Supplementary Information (SI) for Organic & Biomolecular Chemistry.
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Catalytic Stereoselective Synthesis of Cyclopentanones from Donor-Acceptor Cyclopropanes and in situ-generated Ketenes

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NMR and HPLC spectra

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HPLC of (±)-**3c** 41

HPLC of (–)-**3c** 42

HPLC of (+)-**3c** 43

^{1}H & ^{13}C NMR of (–)-**3d** 44

^{1}H & ^{13}C NMR of (+)-**3d** 46

HPLC of (±)-**3d** 48

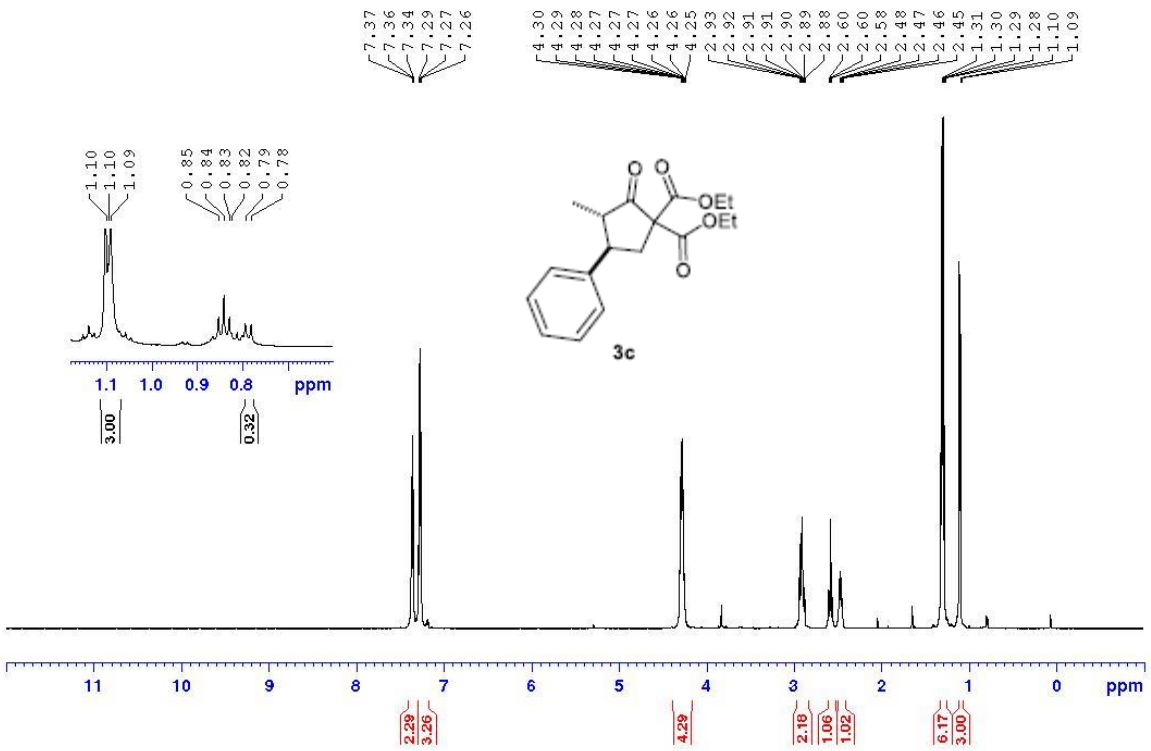
HPLC of (–)-**3d** 49

HPLC of (+)-**3d** 50

^{1}H & ^{13}C NMR of **5d** 51

2D-NOESY for **3c** 53

^{1}H NMR of **3c:**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7.36
7.34
7.29
7.27
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4.30
4.29
4.28
4.27
4.27
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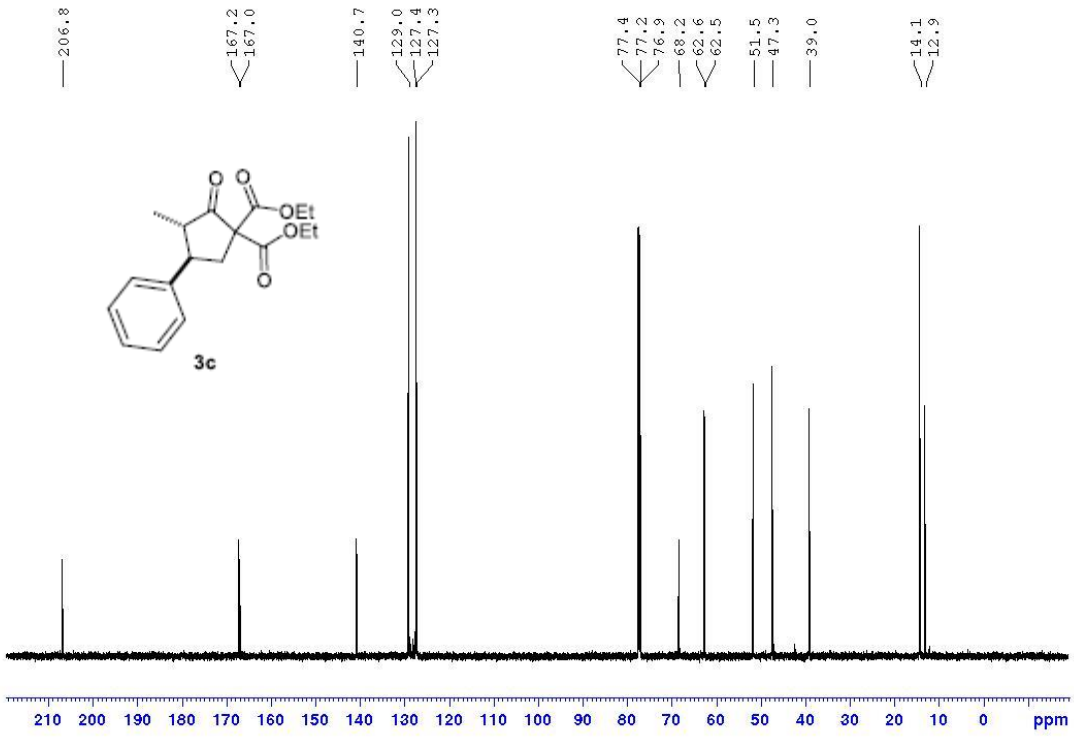
4.26
4.26
4.25
2.93
2.92
2.91
2.91
2.90
2.89
2.88
2.60
2.60
2.58
2.48
2.47
2.46
2.45
1.31
1.30
1.29
1.28
1.10
1.09
1.10
1.10
1.09
0.85
0.84
0.83
0.82
0.79
0.78
OEt
OEt
3c
1.1
1.0
0.9
0.8
ppm
3.00
0.32
11
10
9
8
7
6
5
4
3
2
1
0
ppm
2.29
3.26
4.29
2.10
1.06
1.02
6.17
3.00

^{13}C NMR of **3c**: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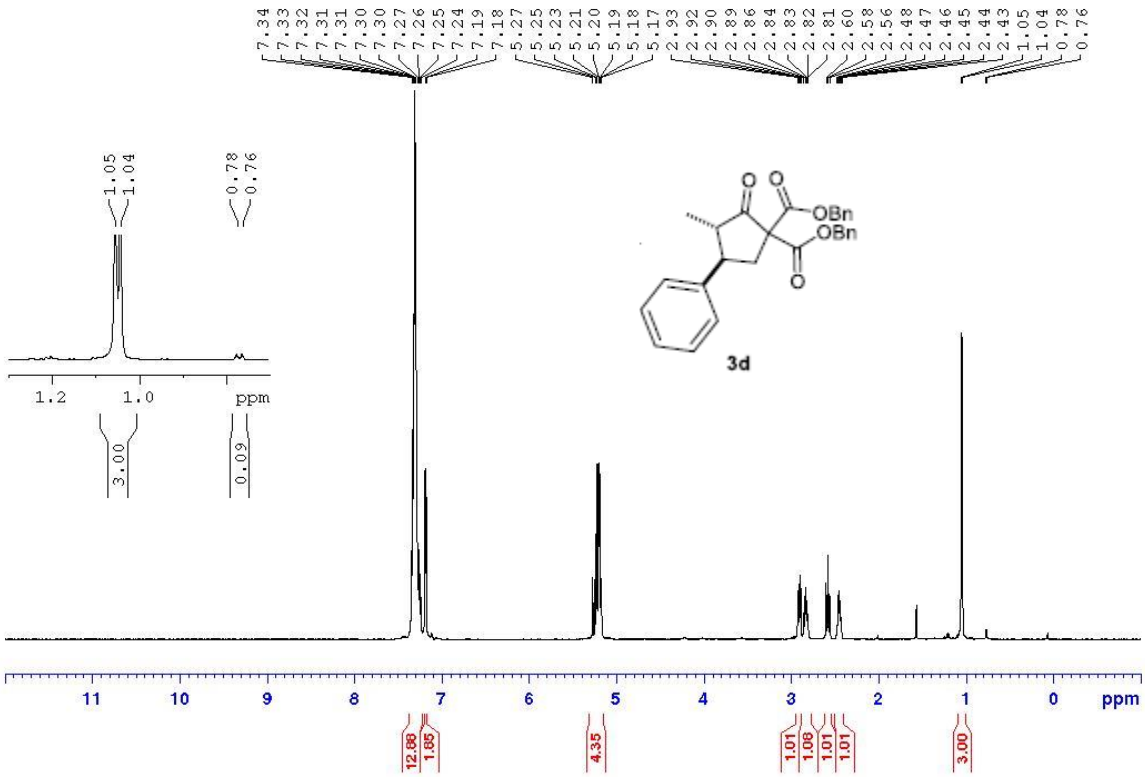
167.0
140.7
129.0
127.4
127.3
77.4
77.2
76.9
68.2
62.6
62.5
51.5
47.3
39.0
14.1
12.9
OEt
OEt
3c
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

^{1}H NMR of **3d:**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7.32
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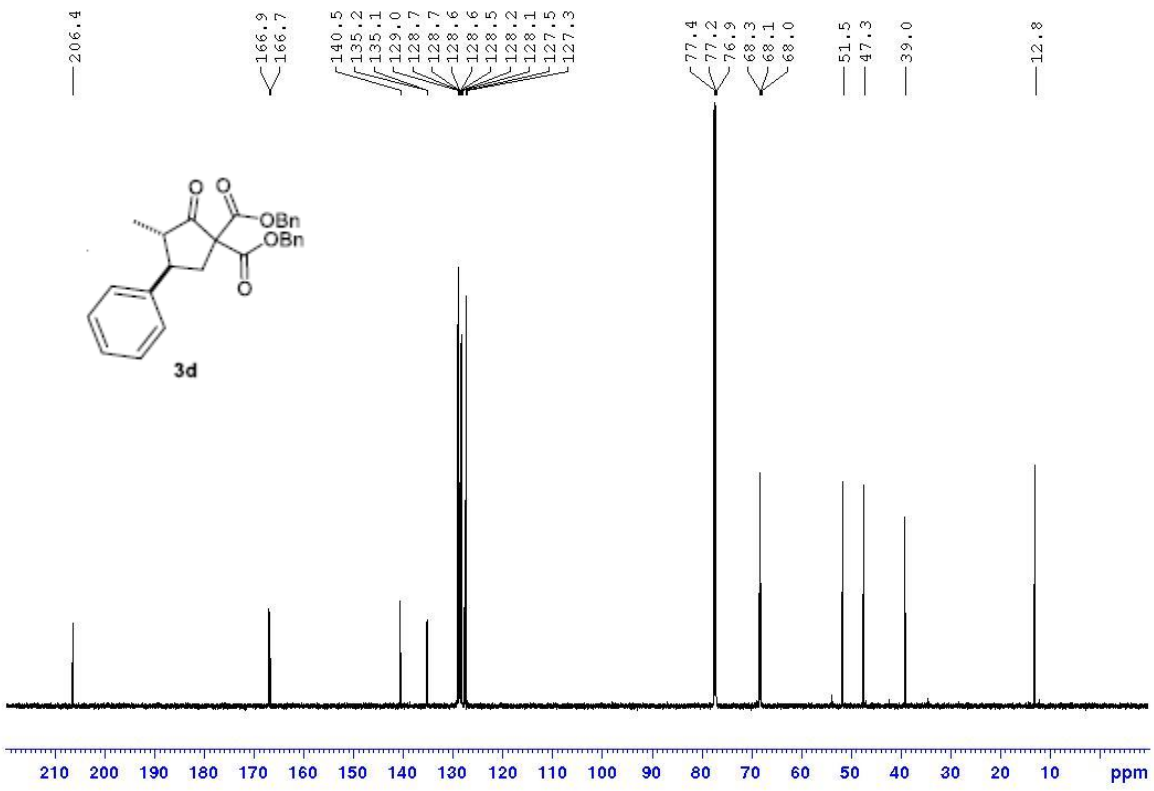
7.31
7.30
7.30
7.27
7.26
7.25
7.24
7.19
7.18
5.27
5.25
5.23
5.21
5.20
5.19
5.18
5.17
2.93
2.92
2.90
2.89
2.86
2.84
2.83
2.82
2.81
2.60
2.58
2.56
2.48
2.47
2.46
2.45
2.44
2.43
1.05
1.04
0.78
0.76
1.05
1.04
0.78
0.76
1.2
1.0
ppm
3.00
0.09
OBn
OBn
3d
11
10
9
8
7
6
5
4
3
2
1
0
ppm
12.86
1.85
4.35
1.01
1.08
1.01
1.01
3.00

^{13}C NMR of **3d:**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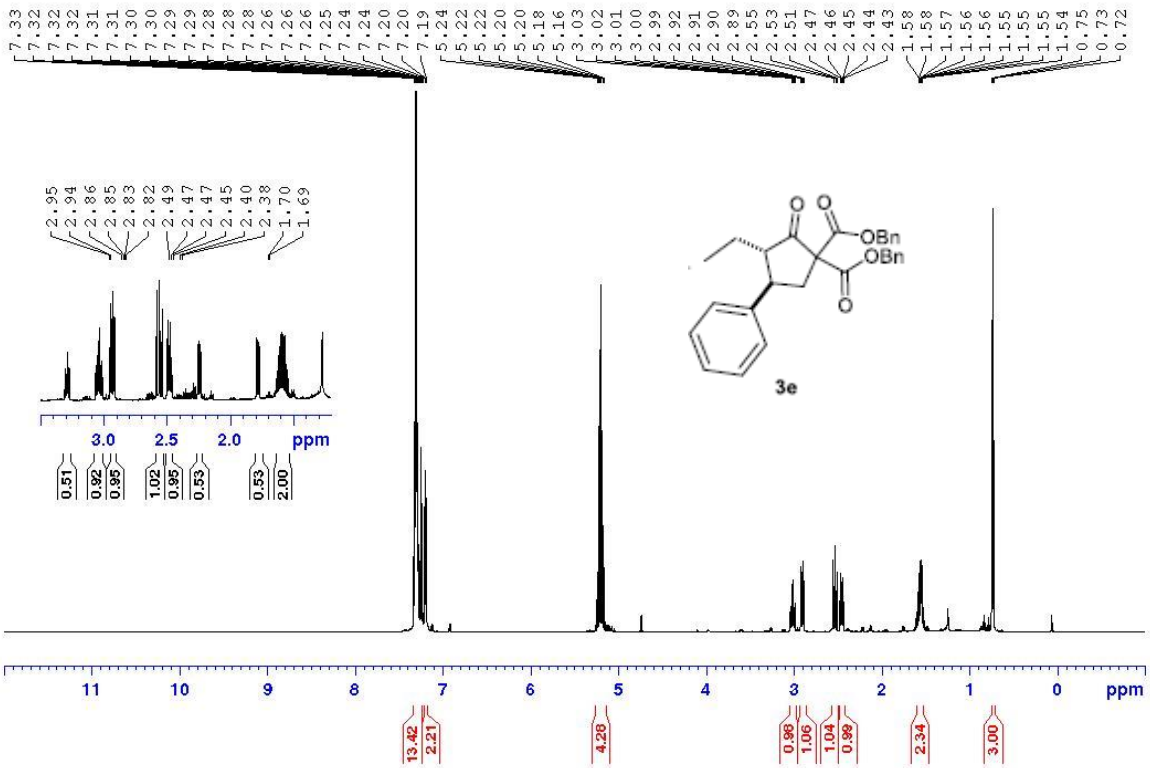
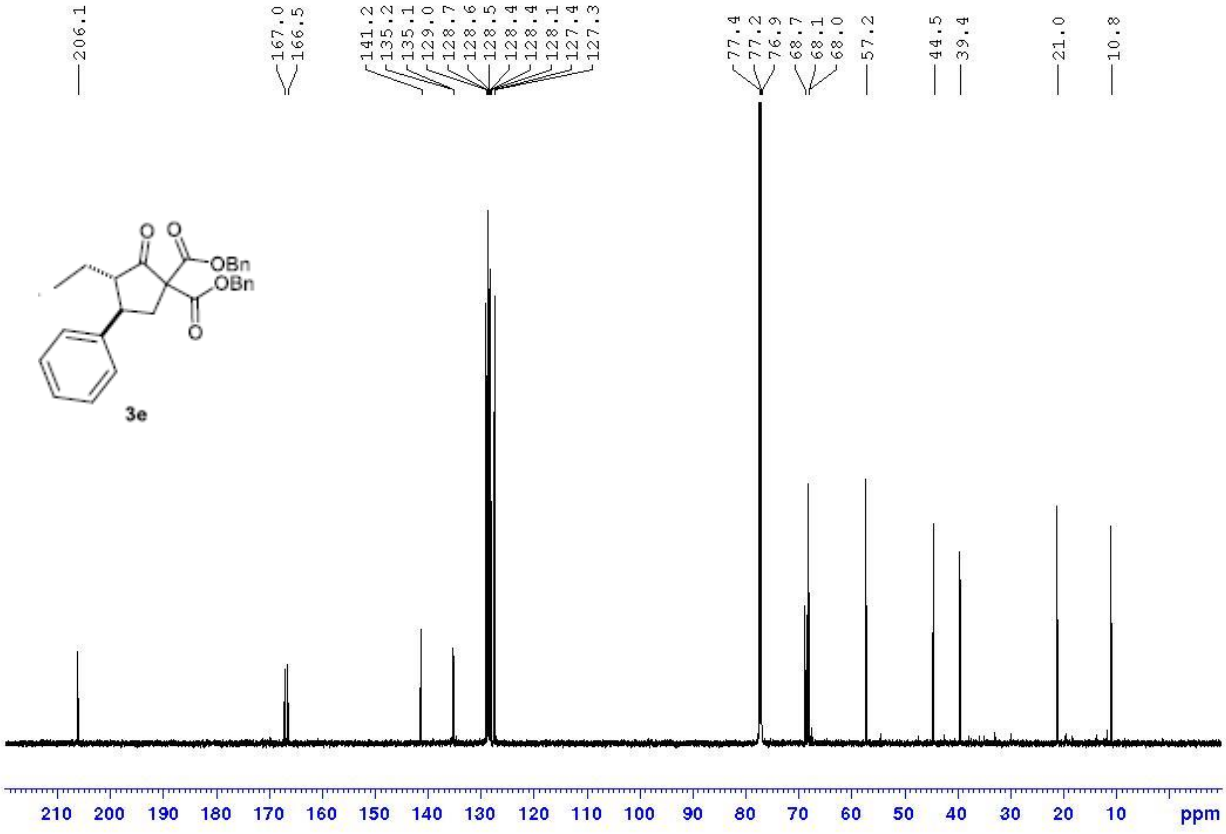
166.7
140.5
135.2
135.1
129.0
128.7
128.7
128.6
128.6
128.5
128.2
128.1
127.5
127.3
77.4
77.2
76.9
68.3
68.1
68.0
51.5
47.3
39.0
12.8
OBn
OBn
3d
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

^{1}H NMR of **3e:**



^{13}C NMR of **3e:**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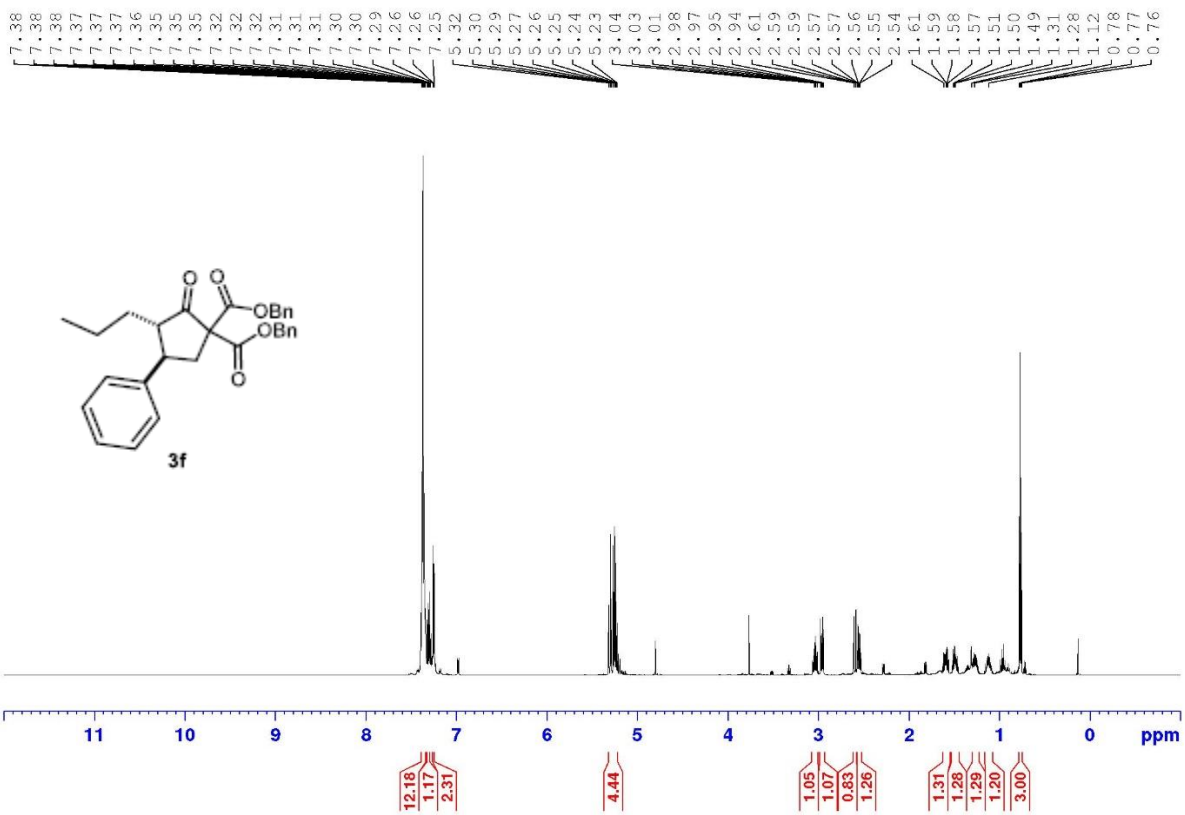
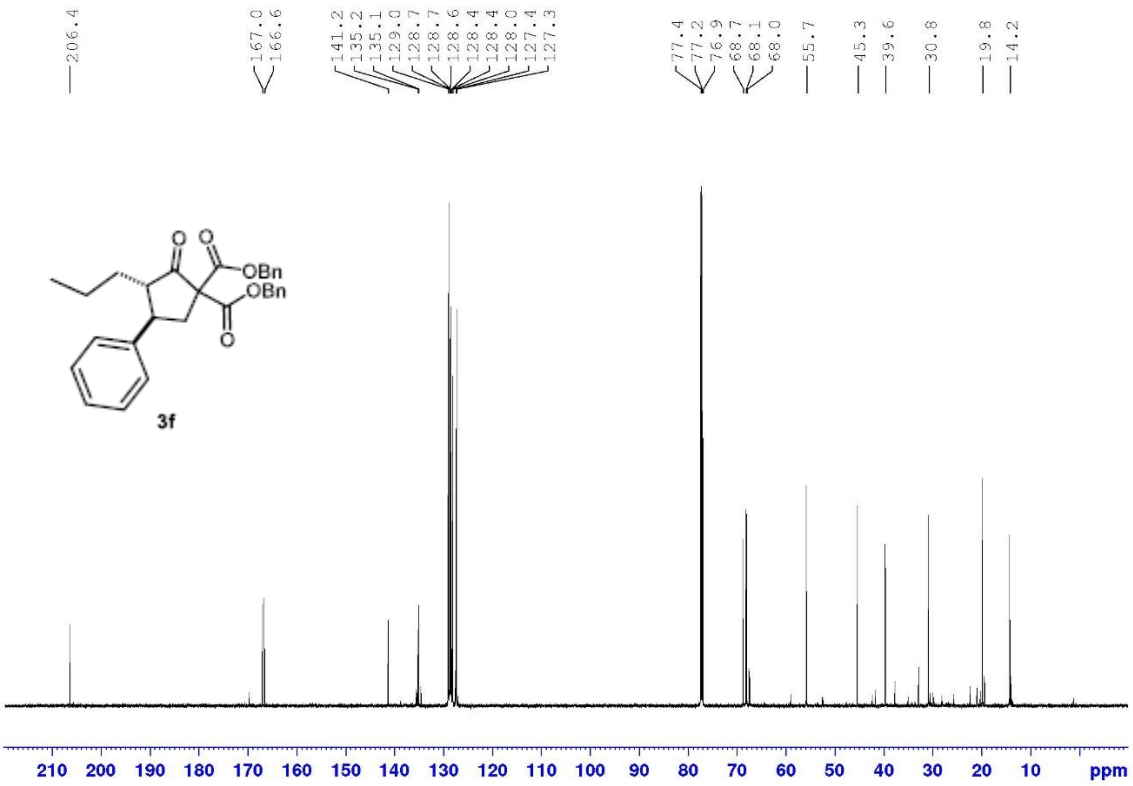
167.0
166.5
141.2
135.2
135.1
129.0
128.7
128.6
128.5
128.4
128.4
128.1
127.4
127.3
77.4
77.2
76.9
68.7
68.1
68.0
57.2
44.5
39.4
21.0
10.8
OBn
OBn
3e
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

