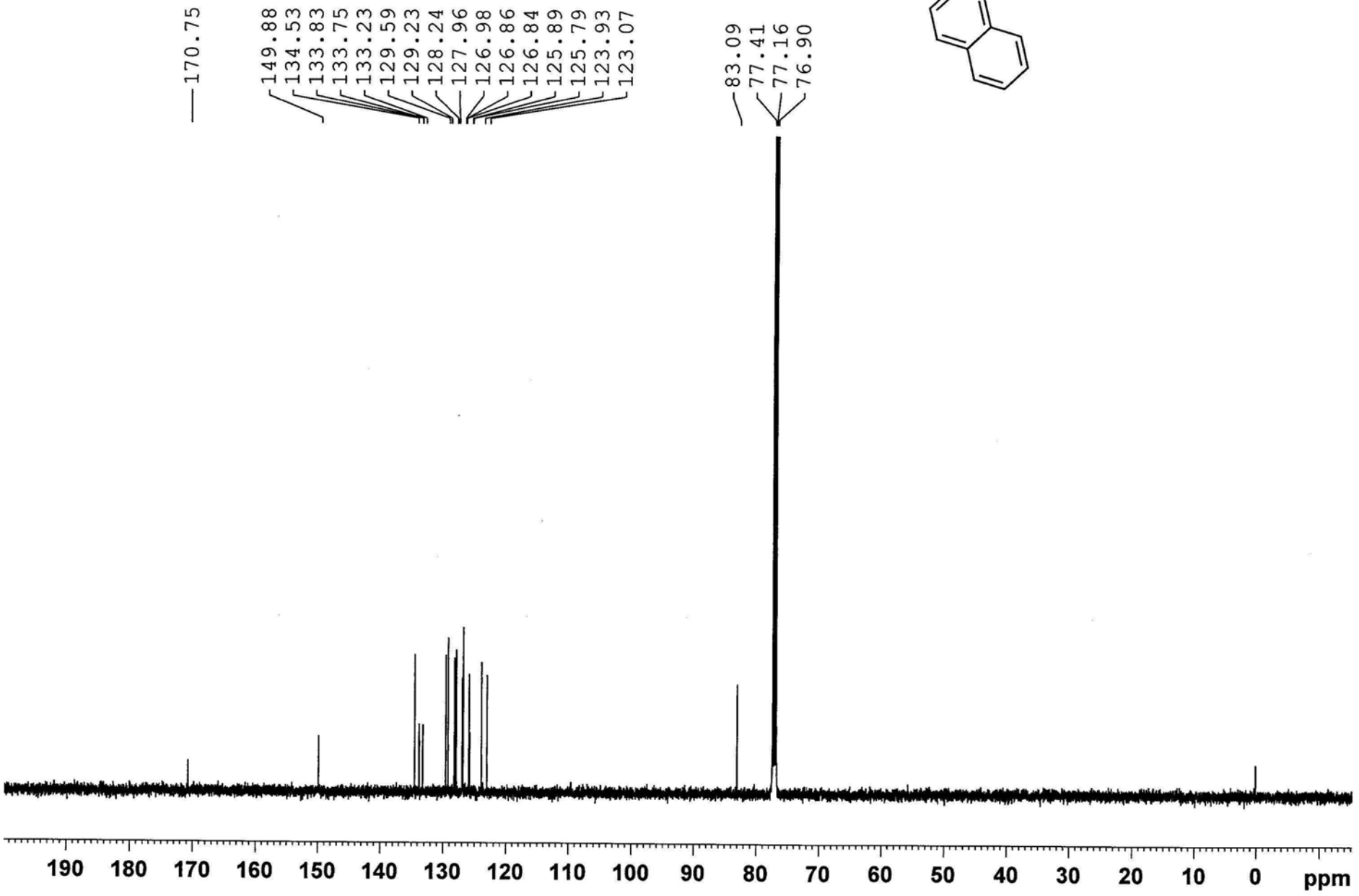

ppm
7.92876
7.90411
7.76330
7.74270
7.73071
7.56102
7.48203
7.44579
7.43507
7.42480
7.41417
7.27012
7.24555
7.16630
6.47963

