Asymmetric Rhodium(I)-Catalyzed C-C Activations with Zwitterionic *Bis*-Phospholane Ligands

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Supplementary Information

Table of Content:

General procedure for substrates synthesis:

The required cyclobutanones were prepared according to the reported procedures:

a) L. R. Krepski, A. Hassner *J. Org. Chem.* **1978**, *43*, 2879–2882;

b) B. D. Johnston, E. Czyzewska, A. C. Oehlschlager *J. Org. Chem.*, **1987**, *52*, 3693–3697;

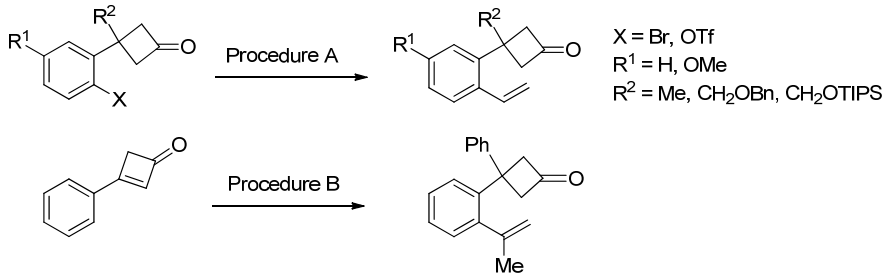
c) W. Cao, I. Erden, R. H. Grow, J. R. Keeffe, J. Song, M. B. Trudell, T. L. Wadsworth, F. Xu, J. Zheng *Can. J. Chem.* **1999**, *77*, 1009;

d) P. P. Shao, F. Ye *Tetrahedron Lett.* **2008**, *49*, 3554–3557;

e) P. E. Pigou, C. H. Schiesser *J. Org. Chem.*, **1988**, *53*, 3841–3843;

f) R. R. Galucci, R. Going *J. Org. Chem.* **1981**, *46*, 2532;

g) K. Sugimoto, R. Hayashi, H. Nemoto, N. Toyooka, Y. Matsuya, *Org. Lett.* **2012**, *14*, 3510-3513.

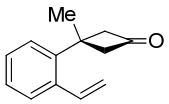


Procedure A (Suzuki coupling):

A dried microwave vial was charged with the respective cyclobutanone (1 equiv.), $Pd(OAc)_2$ (10 mol%), dppf (12 mol%) and potassium vinyltrifluoroborate (3 equiv.). The system was sealed, evacuated and back filled with nitrogen. Dry and degassed mixture of dioxane / n-PrOH (ratio 2:1, 0.3M) and distilled triethylamine (3 equiv.) were added and the mixture was stirred at 110 °C for 6 h. The reaction mixture was filtered through a short pad of celite/silica and poured in sat. aq. NH_4Cl. Aqueous layers were extracted with EtOAc (x3). Combined organic layers were washed with water and brine, dried over $MgSO_4$, filtered and evaporated *in vacuo.* The residue was purified on a silica gel column eluting with the appropriate mixture of pentane/ethyl acetate to afford the desired product in 74–96% yield.

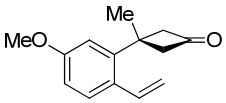
Procedure B (1,4 – addition):

In a flame-dried Schlenk tube containing CuI (66.7 mg, 0.35 mmol) was added a freshly prepared (2-(prop-1-en-2-yl)phenyl)magnesium bromide (0.7 mL, 1.40 mmol, 2M solution in Et_2O) at 0°C and the mixture was stirred for 10 min, then warmed up to room temperature and stirred for additional 10 min. A solution of 3-phenylcyclobut-2-enone (101 mg, 0.7 mmol) in Et_2O (0.7 mL) was added dropwise at 0°C and the reaction mixture was gradually warmed up to room temperature over 3 h. The reaction mixture was carefully quenched at 0°C with aq. NH_4Cl, extracted with Et_2O (x3). The combined organic layers were washed with water and brine, dried over $MgSO_4$, and evaporated *in vacuo.* The residue was purified on a silica gel column eluting with a mixture of pentane/ethyl acetate to afford the desired product (75 mg, 0.29 mmol) in 41% yield.



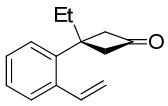
3-methyl-3-(2-vinylphenyl)cyclobutanone (11a)

^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.53–7.51 (m, 1H), 7.31–7.26 (m, 2H), 7.22–7.19 (m, 1H), 6.86 (dd, $^{3}J_{H\text{-}H}$ = 17.3 Hz, $^{3}J_{H\text{-}H}$ = 10.9 Hz, 1H), 5.65 (d, $^{3}J_{H\text{-}H}$ = 17.2 Hz, 1H), 5.32 (d, $^{3}J_{H\text{-}H}$ = 10.9 Hz, 1H), 3.59–3.48 (m, 2H), 3.17–3.07 (m, 2H), 1.60 (s, 3H) ppm; **^{13}C{^{1}H} NMR** (101 MHz, $CDCl_3$) δ = 206.9, 144.7, 136.1, 135.3, 128.0, 127.1, 127.0, 126.6, 116.1, 59.7, 34.4, 29.5 ppm; **IR (ATR)** $\tilde{\nu}$ = 3061, 3025, 2957, 2921, 2865, 1781, 1480, 1444, 1378, 1271, 1183, 1144, 1080, 1057, 993, 915, 757, 650, 551, 477, 427 cm^{-1}; **HRMS (ESI)** *m/z* calc'd. for $[C_{13}H_{15}O]^{+}$: 187.1117, found: 187.1117; R_f = 0.53 (Pentane/Ethyl Acetate 9:1).



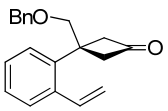
3-(5-methoxy-2-vinylphenyl)-3-methylcyclobutanone (11b)

^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.47 (d, $^{3}J_{H\text{-}H}$ = 8.7 Hz, 1H), 6.85–6.72 (m, 3H), 5.54 (dd, $^{3}J_{H\text{-}H}$ = 17.2 Hz, $^{2}J_{H\text{-}H}$ = 1.4 Hz, 1H), 5.21 (dd, $^{3}J_{H\text{-}H}$ = 10.9 Hz, $^{2}J_{H\text{-}H}$ = 1.4 Hz, 1H), 3.83 (s, 3H), 3.56–3.47 (m, 2H), 3.15–3.05 (m, 2H), 1.59 (s, 3H) ppm; **^{13}C{^{1}H} NMR** (101 MHz, $CDCl_3$) δ = 206.8, 159.3, 146.2, 134.6, 128.8, 128.4, 114.2, 113.0, 111.6, 9.6, 55.3, 34.5, 29.4 ppm; **IR (ATR)** $\tilde{\nu}$ = 3085, 2957, 2921, 2865, 2836, 1779, 1603, 1566, 1485, 1466, 1379, 1325, 1294, 1230, 1185, 1144, 1064, 1046, 1025, 992, 908, 868, 816, 730, 544, 464, 439 cm^{-1}; **HRMS (ESI)** *m/z* calc'd. for $[C_{14}H_{17}O_2]^{+}$: 217.1223, found: 217.1227; R_f = 0.37 (Pentane/Ethyl Acetate 9:1).



3-ethyl-3-(2-vinylphenyl)cyclobutanone (11c)

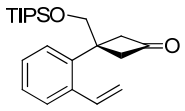
^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.57 – 7.48 (m, 1H), 7.29–7.23 (m, 2H), 7.15–7.04 (m, 1H), 6.86 (dd, $^{3}J_{H\text{-}H}$ = 17.3 Hz, $^{3}J_{H\text{-}H}$ = 10.9 Hz, 1H), 5.62 (dd, $^{3}J_{H\text{-}H}$ = 17.3 Hz, $^{2}J_{H\text{-}H}$ = 1.4 Hz, 1H), 5.29 (dd, $^{3}J_{H\text{-}H}$ = 10.9 Hz, $^{2}J_{H\text{-}H}$ = 1.4 Hz, 1H), 3.54–3.40 (m, 2H), 3.20–3.09 (m, 2H), 1.91 (q, $^{2}J_{H\text{-}H}$ = 7.3 Hz, 2H), 0.76 (t, $^{2}J_{H\text{-}H}$ = 7.3 Hz, 3H) ppm; **^{13}C{^{1}H} NMR** (101 MHz, $CDCl_3$) δ = 207.1, 142.2, 136.4, 135.4, 128.4, 127.3, 127.2, 127.0, 115.9, 57.9, 38.5, 33.6, 9.8 ppm; **IR (ATR)** $\tilde{\nu}$ = 3061, 2964, 2921, 2853, 1779, 1480, 1460, 1381, 1314, 1181, 1142, 1117, 1080, 993, 772, 755, 431 cm^{-1}; **HRMS (ESI)** *m/z* calc'd. for $[C_{14}H_{17}O]^{+}$: 201.1274, found: 201.1274; R_f = 0.56 (Pentane/Ethyl Acetate 9:1).



3-((benzyloxy)methyl)-3-(2-vinylphenyl)cyclobutanone (11d)

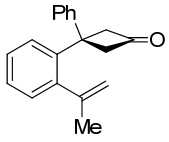
^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.57–7.45 (m, 1H), 7.36–7.24 (m, 5H), 7.25–7.15 (m, 3H), 6.82 (dd, $^{3}J_{H\text{-}H}$ = 17.3 Hz, $^{3}J_{H\text{-}H}$ = 10.9 Hz, 1H), 5.60 (dd, $^{3}J_{H\text{-}H}$ = 17.3 Hz, $^{2}J_{H\text{-}H}$ = 1.4 Hz, 1H), 5.27

(dd, $^{3}J_{H\text{-}H}$ = 10.9 Hz, $^{2}J_{H\text{-}H}$ = 1.4 Hz, 1H), 4.51 (s, 2H), 3.66 (d, $^{2}J_{H\text{-}H}$ = 1.4 Hz, 2H), 3.42 (d, $^{2}J_{H\text{-}H}$ = 1.5 Hz, 4H) ppm; **^{13}C{^{1}H} NMR** (101 MHz, $CDCl_3$) δ = 205.8, 140.7, 137.9, 137.0, 135.4, 128.3, 128.3, 127.8, 127.6, 127.5, 127.4, 127.3, 127.1, 116.2, 76.2, 73.2, 55.8, 38.8 ppm; **IR (ATR)** $\tilde{\nu}$ = 3062, 3028, 2921, 2854, 1782, 1480, 1453, 1374, 1241, 1205, 1090, 1027, 989, 908, 758, 729, 697, 649, 477, 431 cm^{-1}; **HRMS (ESI)** *m/z* calc'd. for $[C_{20}H_{21}O_2]^+$: 293.1536, found: 293.1534; R_f = 0.39 (Pentane/Ethyl Acetate 9:1).



3-(((triisopropylsilyl)oxy)methyl)-3-(2-vinylphenyl)cyclobutanone (11e)

^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.56–7.46 (m, 1H), 7.31–7.27 (m, 2H), 7.24–7.15 (m, 1H), 6.85 (dd, $^{3}J_{H\text{-}H}$ = 17.3 Hz, $^{3}J_{H\text{-}H}$ = 10.9 Hz, 1H), 5.62 (dd, $^{3}J_{H\text{-}H}$ = 17.3 Hz, $^{2}J_{H\text{-}H}$ = 1.5 Hz, 1H), 5.31 (dd, $^{3}J_{H\text{-}H}$ = 10.9 Hz, $^{2}J_{H\text{-}H}$ = 1.5 Hz, 1H), 3.90 (d, $^{2}J_{H\text{-}H}$ = 1.8 Hz, 2H), 3.49 – 3.35 (m, 4H), 1.14–0.93 (m, 21H) ppm; **^{13}C{^{1}H} NMR** (101 MHz, $CDCl_3$) δ = 205.8, 140.6, 137.1, 135.5, 128.4, 127.7, 127.5, 127.1, 116.3, 69.9, 55.1, 40.3, 17.9, 11.9 ppm; **IR (ATR)** $\tilde{\nu}$ = 2941, 2891, 2865, 1787, 1462, 1383, 1373, 1110, 1090, 1068, 1013, 994, 915, 881, 801, 756, 682, 660, 475 cm^{-1}; **HRMS (ESI)** *m/z* calc'd. for $[C_{22}H_{35}O_2Si]^+$: 359.2401, found: 359.2395; R_f = 0.67 (Pentane/Ethyl Acetate 9:1).

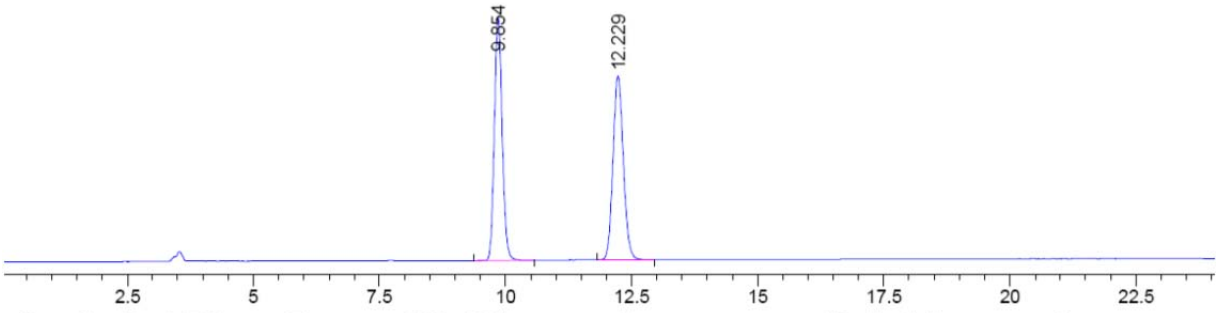


3-phenyl-3-(2-(prop-1-en-2-yl)phenyl)cyclobutanone (11f)

^{1}H NMR (400 MHz, $CDCl_3$) δ = 7.45–7.11 (m, 9H), 4.95 (dq, $^{3}J_{H\text{-}H}$ *= 2.0 Hz,* $^{4}J_{H\text{-}H}$ = 1.6 Hz, 1H), 4.45 (dq, $^{2}J_{H\text{-}H}$ = 2.0 Hz, $^{4}J_{H\text{-}H}$ = 1.0 Hz, 1H), 3.87–3.78 (m, 2H), 3.62–3.53 (m, 2H), 1.79 (dd, $^{4}J_{H\text{-}H}$ = 1.6 Hz, $^{4}J_{H\text{-}H}$ = 1.0 Hz, 3H) ppm; **^{13}C{^{1}H} NMR** (101 MHz, $CDCl_3$) δ = 207.0, 148.5, 145.0, 143.3, 142.8, 129.9, 129.4, 128.5, 127.0, 126.8, 126.2, 125.8, 116.6, 61.7, 41.8, 25.3 ppm; **IR (ATR)** $\tilde{\nu}$ = 3058, 3020, 2973, 2915, 1780, 1639, 1599, 1495, 1446, 1375, 1121, 1082, 1023, 905, 758, 700, 658, 560, 549, 483 cm^{-1}; **HRMS (ESI)** *m/z* calc'd. for $[C_{19}H_{19}O]^+$: 263.1430, found: 263.1428; R_f = 0.56 (Pentane/Ethyl Acetate 9:1).

HPLC-Traces for ketones 14:

(5*S*,9*R*)-5-methyl-8,9-dihydro-5*H*-5,9-methanobenzo[7]annulen-7(6*H*)-one (14a)



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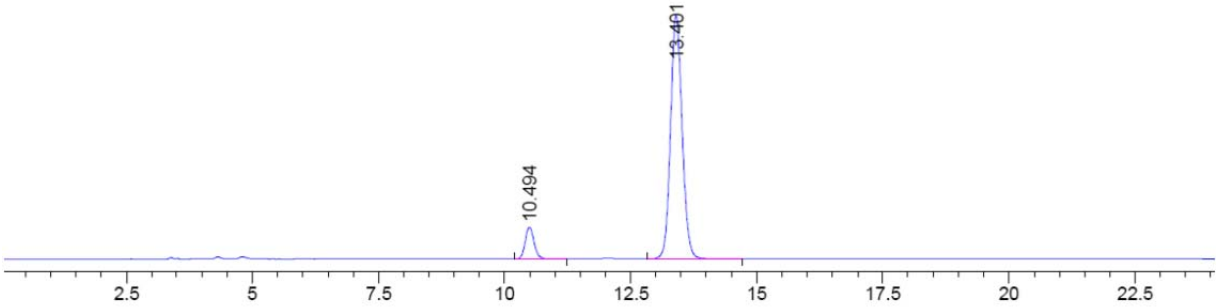
1	9.854	BB	0.1665	439.54361	40.53133	50.1100
2	12.229	BB	0.2227	437.61340	30.61999	49.8900



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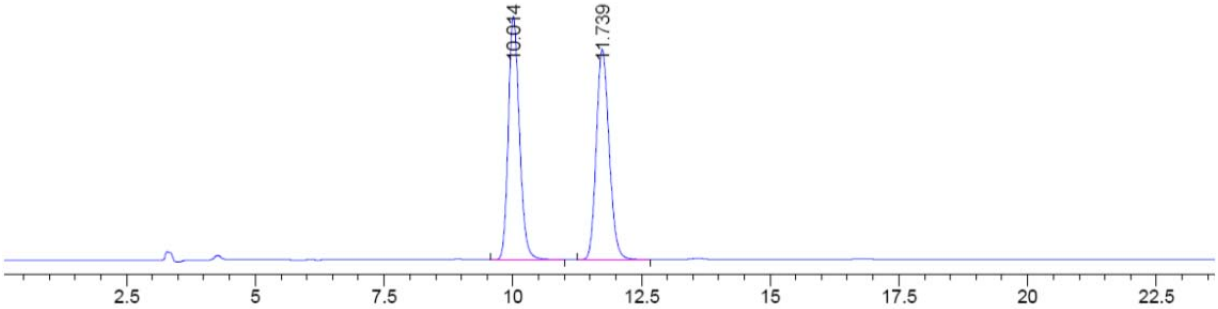
1	10.494	BB	0.1881	71.67528	5.96901	8.9185
2	13.401	BB	0.2467	731.99146	45.74722	91.0815

(5*S*,9*R*)-2-methoxy-9-methyl-8,9-dihydro-5*H*-5,9-methanobenzo[7]annulen-7(6*H*)-one (14b)