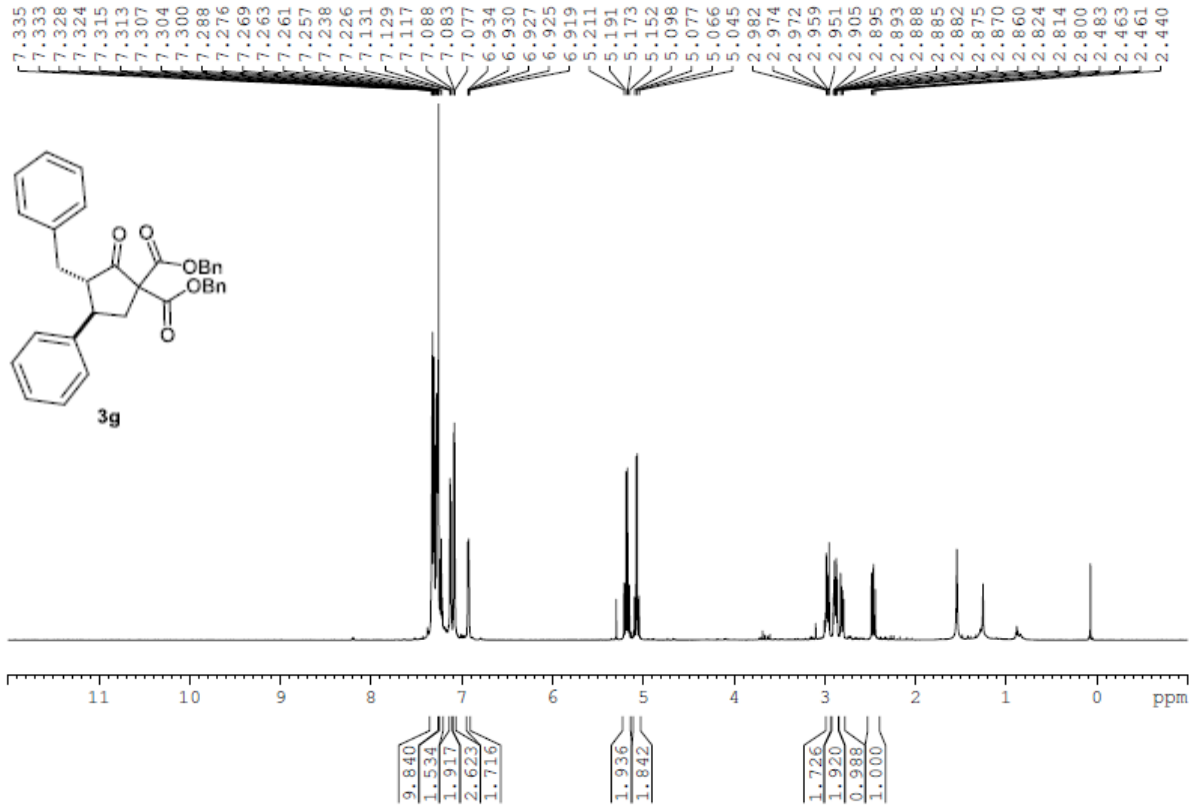
^{1}H NMR of **3f:**


7.38 7.38 7.38 7.37 7.37 7.37 7.36 7.35 7.35 7.35 7.32 7.32 7.32 7.31 7.31 7.31 7.30 7.30 7.29 7.26 7.25
5.32 5.30 5.29 5.27 5.26 5.25 5.24 5.23
3.04 3.03 3.01 2.98 2.97 2.95 2.94 2.61 2.59 2.59 2.57 2.57 2.56 2.55 2.54
1.61 1.59 1.58 1.57 1.51 1.50 1.49 1.31 1.28 1.12 0.78 0.77 0.76
OBn
OBn
3f
11 10 9 8 7 6 5 4 3 2 1 0 ppm
12.18 1.17 2.31
4.44
1.05 1.07 0.83 1.26
1.31 1.28 1.29 1.20 3.00

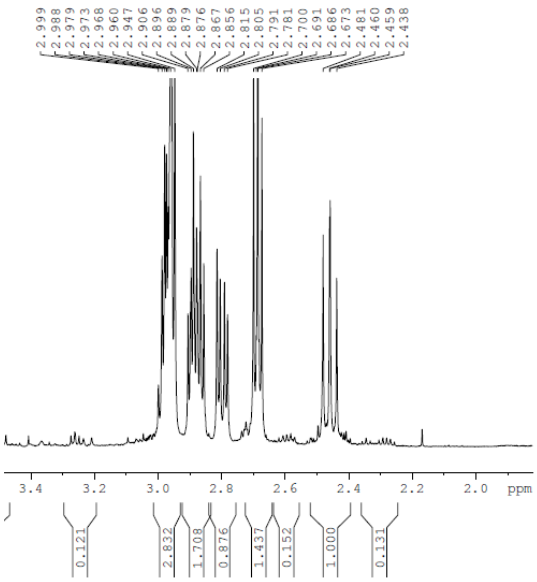
^{13}C NMR of **3f:**


206.4
167.0
166.6
141.2
135.2
135.1
129.0
128.7
128.7
128.6
128.4
128.4
128.0
127.4
127.3
77.4
77.2
76.9
68.7
68.1
68.0
55.7
45.3
39.6
30.8
19.8
14.2
OBn
OBn
3f
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

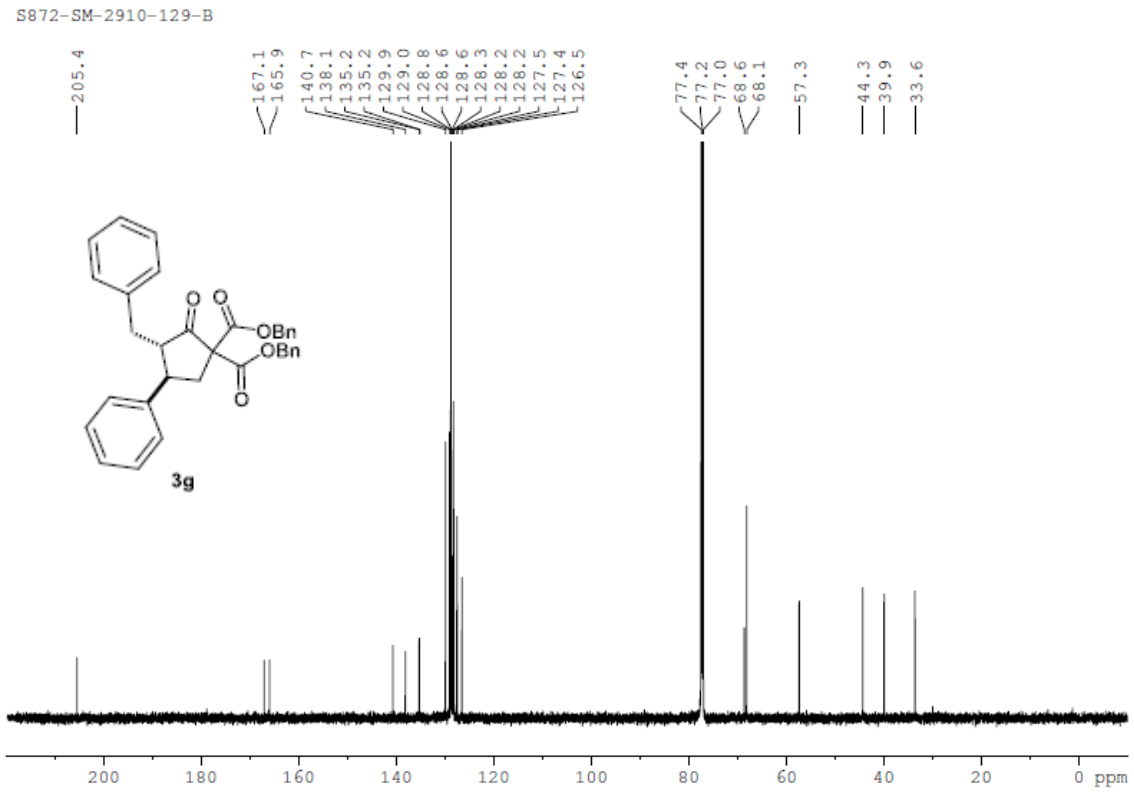
^{1}H NMR of **3g:**



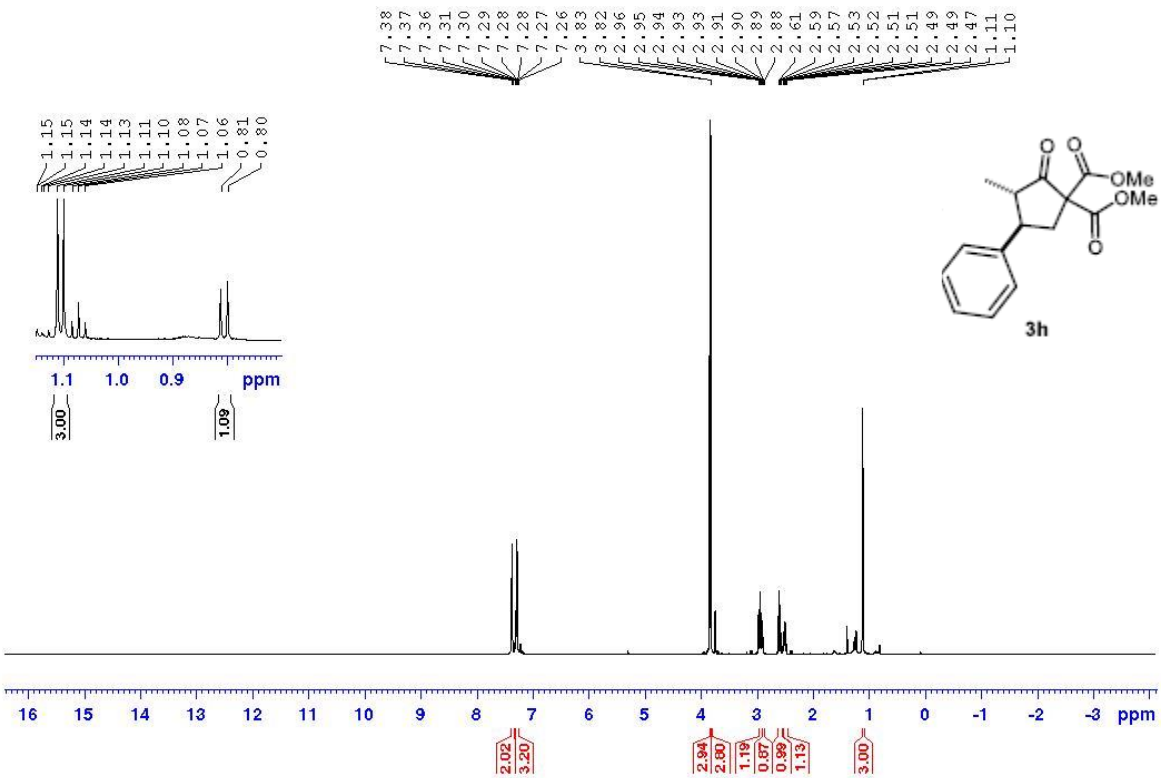
Crude ^{1}H NMR expansion:



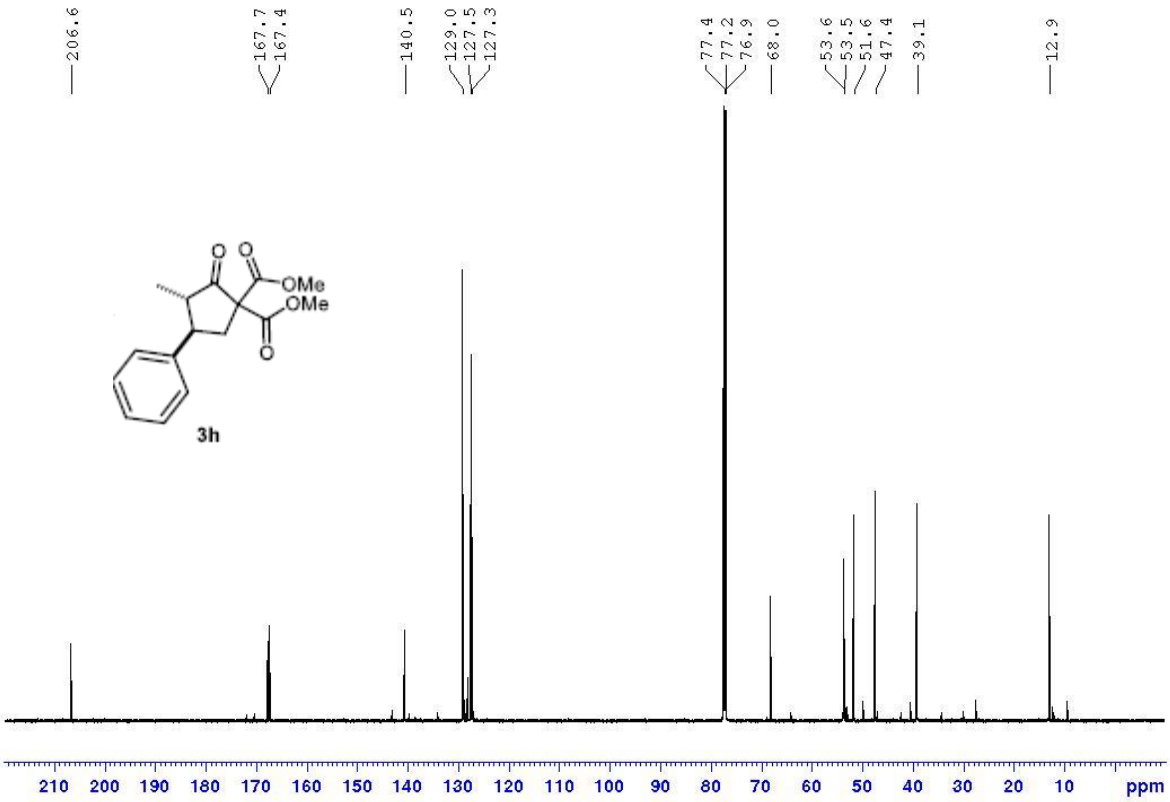
^{13}C NMR of **3g:**


S872-SM-2910-129-B
205.4
167.1
165.9
140.7
138.1
135.2
135.2
129.9
129.0
128.8
128.6
128.6
128.3
128.2
128.2
127.5
127.4
126.5
77.4
77.2
77.0
68.6
68.1
57.3
44.3
39.9
33.6
OBn
OBn
3g
200
180
160
140
120
100
80
60
40
20
0 ppm

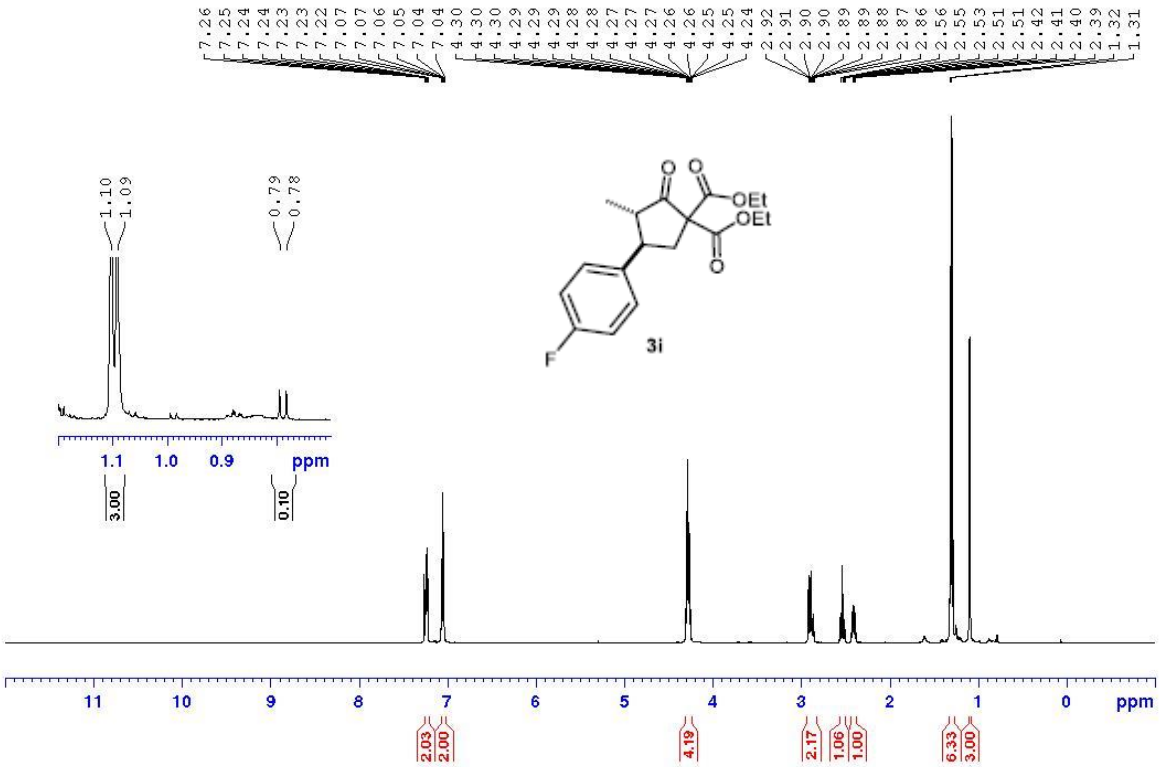
^{1}H NMR of **3h:**


7.38
7.37
7.36
7.31
7.30
7.29
7.28
7.28
7.27
7.26
3.83
3.82
2.96
2.95
2.94
2.93
2.93
2.91
2.90
2.89
2.88
2.61
2.59
2.57
2.53
2.52
2.51
2.51
2.49
2.49
2.47
1.11
1.10
1.15
1.15
1.14
1.14
1.13
1.11
1.10
1.08
1.07
1.06
0.81
0.80
1.1
1.0
0.9
ppm
3.00
1.09
OMe
OMe
3h
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
0
-1
-2
-3
ppm
2.02
3.20
2.94
2.80
1.19
0.67
0.98
1.13
3.00

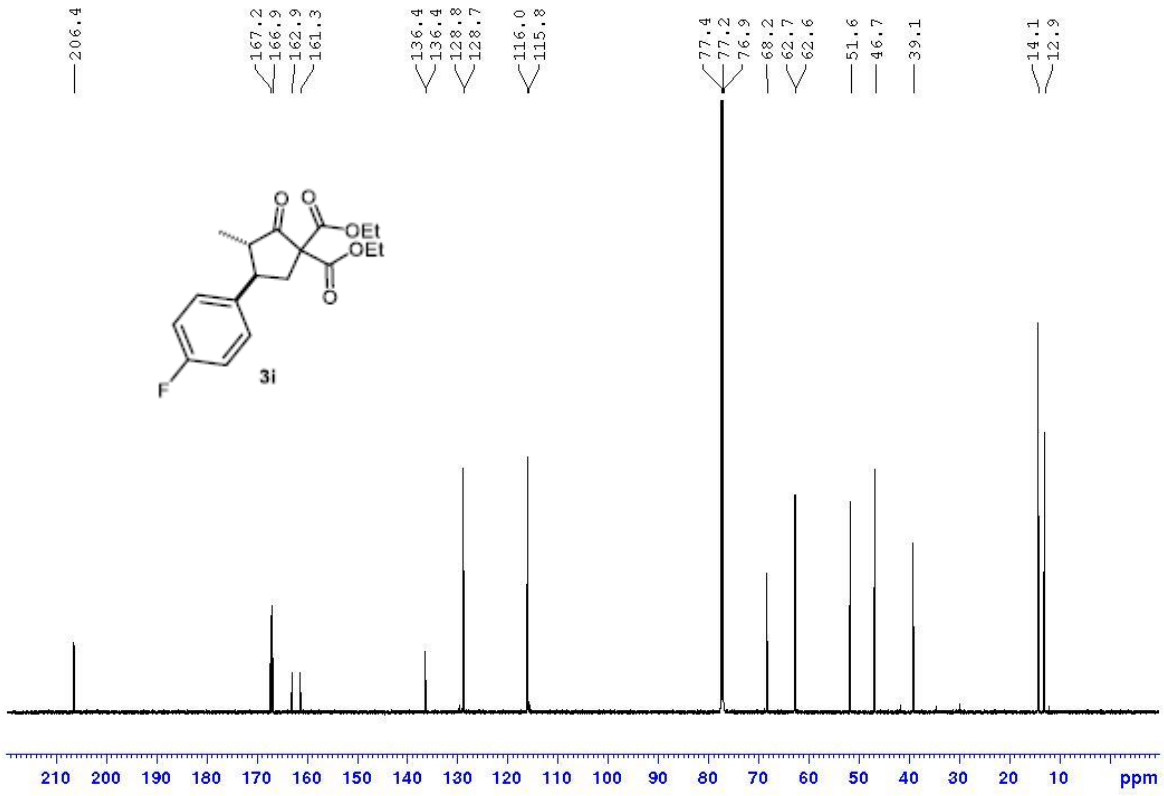
^{13}C NMR of **3h:**


206.6
167.7
167.4
140.5
129.0
127.5
127.3
77.4
77.2
76.9
68.0
53.6
53.5
51.6
47.4
39.1
12.9
OMe
OMe
3h
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