O
O

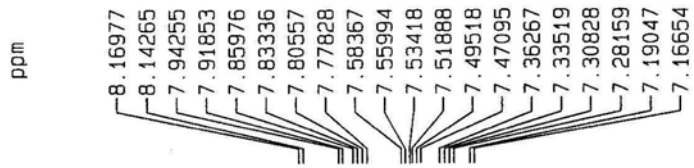

Integral
1.1904
4.1544
4.4995
1.0995
1.7164
1.0000
ppm
9
8
7
6
5
4
3
2
1
0

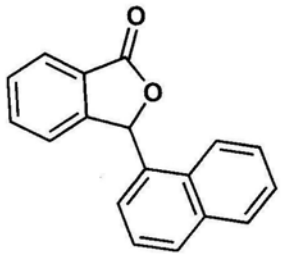
3i ^{13}C NMR

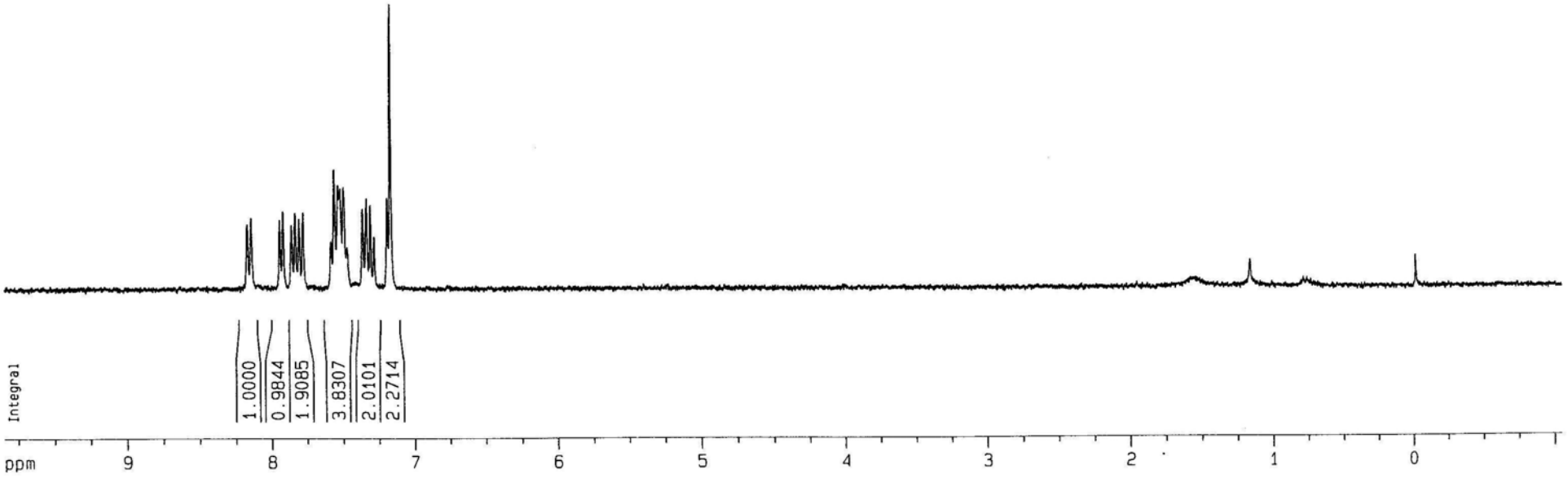



170.75
149.88
134.53
133.83
133.75
133.23
129.59
129.23
128.24
127.96
126.98
126.86
126.84
125.89
125.79
123.93
123.07
83.09
77.41
77.16
76.90
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