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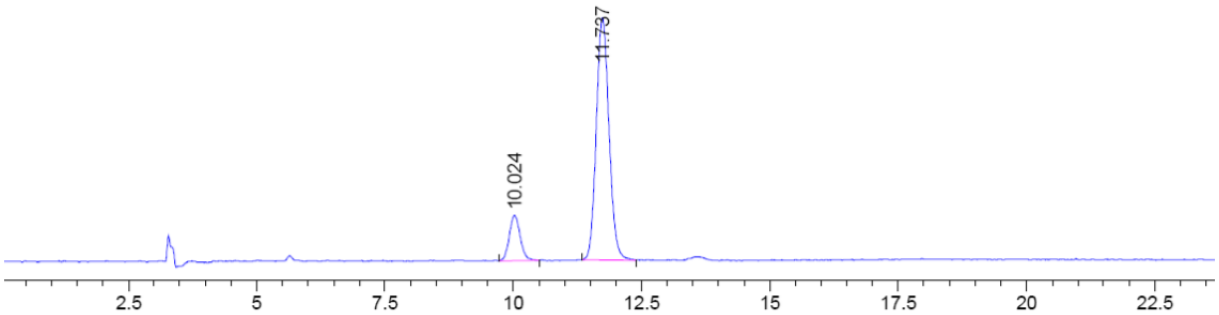
2	11.739	BB	0.2680	2122.33618	122.57774	49.9796



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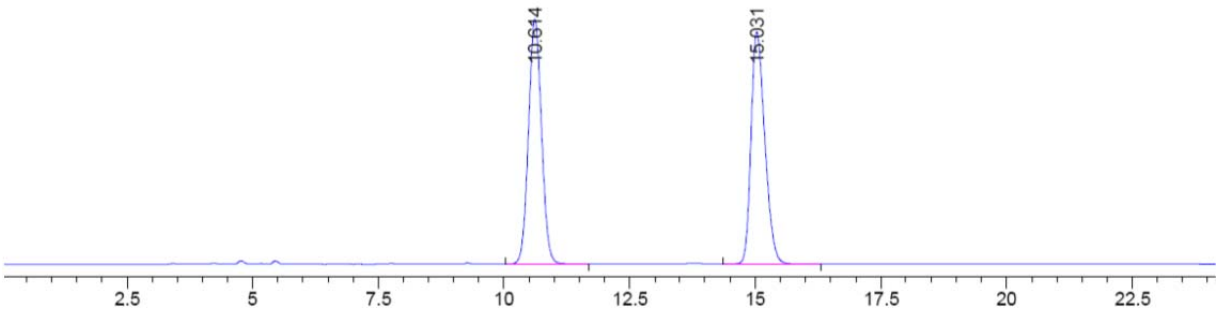
1	10.024	BB	0.2258	38.96115	2.61597	13.9343
2	11.737	BB	0.2646	240.64484	13.99832	86.0657

(5*S*,9*R*)-5-ethyl-8,9-dihydro-5*H*-5,9-methanobenzo[7]annulen-7(6*H*)-one (14c)



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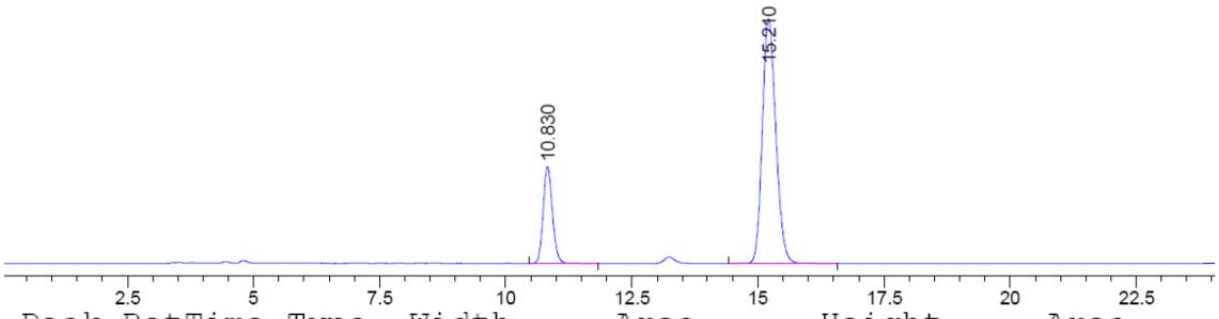

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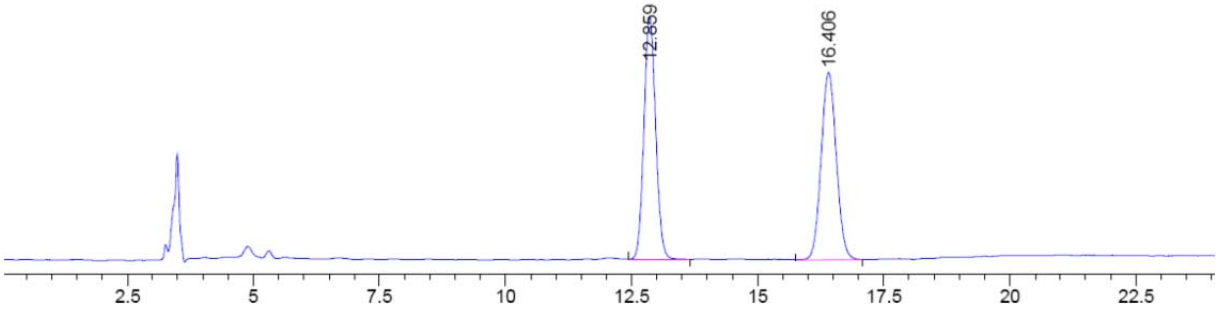
2	15.210	BB	0.2891	1373.49219	73.80065	78.5554

(5*R*,9*R*)-5-((benzyloxy)methyl)-8,9-dihydro-5*H*-5,9-methanobenzo[7]annulen-7(6*H*)-one (14d)



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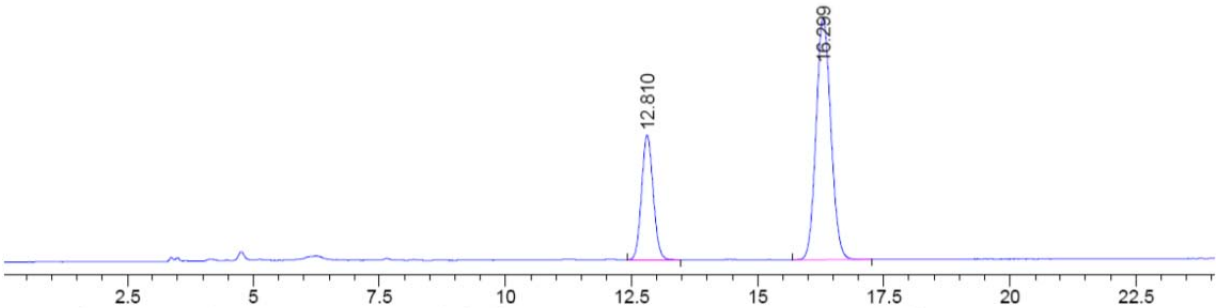

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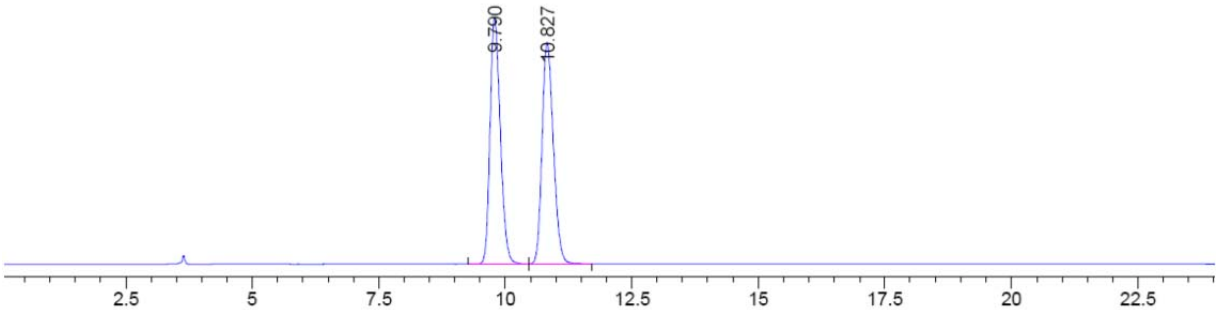
1	12.810	BB	0.2470	325.54828	20.31510	28.8265
2	16.299	BB	0.3181	803.79028	39.38016	71.1735

(5*R*,9*R*)-5-(((triisopropylsilyl)oxy)methyl)-8,9-dihydro-5*H*-5,9-methanobenzo[7]annulen-7(6*H*)-one (14e)



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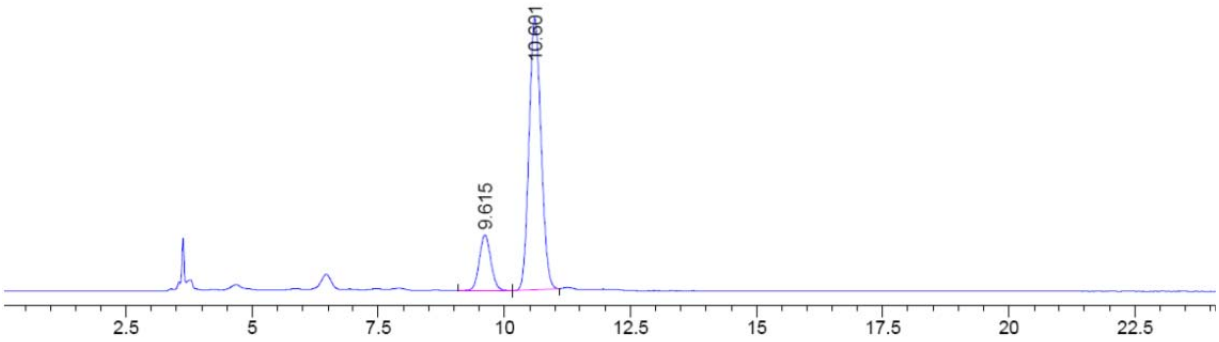
1	9.790	BB	0.2164	1770.25574	125.59419	50.2430
2	10.827	BB	0.2409	1753.13440	113.06232	49.7570



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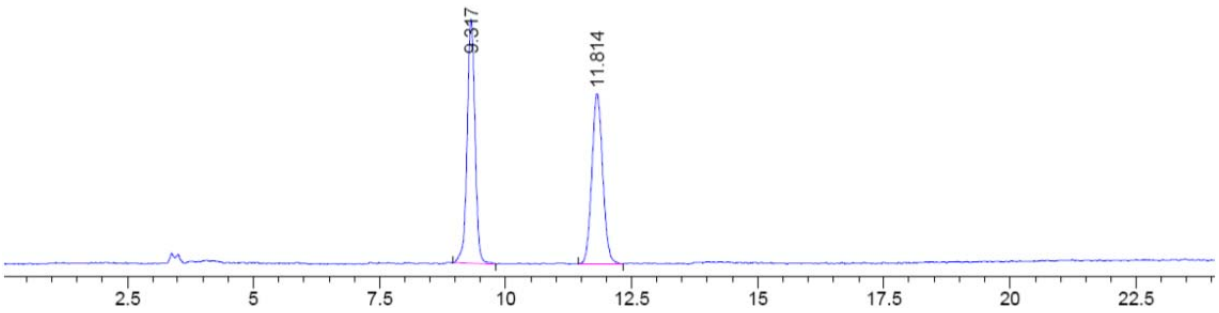
2	10.601	BB	0.2593	988.31720	59.64160	83.3177

(5*S*,9*S*)-5-methyl-9-phenyl-8,9-dihydro-5*H*-5,9-methanobenzo[7]annulen-7(6*H*)-one (14f)



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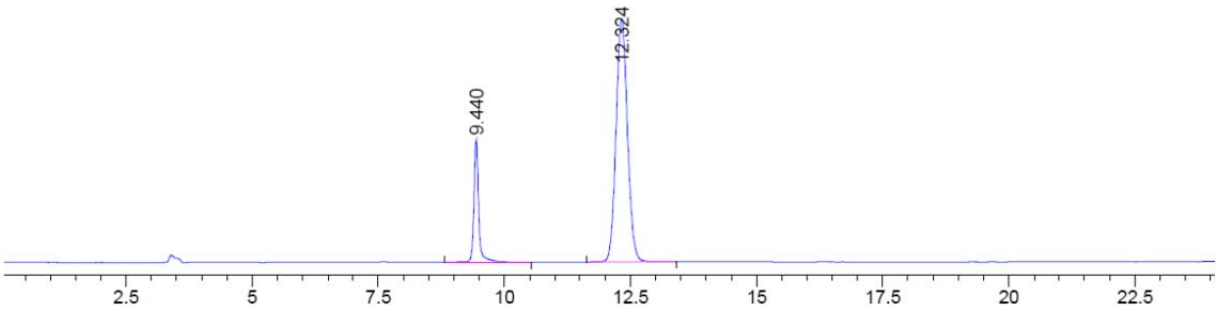

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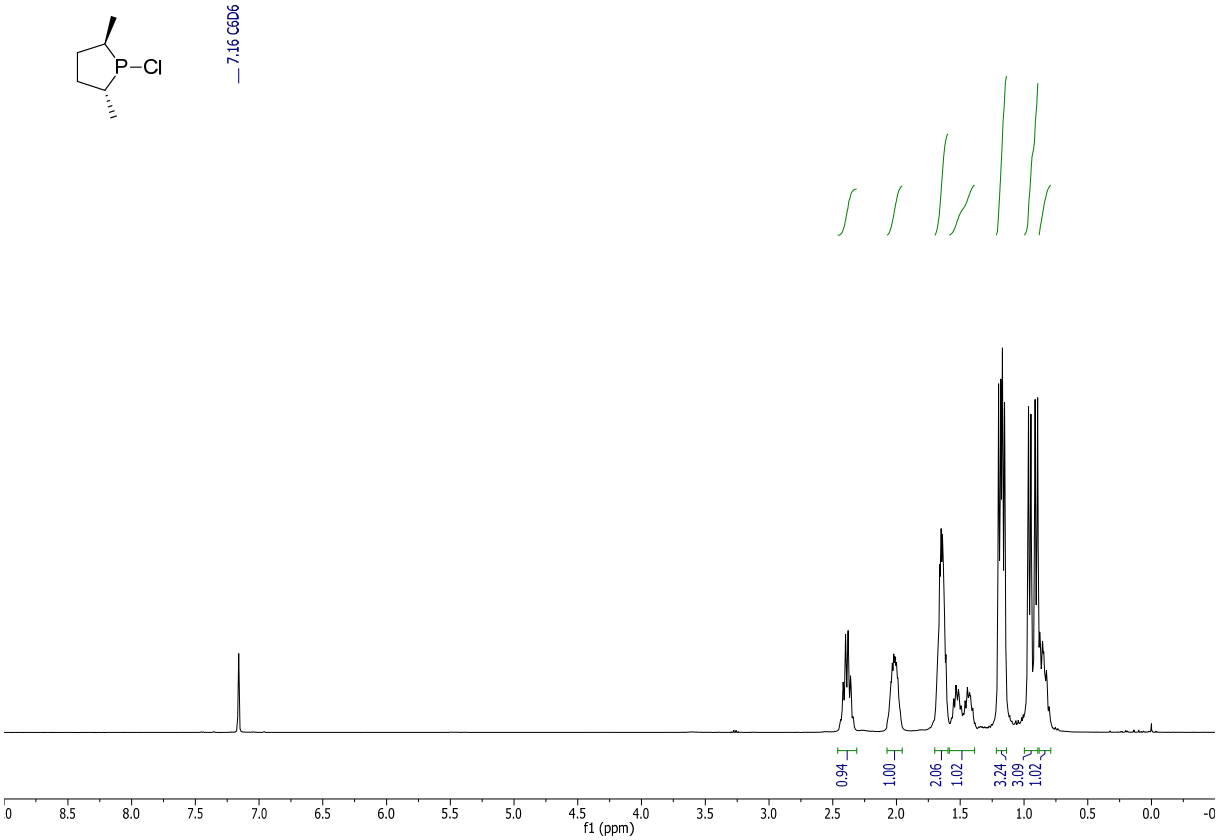
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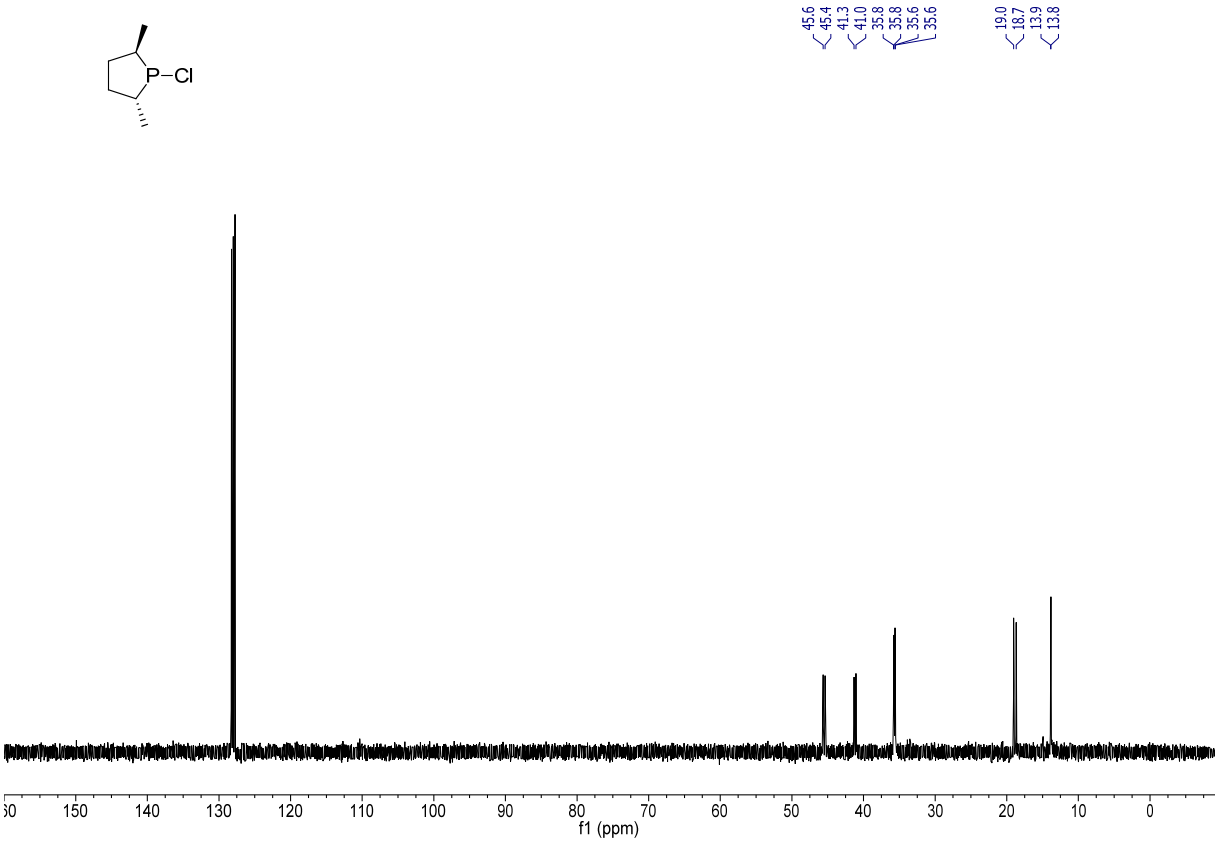
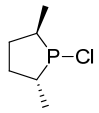
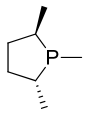
0.94
1.00
2.06
1.02
3.24
3.09
1.02
f1 (ppm)