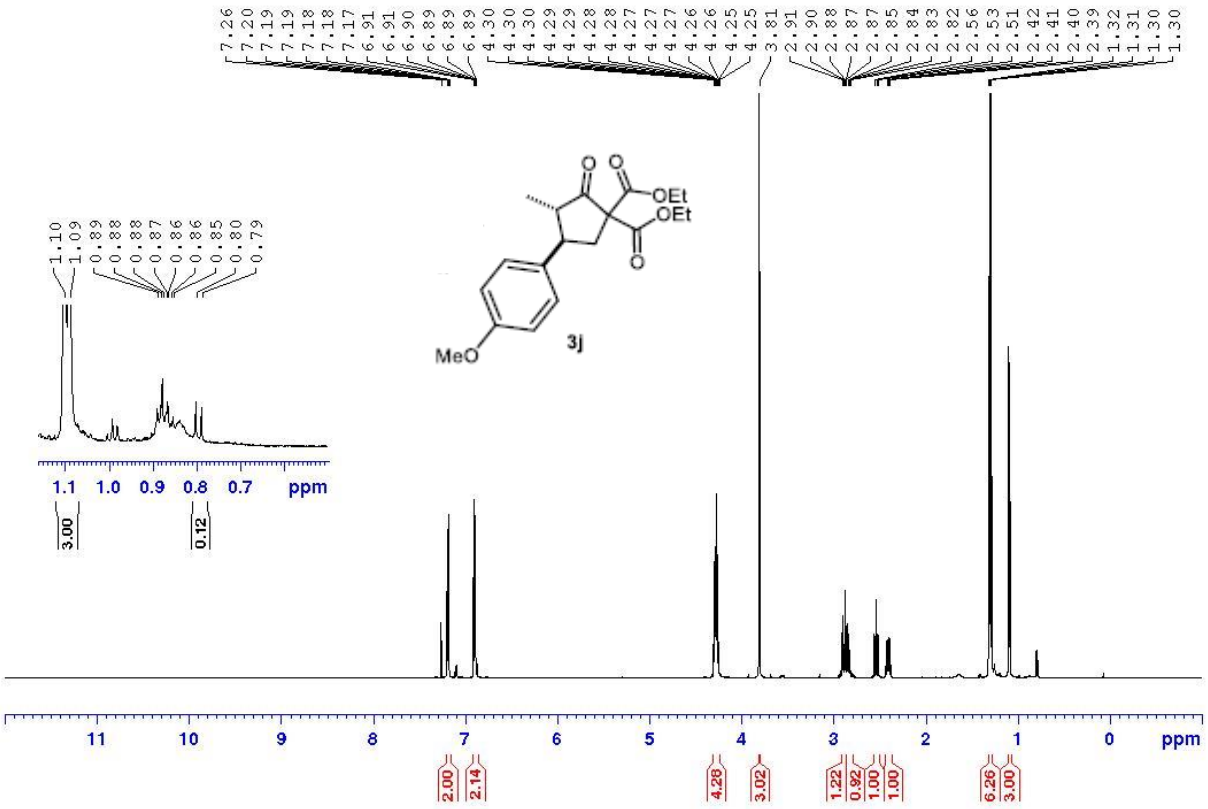
^{1}H NMR of **3i:**


7.26 7.25 7.24 7.24 7.23 7.23 7.22 7.07 7.07 7.06 7.05 7.04 7.04
4.30 4.30 4.29 4.29 4.29 4.28 4.28 4.27 4.27 4.27 4.26 4.26 4.25 4.25 4.24
2.92 2.91 2.90 2.90 2.89 2.89 2.88 2.87 2.86 2.56 2.55 2.53 2.51 2.51 2.42 2.41 2.40 2.39 1.32 1.31
1.10 1.09
0.79 0.78
OEt
OEt
F
3i
1.1 1.0 0.9 ppm
3.00
0.10
11 10 9 8 7 6 5 4 3 2 1 0 ppm
2.03 2.00 4.19 2.17 1.06 1.00 6.33 3.00

^{13}C NMR of **3i:**


206.4
167.2
166.9
162.9
161.3
136.4
136.4
128.8
128.7
116.0
115.8
77.4
77.2
76.9
68.2
62.7
62.6
51.6
46.7
39.1
14.1
12.9
OEt
OEt
O
O
O
O
F
3i
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

^{1}H NMR of **3j:**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7.19
7.18
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7.17
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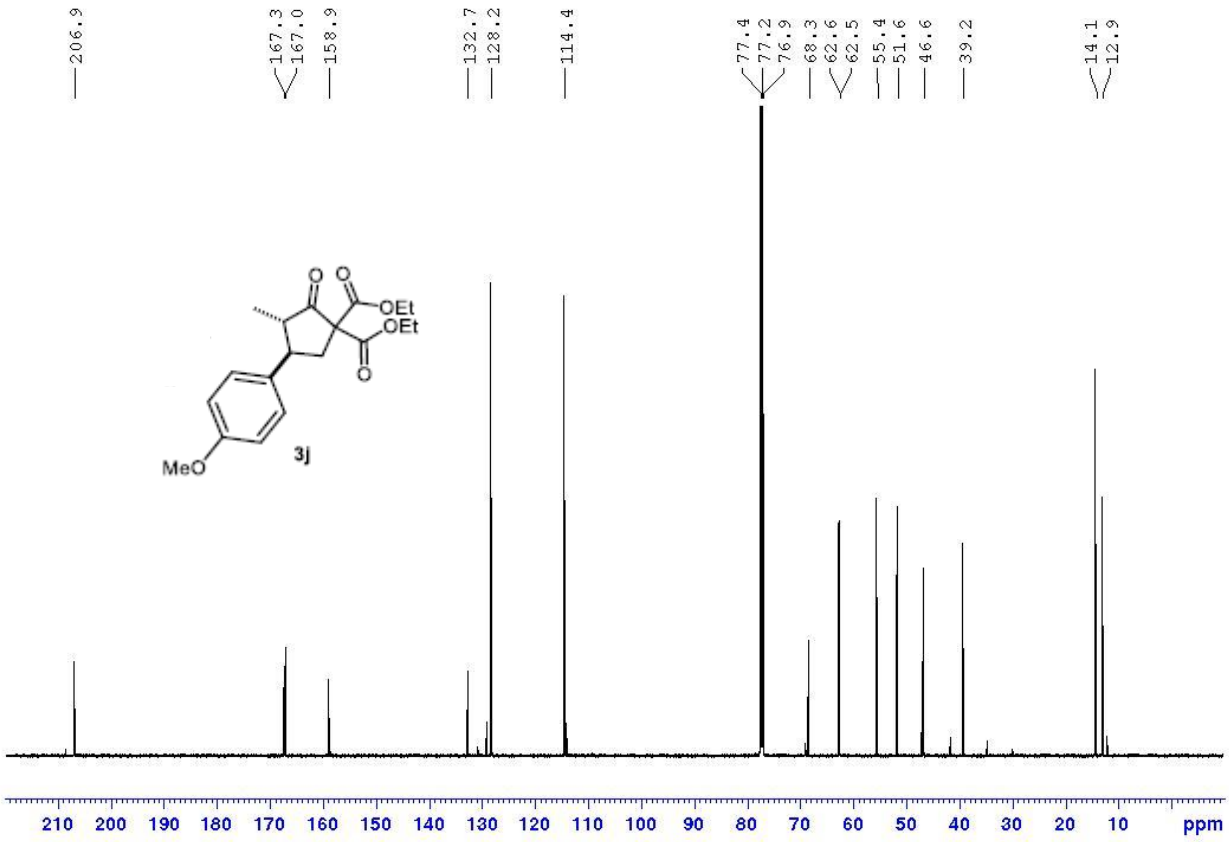
6.90
6.89
6.89
4.30
4.30
4.29
4.29
4.28
4.28
4.27
4.27
4.27
4.26
4.26
4.25
4.25
3.81
2.91
2.90
2.88
2.87
2.87
2.85
2.84
2.83
2.82
2.56
2.53
2.51
2.42
2.41
2.40
2.39
1.32
1.31
1.30
1.30
1.10
1.09
0.89
0.88
0.88
0.87
0.86
0.86
0.85
0.80
0.79
OEt
OEt
MeO
3j
1.1 1.0 0.9 0.8 0.7 ppm
3.00
0.12
11 10 9 8 7 6 5 4 3 2 1 0 ppm
2.00
2.14
4.26
3.02
1.22
0.92
1.00
1.00
6.26
3.00

^{13}C NMR of **3j:**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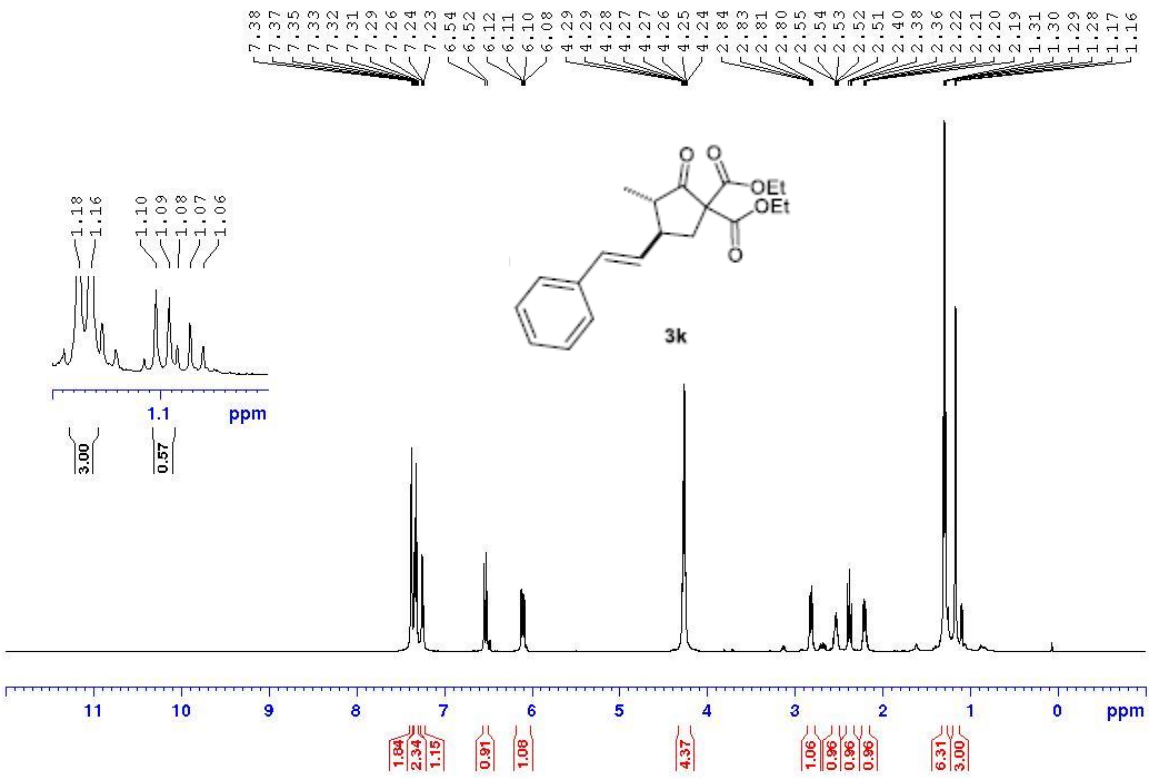
167.0
158.9
132.7
128.2
114.4
77.4
77.2
76.9
68.3
62.6
62.5
55.4
51.6
46.6
39.2
14.1
12.9
OEt
OEt
MeO
3j
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

^{1}H NMR of **3k:**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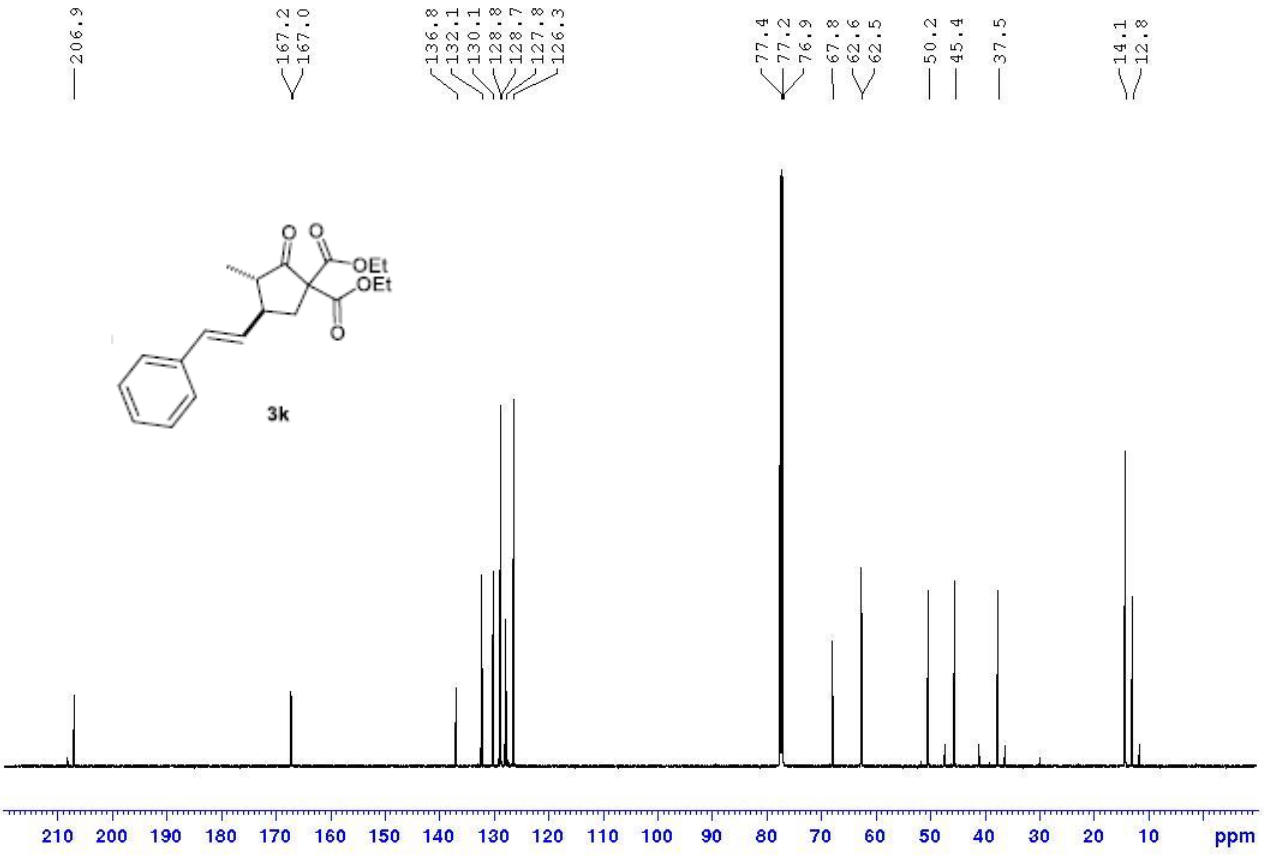
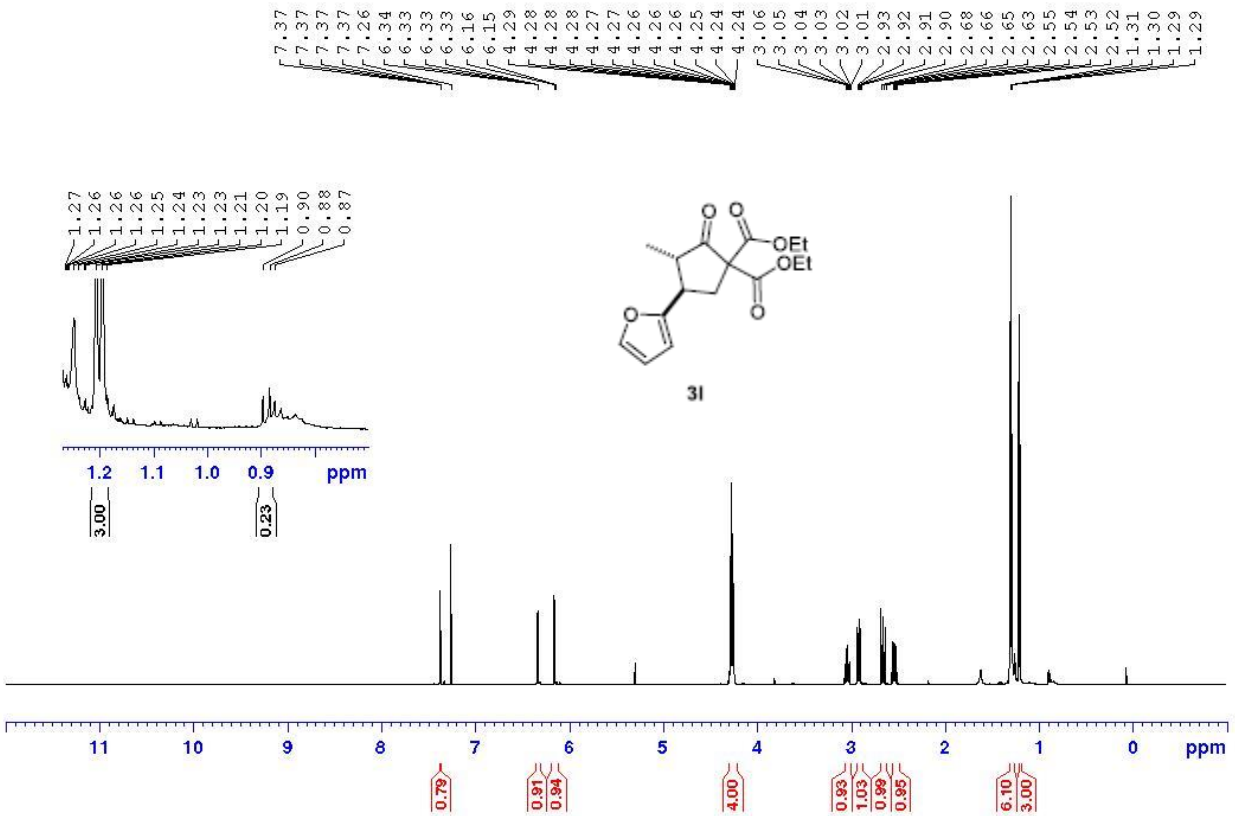
7.35
7.33
7.32
7.31
7.29
7.26
7.24
7.23
6.54
6.52
6.12
6.11
6.10
6.08
4.29
4.29
4.28
4.27
4.27
4.26
4.25
4.24
2.84
2.83
2.81
2.80
2.55
2.54
2.53
2.52
2.51
2.40
2.38
2.36
2.22
2.21
2.20
2.19
1.31
1.30
1.29
1.28
1.17
1.16
OEt
OEt
3k
1.18
1.16
1.10
1.09
1.08
1.07
1.06
1.1
ppm
3.00
0.57
11
10
9
8
7
6
5
4
3
2
1
0
ppm
1.84
2.34
1.15
0.91
1.06
4.37
1.06
0.96
0.96
0.96
6.31
3.00

^{13}C NMR of **3k:**


206.9
167.2
167.0
136.8
132.1
130.1
128.8
128.7
127.8
126.3
77.4
77.2
76.9
67.8
62.6
62.5
50.2
45.4
37.5
14.1
12.8
OEt
OEt
3k
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