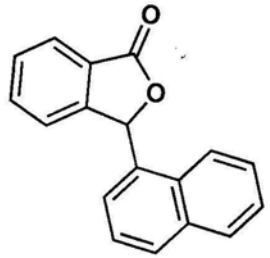
3j ^{1}H NMR

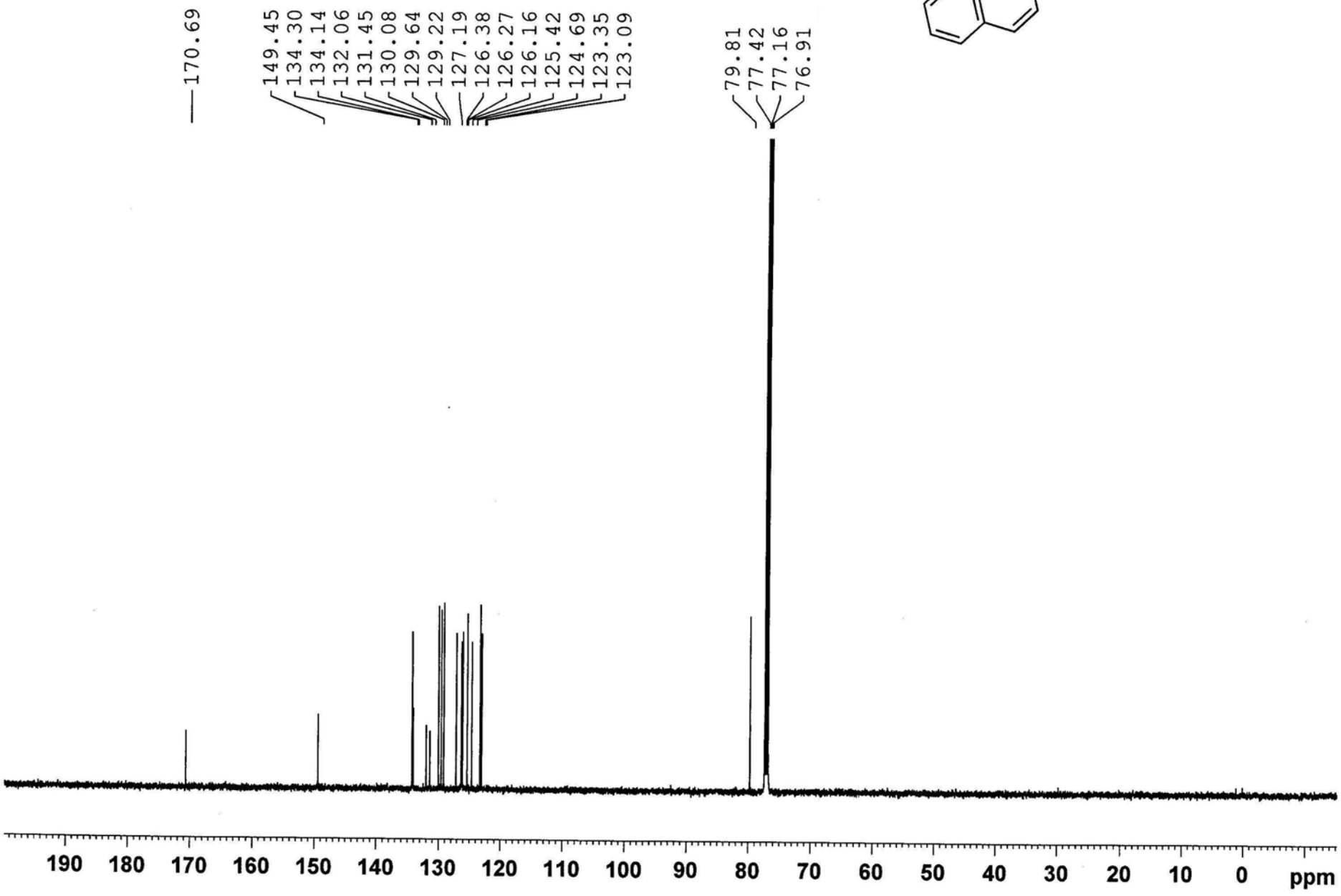

ppm
8.16977
8.14265
7.94255
7.91853
7.85976
7.83336
7.80557
7.77828
7.58367
7.55994
7.53418
7.51888
7.49518
7.47095
7.36267
7.33519
7.30828
7.28159
7.19047
7.16654