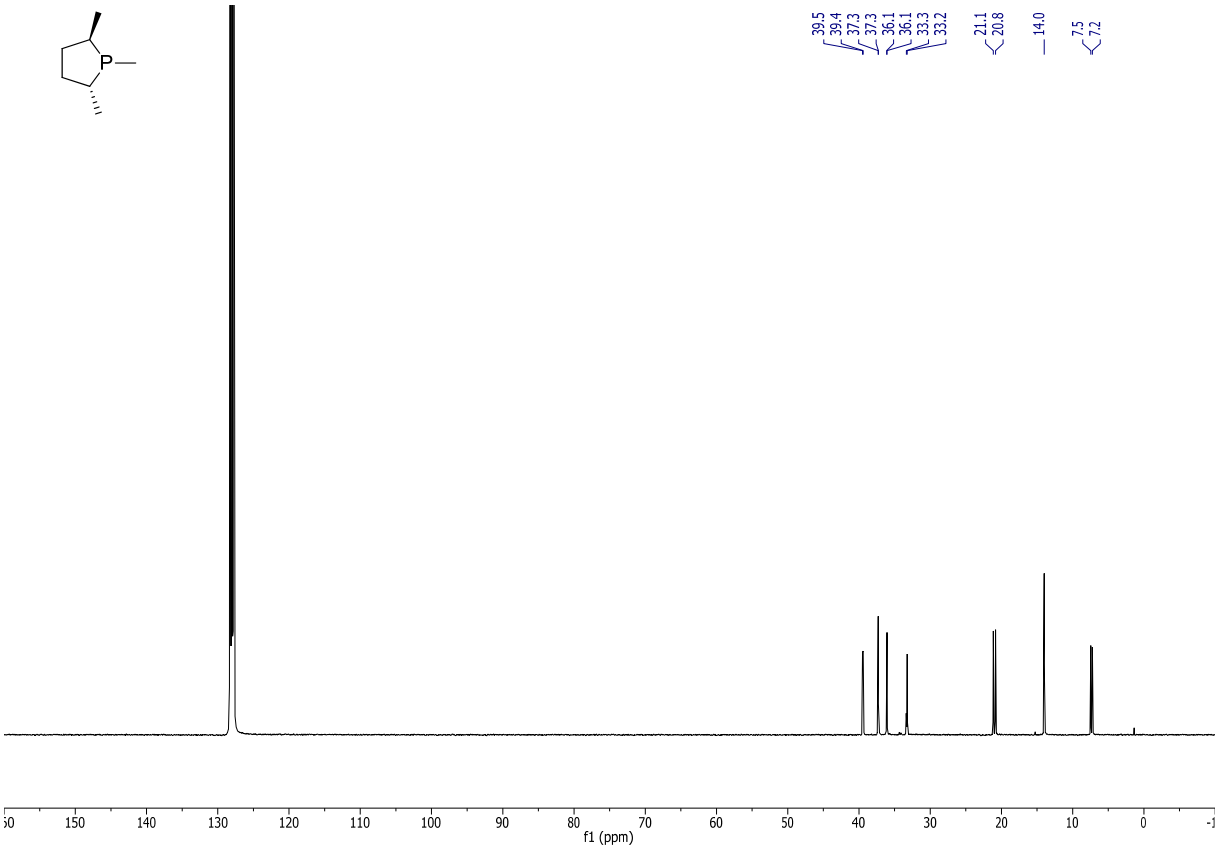

P–Cl
45.6
45.4
41.3
41.0
35.8
35.8
35.6
35.6
19.0
18.7
13.9
13.8
f1 (ppm)

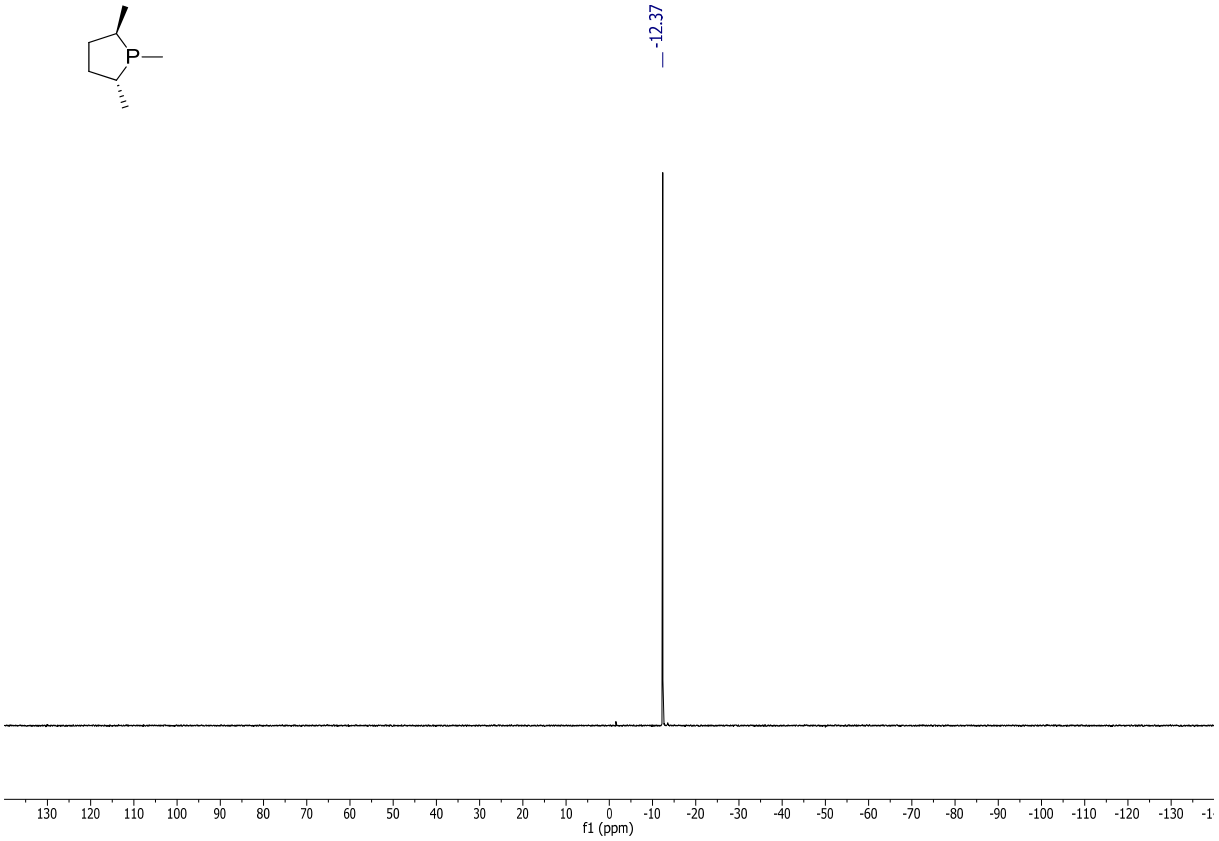

P–Cl

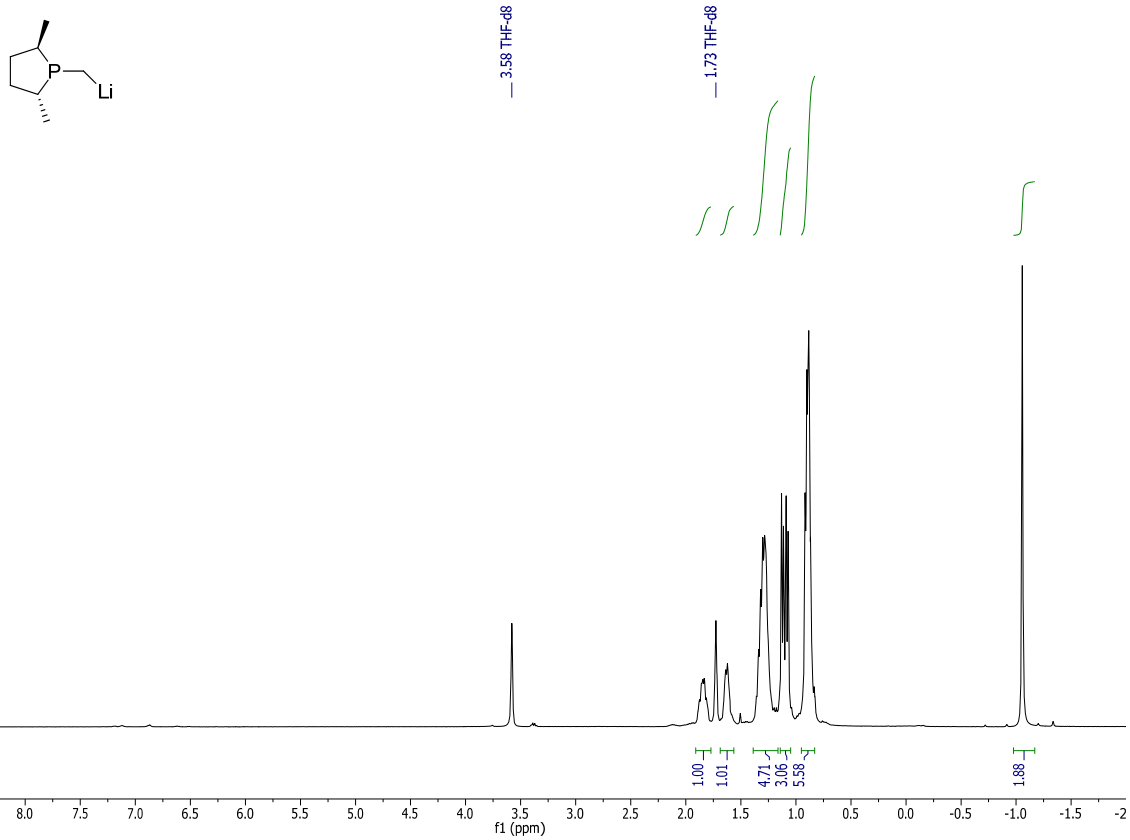
142.24
195 190 185 180 175 170 165 160 155 150 145 140 135 130 125 120 115 110 105 100 95 90 85
f1 (ppm)

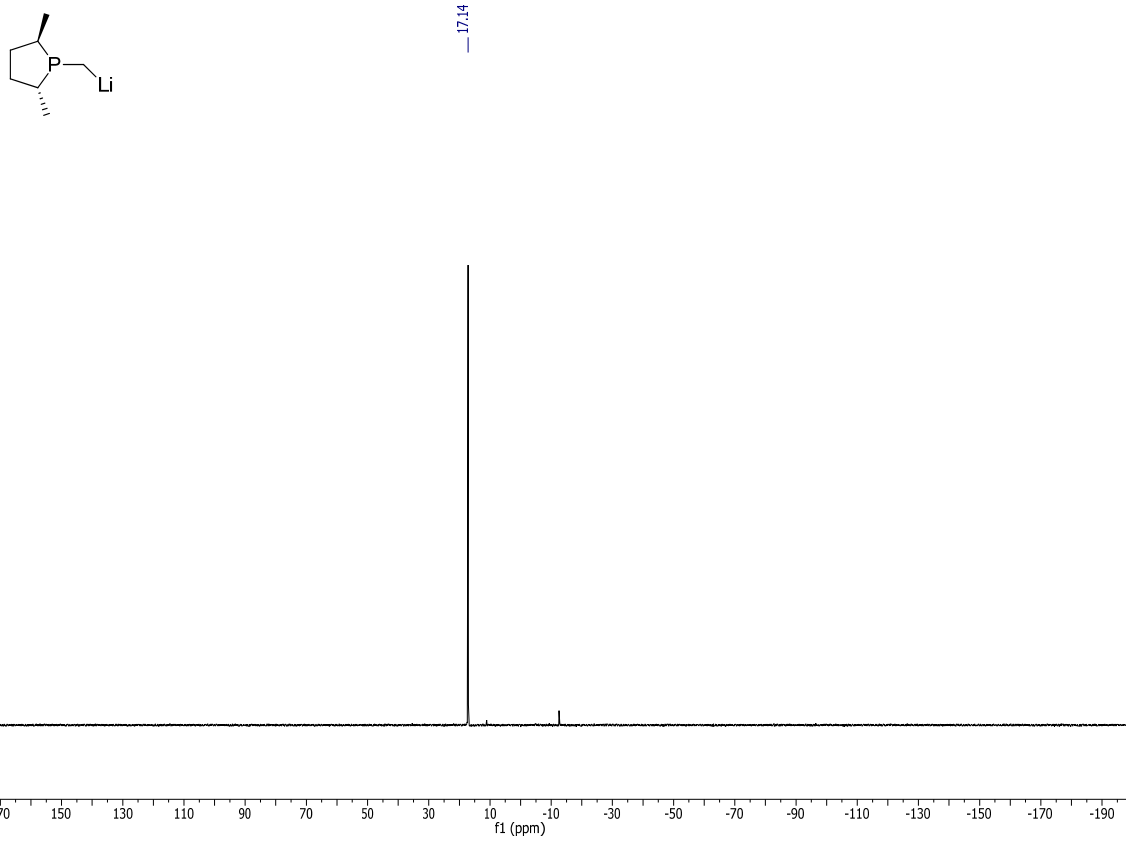

P—

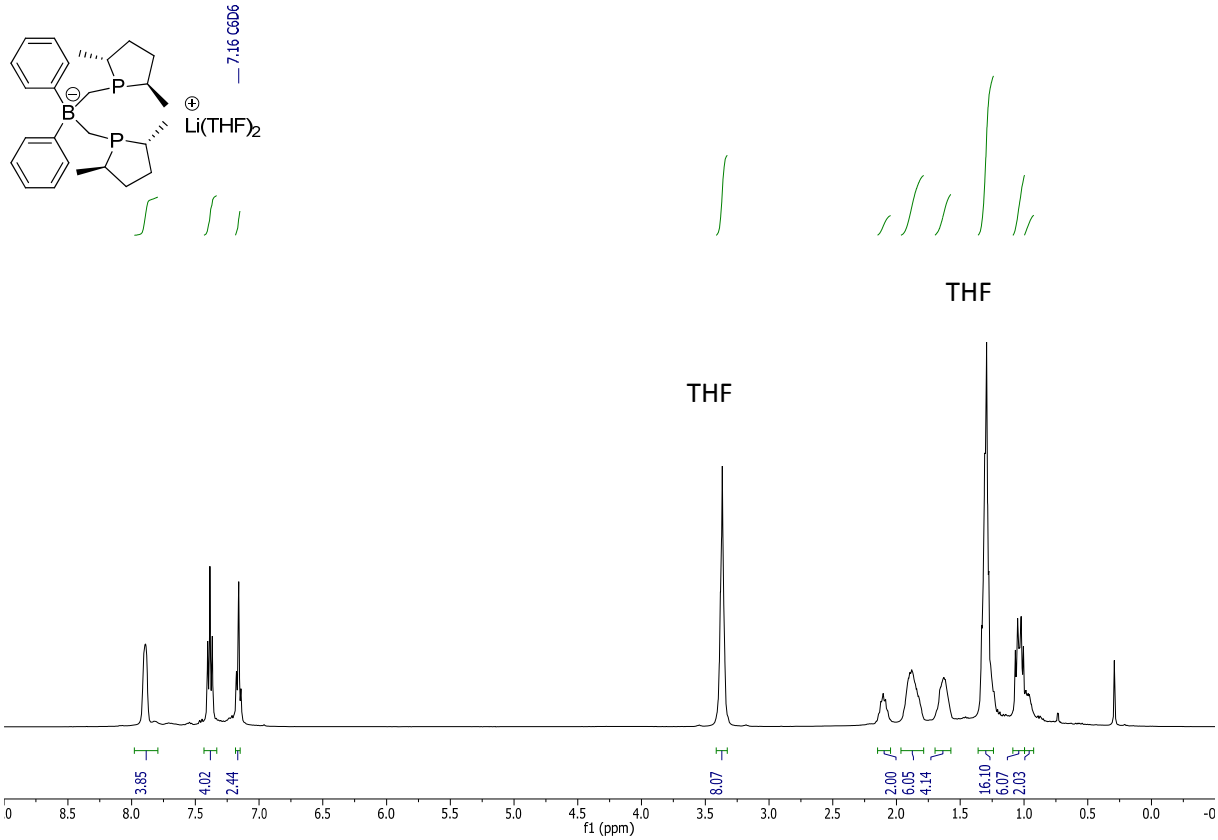
7.16 C6D6
2.00
1.12
0.98
1.28
3.09
1.12
3.34
3.32
0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0
f1 (ppm)

