Supplementary Information

Pd(II)-Catalyzed Cycloisomerization/Dipolar Cycloaddition Cascade of *N*-Arylnitrone Alkynes with Olefins

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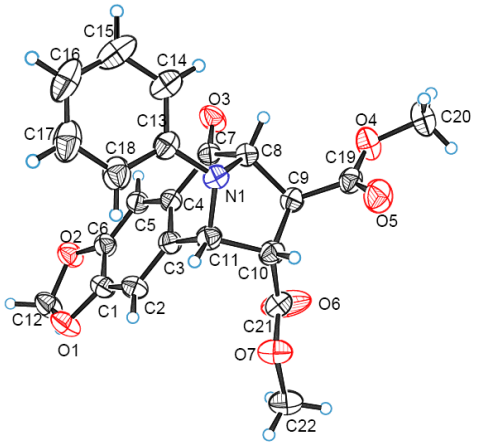
I. X-ray single crystal data for 3n.



Empirical formula	$C_{45}H_{40}Cl_2N_2O_{14}$
Formula weight	903.69
Temperature/K	291(2)
Crystal system	monoclinic
Space group	C2/c
a/Å	16.7819(4)
b/Å	9.7325(3)
c/Å	25.9139(8)
α/°	90
β/°	100.705(3)
γ/°	90
Volume/Å^3	4158.9(2)
Z	4
ρ_{calc}g/cm^3	1.443
μ/mm^{-1}	2.035
F(000)	1880.0
Crystal size/mm^3	0.260 × 0.220 × 0.220
Radiation	CuKα (λ = 1.54184)
2Θ range for data collection/°	10.554 to 140.124
Index ranges	$-19 \leq h \leq 20$, $-3 \leq k \leq 11$, $-31 \leq l \leq 29$
Reflections collected	6960
Independent reflections	3745 [R_{int} = 0.0205, R_{sigma} = 0.0306]
Data/restraints/parameters	3745/0/287
Goodness-of-fit on F^2	1.088
Final R indexes [I>=2σ (I)]	R_1 = 0.0680, wR_2 = 0.1878
Final R indexes [all data]	R_1 = 0.0761, wR_2 = 0.1967
Largest diff. peak/hole / e Å^{-3}	0.64/-0.66
CCDC	1401612

II. NMR Spectra

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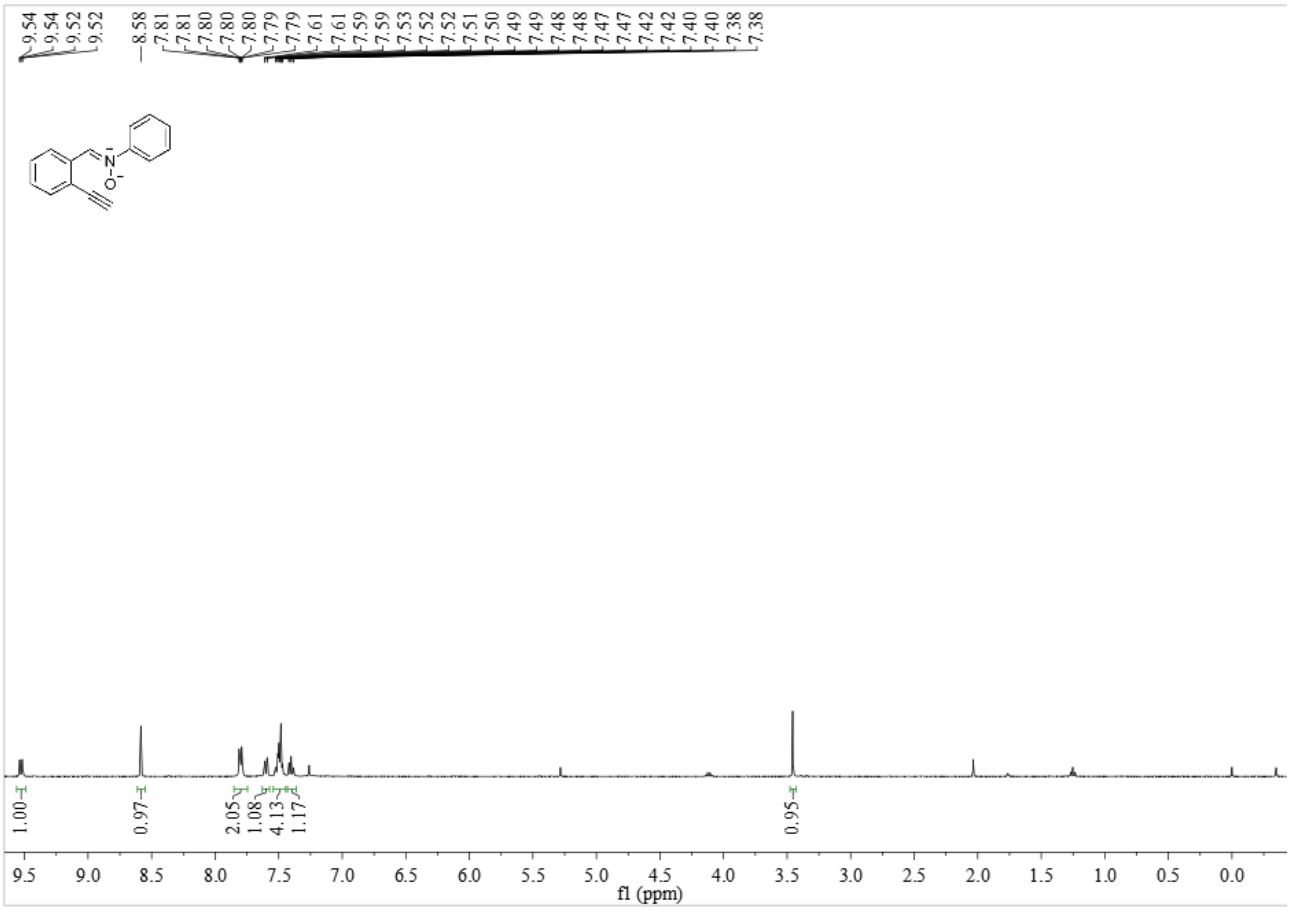

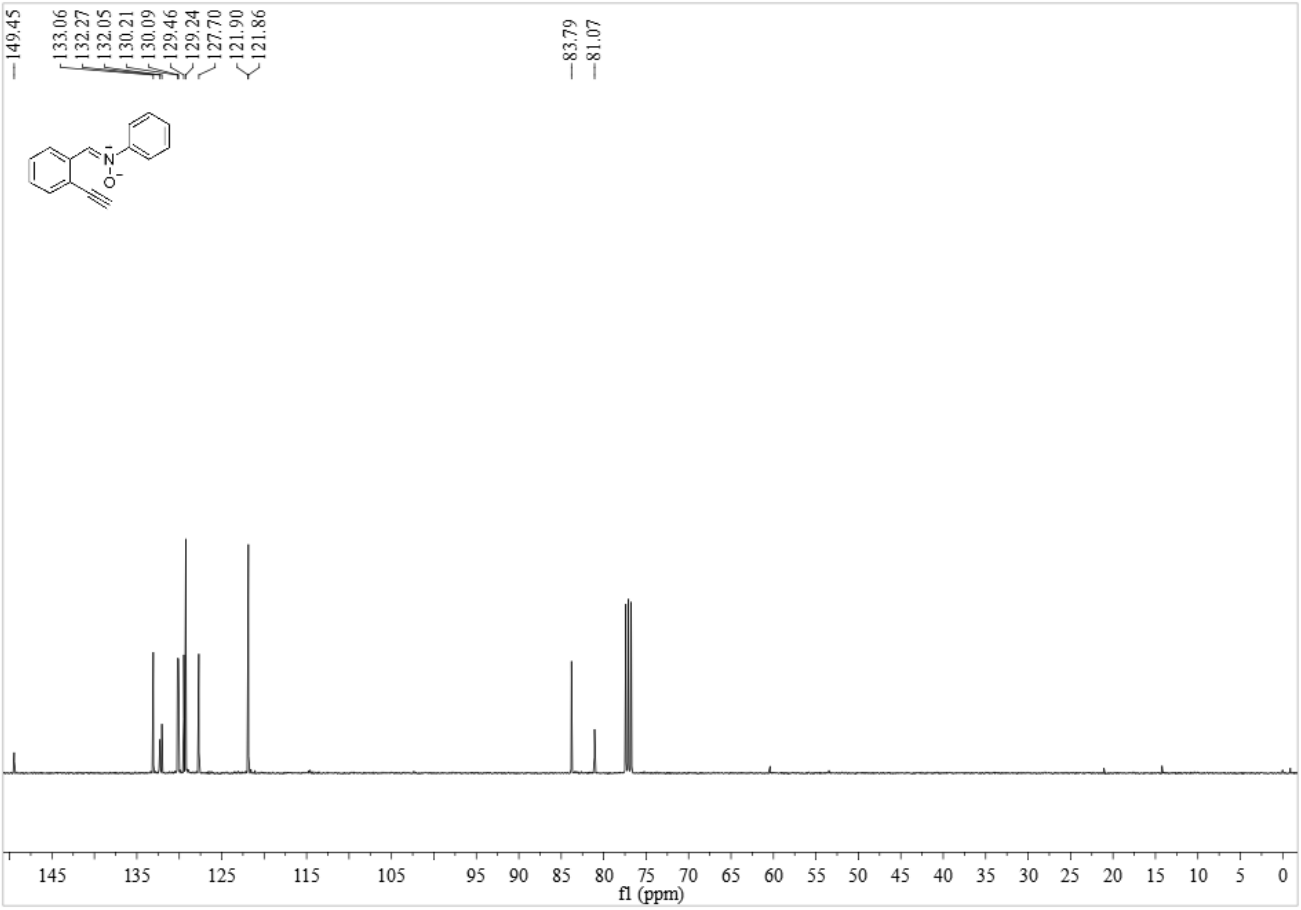

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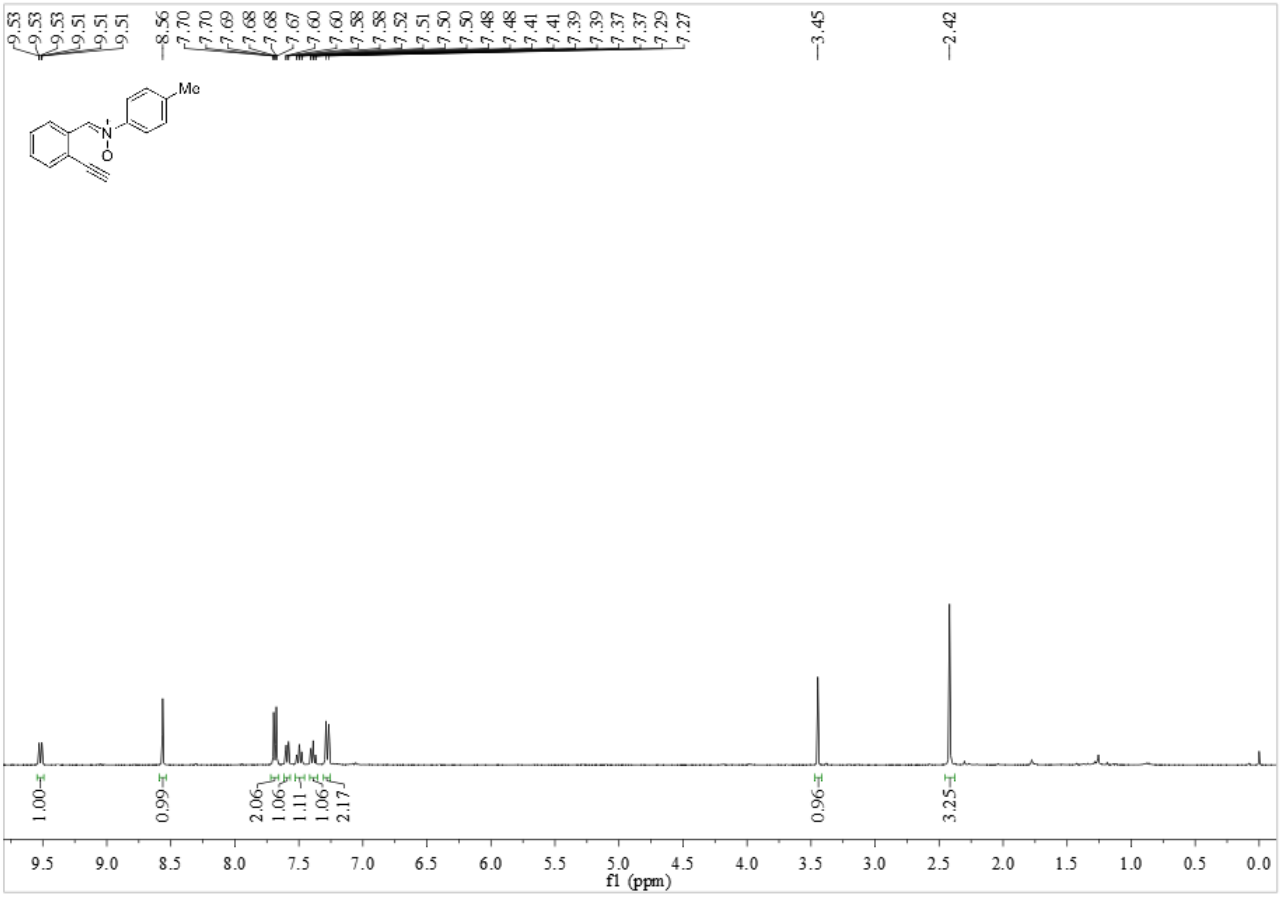
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O
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0.99
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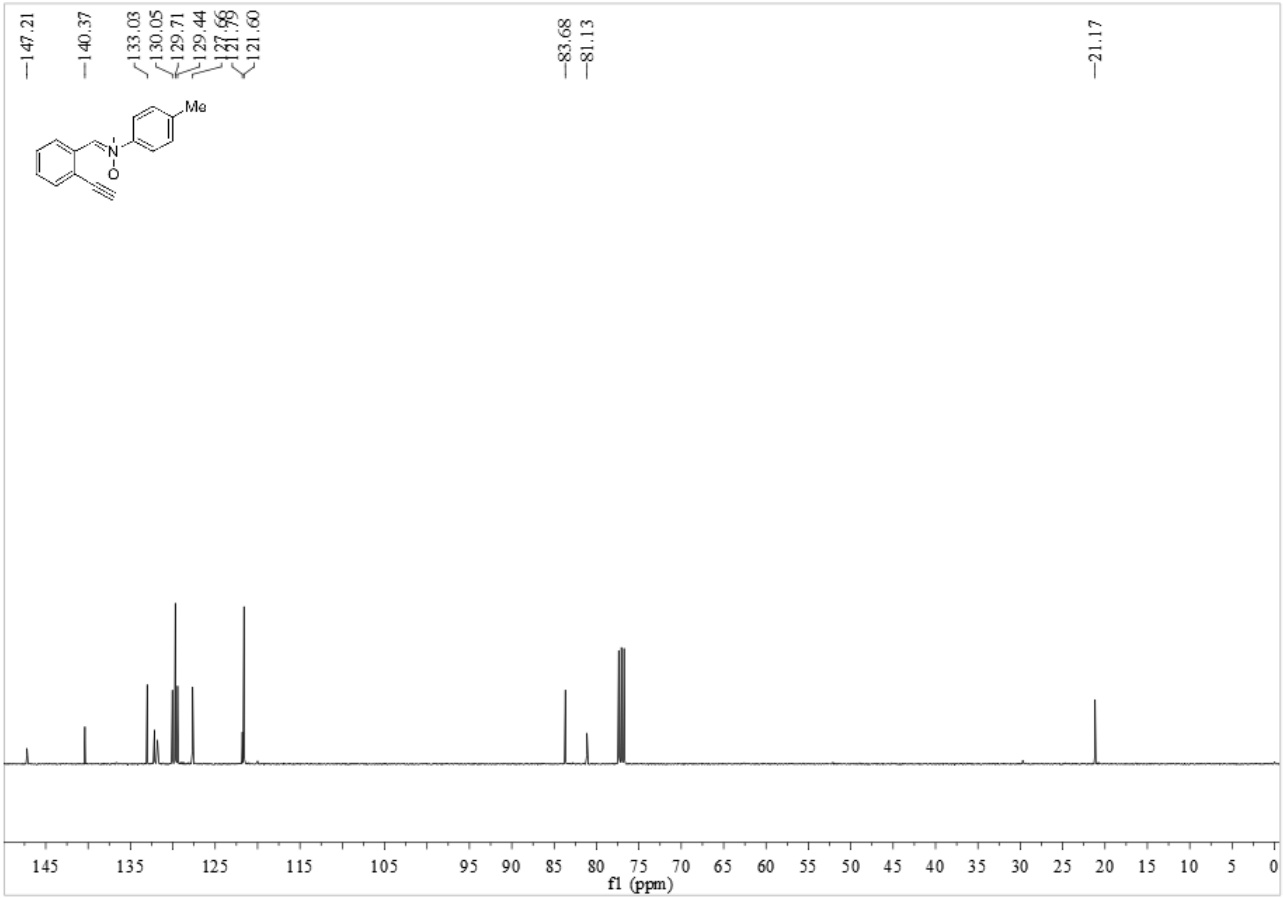
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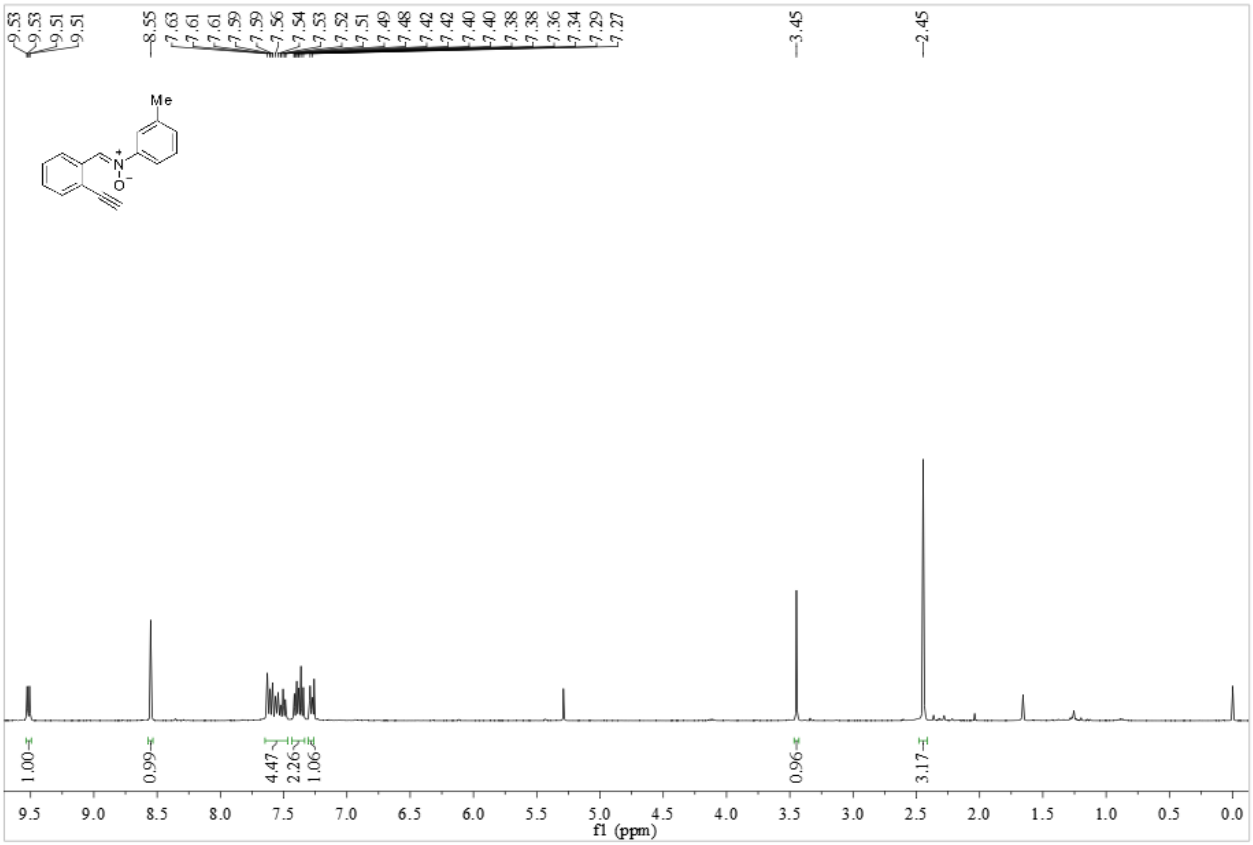
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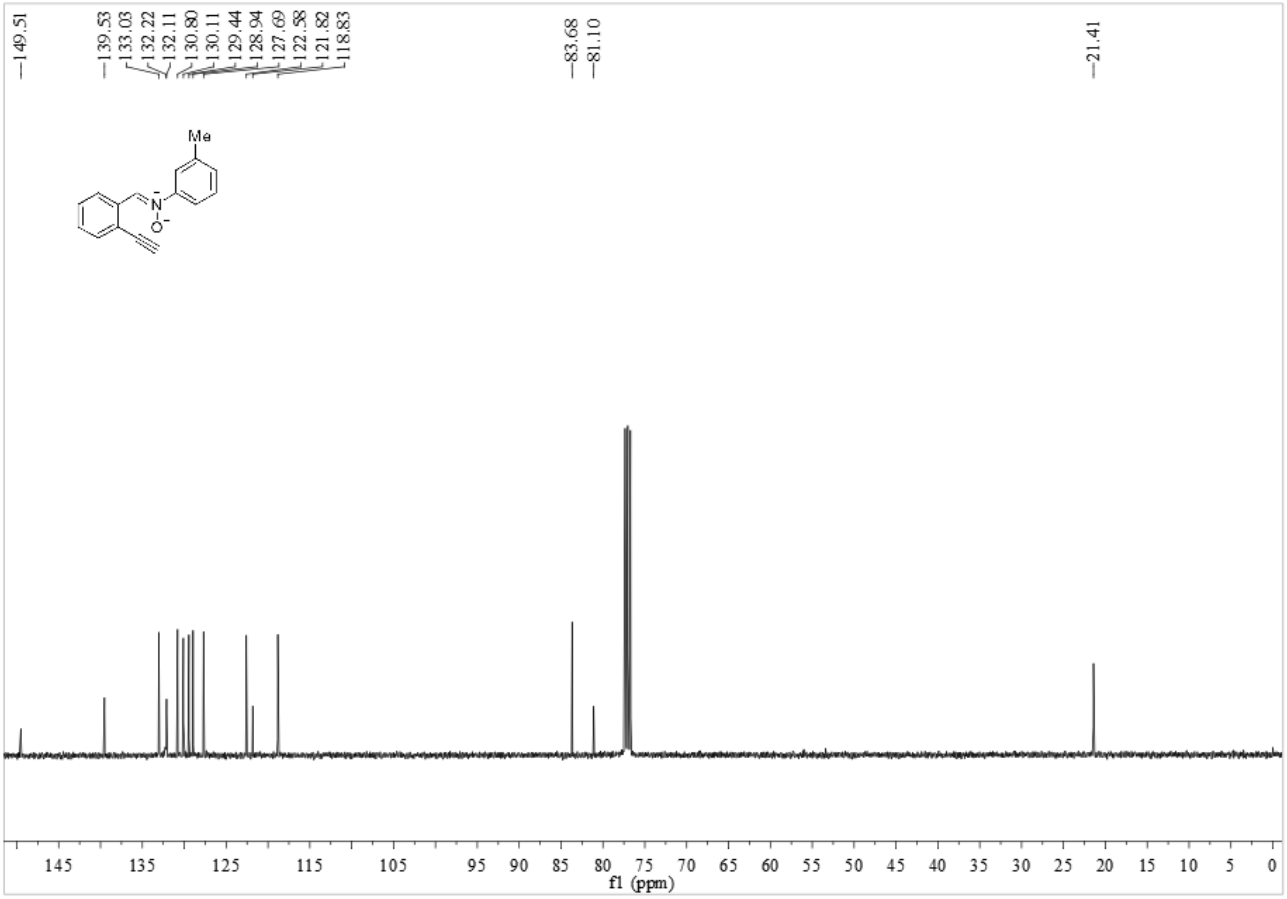
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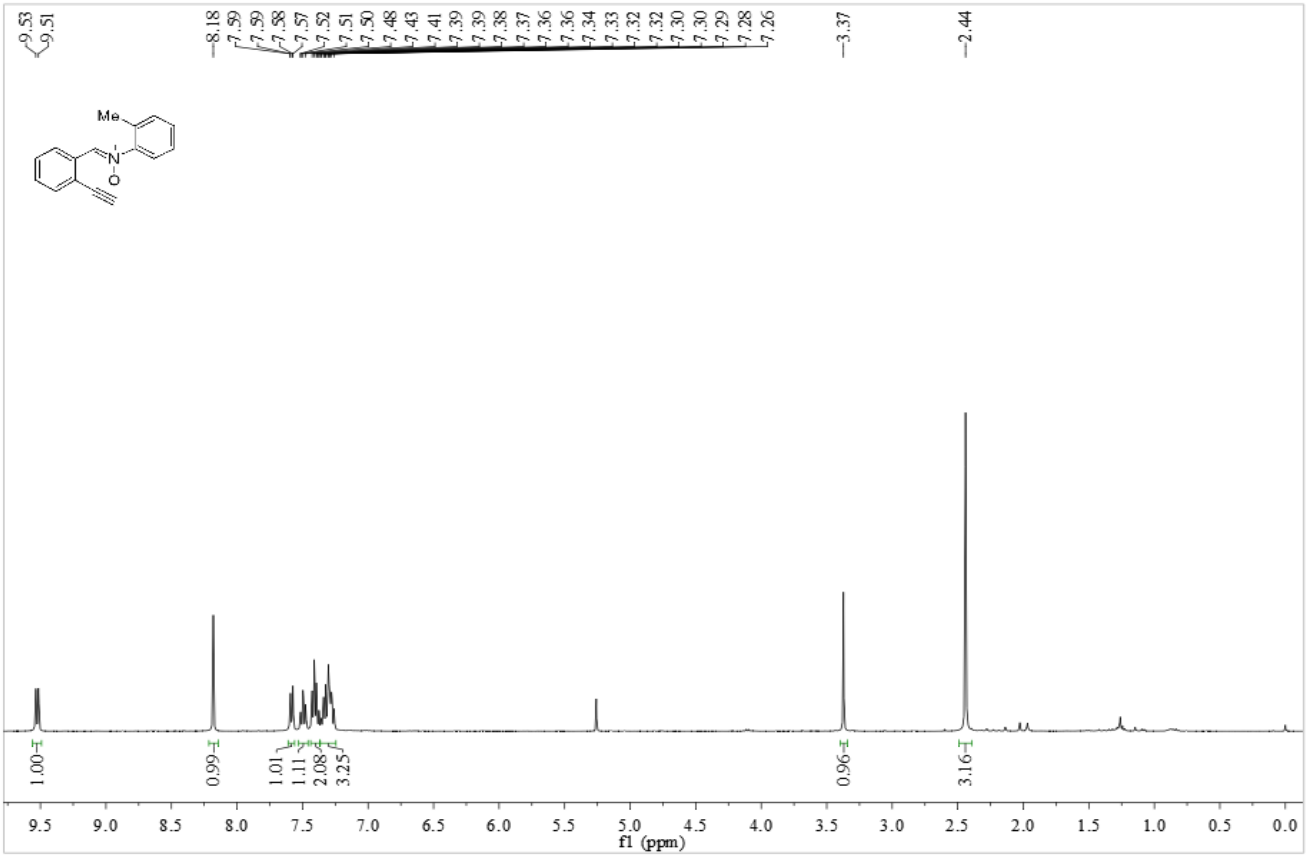
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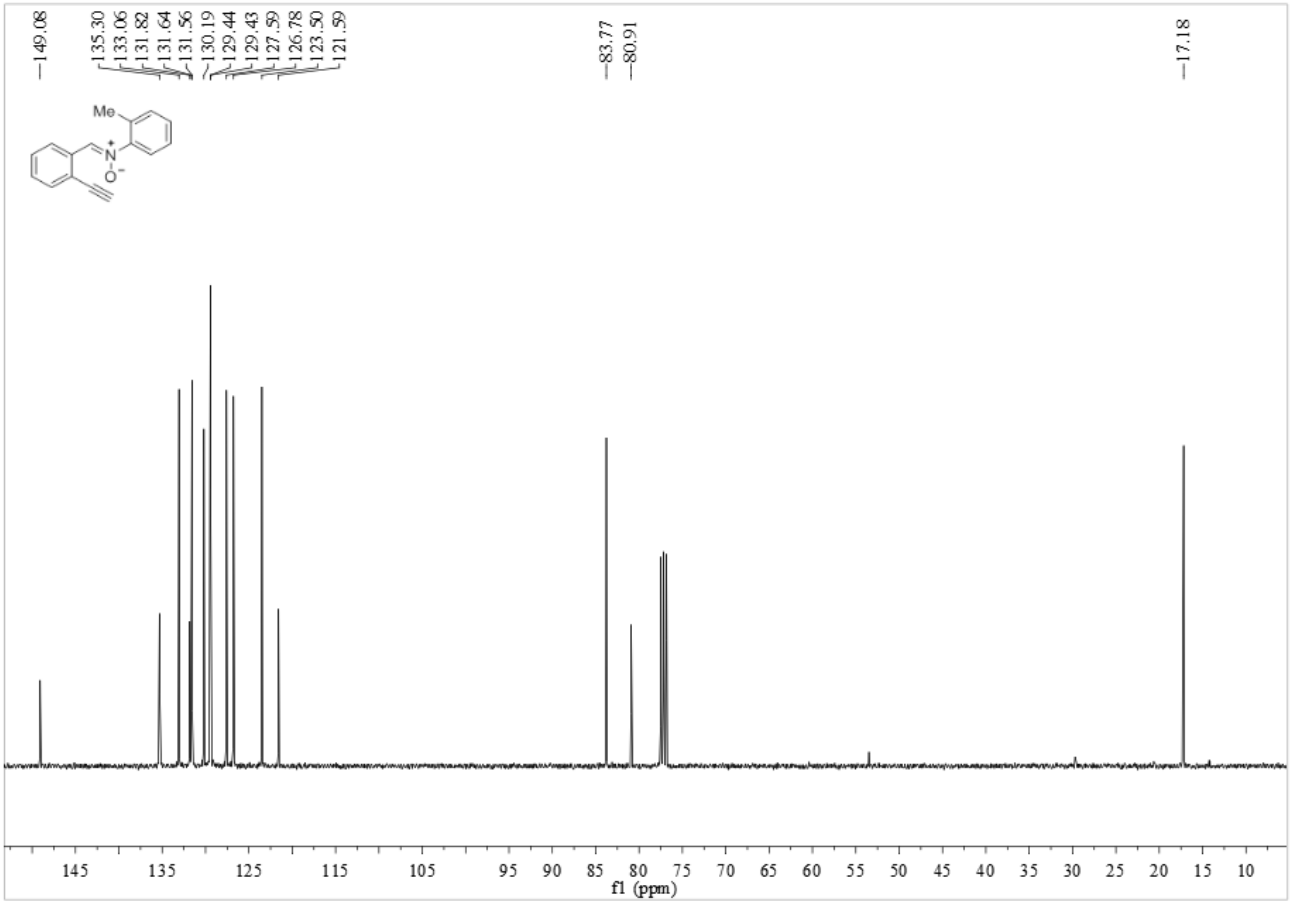
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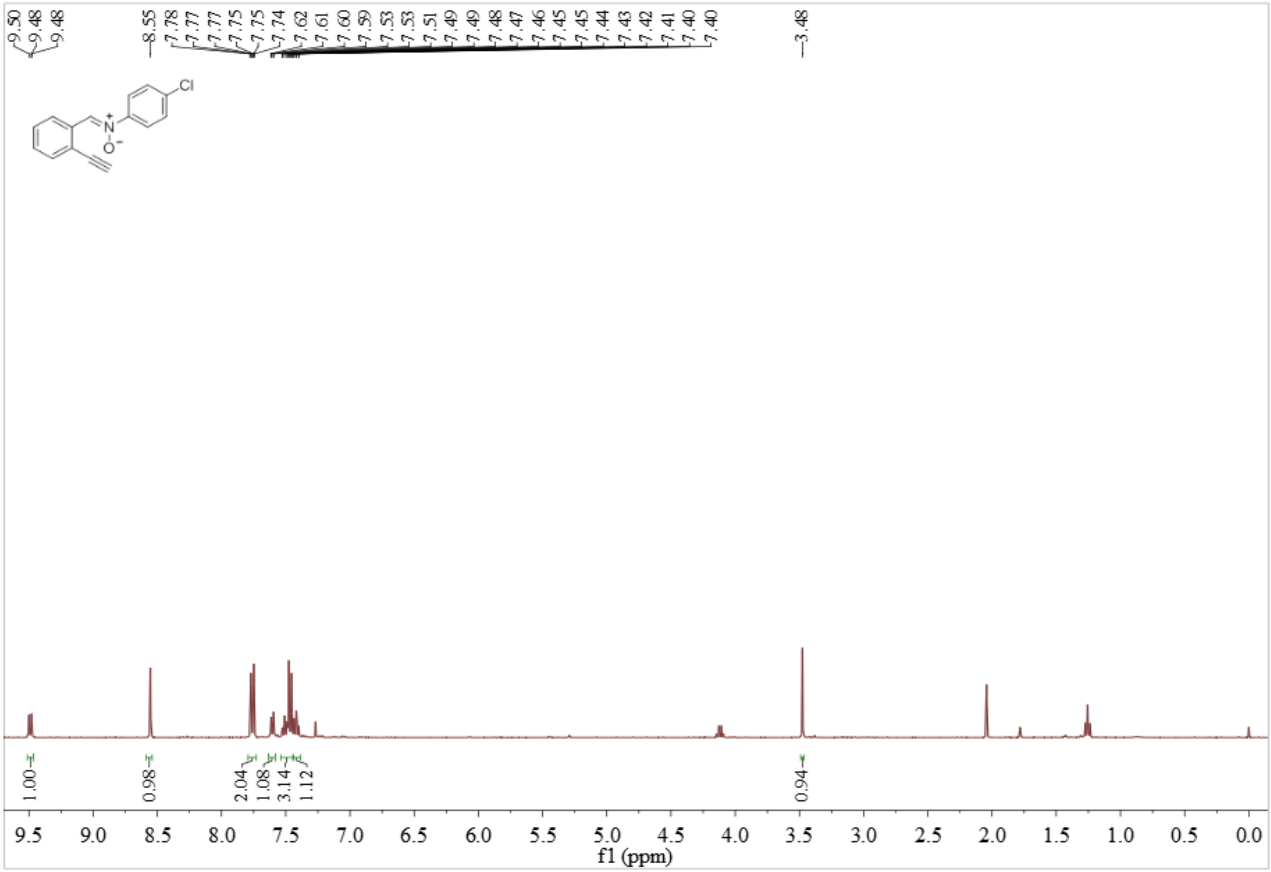
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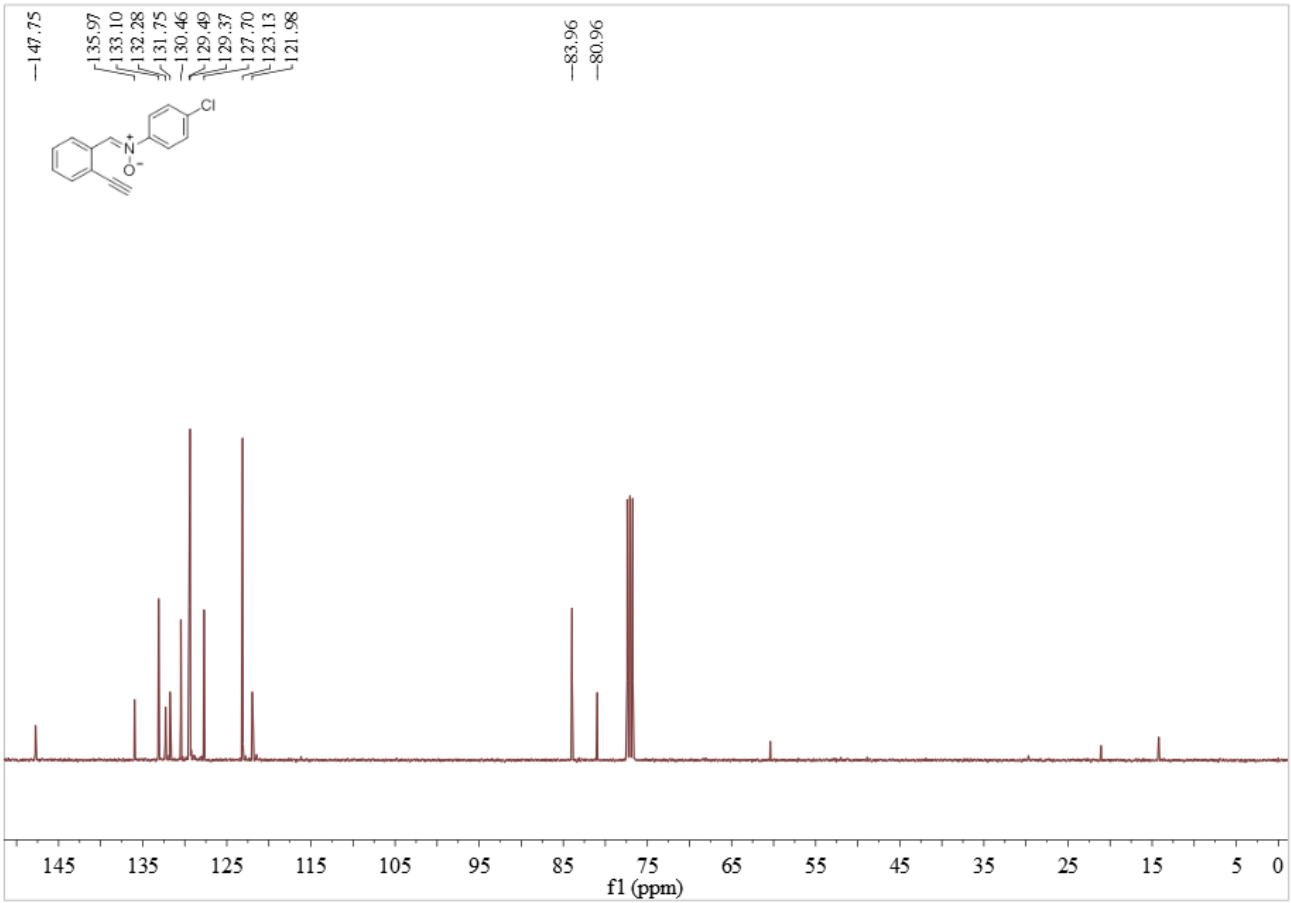
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f1 (ppm)

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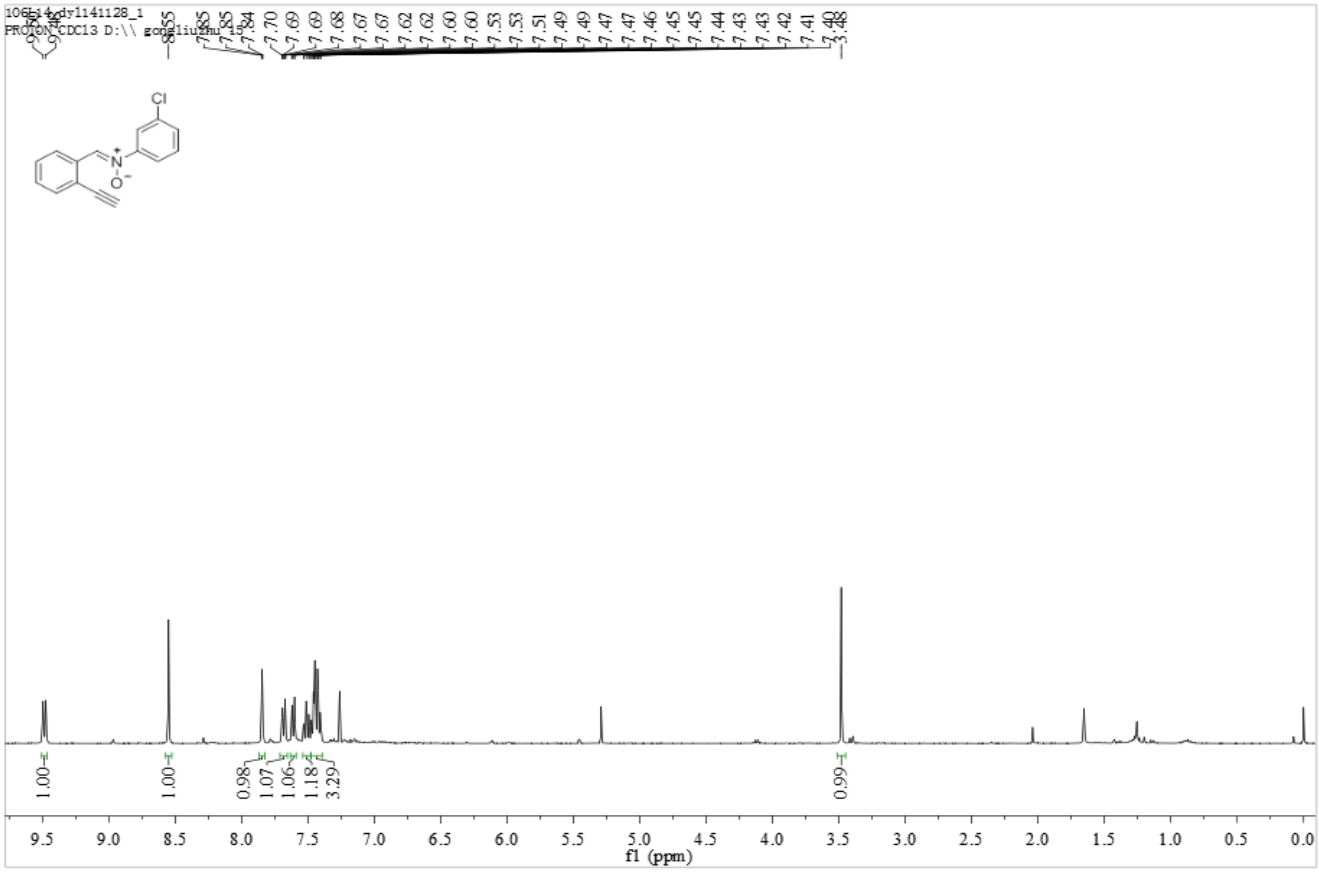
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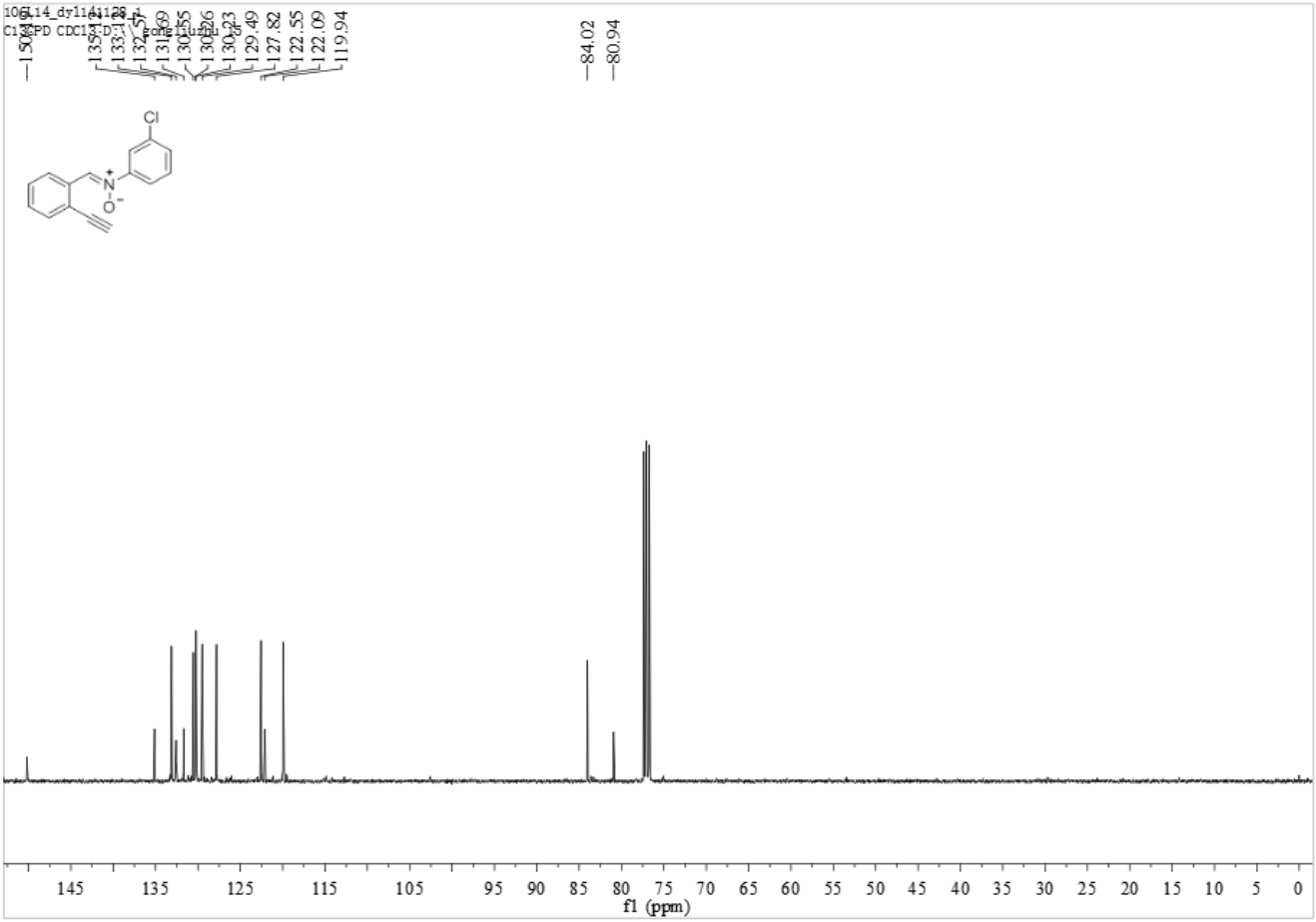
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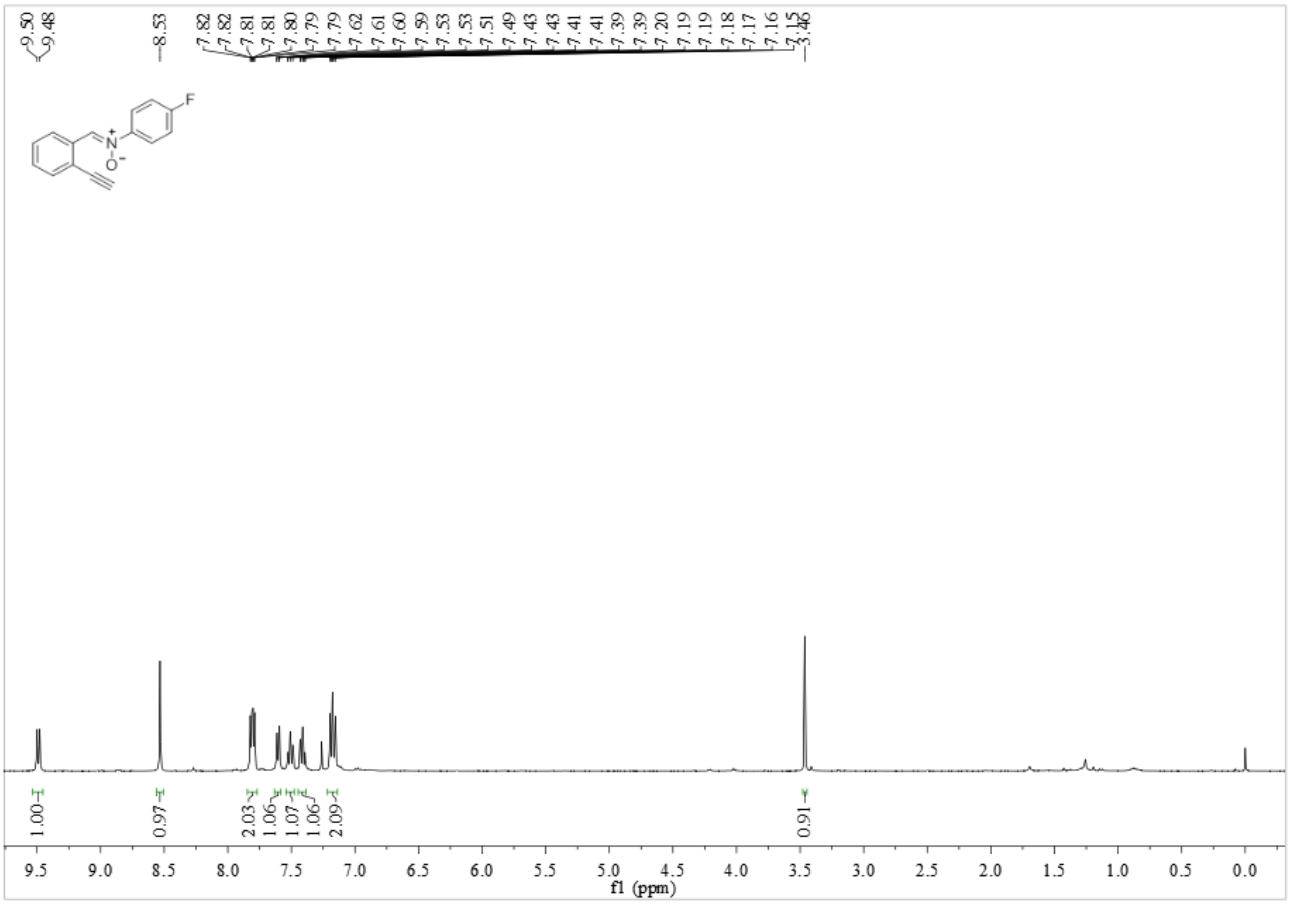
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7.41
7.39
7.39
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7.18
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7.15
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F
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O
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2.03
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1.07
1.06
2.09
0.91
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
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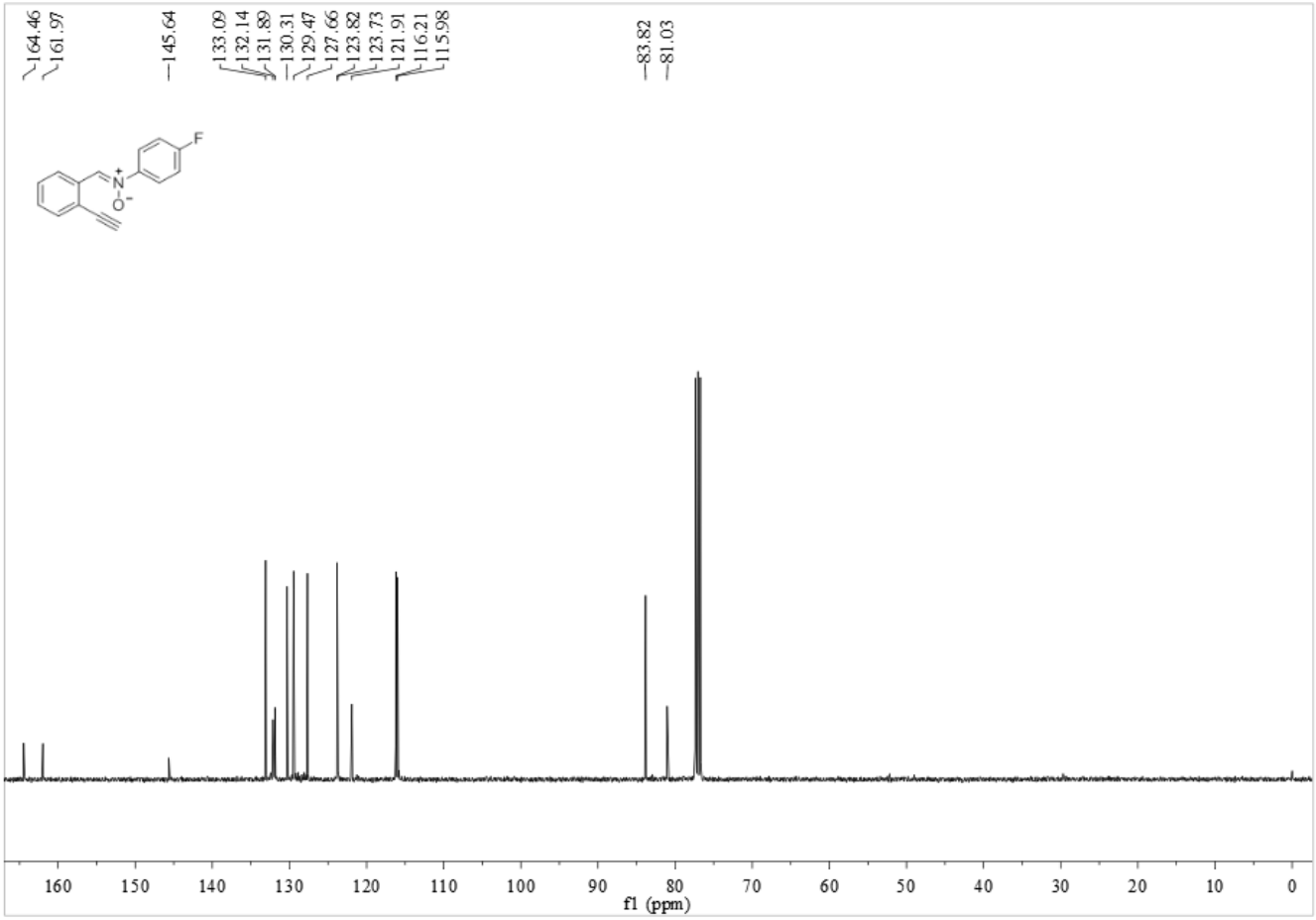
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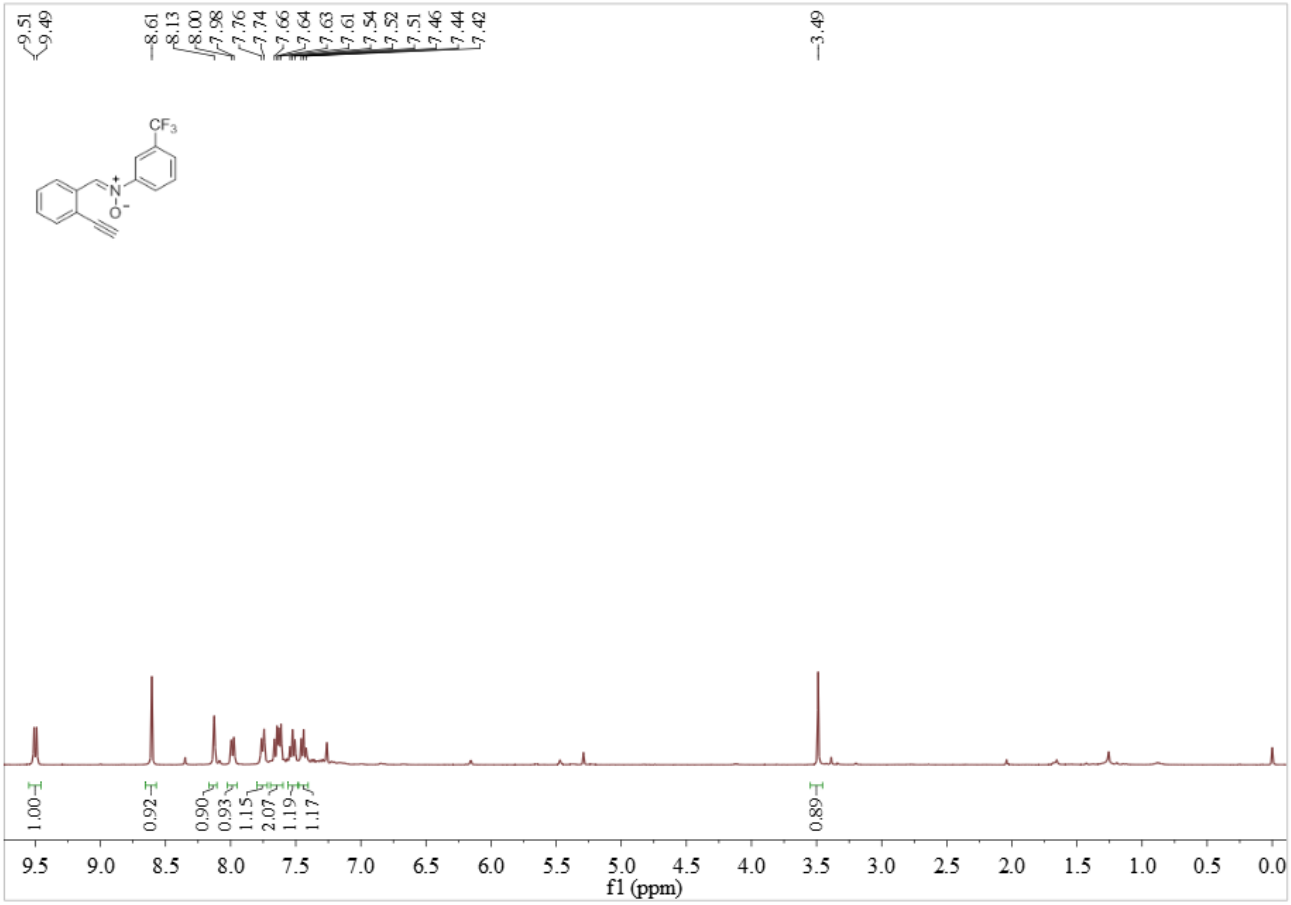
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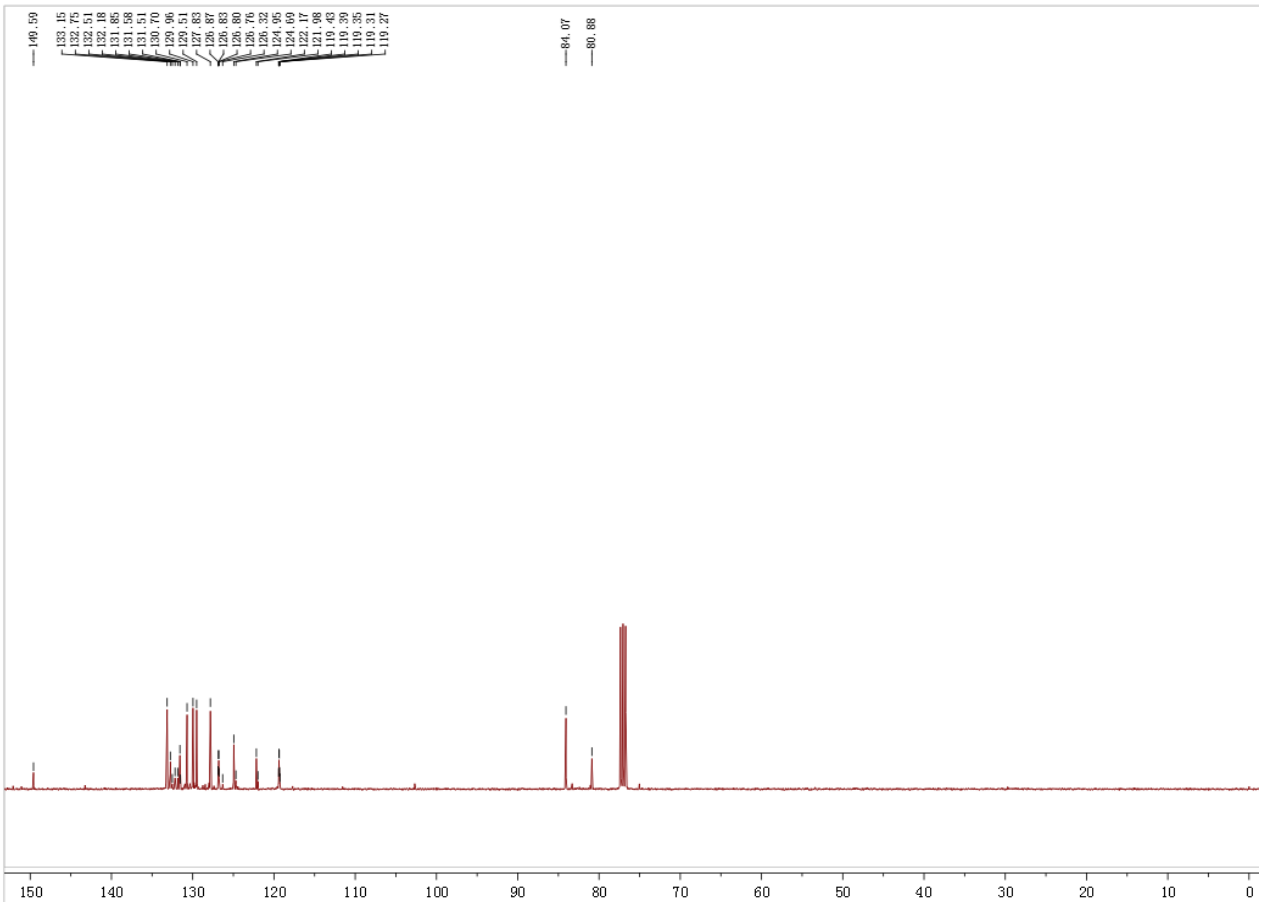
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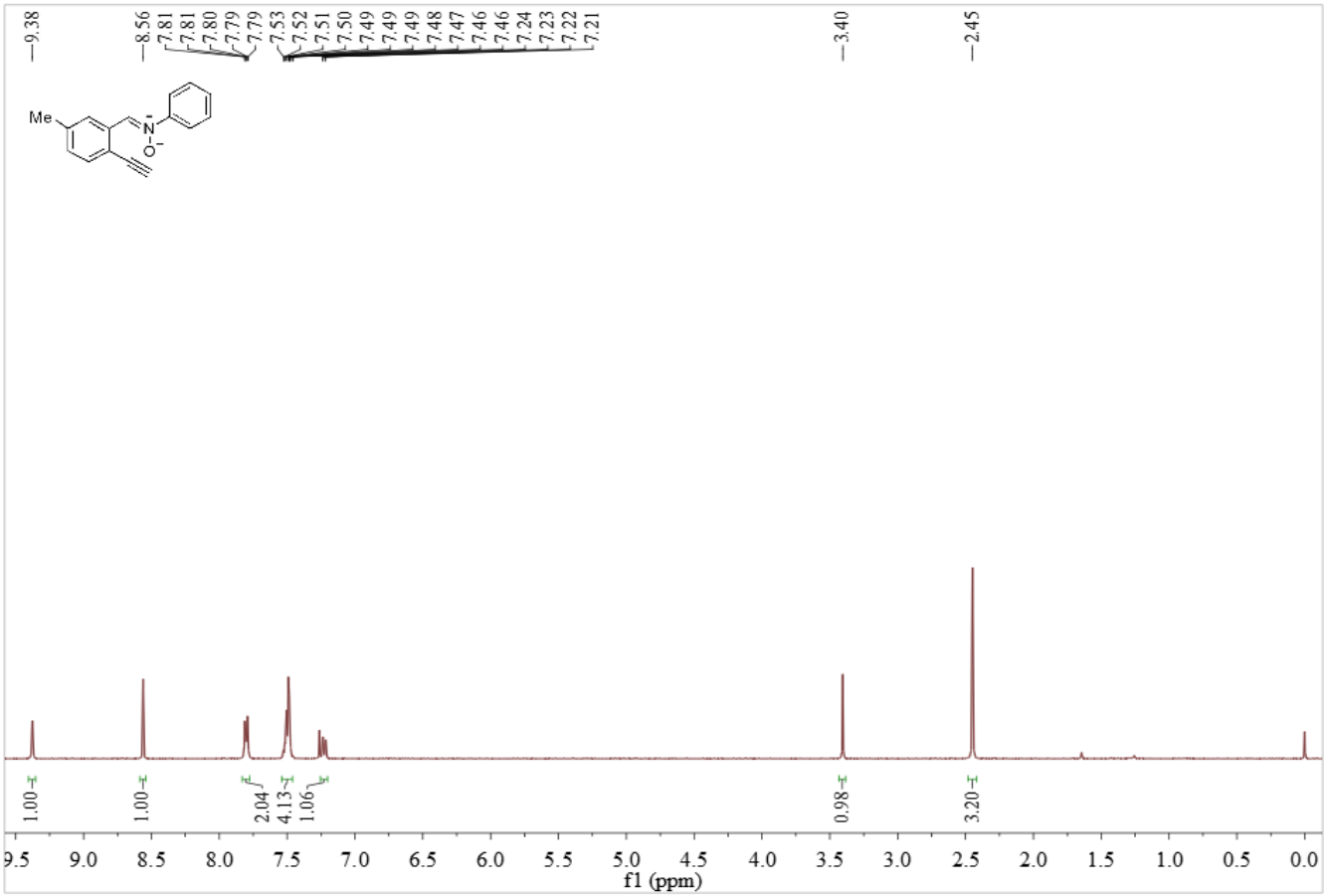
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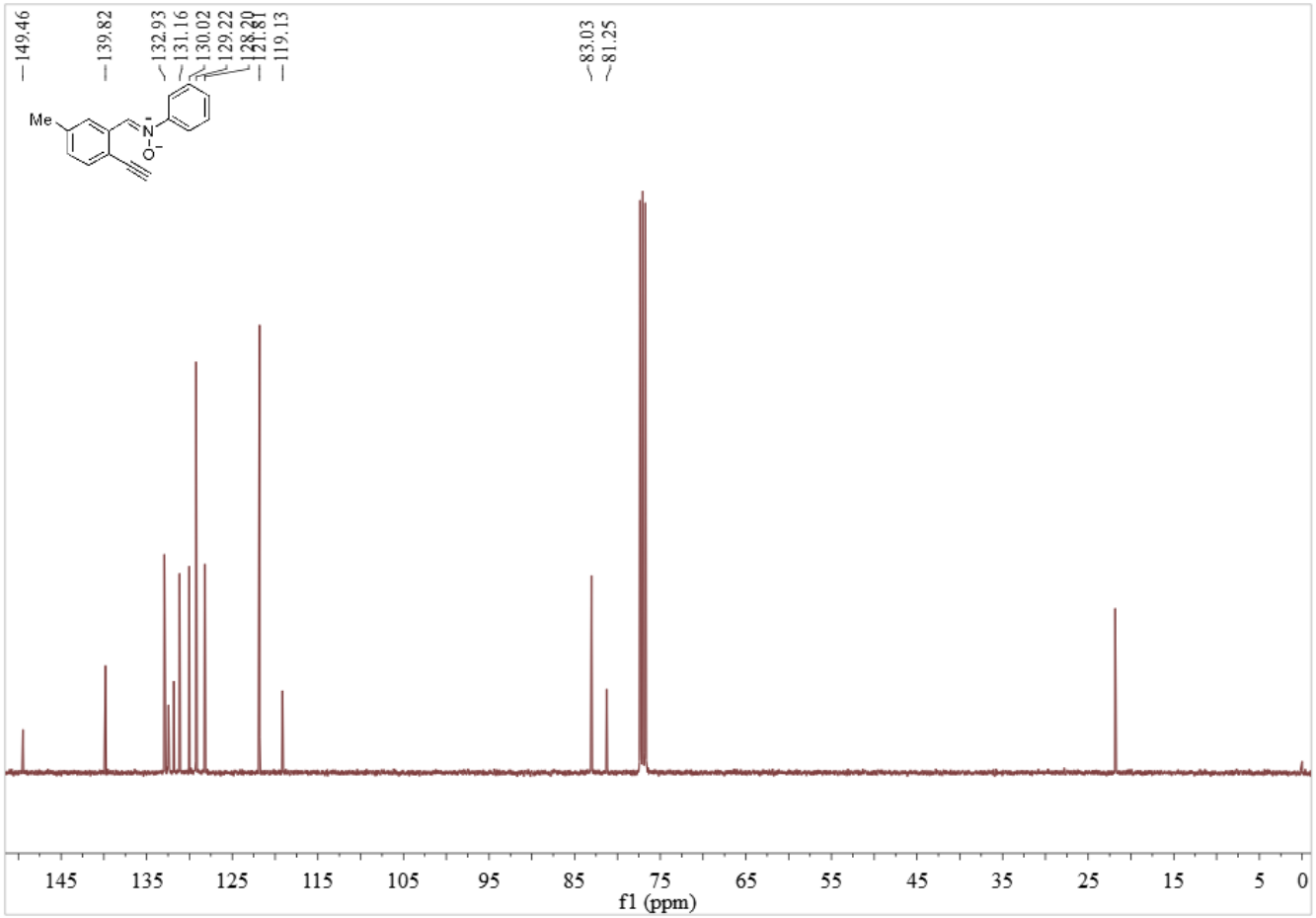
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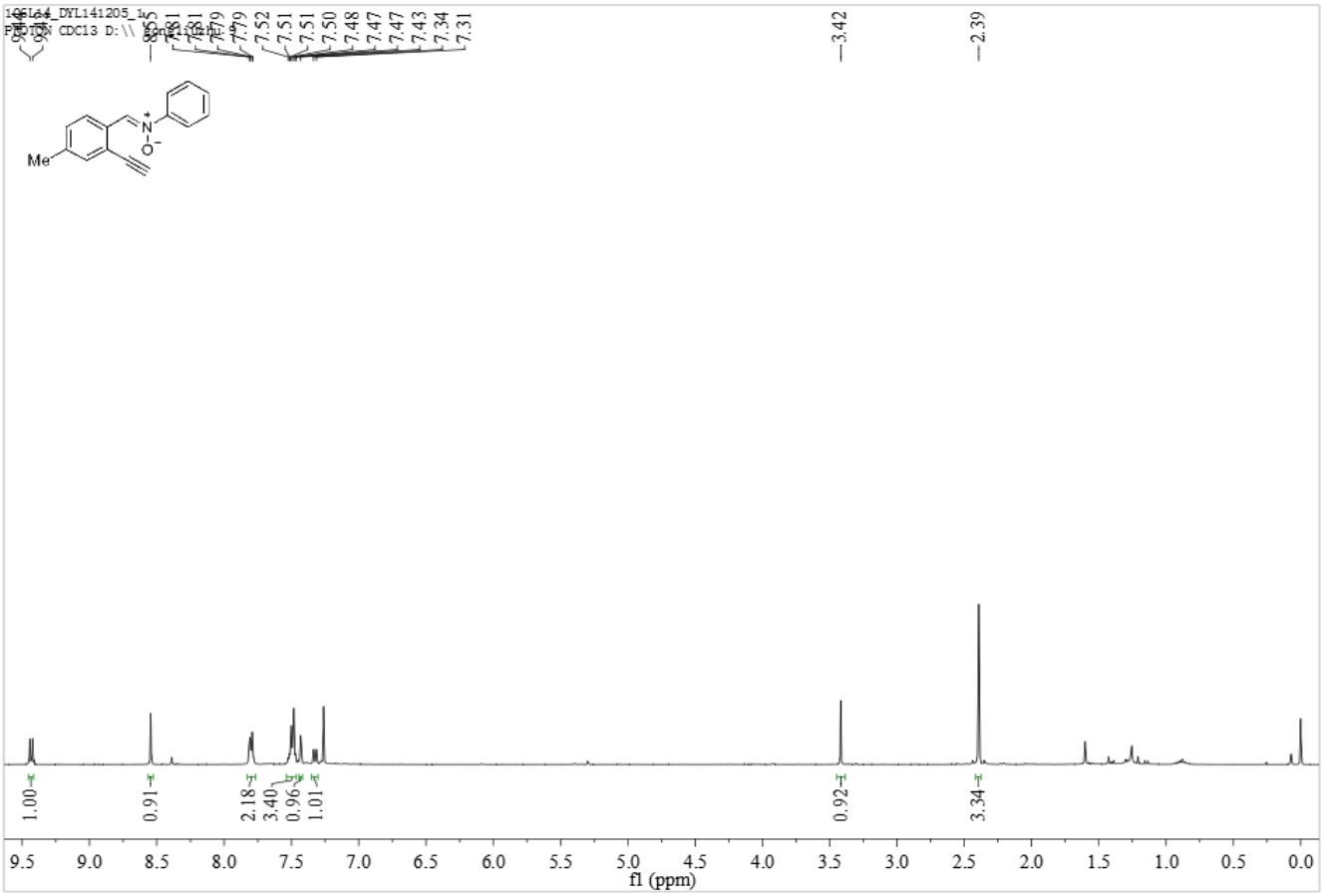
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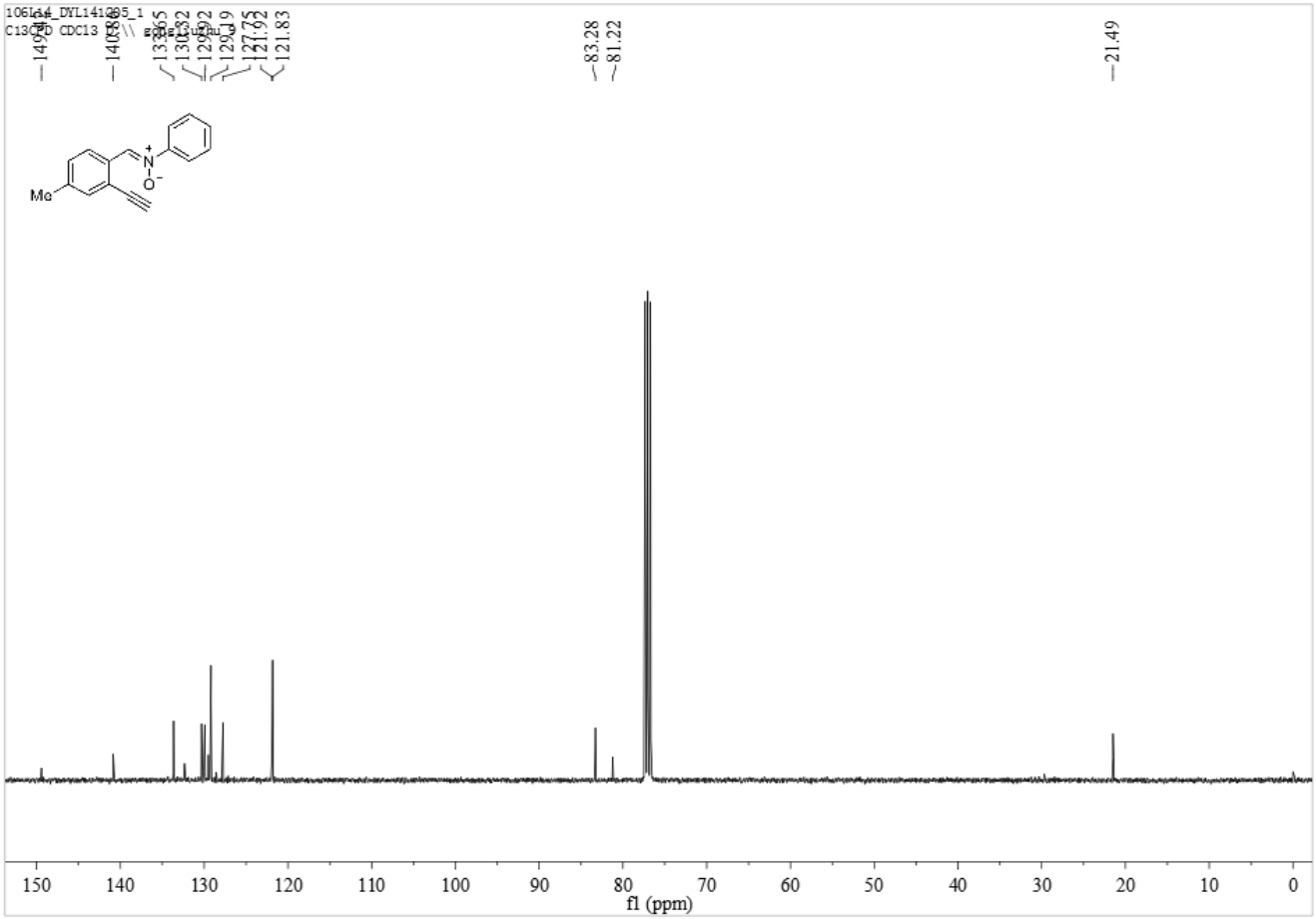
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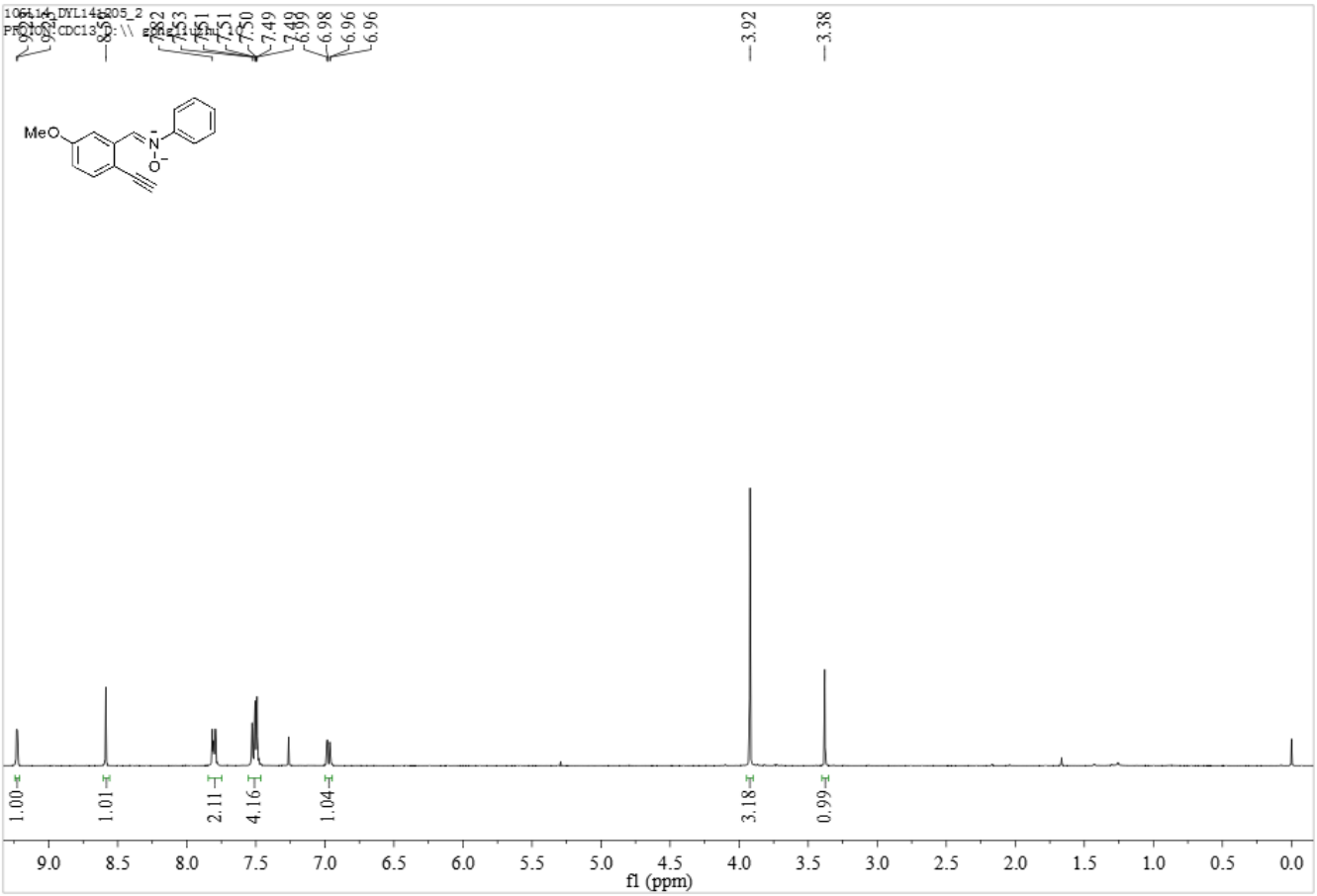
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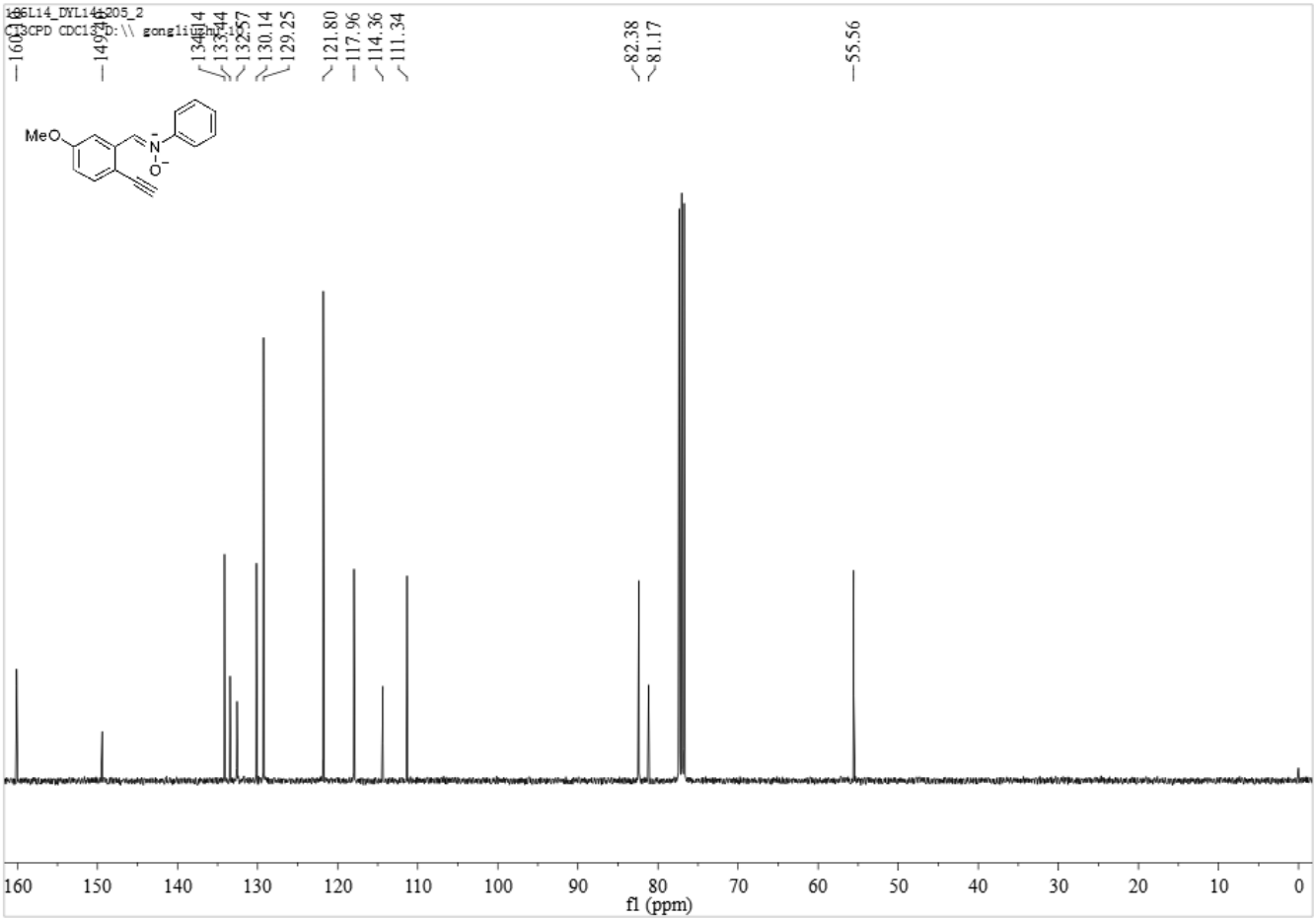
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f1 (ppm)

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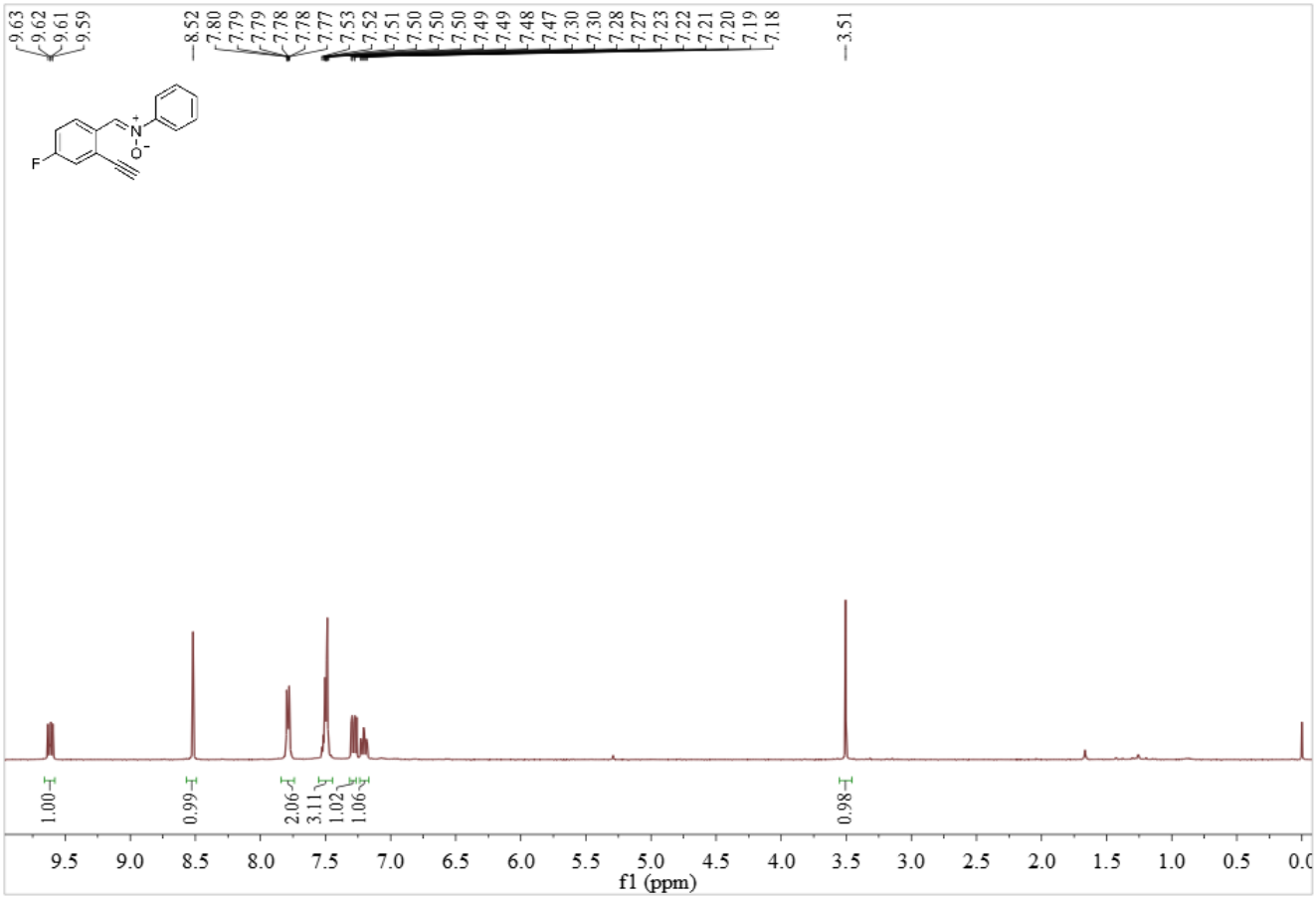
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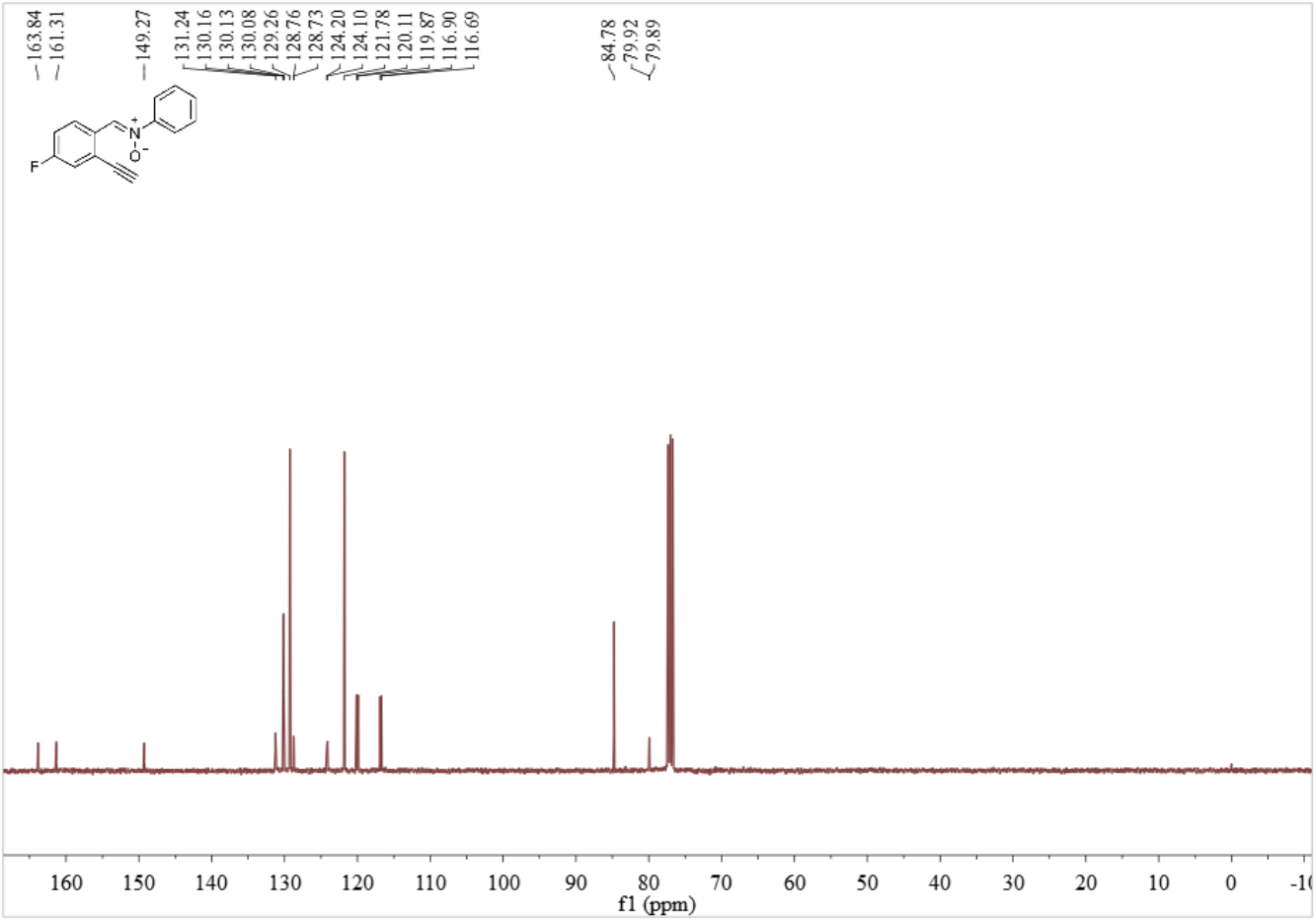
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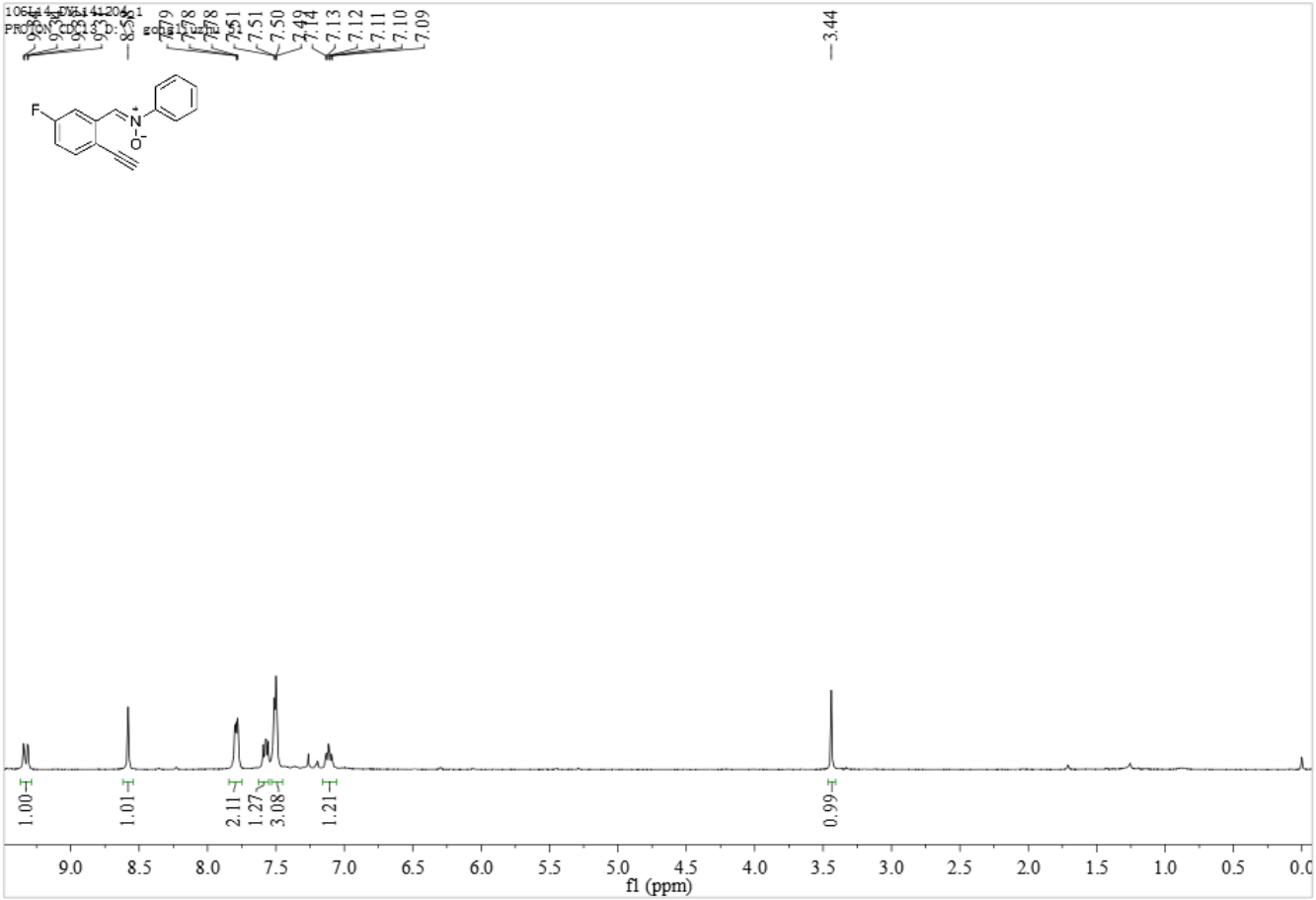
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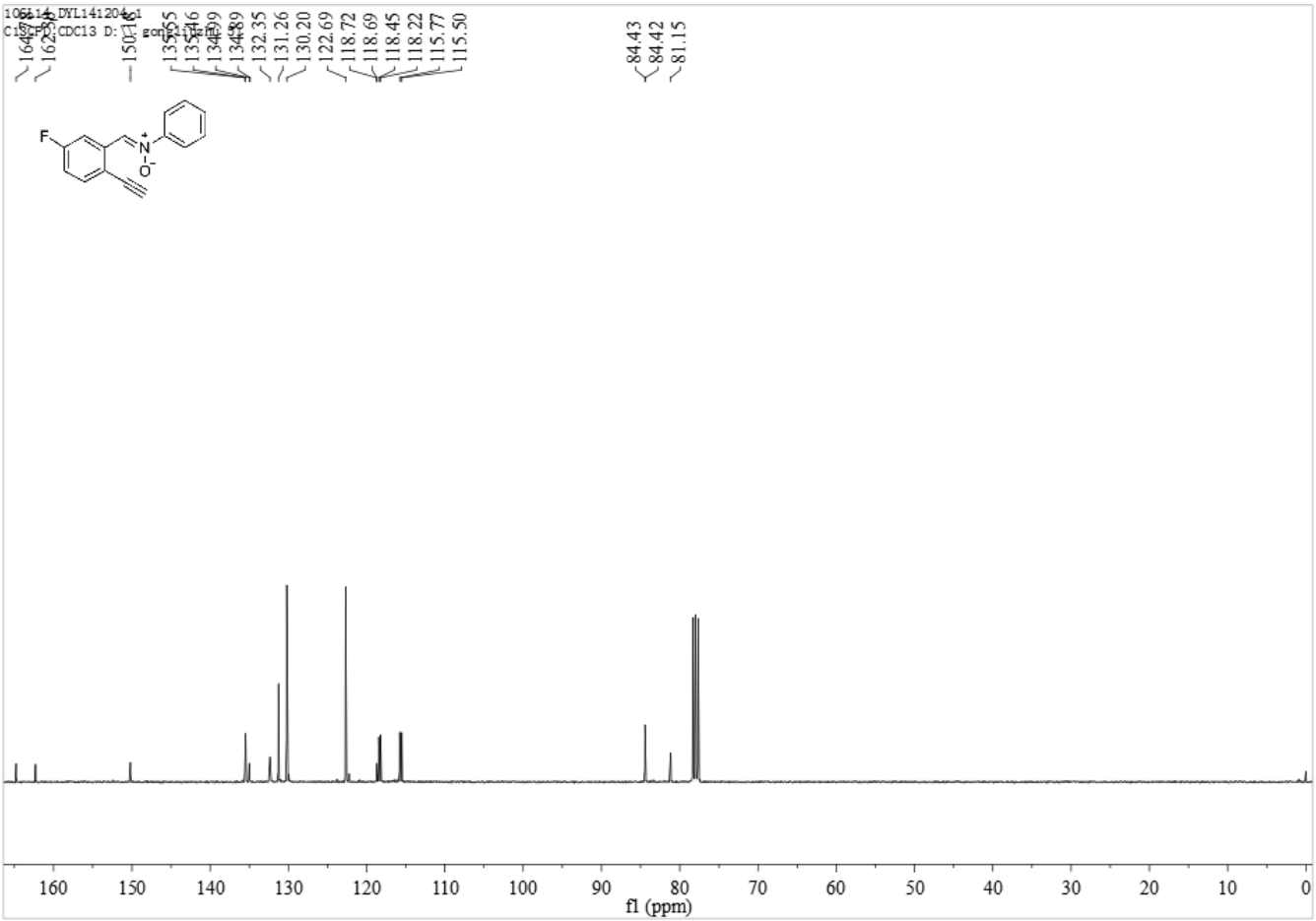
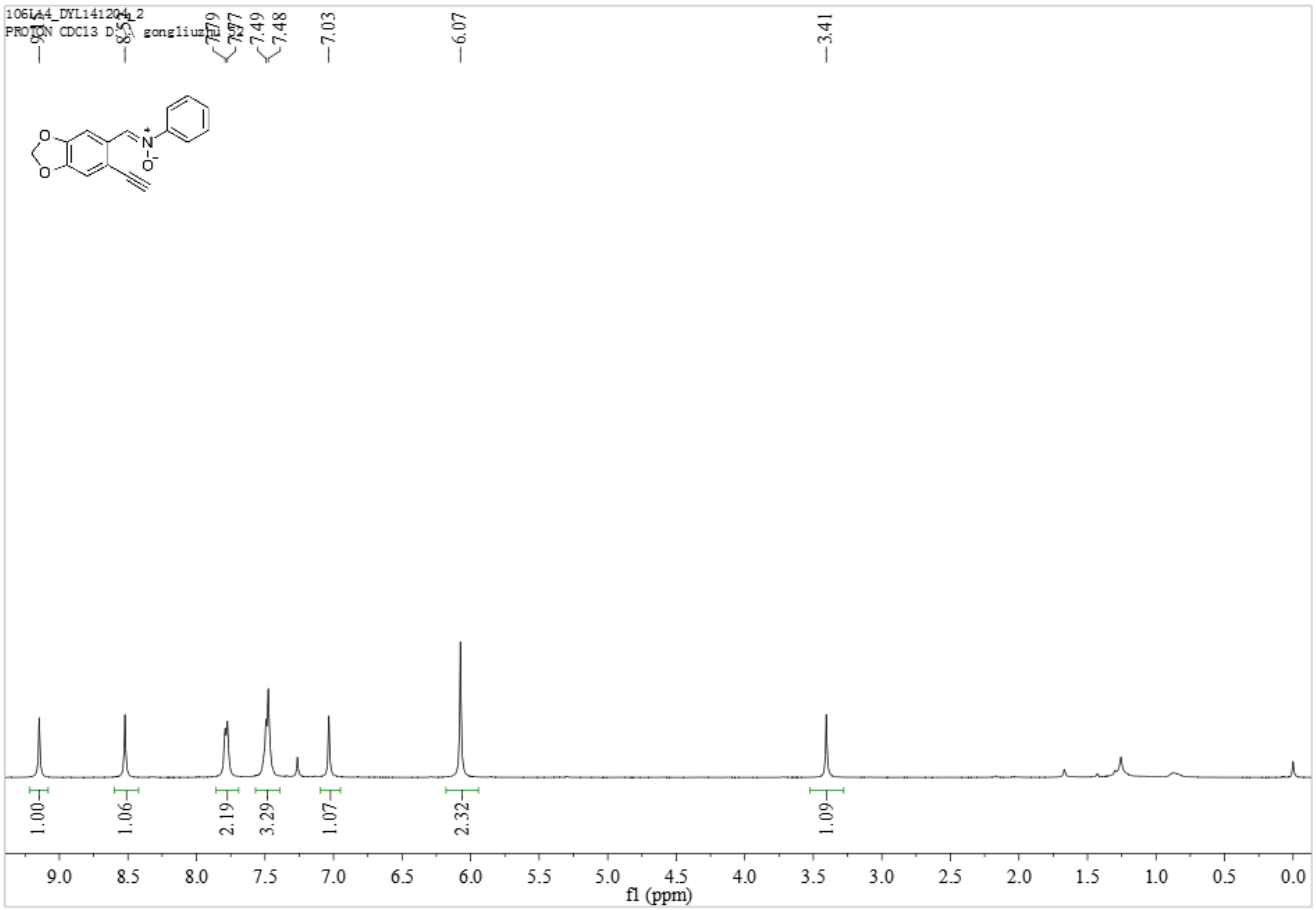
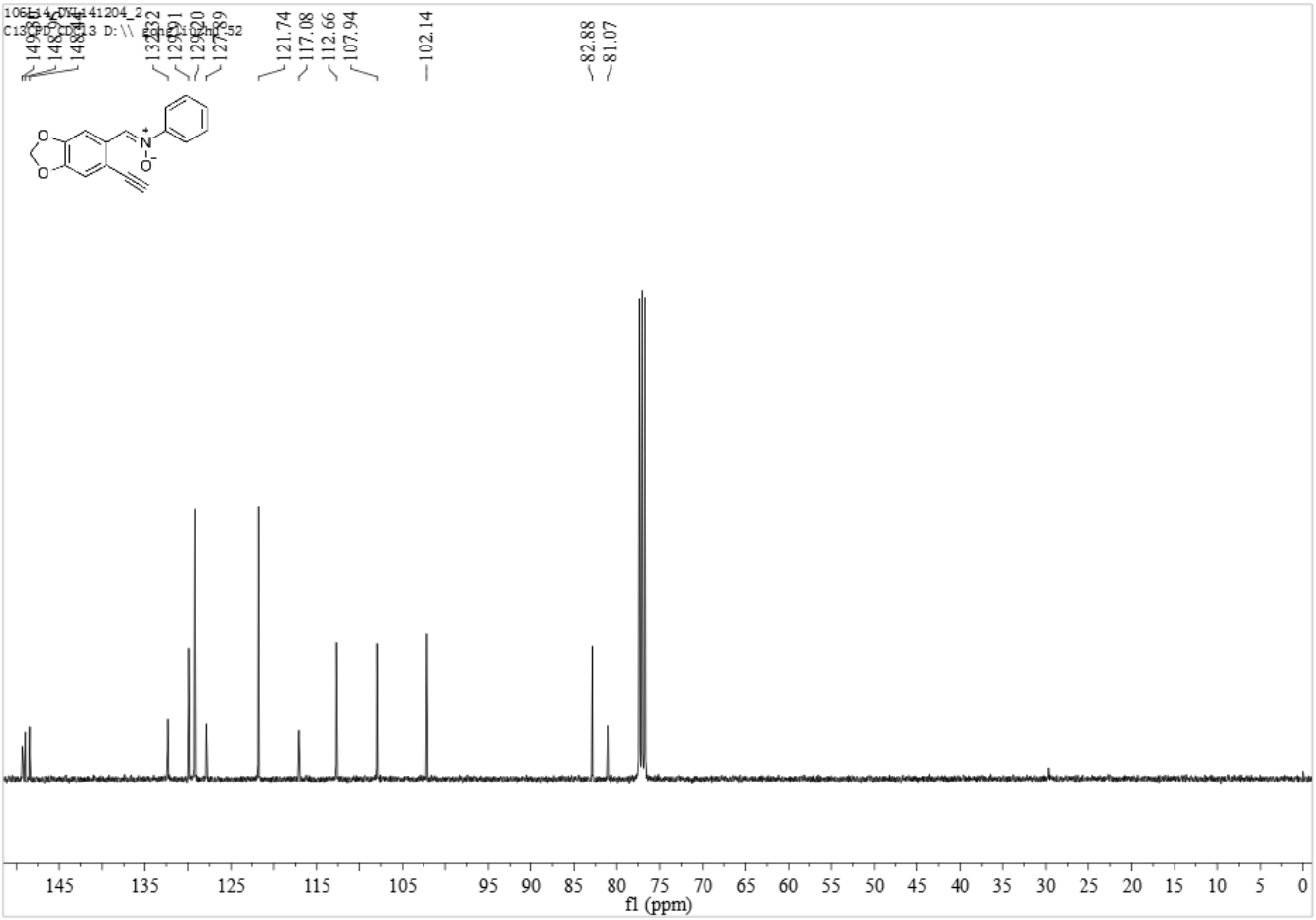
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

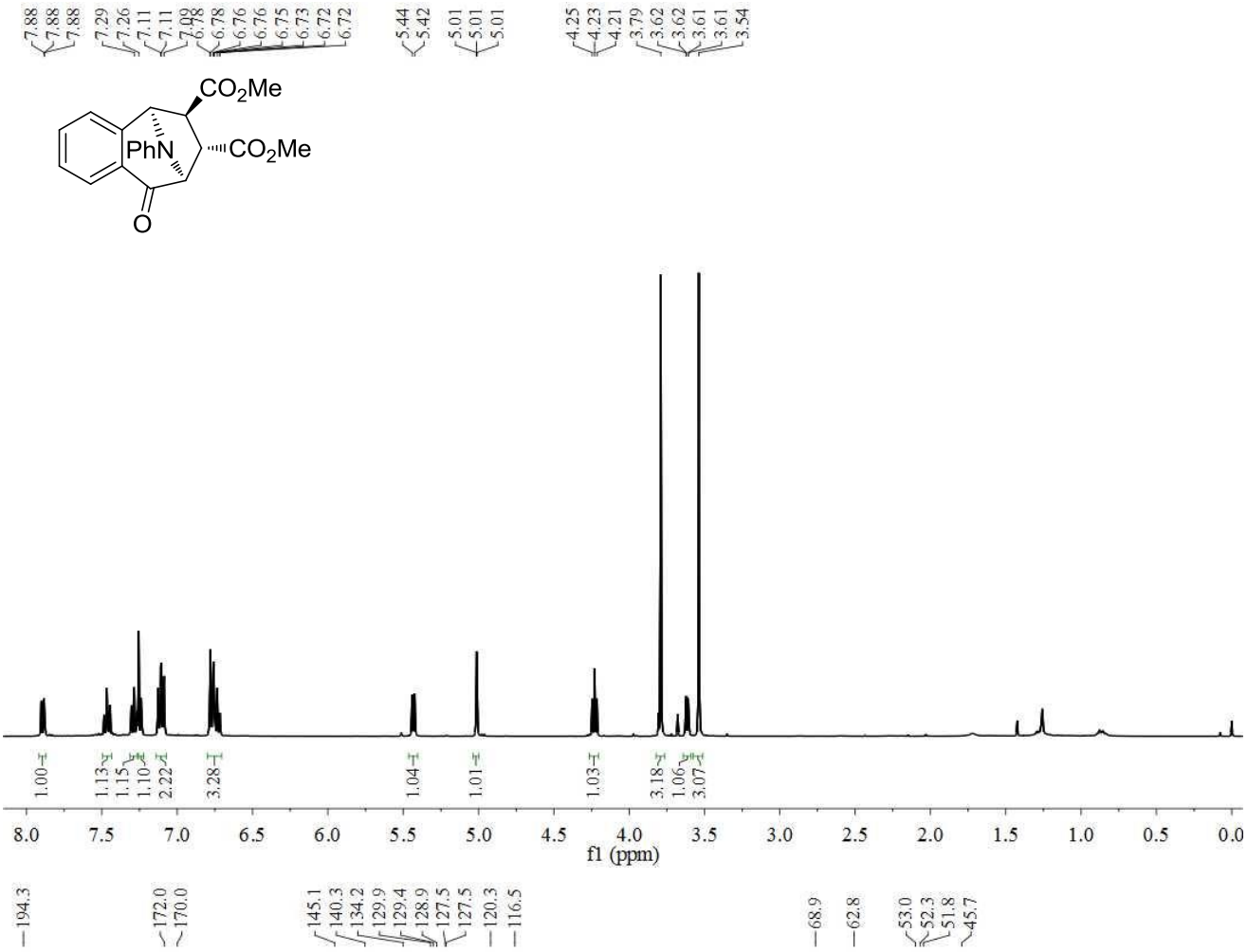

150.16
135.55
135.46
134.99
134.89
132.35
131.26
130.20
122.69
118.72
118.69
118.45
118.22
115.77
115.50
84.43
84.42
81.15
160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

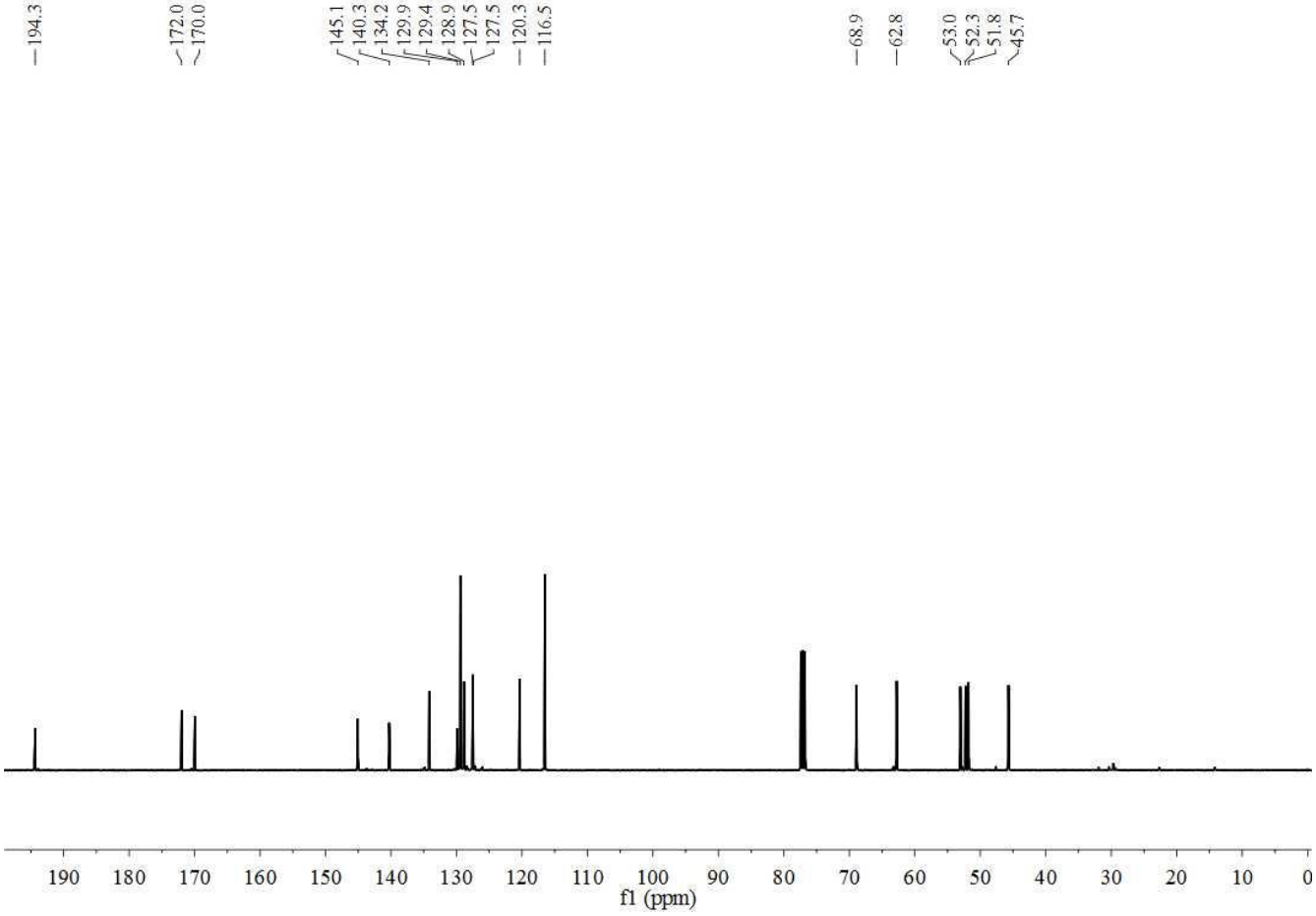
1n


106LA4_DYL141204_2
PROTON CDCl3 D:\ gongliuzi
9.00
8.53
7.79
7.77
7.49
7.48
7.03
6.07
3.41
1.00
1.06
2.19
3.29
1.07
2.32
1.09
f1 (ppm)

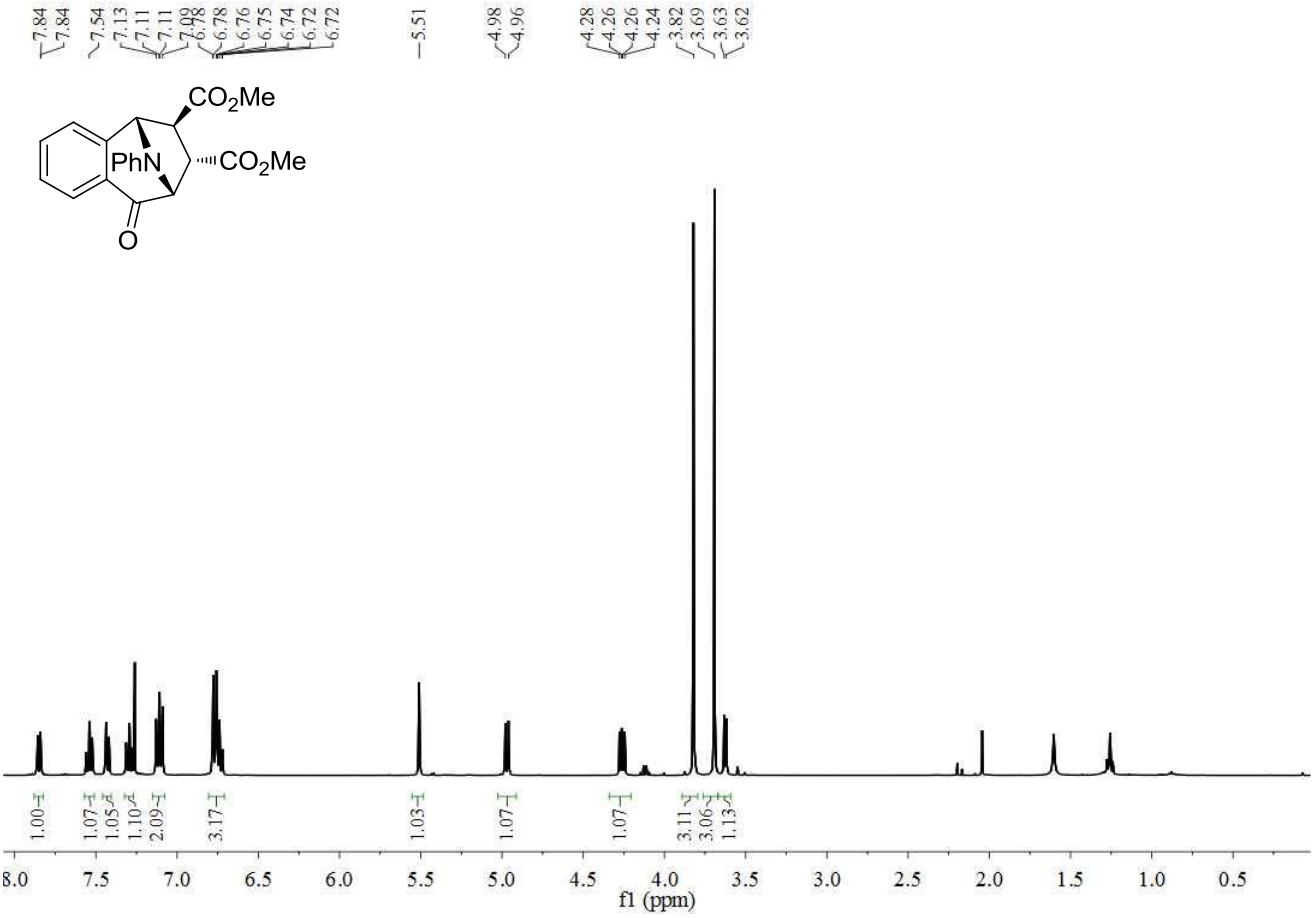

106LA4_DYL141204_2
C13CPD CDCl3 D:\
121.74
117.08
112.66
107.94
102.14
82.88
81.07
f1 (ppm)

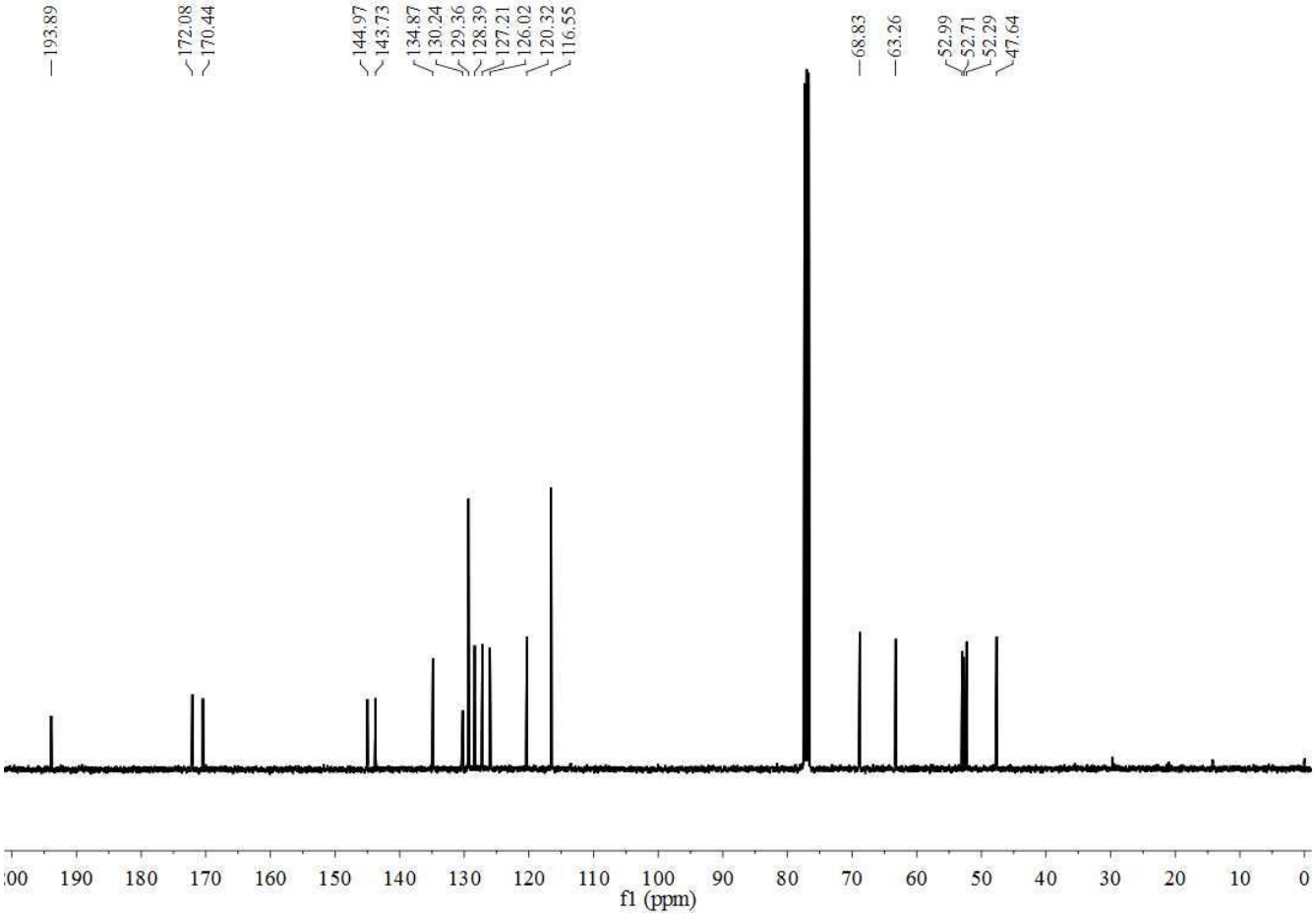
3a


7.88
7.88
7.88
7.29
7.26
7.11
7.11
7.09
6.78
6.78
6.76
6.76
6.75
6.73
6.72
6.72
5.44
5.42
5.01
5.01
5.01
4.25
4.23
4.21
3.79
3.62
3.62
3.61
3.61
3.54
CO2Me
PhN
CO2Me
O
1.00
1.13
1.15
1.10
2.22
3.28
1.04
1.01
1.03
3.18
1.06
3.07
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)

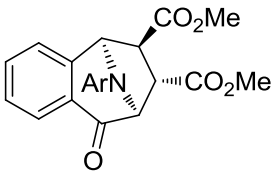

194.3
172.0
170.0
145.1
140.3
134.2
129.9
129.4
128.9
127.5
127.5
120.3
116.5
68.9
62.8
53.0
52.3
51.8
45.7
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

3a'

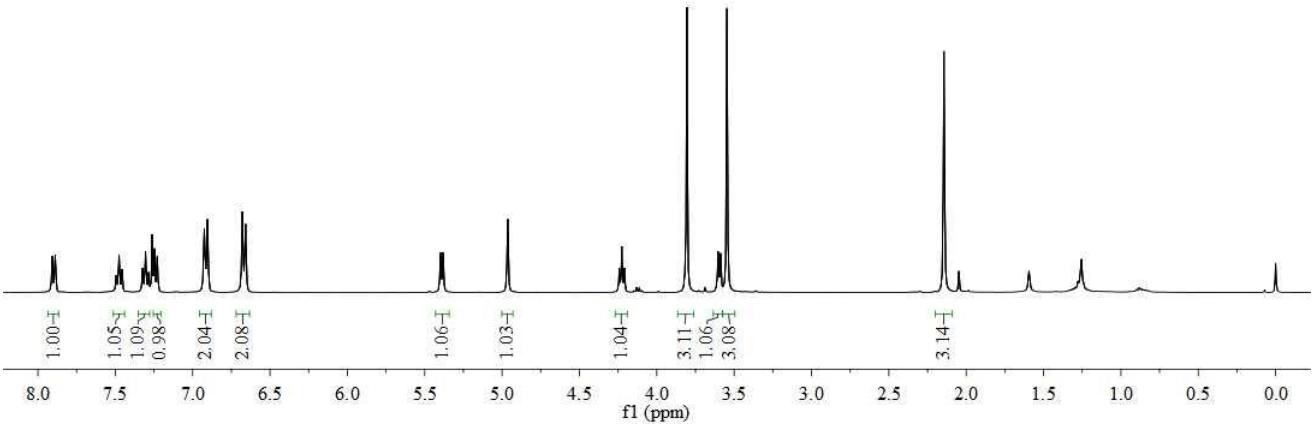

7.84
7.84
7.54
7.13
7.11
7.11
7.09
6.78
6.78
6.76
6.75
6.74
6.72
6.72
5.51
4.98
4.96
4.28
4.26
4.26
4.24
3.82
3.69
3.63
3.62
CO2Me
PhN
CO2Me
O
1.00
1.07
1.05
1.10
2.09
3.17
1.03
1.07
1.07
3.11
3.06
1.13
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

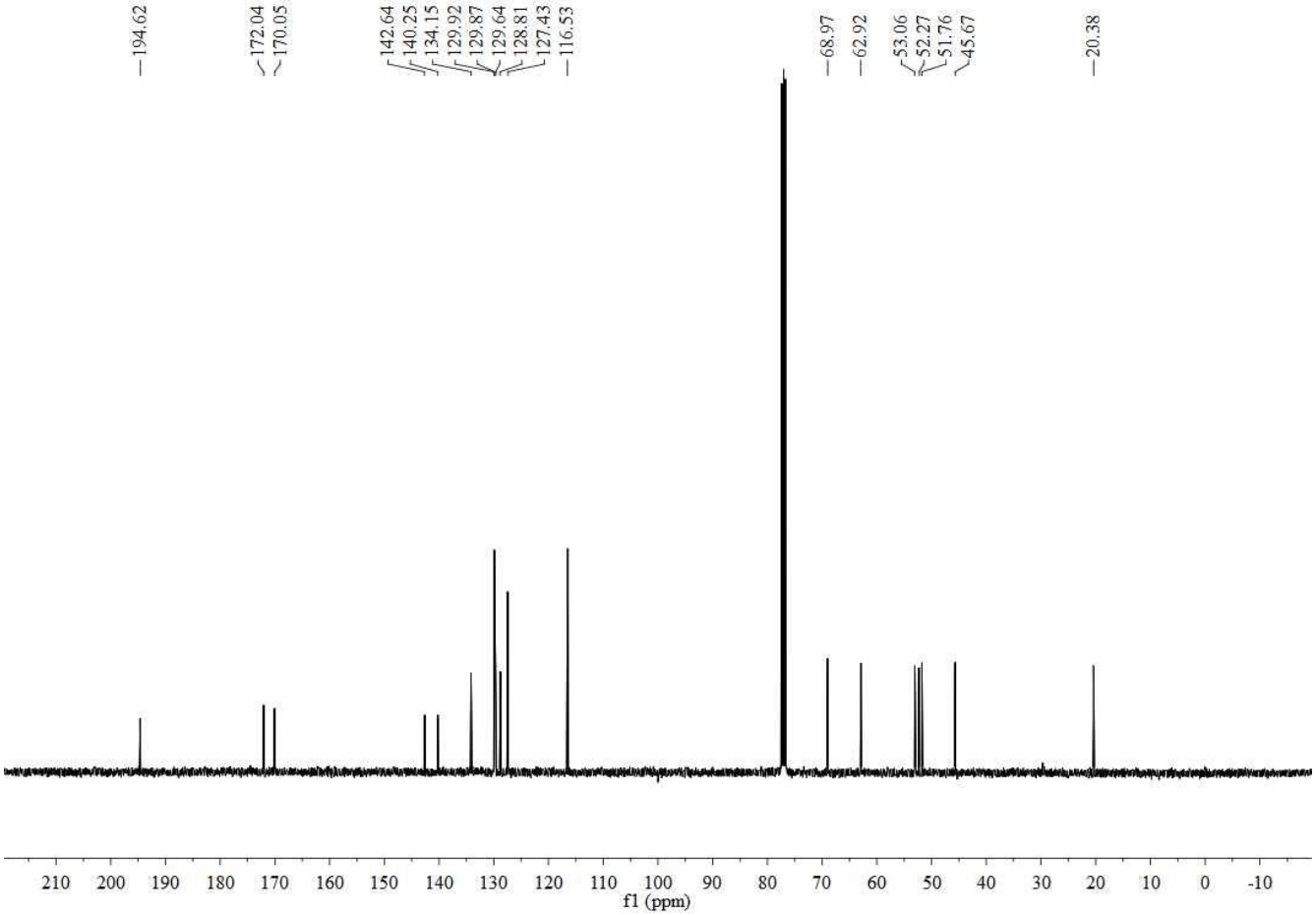

193.89
172.08
170.44
144.97
143.73
134.87
130.24
129.36
128.39
127.21
126.02
120.32
116.55
68.83
63.26
52.99
52.71
52.29
47.64
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

3b

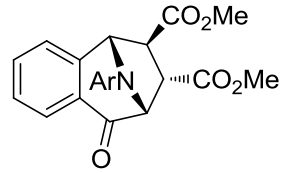


Ar = 4-MeC_6H_4

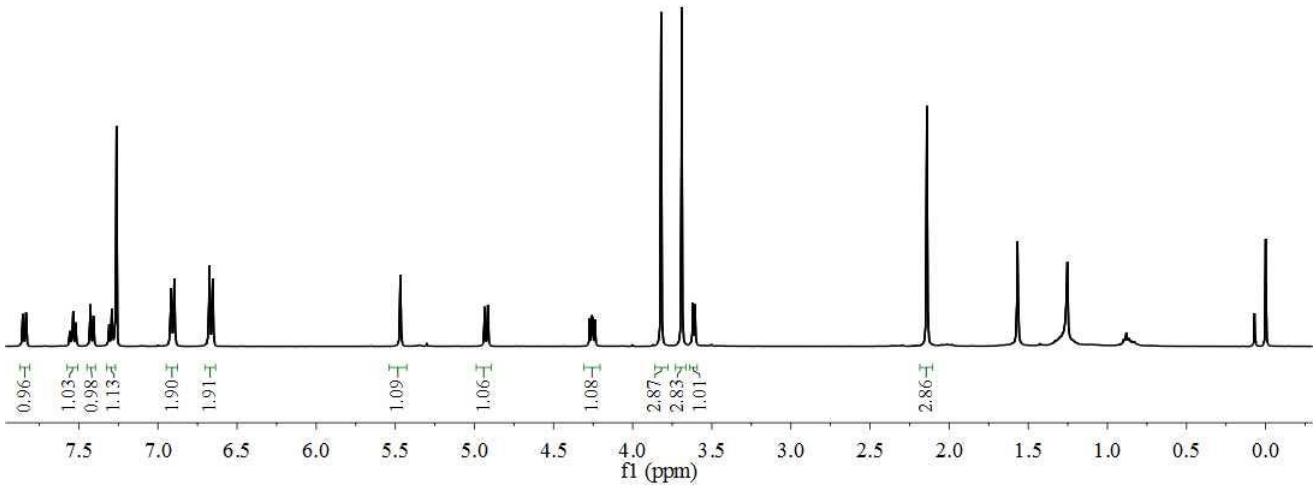


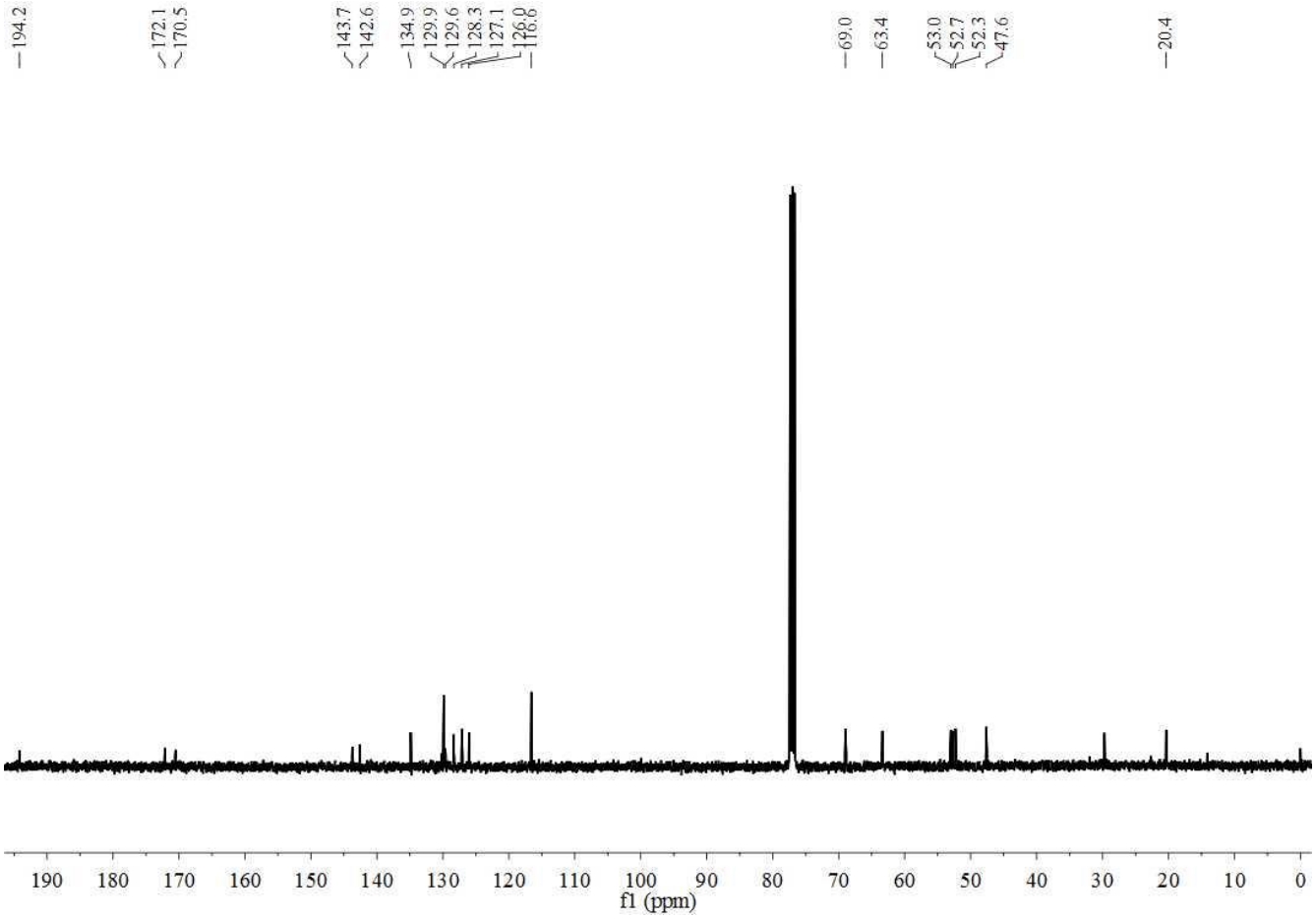


3b'

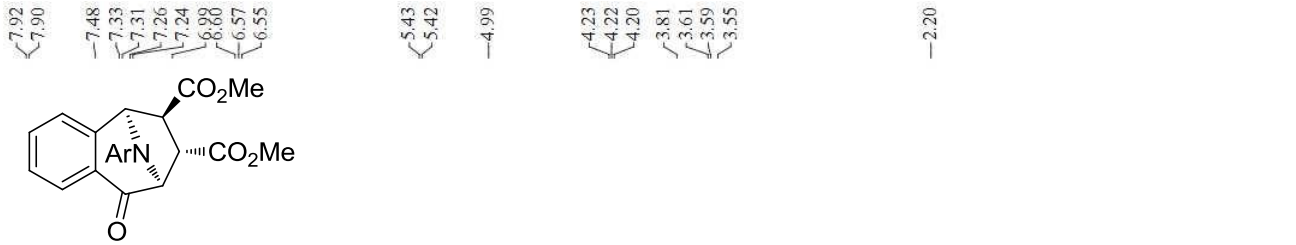


Ar = 4-MeC_6H_4

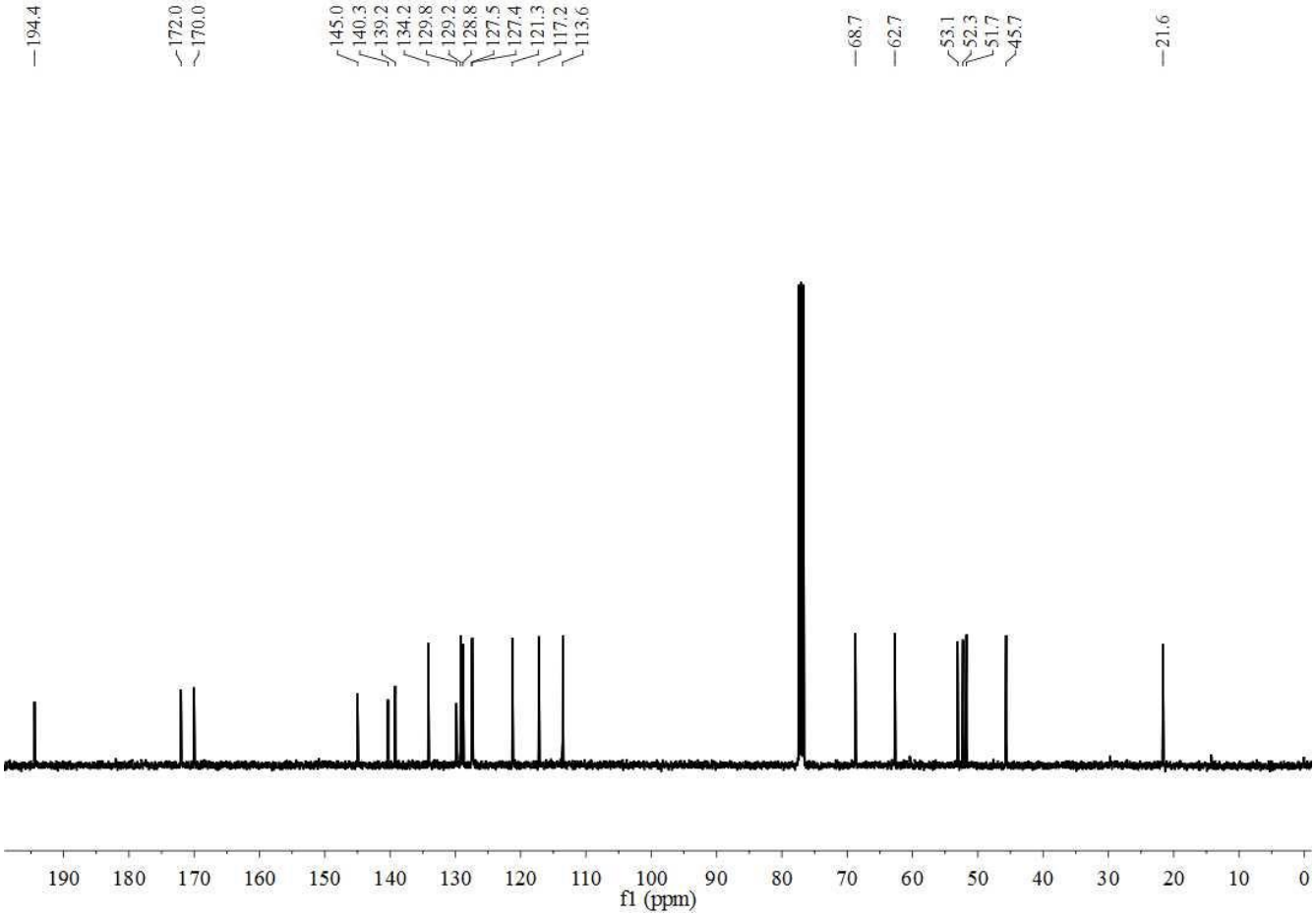




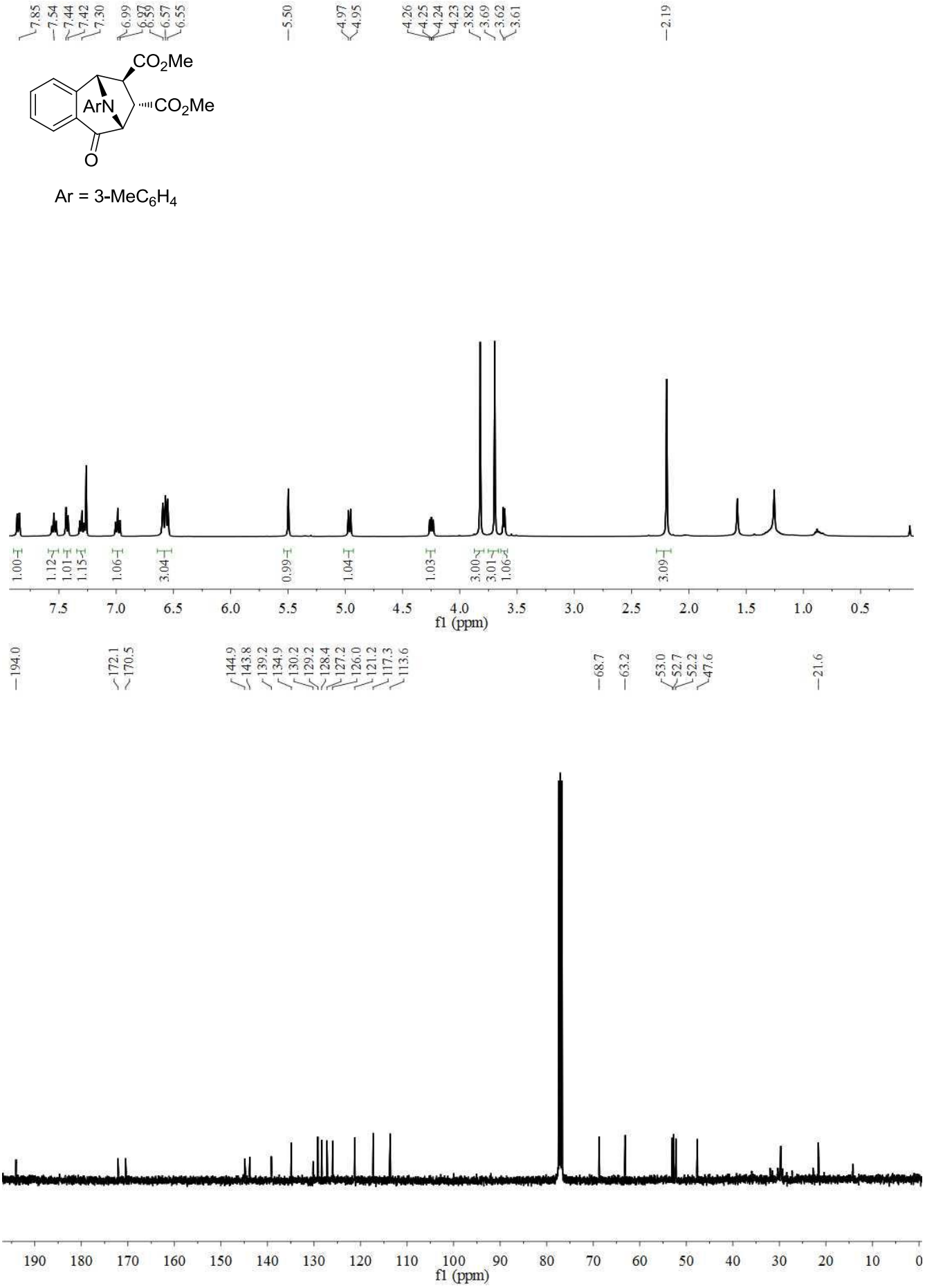
3c



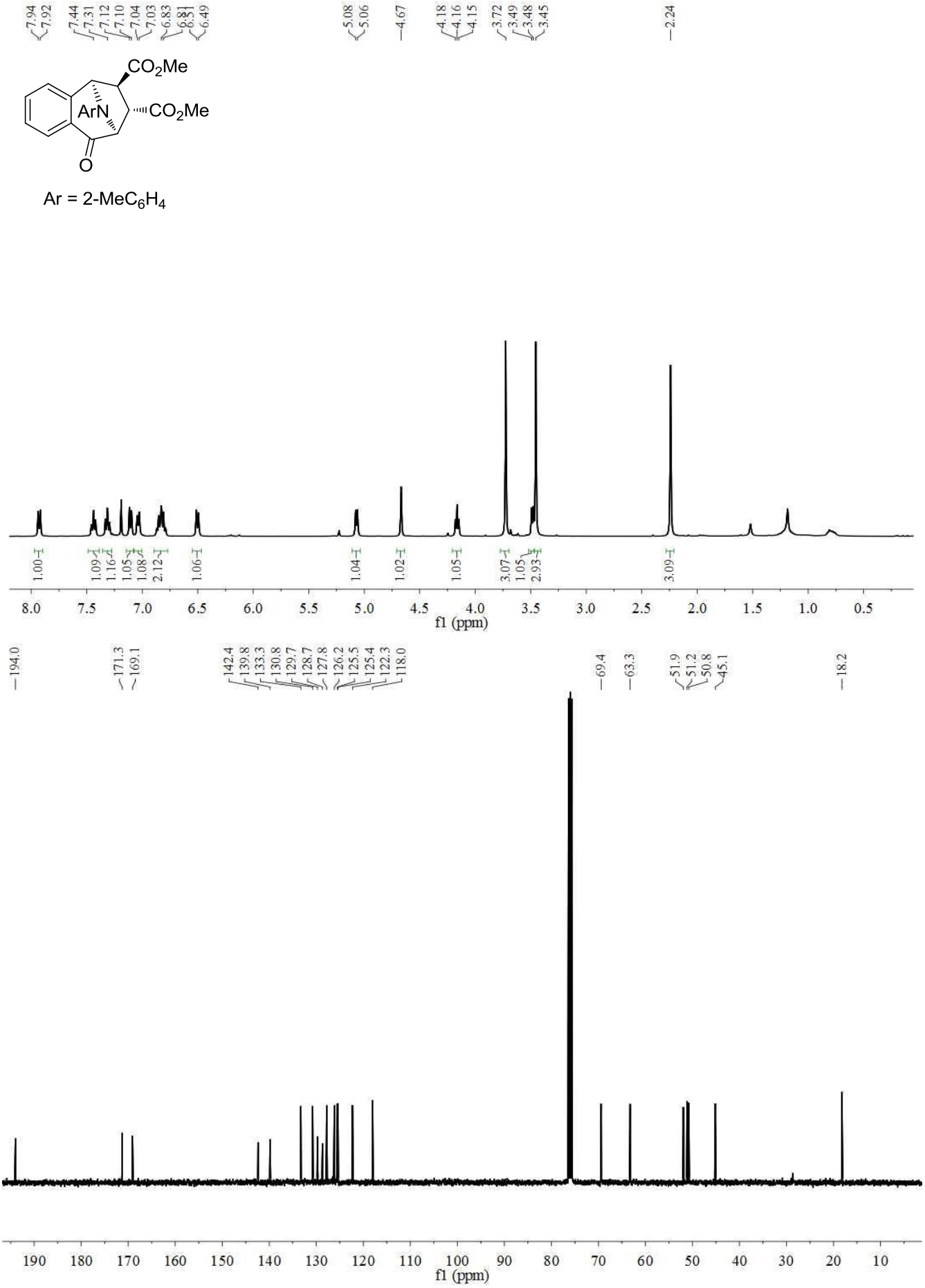
Ar = 3-MeC$_6$H$_4$



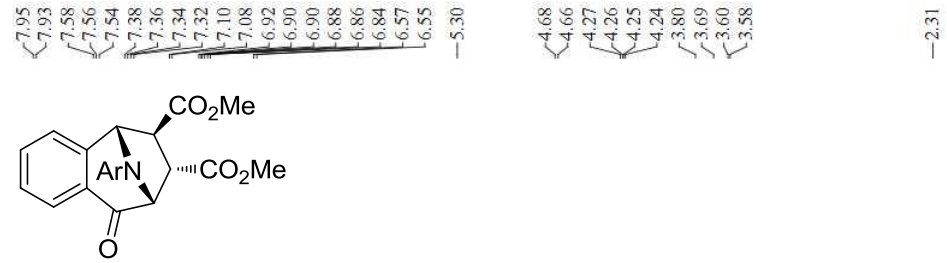
3c'


7.85
7.54
7.44
7.42
7.30
6.99
6.87
6.59
6.57
6.55
5.50
4.97
4.95
4.26
4.25
4.24
4.23
3.82
3.69
3.62
3.61
2.19
CO2Me
ArN
CO2Me
O
Ar = 3-MeC6H4
1.00
1.12
1.01
1.15
1.06
3.04
0.99
1.04
1.03
3.00
3.01
1.06
3.09
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)
194.0
172.1
170.5
144.9
143.8
139.2
134.9
130.2
129.2
128.4
127.2
126.0
121.2
117.3
113.6
68.7
63.2
53.0
52.7
52.2
47.6
21.6
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

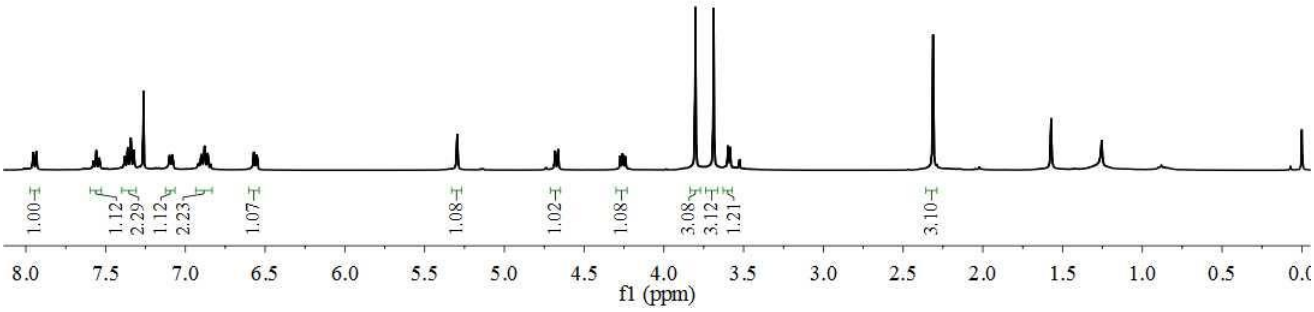
3d

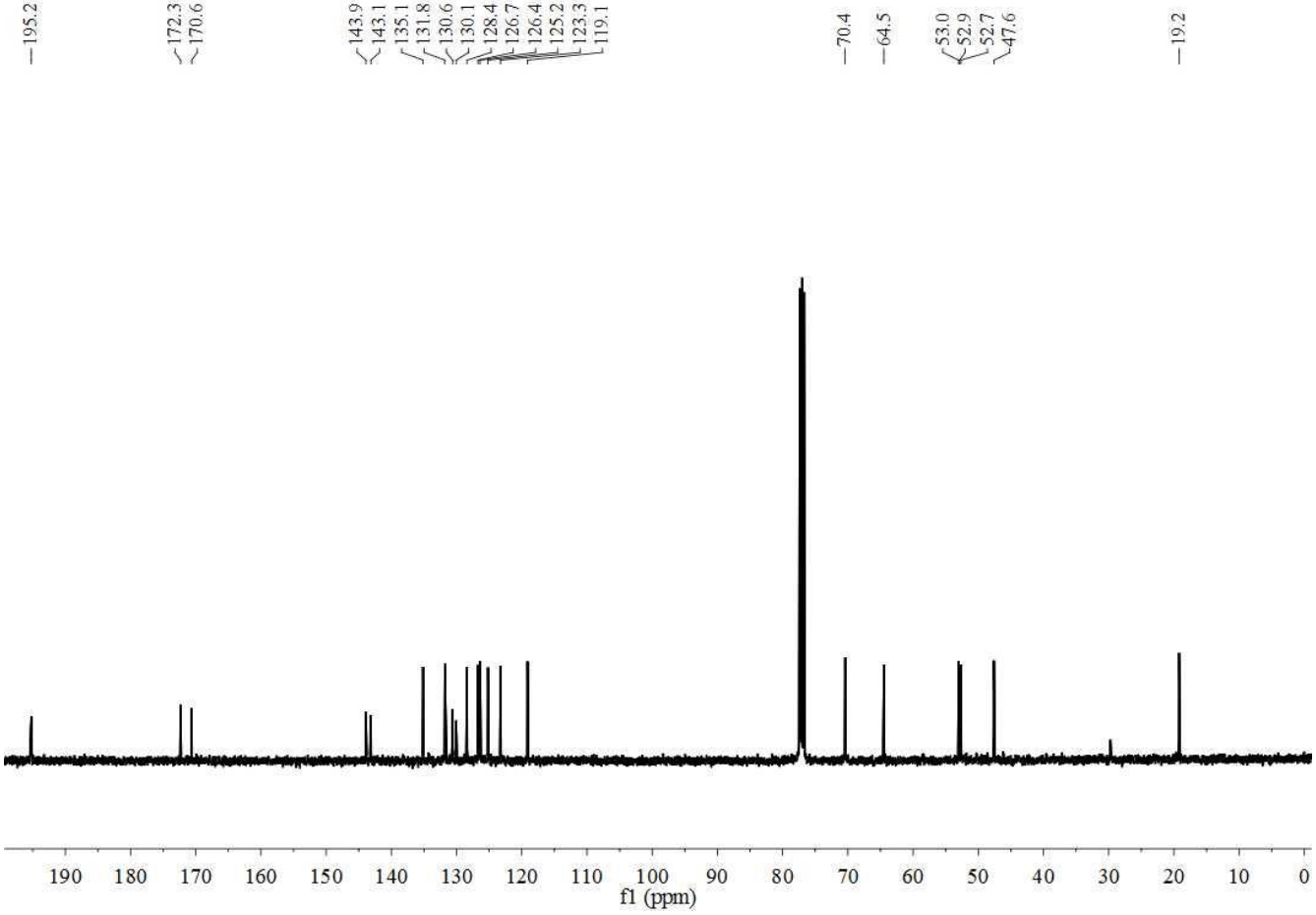

7.94
7.92
7.44
7.31
7.12
7.10
7.04
7.03
6.83
6.81
6.49
5.08
5.06
4.67
4.18
4.16
4.15
3.72
3.49
3.48
3.45
2.24
CO2Me
ArN
CO2Me
O
Ar = 2-MeC6H4
1.00
1.09
1.16
1.05
1.08
2.12
1.06
1.04
1.02
1.05
3.07
1.05
2.93
3.09
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)
194.0
171.3
169.1
142.4
139.8
133.3
130.8
129.7
128.7
127.8
126.2
125.5
125.4
122.3
118.0
69.4
63.3
51.9
51.2
50.8
45.1
18.2
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

3d'



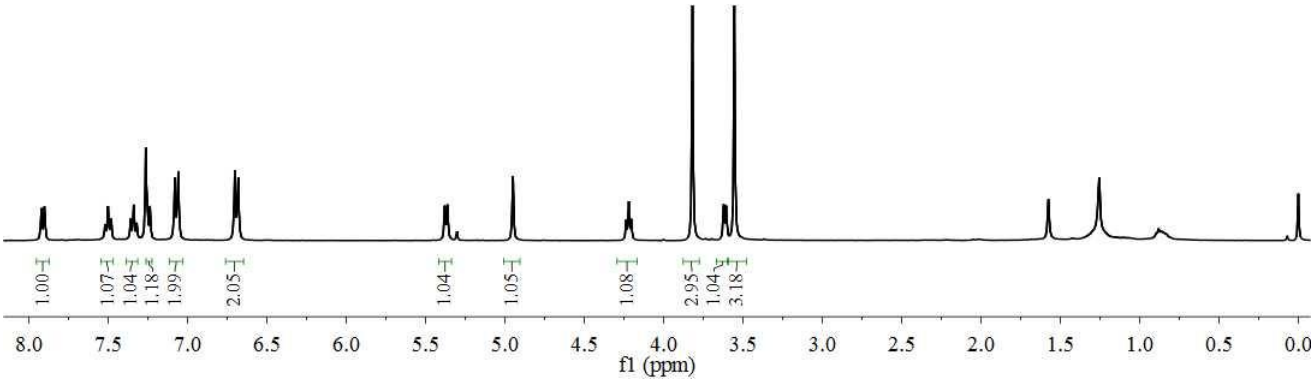
Ar = 2-MeC_6H_4

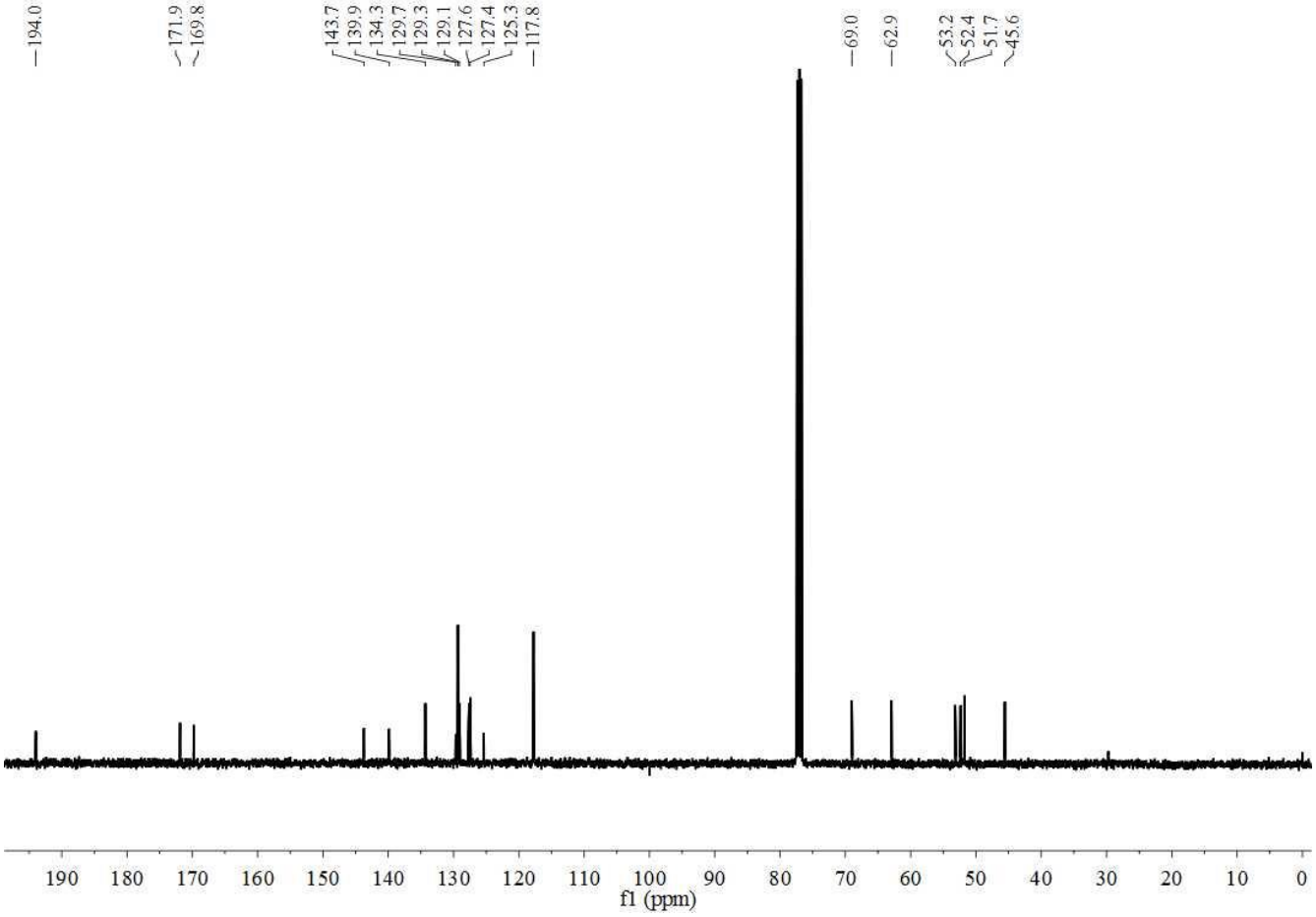




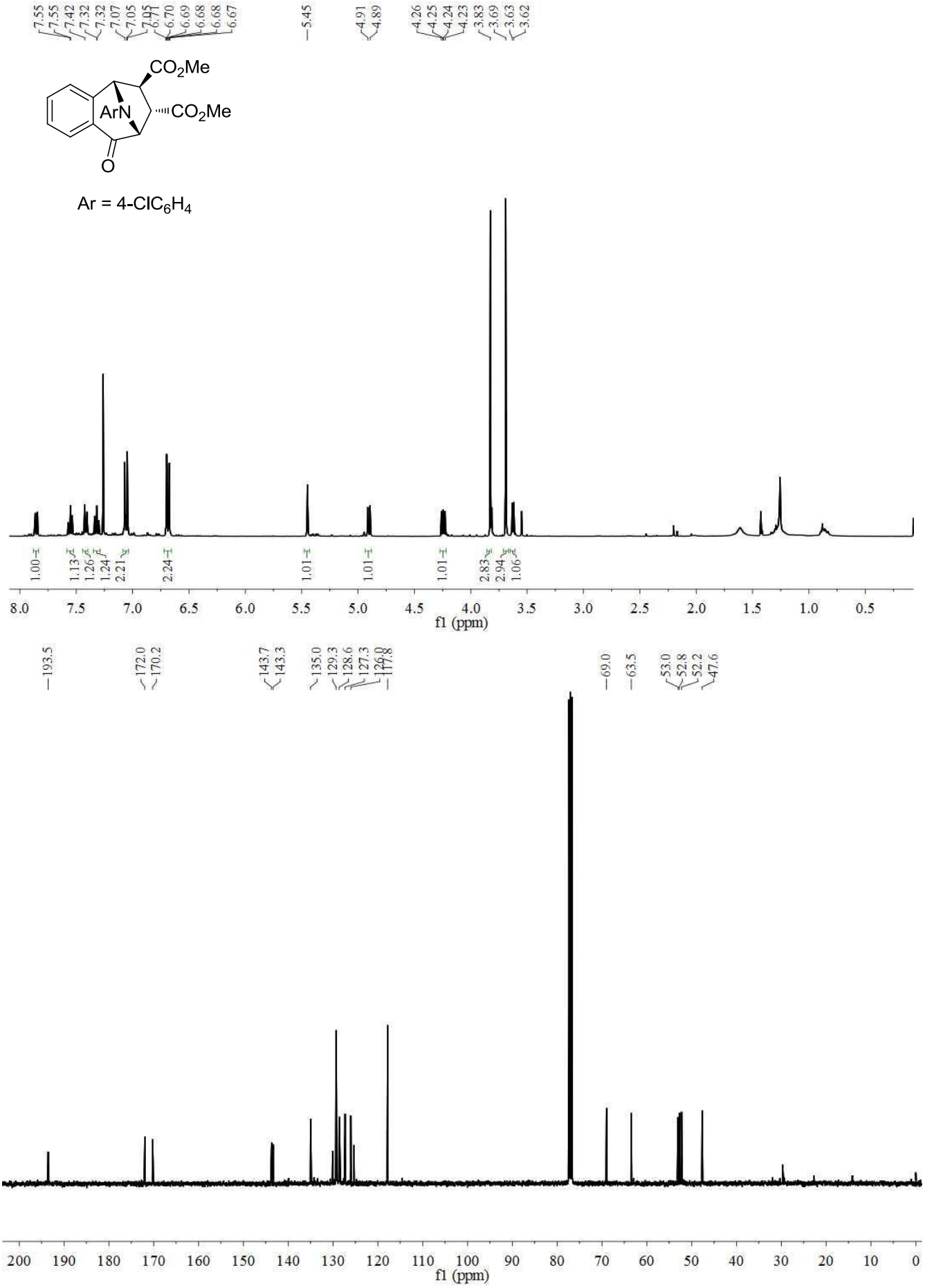
3e

Ar = 4-ClC_6H_4

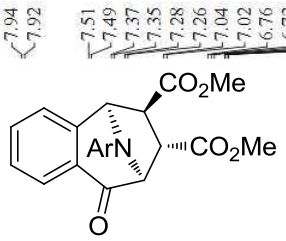




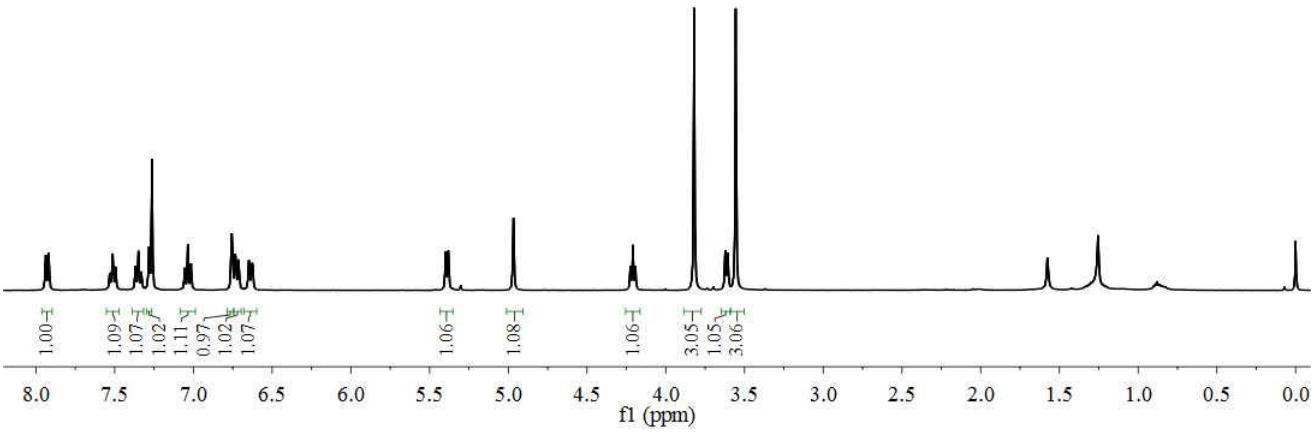
3e'

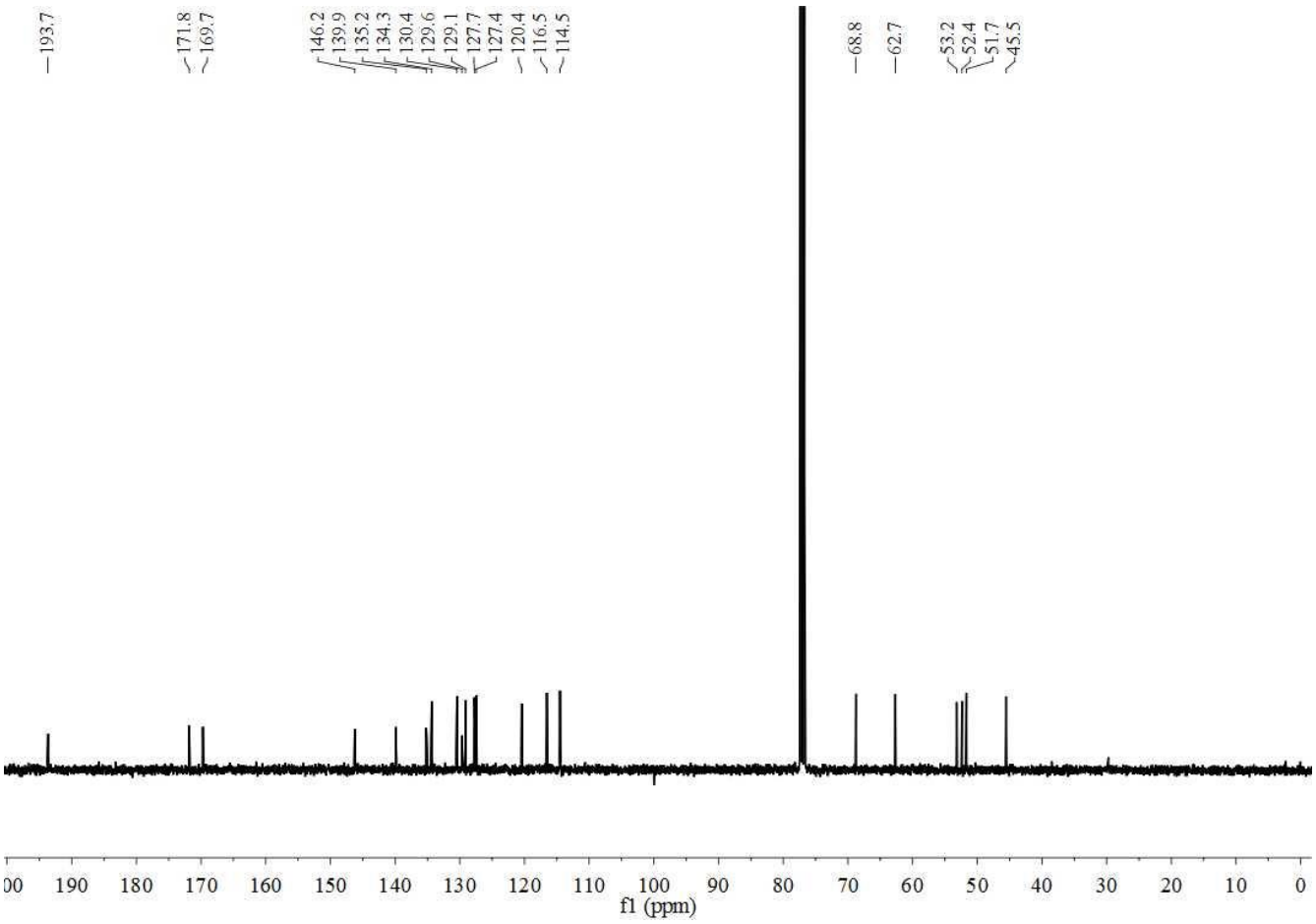

7.55
7.55
7.42
7.32
7.32
7.07
7.05
6.71
6.70
6.69
6.68
6.68
6.67
5.45
4.91
4.89
4.26
4.25
4.24
4.23
3.83
3.69
3.63
3.62
CO2Me
ArN
CO2Me
O
Ar = 4-ClC6H4
1.00
1.13
1.26
1.24
2.21
2.24
1.01
1.01
1.01
2.83
2.94
1.06
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)
193.5
172.0
170.2
143.7
143.3
135.0
129.3
128.6
127.3
126.0
117.8
69.0
63.5
53.0
52.8
52.2
47.6
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

3f

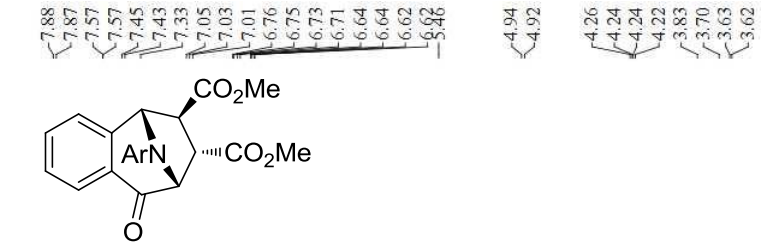


Ar = 3-ClC_6H_4

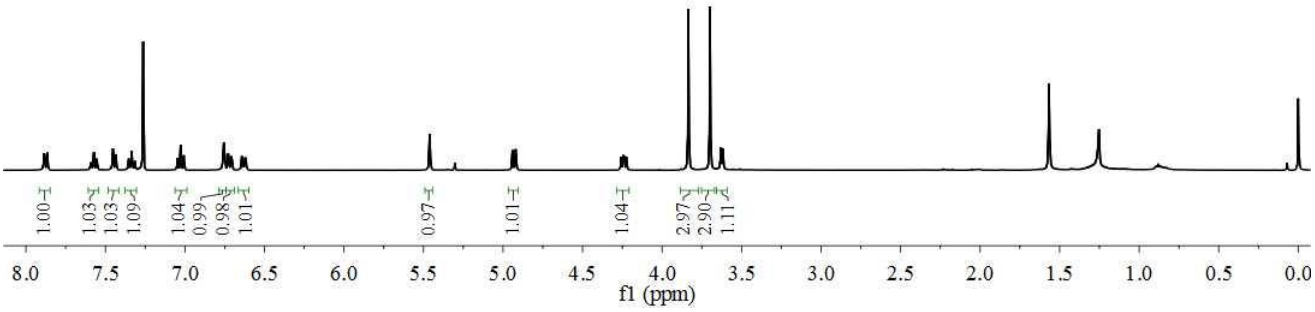


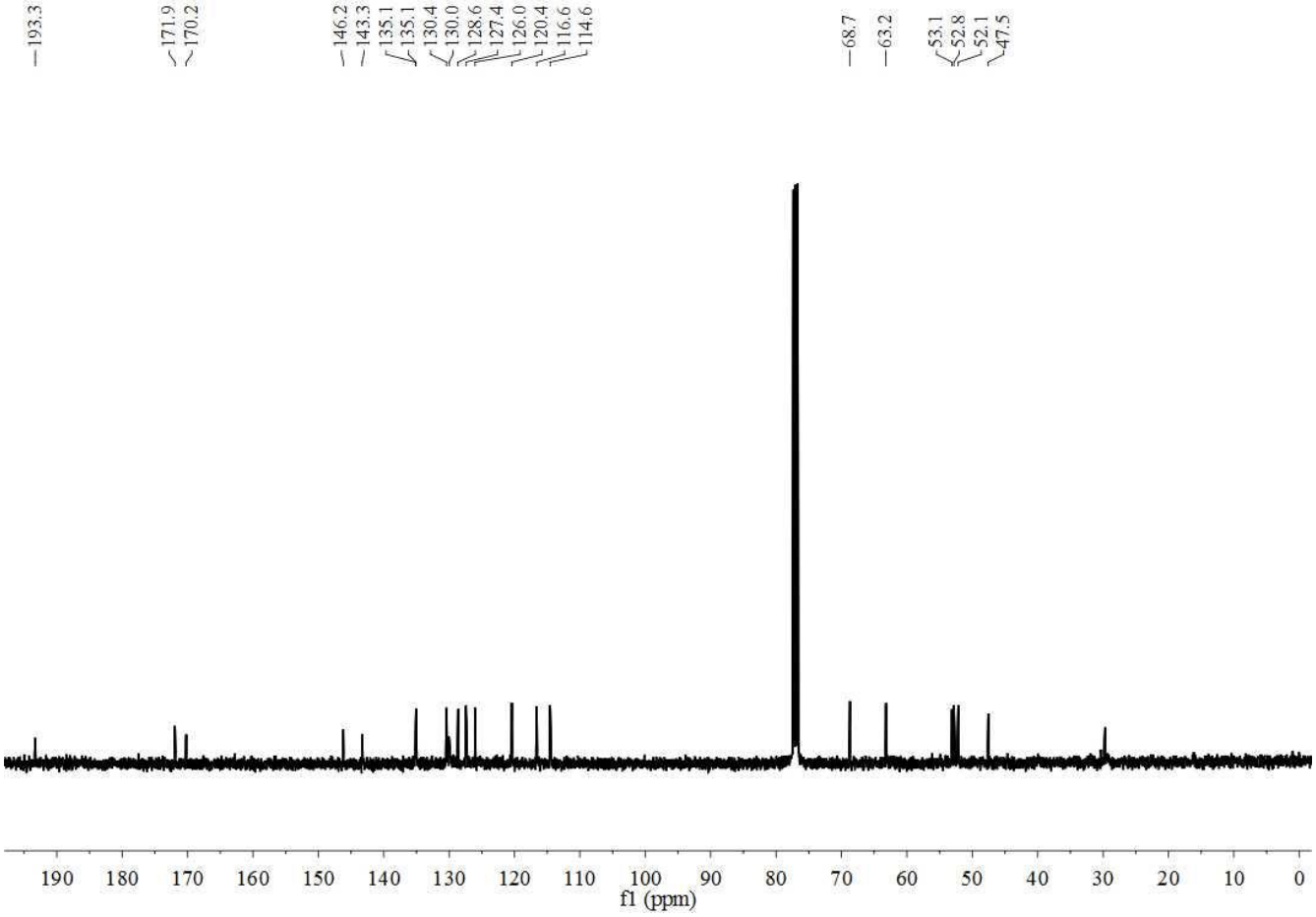


3f'

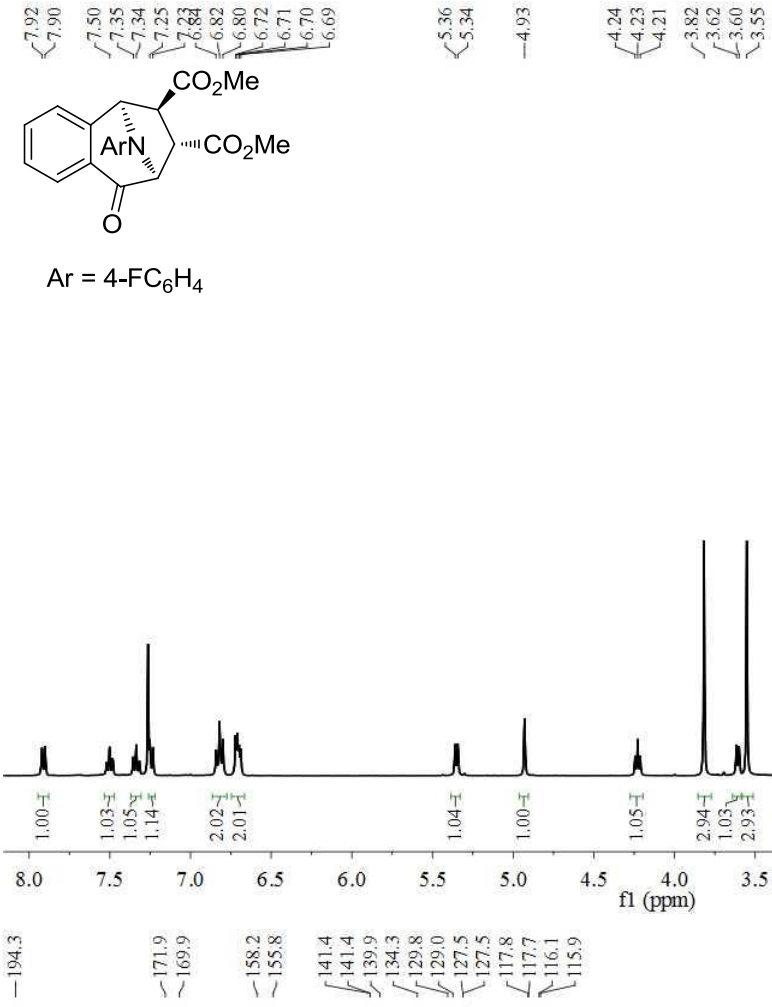


Ar = 3-ClC_6H_4

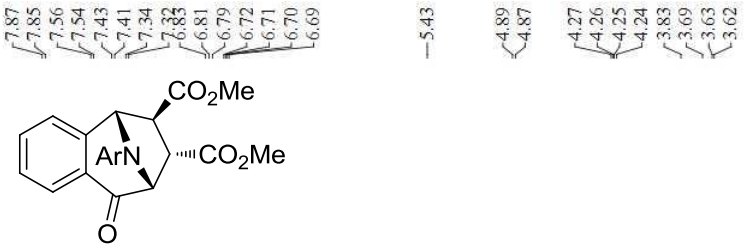




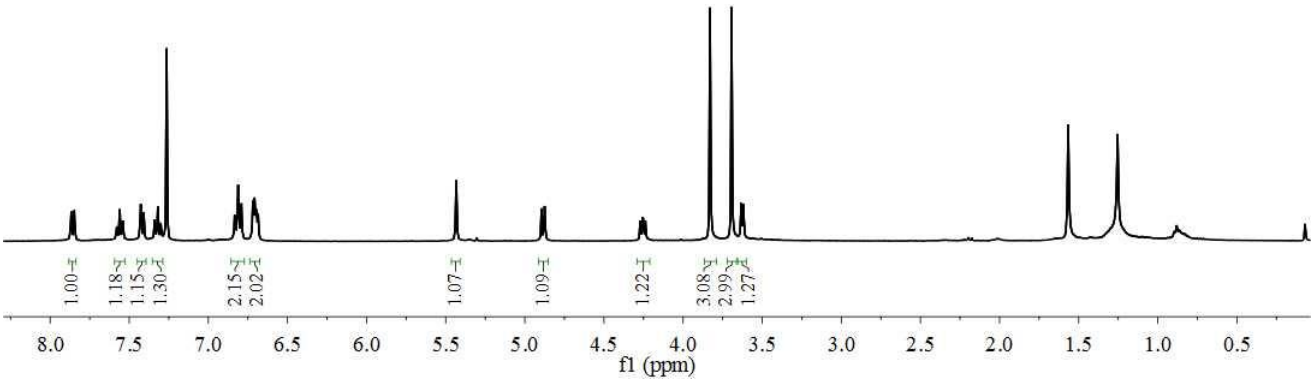
3g

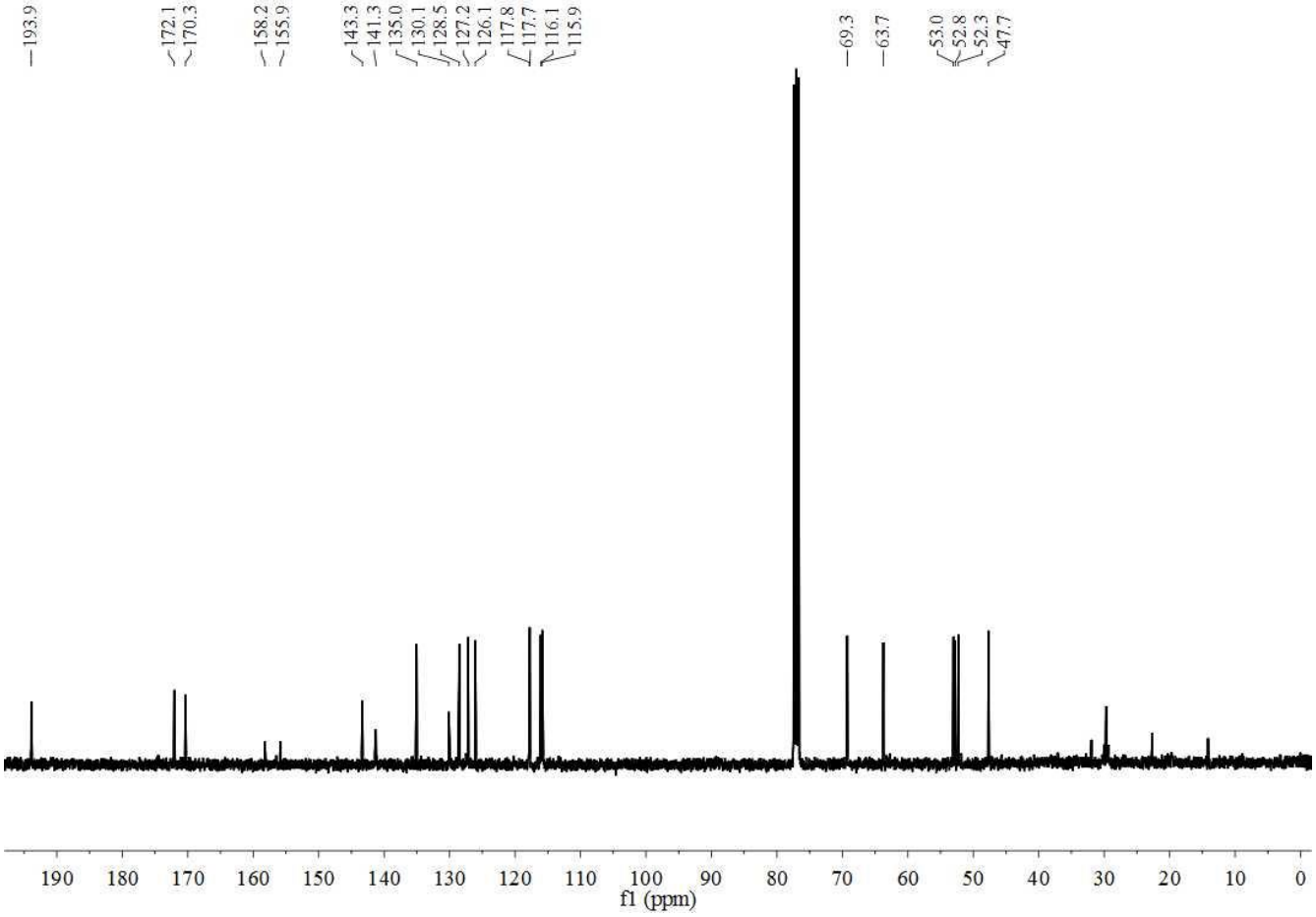

7.92
7.90
7.50
7.35
7.34
7.25
6.82
6.80
6.72
6.71
6.70
6.69
5.36
5.34
4.93
4.24
4.23
4.21
3.82
3.62
3.60
3.55
CO2Me
ArN
CO2Me
O
Ar = 4-FC6H4
1.00
1.03
1.05
1.14
2.02
2.01
1.04
1.00
1.05
2.94
1.03
2.93
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)
194.3
171.9
169.9
158.2
155.8
141.4
141.4
139.9
134.3
129.8
129.0
127.5
127.5
117.8
117.7
116.1
115.9
69.3
63.2
53.1
52.3
51.8
45.7
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

3g'

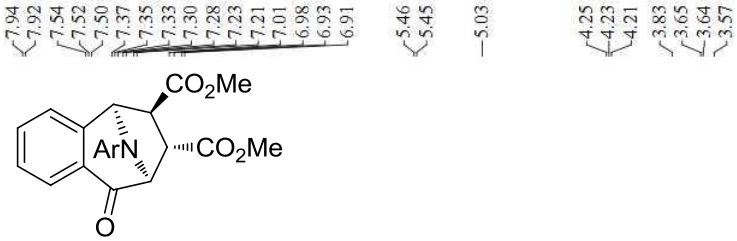

7.87
7.85
7.56
7.54
7.43
7.41
7.34
7.32
6.83
6.81
6.79
6.72
6.71
6.70
6.69
5.43
4.89
4.87
4.27
4.26
4.25
4.24
3.83
3.69
3.63
3.62
CO2Me
ArN
CO2Me
O

Ar = 4-FC_6H_4

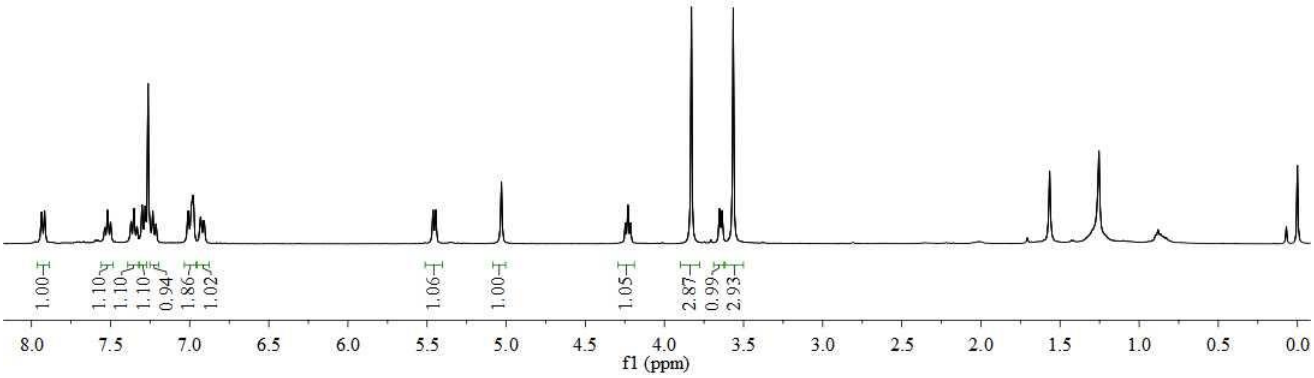

1.00
1.18
1.15
1.30
2.15
2.02
1.07
1.09
1.22
3.08
2.99
1.27
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)

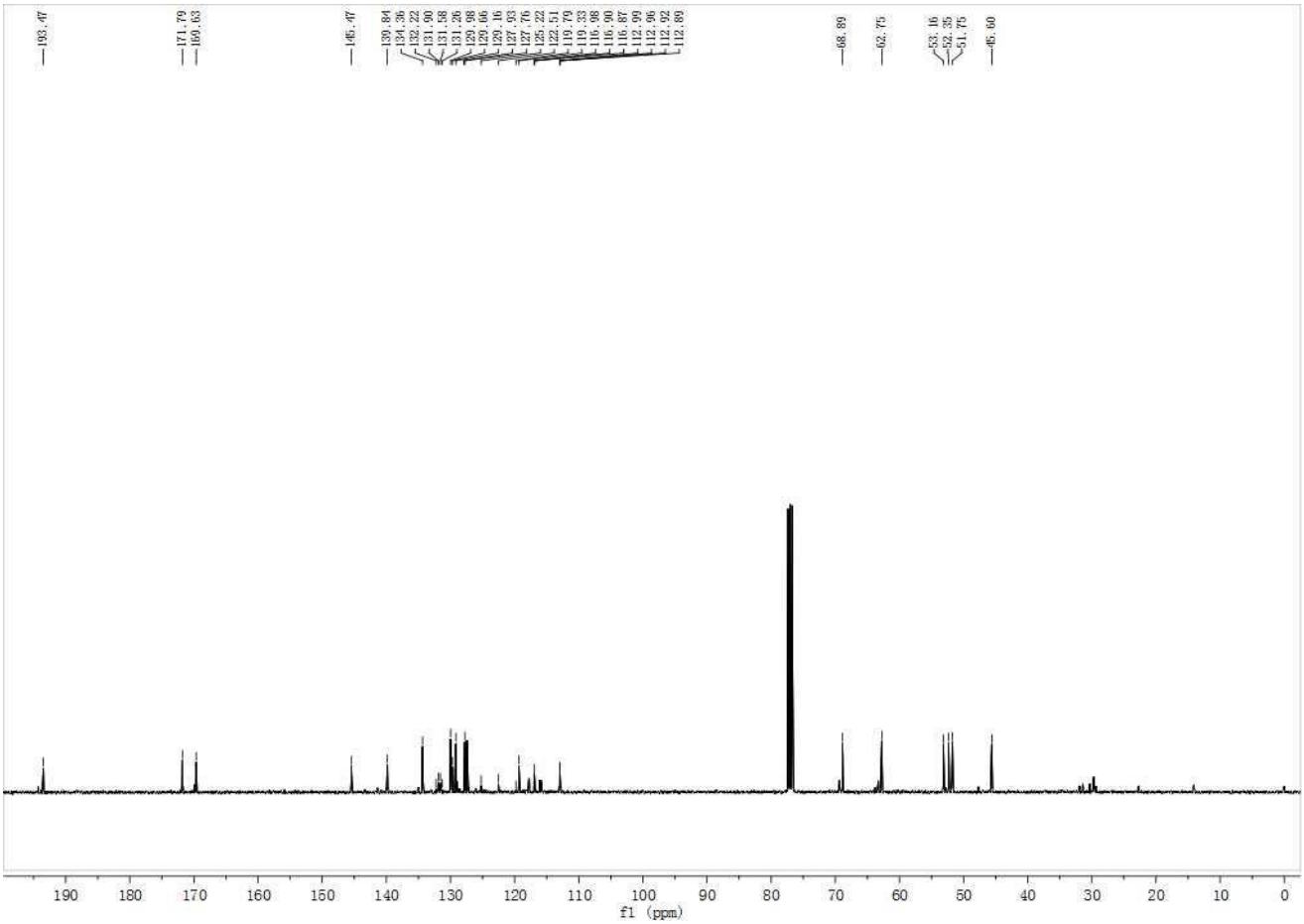

193.9
172.1
170.3
158.2
155.9
143.3
141.3
135.0
130.1
128.5
127.2
126.1
117.8
117.7
116.1
115.9
69.3
63.7
53.0
52.8
52.3
47.7
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

3h

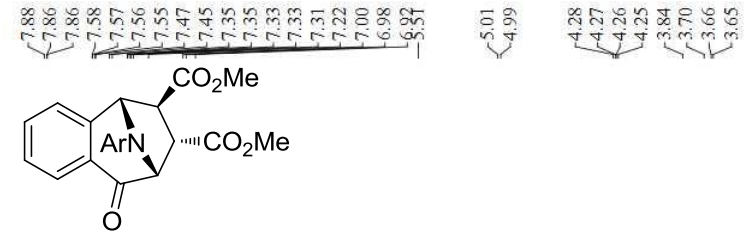


Ar = 3-$CF_3C_6H_4$

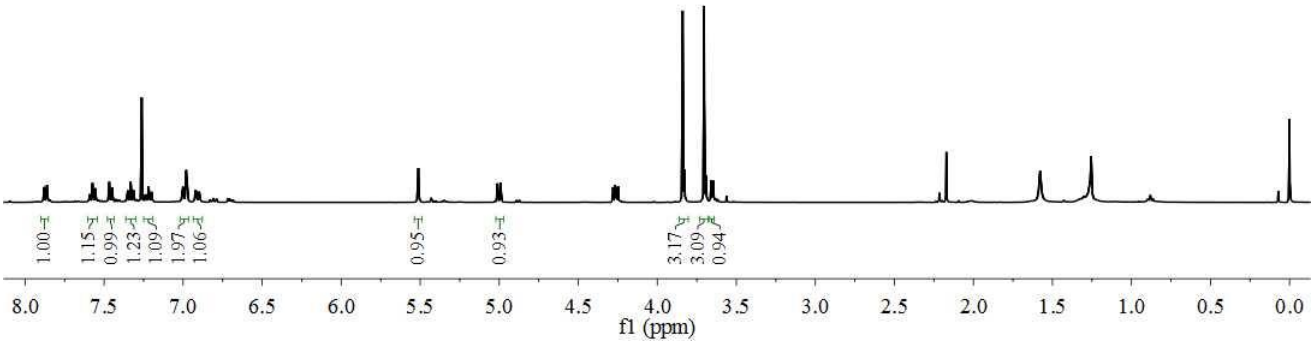


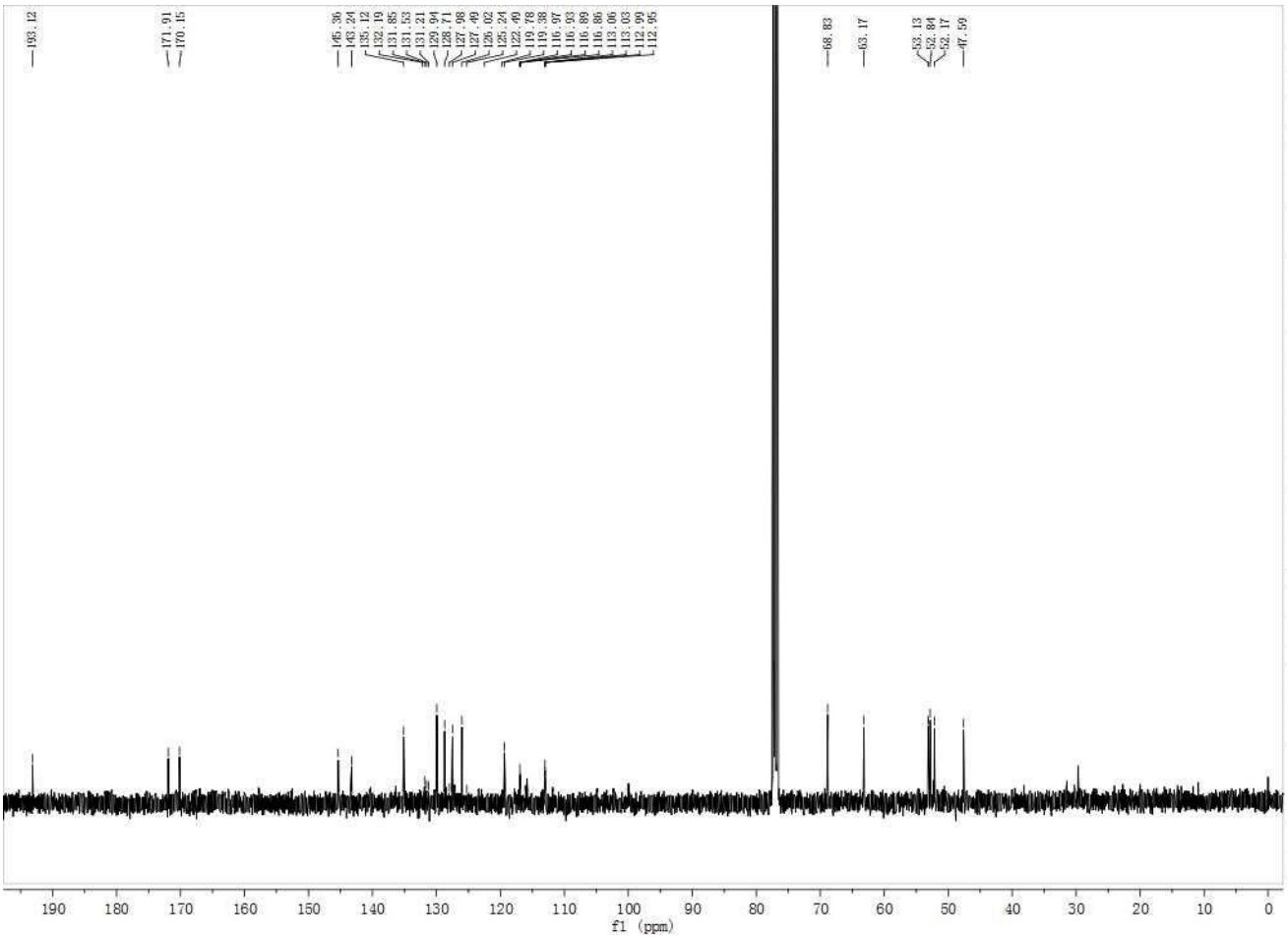


3h'

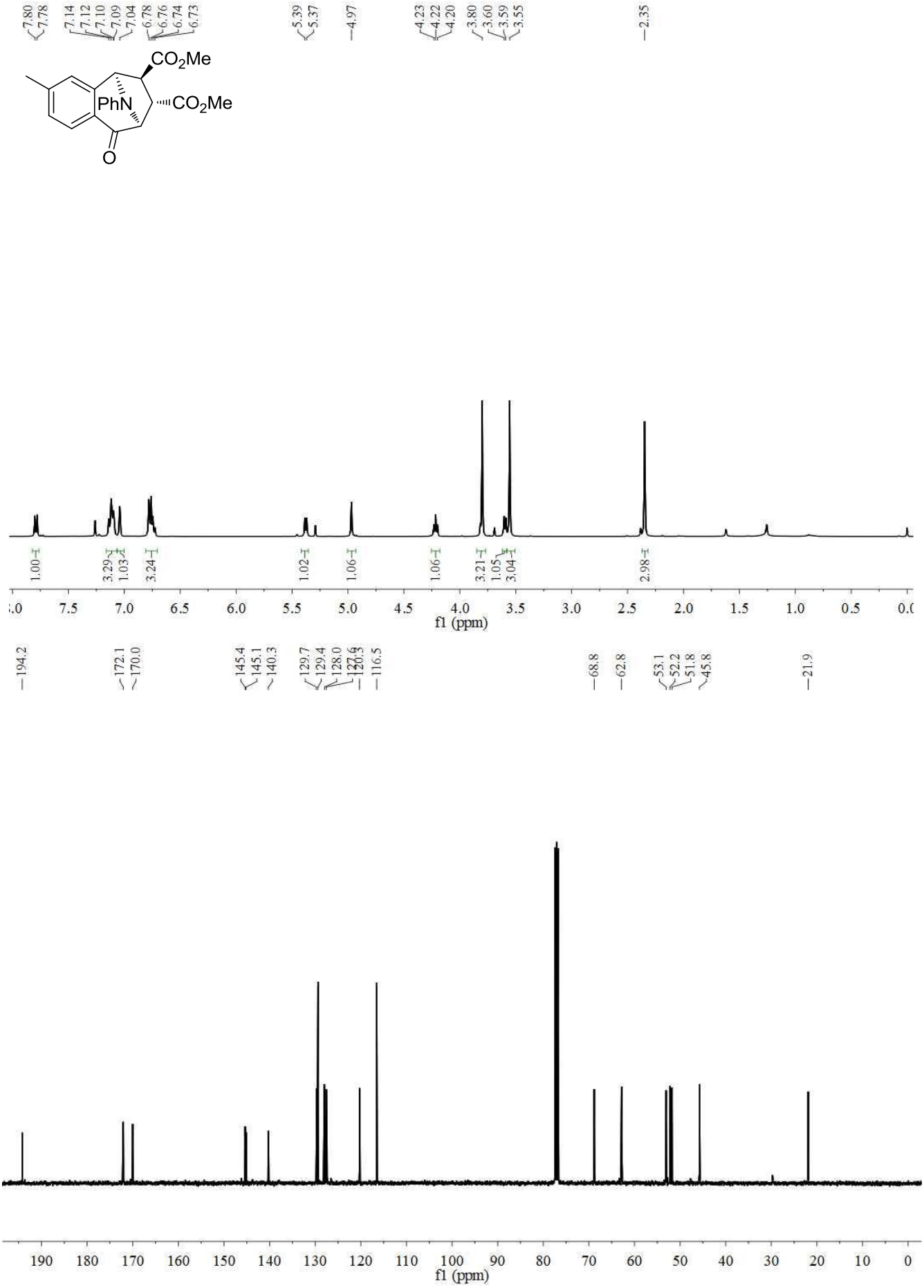


Ar = 3-$CF_3C_6H_4$

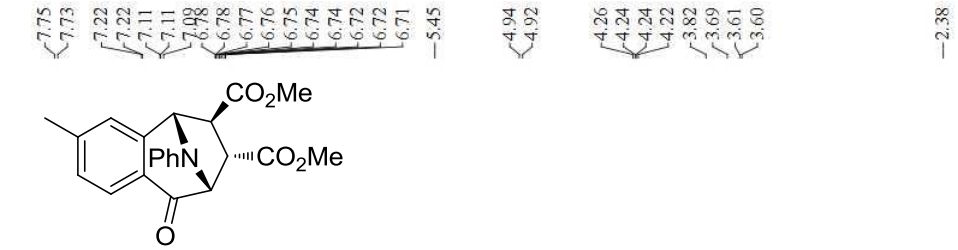


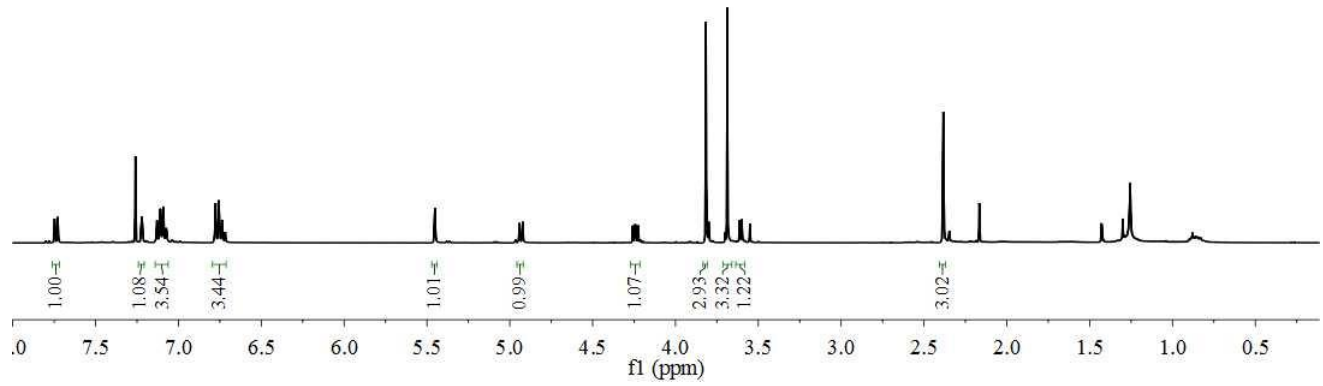


3i


7.80
7.78
7.14
7.12
7.10
7.09
7.04
6.78
6.76
6.74
6.73
5.39
5.37
4.97
4.23
4.22
4.20
3.80
3.60
3.59
3.55
2.35
CO2Me
PhN
CO2Me
O
1.00
3.29
1.03
3.24
1.02
1.06
1.06
3.21
1.05
3.04
2.98
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)
194.2
172.1
170.0
145.4
145.1
140.3
129.7
129.4
128.0
116.5
68.8
62.8
53.1
52.2
51.8
45.8
21.9
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

3i'


7.75
7.73
7.22
7.22
7.11
7.11
7.09
6.78
6.78
6.77
6.76
6.75
6.74
6.74
6.72
6.72
6.71
5.45
4.94
4.92
4.26
4.24
4.24
4.22
3.82
3.69
3.61
3.60
2.38
CO2Me
PhN
CO2Me
O


1.00
1.08
3.54
3.44
1.01
0.99
1.07
2.93
3.32
1.22
3.02
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)

193.7
172.2
170.5
146.1
145.1
143.8
129.3
129.3
127.8
127.3
126.5
120.2
116.6
68.8
63.4
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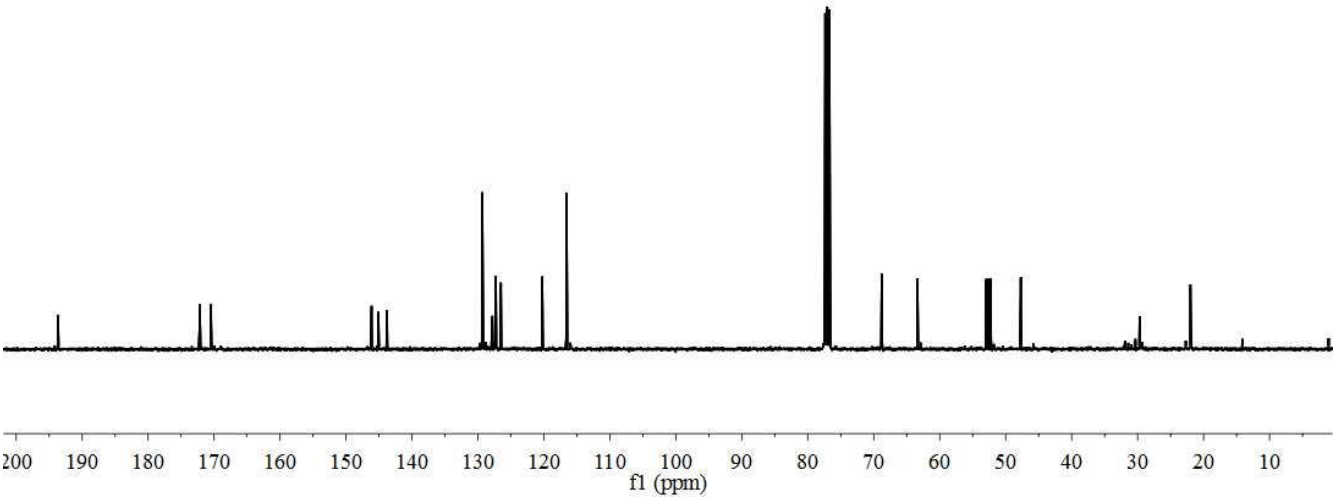
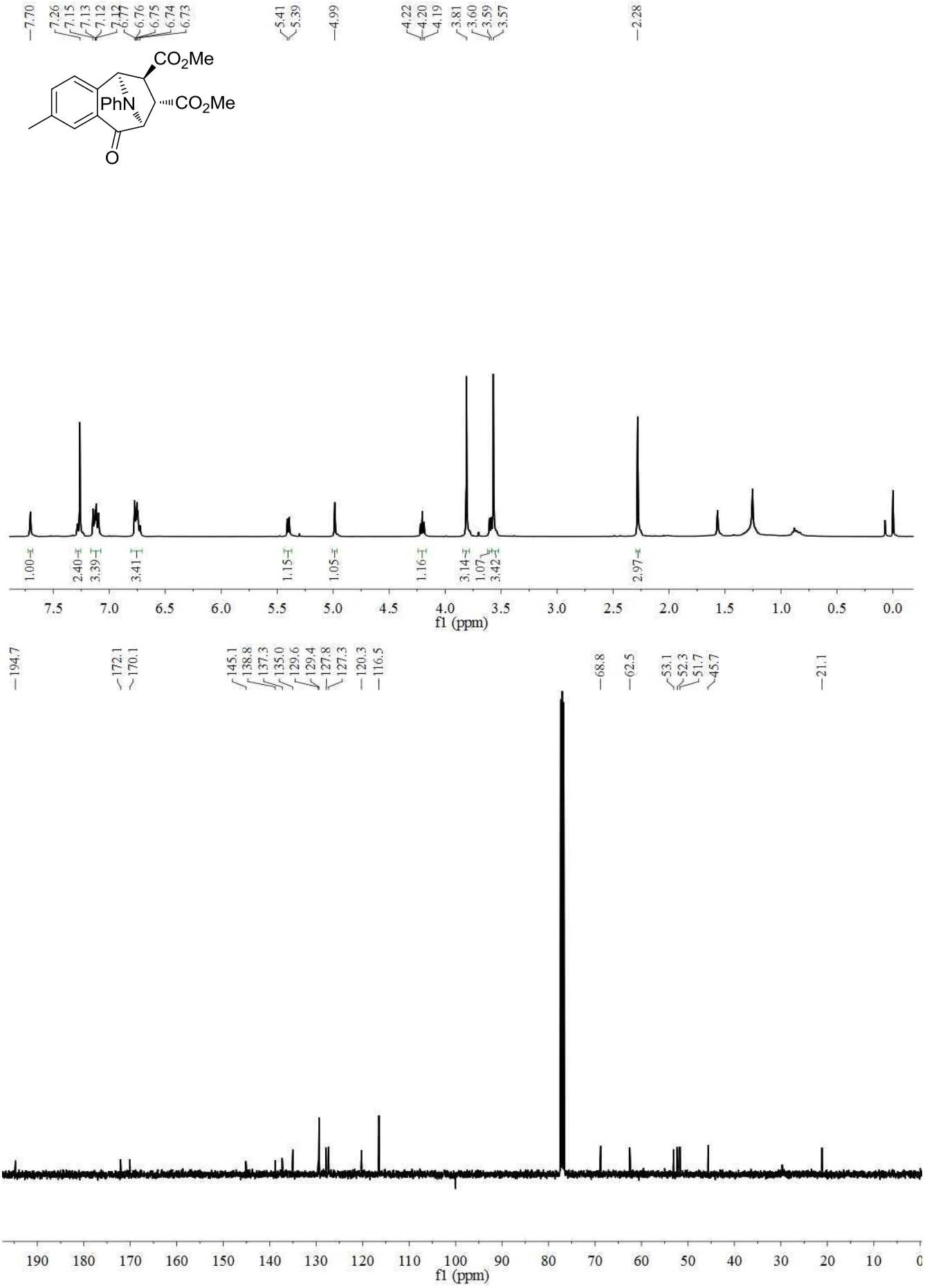
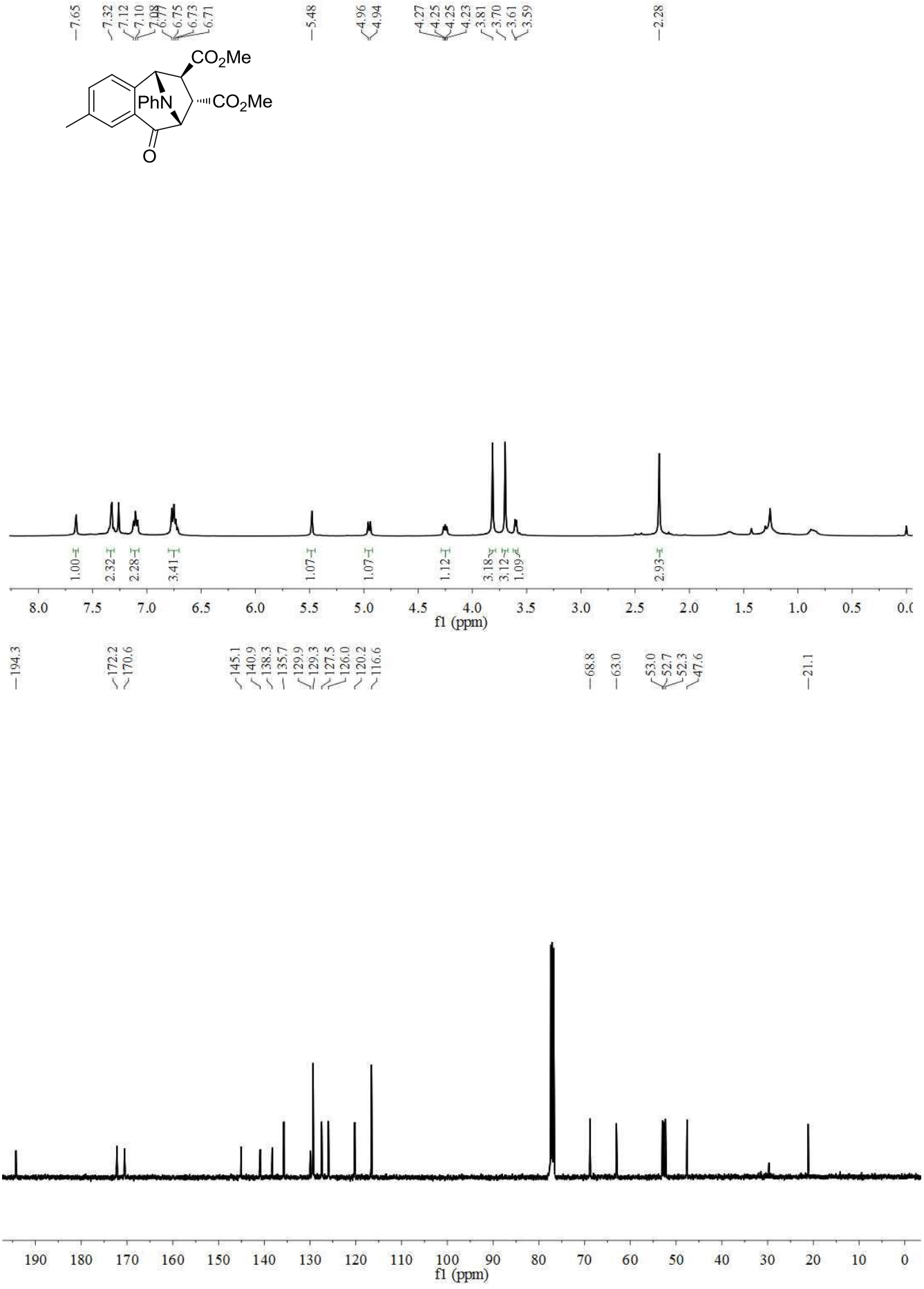
52.3
47.7
22.0


190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
f1 (ppm)

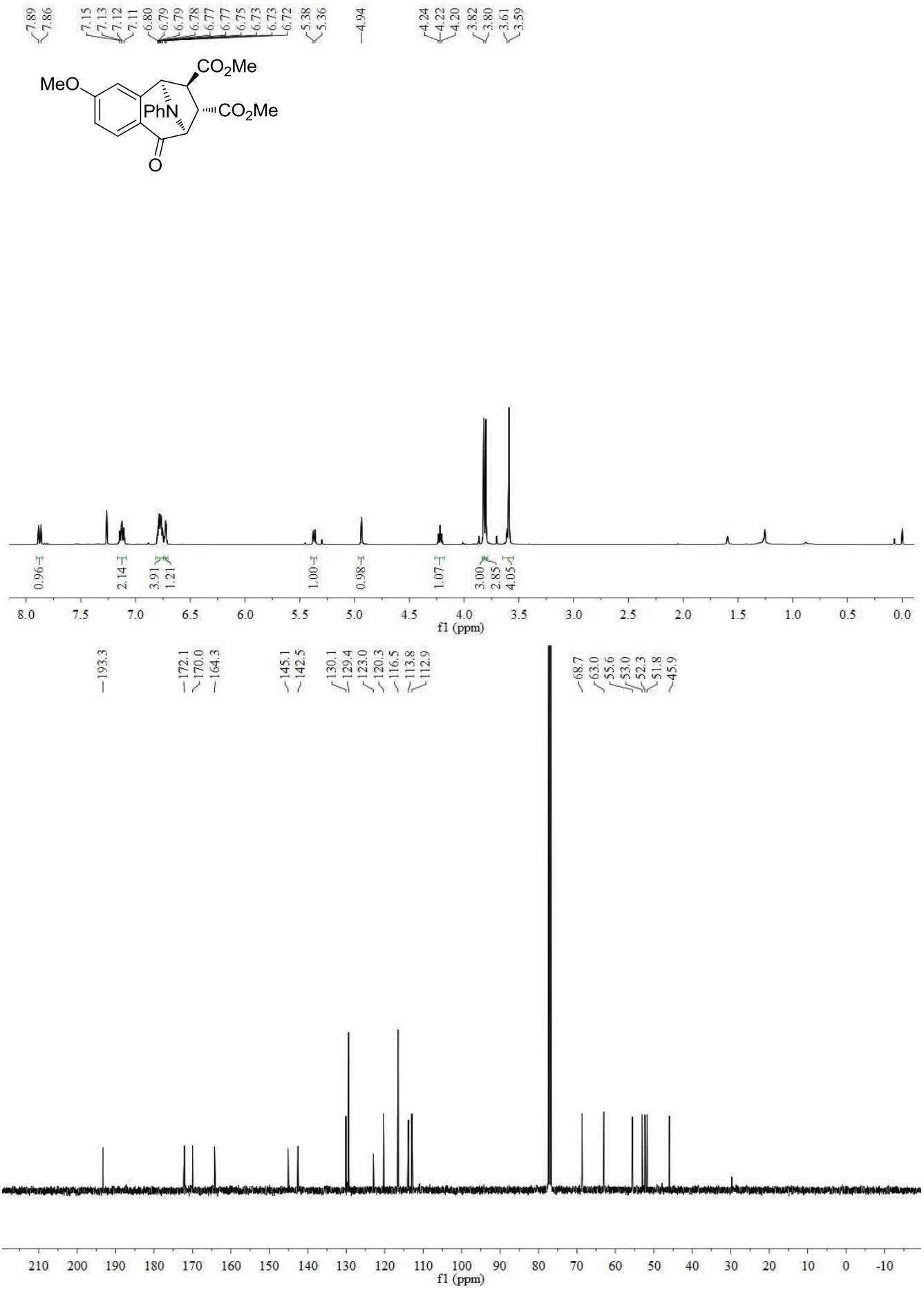
3j


7.70
7.26
7.15
7.13
7.12
7.12
6.77
6.76
6.75
6.74
6.73
5.41
5.39
4.99
4.22
4.20
4.19
3.81
3.60
3.59
3.57
2.28
CO2Me
PhN
CO2Me
O
1.00
2.40
3.39
3.41
1.15
1.05
1.16
3.14
1.07
3.42
2.97
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)
194.7
172.1
170.1
145.1
138.8
137.3
135.0
129.6
129.4
127.8
127.3
120.3
116.5
68.8
62.5
53.1
52.3
51.7
45.7
21.1
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

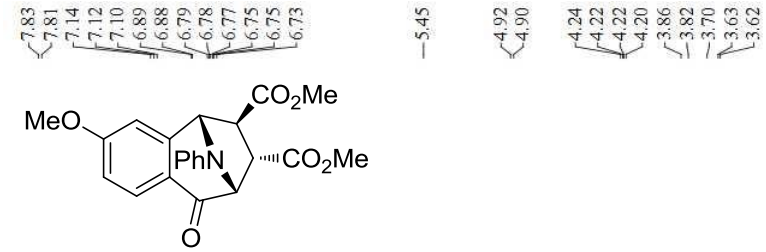
3j'

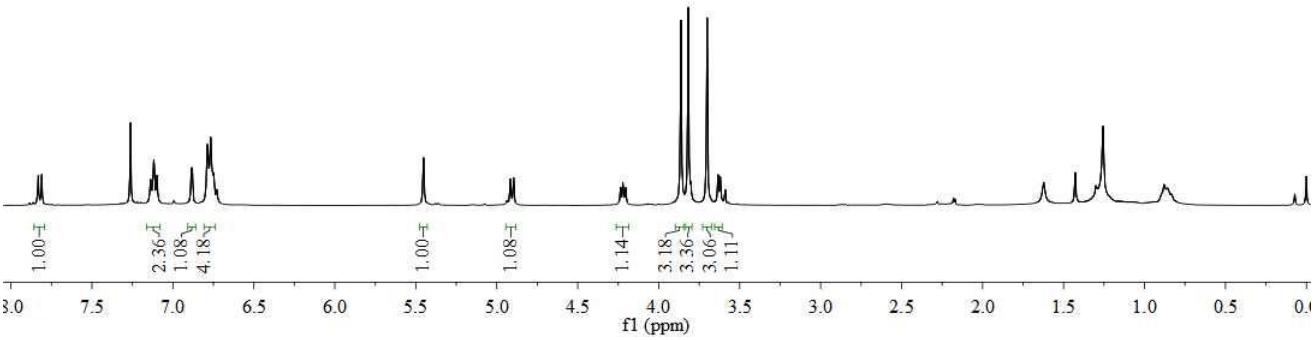

7.65
7.32
7.12
7.10
7.08
6.77
6.75
6.73
6.71
5.48
4.96
4.94
4.27
4.25
4.25
4.23
3.81
3.70
3.61
3.59
2.28
CO2Me
PhN
CO2Me
O
1.00
2.32
2.28
3.41
1.07
1.07
1.12
3.18
3.12
1.09
2.93
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)
194.3
172.2
170.6
145.1
140.9
138.3
135.7
129.9
129.3
127.5
126.0
120.2
116.6
68.8
63.0
53.0
52.7
52.3
47.6
21.1
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

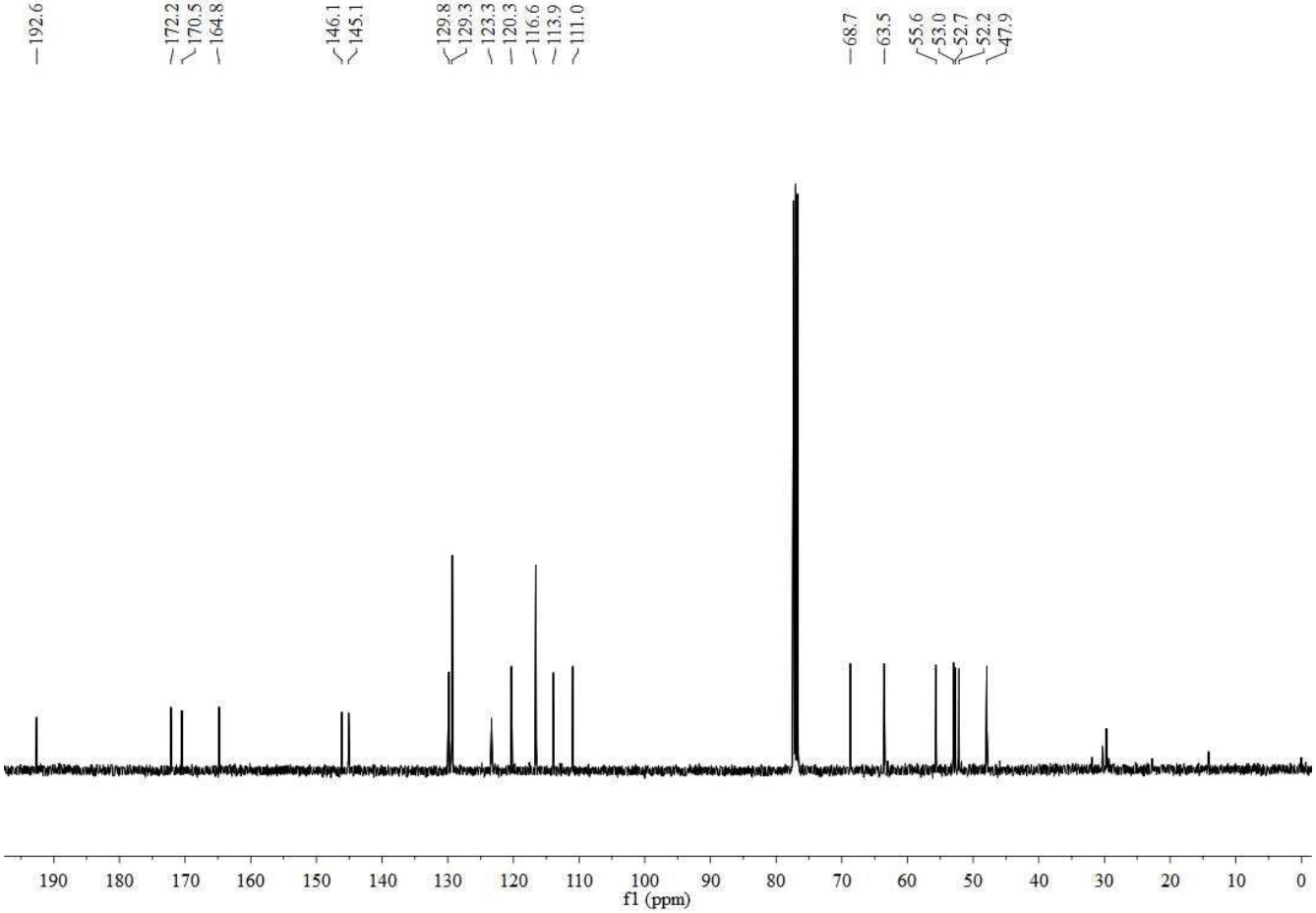
3k


7.89
7.86
7.15
7.13
7.12
7.11
6.80
6.79
6.79
6.78
6.77
6.77
6.75
6.73
6.73
6.72
5.38
5.36
4.94
4.24
4.22
4.20
3.82
3.80
3.61
3.59
MeO
CO2Me
PhN
CO2Me
O
0.96
2.14
3.91
1.21
1.00
0.98
1.07
3.00
2.85
4.05
f1 (ppm)
193.3
172.1
170.0
164.3
145.1
142.5
130.1
129.4
123.0
120.3
116.5
113.8
112.9
68.7
63.0
55.6
53.0
52.3
51.8
45.9
f1 (ppm)

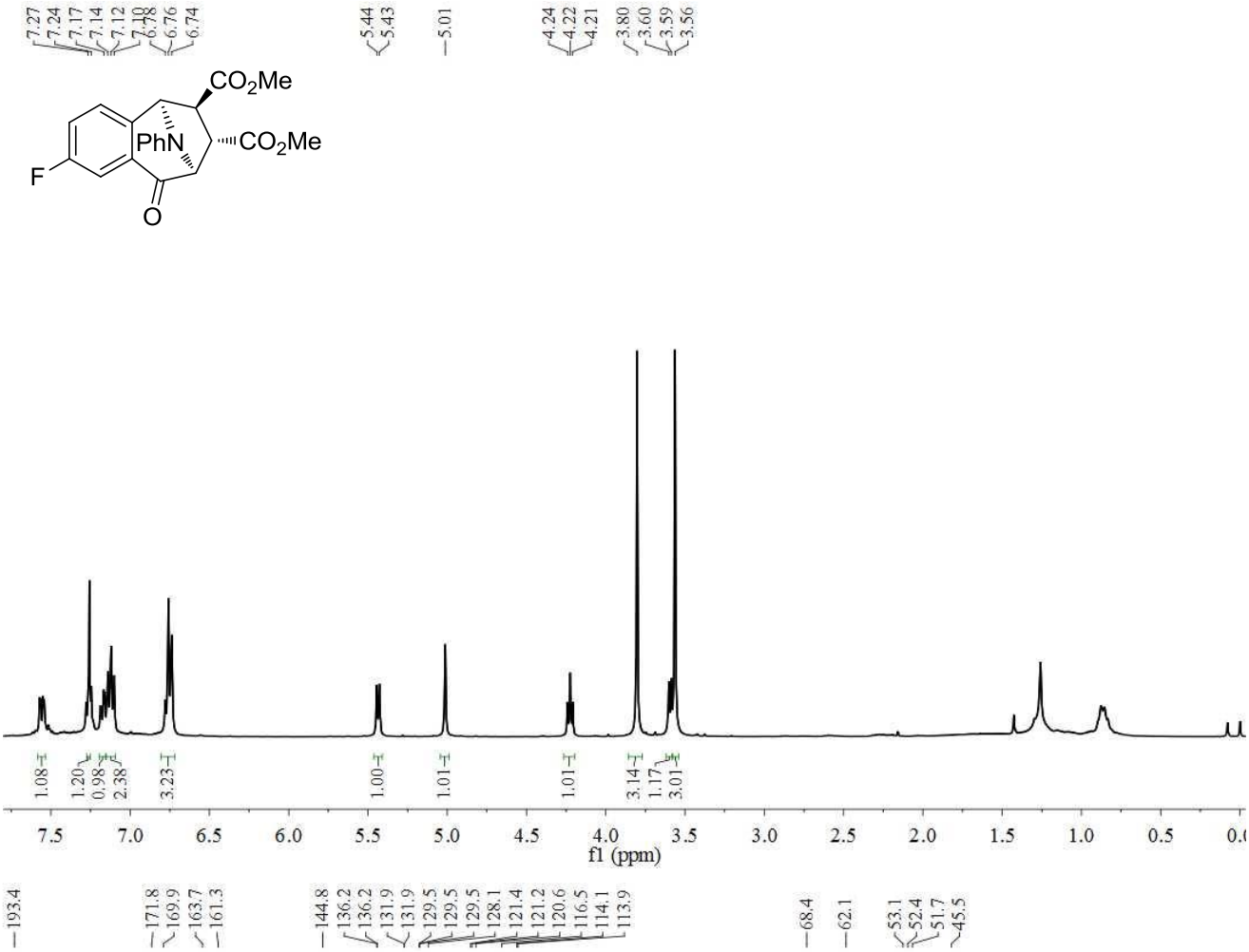
3k'

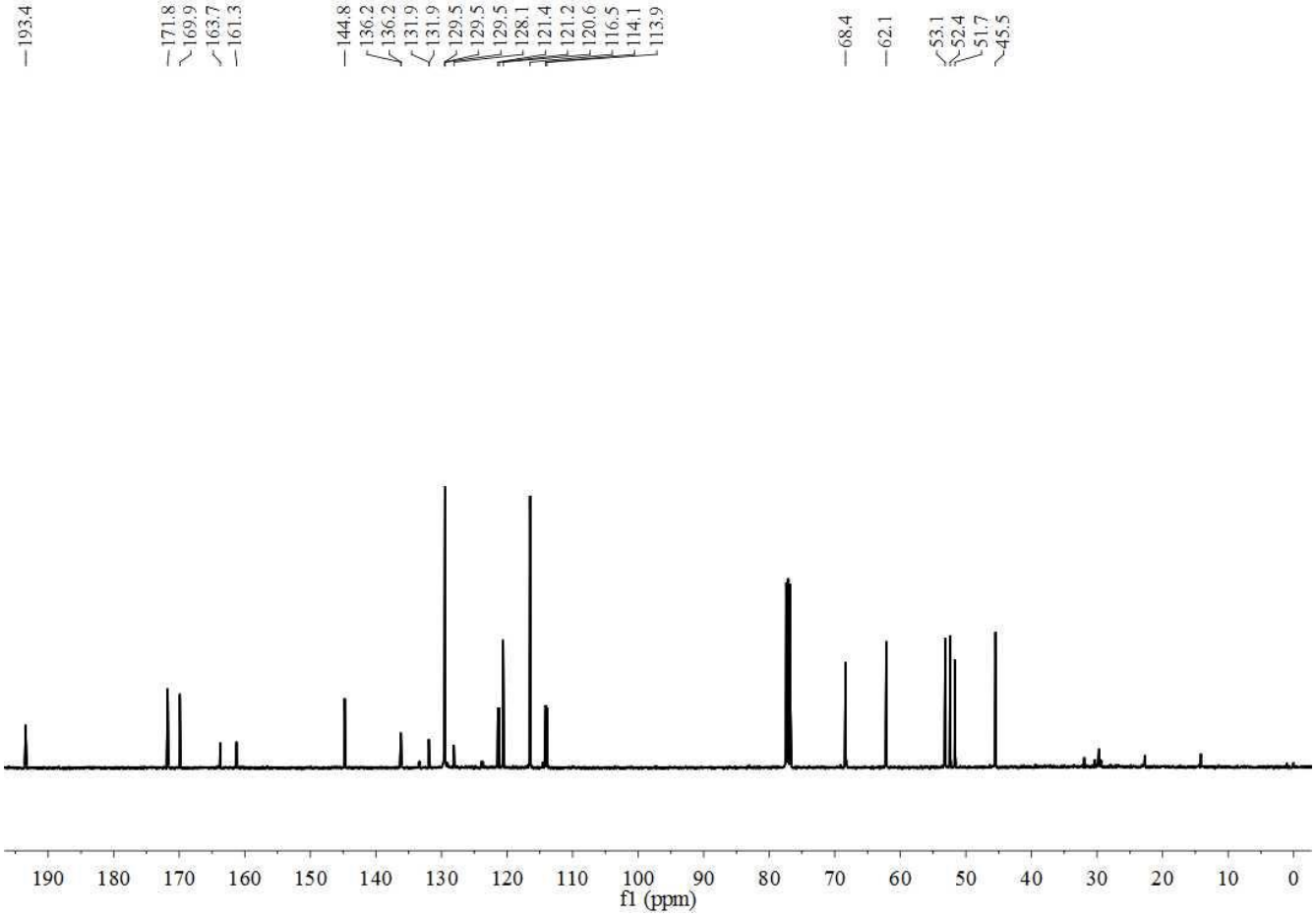

7.83
7.81
7.14
7.12
7.10
6.89
6.88
6.79
6.78
6.77
6.75
6.75
6.73
5.45
4.92
4.90
4.24
4.22
4.22
4.20
3.86
3.82
3.70
3.63
3.62
MeO
CO2Me
PhN
CO2Me
O


1.00
2.36
1.08
4.18
1.00
1.08
1.14
3.18
3.36
3.06
1.11
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

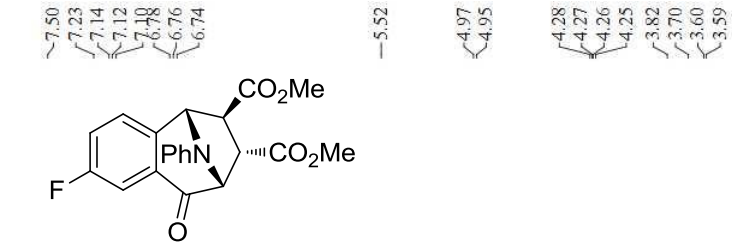

192.6
172.2
170.5
164.8
146.1
145.1
129.8
129.3
123.3
120.3
116.6
113.9
111.0
68.7
63.5
55.6
53.0
52.7
52.2
47.9
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

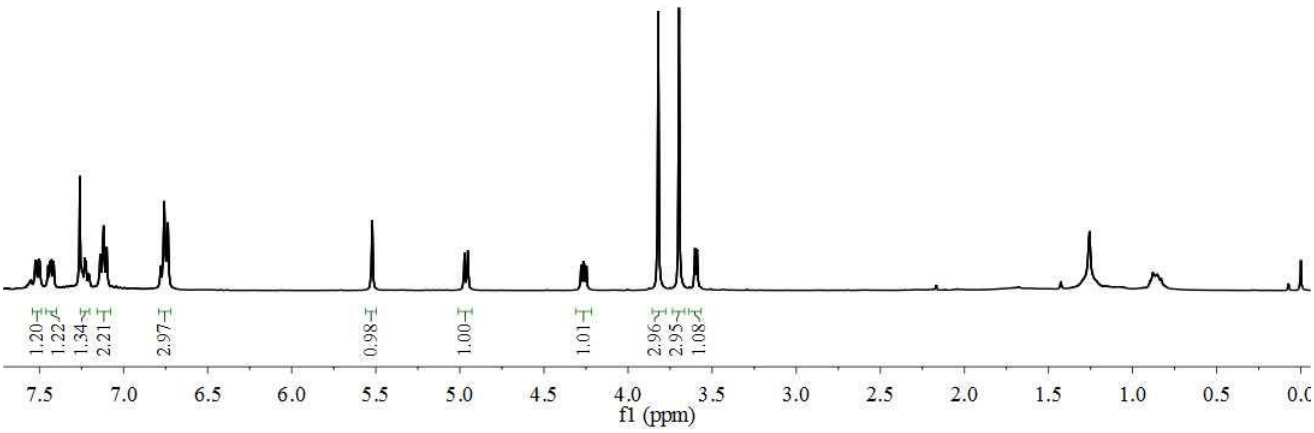
3l

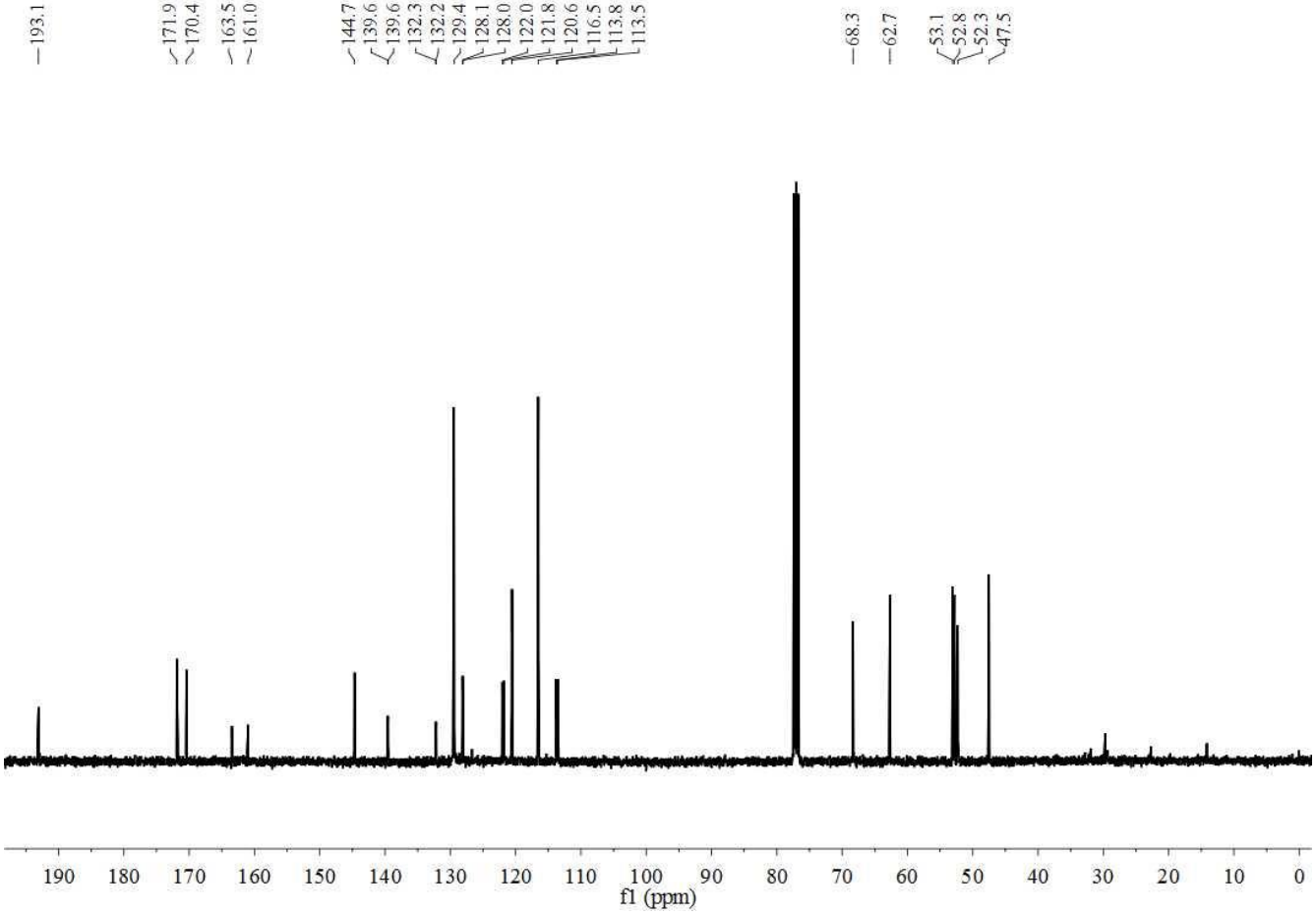

7.27
7.24
7.17
7.14
7.12
7.10
6.78
6.76
6.74
5.44
5.43
5.01
4.24
4.22
4.21
3.80
3.60
3.59
3.56
CO2Me
PhN
CO2Me
F
O
1.08
1.20
0.98
2.38
3.23
1.00
1.01
1.01
3.14
1.17
3.01
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)


193.4
171.8
169.9
163.7
161.3
144.8
136.2
136.2
131.9
131.9
129.5
129.5
129.5
128.1
121.4
121.2
120.6
116.5
114.1
113.9
68.4
62.1
53.1
52.4
51.7
45.5
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

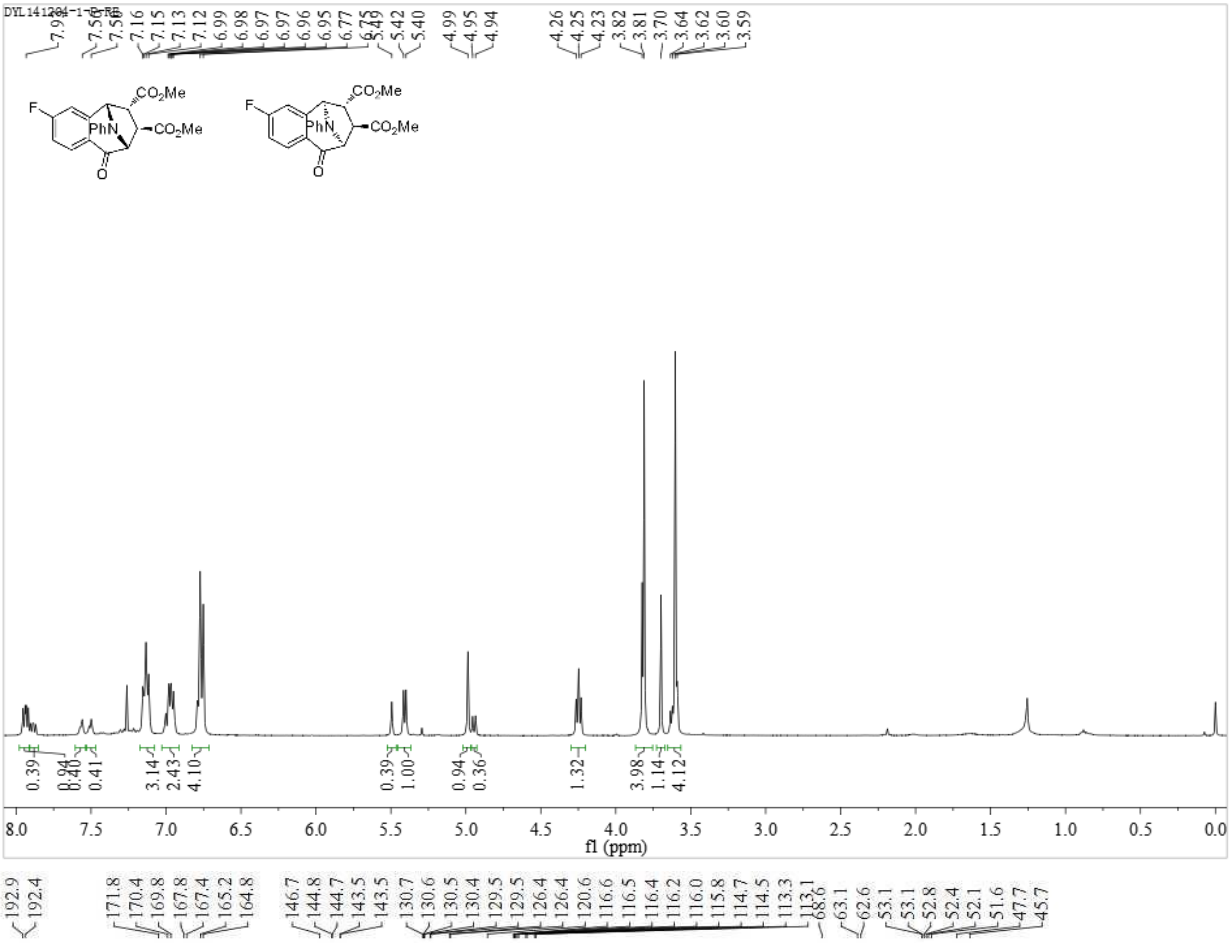
3l'

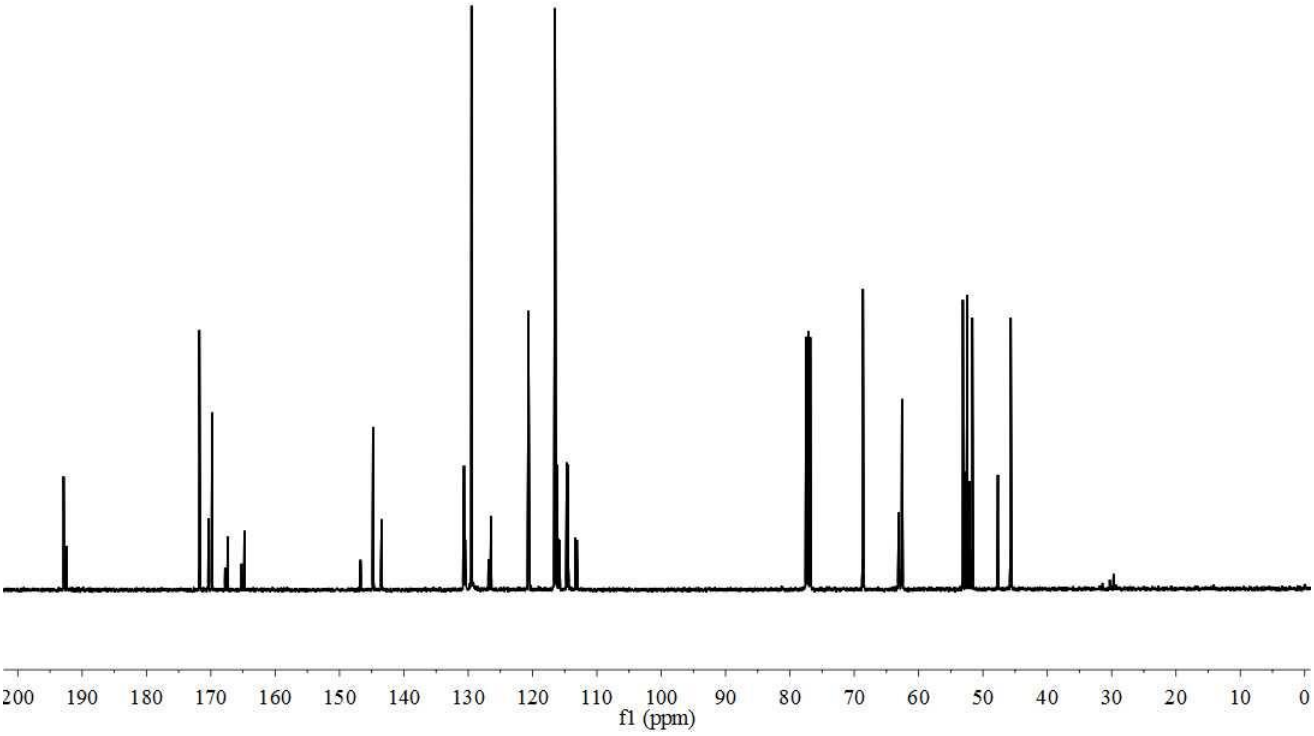

7.50
7.23
7.14
7.12
7.10
6.78
6.76
6.74
5.52
4.97
4.95
4.28
4.27
4.26
4.25
3.82
3.70
3.60
3.59
CO2Me
PhN
CO2Me
F
O


1.20
1.22
1.34
2.21
2.97
0.98
1.00
1.01
2.96
2.95
1.08
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)

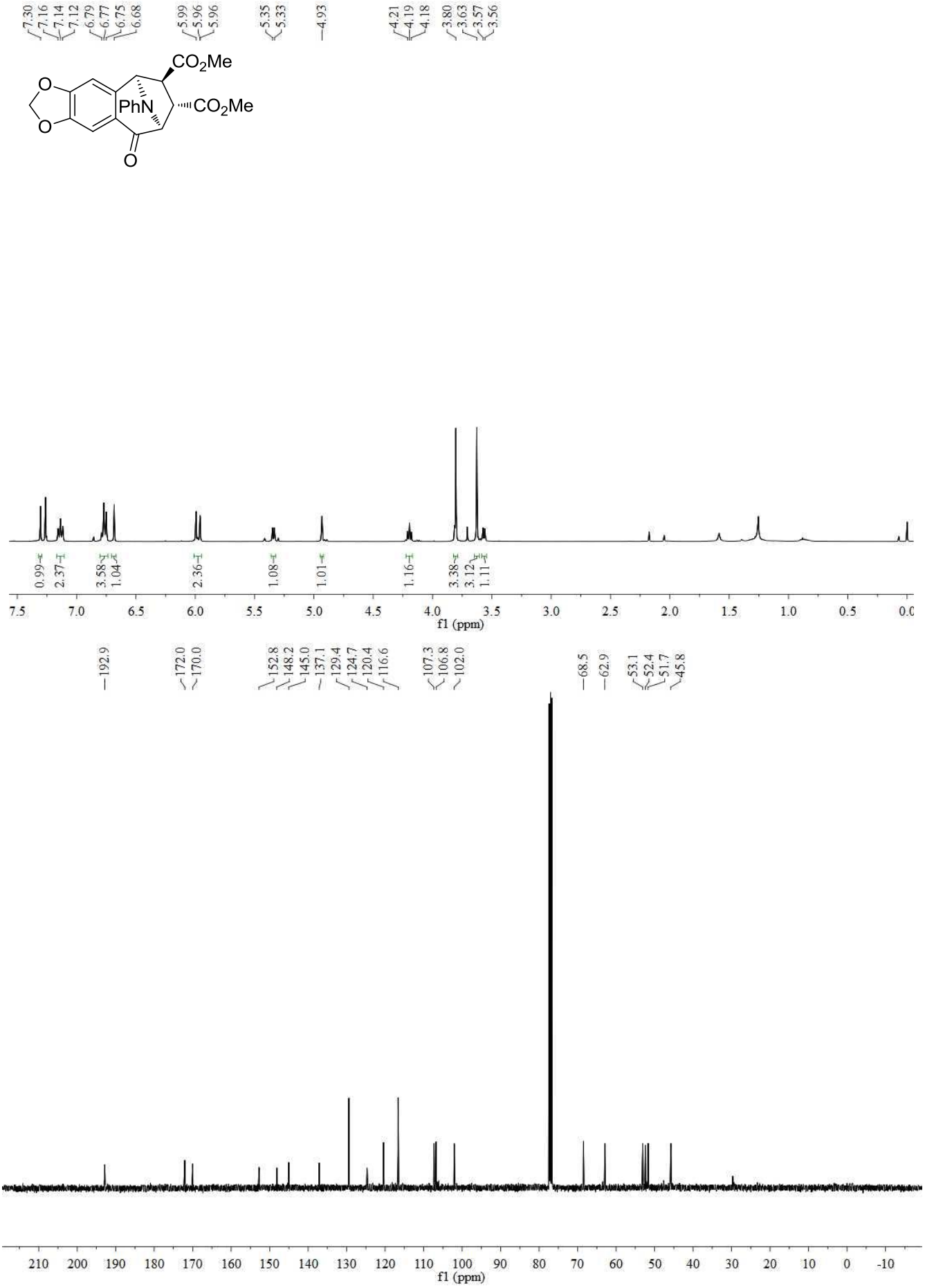

193.1
171.9
170.4
163.5
161.0
144.7
139.6
139.6
132.3
132.2
129.4
128.1
128.0
122.0
121.8
120.6
116.5
113.8
113.5
68.3
62.7
53.1
52.8
52.3
47.5
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

3m and 3m’

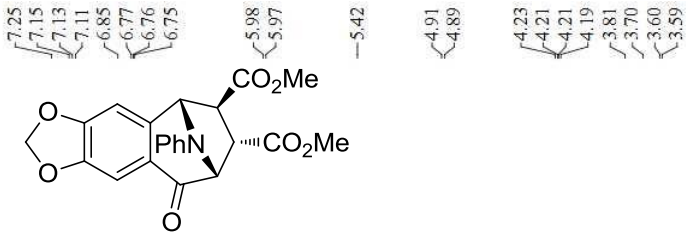


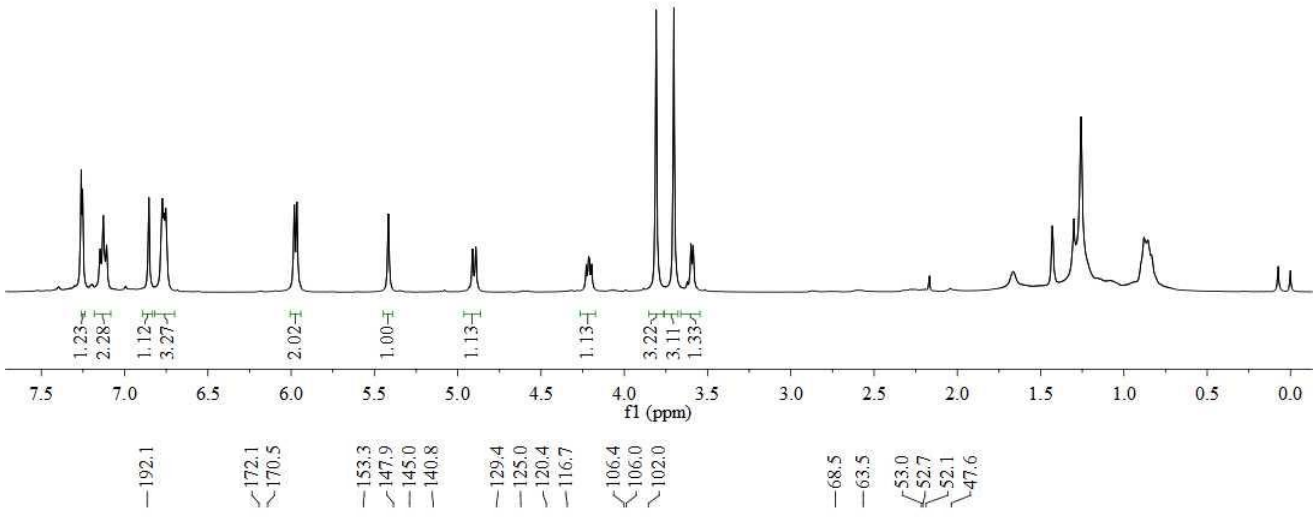


3n

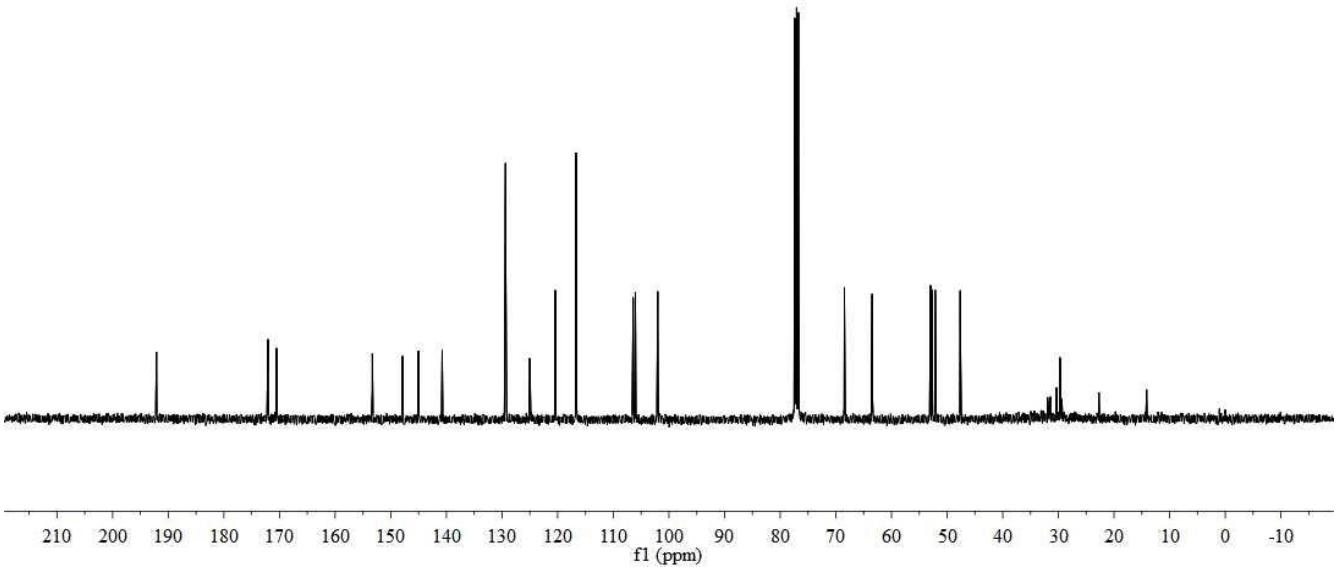

7.30
7.16
7.14
7.12
6.79
6.77
6.75
6.68
5.99
5.96
5.96
5.35
5.33
4.93
4.21
4.19
4.18
3.80
3.63
3.57
3.56
CO2Me
PhN
CO2Me
O
O
O
0.99
2.37
3.58
1.04
2.36
1.08
1.01
1.16
3.38
3.12
1.11
f1 (ppm)
192.9
172.0
170.0
152.8
148.2
145.0
137.1
129.4
124.7
120.4
116.6
107.3
106.8
102.0
68.5
62.9
53.1
52.4
51.7
45.8
f1 (ppm)

3n'

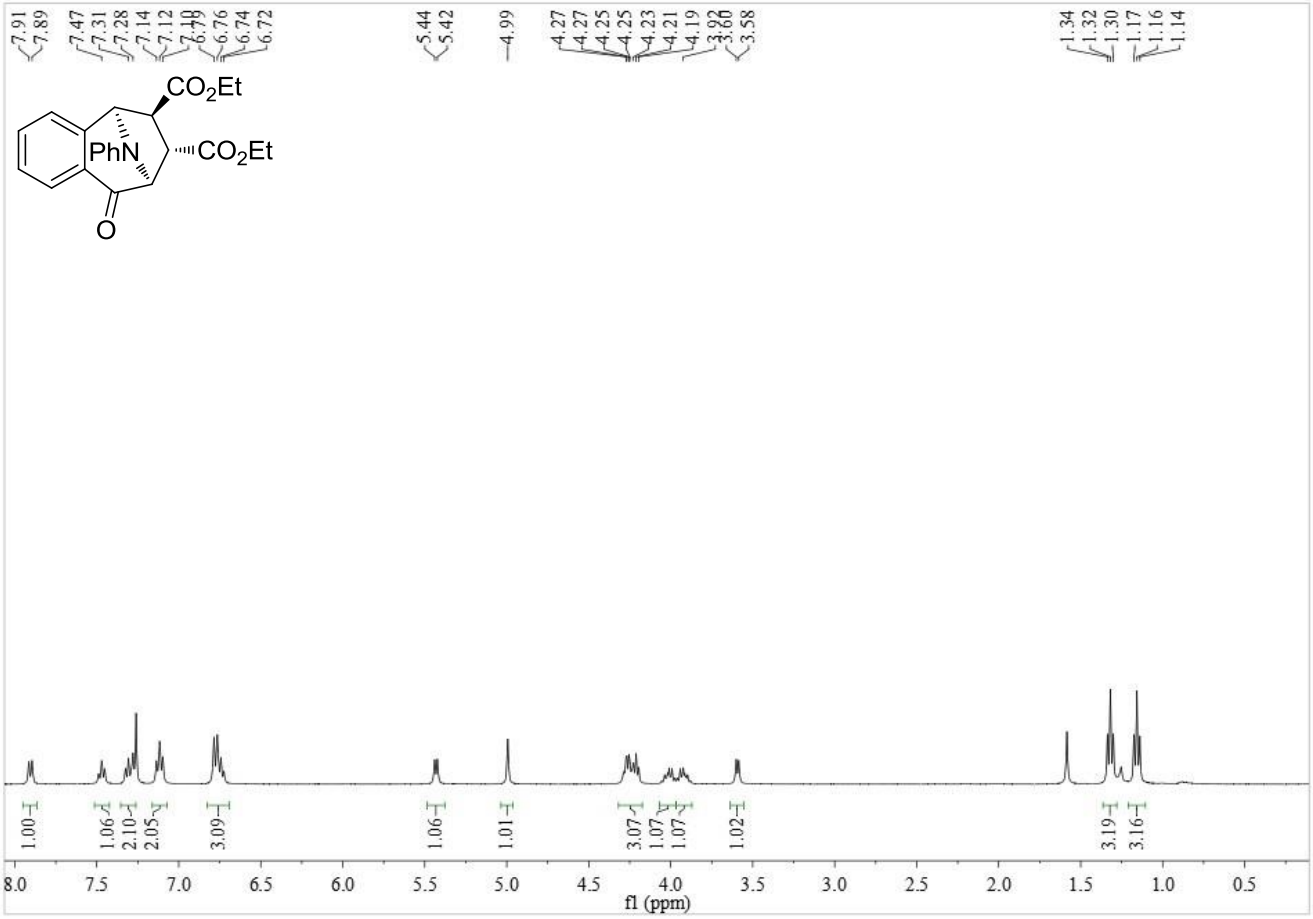

7.25
7.15
7.13
7.11
6.85
6.77
6.76
6.75
5.98
5.97
5.42
4.91
4.89
4.23
4.21
4.21
4.19
3.81
3.70
3.60
3.59
CO2Me
CO2Me
PhN
O
O
O

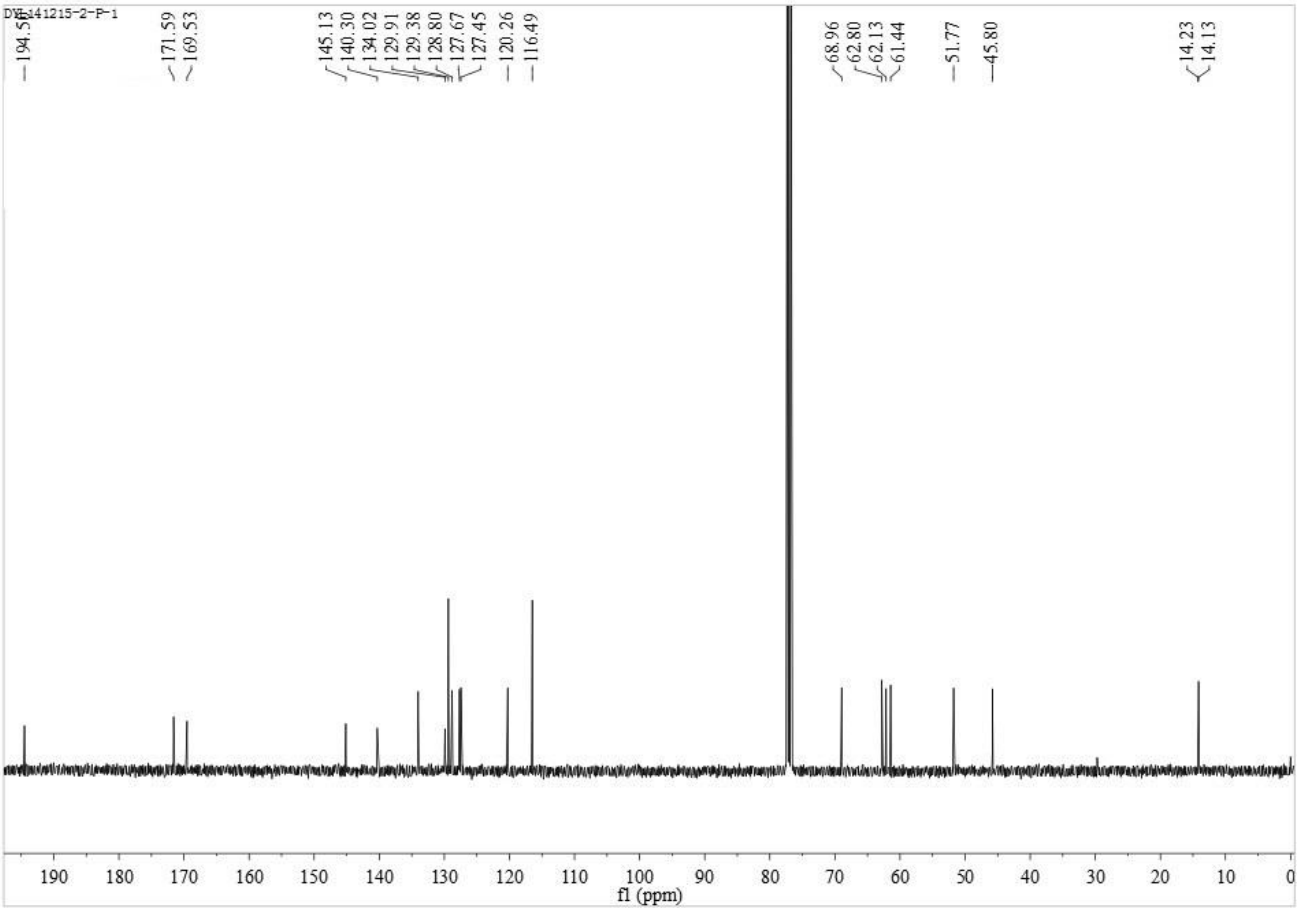

1.23
2.28
1.12
3.27
2.02
1.00
1.13
1.13
3.22
3.11
1.33
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)

192.1
172.1
170.5
153.3
147.9
145.0
140.8
129.4
125.0
120.4
116.7
106.4
106.0
102.0
68.5
63.5
53.0
52.7
52.1
47.6

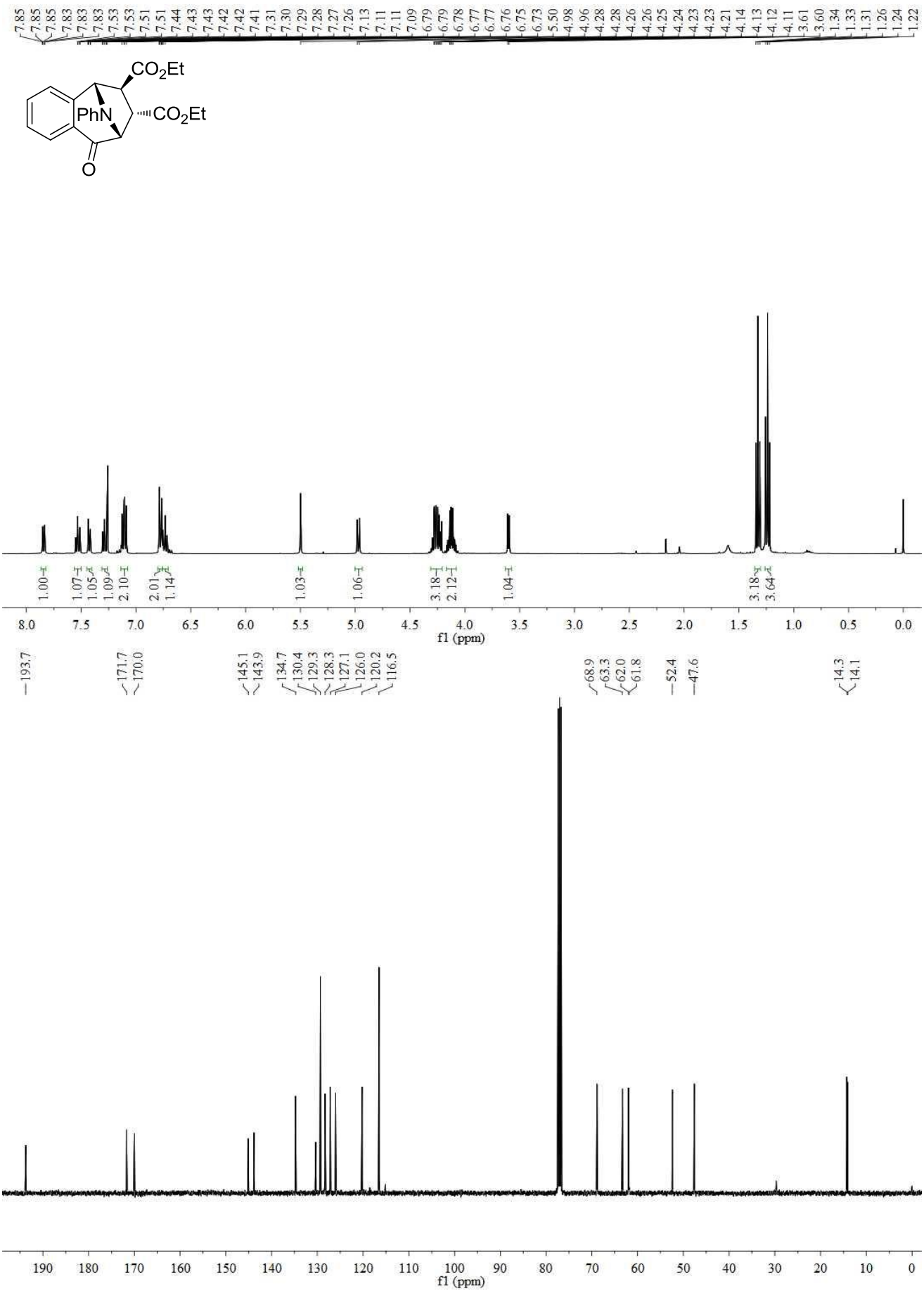

210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

3o

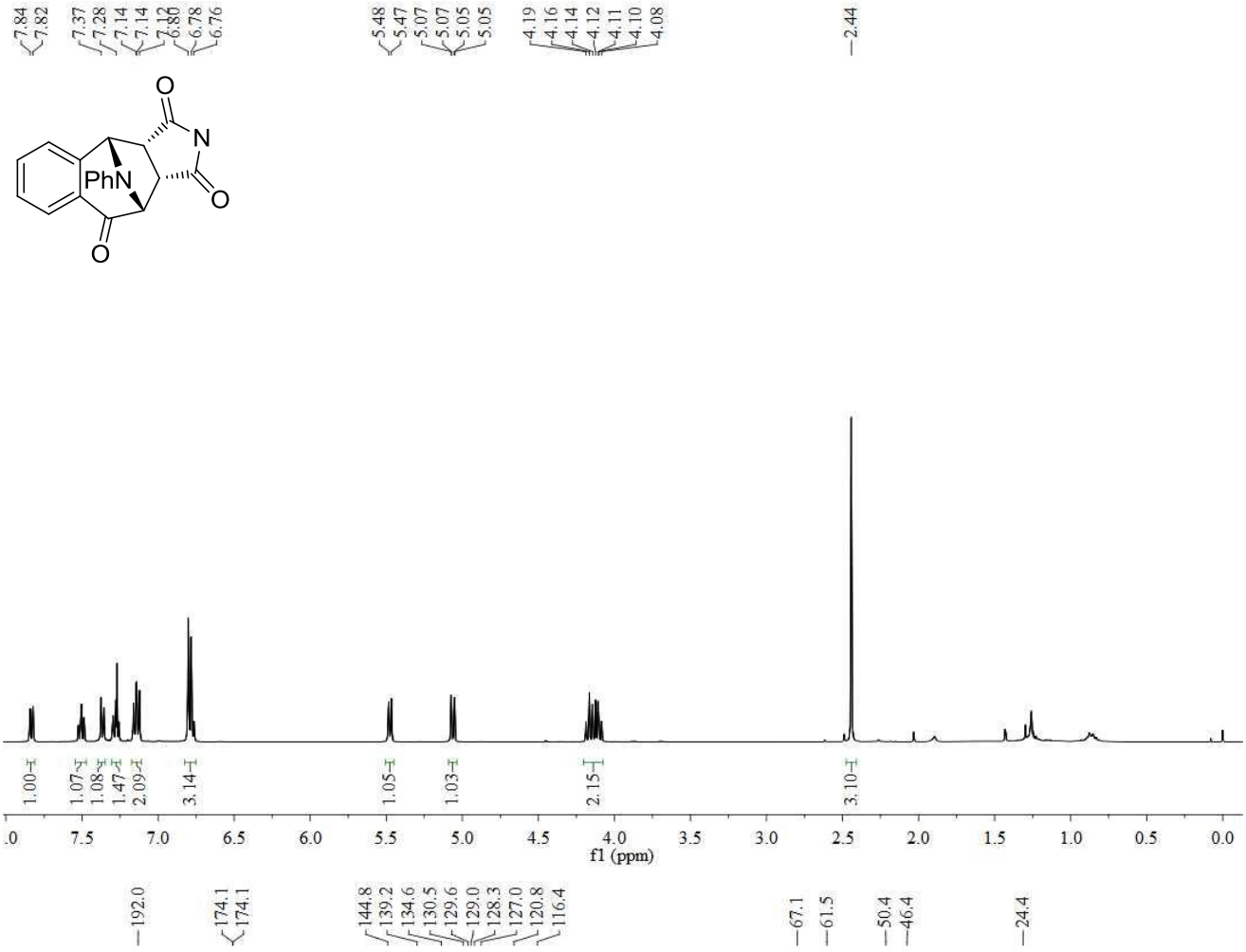

7.91
7.89
7.47
7.31
7.28
7.14
7.12
7.10
6.79
6.76
6.74
6.72
5.44
5.42
4.99
4.27
4.27
4.25
4.25
4.23
4.21
4.19
3.92
3.60
3.58
1.34
1.32
1.30
1.17
1.16
1.14
CO2Et
PhN
CO2Et
O
1.00
1.06
2.10
2.05
3.09
1.06
1.01
3.07
1.07
1.07
1.02
3.19
3.16
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
f1 (ppm)

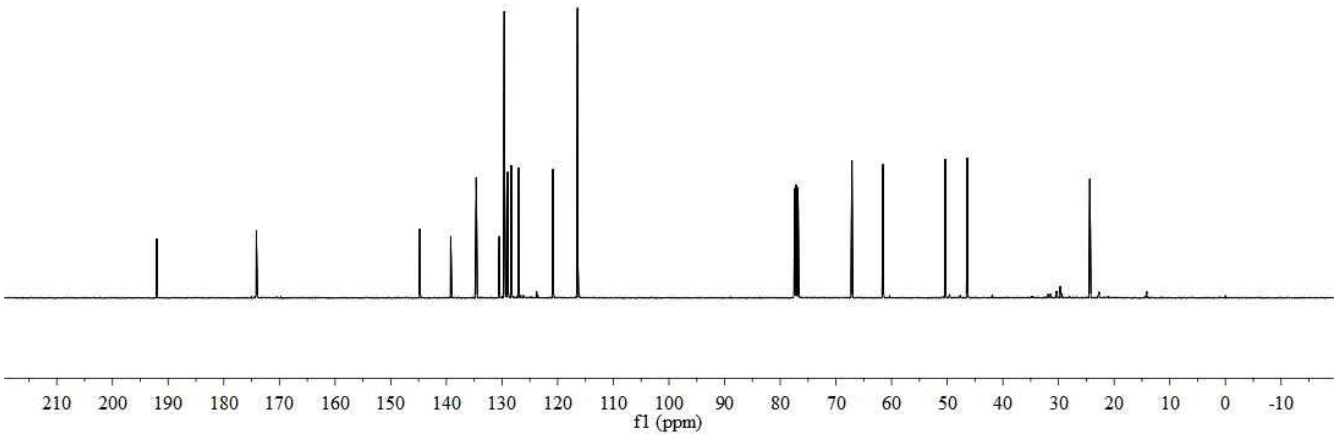

DYL141215-2-P-1
194.56
171.59
169.53
145.13
140.30
134.02
129.91
129.38
128.80
127.67
127.45
120.26
116.49
68.96
62.80
62.13
61.44
51.77
45.80
14.23
14.13
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

3o'

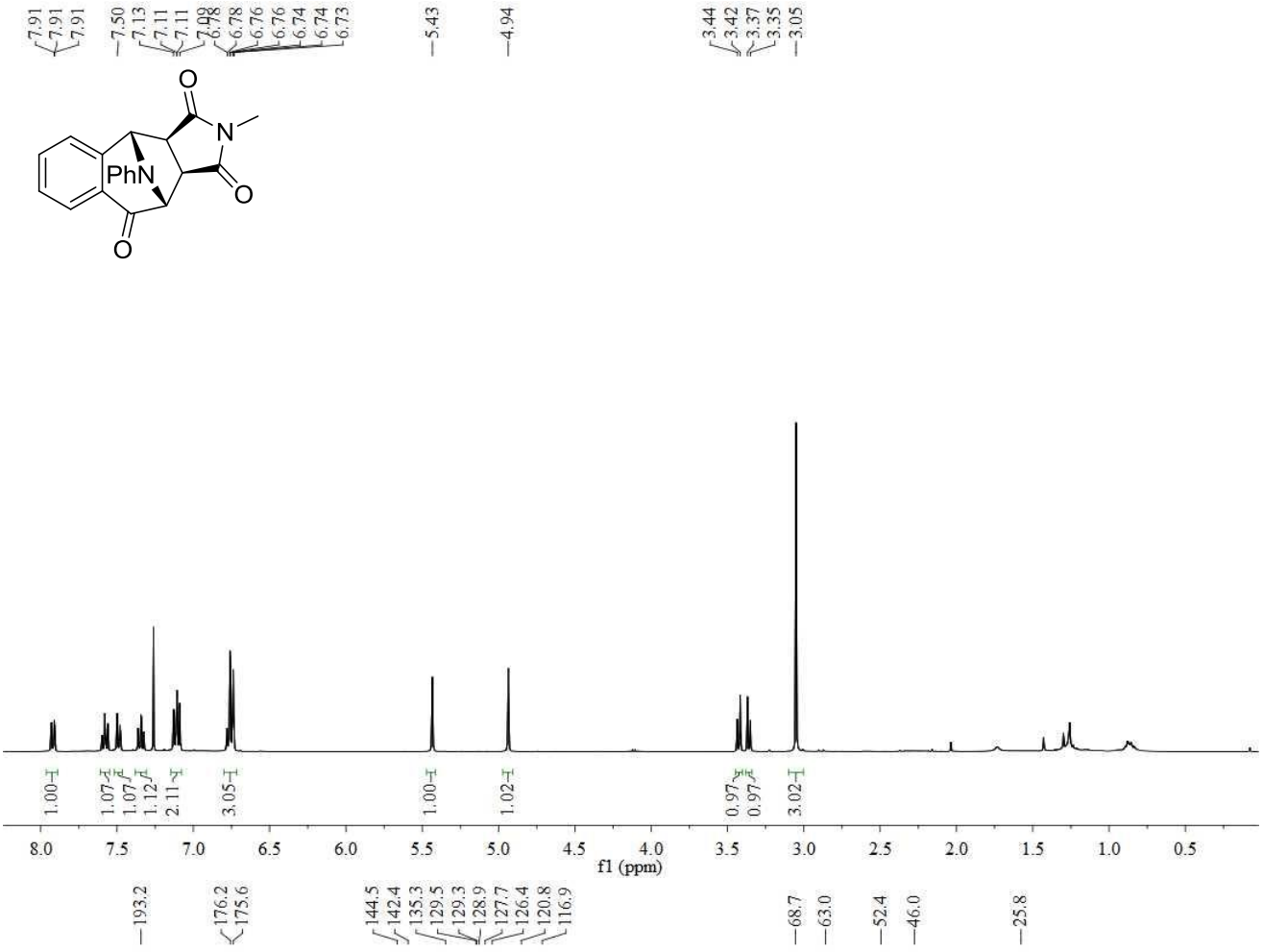

7.85 7.85 7.85 7.83 7.83 7.53 7.53 7.51 7.51 7.44 7.43 7.43 7.42 7.42 7.41 7.31 7.30 7.29 7.28 7.27 7.26 7.13 7.11 7.11 7.09 6.79 6.79 6.78 6.77 6.77 6.76 6.75 6.73 5.50 4.98 4.96 4.28 4.26 4.26 4.25 4.24 4.23 4.23 4.21 4.14 4.13 4.12 4.11 3.61 3.60 1.34 1.33 1.31 1.26 1.24 1.22
CO2Et
PhN
CO2Et
O
1.00 1.07 1.05 1.09 2.10 2.01 1.14 1.03 1.06 3.18 2.12 1.04 3.18 3.64
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)
193.7 171.7 170.0 145.1 143.9 134.7 130.4 129.3 128.3 127.1 126.0 120.2 116.5 68.9 63.3 62.0 61.8 52.4 47.6 14.3 14.1
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0
f1 (ppm)

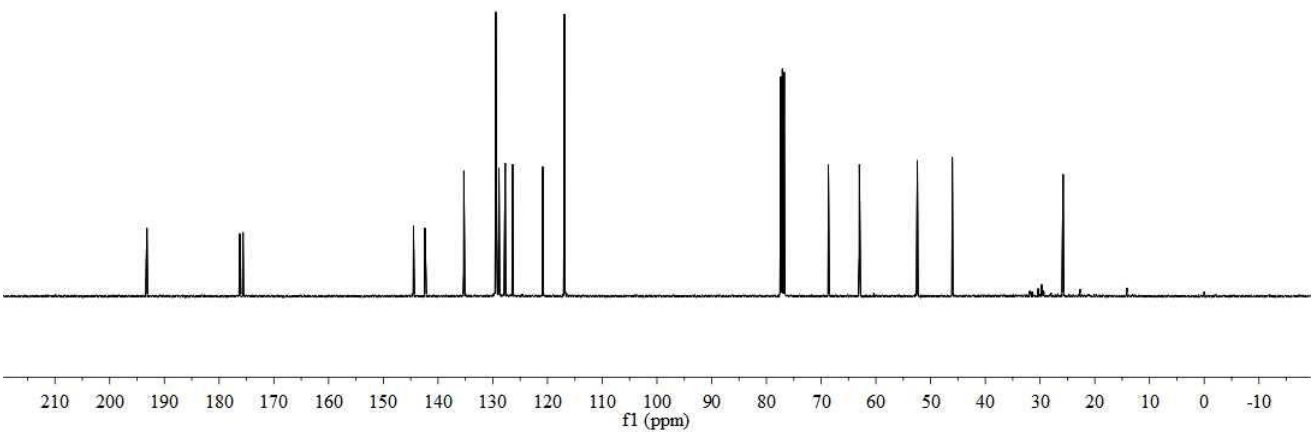
3p


7.84
7.82
7.37
7.28
7.14
7.14
7.12
6.80
6.78
6.76
5.48
5.47
5.07
5.07
5.05
5.05
4.19
4.16
4.14
4.12
4.11
4.10
4.08
2.44
O
N
PhN
O
O
1.00
1.07
1.08
1.47
2.09
3.14
1.05
1.03
2.15
3.10
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
f1 (ppm)


192.0
174.1
174.1
144.8
139.2
134.6
130.5
129.6
129.0
128.3
127.0
120.8
116.4
67.1
61.5
50.4
46.4
24.4
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-10
f1 (ppm)

3p'


7.91
7.91
7.91
7.50
7.13
7.11
7.11
7.09
6.78
6.78
6.76
6.76
6.74
6.74
6.73
5.43
4.94
3.44
3.42
3.37
3.35
3.05
O
N
PhN
O
O
1.00
1.07
1.07
1.12
2.11
3.05
1.00
1.02
0.97
0.97
3.02
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
f1 (ppm)
193.2
176.2
175.6
144.5
142.4
135.3
129.5
129.3
128.9
127.7
126.4
120.8
116.9
68.7
63.0
52.4
46.0
25.8


210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)