O
O

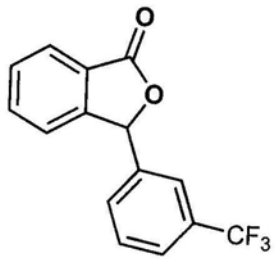

Integral
1.0000
0.9844
1.9085
3.8307
2.0101
2.2714
ppm
9
8
7
6
5
4
3
2
1
0

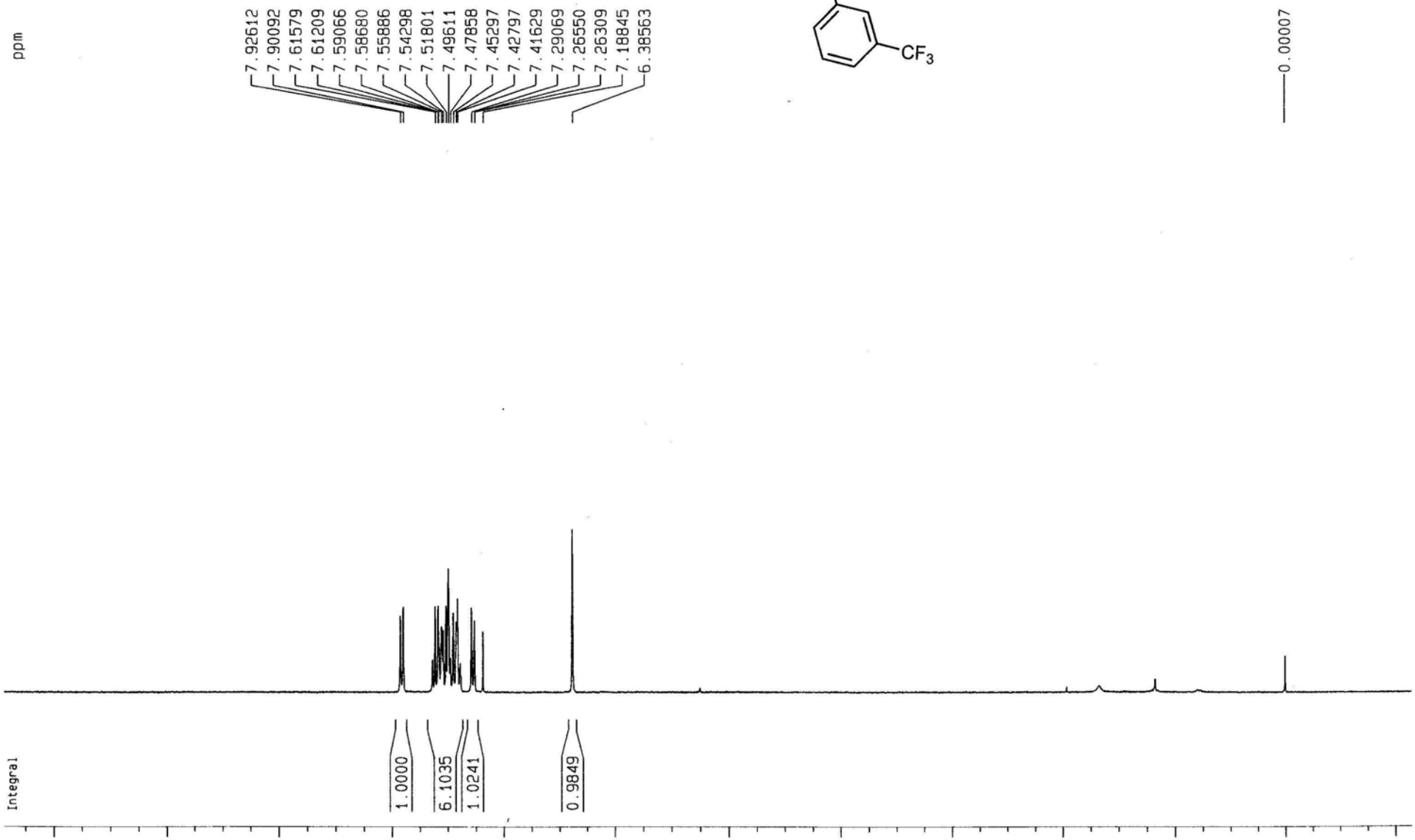
3j ^{13}C NMR



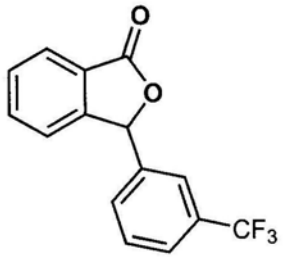

170.69
149.45
134.30
134.14
132.06
131.45
130.08
129.64
129.22
127.19
126.38
126.27
126.16
125.42
124.69
123.35
123.09
79.81
77.42
77.16
76.91
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm

3k ^{1}H NMR

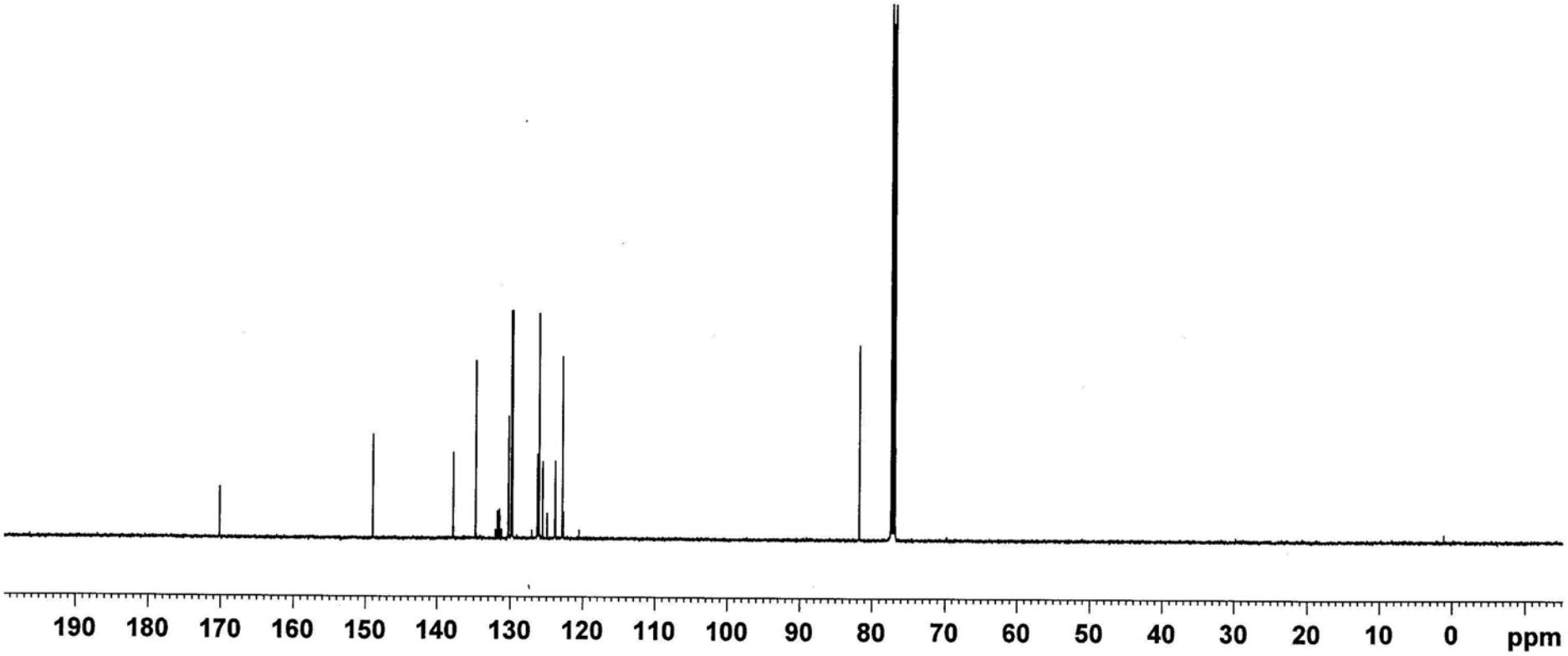

O
O
CF3


ppm
7.92612
7.90092
7.61579
7.61209
7.59066
7.58680
7.55886
7.54298
7.51801
7.49611
7.47858
7.45297
7.42797
7.41629
7.29069
7.26550
7.26309
7.18845
6.38563
0.00007
Integral
1.0000
6.1035
1.0241
0.9849
ppm
10
9
8
7
6
5
4
3
2
1
0