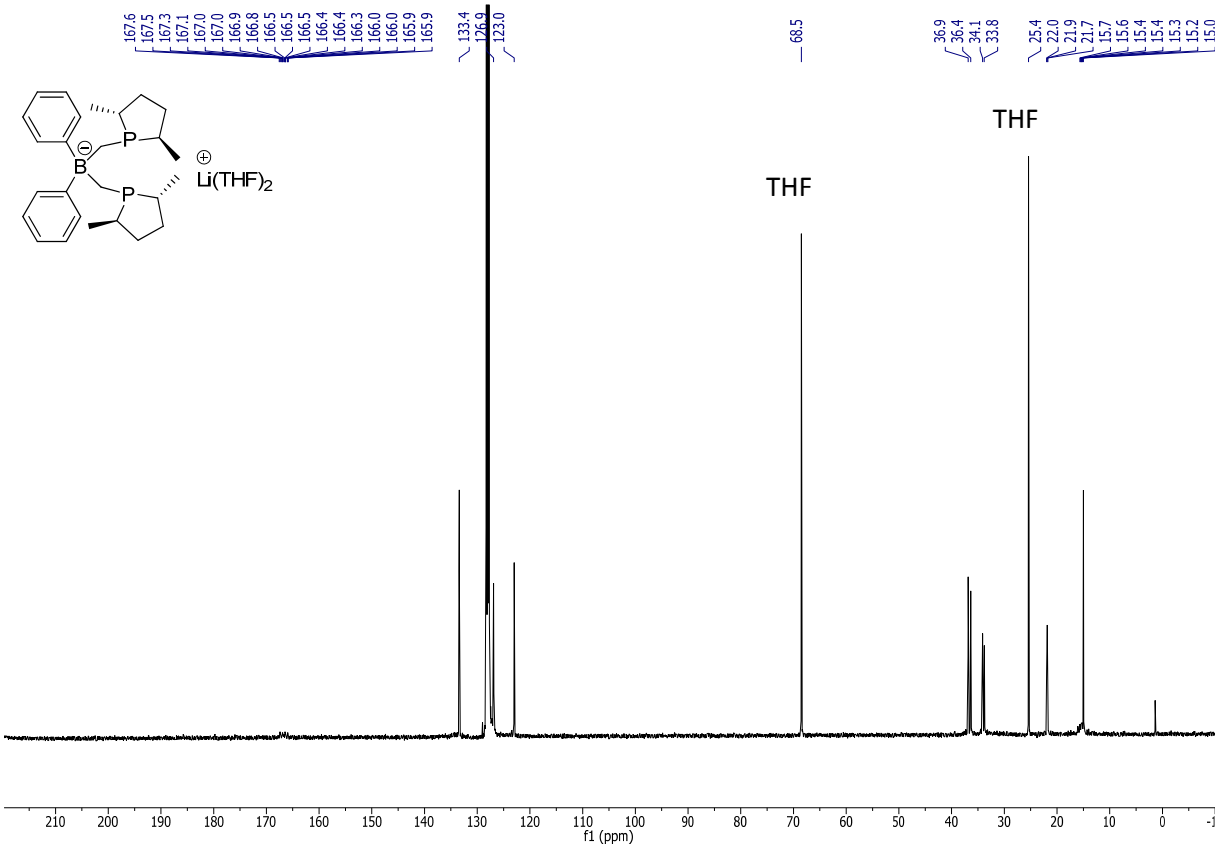

39.5
39.4
37.3
37.3
36.1
36.1
33.3
33.2
21.1
20.8
14.0
7.5
7.2
f1 (ppm)

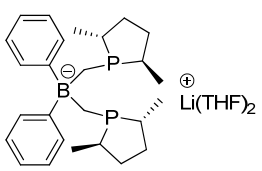

-12.37
f1 (ppm)


P
Li
3.58 THF-d8
1.73 THF-d8
1.00
1.01
4.71
3.06
5.58
1.88
f1 (ppm)

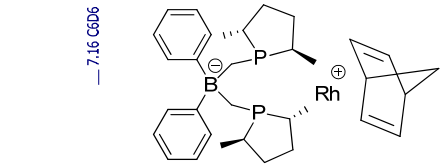

P
Li
17.14
f1 (ppm)


7.16 C6D6
Li(THF)2
THF
THF
3.85
4.02
2.44
8.07
2.00
6.05
4.14
16.10
6.07
2.03
f1 (ppm)

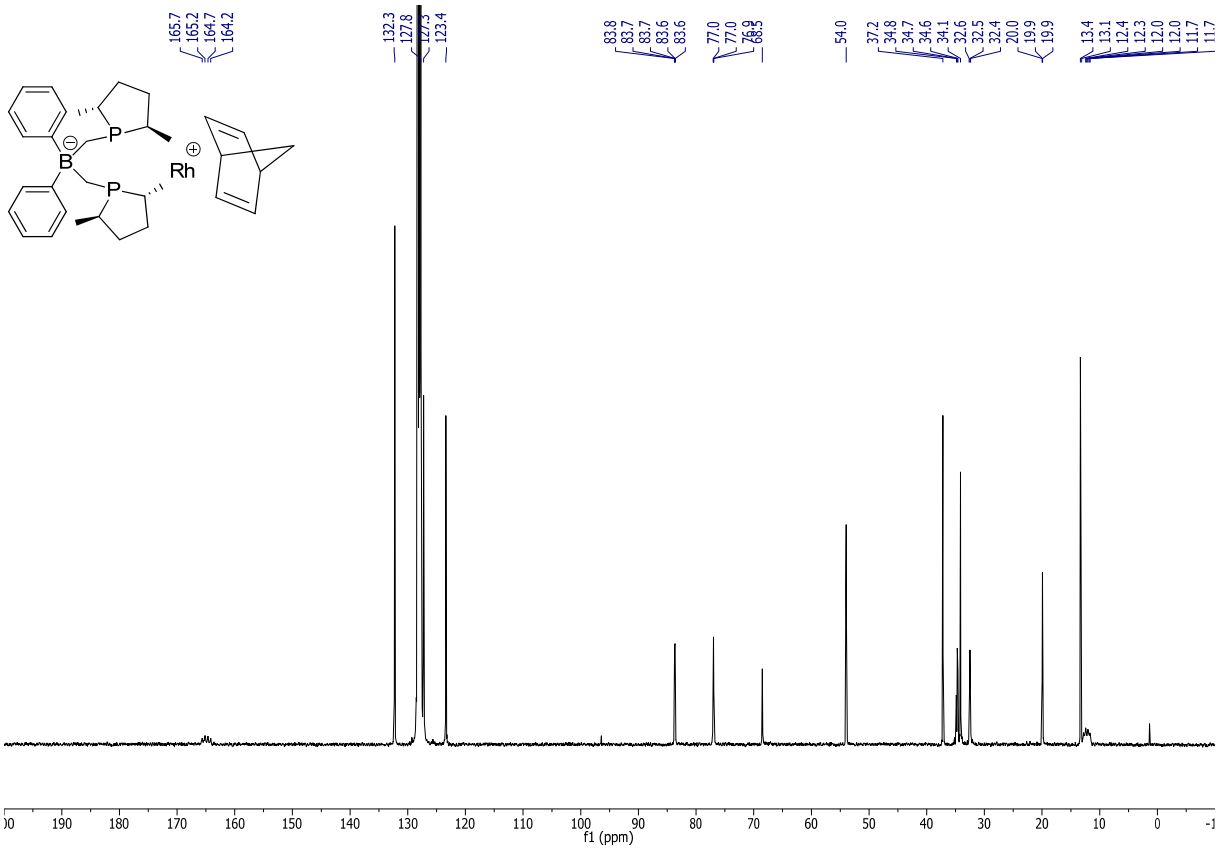

167.6
167.5
167.3
167.1
167.0
167.0
166.9
166.8
166.5
166.5
166.5
166.4
166.4
166.3
166.0
166.0
165.9
165.9
133.4
126.9
123.0
68.5
36.9
36.4
34.1
33.8
25.4
22.0
21.9
21.7
15.7
15.6
15.4
15.4
15.3
15.2
15.0
Li(THF)2
THF
THF
f1 (ppm)

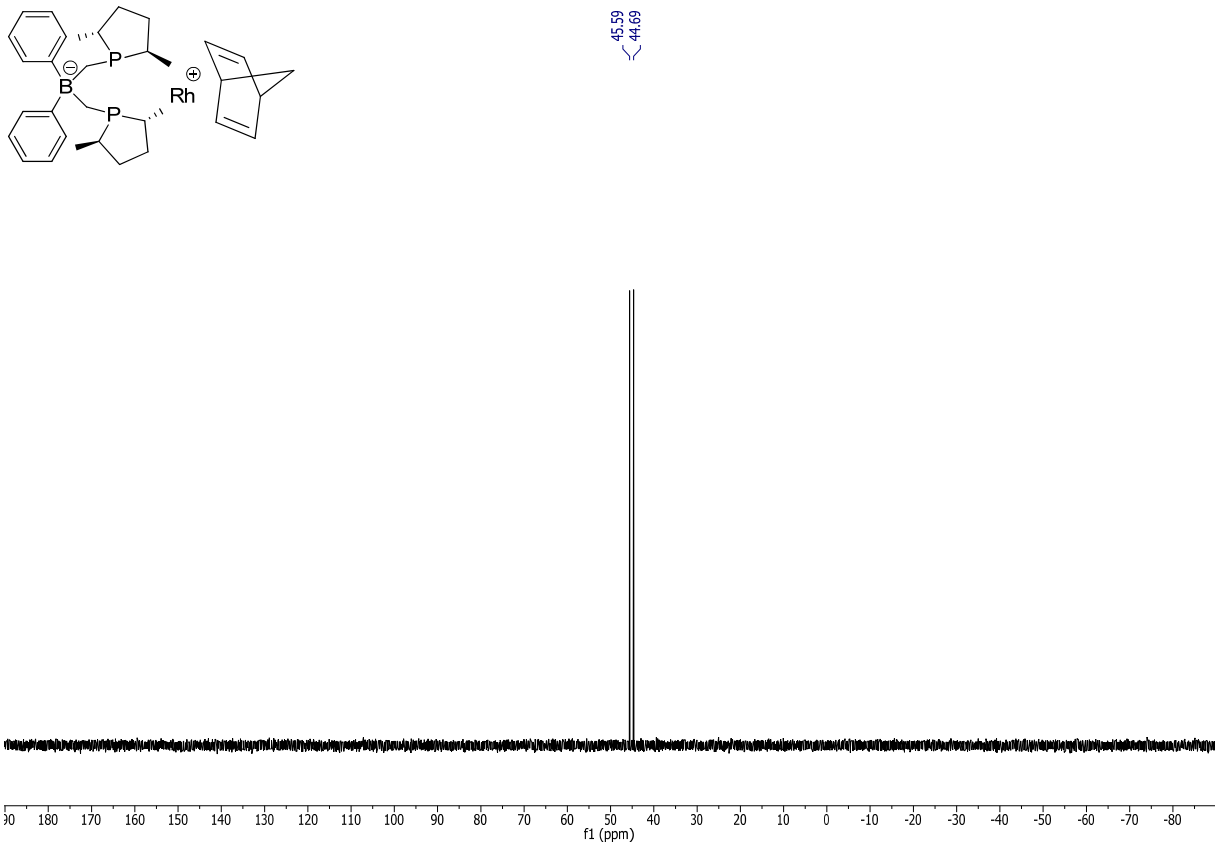

P
B
P
Li(THF)2

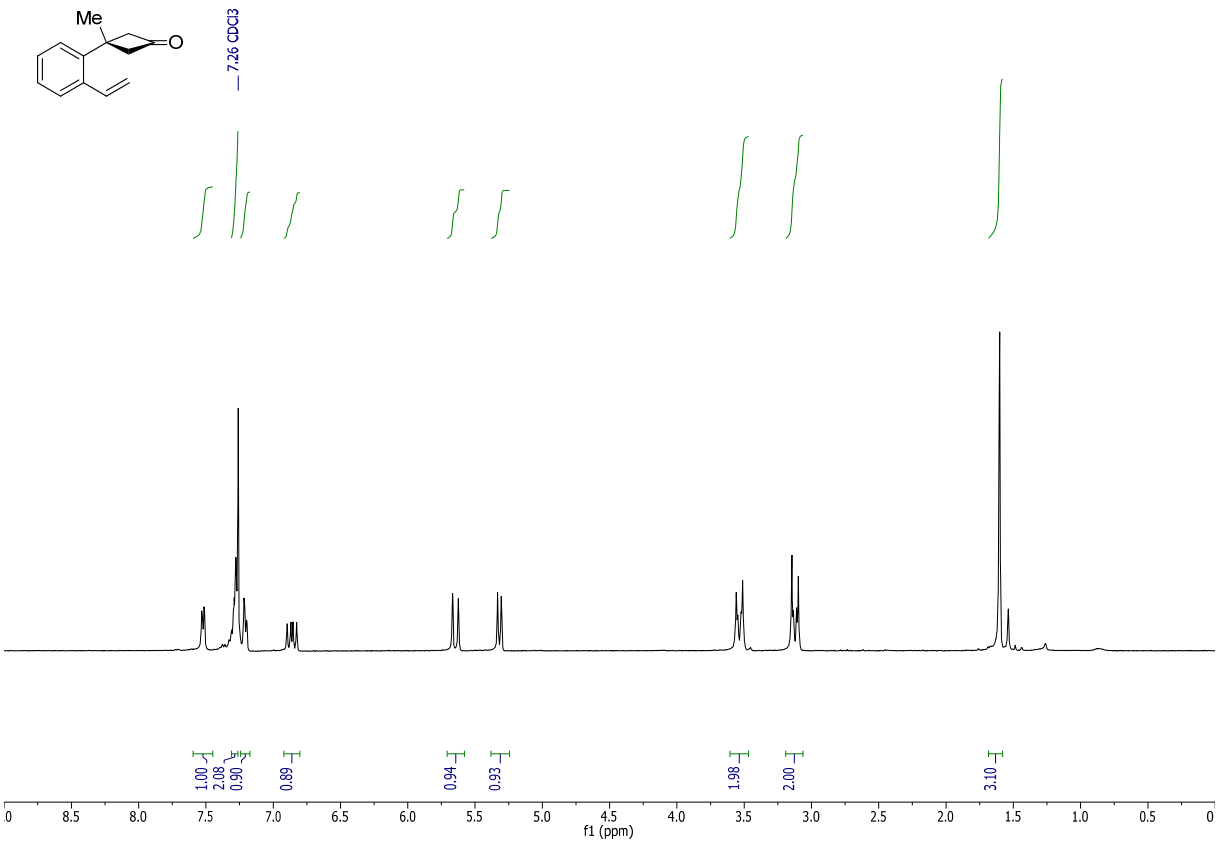
2.80
130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -1
f1 (ppm)

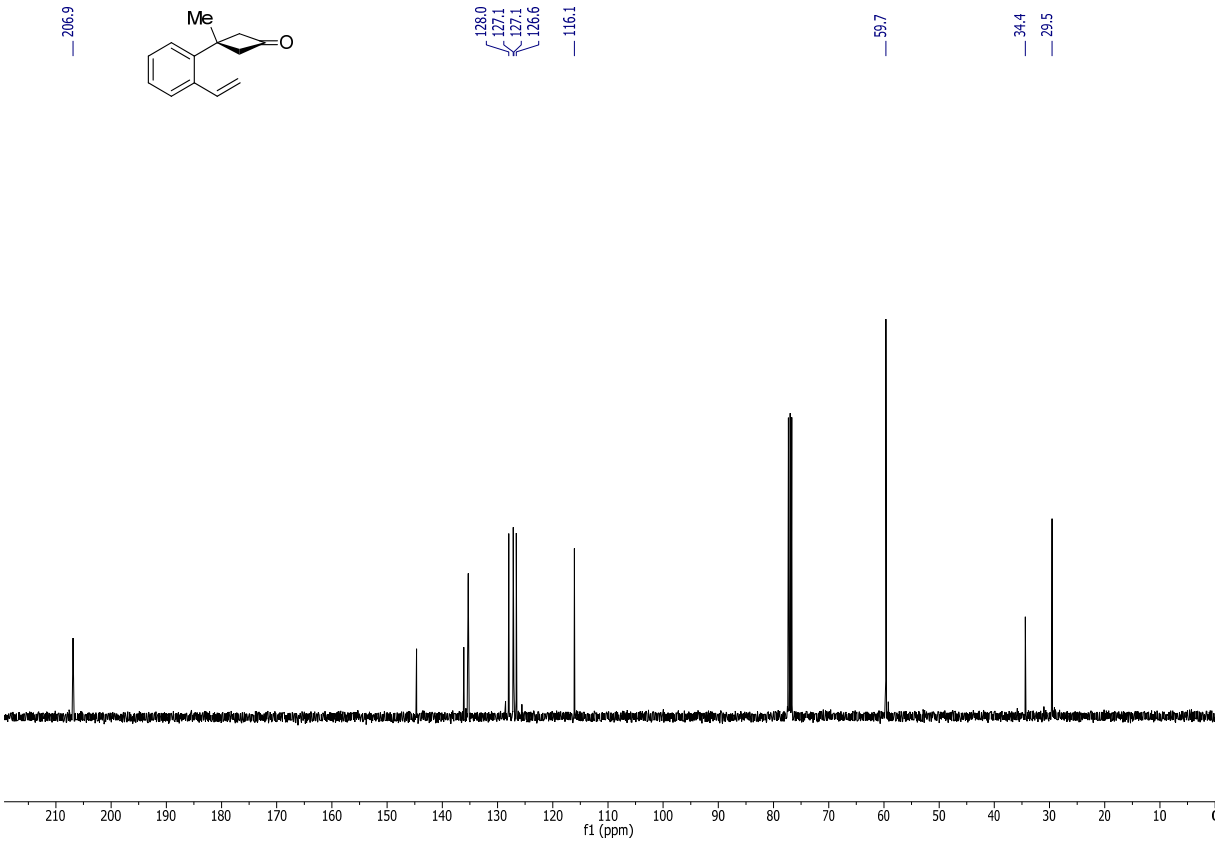

P
B
P
Rh

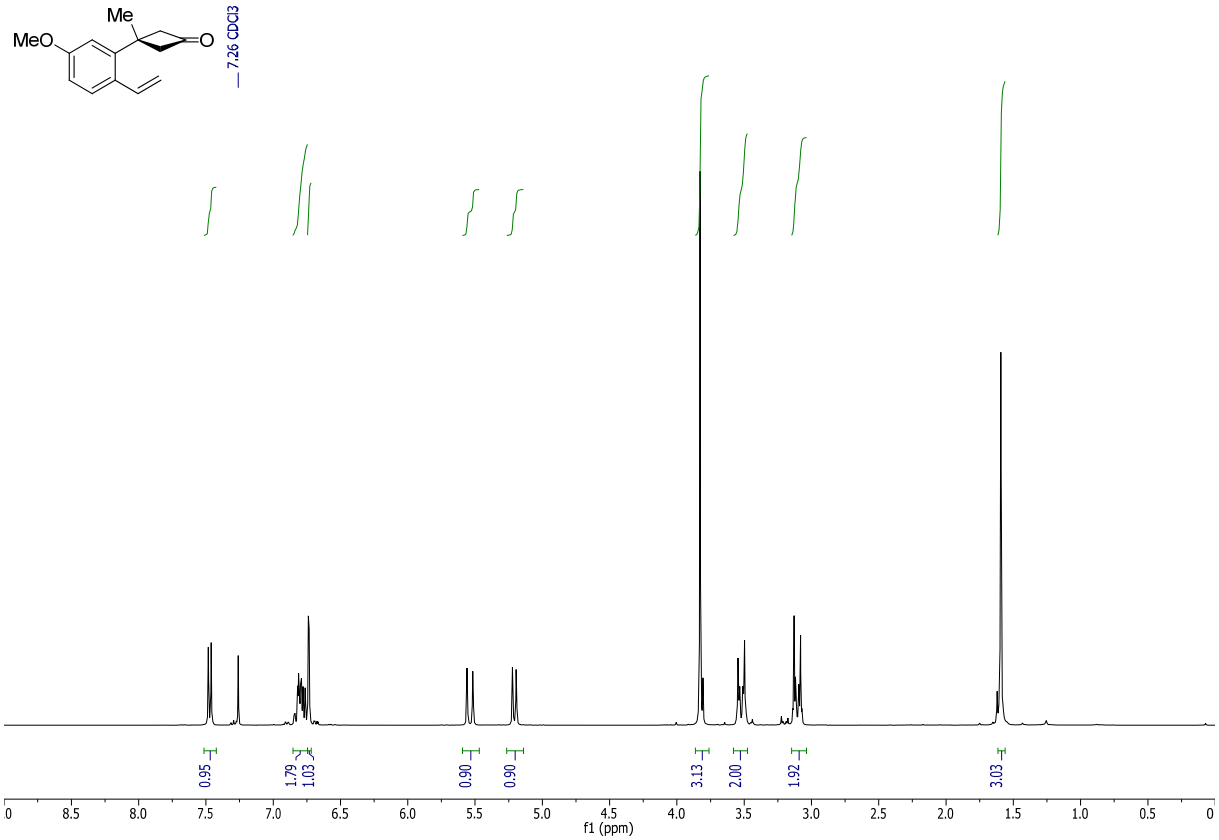
7.16 C6D6
4.06
4.04
2.09
2.01
1.99
1.95
2.06
2.02
4.18
9.95
4.09
5.99
2.03
0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0
f1 (ppm)