^{1}H NMR of **3l:**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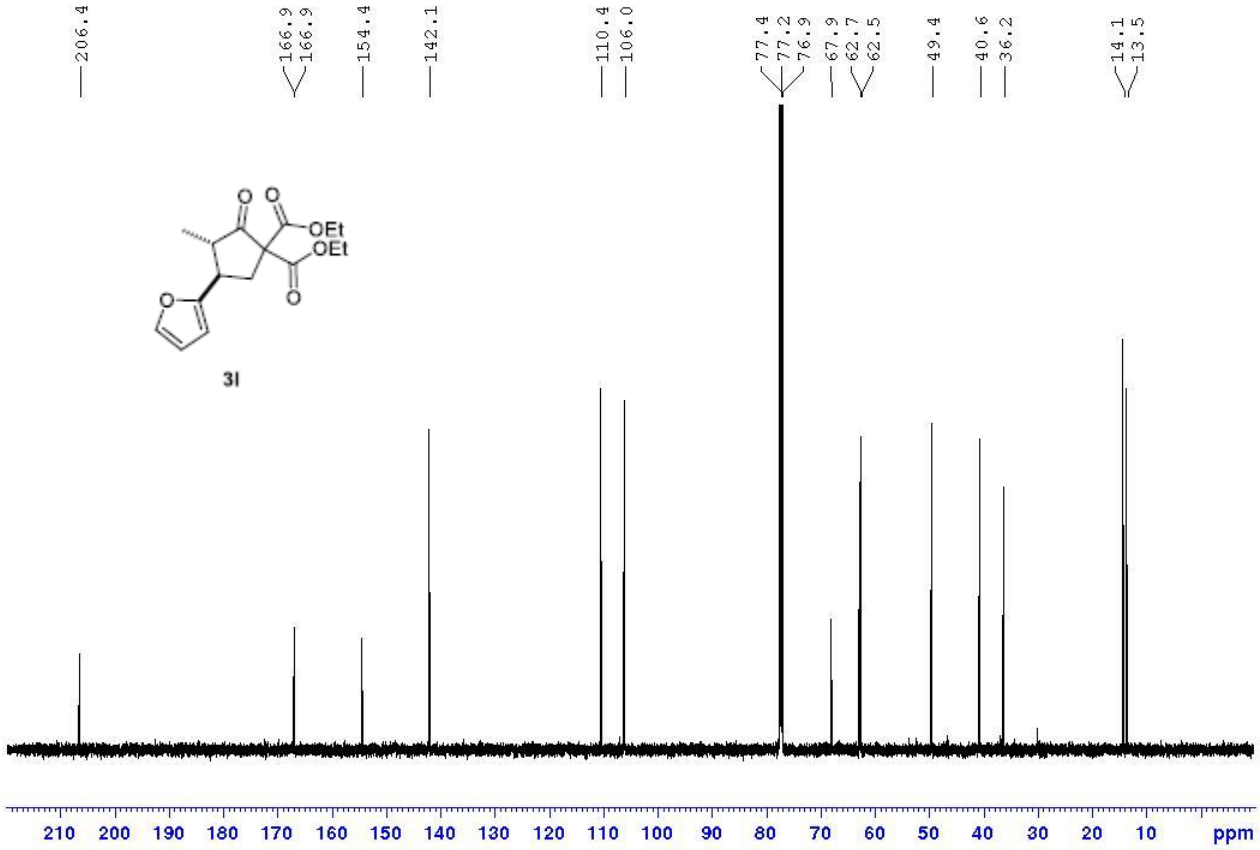
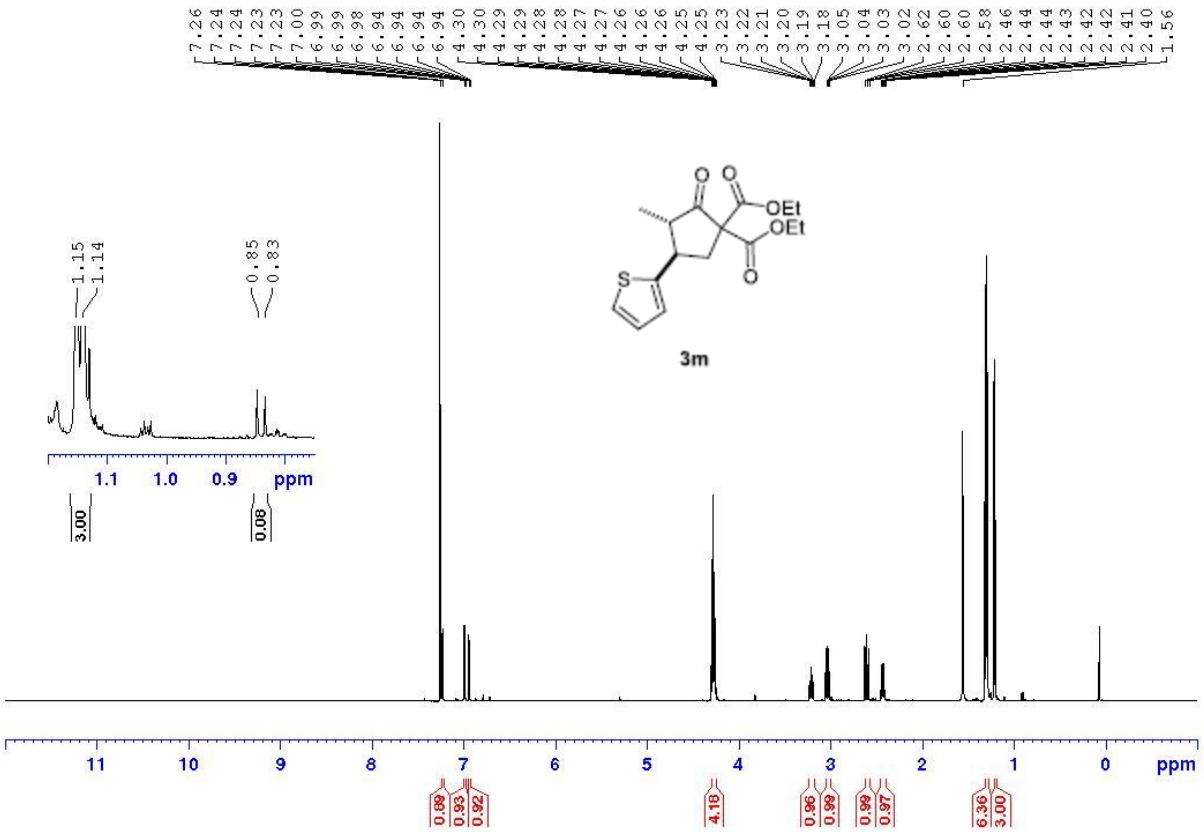
7.37
7.37
7.37
7.26
6.34
6.33
6.33
6.33
6.16
6.15
4.29
4.28
4.28
4.28
4.27
4.27
4.26
4.26
4.26
4.25
4.24
4.24
3.06
3.05
3.04
3.03
3.02
3.01
2.93
2.92
2.91
2.90
2.68
2.66
2.65
2.63
2.55
2.54
2.53
2.52
1.31
1.30
1.29
1.29
1.27
1.26
1.26
1.26
1.25
1.24
1.23
1.23
1.21
1.20
1.19
0.90
0.88
0.87
OEt
OEt
3l
1.2
1.1
1.0
0.9
ppm
3.00
0.23
11
10
9
8
7
6
5
4
3
2
1
0
ppm
0.79
0.91
0.94
4.00
0.93
1.03
0.99
0.95
6.10
3.00

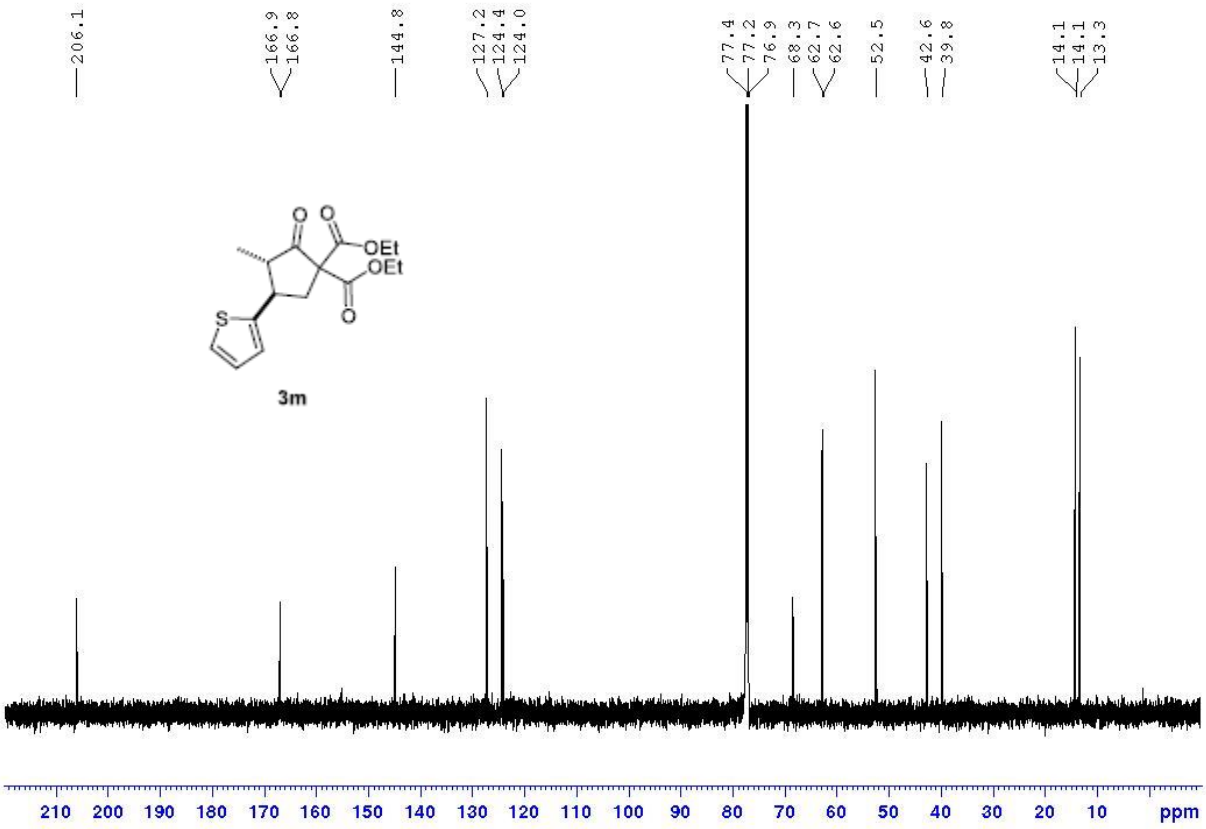
^{13}C NMR of **3l:**


206.4
166.9
166.9
154.4
142.1
110.4
106.0
77.4
77.2
76.9
67.9
62.7
62.5
49.4
40.6
36.2
14.1
13.5
OEt
OEt
3l
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

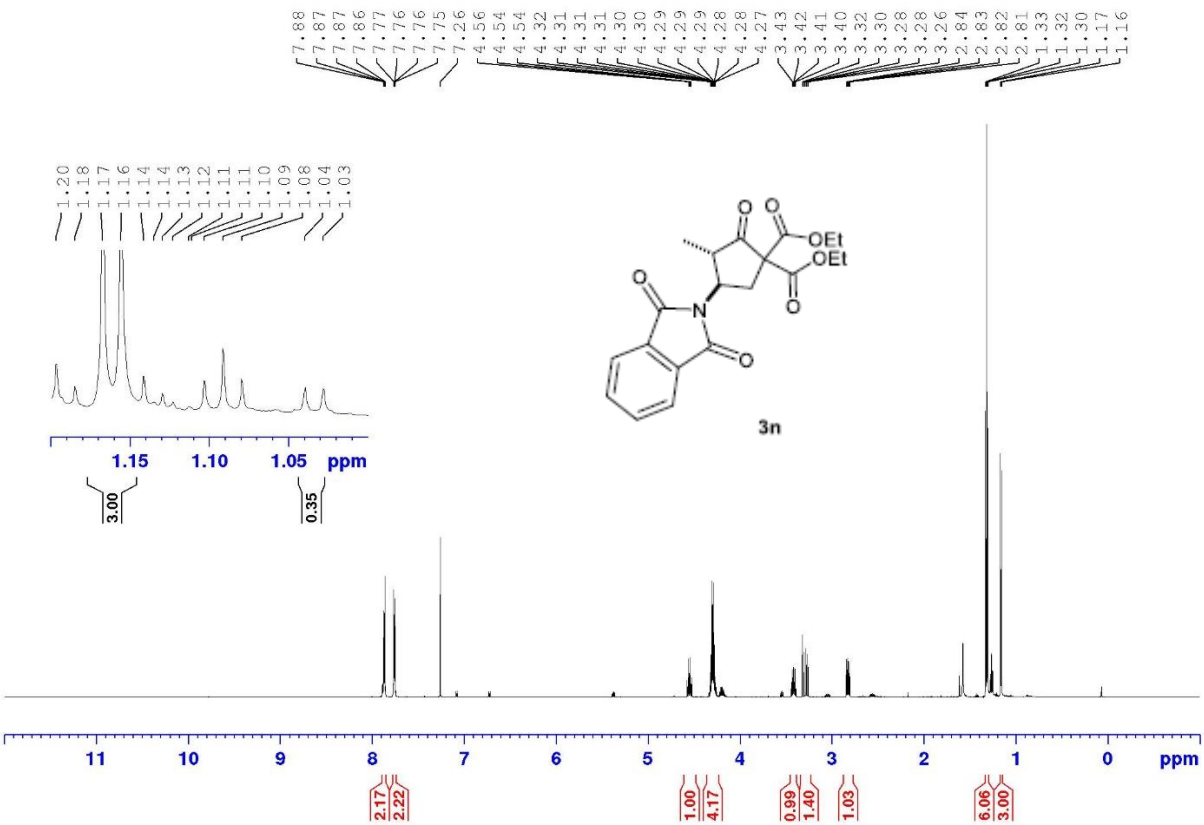
^{1}H NMR of **3m:**


7.26
7.24
7.24
7.23
7.23
7.00
6.99
6.98
6.94
6.94
6.94
4.30
4.29
4.29
4.28
4.27
4.27
4.26
4.26
4.25
3.23
3.22
3.21
3.20
3.19
3.18
3.05
3.04
3.03
3.02
2.62
2.60
2.60
2.58
2.46
2.44
2.44
2.43
2.42
2.42
2.41
2.40
1.56
1.15
1.14
0.85
0.83
1.1
1.0
0.9
ppm
3.00
0.08
OEt
OEt
S
3m
11
10
9
8
7
6
5
4
3
2
1
0
ppm
0.89
0.93
0.92
4.18
0.96
0.99
0.99
0.97
6.36
3.00

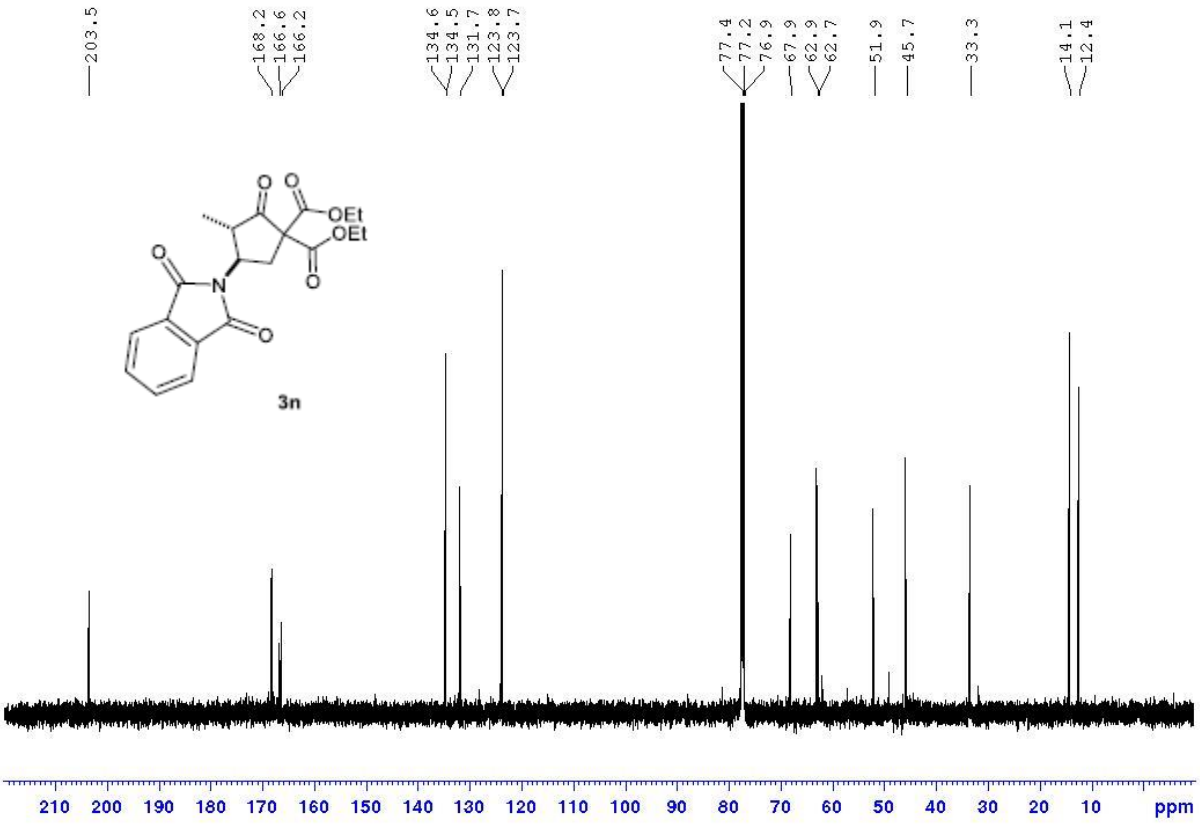
^{13}C NMR of **3m:**


206.1
166.9
166.8
144.8
127.2
124.4
124.0
77.4
77.2
76.9
68.3
62.7
62.6
52.5
42.6
39.8
14.1
14.1
13.3
OEt
OEt
S
3m
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

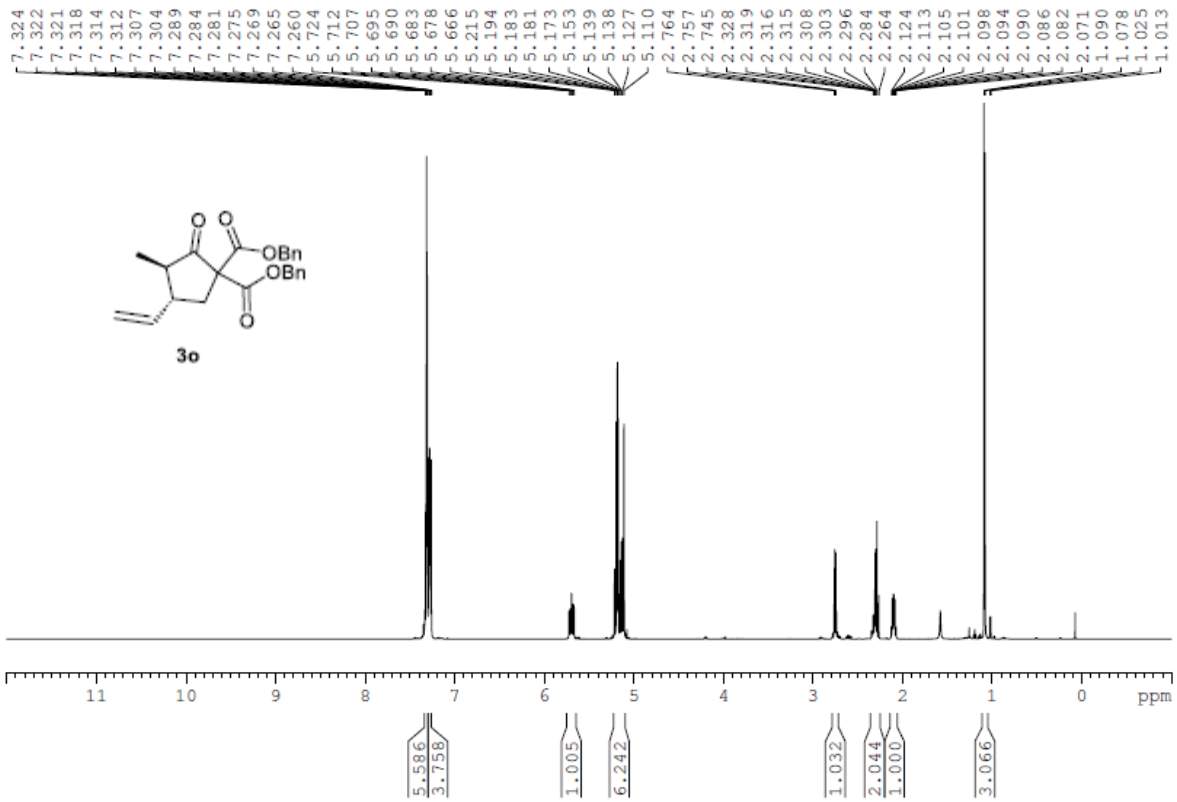
^{1}H NMR of **3n:**


7.88
7.87
7.87
7.86
7.77
7.76
7.76
7.75
7.26
4.56
4.54
4.54
4.32
4.31
4.31
4.31
4.30
4.30
4.29
4.29
4.29
4.28
4.28
4.27
3.43
3.42
3.41
3.40
3.32
3.30
3.28
3.28
3.26
2.84
2.83
2.82
2.81
1.33
1.32
1.30
1.17
1.16
1.20
1.18
1.17
1.16
1.14
1.14
1.13
1.12
1.11
1.11
1.10
1.09
1.08
1.04
1.03
OEt
OEt
3n
1.15
1.10
1.05
ppm
3.00
0.35
11
10
9
8
7
6
5
4
3
2
1
0
ppm
2.17
2.22
1.00
4.17
0.99
1.40
1.03
6.06
3.00

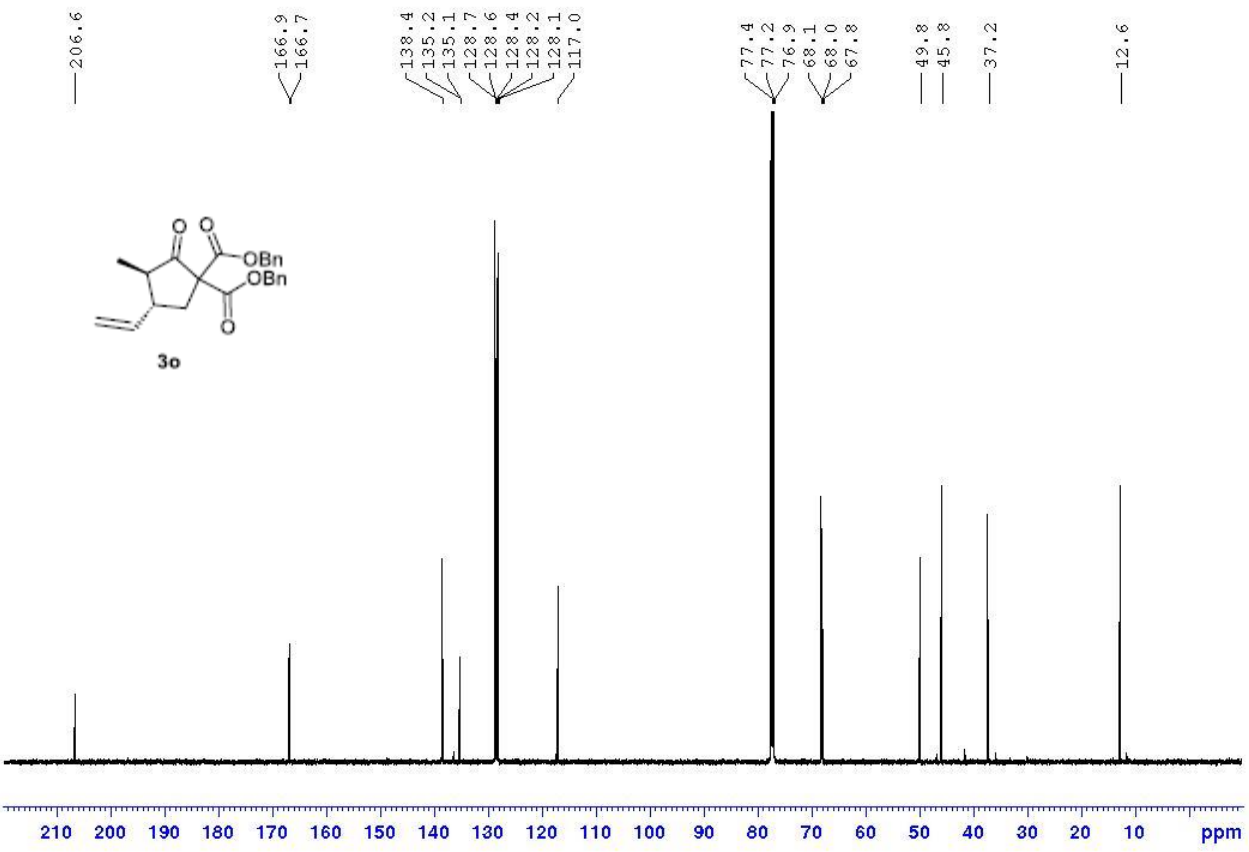
^{13}C NMR of **3n:**


203.5
168.2
166.6
166.2
134.6
134.5
131.7
123.8
123.7
77.4
77.2
76.9
67.9
62.9
62.7
51.9
45.7
33.3
14.1
12.4
OEt
OEt
3n
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