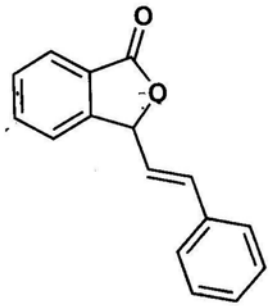
3k ^{13}C NMR

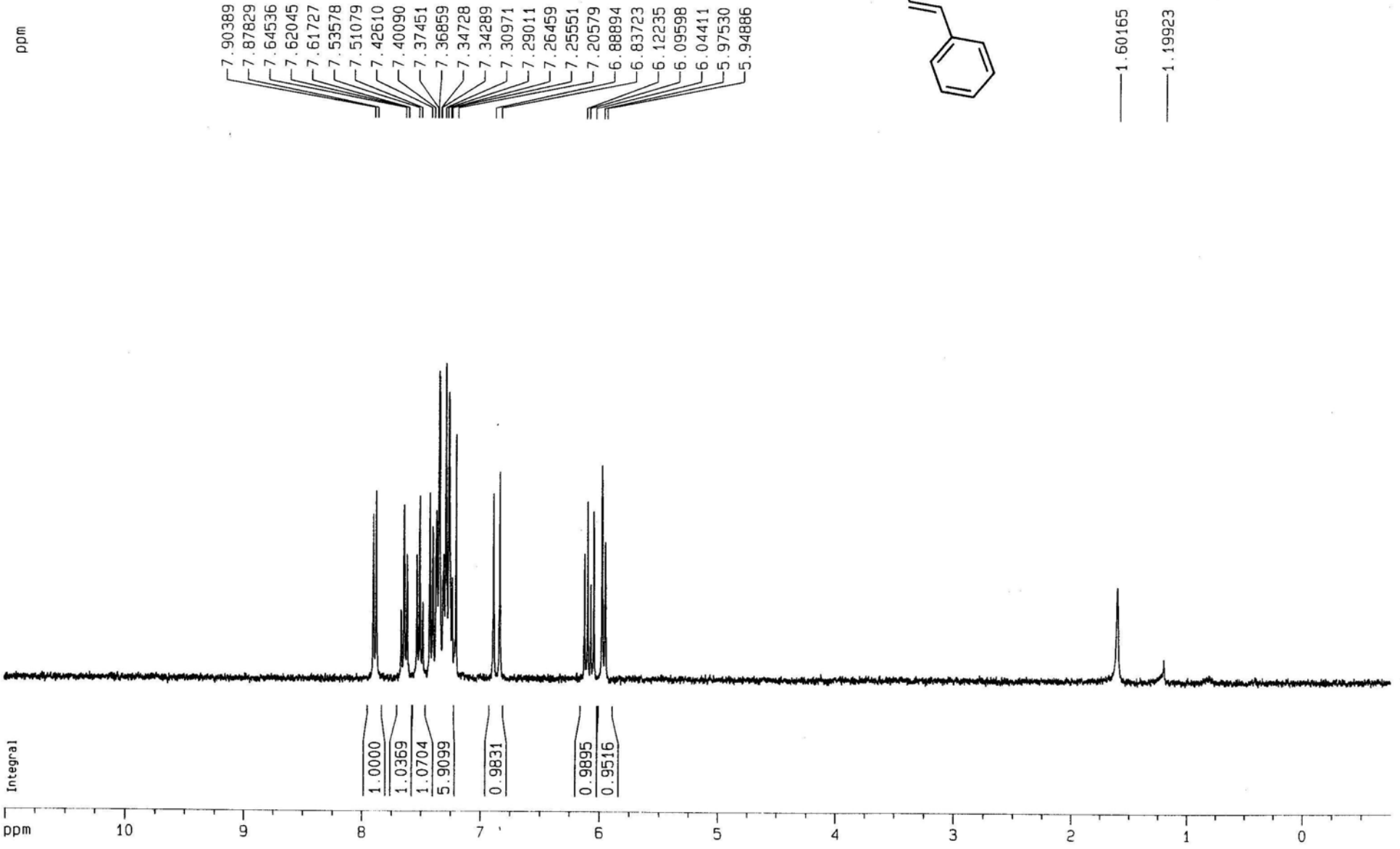


170.17
148.99
137.80
134.75
132.03
131.77
131.51
131.24
130.29
127.08
126.32
126.29
126.27
126.24
125.57
124.92
123.89
123.86
123.82
123.80
122.86
122.75
120.58
81.78
77.41
77.16
76.90