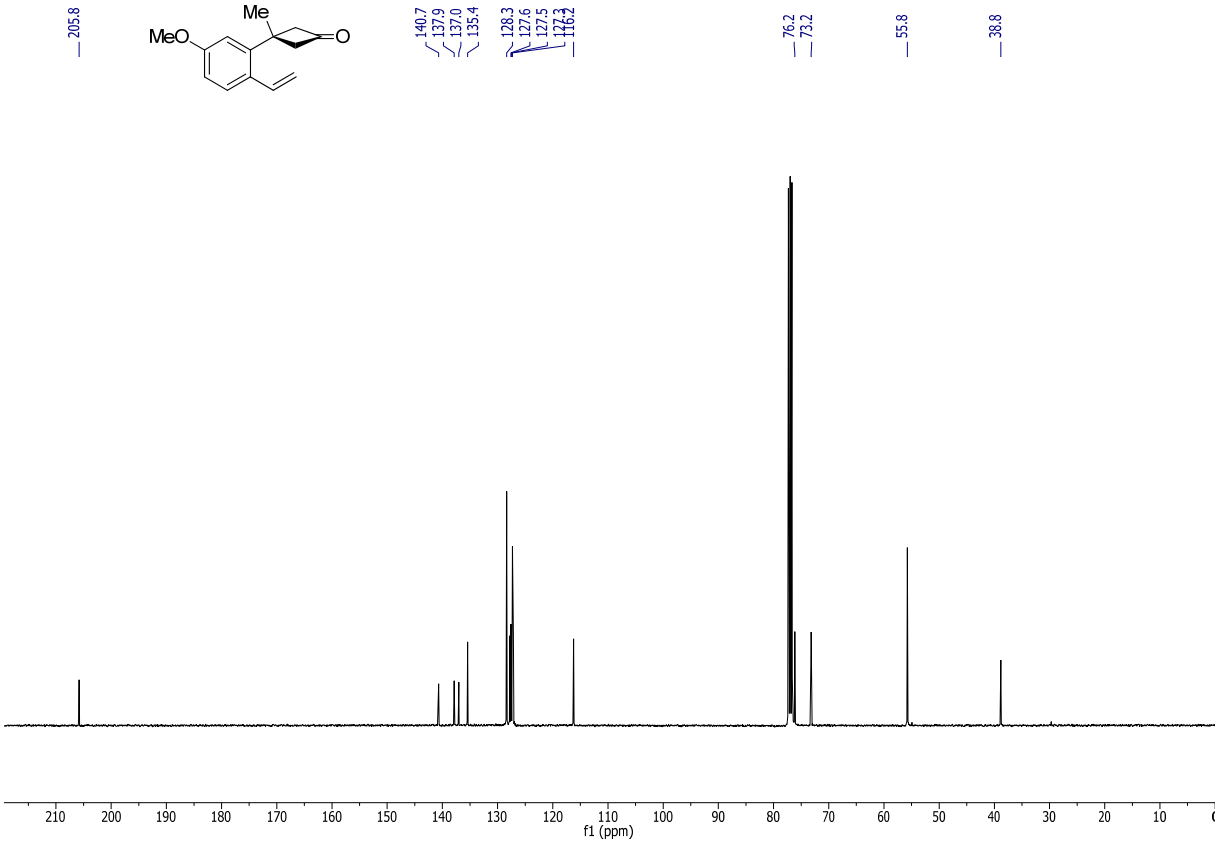

165.7
165.2
164.7
164.2
132.3
127.8
127.3
123.4
83.8
83.7
83.7
83.6
83.6
77.0
77.0
76.9
68.5
54.0
37.2
34.8
34.7
34.6
34.1
32.6
32.5
32.4
20.0
19.9
19.9
13.4
13.1
12.4
12.3
12.0
12.0
11.7
11.7
P
B
P
Rh
f1 (ppm)

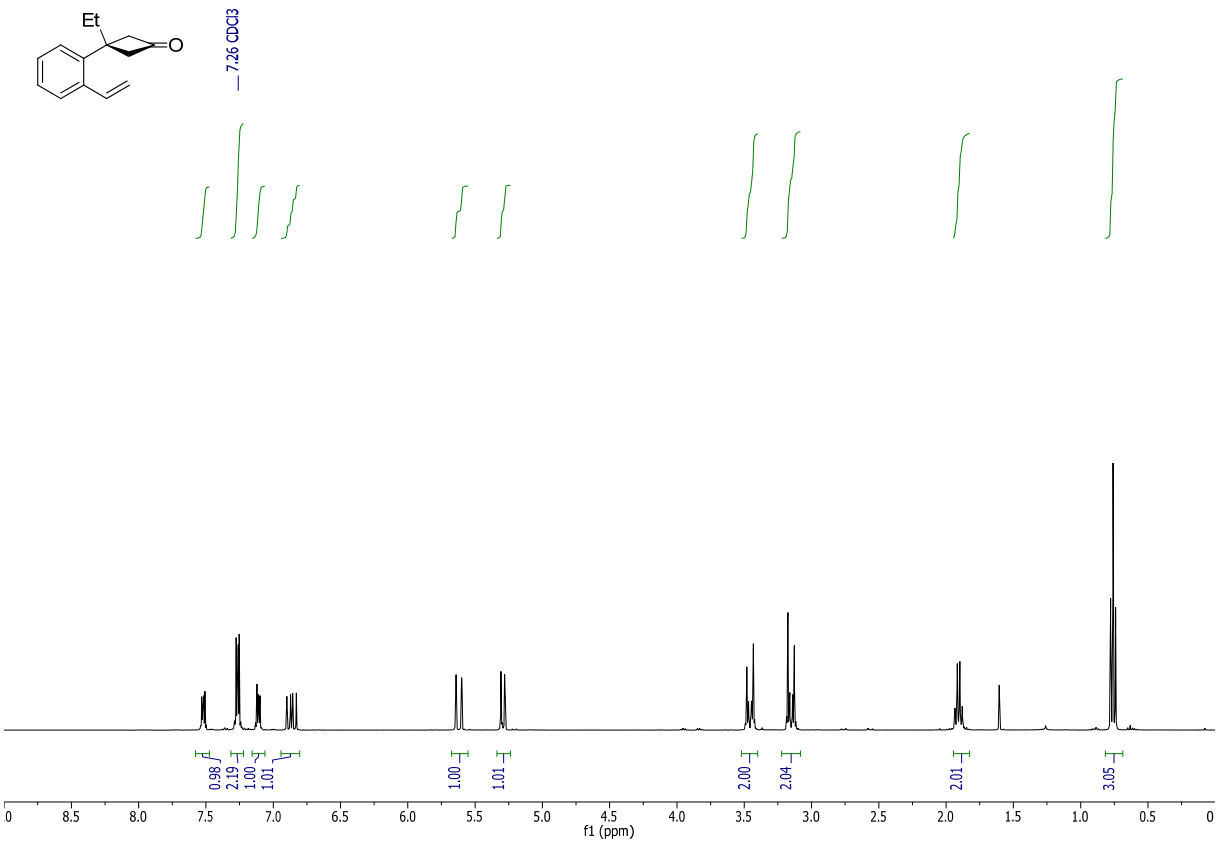

45.59
44.69
P
B
P
Rh
f1 (ppm)

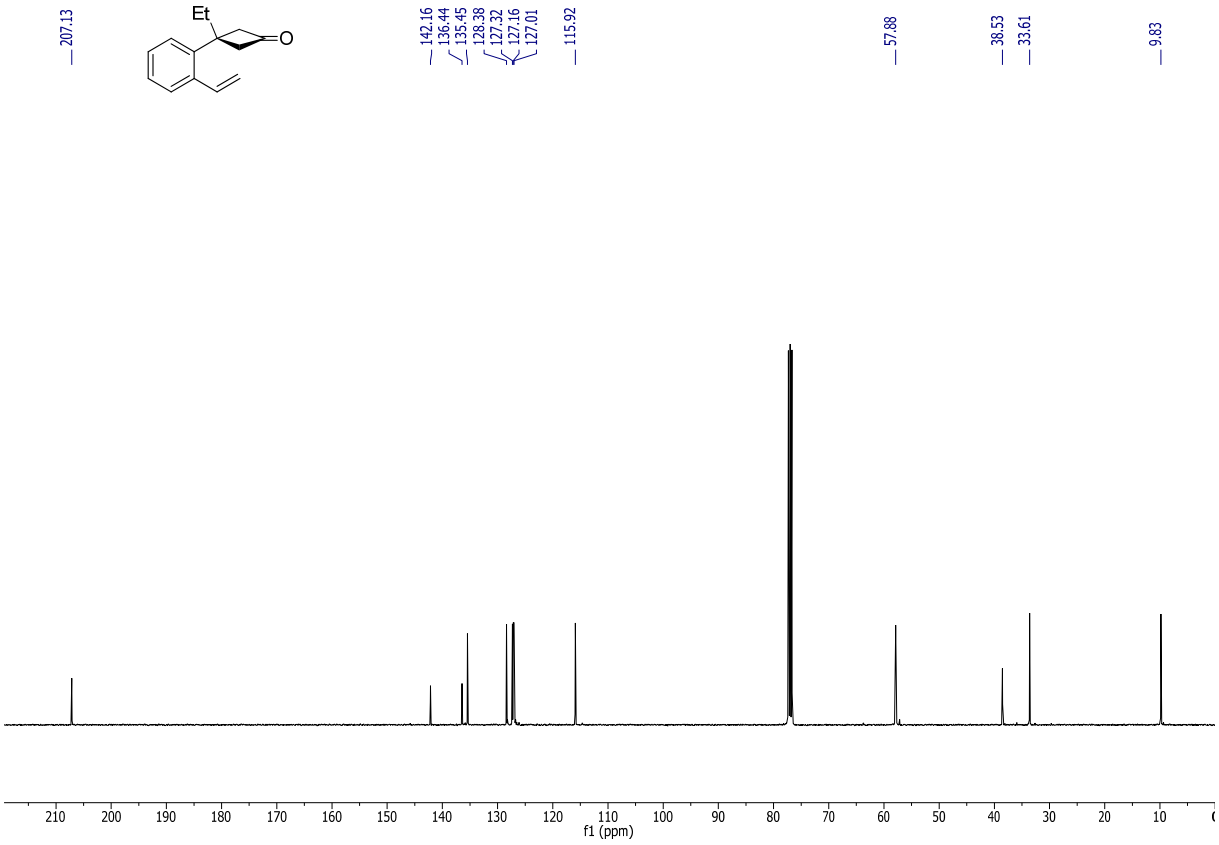

Me
O
7.26 CDCl3
1.00
2.08
0.90
0.89
0.94
0.93
1.98
2.00
3.10
f1 (ppm)

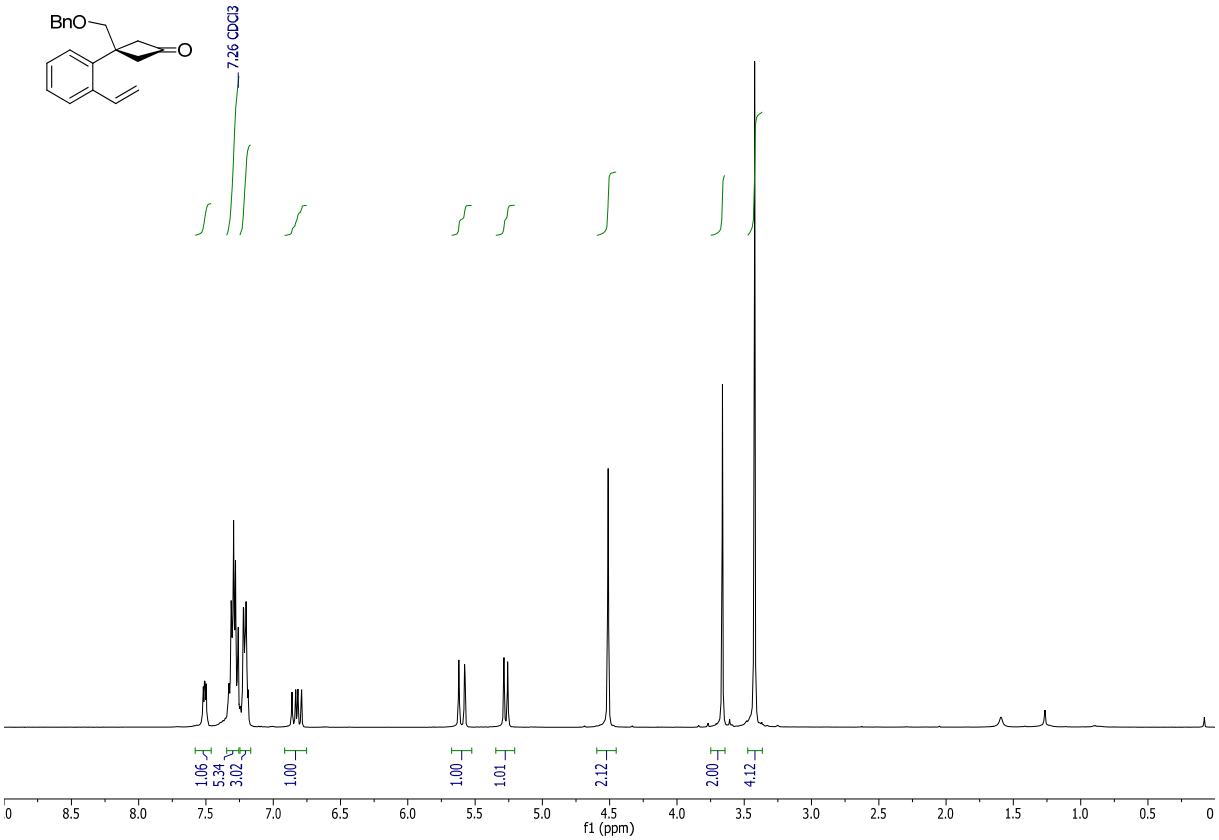

206.9
Me
O
128.0
127.1
127.1
126.6
116.1
59.7
34.4
29.5
f1 (ppm)

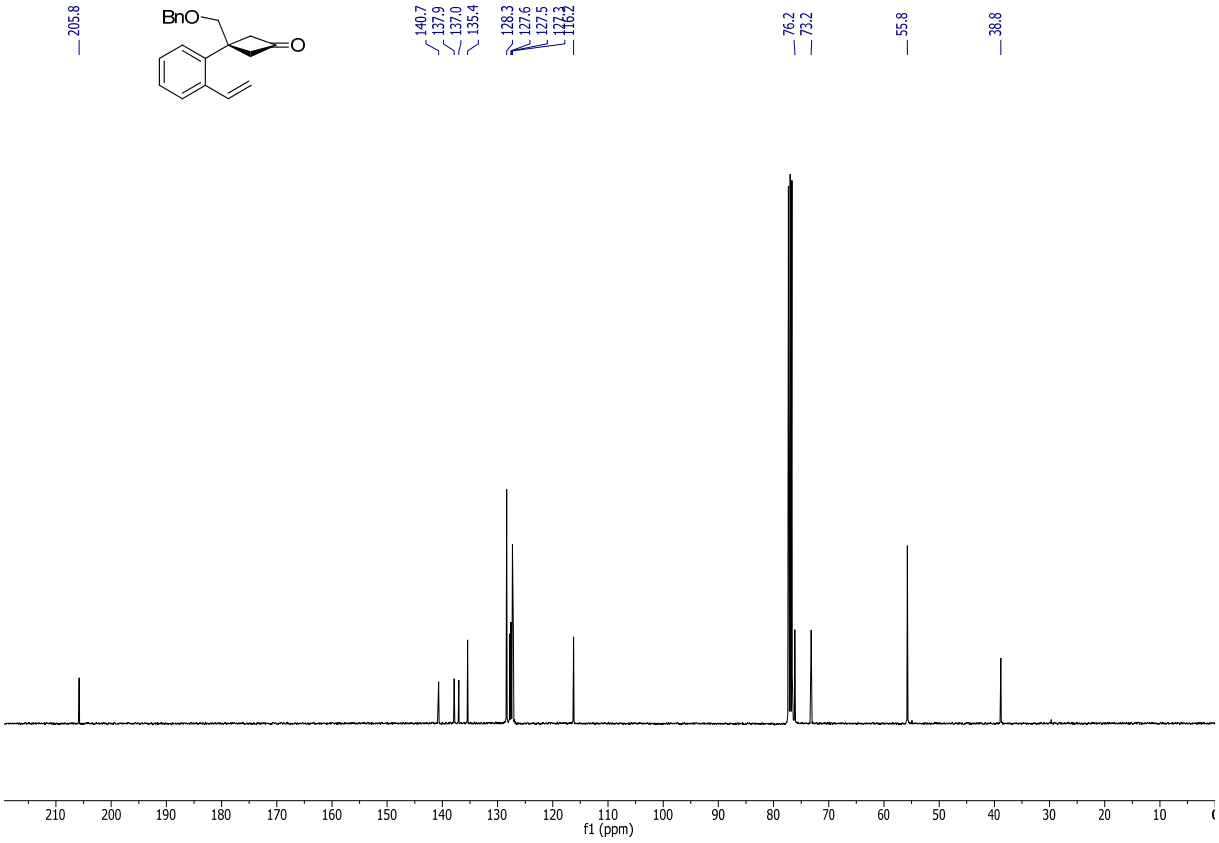

Me
MeO
O
7.26 CDCl3
0.95
1.79
1.03
0.90
0.90
3.13
2.00
1.92
3.03
f1 (ppm)

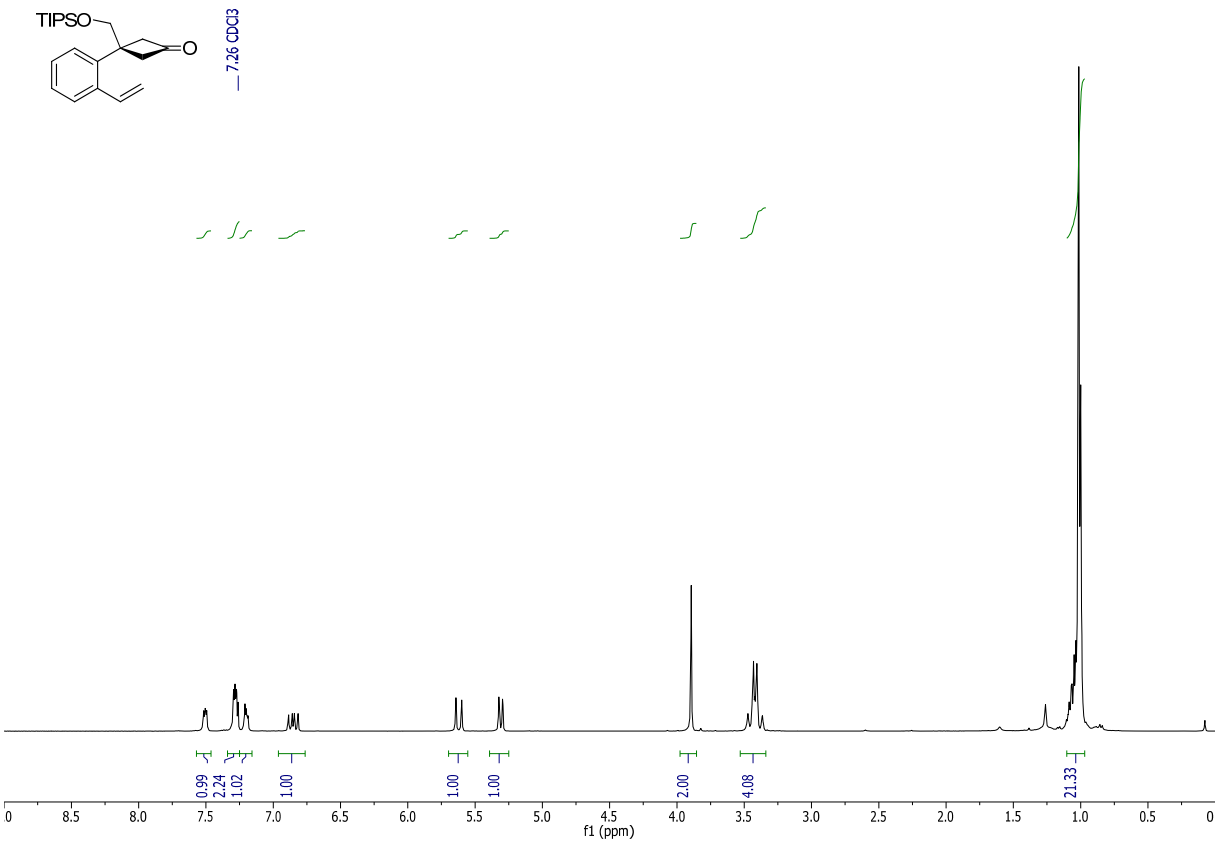

205.8
Me
MeO
O
140.7
137.9
137.0
135.4
128.3
127.6
127.5
76.2
73.2
55.8
38.8
f1 (ppm)

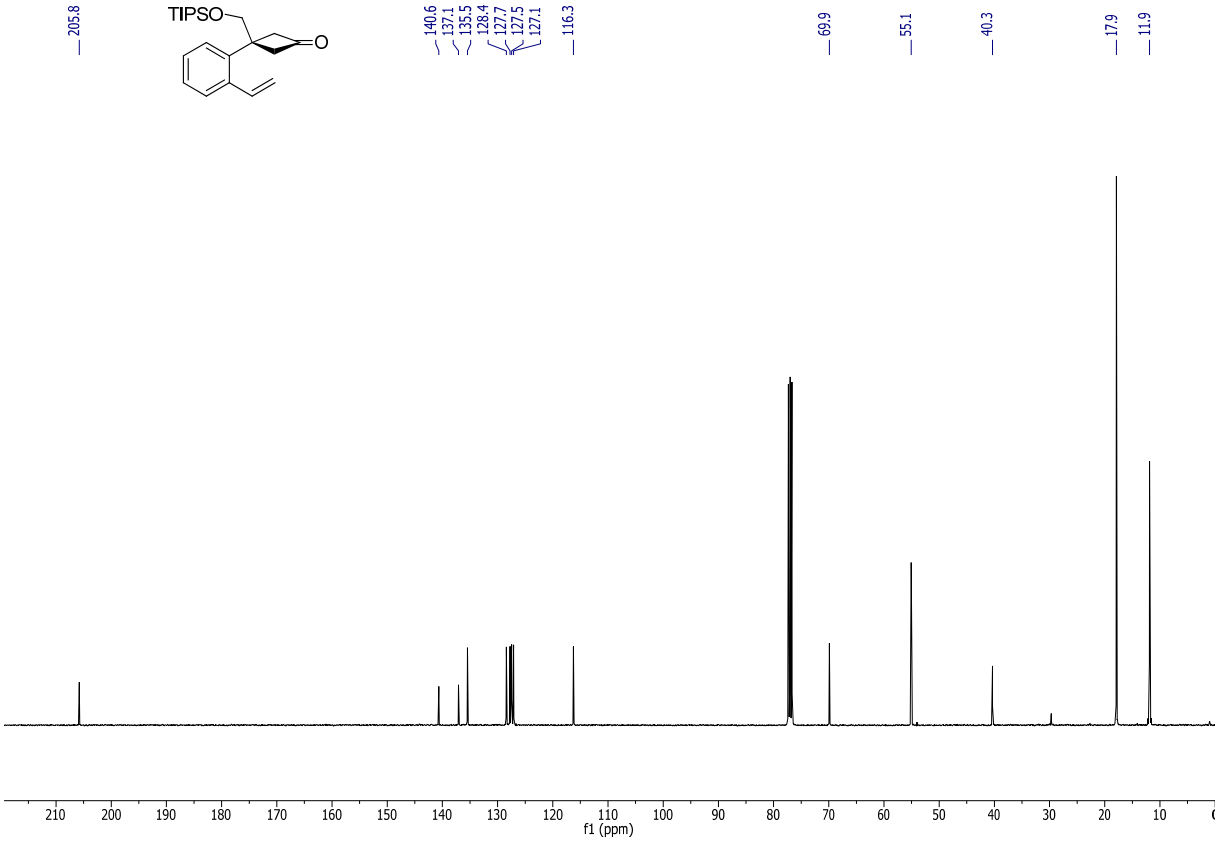

Et
O
7.26 CDCl3
0.98
2.19
1.00
1.01
1.00
1.01
2.00
2.04
2.01
3.05
f1 (ppm)

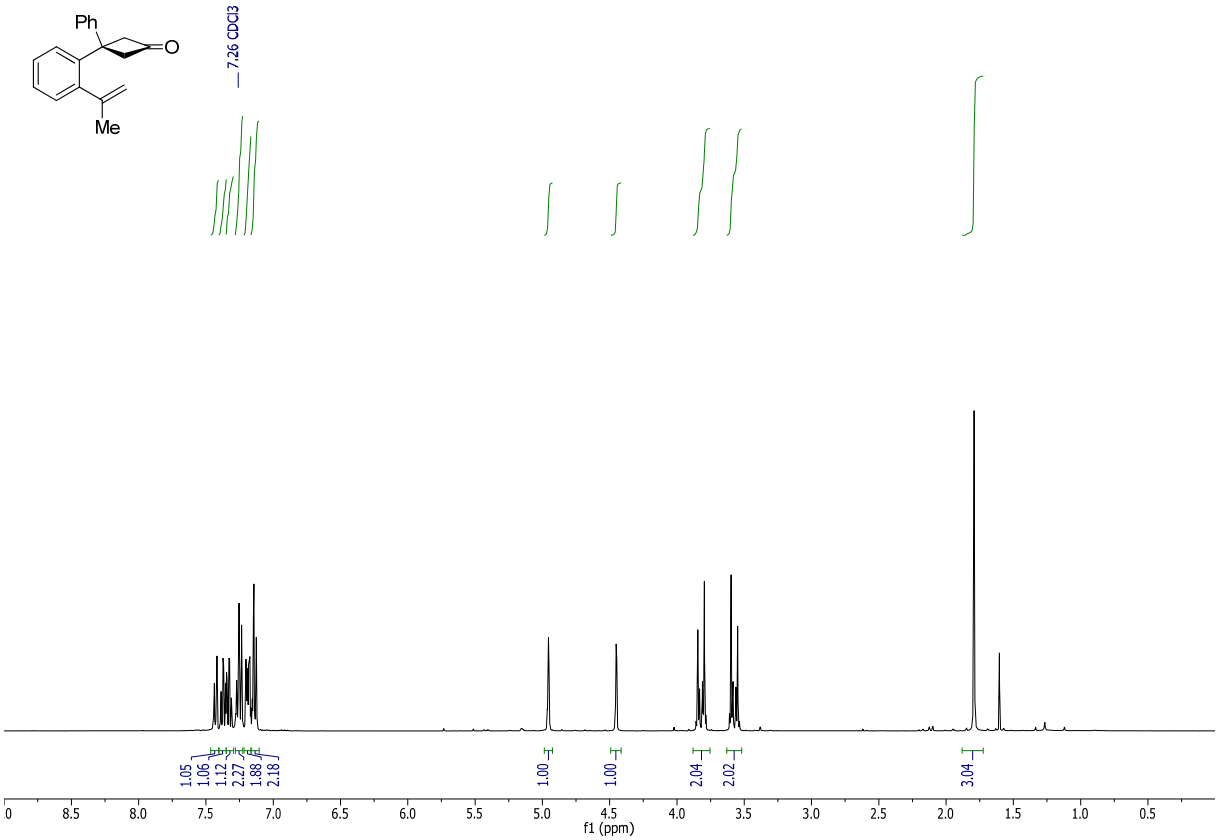

207.13
Et
O
142.16
136.44
135.45
128.38
127.32
127.16
127.01
115.92
57.88
38.53
33.61
9.83
f1 (ppm)

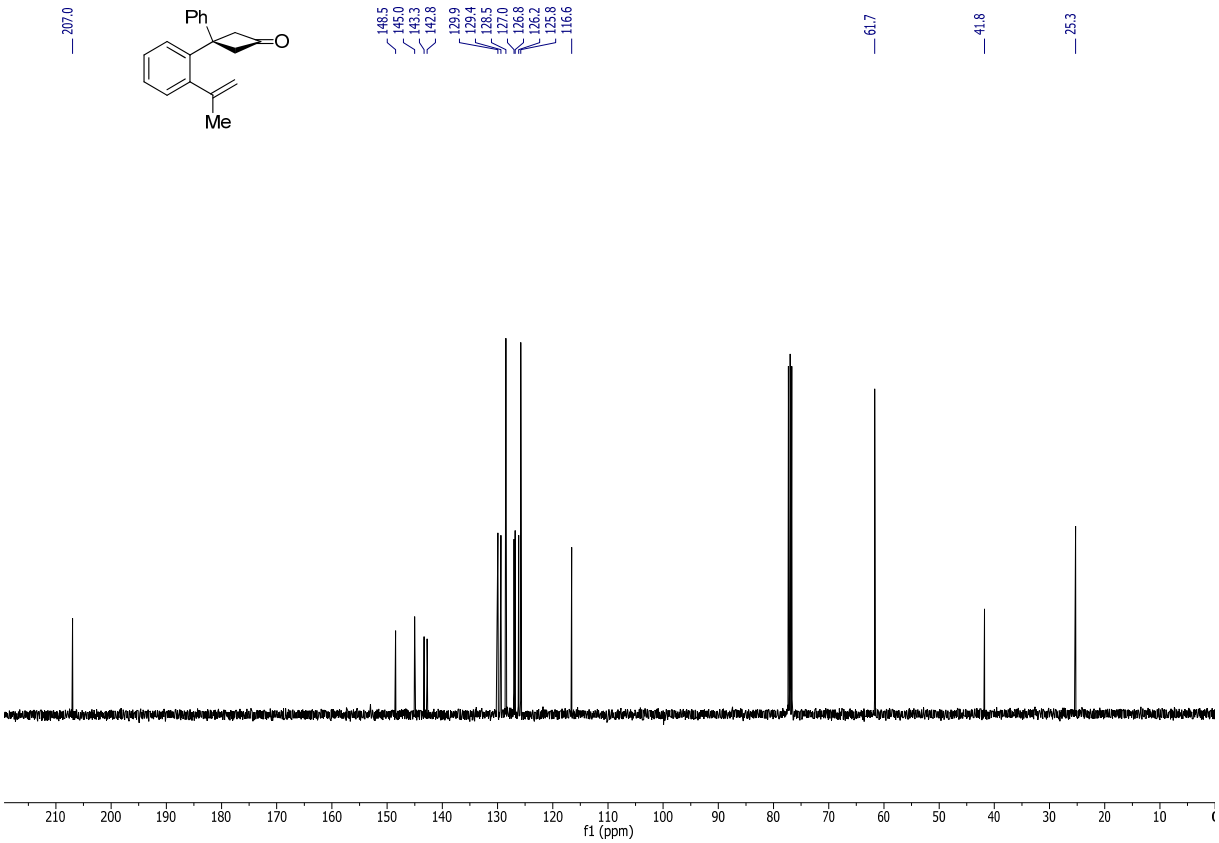

BnO
O
7.26 CDCl3
1.06
5.34
3.02
1.00
1.00
1.01
2.12
2.00
4.12
f1 (ppm)

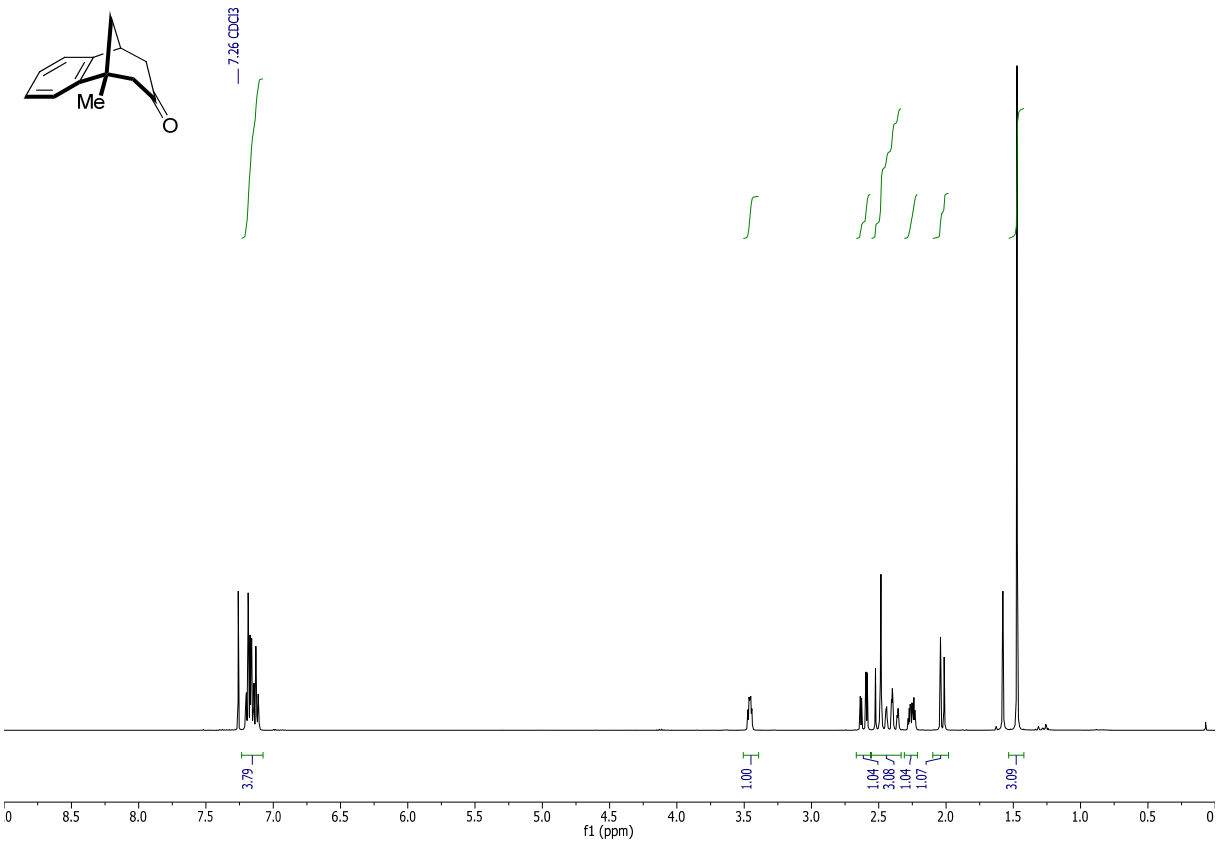

205.8
BnO
O
140.7
137.9
137.0
135.4
128.3
127.6
127.5
116.2
76.2
73.2
55.8
38.8
f1 (ppm)

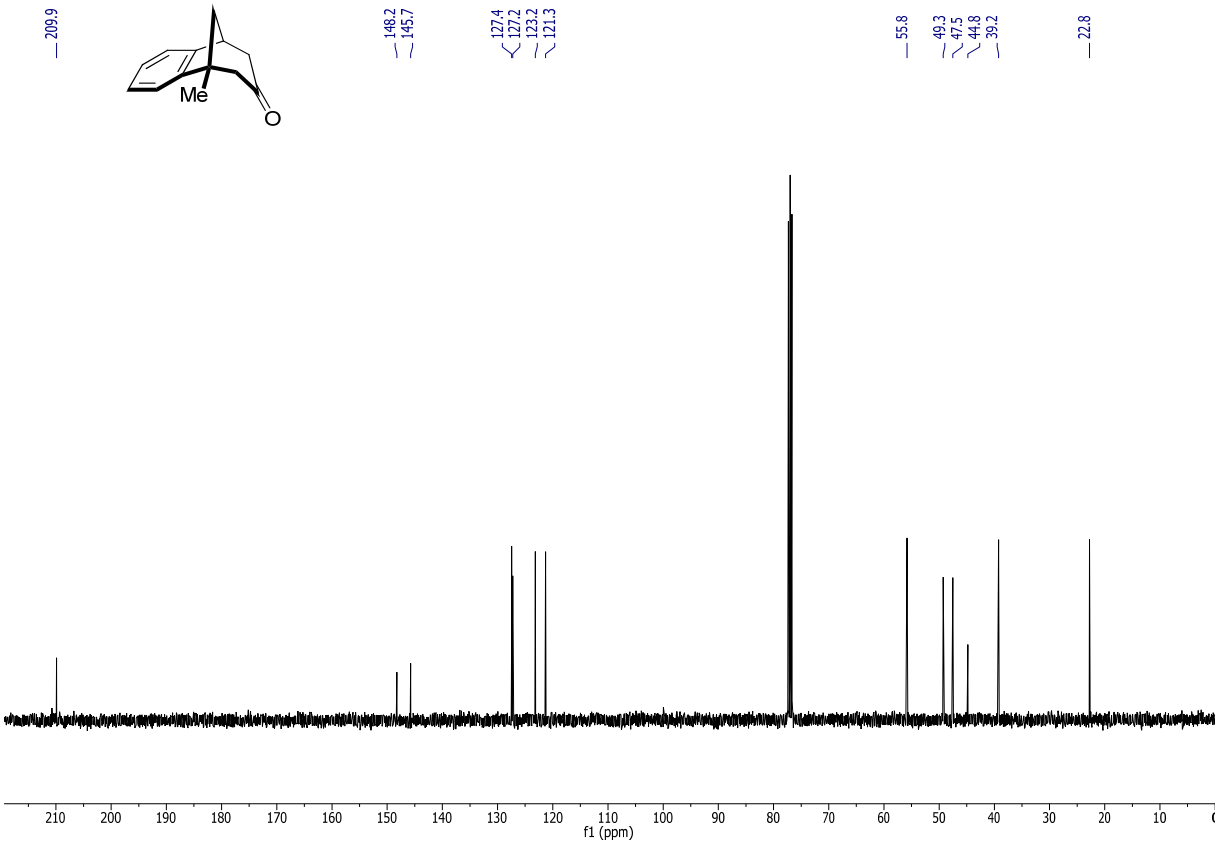

TIPSO
O
7.26 CDCl3
0.99
2.24
1.02
1.00
1.00
1.00
2.00
4.08
21.33
f1 (ppm)

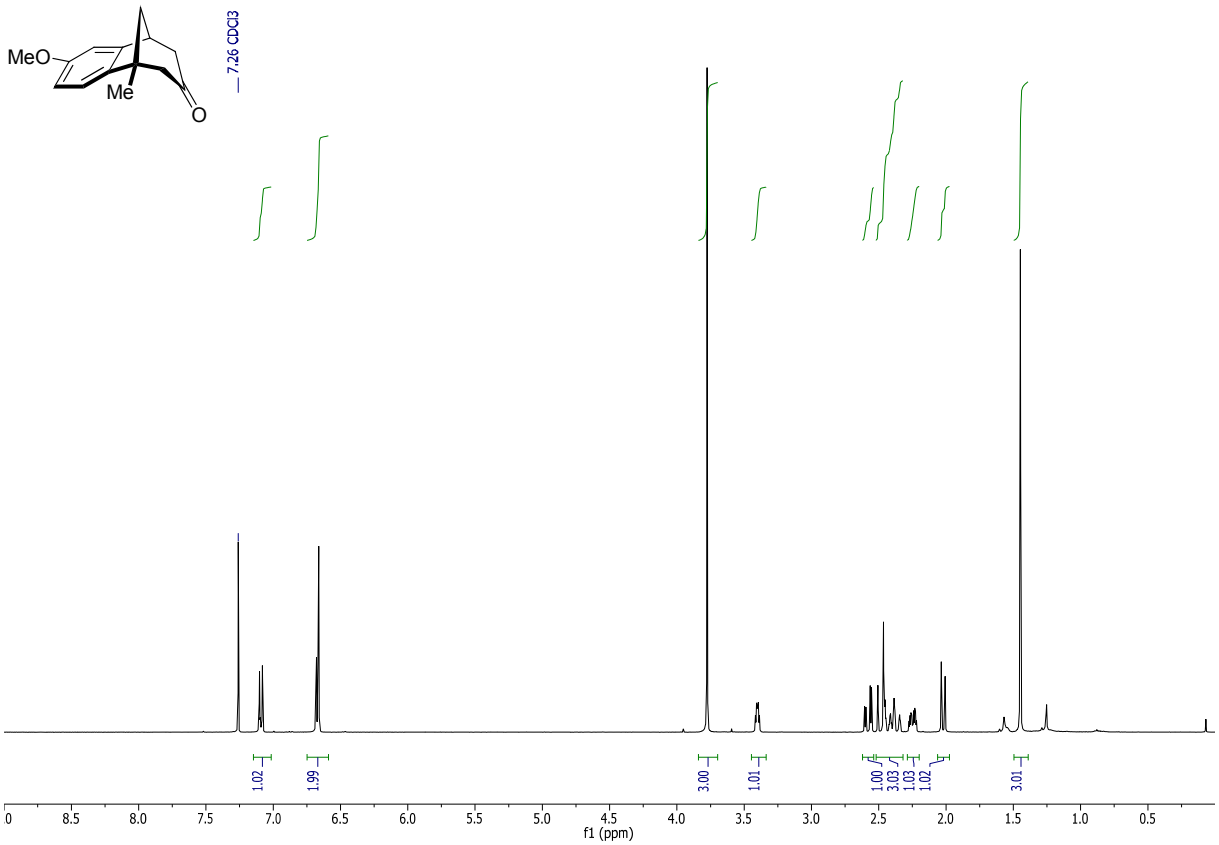

205.8
TIPSO
O
140.6
137.1
135.5
128.4
127.7
127.5
127.1
116.3
69.9
55.1
40.3
17.9
11.9
f1 (ppm)

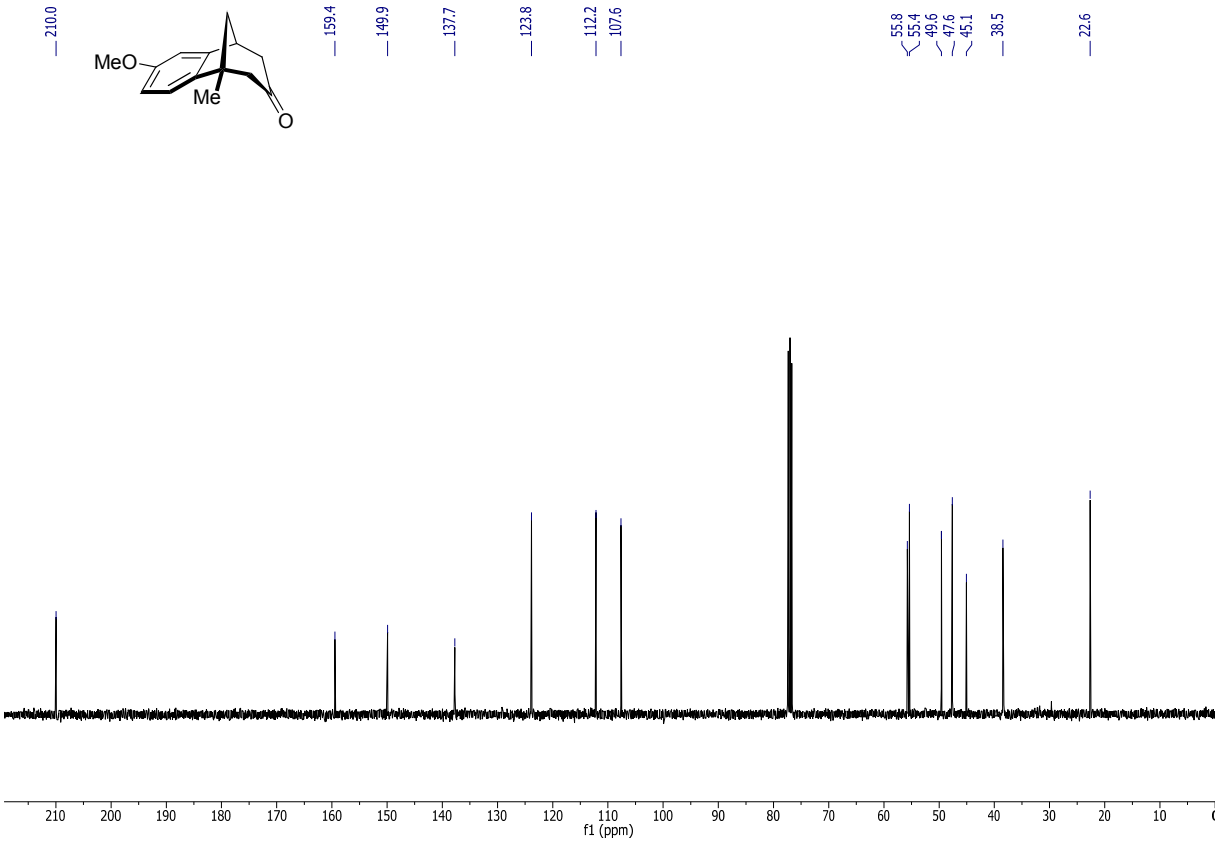

Ph
O
Me
7.26 CDCl3
1.05
1.06
1.12
2.27
1.88
2.18
1.00
1.00
2.04
2.02
3.04
f1 (ppm)

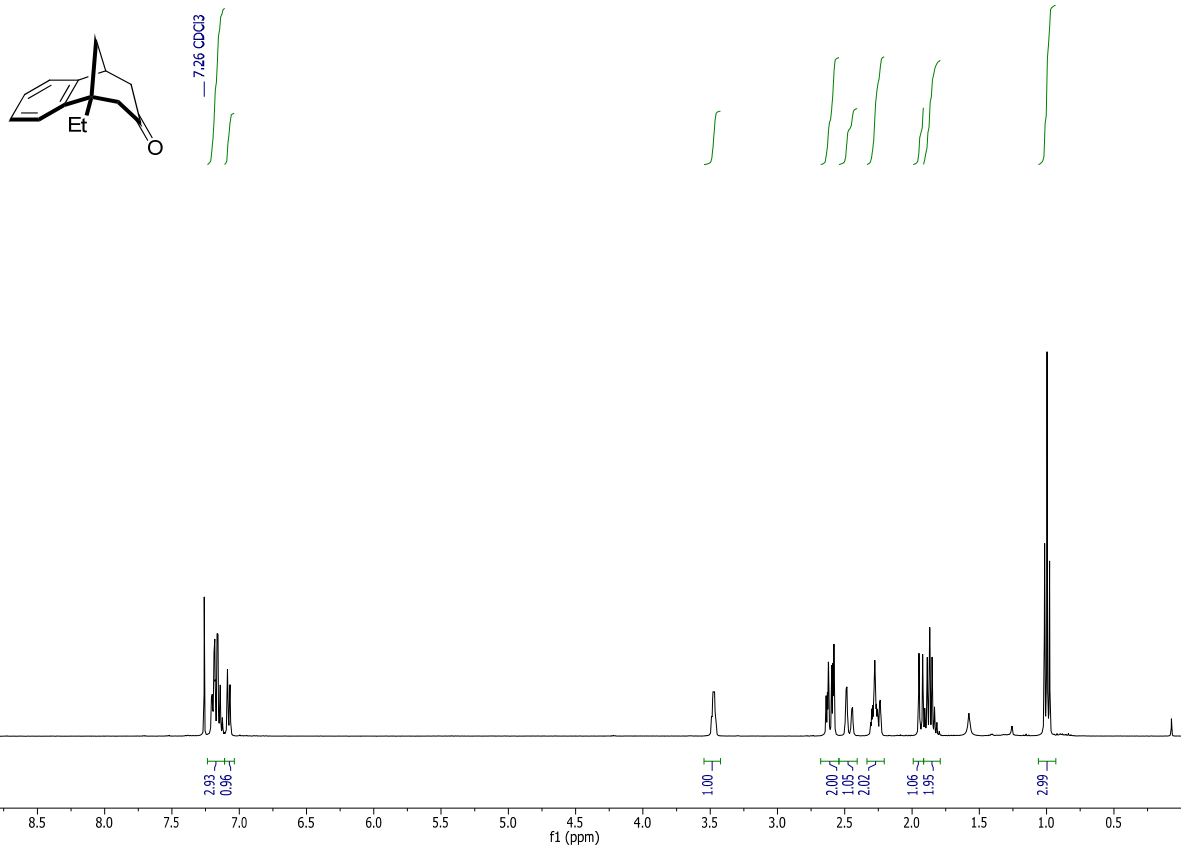

207.0
Ph
O
Me
148.5
145.0
143.3
142.8
129.9
129.4
128.5
127.0
126.8
126.2
125.8
116.6
61.7
41.8
25.3
f1 (ppm)

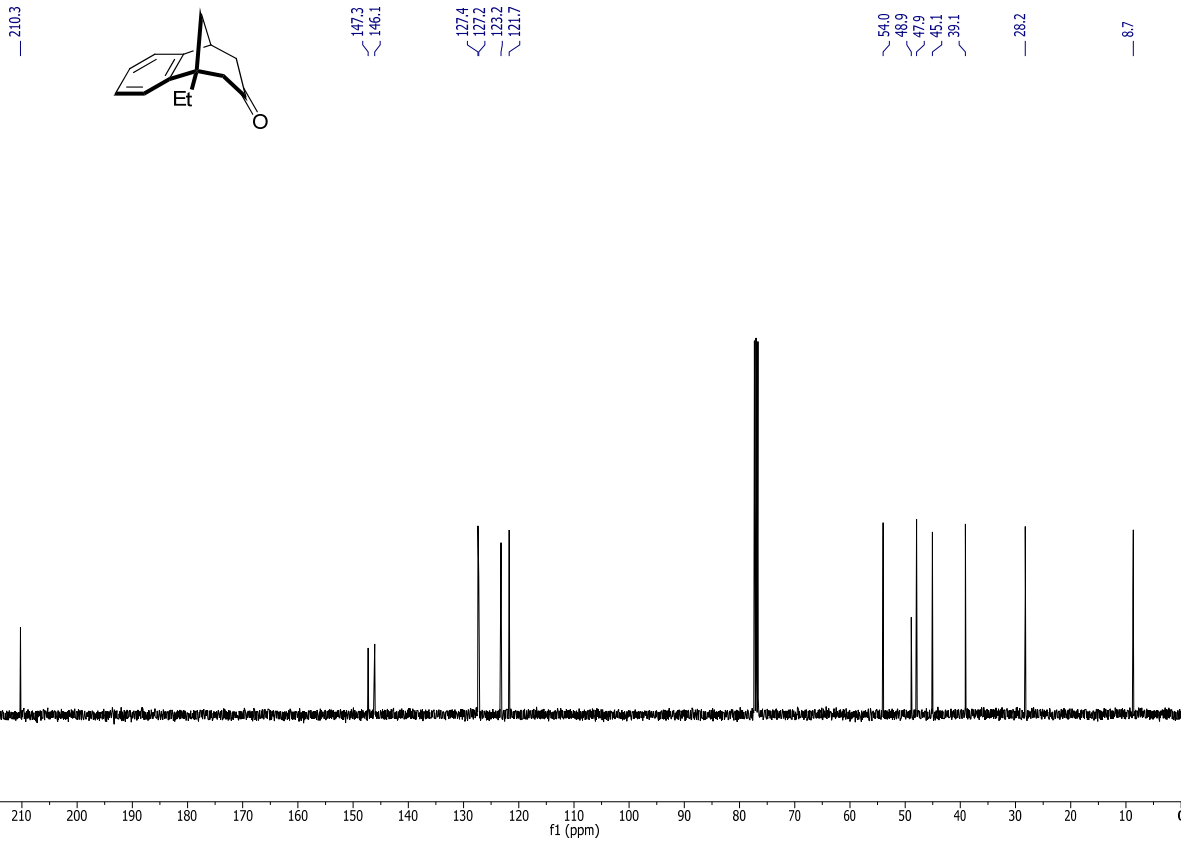

Me
O
7.26 CDCl3
3.79
1.00
1.04
3.08
1.04
1.07
3.09
f1 (ppm)

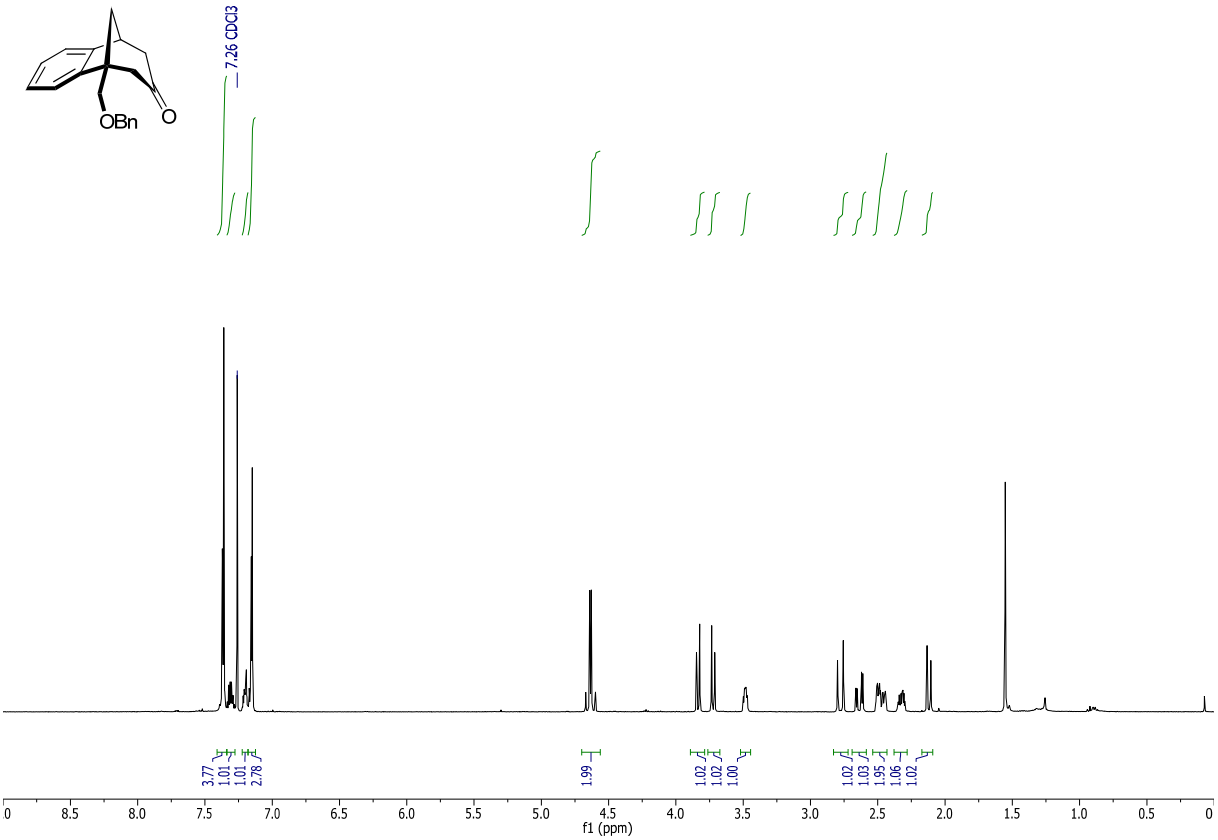

209.9
148.2
145.7
127.4
127.2
123.2
121.3
55.8
49.3
47.5
44.8
39.2
22.8
Me
O
f1 (ppm)

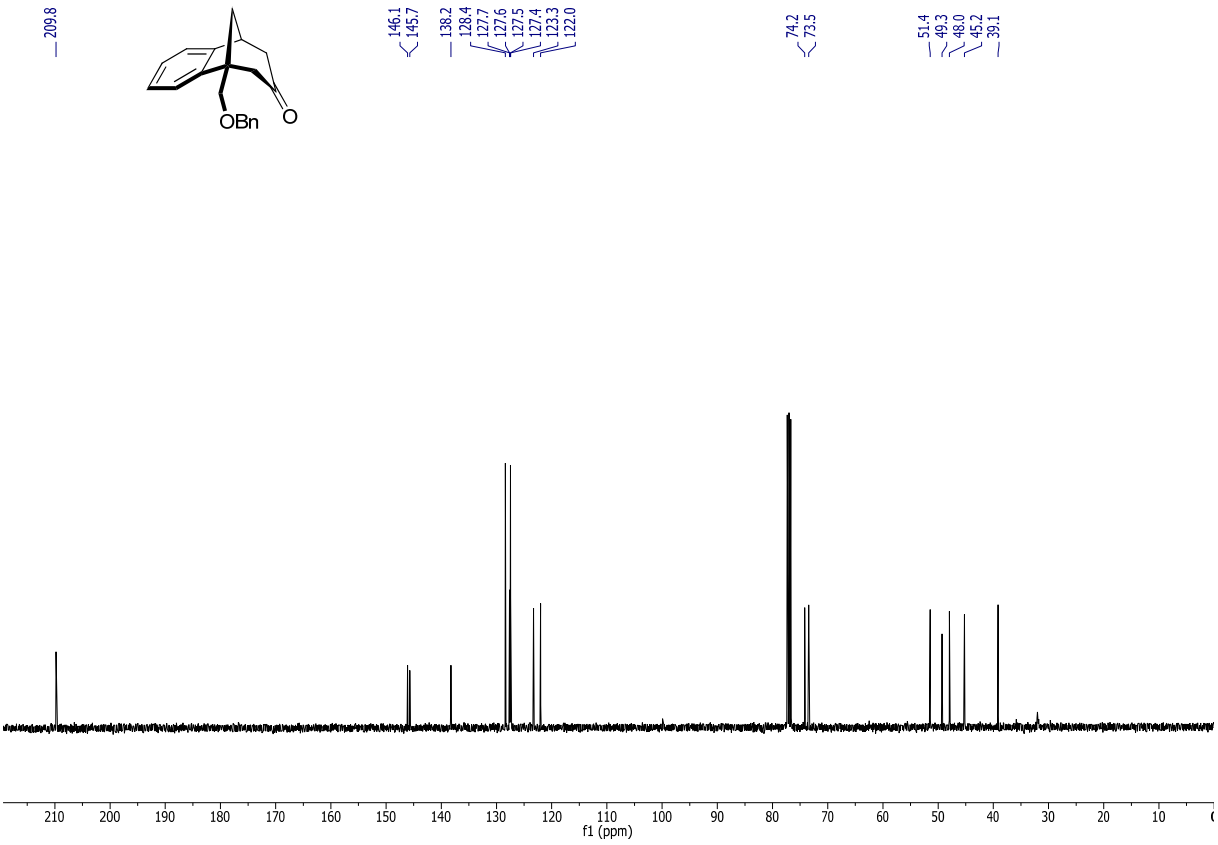

MeO
Me
O
7.26 CDCl3
1.02
1.99
3.00
1.01
1.00
3.03
1.03
1.02
3.01
f1 (ppm)

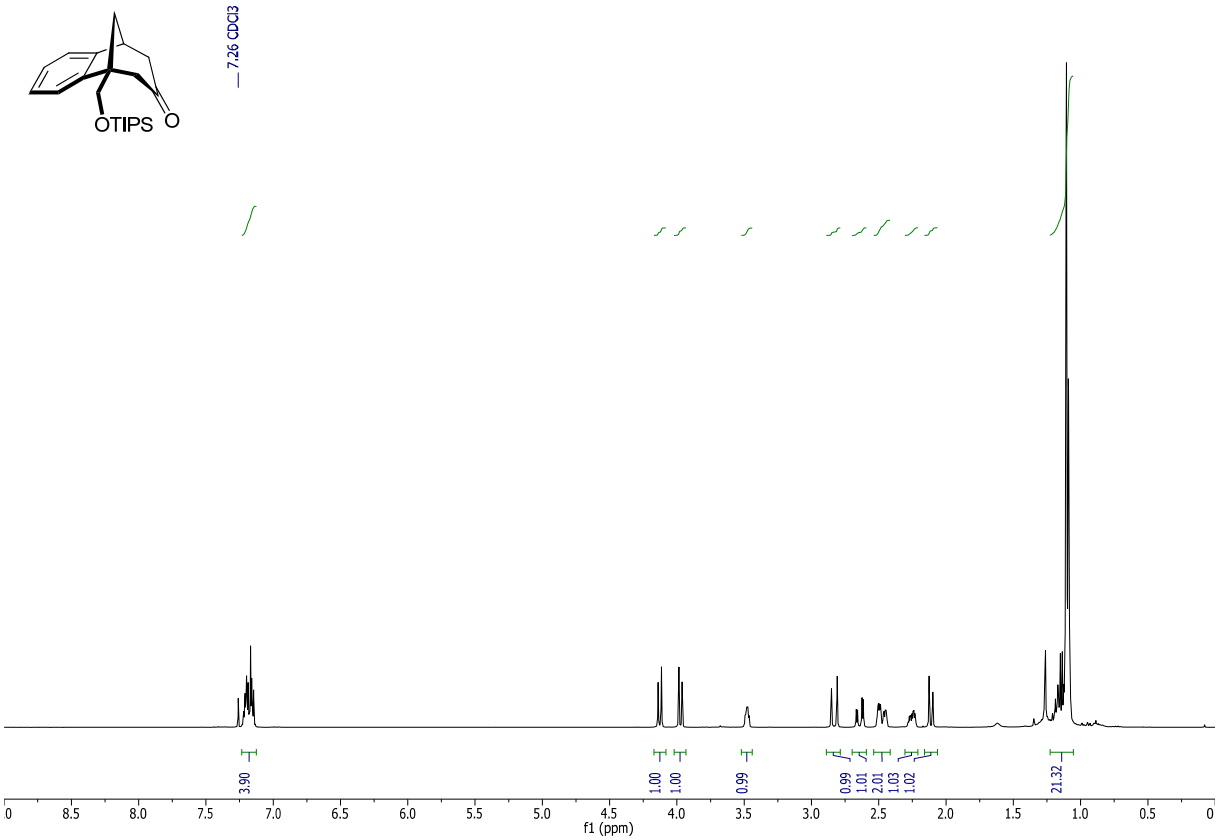

MeO
Me
O
210.0
159.4
149.9
137.7
123.8
112.2
107.6
55.8
55.4
49.6
47.6
45.1
38.5
22.6
f1 (ppm)

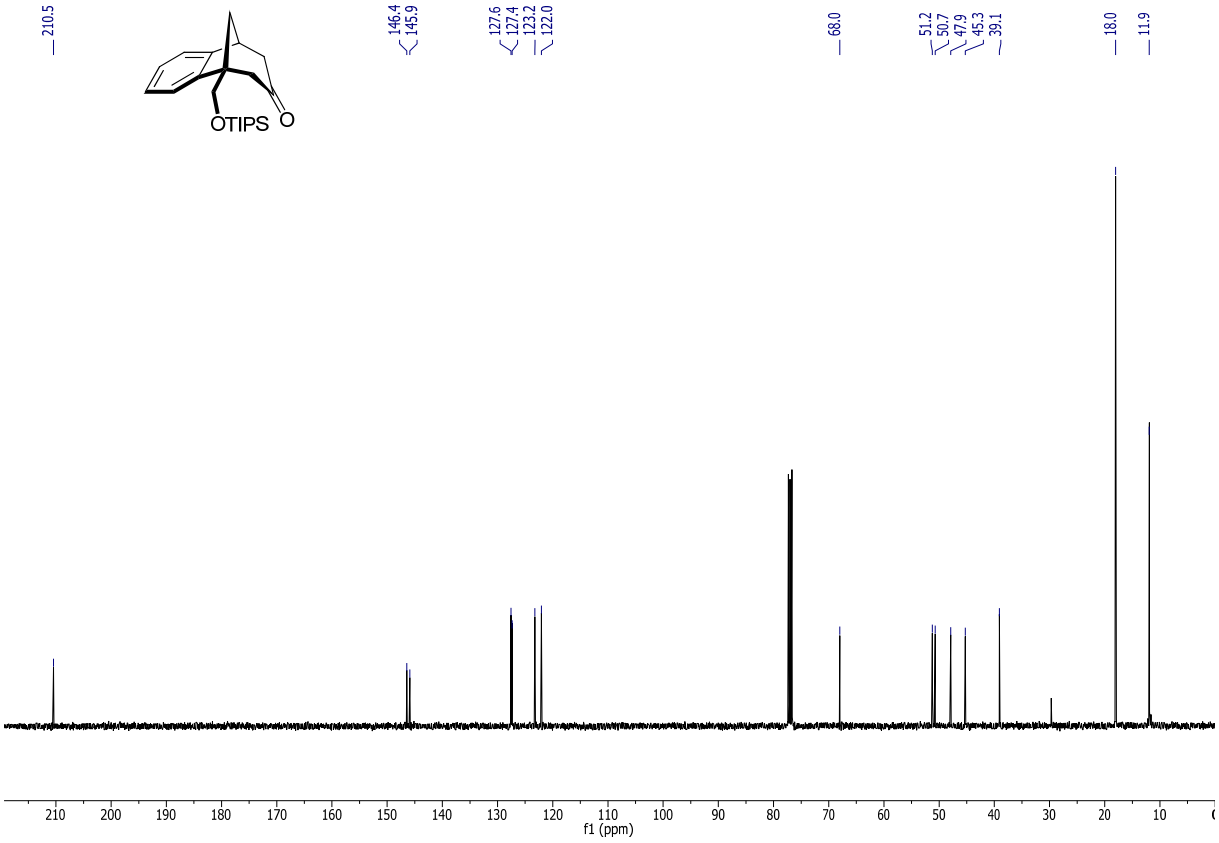

Et
O
7.26 CDCl3
2.93
0.96
1.00
2.00
1.05
2.02
1.06
1.95
2.99
f1 (ppm)

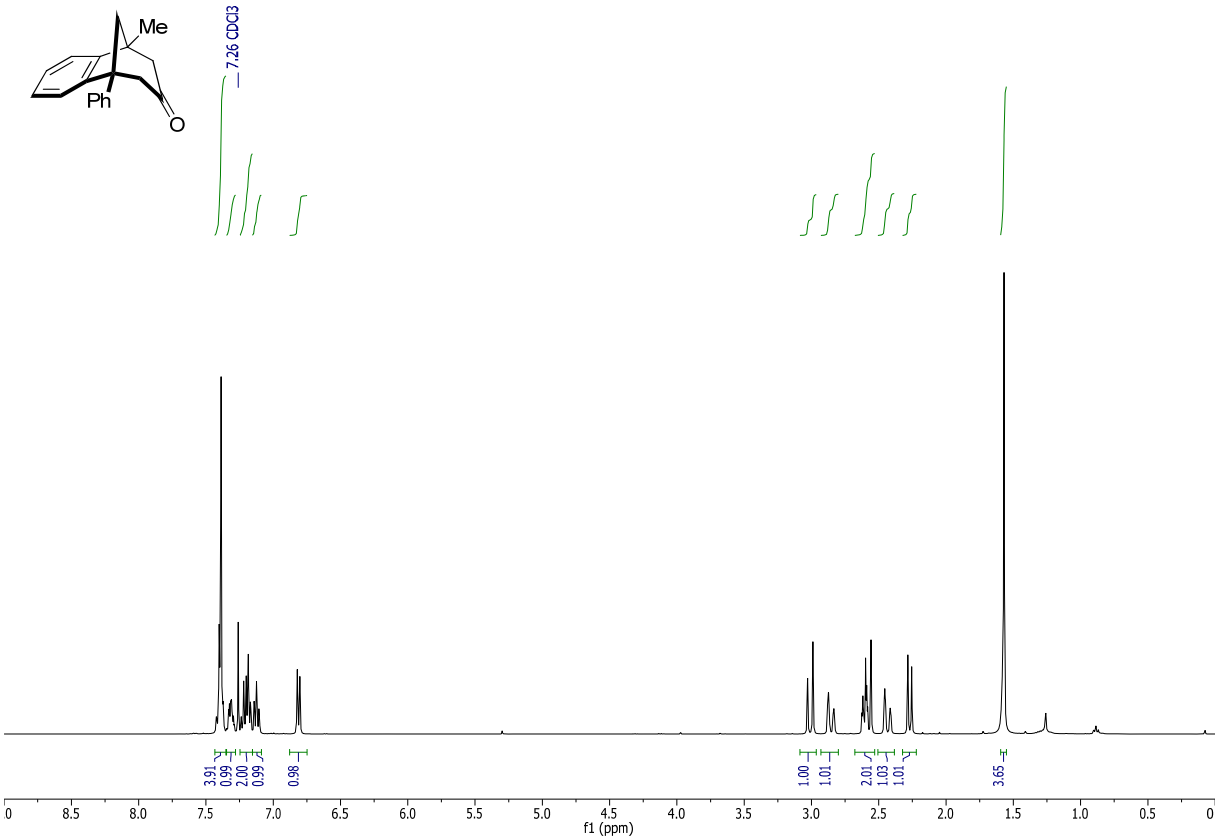

210.3
Et
O
147.3
146.1
127.4
127.2
123.2
121.7
54.0
48.9
47.9
45.1
39.1
28.2
8.7
f1 (ppm)

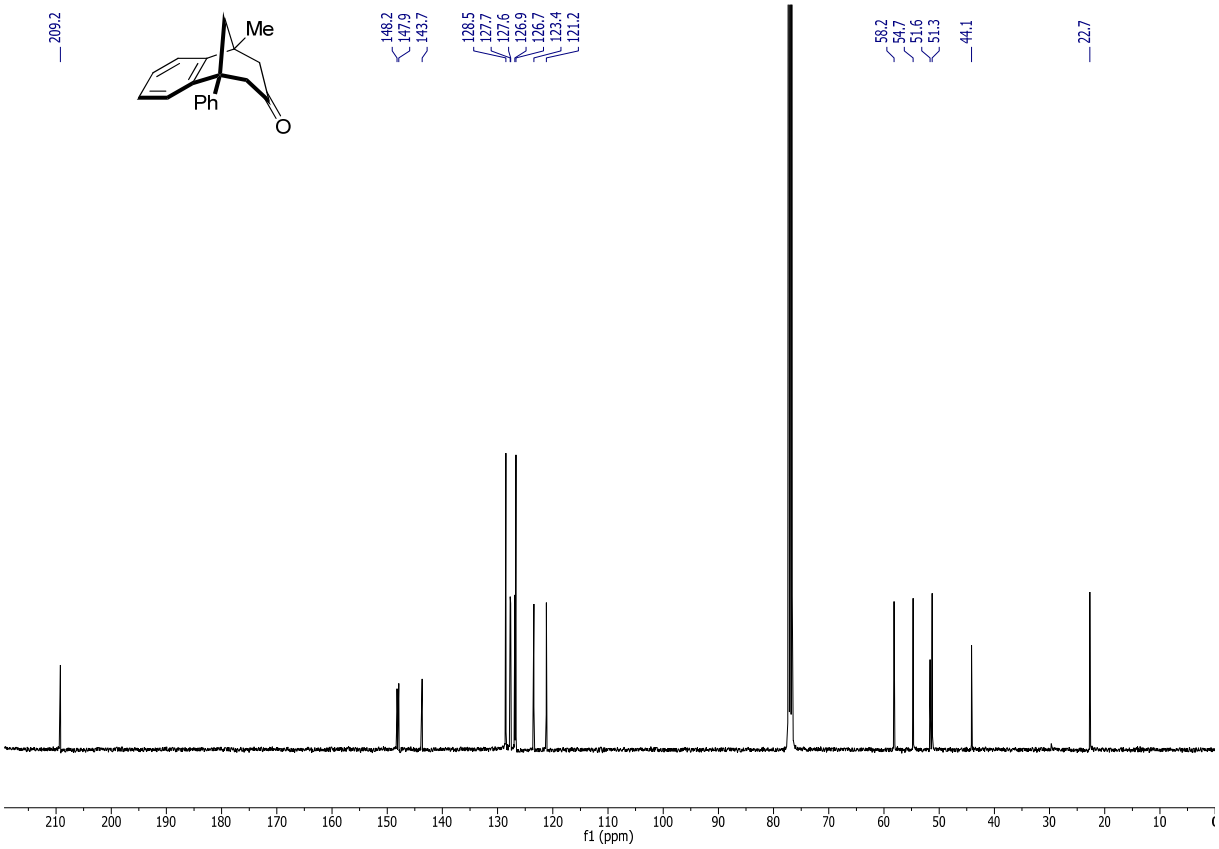

OBn
O
7.26 CDCl3
3.77
1.01
1.01
2.78
1.99
1.02
1.02
1.00
1.02
1.03
1.95
1.06
1.02
f1 (ppm)


209.8
OBn
O
146.1
145.7
138.2
128.4
127.7
127.6
127.5
127.4
123.3
122.0
74.2
73.5
51.4
49.3
48.0
45.2
39.1
f1 (ppm)


OTIPS
O
7.26 CDCl3
3.90
1.00
1.00
0.99
0.99
1.01
2.01
1.03
1.02
21.32
f1 (ppm)


OTIPS
O
210.5
146.4
145.9
127.6
127.4
123.2
122.0
68.0
51.2
50.7
47.9
45.3
39.1
18.0
11.9
f1 (ppm)


Me
Ph
O
7.26 CDCl3
3.91
0.99
2.00
0.99
0.98
1.00
1.01
2.01
1.03
1.01
3.65
f1 (ppm)


209.2
Me
Ph
O
148.2
147.9
143.7
128.5
127.7
127.6
126.9
126.7
123.4
121.2
58.2
54.7
51.6
51.3
44.1
22.7
f1 (ppm)