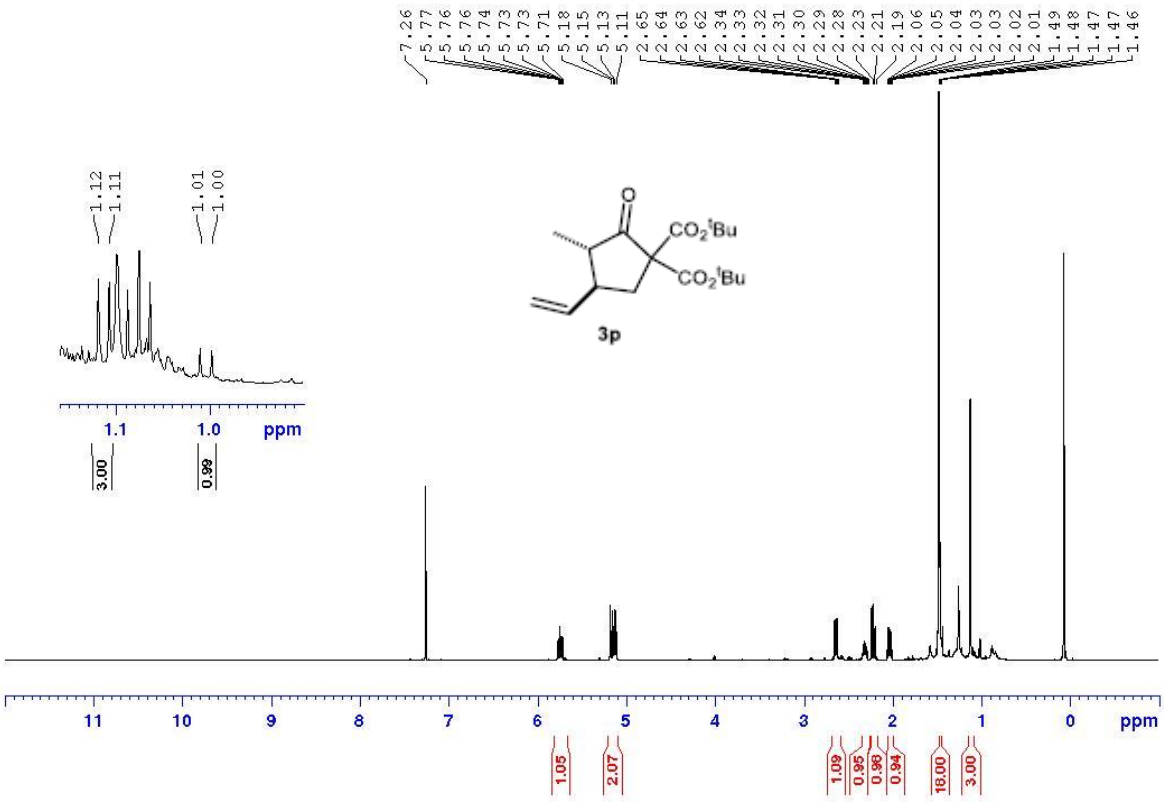
^{1}H NMR of **3o:**


7.324
7.322
7.321
7.318
7.314
7.312
7.307
7.304
7.289
7.284
7.281
7.275
7.269
7.265
7.260
5.724
5.712
5.707
5.695
5.690
5.683
5.678
5.666
5.215
5.194
5.183
5.181
5.173
5.153
5.139
5.138
5.127
5.110
2.764
2.757
2.745
2.328
2.319
2.316
2.315
2.308
2.303
2.296
2.284
2.264
2.124
2.113
2.105
2.101
2.098
2.094
2.090
2.086
2.082
2.071
1.090
1.078
1.025
1.013
OBn
OBn
3o
11
10
9
8
7
6
5
4
3
2
1
0
ppm
5.586
3.758
1.005
6.242
1.032
2.044
1.000
3.066

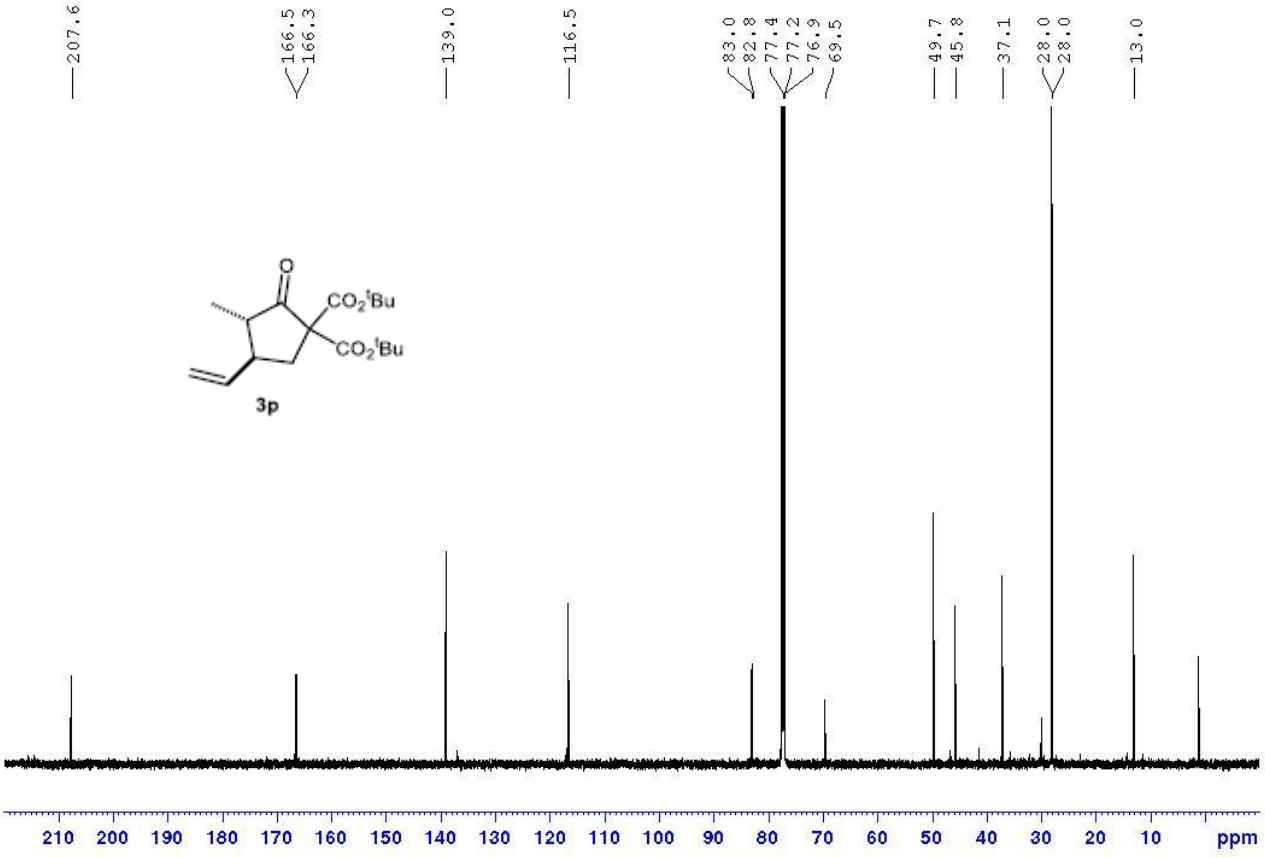
^{13}C NMR of **3o:**


206.6
166.9
166.7
138.4
135.2
135.1
128.7
128.6
128.4
128.2
128.1
117.0
77.4
77.2
76.9
68.1
68.0
67.8
49.8
45.8
37.2
12.6
OBn
OBn
3o
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

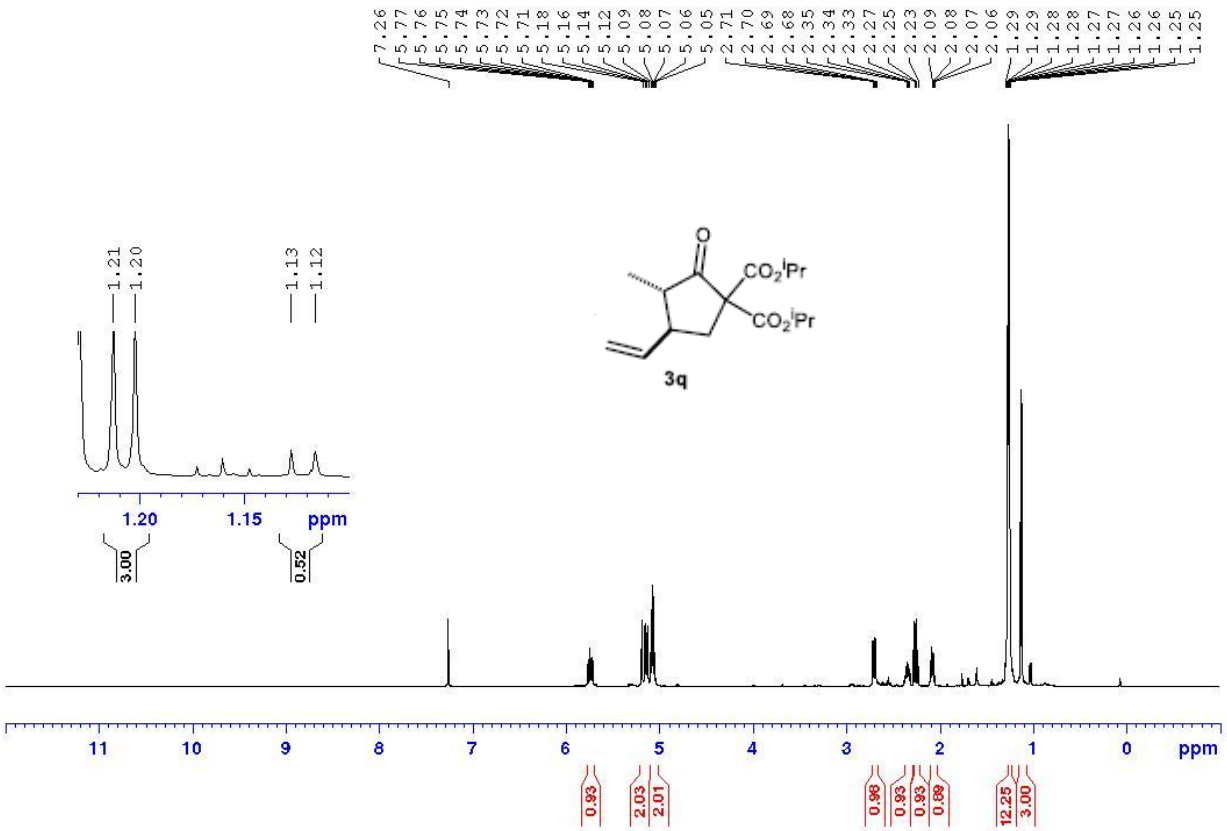
^{1}H NMR of **3p:**


7.26
5.77
5.76
5.76
5.74
5.73
5.73
5.71
5.18
5.15
5.13
5.11
2.65
2.64
2.63
2.62
2.34
2.33
2.32
2.31
2.30
2.29
2.28
2.23
2.21
2.19
2.06
2.05
2.04
2.03
2.03
2.02
2.01
1.49
1.48
1.47
1.47
1.46
1.12
1.11
1.01
1.00
CO2tBu
CO2tBu
3p
1.1
1.0
ppm
3.00
0.99
11
10
9
8
7
6
5
4
3
2
1
0
ppm
1.05
2.07
1.09
0.95
0.96
0.94
18.00
3.00

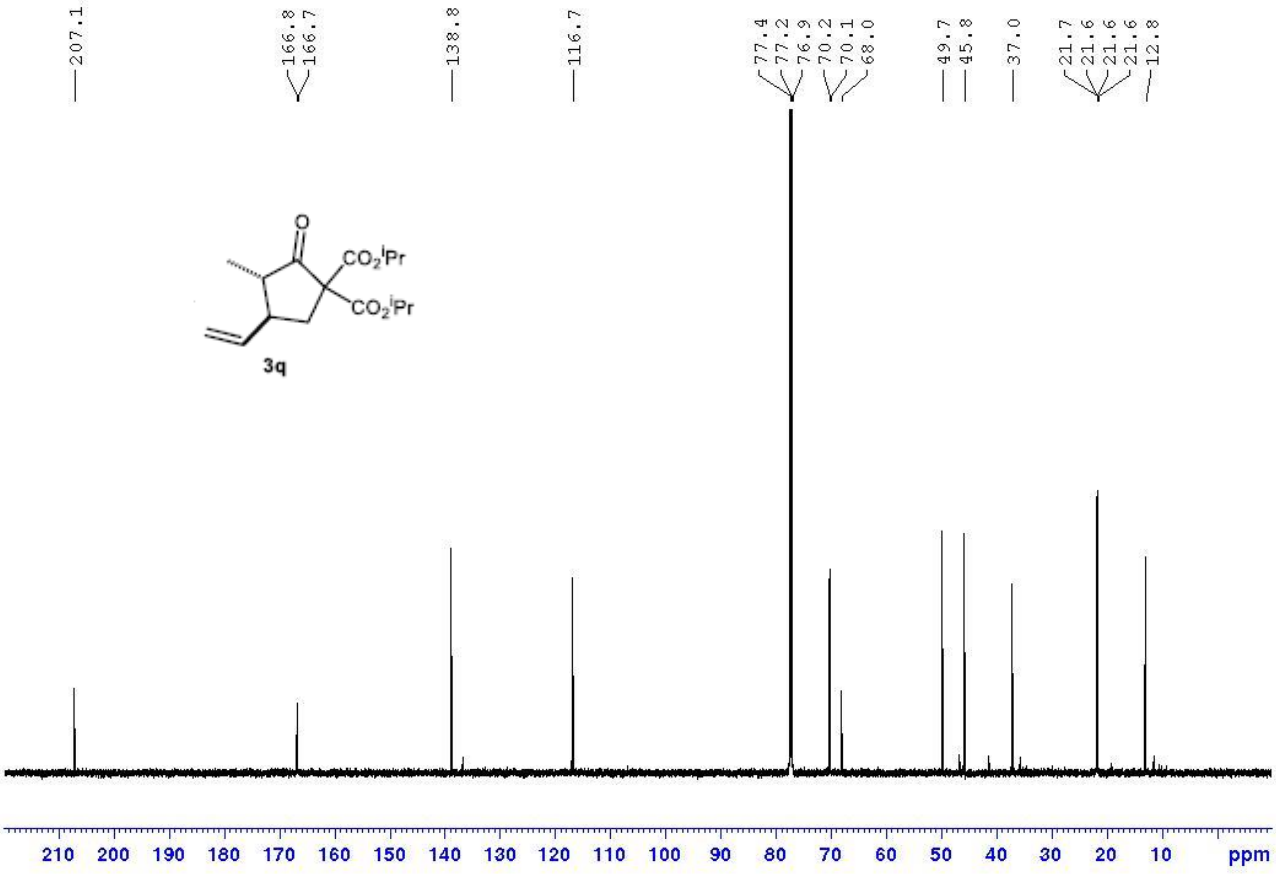
^{13}C NMR of **3p:**


207.6
166.5
166.3
139.0
116.5
83.0
82.8
77.4
77.2
76.9
69.5
49.7
45.8
37.1
28.0
28.0
13.0
O
CO2tBu
CO2tBu
3p
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

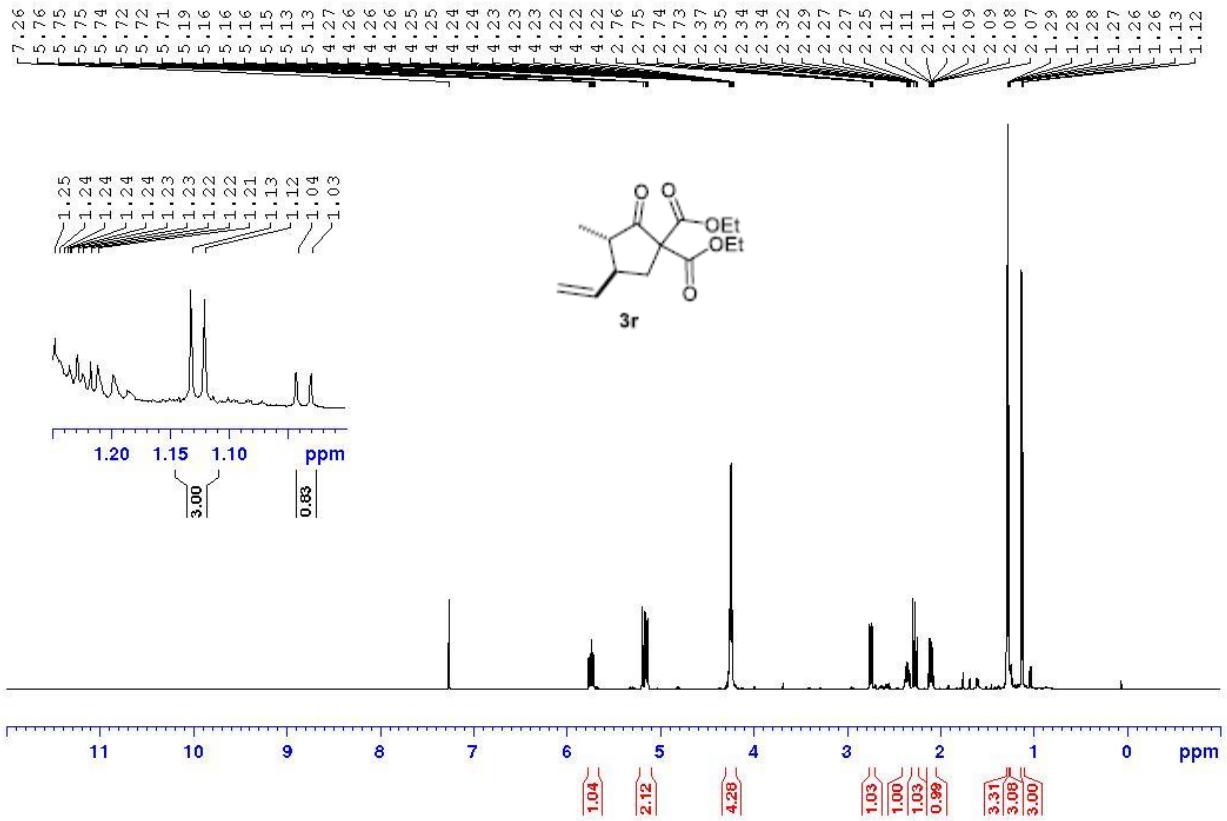
^{1}H NMR of **3q:**


7.26
5.77
5.76
5.75
5.74
5.73
5.72
5.71
5.18
5.16
5.14
5.12
5.09
5.08
5.07
5.06
5.05
2.71
2.70
2.69
2.68
2.35
2.34
2.33
2.27
2.25
2.23
2.09
2.08
2.07
2.06
1.29
1.29
1.28
1.28
1.27
1.27
1.26
1.26
1.25
1.25
1.21
1.20
1.13
1.12
1.20
1.15
ppm
3.00
0.52
O
CO2iPr
CO2iPr
3q
11
10
9
8
7
6
5
4
3
2
1
0
ppm
0.93
2.03
2.01
0.96
0.93
0.93
0.89
12.25
3.00

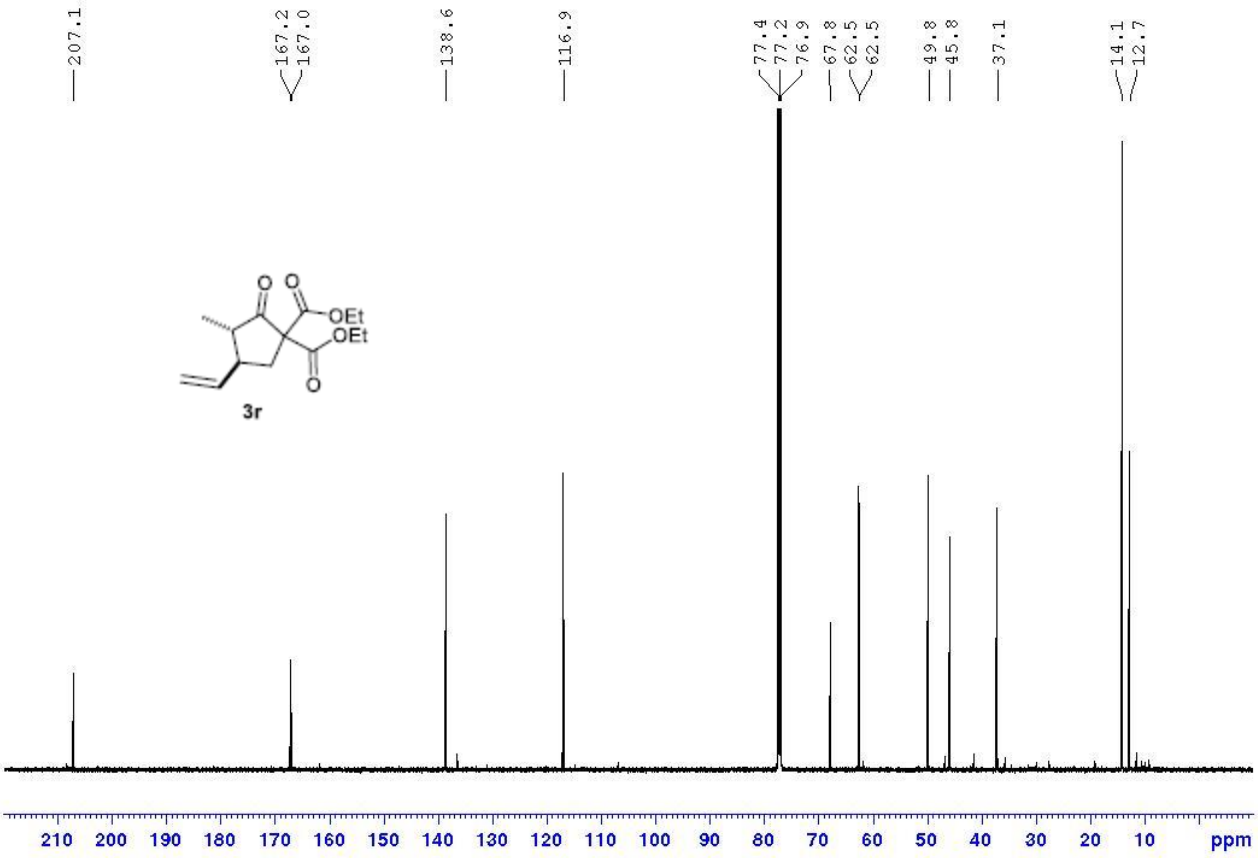
^{13}C NMR of **3q:**


207.1
166.8
166.7
138.8
116.7
77.4
77.2
76.9
70.2
70.1
68.0
49.7
45.8
37.0
21.7
21.6
21.6
21.6
12.8
O
CO2iPr
CO2iPr
3q
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

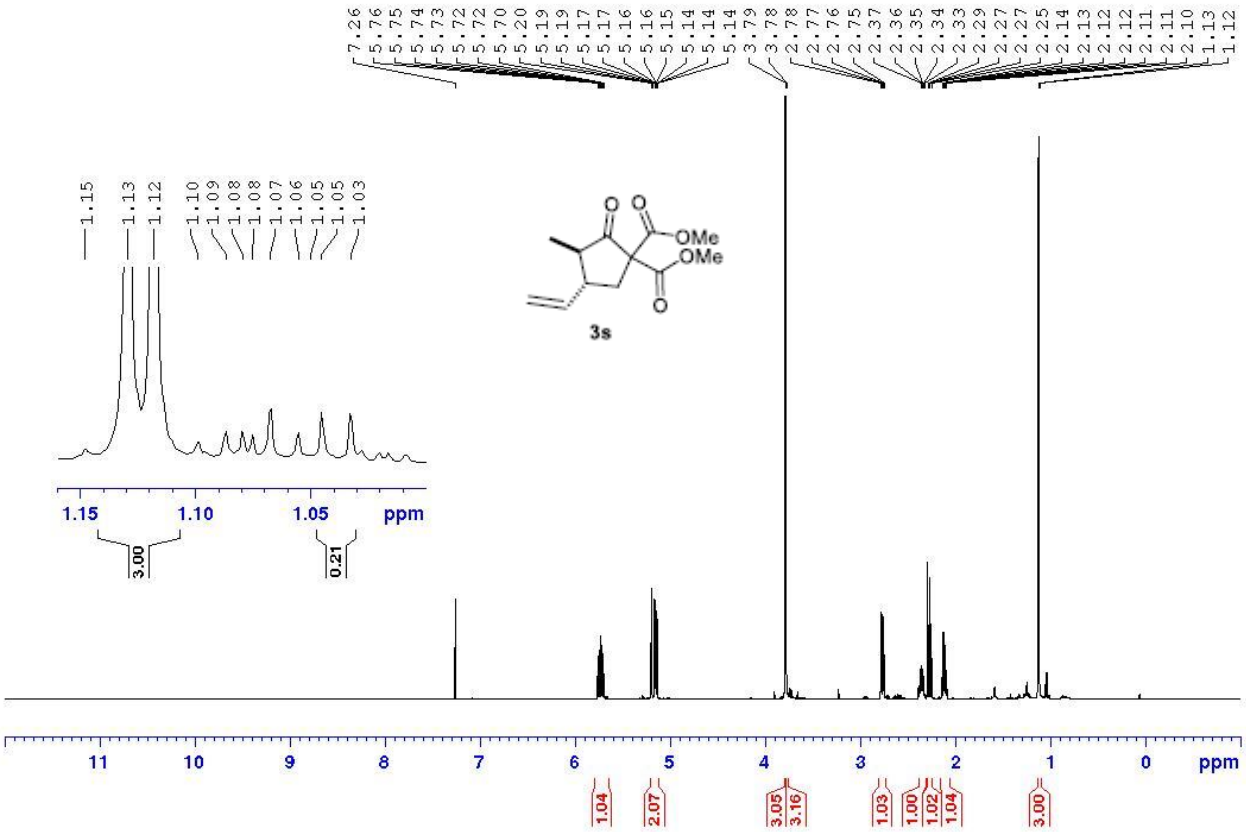
^{1}H NMR of **3r:**


7.26 5.76 5.75 5.75 5.74 5.72 5.72 5.71 5.19 5.16 5.16 5.16 5.15 5.13 5.13 4.27 4.26 4.26 4.26 4.25 4.25 4.24 4.24 4.24 4.23 4.23 4.23 4.22 4.22 4.22 4.22 2.76 2.75 2.74 2.73 2.37 2.35 2.34 2.34 2.32 2.29 2.27 2.27 2.25 2.12 2.11 2.11 2.10 2.09 2.09 2.08 2.07 1.29 1.28 1.28 1.27 1.26 1.26 1.13 1.12
1.25 1.24 1.24 1.24 1.24 1.24 1.23 1.23 1.22 1.22 1.21 1.13 1.12 1.04 1.03
OEt
OEt
3r
1.20 1.15 1.10 ppm
3.00
0.83
11 10 9 8 7 6 5 4 3 2 1 0 ppm
1.04
2.12
4.28
1.03
1.00
1.03
0.99
3.31
3.08
3.00

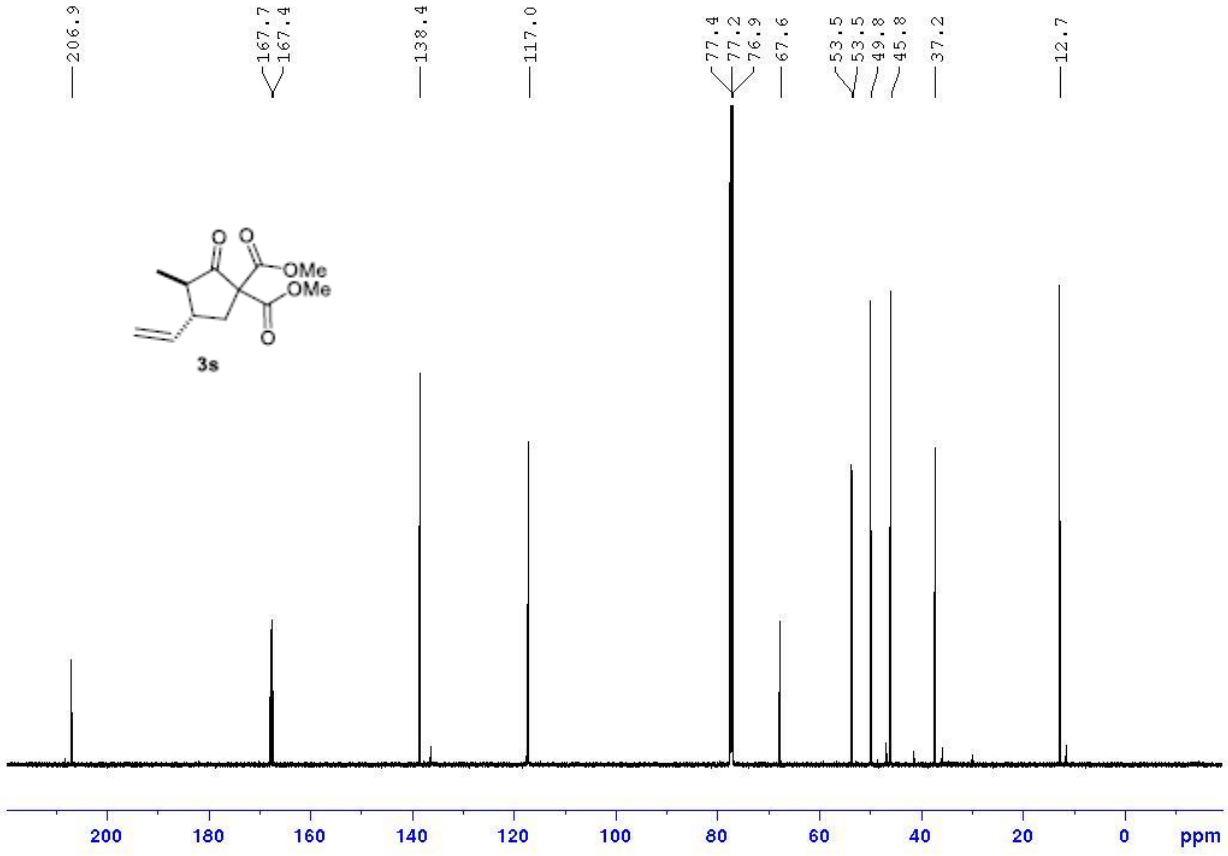
^{13}C NMR of **3r:**


207.1
167.2
167.0
138.6
116.9
77.4
77.2
76.9
67.8
62.5
62.5
49.8
45.8
37.1
14.1
12.7
OEt
OEt
3r
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

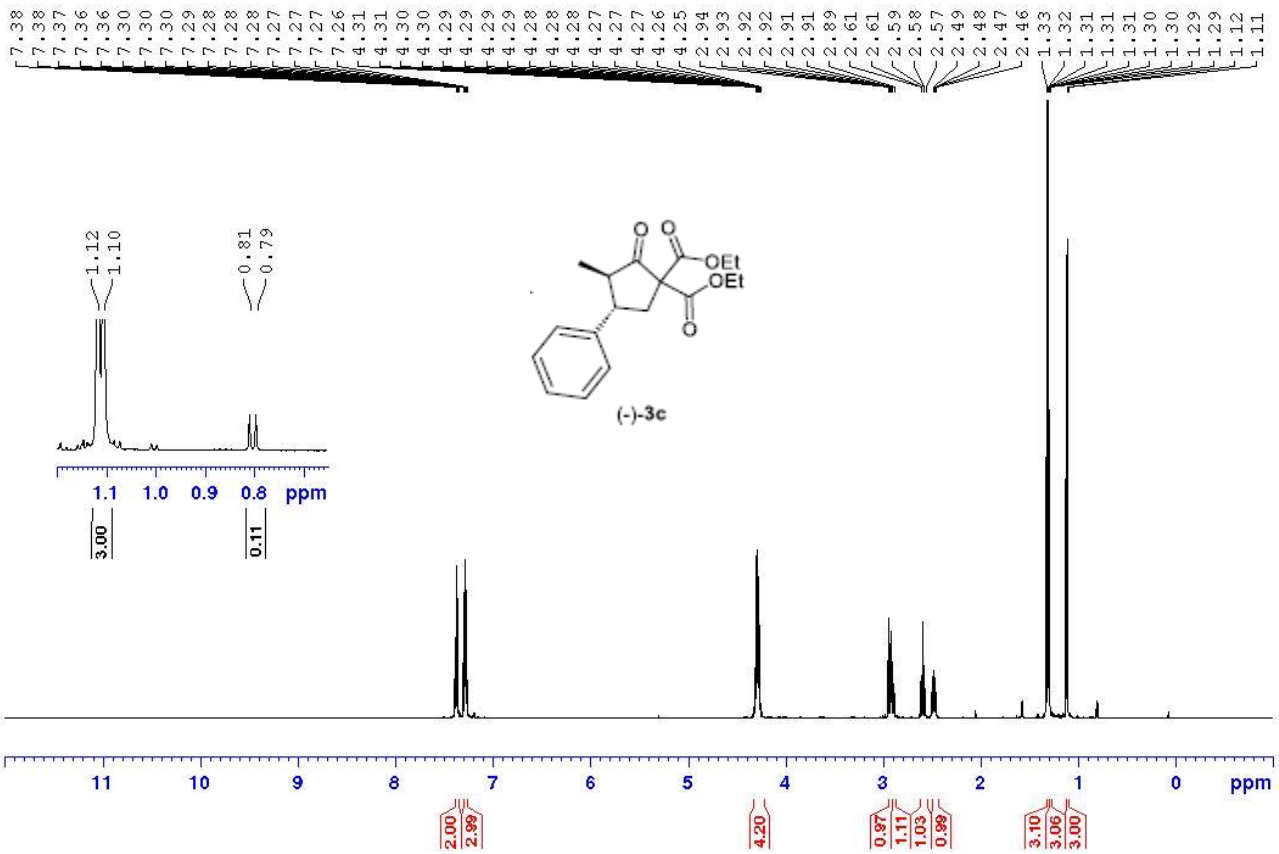
^{1}H NMR of **3s:**


7.26
5.76
5.75
5.74
5.73
5.72
5.72
5.70
5.20
5.19
5.19
5.17
5.17
5.16
5.16
5.15
5.14
5.14
5.14
3.79
3.78
2.78
2.77
2.76
2.75
2.37
2.36
2.35
2.34
2.33
2.29
2.27
2.27
2.25
2.14
2.13
2.12
2.12
2.11
2.11
2.10
1.13
1.12
1.15
1.13
1.12
1.10
1.09
1.08
1.08
1.07
1.06
1.05
1.05
1.03
OMe
OMe
O
O
O
O
3s
1.15
1.10
1.05
ppm
3.00
0.21
11
10
9
8
7
6
5
4
3
2
1
0
ppm
1.04
2.07
3.05
3.16
1.03
1.00
1.02
1.04
3.00

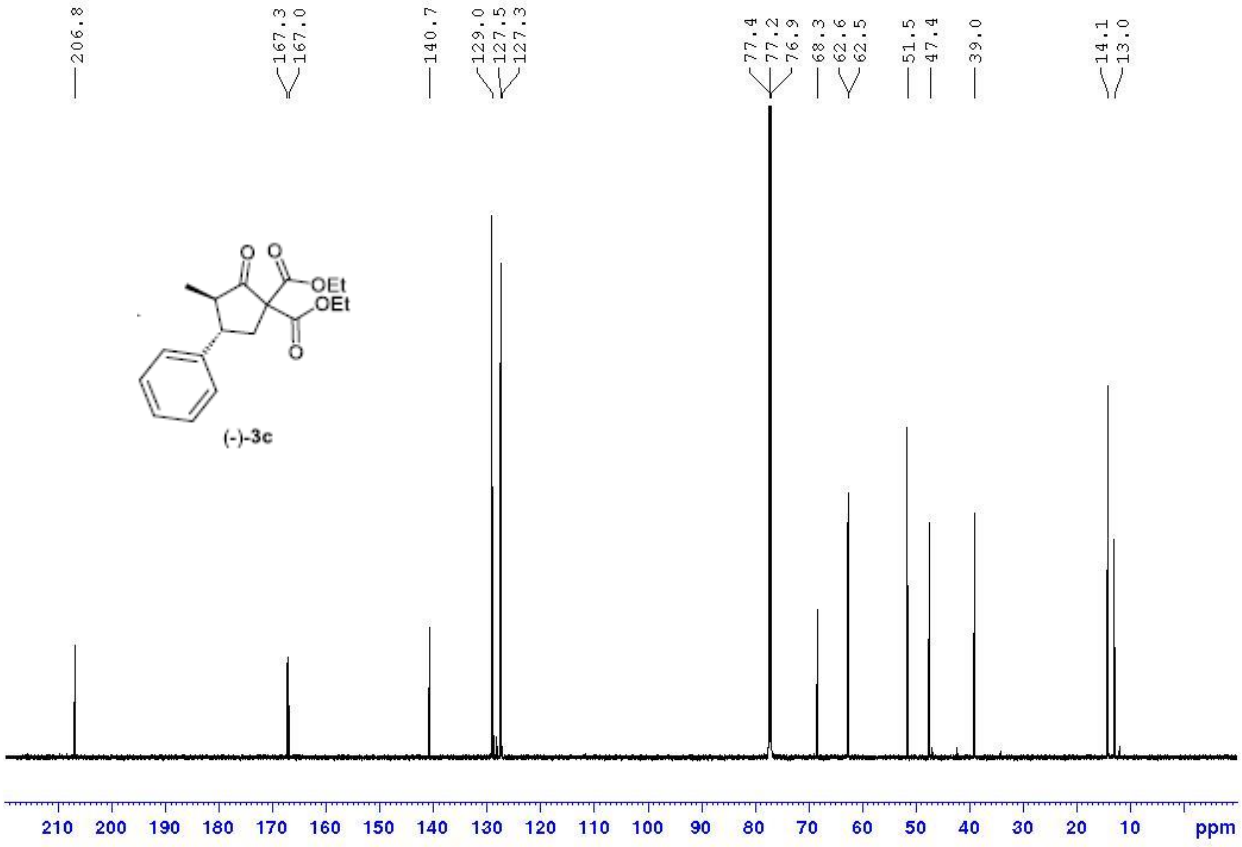
^{13}C NMR of **3s:**


206.9
167.7
167.4
138.4
117.0
77.4
77.2
76.9
67.6
53.5
53.5
49.8
45.8
37.2
12.7
OMe
OMe
3s
200
180
160
140
120
100
80
60
40
20
0
ppm

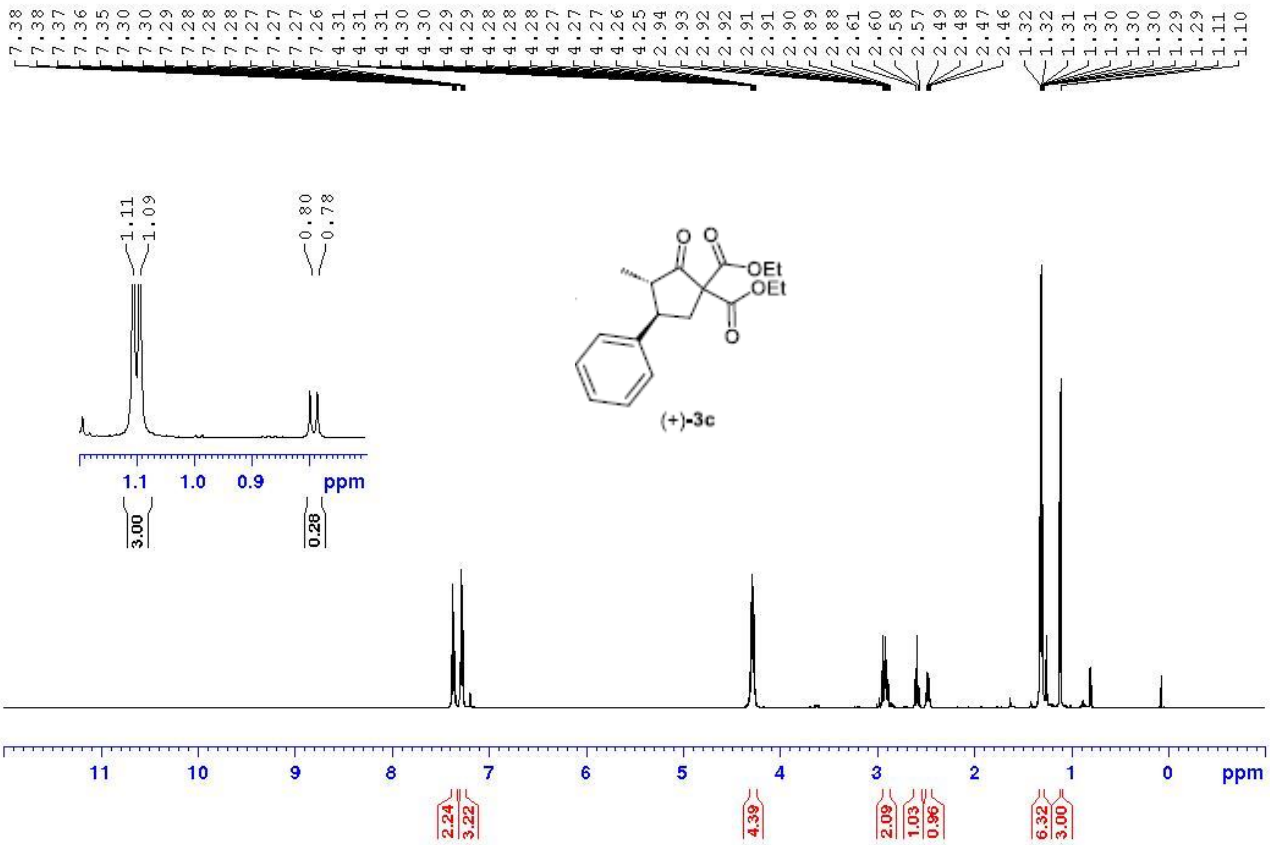
^{1}H NMR of (–)-**3c:**


7.38 7.38 7.37 7.36 7.36 7.30 7.30 7.29 7.28 7.28 7.27 7.27 7.27 7.26 4.31 4.31 4.30 4.30 4.29 4.29 4.29 4.29 4.28 4.28 4.28 4.27 4.27 4.27 4.26 4.25 2.94 2.93 2.92 2.92 2.91 2.91 2.89 2.61 2.61 2.59 2.58 2.57 2.49 2.48 2.47 2.46 1.33 1.32 1.31 1.31 1.31 1.30 1.30 1.29 1.29 1.12 1.11
1.12 1.10 0.81 0.79
1.1 1.0 0.9 0.8 ppm
3.00 0.11
OEt OEt
(-)-3c
11 10 9 8 7 6 5 4 3 2 1 0 ppm
2.00 2.99 4.20 0.97 1.11 1.03 0.98 3.10 3.06 3.00

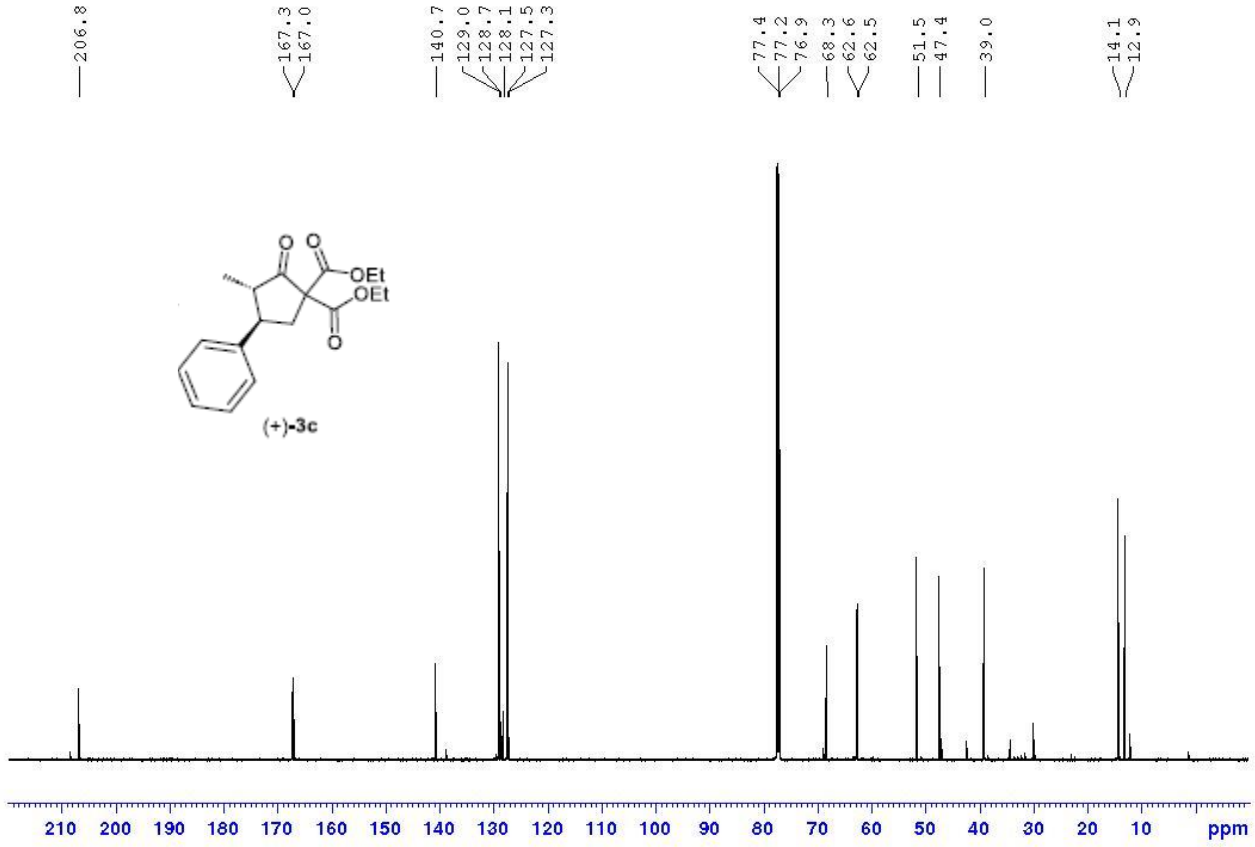
^{13}C NMR of (–)-**3c:**


206.8
167.3
167.0
140.7
129.0
127.5
127.3
77.4
77.2
76.9
68.3
62.6
62.5
51.5
47.4
39.0
14.1
13.0
OEt
OEt
(-)-3c
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

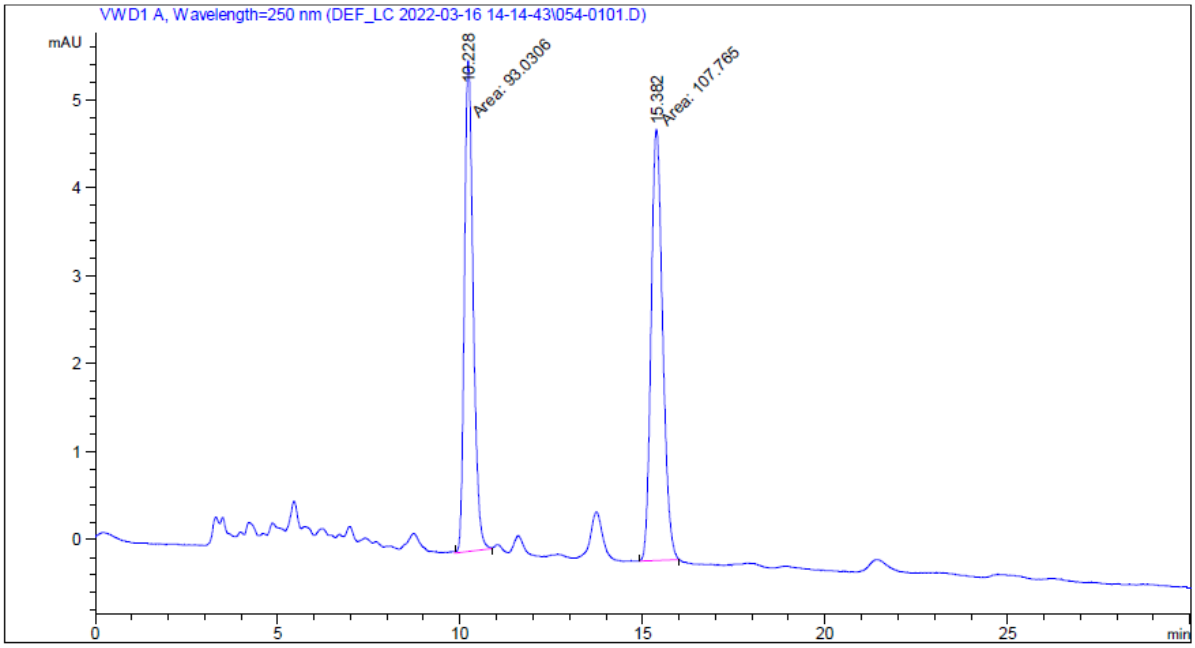
^{1}H NMR of (+)-**3c:**


7.38 7.38 7.37 7.36 7.35 7.30 7.30 7.29 7.28 7.28 7.28 7.27 7.27 7.27 7.26 4.31 4.31 4.31 4.30 4.30 4.29 4.29 4.28 4.28 4.28 4.27 4.27 4.27 4.27 4.26 4.25 2.94 2.93 2.92 2.92 2.91 2.91 2.90 2.89 2.88 2.61 2.60 2.58 2.57 2.49 2.48 2.47 2.46 1.32 1.32 1.31 1.31 1.30 1.30 1.29 1.29 1.11 1.10
1.11
1.09
0.80
0.78
1.1
1.0
0.9
ppm
3.00
0.28
OEt
OEt
(+)-3c
11
10
9
8
7
6
5
4
3
2
1
0
ppm
2.24
3.22
4.39
2.09
1.03
0.96
6.32
3.00

^{13}C NMR of (+)-**3c:**


206.8
167.3
167.0
140.7
129.0
128.7
128.1
127.5
127.3
77.4
77.2
76.9
68.3
62.6
62.5
51.5
47.4
39.0
14.1
12.9
OEt
OEt
(+)-3c
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

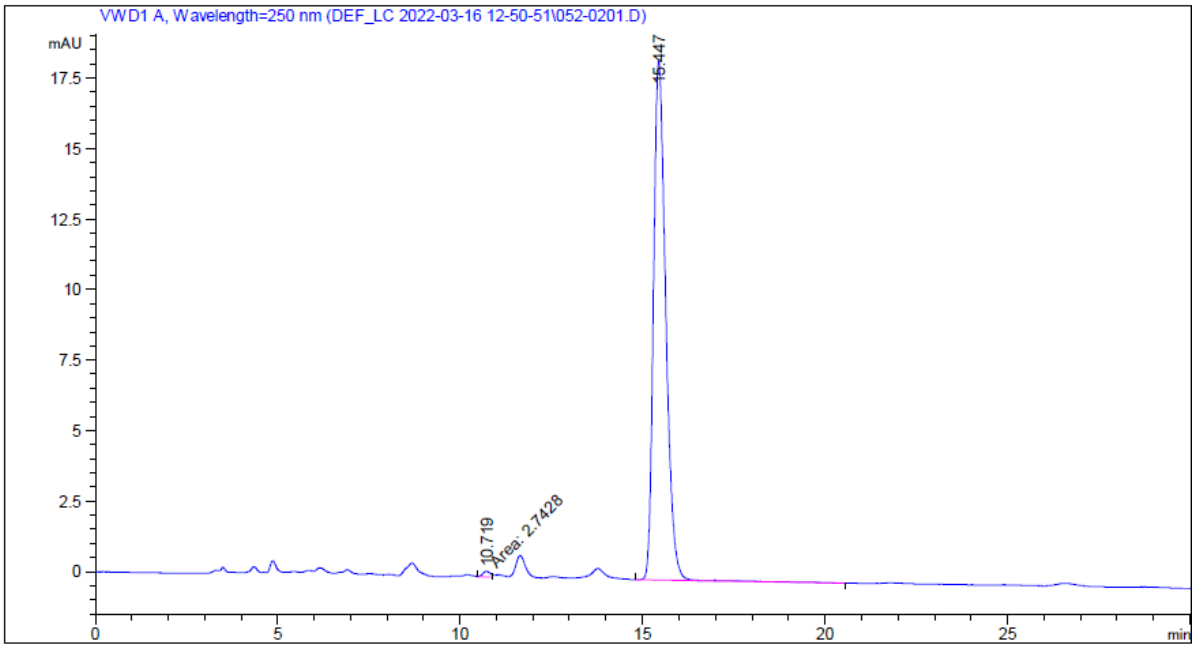
HPLC of (±)-**3c:**



Signal 1: VWD1 A, Wavelength=250 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.228	MM	0.2772	93.03059	5.59353	46.3310
2	15.382	MM	0.3662	107.76504	4.90498	53.6690
Totals :				200.79562	10.49851	

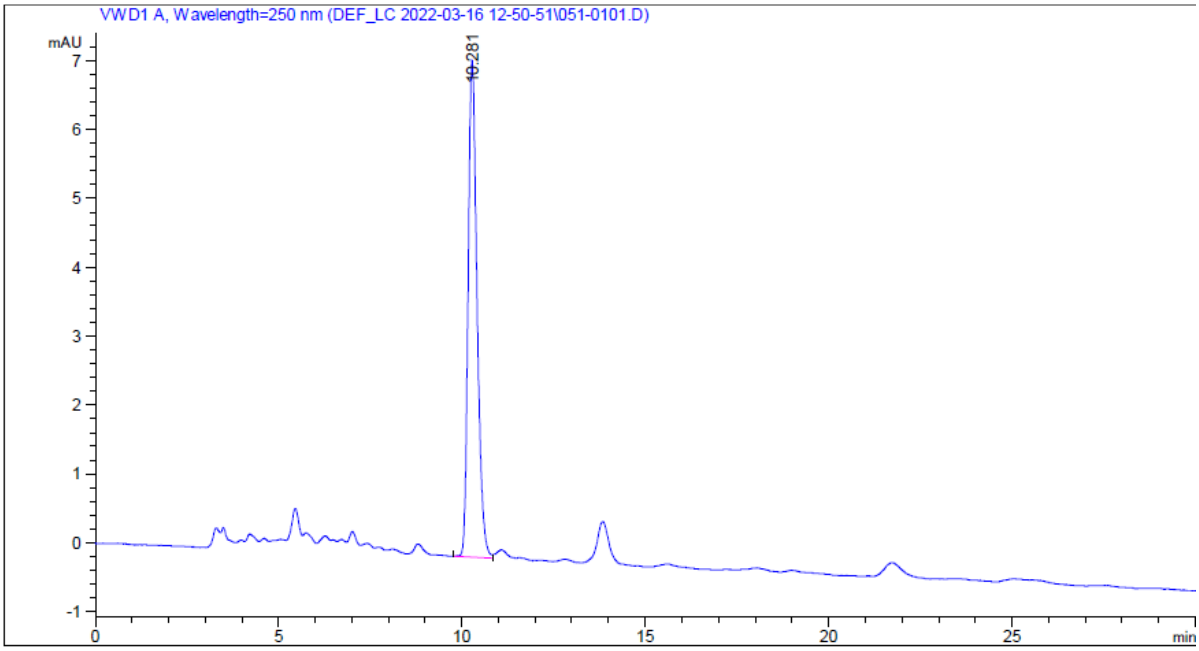
HPLC of (–)-**3c:**



Signal 1: VWD1 A, Wavelength=250 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.719	MF	0.2333	2.74280	1.95942e-1	0.6335
2	15.447	BB	0.3661	430.19397	18.40050	99.3665
Totals :				432.93677	18.59644	

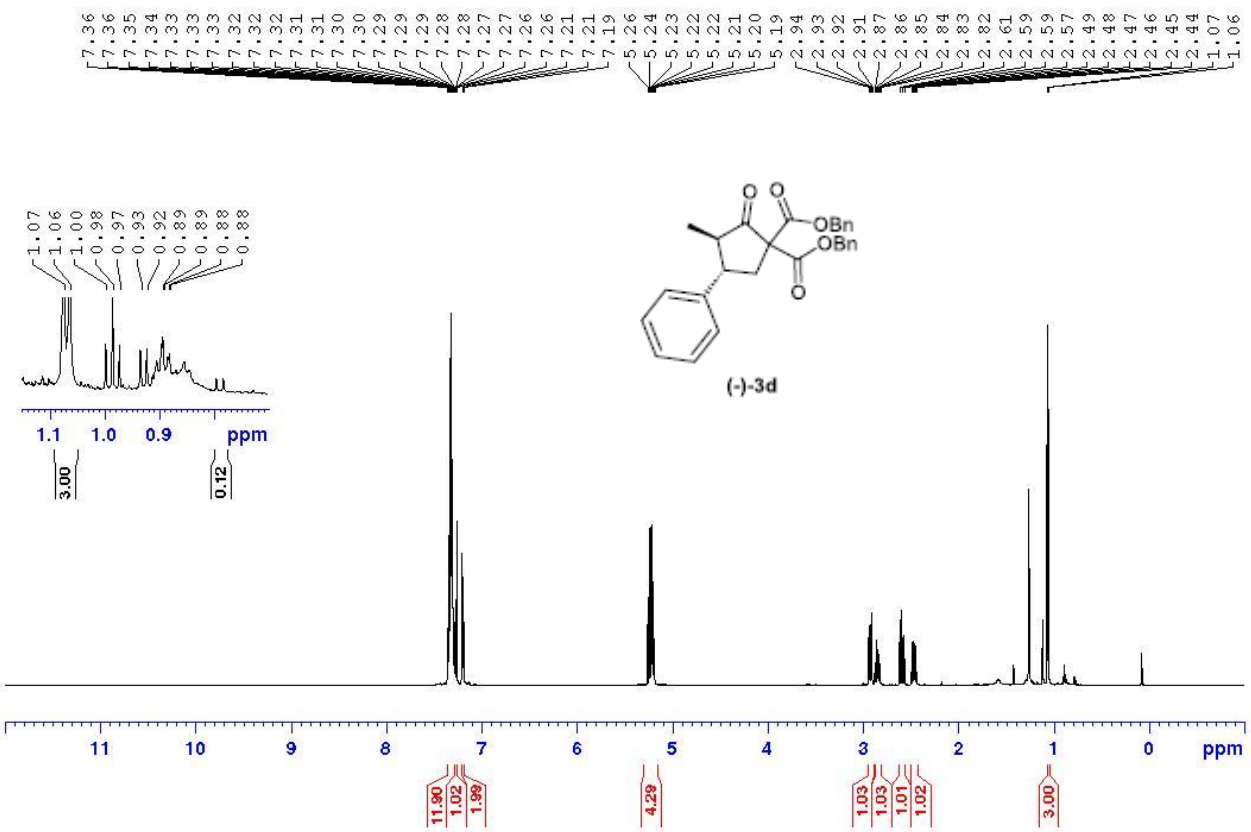
HPLC of (+)-**3c:**



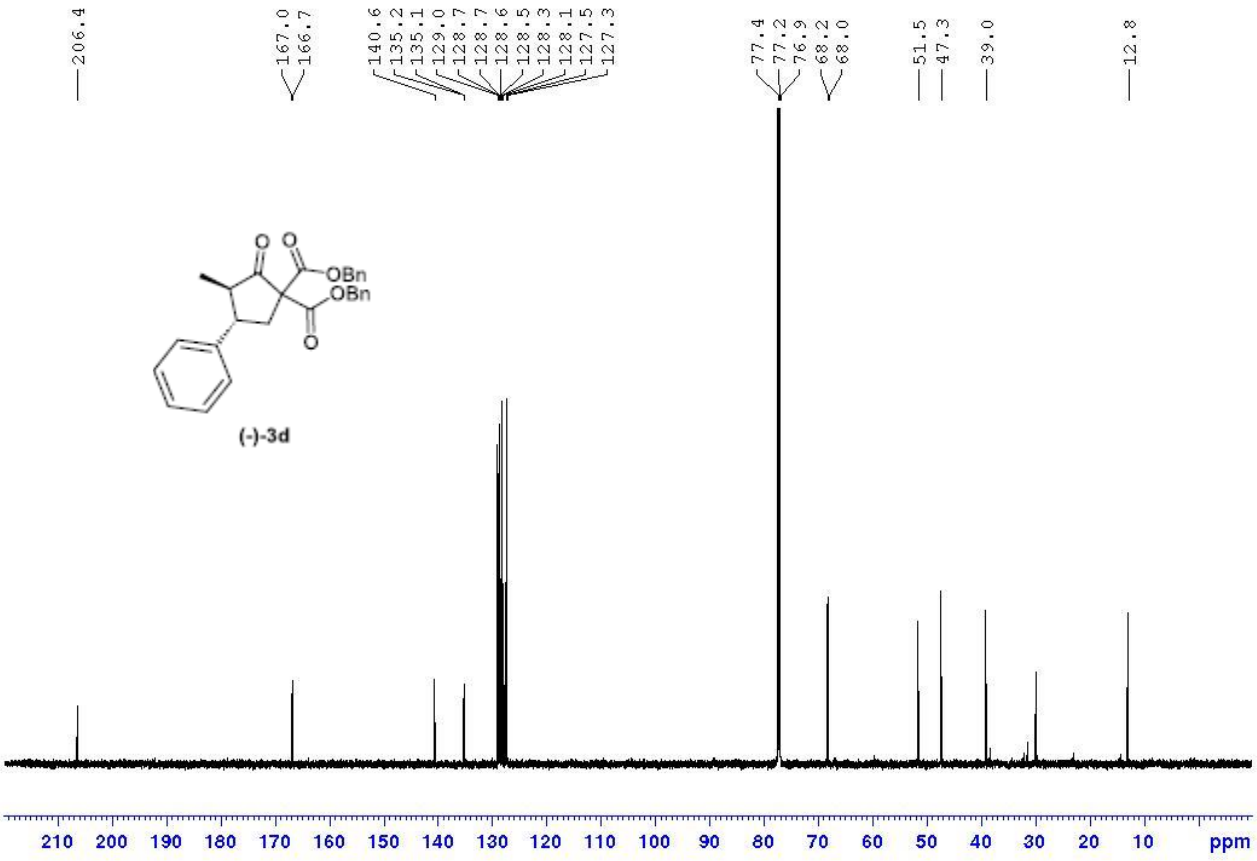
Signal 1: VWD1 A, Wavelength=250 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.281	BV	0.2678	121.96597	7.19289	100.0000
Totals :				121.96597	7.19289	

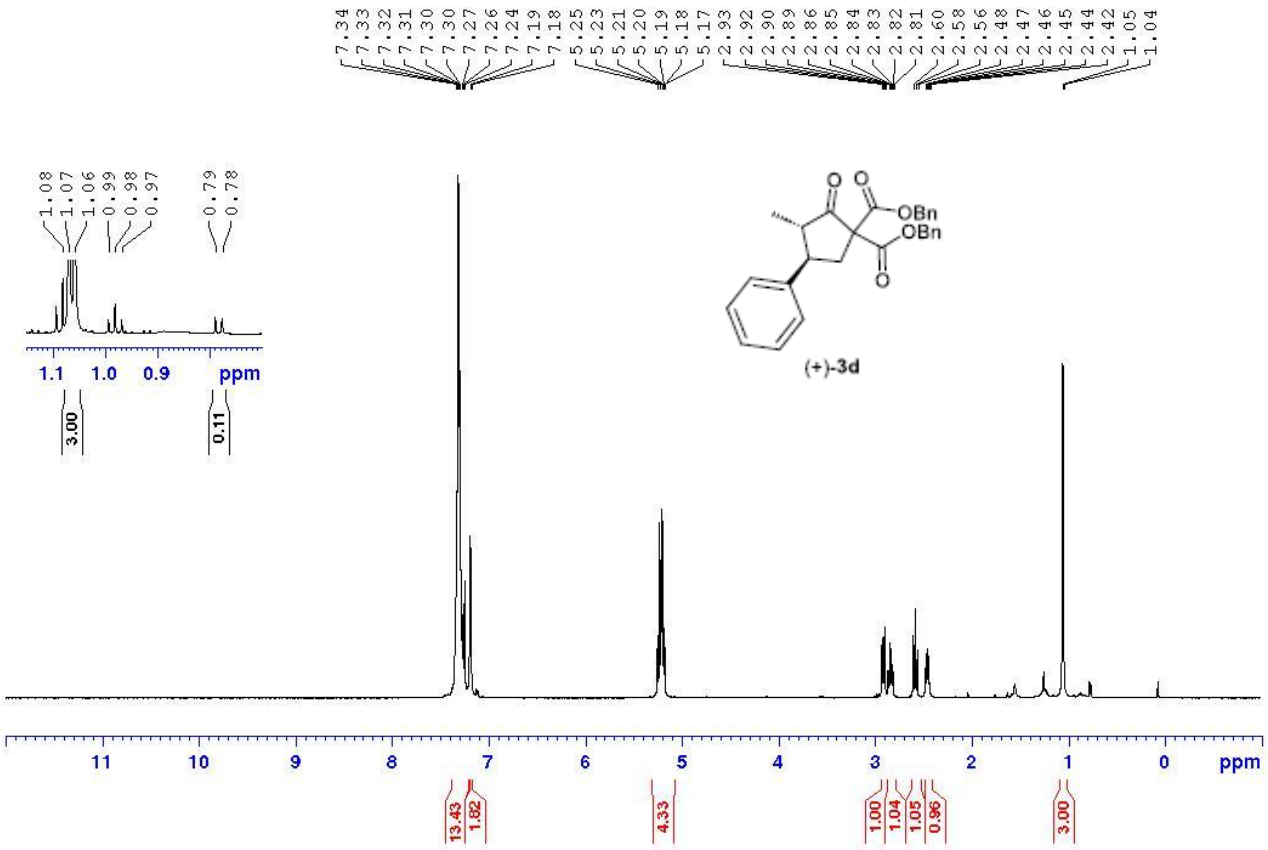
^{1}H NMR of (–)-**3d:**


7.36 7.36 7.35 7.34 7.33 7.33 7.33 7.32 7.32 7.32 7.31 7.31 7.30 7.30 7.29 7.29 7.29 7.28 7.28 7.27 7.27 7.26 7.26 7.21 7.21 7.19 5.26 5.24 5.23 5.22 5.21 5.20 5.19 2.94 2.93 2.92 2.91 2.87 2.86 2.85 2.84 2.83 2.82 2.61 2.59 2.59 2.57 2.49 2.48 2.47 2.46 2.45 2.44 1.07 1.06
1.07 1.06 1.00 0.98 0.97 0.93 0.92 0.89 0.89 0.88 0.88
1.1 1.0 0.9 ppm
3.00 0.12
OBn OBn
(-)-3d
11 10 9 8 7 6 5 4 3 2 1 0 ppm
11.90 1.02 1.99 4.29 1.03 1.03 1.01 1.02 3.00

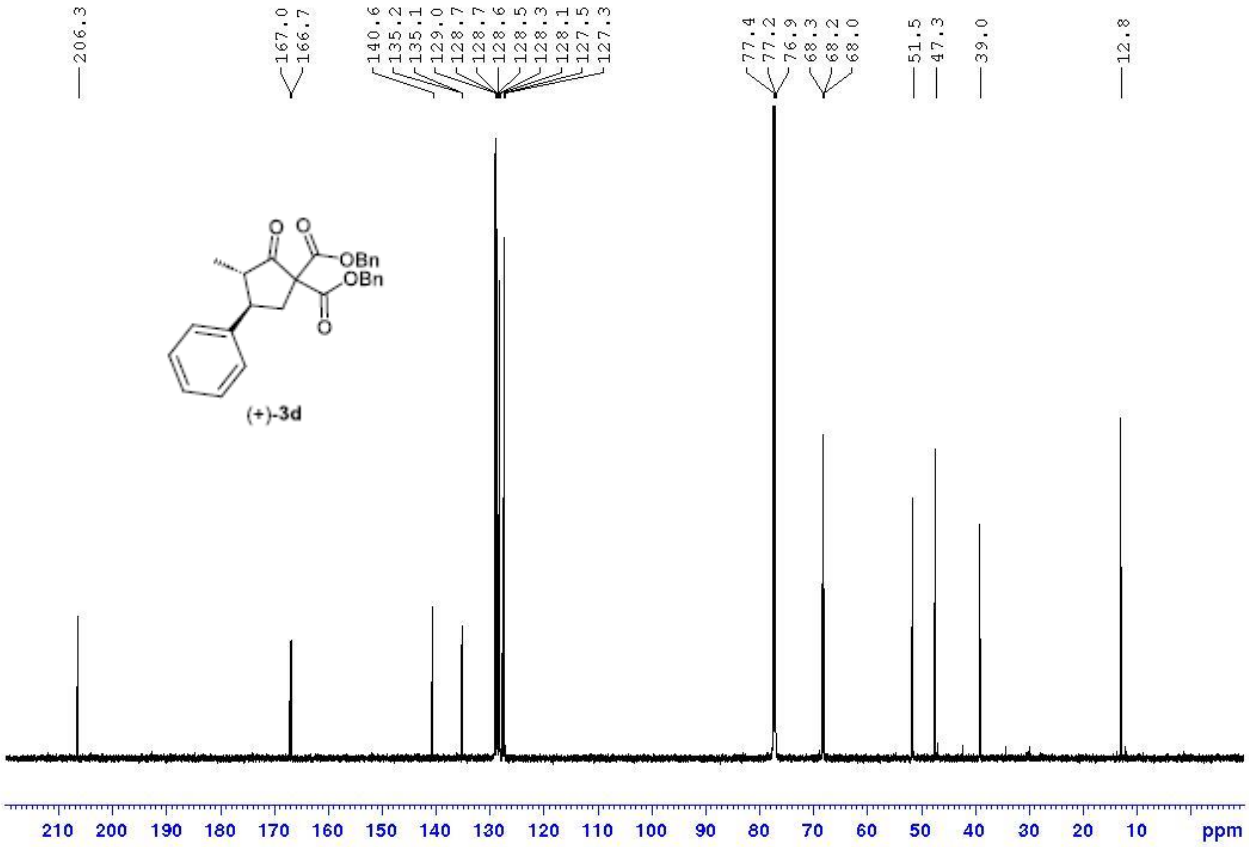
^{13}C NMR of (–)-**3d:**


206.4
167.0
166.7
140.6
135.2
135.1
129.0
128.7
128.7
128.6
128.5
128.3
128.1
127.5
127.3
77.4
77.2
76.9
68.2
68.0
51.5
47.3
39.0
12.8
OBn
OBn
(-)-3d
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

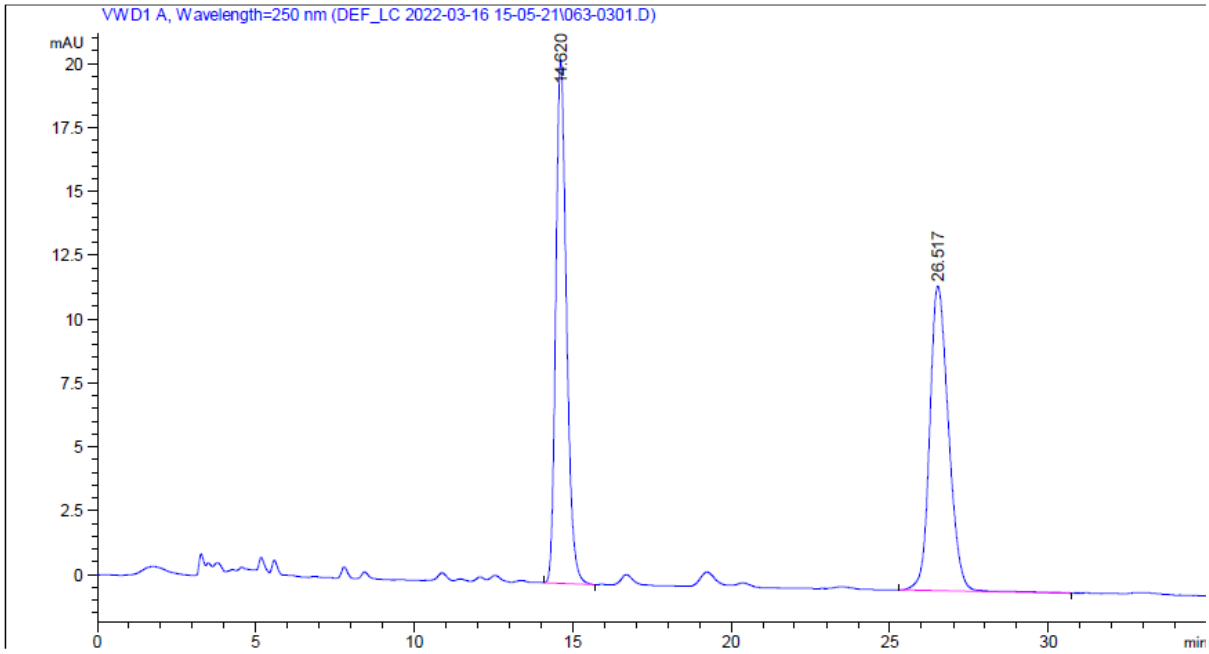
^{1}H NMR of (+)-**3d:**


7.34
7.33
7.32
7.31
7.30
7.30
7.27
7.26
7.24
7.19
7.18
5.25
5.23
5.21
5.20
5.19
5.18
5.17
2.93
2.92
2.90
2.89
2.86
2.85
2.84
2.83
2.82
2.81
2.60
2.58
2.56
2.48
2.47
2.46
2.45
2.44
2.42
1.05
1.04
1.08
1.07
1.06
0.99
0.98
0.97
0.79
0.78
1.1
1.0
0.9
ppm
3.00
0.11
OBn
OBn
(+)-3d
11
10
9
8
7
6
5
4
3
2
1
0
ppm
13.43
1.82
4.33
1.00
1.04
1.05
0.96
3.00

^{13}C NMR of (+)-**3d:**


206.3
167.0
166.7
140.6
135.2
135.1
129.0
128.7
128.7
128.6
128.5
128.3
128.1
127.5
127.3
77.4
77.2
76.9
68.3
68.2
68.0
51.5
47.3
39.0
12.8
OBn
OBn
(+)-3d
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

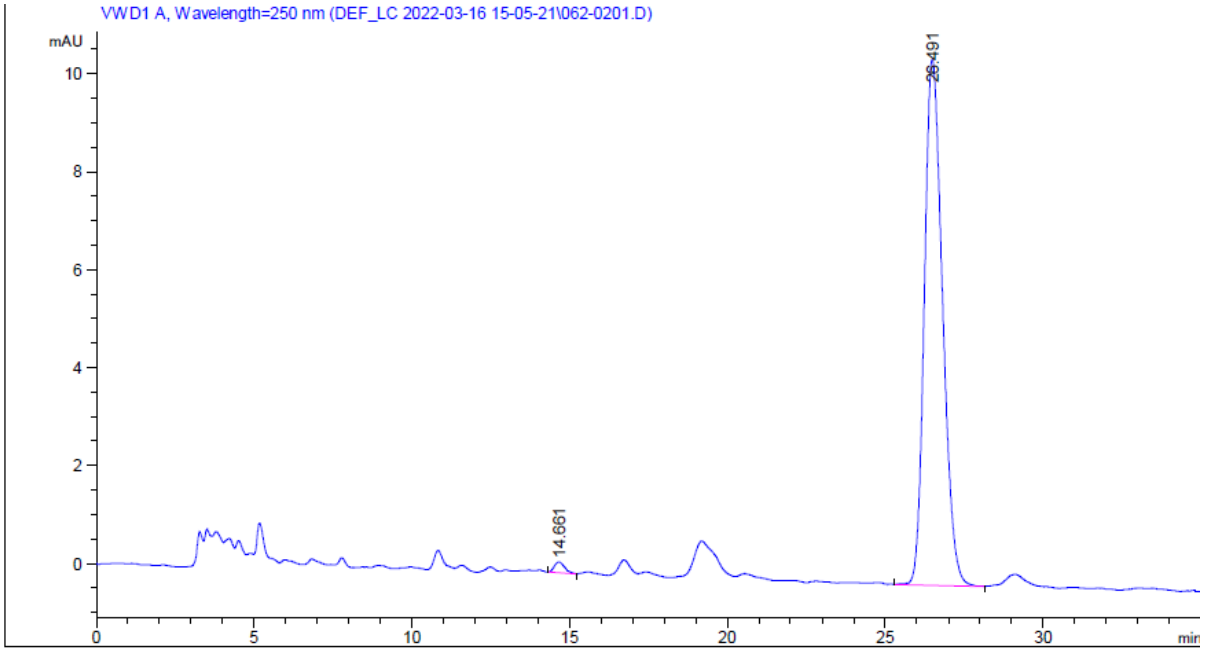
HPLC of (±)-**3d:**



Signal 1: VWD1 A, Wavelength=250 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.620	BB	0.3606	478.25949	20.42881	49.3090
2	26.517	BB	0.6327	491.66290	11.92206	50.6910
Totals :				969.92239	32.35087	

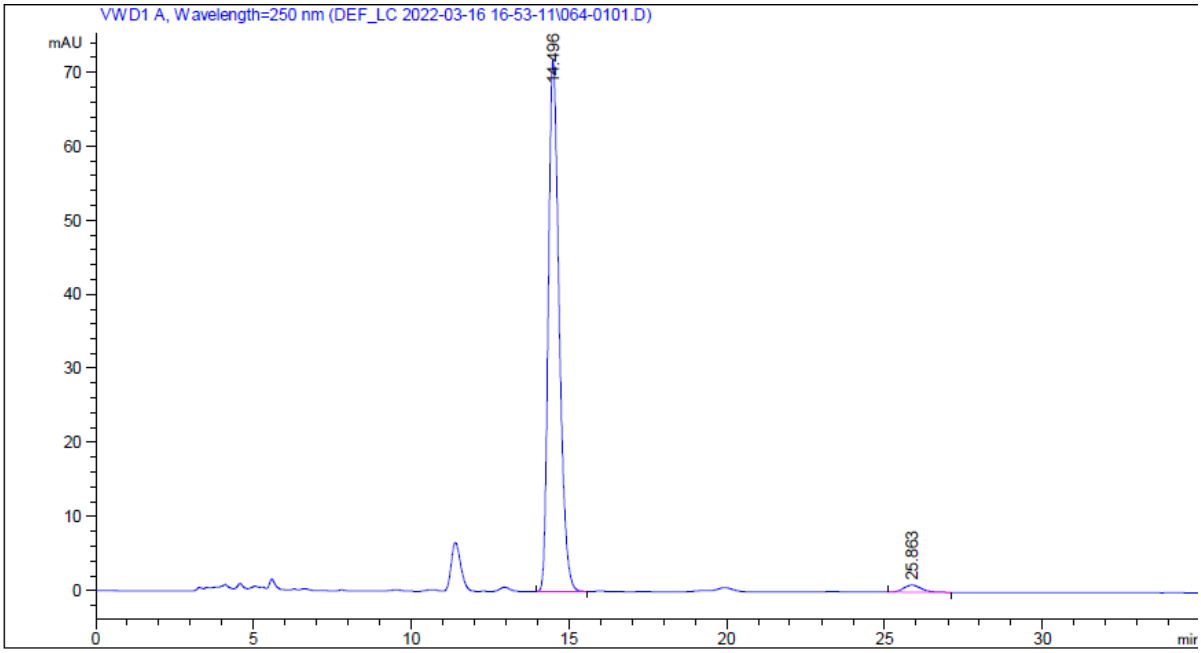
HPLC of (–)-**3d:**



Signal 1: VWD1 A, Wavelength=250 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.661	BB	0.3508	4.99249	2.21160e-1	1.1506
2	26.491	BB	0.6236	428.90231	10.73315	98.8494
Totals :				433.89480	10.95431	

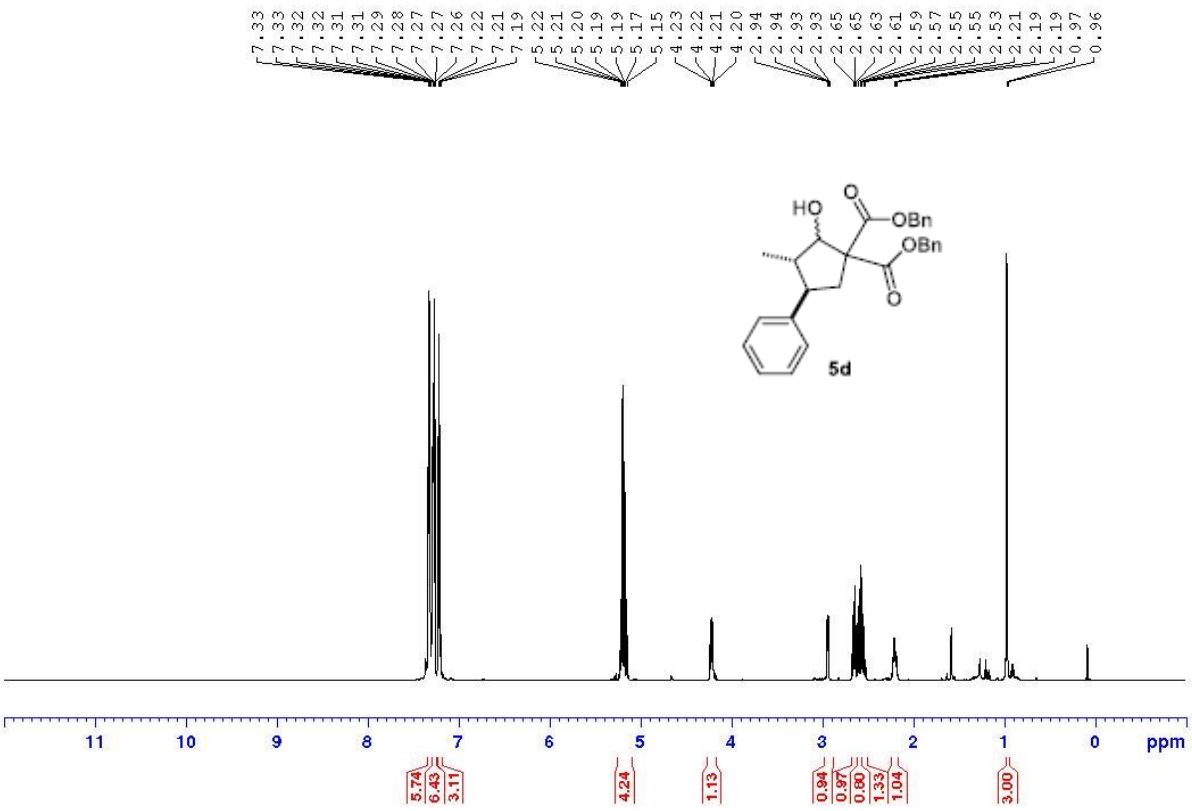
HPLC of (+)-**3d:**



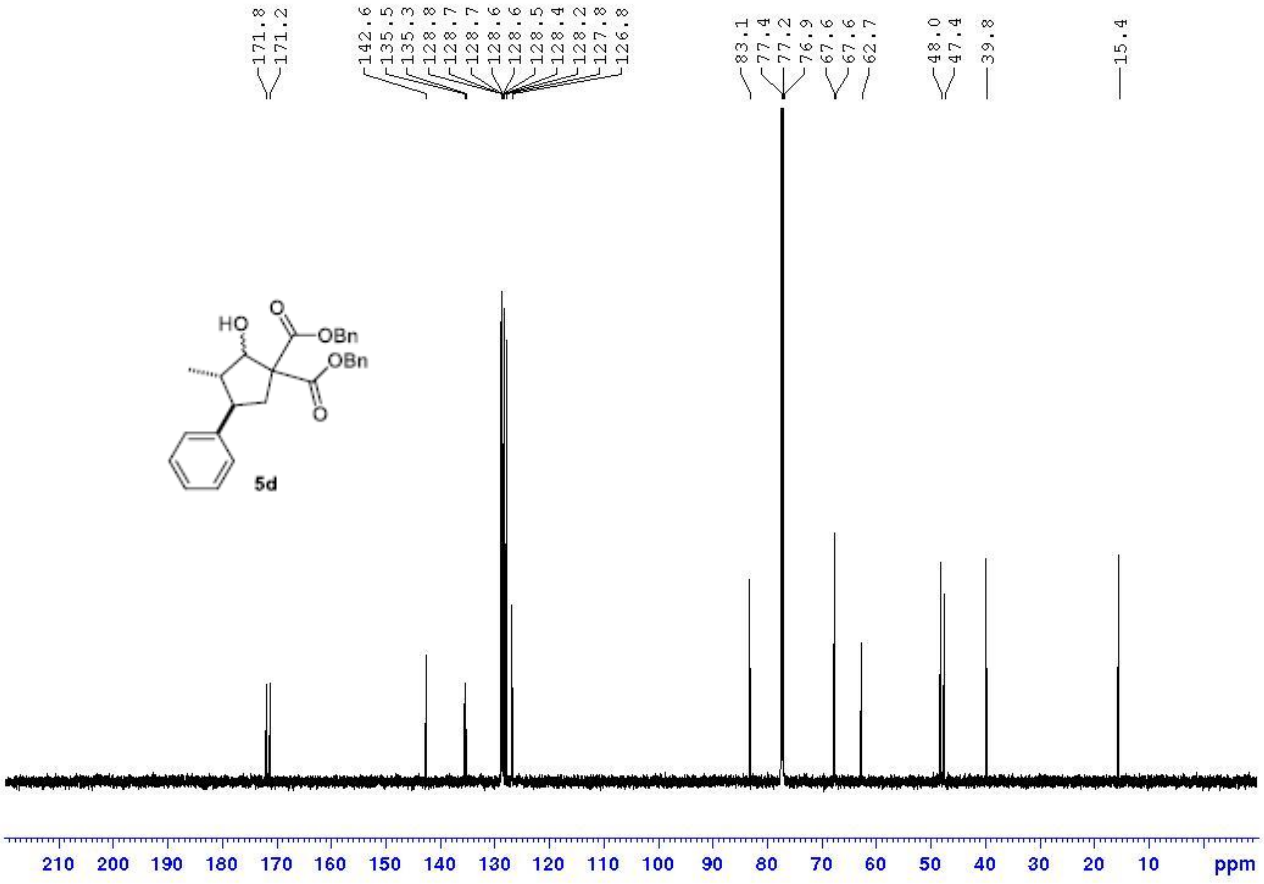
Signal 1: VWD1 A, Wavelength=250 nm

Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	14.496	BB	0.3711	1707.18237	71.68500	97.8788
2	25.863	BB	0.5953	36.99744	9.60217e-1	2.1212
Totals :				1744.17982	72.64521	

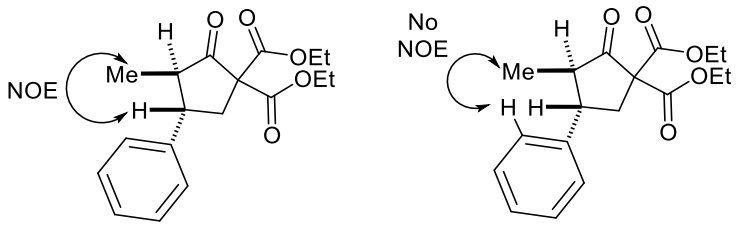
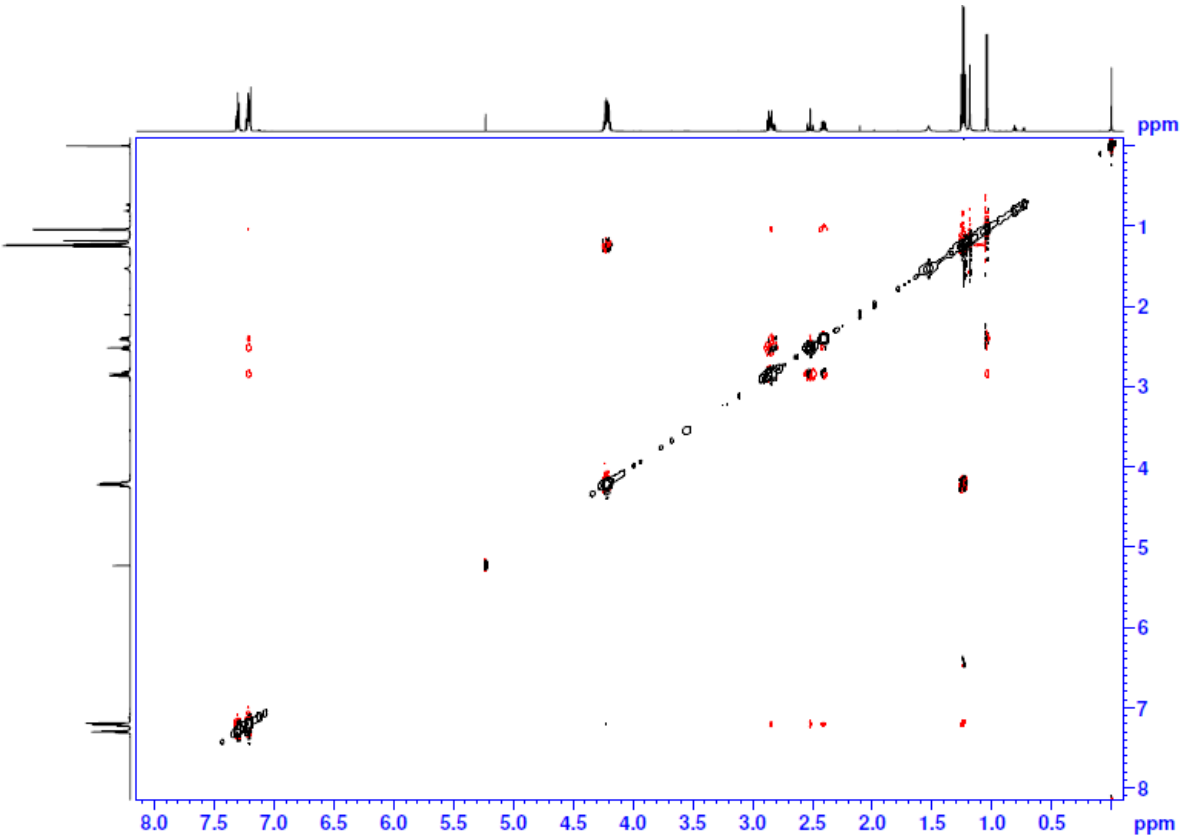
^{1}H NMR of **5d:**


7.33
7.33
7.32
7.32
7.31
7.31
7.29
7.28
7.27
7.27
7.26
7.22
7.21
7.19
5.22
5.21
5.20
5.19
5.19
5.17
5.15
4.23
4.22
4.21
4.20
2.94
2.94
2.93
2.93
2.65
2.65
2.63
2.61
2.59
2.57
2.55
2.55
2.53
2.21
2.19
2.19
0.97
0.96
HO
O
OBn
OBn
O
5d
11
10
9
8
7
6
5
4
3
2
1
0
ppm
5.74
6.43
3.11
4.24
1.13
0.94
0.97
0.80
1.33
1.04
3.00

^{13}C NMR of **5d:**


171.8
171.2
142.6
135.5
135.3
128.8
128.7
128.7
128.6
128.6
128.5
128.4
128.2
127.8
126.8
83.1
77.4
77.2
76.9
67.6
67.6
62.7
48.0
47.4
39.8
15.4
HO
OBn
OBn
5d
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

2D NOESY for 3c:



3c