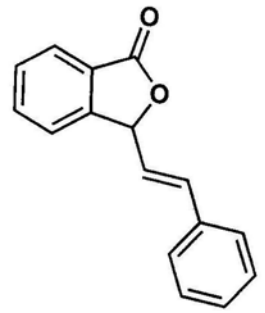


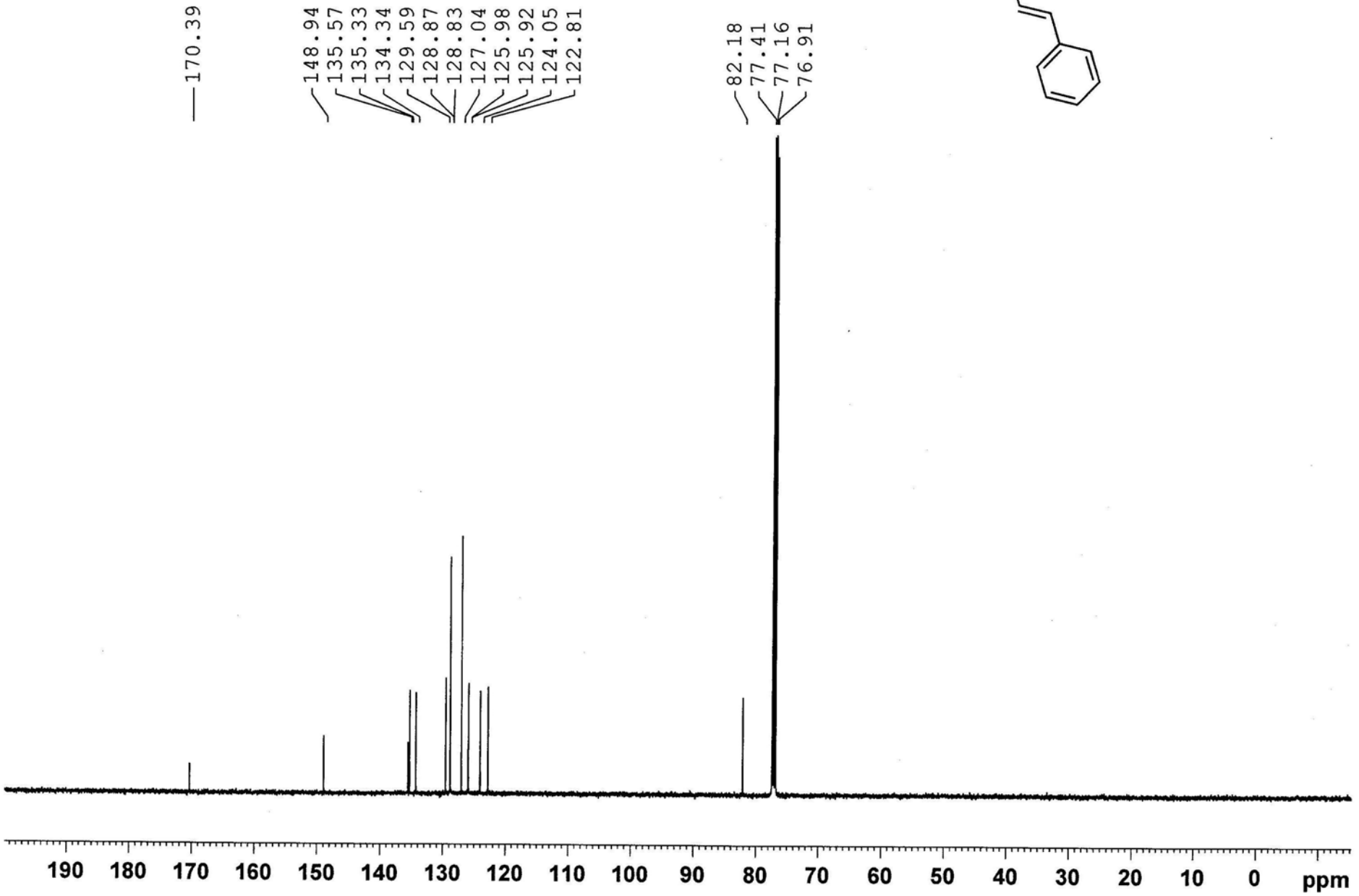
3l ^{1}H NMR




ppm
7.90389
7.87829
7.64536
7.62045
7.61727
7.53578
7.51079
7.42610
7.40090
7.37451
7.36859
7.34728
7.34289
7.30971
7.29011
7.26459
7.25551
7.20579
6.88894
6.83723
6.12235
6.09598
6.04411
5.97530
5.94886
1.60165
1.19923
Integral
1.0000
1.0369
1.0704
5.9099
0.9831
0.9895
0.9516
ppm
10
9
8
7
6
5
4
3
2
1
0

3l ^{13}C NMR




170.39
148.94
135.57
135.33
134.34
129.59
128.87
128.83
127.04
125.98
125.92
124.05
122.81
82.18
77.41
77.16
76.91
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm