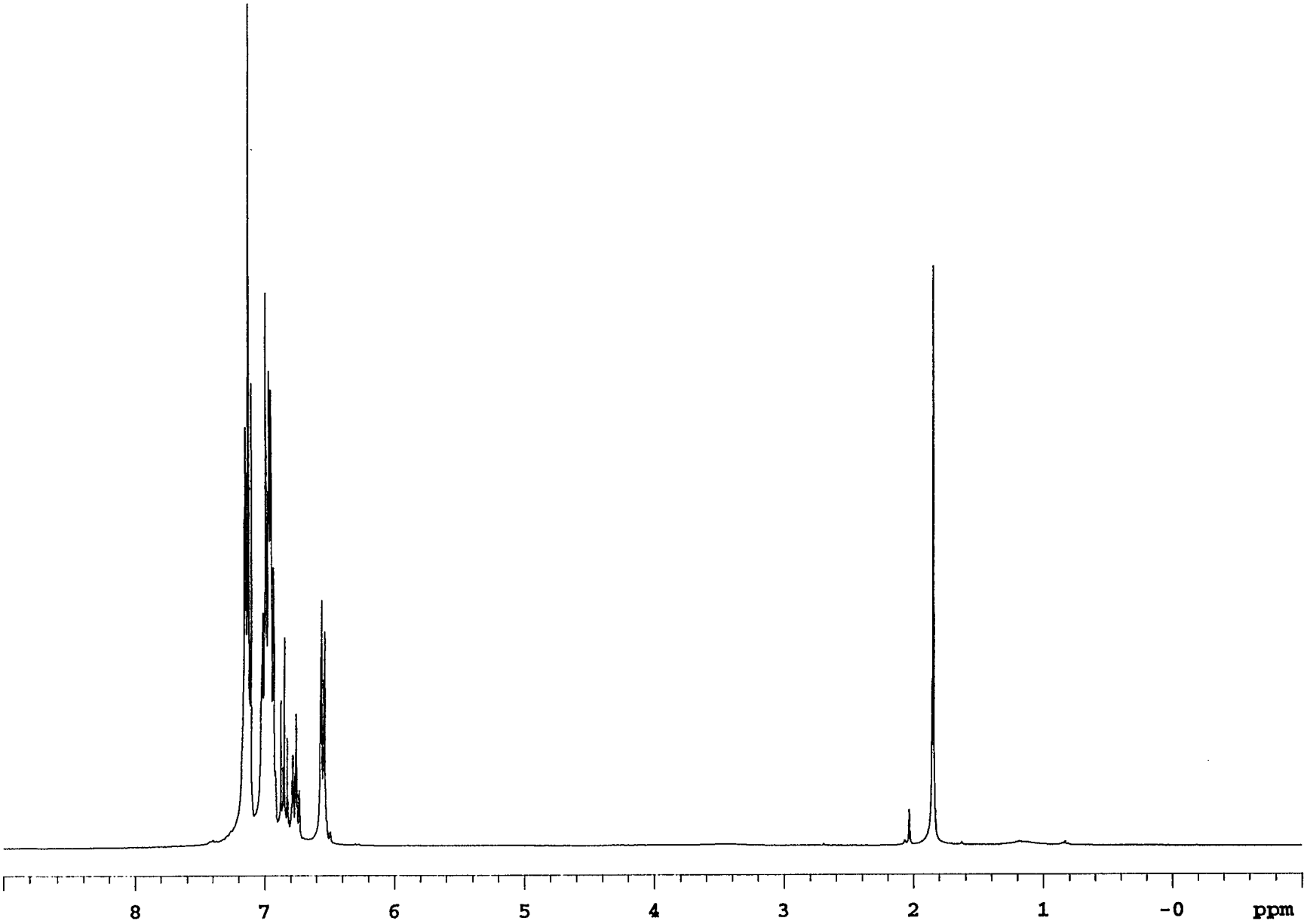
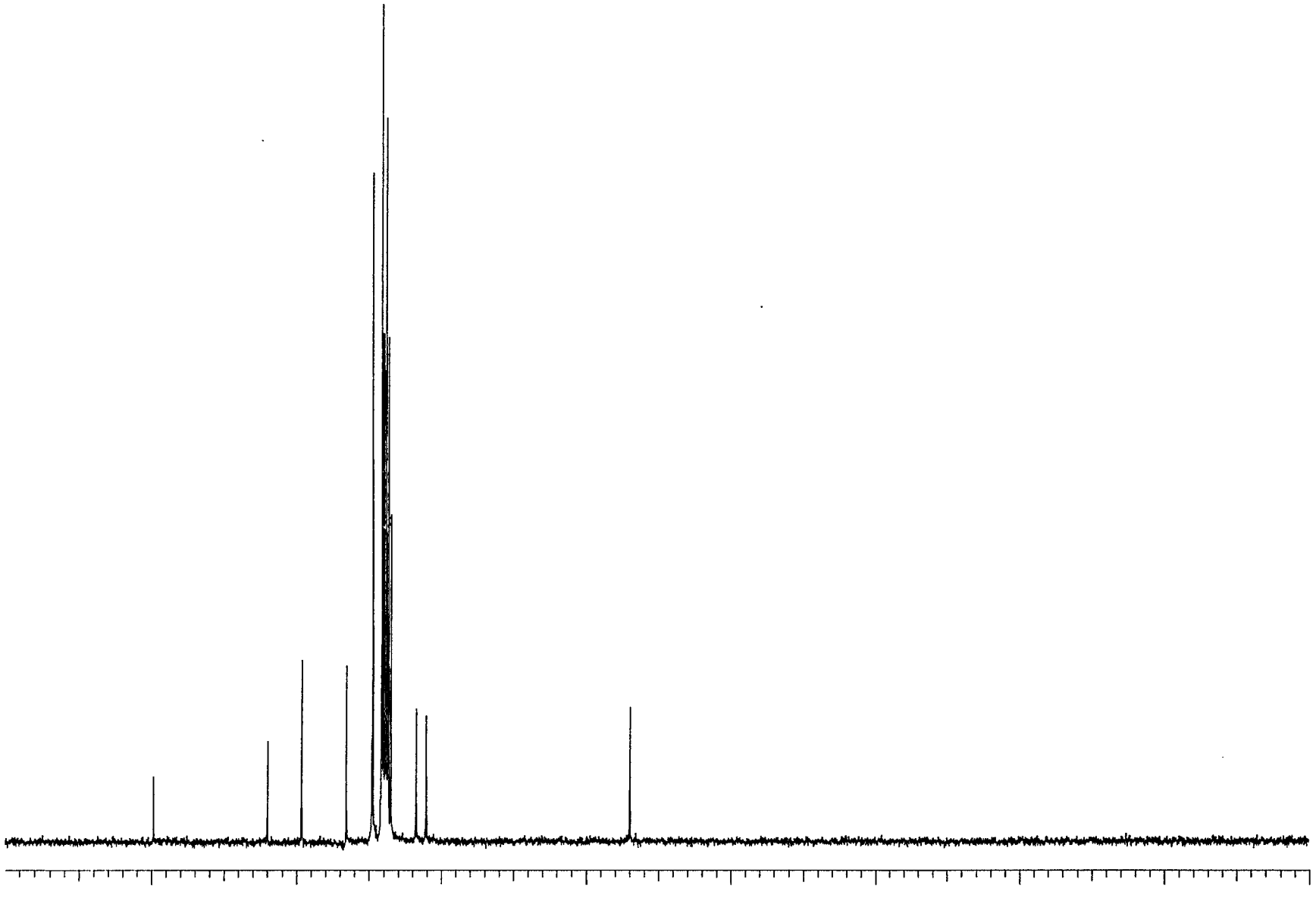
ORGANOMETALLICS
Organometallics, 1998, 17(17), 3636-3638, DOI:10.1021/om980287x

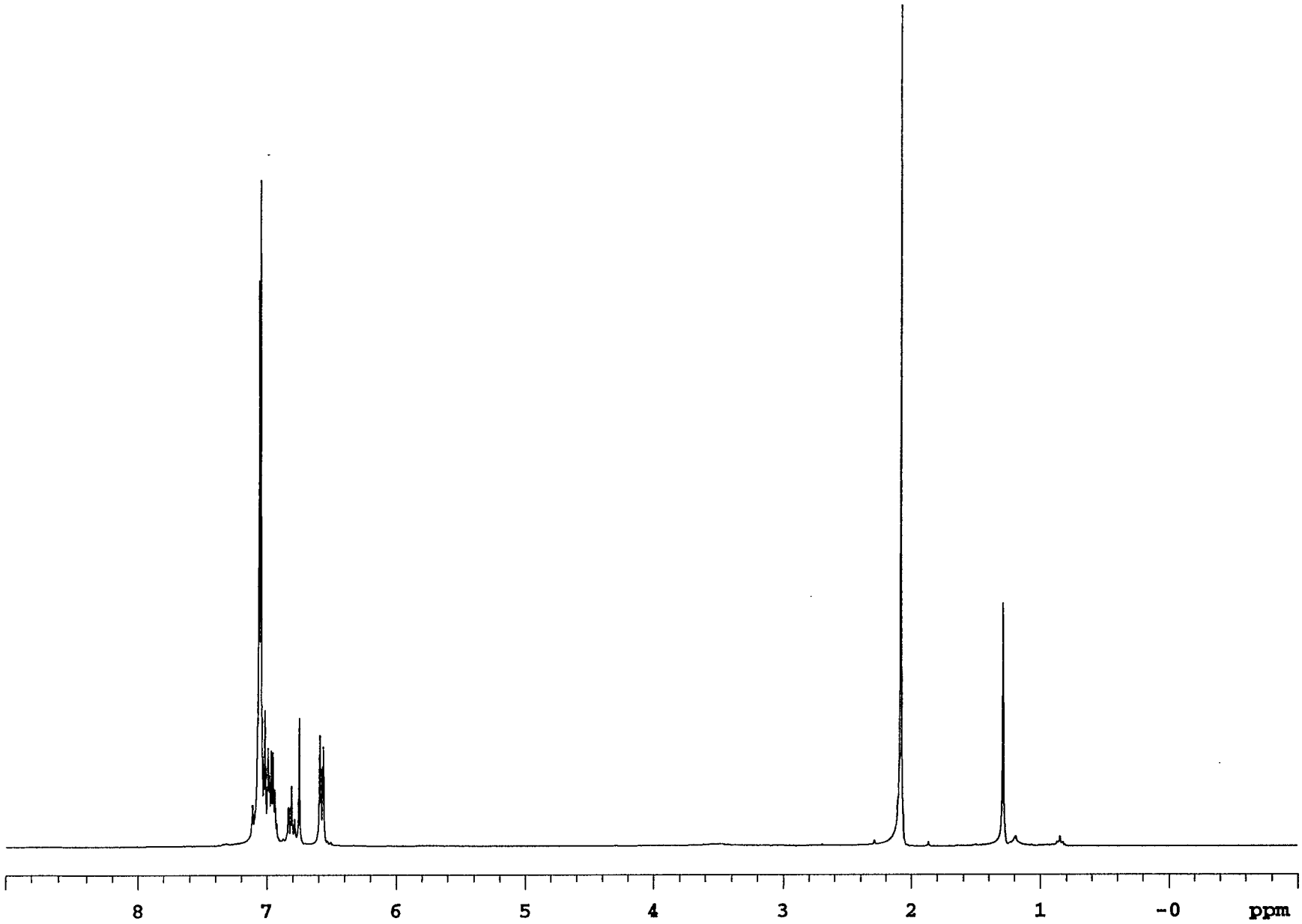


INDEX	FREQUENCY	PPM	HEIGHT
1	2148.406	7.162	59.0
2	2146.941	7.157	72.8
3	2140.105	7.135	146.1
4	2135.222	7.118	26.0
5	2132.536	7.109	80.4
6	2108.365	7.029	31.6
7	2106.656	7.023	40.5
8	2104.947	7.017	27.9
9	2100.063	7.001	96.3
10	2092.250	6.975	82.5
11	2088.344	6.962	76.3
12	2087.367	6.959	79.3
13	2081.752	6.940	48.4
14	2079.554	6.933	38.2
15	2076.380	6.922	12.6
16	2074.915	6.917	13.1
17	2062.464	6.876	25.2
18	2054.895	6.851	36.3
19	2047.326	6.825	18.7
20	2034.874	6.784	15.7
21	2027.549	6.759	22.8
22	2020.225	6.735	9.7
23	1968.464	6.562	42.7
24	1960.895	6.537	37.2
25	1947.466	6.492	2.6
26	609.006	2.030	6.2
27	553.827	1.846	100.8

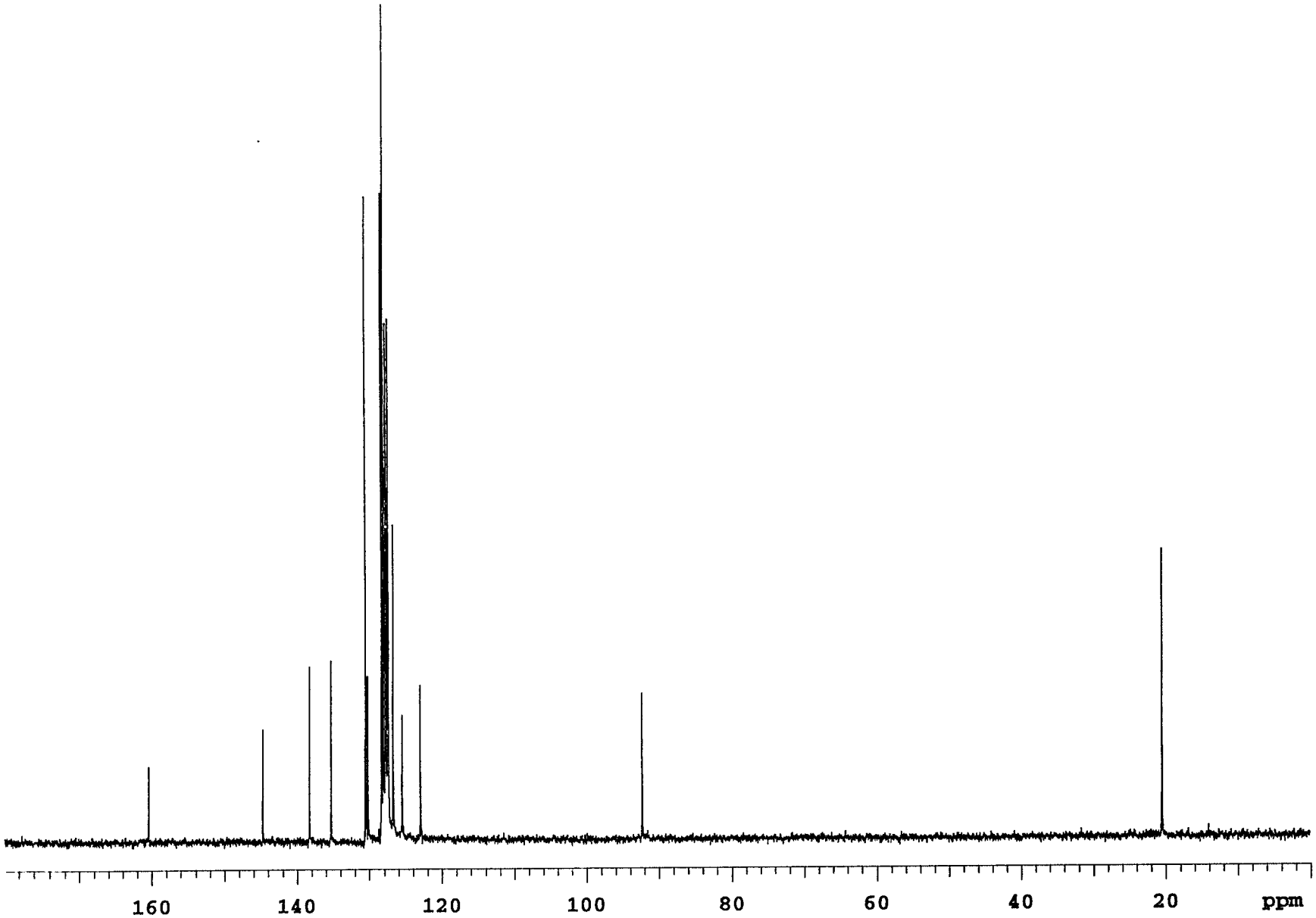
3a



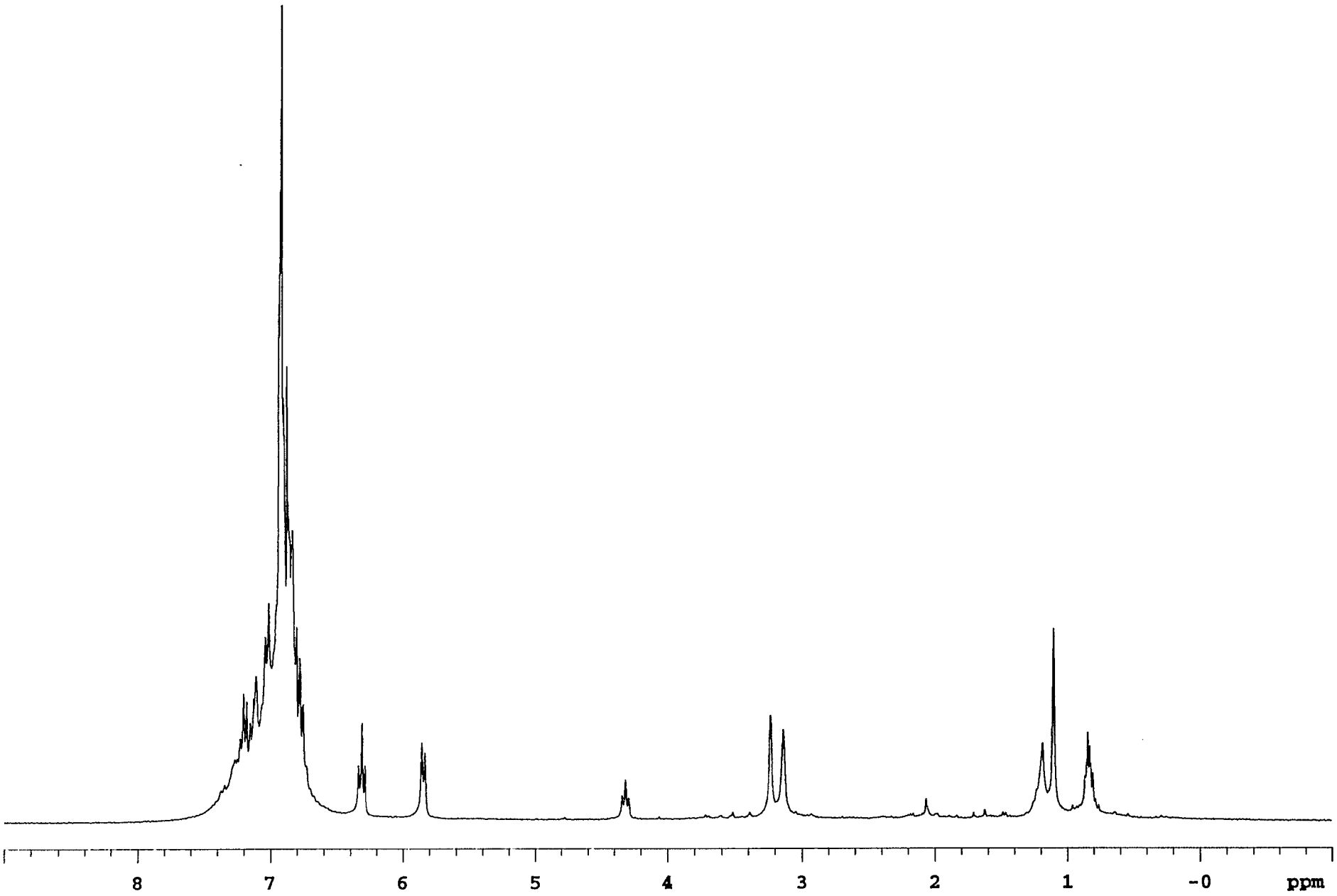
INDEX	FREQUENCY	PPM	HEIGHT
1	12051.165	159.777	11.1
2	10860.604	143.992	18.3
3	10509.077	139.331	31.4
4	10044.738	133.175	30.3
5	9772.783	129.570	117.4
6	9768.754	129.516	69.1
7	9679.109	128.328	144.0
8	9658.964	128.061	88.8
9	9635.798	127.753	124.9
10	9610.617	127.420	89.5
11	9583.421	127.059	59.4
12	9313.480	123.480	23.9
13	9211.749	122.131	22.7
14	7087.475	93.967	23.8



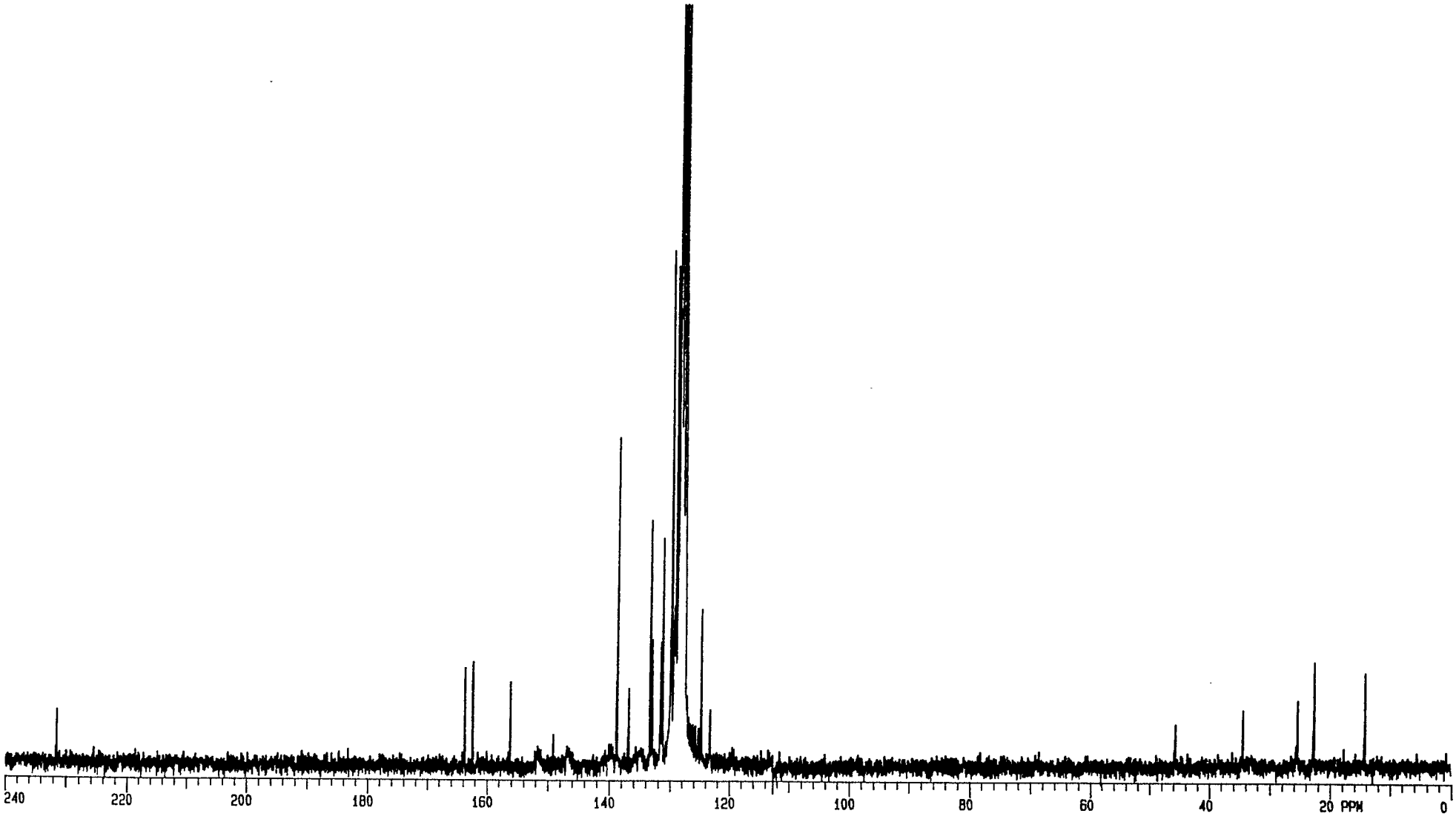
INDEX	FREQUENCY	PPM	HEIGHT
1	2133.757	7.113	7.1
2	2133.024	7.111	7.2
3	2120.084	7.068	98.5
4	2115.934	7.054	115.8
5	2104.947	7.017	23.5
6	2097.622	6.993	16.9
7	2094.692	6.983	12.3
8	2090.541	6.969	16.5
9	2086.147	6.955	16.2
10	2081.508	6.939	9.8
11	2077.357	6.925	4.1
12	2049.767	6.833	6.8
13	2042.687	6.810	10.4
14	2035.606	6.786	4.8
15	2024.619	6.750	22.1
16	1976.521	6.589	19.1
17	1968.708	6.563	17.2
18	623.655	2.079	146.1
19	385.359	1.285	42.1


160
140
120
100
80
60
40
20
ppm

INDEX	FREQUENCY	PPM	HEIGHT
1	12102.534	160.458	11.1
2	10917.010	144.740	17.5
3	10430.512	138.290	27.5
4	10204.890	135.299	30.0
5	9843.290	130.504	104.9
6	9819.116	130.184	26.3
7	9680.116	128.341	108.0
8	9657.957	128.047	144.0
9	9634.791	127.740	93.3
10	9621.696	127.566	59.5
11	9610.617	127.420	93.4
12	9560.255	126.752	50.2
13	9465.574	125.497	22.0
14	9278.227	123.013	26.5
15	6965.599	92.351	23.5
16	1546.635	20.506	49.4


8
7
6
5
4
3
2
1
-0
ppm

INDEX	FREQUENCY	PPM	HEIGHT	INDEX	FREQUENCY	PPM	HEIGHT
1	2211.643	7.373	5.8	34	942.034	3.141	16.8
2	2203.341	7.345	6.8	35	913.712	3.046	1.8
3	2179.902	7.267	11.5	36	879.774	2.933	1.4
4	2175.263	7.252	11.5	37	654.419	2.182	1.5
5	2168.671	7.230	15.1	38	648.803	2.163	1.5
6	2161.835	7.207	23.4	39	619.993	2.067	4.1
7	2154.266	7.182	21.9	40	594.112	1.981	1.5
8	2145.965	7.154	18.1	41	512.076	1.707	1.6
9	2138.884	7.131	22.6	42	486.928	1.623	2.0
10	2133.757	7.113	26.5	43	446.642	1.489	1.7
11	2113.736	7.047	33.8	44	440.294	1.468	1.6
12	2106.412	7.022	40.0	45	356.549	1.189	14.3
13	2081.996	6.941	95.3	46	331.889	1.106	35.1
14	2076.625	6.923	200.0	47	288.185	0.961	2.9
15	2071.986	6.908	78.0	48	278.907	0.930	2.7
16	2064.661	6.883	83.8	49	259.863	0.866	8.3
17	2059.045	6.864	52.4	50	253.759	0.846	16.2
18	2052.697	6.843	53.5	51	249.609	0.832	13.7
19	2042.443	6.809	35.6	52	246.923	0.823	10.6
20	2035.362	6.785	29.9	53	242.284	0.808	8.8
21	2034.141	6.781	29.1	54	235.936	0.787	4.0
22	2027.061	6.758	21.4	55	228.611	0.762	3.0
23	1901.321	6.339	10.2	56	192.232	0.641	1.7
24	1893.752	6.313	17.9	57	163.422	0.545	1.3
25	1886.183	6.288	10.2				
26	1757.025	5.858	14.4				
27	1749.944	5.834	12.5				
28	1302.652	4.343	4.8				
29	1295.327	4.318	7.6				
30	1287.758	4.293	4.3				
31	1055.566	3.519	1.7				
32	1017.478	3.392	1.7				
33	970.844	3.237	19.4				



INDEX	FREQUENCY	PPM	HEIGHT	INDEX	FREQUENCY	PPM	HEIGHT
1	18808.015	249.377	5.134		1714.598	22.734	9.9
2	18228.927	241.699	4.935		1703.154	22.582	-5.9
3	17431.631	231.127	11.036		1166.791	15.471	-7.6
4	12328.173	163.460	11.437		1058.450	14.034	9.2
5	12219.832	162.024	15.1				
6	11754.425	155.853	11.7				
7	11219.588	148.761	9.3				
8	11098.277	147.153	5.2				
9	10439.840	138.423	48.1				
10	10298.692	136.551	12.4				
11	10027.077	132.950	38.3				
12	10004.951	132.656	24.9				
13	9901.188	131.281	23.4				
14	9876.774	130.957	49.0				
15	9766.907	129.500	115.8				
16	9725.707	128.954	50.1				
17	9711.211	128.762	118.2				
18	9689.085	128.468	84.1				
19	9658.566	128.064	1688.6				
20	9634.151	127.740	1677.6				
21	9610.500	127.426	1630.3				
22	9594.477	127.214	74.6				
23	9379.322	124.361	29.4				
24	9267.929	122.884	17.0				
25	7542.108	100.001	-5.4				
26	4097.331	54.327	-6.3				
27	3593.012	47.640	-6.7				
28	3420.583	45.354	5.9				
29	3325.212	44.089	-7.3				
30	2386.005	31.636	8.1				
31	2139.568	28.369	-7.1				
32	1896.946	25.152	11.4				
33	1870.243	24.798	-7.3				

[Zr(OC$_6$HMe$_2$-3,5-Ph$_2$-2,6)$_2$(CH$_2$Ph)$_2$] (1a):

To a solution of [Zr(CH$_2$Ph)$_4$] (1.0g, mmol) in toluene (20 mL) was added 2,6-diphenyl-3,5-dimethylphenol (1.2g, mmol). The resulting mixture was stirred for 12 hrs before the solvent was removed in vacuo to yield the crude product. The crude product was recrystallized from a benzene solution layered with pentane. Yield 0.61g (34%). Anal. Calcd for $C_{54}H_{48}O_2Zr$: C, 79.08; H, 5.90. Found: C, 79.05; H, 6.09. ^{1}H NMR (C_6D_6, 30°C): δ 1.05 (s, Zr-C*H*$_2$); 2.13 (s, *meta*-C*H*$_3$); 6.78 (s, *para*-CH); 6.44 (d), 6.8-7.3 (m, aromatics). Selected ^{13}C NMR (C_6D_6, 30°C): δ 21.3 (*meta* *C*H$_3$); 68.7 (Zr-*C*H$_2$); 158.0 (Zr-O-*C*).

[Zr(OC$_6$H$_3$^{t}Bu$_2$-2,6)$_2$(CH$_2$Ph)$_2$] (1b)[5a]

[Zr(OC$_6$HMe$_2$-3,5-Ph$_2$-2,6)$_2$(CH$_2$Ph)][η^6-C$_6$H$_5$CH$_2$B(C$_6$F$_5$)$_3$] (2a):

A sample of [Zr(OC$_6$HMe$_2$-3,5-Ph$_2$-2,6)$_2$(CH$_2$Ph)$_2$] (**1**) (200 mg, 0.24 mmol) was placed in a solvent sealed flask along with one equivalent of B(C$_6$F$_5$)$_3$ (125mg, 0.24 mmol) and approximately 1 mL of benzene. The flask was left undisturbed for 12 hours and then the solution was evacuated to dryness. The resulting material was redissolved in a minimal amount of benzene and layered with hexane affording yellow crystals of [Zr(OC$_6$HMe$_2$-3,5-Ph$_2$-2,6)$_2$(CH$_2$Ph)][η^6-C$_6$H$_5$CH$_2$B(C$_6$F$_5$)$_3$] (**2**) in 79% yield (252 mg). ^{1}H NMR (C_6D_6, 30°C): δ 6.91 [t, ^{3}J(^{1}H-^{1}H) = 7.7 Hz, *meta-Ph*)]; 6.72 [t, ^{3}J(^{1}H-^{1}H) = 7.4 Hz, *para-Ph*)]; 6.34 [d, ^{3}J(^{1}H-^{1}H) = 7.2 Hz, *ortho-Ph*)]; 6.10 [d, ^{3}J(^{1}H-^{1}H) = 7.3 Hz, *ortho* Zr-η^6-C$_6$*H*$_5$]; 5.00 [t, ^{3}J(^{1}H-^{1}H) = 7.4 Hz, *meta* Zr-η^6-C$_6$*H*$_5$]; 4.71 [t, ^{3}J(^{1}H-^{1}H) = 7.3 Hz, *para* Zr-η^6-C$_6$*H*$_5$]; 2.97 [(br), BC*H*$_2$]; 1.88 (s, *meta*-C*H*$_3$); 0.83 (s, Zr-C*H*$_3$). Selected ^{13}C NMR (C_6D_6, 30°C): δ 163.0 (Zr-O-*C*); 70.6 (Zr-*C*H$_2$); 20.7 (*meta* *C*H$_3$).

[Zr(OC$_6$H$_3$^{t}Bu$_2$-2,6)$_2$(CH$_2$Ph)][η^6-C$_6$H$_5$CH$_2$B(C$_6$F$_5$)$_3$] (2b):

180 mg of [Zr(OC$_6$H$_3$^{t}Bu$_2$-2,6)$_2$(CH$_2$Ph)$_2$] (0.29 mmol) and 165 mg (0.32 mmol) of B(C$_6$F$_5$)$_3$ were dissolved in 2 mL of benzene in a solvent sealed flask forming a yellow solution. The solution was left undisturbed for 16 hours and then evacuated to dryness affording a yellow glassy solid. This solid was redissolved in fresh benzene and layered with pentane affording 260 mg (75%) of as yellow crystals. Anal. Cald for $ZrC_{60}H_{56}BF_{15}O_2$: C, 60.25; H, 4.72. Found: C, 55.47; H, 4.60. ^{1}H NMR (C_6D_6, 30°C): δ 7.05-7.20 (aromatics); 6.98 (d), 6.87 [d, ^{3}J(^{1}H-^{1}H) = 7.7 and 7.9 Hz, *ortho* Zr-η^6-C$_6$*H*$_5$]; 6.81 (t), 6.75 [t, ^{3}J(^{1}H-^{1}H) = 7.8 and 7.9 Hz, *meta*- Ti-O-*Ph*)]; 6.16 (t), 5.89 (t), 5.66 [t, ^{3}J(^{1}H-^{1}H) = 7.6, 7.6, and 7.3 Hz, *meta* and *para* Zr-η^6-C$_6$*H*$_5$]; 3.39 (br), 3.19 (br, B-C*H*$_2$); 1.98 (AB, 17.2 and 21.5 Hz); 1.23 (s), 1.18 (s, ^{t}*Bu*). Selected ^{13}C NMR (C_6D_6, 30°C): δ 163.9, 161.2 (Zr-O-*C*); 82.0 (Zr-*C*H$_2$); 43.9 (B-*C*H$_2$); 35.4, 35.3 [*C*-(CH$_3$)$_3$]; 32.3, 31.2 [C-(*C*H$_3$)$_3$].

[Ti(OC$_6$H$_3$Ph$_2$-2,6)$_2$(CH$_2$Ph)$_2$] (3a):

A sample of [Ti(OC$_6$H$_3$Ph$_2$-2.6)$_2$Cl$_2$] (1.00 g. 1.46 mmol) was dissolved in a benzene solution along with 1.3 equivalents of Mg(CH$_2$Ph)$_2$(thf)$_2$ (0.66 g. 1.89 mmol). The dark red solution was stirred for several hours and then evacuated to dryness. The resulting red solid was redissolved in benzene. filtered. and evacuated to dryness affording a red solid which was redissolved in a minimal amount of benzene and layered with hexane affording [Ti(OC$_6$H$_3$Ph$_2$-2.6)$_2$(CH$_2$Ph)$_2$] (**3a**) as a red powder in 89% yield (1.89 g). Anal. Calcd for C$_{50}$H$_{40}$O$_2$Ti: C. 83.32; H. 5.59: Found: C. 82.39; H. 5.44. ^{1}H NMR (C$_6$D$_6$. 30°C): δ 6.70-7.30 (aromatics): 6.63 (d. *ortho*-CH$_2$*Ph*): 1.91 (s. Ti-C*H*$_2$).

[Ti(OC$_6$HMe$_2$-3,5-Ph$_2$-2,6)$_2$(CH$_2$Ph)$_2$] (3b):

An identical procedure to that used in the synthesis of **3a** was used for **3b** starting with [Ti(OC$_6$HPh$_2$-2.6-Me$_2$-3,5)$_2$Cl$_2$] (1.00 g. 1.50 mmol). The synthesis afforded [Ti(OC$_6$HMe$_2$-3.5-Ph$_2$-2.6)$_2$(CH$_2$Ph)$_2$] (**3b**) as a red powder in 87% yield (1.04 g). Anal. Calcd for C$_{54}$H$_{48}$O$_2$Ti: C. 83.49; H. 6.23; Found: C. 83.15; H. 6.17. ^{1}H NMR (C$_6$D$_6$. 30°C): δ 6.80-7.50 (aromatics); 6.64 (d. *ortho*-CH$_2$*Ph*): 2.13 (s. *meta* C*H*$_3$): 1.34 (s. Ti-C*H*$_2$). Selected ^{13}C NMR (C$_6$D$_6$. 30°C): δ 160.8 (Ti-O-*C*); 92.7 (Ti-*C*H$_2$. ^{1}J(^{13}C-^{1}H) = 131 Hz): 20.8 (*meta* *C*H$_3$).

[Ti(OC$_6$H$_3$Ph$_2$-2,6)$_2$(CH$_2$Ph)][η^6-C$_6$H$_5$CH$_2$B(C$_6$F$_5$)$_3$] (4a):

A sample of [Ti(OC$_6$H$_3$Ph$_2$-2.6)$_2$(CH$_2$Ph)$_2$] (**3a**). (1.00 g. 1.39 mmol) was placed in a solvent sealed flask along with 1.3 equivalents of trispentafluorophenylboron (0.92 g. 1.80 mmol) and 5 mL of benzene. The reaction solution immediately turned red in color. The flask was left undisturbed for 12 hours and then the solution was evacuated to dryness. The resulting red solid was redissolved in minimal benzene and layered with hexane affording [Ti(OC$_6$H$_3$Ph$_2$-2.6)$_2$(CH$_2$Ph)][η^6-C$_6$H$_5$CH$_2$B(C$_6$F$_5$)$_3$] (**4a**) as dark red crystals in 63% yield (1.07 g). Anal Calcd for C$_{68}$H$_{40}$BF$_{15}$O$_2$Ti: C. 66.26: H. 3.27. Found: C. 66.32: H. 3.28. ^{1}H NMR (C$_6$D$_6$. 30°C): δ 6.75-7.30 (aromatics): 6.65 [d. ^{3}J(^{1}H-^{1}H) = 7.0 Hz. *ortho* Ti-CH$_2$*Ph*]: 6.07 [d. ^{3}J(^{1}H-^{1}H) = 6.7 Hz. *ortho* Ti-η^6-C$_6$*H*$_5$]: 4.78 [t. ^{3}J(^{1}H-^{1}H) = 7.5 Hz. *meta* Ti-η^6-C$_6$*H*$_5$]; 4.43 [t. ^{3}J(^{1}H-^{1}H) = 7.3 Hz. *para* Ti-η^6-C$_6$*H*$_5$]: 2.77 (br. B-C*H*$_2$): 2.12 (s. Ti-C*H*$_2$). Selected ^{13}C NMR (C$_6$D$_6$. 30°C): δ 161.9 (Ti-O-*C*): 151.0 (br. B-*C*H$_2$); 146.2 (*ipso* BCH$_2$-Ph): 101.2 (Ti*C*H$_2$).

[Ti(OC$_6$HMe$_2$-3,5-Ph$_2$-2,6)$_2$(CH$_2$Ph)][η^6-C$_6$H$_5$CH$_2$B(C$_6$F$_5$)$_3$] (4b).

An identical procedure to that used for the synthesis of **4a** was attempted for the synthesis of **4b** however only reddish oils could be isolated which were found to have broad ^{1}H NMR resonances with similar chemical shifts as that of **4a**. ^{1}H NMR (C$_6$D$_6$. 30°C): δ 6.60-7.40 (aromatics): 6.39 (d. *ortho* Ti-CH$_2$*Ph*): 5.85 (br. *ortho* Ti-η^6-C$_6$*H*$_5$): 4.75 (br t. *meta* Ti-η^6-C$_6$*H*$_5$): 3.49 (br. *para* Ti-η^6-C$_6$*H*$_5$): 2.93 (br. B-C*H*$_2$): 1.89 (s. Ti-

CH_2): 1.86 (s. *meta* CH_3). For subsequent reactions **4b** was generated in situ and found to undergo similar chemistry as solid **4a** thereby further confirming its synthesis here.

[Ti($OC_6H_3Ph_2$-2,6)$_2$(C{CH_3}C{Ph}CH_2{η^6-C_6H_5})][$PhCH_2B(C_6F_5)_3$] (5a):

A sample of [Ti($OC_6H_3Ph_2$-2.6)$_2$(CH_2Ph)][η^6-$C_6H_5CH_2B(C_6F_5)_3$] (**4a**) (130 mg. 0.105 mmol) was dissolved in 2 mL of benzene in a round bottomed flask. To this solution was added 1.0 equivalents of 1-phenylpropyne (13.2 μL. 0.105 mmol). The color of this solution slowly turned from red to orange over the course of an hour. This orange solution was evacuated to dryness affording [Ti($OC_6H_3Ph_2$-2.6)$_2$(C{CH_3}C{Ph}CH_2{η^6-C_6H_5})][$B(C_6F_5)_3(CH_2Ph)$] (**5a**) as a red glassy solid in 64% yield (90 mg). Anal Calcd for $C_{77}H_{48}BF_{15}O_2Ti$: C, 68.56; H. 3.59. Found: C, 65.59: H. 3.87. ^{1}H NMR (C_6D_6. 30°C): δ 6.60-7.40 (aromatics); 6.29 [t. $^3J(^1H\text{-}^1H)$ = 7.6 Hz. *meta* Ti-η^6-C_6H_5]: 5.82 [d. $^3J(^1H\text{-}^1H)$ = 7.4 Hz. *ortho* Ti-η^6-C_6H_5]: 4.29 [t. $^3J(^1H\text{-}^1H)$ = 7.4 Hz. *para* Ti-η^6-C_6H_5]: 3.22 (s. CH_2); 3.17 (s. B-CH_2): 1.87 (s. CH_3). Selected ^{13}C NMR (C_6D_6. 30°C): δ 231.6 (Ti-*C*{CH_3}); 163.8 (Ti-O-*C*); 45.7 (*C*H_2); 34.3 (Ti-C{*C*H_3}). Attempts to isolate **5a** as a crystalline solid have thus far been unsuccessful.

[Ti(OC_6HMe_2-3,5-Ph_2-2,6)$_2$(C{CH_3}C{Ph}CH_2{η^6-C_6H_5})][$PhCH_2B(C_6F_5)_3$] (5b):

A sample of [Ti(OC_6HPh_2-2.6-Me_2-3.5)$_2$(CH_2Ph)$_2$] (**3b**) was placed in a NMR tube along with $B(C_6F_5)_3$ and C_6D_6. After several minutes excess 1-phenylpropyne was added. ^{1}H NMR (C_6D_6. 30°C): δ 6.60-7.60 (aromatics): 6.53 [t. $^3J(^1H\text{-}^1H)$ = 8.0 Hz. *meta* Ti-η^6-C_6H_5]: 5.99 [d. $^3J(^1H\text{-}^1H)$ = 6.0 Hz. *ortho* Ti-η^6-C_6H_5]: 4.51 [t. $^3J(^1H\text{-}^1H)$ = 8.0 Hz. *para* Ti-η^6-C_6H_5]: 3.32 (br s. B-CH_2): 3.21 (s. CH_2); 1.88 (s. *meta* CH_3): 1.03 (s. CH_3). Attempts to isolate **5b** as a solid and not as an oil have thus far been unsuccessful.

[Ti($OC_6H_3Ph_2$-2,6)$_2$(CH_2CH{CH_2Ph}CH_2{η^6-C_6H_5})][$PhCH_2B(C_6F_5)_3$] (6a):

A sample of [Ti($OC_6H_3Ph_2$-2.6)$_2$(CH_2Ph)][$B(C_6F_5)_3(CH_2${η^6-C_6H_5})] (**4a**) (20mg. 0.016 mmol) was placed in a NMR tube along with deuterated benzene and 1.5 equivalents of allylbenzene (3.22 μL, 0.024 mmol) resulting in a red solution. ^{1}H NMR (C_6D_6. 30°C): δ 6.50-7.50 (aromatics): 6.16 (d). 6.08 [d. $^3J(^1H\text{-}^1H)$ = 7.5 and 7.6 Hz. *ortho* Ti-η^6-C_6H_5]: 5.43 (t). 5.27 (t). 5.12 [t. $^3J(^1H\text{-}^1H)$ = 6.9. 7.8. and 7.4 Hz. *meta* and *para* Ti-η^6-C_6H_5]: 3.28 (s. B-CH_2Ph): 1.40-2.60 (aliphatics): 0.26 (dd. Ti-CH_2). Selected ^{13}C NMR (C_6D_6. 30°C): δ 161.9. 161.6 (Ti-O-*C*): 58.3 (Ti-*C*H_2): 45.0 (Ti-CH_2*C*H): 40.5. 39.7 (*C*H_2Ph. *C*H_2{η^6-C_6H_5}): 32.7 (br. B-*C*H_2).Attempts to isolate **6a** as a solid and not as an oil have thus far been unsuccessful.

[Ti(OC_6HMe_2-3,5-Ph_2-2,6)$_2$(CH_2CH{CH_2Ph}CH_2{η^6-C_6H_5})][$B(C_6F_5)_3(CH_2Ph)$] (6b):

A sample of [Ti(OC_6HPh_2-2.6-Me_2-3.5)$_2$(CH_2Ph)$_2$] (**3b**) (200 mg. 0.026 mmol) and 1.3 equivalents of $B(C_6F_5)_3$ (171 mg. 0.33 mmol) were dissolved in benzene and after 10 minutes 1.1 equivalents of allylbenzene (37.5 mL. 0.28 mmol) were added to this

stirred red solution. The solution was stirred for an additional 30 minutes and then evacuated to dryness leaving (**6b**) as a yellow-orange solid. ^{1}H NMR (C_6D_6, 30°C): δ 6.65-7.40 (aromatics): 6.58 (d), 6.37 [d. $^3J(^1H\text{-}^1H)$ = 7.6 and 7.0 Hz. *ortho* Ti-η^6-C_6H_5]; 6.14 (t), 5.63 (t), 5.03 [t. $^3J(^1H\text{-}^1H)$ = 6.8, 7.7, and 6.9 Hz. *meta* and *para* Ti-η^6-C_6H_5]: 3.35 (s. B-CH_2Ph): 1.94 (s), 1.82 (s. *meta* CH_3): 1.40-2.50 (aliphatics): 0.00 (dd. Ti-CH_2). Selected ^{13}C NMR (C_6D_6, 30°C): δ 162.5, 161.6 (Ti-O-*C*); 58.4 (Ti-*C*H$_2$); 44.9 (Ti-CH$_2$*C*H): 40.2 (*C*H$_2$Ph, *C*H$_2${ η^6-C_6H_5}); 20.8, 20.6 (*meta* *C*H$_3$).

[Ti(OC$_6$H$_3$Ph$_2$-2,6)$_2$(CH$_2$CH{CH$_3$}CH$_2${η^6-Ph})][PhCH$_2$B(C$_6$F$_5$)$_3$] (7a):

A sample of **4a** was placed in a NMR tube along with C_6D_6 causing the formation of a red solution. Propene (1 atm) was added to the solution via a glass manifold causing the solution to become orange in color after a few minutes. Excess propene was then removed by evacuation of the solution to dryness leaving an orange oil. This oil was redissolved in C_6D_6. The ^{1}H NMR spectrum matches exactly with the ^{1}H NMR spectrum of **11a** except for the signal for the boron containing anion which was δ 3.28 for B-CH_2 for **7a**.

[Ti(OC$_6$H$_3$Ph$_2$-2,6)$_2$(CH$_2$CH{(CH$_2$)$_3$CH$_3$}CH$_2${η^6-C$_6$H$_5$})][PhCH$_2$B(C$_6$F$_5$)$_3$] (8a):

A sample of [Ti(OC$_6$H$_3$Ph$_2$-2.6)$_2$(CH$_2$Ph)][B(C$_6$F$_5$)$_3$(CH$_2${η^6-C$_6$H$_5$})] (**4a**) (40mg. 0.03 mmol) was placed in a NMR tube along with deuterated benzene and 2 equivalents of 1-hexene (8.1 μL. 0.06 mmol) resulting in an orange solution. ^{1}H NMR (C_6D_6, 30°C): δ 6.50-7.50 (aromatics): 6.37 (d), 6.30 [d. $^3J(^1H\text{-}^1H)$ = 7.9 and 7.8 Hz. *ortho* Ti-η^6-C_6H_5]: 5.63 (t), 5.16 (t), 4.86 [t. $^3J(^1H\text{-}^1H)$ = 7.6, 7.4, and 7.6 Hz. *meta* and *para* Ti-η^6-C_6H_5]: 3.22 (s. B-CH_2Ph): 0.20-1.50 (aliphatics). Selected ^{13}C NMR (C_6D_6, 30°C): δ 161.9, 161.4 (Ti-O-*C*): 58.7 (Ti-*C*H): 44.1 (*C*H$_2$CH$_2${η^6-C$_6$H$_5$}): 39.7 (Ti-CH*C*H$_2$): 36.8 (CH$_2$*C*H$_2${η^6-C$_6$H$_5$}): 29.8 (Ti-CHCH$_2$*C*H$_2$): 23.3 (Ti-CHCH$_2$CH$_2$*C*H$_2$): 14.3 (Ti-CHCH$_2$ CH$_2$CH$_2$*C*H$_3$).

[Ti(OC$_6$HMe$_2$-3,5-Ph$_2$-2,6)$_2$(CH$_2$CH{(CH$_2$)$_3$CH$_3$}CH$_2${η^6-Ph})][PhCH$_2$B(C$_6$F$_5$)$_3$] (8b):

A sample of [Ti(OC$_6$HPh$_2$-2.6-Me$_2$-3.5)$_2$(CH$_2$Ph)$_2$] (**3b**) (60 mg. 0.08 mmol) was placed in a NMR tube along with B(C$_6$F$_5$)$_3$ (50 mg. 0.10 mmol), deuterated benzene, and 2 equivalents of 1-hexene (19.3 μL. 0.15 mmol) resulting in a red solution. ^{1}H NMR (C_6D_6, 30°C): δ 6.95-7.60 (aromatics): 6.77(s), 6.73 (s. *para* OC$_6$*H*): 6.87 (d), 6.58 [d. $^3J(^1H\text{-}^1H)$ = 7.3 and 7.3 Hz. *ortho* Ti-η^6-C_6H_5]: 5.81 (t), 5.68 (t), 5.08 [t. $^3J(^1H\text{-}^1H)$ = 7.3, 7.3, and 7.0 Hz. *meta* and *para* Ti-η^6-C_6H_5]: 3.37 (s. B-CH_2Ph): 1.94 (s), 1.87 (s. *meta* CH_3): 0.00-2.50 (aliphatics). Evacuation of this solution to dryness afforded a yellow/orange glassy solid which only produced an oil from recrystallization attempts.

[Ti(OC$_6$H$_3$Ph$_2$-2,6)$_2$(CH$_3$)$_2$] (9a)[5]:

A procedure identical to the one used for **9b** was used to prepare **9a**. The amounts of reagents used was as follows: $TiCl_4$ (1 mL. 9.12 mmol); MeLi (26.1 mL. 36.5 mmol); 2.6-diphenylphenol (4.35 g. 17.1 mmol). $[Ti(OC_6H_3Ph_2\text{-}2.6)_2(CH_3)_2]$ (**9a**) was isolated as a greenish solid in 74% yield (3.84 g). Anal Cald for $TiC_{38}H_{32}O_2$: C. 80.28: H. 5.67. Found: C. 80.14: H. 5.69. ^1H NMR (C_6D_6. 30°C): δ 6.70-7.50 (aromatics): 0.60 (Ti-C*H*$_3$). Selected ^{13}C NMR (C_6D_6. 30°C): 66.1 (Ti-*C*H$_3$. $^1J(^{13}C\text{-}^1H)$ = 125 Hz).

$[Ti(OC_6HMe_2\text{-}3,5\text{-}Ph_2\text{-}2,6)_2(CH_3)_2]$ (9b):

A 1 L three neck flask fitted with a nitrogen adapter was charged with 400 mL of distilled anhydrous diethylether and 2 mL of $TiCl_4$ (18.2 mmol) and cooled to -78°C using dry ice/acetone. Once the desired temperature was achieved 4 equivalents of MeLi (1.4 M in Et_2O. 52.11 mL. 73 mmol) was slowly added to the stirred solution. The solution turned red upon MeLi addition. After 15 minutes of stirring the dark solution at -78°C 2 equivalents of 2.6-diphenyl-3.5-dimethylphenol (9.86 g. 36.0 mmol) were added with continued stirring. The dark solution was slowly allowed to warm to room temperature. The diethylether was removed in vacuo and the resulting dark residue was dissolved in benzene. filtered to remove LiCl. and evacuated to dryness. The resulting dark solid was recrystallized from a benzene/hexane mixture (30/70) affording $[Ti(OC_6HMe_2\text{-}3.5\text{-}Ph_2\text{-}2.6)_2(CH_3)_2]$ (**9b**) as a dark green powder in 50% yield (5.61 g). ^1H NMR (C_6D_6. 30°C): δ 6.90-7.40 (aromatics): 6.83 (s. *para*-OC_6H): 2.12 (s. *meta* CH_3): 0.24 (Ti-C*H*$_3$). Selected ^{13}C NMR (C_6D_6. 30°C): δ 160.7 (Ti-O-*C*): 64.7 (Ti-*C*H$_3$): 21.2 (*meta* C*H*$_3$).

$[Ti(OC_6HBu^t_2\text{-}3,5\text{-}Ph_2\text{-}2,6)_2(CH_3)_2]$ (9c):

A procedure identical to the one used for **9b** was used to prepare **9c**. The amounts of reagents used was as follows: $TiCl_4$ (1 mL. 9.12 mmol); MeLi (26.1 mL. 36.5 mmol); 3.5-di-*tert*-butyl-2.6-diphenylphenol (4.35 g. 17.1 mmol). $[Ti(OC_6HBu^t_2\text{-}3.5\text{-}Ph_2\text{-}2.6)_2(CH_3)_2]$ (**9c**) was isolated as a yellow solid in 27% yield (3.84 g). ^1H NMR (C_6D_6. 30°C): δ 7.69 (s. *para*-OC_6H): 6.90-7.40 (aromatics): 1.31 [s. $C(CH_3)_3$]: 0.06 (s.Ti-C*H*$_3$). Selected ^{13}C NMR (C_6D_6. 30°C): δ 162.4 (Ti-O-*C*): 64.9 (Ti-*C*H$_3$. $^1J(^{13}C\text{-}^1H)$ = 125.2 Hz): 37.5 [*C*$(CH_3)_3$]: 33.1 [C(*C*H$_3$)$_3$].

$[Ti(OC_6HBu^t_2\text{-}4,6\text{-}Ph\text{-}2)_2(CH_3)_2]$ (9d):

A procedure identical to the one used for **9b** was used to prepare **9d**. The amounts of reagents used was as follows: $TiCl_4$ (1 mL. 9.12 mmol); MeLi (26.1 mL. 36.5 mmol); 4.6-di-*tert*-butyl-2-phenylphenol (4.35 g. 17.1 mmol). $[Ti(OC_6HBu^t_2\text{-}4.6\text{-}Ph\text{-}2)_2(CH_3)_2]$ (**9d**) was isolated as a green solid in 47% yield (3.84 g). ^1H NMR (C_6D_6. 30°C): δ 7.59 (d). 7.33 (d. *meta-H*): 6.80-7.30 (aromatics): 1.71 [s. *ortho*-C(C*H*$_3$)$_3$]: 1.31 [s. *para*-C(C*H*$_3$)$_3$]: 1.10 (s.Ti-C*H*$_3$). Selected ^{13}C NMR (C_6D_6. 30°C): δ 160.3 (Ti-O-*C*): 67.6 (Ti-*C*H$_3$. $^1J(^{13}C\text{-}^1H)$ = 124.0 Hz): 35.9 [*ortho*-*C*$(CH_3)_3$]: 34.7 [*para*-C(*C*H$_3$)$_3$]: 31.8 [*ortho*-C(C*H*$_3$)$_3$]: 30.9 [*para*-*C*$(CH_3)_3$].

[Ti(OC$_6$H$_3$Ph$_2$-2,6)$_2$(CH$_3$)][CH$_3$B(C$_6$F$_5$)$_3$] (10a):

Equimolar amounts of **9a** and B(C$_6$F$_5$)$_3$ were placed in a NMR tube along with C$_6$D$_6$. Unlike **10b** no initial abstraction product was seen in the ^{1}H NMR but rather the decomposition products **12a** and **13** along with a small amount of [Ti(OC$_6$H$_3$Ph$_2$-2,6)$_3$(CH$_3$)]. ^{1}H NMR (C$_6$D$_6$, 30°C): δ 6.75-7.30 (aromatics); -0.36 [s, (ArO)$_3$TiC*H*$_3$].

[Ti(OC$_6$HMe$_2$-3,5-Ph$_2$-2,6)$_2$(CH$_3$)][CH$_3$B(C$_6$F$_5$)$_3$] (10b):

A sample of **9b** was placed in a NMR tube along with B(C$_6$F$_5$)$_3$ and C$_6$D$_6$ forming a red solution instantaneously. ^{1}H NMR (C$_6$D$_6$, 30°C): δ 6.80-7.30 (aromatics); 6.77 (s, *para-H*); 1.92 (s, *meta*-C*H*$_3$); 1.27 (br, B-C*H*$_3$); 0.01 (s, Ti-C*H*$_3$). Within an hour the decomposition products **12b** and **13** had formed along with a minor amount of [Ti(OC$_6$HMe$_2$-3,5-Ph$_2$-2,6)$_3$(CH$_3$)]. ^{1}H NMR(C$_6$D$_6$,30°C): δ 6.70-7.50 (aromatics); 1.90 (s, *meta* C*H*$_3$); -0.97 (s, Ti-C*H*$_3$).

[Ti(OC$_6$H$_3$Ph$_2$-2,6)$_2$(CH$_2$CH{CH$_3$}CH$_2${η^6-C$_6$H$_5$})][CH$_3$B(C$_6$F$_5$)$_3$] (11a):

A sample of **9a** (50 mg, 0.09 mmol) was placed in a NMR tube along with 2 equivalents of allylbenzene (24 μL, 0.18 mmol) and approximately 0.5 mL of C$_6$D$_6$. To this solution was added 60 mg of B(C$_6$F$_5$)$_3$ (0.11 mmol) dissolved in approximately 0.5 mL of C$_6$D$_6$ resulting in a dark red solution. ^{1}H NMR (C$_6$D$_6$, 30°C): δ 6.80-7.50 (aromatics); 6.49 (d), 6.33 [d, ^{3}J(^{1}H-^{1}H) = 8.0 and 7.8 Hz, *ortho* Ti-η^6-C$_6$*H*$_5$]; 5.79 (t), 5.24 (t), 4.79 [t, ^{3}J(^{1}H-^{1}H) = 7.7, 7.3, and7.5 Hz, *meta* and *para* Ti-η^6-C$_6$*H*$_5$]; 1.35 (m, C*H*$_2$Ph); 1.18 (m, C*H*CH$_3$); 0.98 (br, B-C*H*$_3$); 0.21 (m, Ti-CH$_2$); 0.21 [d, ^{3}J(^{1}H-^{1}H) = 5.9 Hz, CHC*H*$_3$). Selected ^{13}C NMR (C$_6$D$_6$, 30°C): δ 162.1, 161.3 (Ti-O-*C*); 70.4 (Ti-*C*H$_2$); 53.2 (Ti-CH$_2$*C*H); 33.2 (*C*H$_2$-η^6-C$_6$H$_5$); 24.5 (B-*C*H$_3$); 13.6 (CH*C*H$_3$).

[Ti(OC$_6$HMe$_2$-3,5-Ph$_2$-2,6)$_2$(CH$_2$CH{CH$_3$}CH$_2${η^6-C$_6$H$_5$})][CH$_3$B(C$_6$F$_5$)$_3$] (11b):

A sample of **9b** (50 mg, 0.08 mmol) was placed in a NMR tube along with 2 equivalents of allylbenzene (21 μL, 0.16 mmol) and approximately 0.5 mL of C$_6$D$_6$. To this solution was added 50 mg of B(C$_6$F$_5$)$_3$ (0.10 mmol) dissolved in approximately 0.5 mL of C$_6$D$_6$ resulting in a dark red solution. ^{1}H NMR (C$_6$D$_6$, 30°C): δ 6.75-7.40 (aromatics); 6.66 (d), 6.52 [d, ^{3}J(^{1}H-^{1}H) = 7.7 and 7.4 Hz, *ortho* Ti-η^6-C$_6$*H*$_5$]; 5.71 (t), 5.52 (t), 5.30 [t, ^{3}J(^{1}H-^{1}H) = 8.0, 6.7, and7.3 Hz, *meta* and *para* Ti-η^6-C$_6$*H*$_5$]; 1.93 (s), 1.89 (s, *meta* C*H*$_3$); 0.98 (br, B-C*H*$_3$); 0.00-2.10 (other aliphatics). Selected ^{13}C NMR (C$_6$D$_6$, 30°C): δ 162.1, 161.7 (Ti-O-*C*); 70.5 (Ti-*C*H$_2$); 53.1 (Ti-CH$_2$*C*H); 33.2 (*C*H$_2$-η^6-C$_6$H$_5$); 24.5 (B-*C*H$_3$); 24.0 (CH*C*H$_3$); 20.8, 20.5 (*meta* *C*H$_3$).

[Ti(OC$_6$H$_3$Ph$_2$-2,6)$_2$(CH$_3$)(C$_6$F$_5$)] (12a):

^{1}H NMR (C$_6$D$_6$, 30°C): δ 6.75-7.30 (aromatics); 0.75 (t, ^{5}J(^{19}F-^{1}H) = 1.5 Hz, Ti-C*H*$_3$).

[Ti(OC$_6$HMe$_2$-3,5-Ph$_2$-2,6)$_2$(CH$_3$)(C$_6$F$_5$)] (12b):

^{1}H NMR(C_6D_6,30°C): δ 6.70-7.50 (aromatics): 2.04 (s. *meta* CH_3): 0.52 (t. $^5J(^{19}F\text{-}^1H)$ = 1.5 Hz. Ti-CH_3).

$[CH_3B(C_6F_5)_2]$ (13):

^{1}H NMR(C_6D_6,30°C):δ 1.27 [p. $^5J(^{19}F\text{-}^1H)$ = 1.7 Hz. $CH_3B(C_6F_5)_2$].

Polymerization Experiments.

In the drybox a sample of **9** (0.089 mmol) was dissolved in 3 mL of toluene along with $B(C_6F_5)_3$ (0.097 mmol) in a solvent sealed flask containing a stir bar. The flask was quickly brought out of the drybox and chilled to 0°C using an ice-acetone bath. While the mixture was vigorously stirred one atmosphere of monomer was placed upon the contents of the flask for fifteen minutes. After this time methanol was added to quench the reaction and precipitate any polymer formed. The polymers which formed were washed with methanol and dried under vacuum affording either viscous colorless oils (polypropylene) or white solids (polyethylene).

$[Zr(OC_6HMe_2\text{-}3,5\text{-}Ph_2\text{-}2,6)_2(CH_2Ph)][\eta^6\text{-}C_6H_5CH_2B(C_6F_5)_3]$ (2a).

EXPERIMENTAL

DATA COLLECTION

A orange plate of $C_{84}H_{55}ZrBF_{15}O_2$ having approximate dimensions of 0.50 x 0.48 x 0.36 mm was mounted on a glass fiber in a random orientation. Preliminary examination and data collection were performed with Mo K_α radiation (λ = 0.71073 Å) on an Enraf-Nonius CAD4 computer controlled kappa axis diffractometer equipped with a graphite crystal, incident beam monochromator.

Cell constants and an orientation matrix for data collection were obtained from least-squares refinement, using the setting angles of 25 reflections in the range $16 < \theta < 19°$, measured by the computer controlled diagonal slit method of centering. The triclinic cell parameters and calculated volume are: a = 13.358(2), b = 14.722(4), c = 20.596(7)Å, α = 71.45(2), β = 77.55(2), γ = 72.18(2)°, V = 3624.1Å^3. For Z = 2 and F.W. = 1483.38 the calculated density is 1.36 g/cm^3. As a check on crystal quality, omega scans of several intense reflections were measured; the width at half-height was 0.56° with a take-off angle of 3.0° indicating moderate crystal quality. The space group was determined by the program ABSEN(ref 1). There were no_ systematic absences; the space group was determined to be P1(# 2).

The data were collected at a temperature of 203 ± 1 K using the ω-2θ scan technique. The scan rate varied from 3 to 16°/min (in omega). The variable scan rate allows rapid data collection for intense reflections where a fast scan rate is used and assures good counting statistics for weak reflections where a slow scan rate is used. Data were collected to a maximum 2θ of 45.3°. The scan range (in deg.) was determined as a function of θ to correct for the separation of the Kα doublet(ref 2); the scan width was calculated as follows:

$$\omega \text{ scan width} = 0.56 + 0.650 \tan\theta$$

Moving-crystal moving-counter background counts were made by scanning an additional 25% above and below this range. Thus the ratio of peak counting time to background counting time was 2:1. The counter aperture was also adjusted as a function of θ. The horizontal aperture width ranged from 1.4 to 1.8 mm; the vertical aperture was set at 4.0 mm. The diameter of the incident beam collimator was 0.7 mm and the crystal to detector distance was 21cm. For intense reflections an attenuator was automatically inserted in front of the detector; the attenuator factor was 13.2.

DATA REDUCTION

A total of 9916 reflections were collected, of which 9594 were unique. As a check on crystal and electronic stability 3 representative reflections were measured every 83 min. The intensities of these standards remained constant within experimental error throughout data collection. No decay correction was applied.

Lorentz and polarization corrections were applied to the data. The linear absorption coefficient is 2.3 /cm for Mo K radiation. An empirical absorption correction based on the method of Walker and Stuart (ref 3) was applied. Transmission coefficients ranged from 0.564 to 0.920 with an average value of 0.818. Intensities of equivalent reflections were averaged. The agreement factor for the averaging was 2.9% based on intensity.

STRUCTURE SOLUTION AND REFINEMENT

The structure was solved using the structure solution program PATTY in DIRDIF92 (ref 4). The remaining atoms were located in succeeding difference Fourier syntheses. Hydrogen atoms were included in the refinement but restrained to ride on the atom to which they are bonded. The structure was refined in full-matrix least-squares where the function minimized was $\Sigma w(|Fo|^2-|Fc|^2)^2$ and the weight w is defined as $w=1/[\sigma^2(Fo^2)+(0.1243P)^2+0.0000P]$ where $P=(Fo^2+2Fc^2)/3$

Scattering factors were taken from the "International Tables for Crystallography" (ref 5). 9594 reflections were used in the refinements. However, only reflections with $F_o^2>2\sigma(F_o^2)$ were used in calculating R. The final cycle of refinement included 841 variable parameters and converged (largest parameter shift was 0.01 times its esd) with unweighted and weighted agreement factors of:

$$R1 = \Sigma\ |Fo - Fc|\ /\ \Sigma\ Fo = 0.055$$

$$R2 = SQRT\ (\ \Sigma\ w\ (\ Fo^2 - Fc^2\ )^2\ /\ \Sigma\ w\ (Fo^2)^2\ ) = 0.160$$

The standard deviation of an observation of unit weight was 1.12. The highest peak in the final difference Fourier had a height of 0.51 e/A^3. The minimum negative peak had a height of -0.80 e/A^3.

Refinement was performed on a AlphaServer 2100 using SHELX-93 (ref 6). Crystallographic drawings were done using programs ORTEP (ref 7) and/or PLUTON (ref 8).

---

(1) P. C. McArdle, J. Appl. Cryst., 239, 306 (1996).
(2) "CAD4 Operations Manual", Enraf-Nonius, Delft, (1977)
(3) N. Walker and D. Stuart,Acta Crystallogr., A39, 158 (1983).
(4) P. T. Beurskens, G. Admirall, G. Beurskens, W. P. Bosman S. Garcia-Granda, R. O. Gould, J. M. M. Smits, and C. Smykalla, The DIRDIF92 Program System, Technical Report. Crystallography Laboratory, Univ. of Nijmegen, The Netherlands, (1992)
(5) "International Tables for Crystallography", Vol. C, Kluwer

Academic Publishers, Dordrecht, The Netherlands, (199)2, Tables 4.2.6.8 and 6.1.1.4
(6) G. M. Sheldrick, SHELXS93. A Program for Crystal Structure Refinement. Univ. of Gottingen, Germany, (1993)
(7) C. K. Johnson, ORTEPII, Report ORNL-5138, Oak Ridge National Laboratory, Tennessee, USA (1976)
(8) A. L Spek, PLUTON. Molecular Graphics Program. Univ. of Ultrecht, The Netherlands (1991)

CRYSTALLOGRAPHIC DATA FOR

$Zr(O\text{-}2,6Ph\text{-}3,5Me\text{-}Ph)_2(CH_2Ph)(C_6H_5B(C_6F_5)_3)\cdot 2C_6$

$ZrF_{15}O_2C_{84}BH_{55}$	formula weight 1483.38
a = 13.3579(17)Å	space group $P\bar{1}$ (No. 2)
b = 14.722(4)Å	T = 203. K
c = 20.596(7)Å	λ = 0.71073Å
α = 71.45(2)°	ρ_{calc} = 1.359g cm^{-3}
β = 77.548(19)°	μ = 0.231mm^{-1}
γ = 72.175(17)°	transmission coeff = 0.564-0.920
V = 3624.1(17)$Å^3$	$R(F_o)^a$ = 0.055
Z = 2	$R_w(F_o^2)^b$ = 0.160

---

[a] $R = \Sigma \, ||F_o| - |F_c|| / \Sigma \, |F_o|$ for $F_o^2 > 2\sigma(F_o^2)$

[b] $R_w = [\Sigma \, w \, (|F_o^2| - |F_c^2|)^2 / \Sigma \, w \, |F_o^2|^2]^{1/2}$

CRYSTAL DATA AND DATA COLLECTION PARAMETERS for

$Zr(O\text{-}2,6Ph\text{-}3,5Me\text{-}Ph)_2(CH_2Ph)(C_6H_5B(C_6F_5)_3)\cdot 2C_6$

formula	$ZrF_{15}O_2C_{84}BH_{55}$
formula weight	1483.38
space group	P1 (No. 2)
a, Å	13.3579(17)
b, Å	14.722(4)
c, Å	20.596(7)
α, deg	71.45(2)
β, deg	77.548(19)
γ, deg	72.175(17)
V, Å^3	3624.1(17)
Z	2
d_{calc}, g cm^{-3}	1.359
crystal dimensions, mm	0.50x0.48x0.36
temperature, K	203.
radiation (wavelength)	Mo K_α(0.71073Å)
monochromator	graphite
linear abs coef, mm^{-1}	0.231
absorption correction applied	empirical[a]
transmission factors: min, max	0.56, 0.92
diffractometer	Enraf-Nonius CAD4
scan method	ω-2θ
h, k, l range	-14 to 14 -15 to 15 0 to 22
2θ range, deg	5.14-45.34
scan width, deg	0.56 + 0.65tan(θ)
take-off angle, deg	3.00
programs used	SHELXL-93
F_{000}	1510.0
weighting	
$w=1/[\sigma^2(Fo^2)+(0.1243P)^2+0.0000P]$ where $P=(Fo^2+2Fc^2)/3$	
data collected	9916
unique data	9594
R_{int}	0.029
data used in refinement	9594
cutoff used in R-factor calculations	$F_o^2>2\sigma(F_o^2)$
number of variables	841
largest shift/esd in final cycle	0.01
$R(F_o)$	0.055
$R_w(F_o^2)$	0.160
goodness of fit	1.121

[a] Walker, N.; Stuart, D. Acta Crystallogr., Sect. A 1983, A39, 158.

Positional Parameters and Their Estimated Standard Deviations

for $Zr(O\text{-}2,6Ph\text{-}3,5Me\text{-}Ph)_2(CH_2Ph)(C_6H_5B(C_6F_5)_3)\cdot 2C_6$

Atom	x	y	z	U(Å^2)
Zr	0.14988(3)	0.03422(3)	0.20619(2)	0.03384(18)
F112	0.0575(2)	0.3581(2)	0.3713(2)	0.0601(13)
F113	-0.0099(3)	0.4093(3)	0.4885(2)	0.0877(17)
F114	0.1295(4)	0.3787(3)	0.5773(2)	0.112(2)
F115	0.3383(3)	0.2948(3)	0.5445(2)	0.0945(17)
F116	0.4077(2)	0.2417(2)	0.42893(14)	0.0590(12)
F122	0.4276(2)	0.3697(2)	0.29762(14)	0.0524(12)
F123	0.4379(2)	0.5444(2)	0.2104(2)	0.0760(15)
F124	0.2846(3)	0.6488(2)	0.1270(2)	0.0903(15)
F125	0.1209(3)	0.5712(2)	0.1332(2)	0.0787(13)
F126	0.1089(2)	0.3987(2)	0.21798(14)	0.0552(12)
F132	0.3447(2)	0.0830(2)	0.42904(14)	0.0599(12)
F133	0.5261(3)	-0.0560(2)	0.4225(2)	0.0844(15)
F134	0.6642(3)	-0.0465(3)	0.3034(2)	0.0987(17)
F135	0.6146(2)	0.1100(3)	0.1904(2)	0.0837(17)
F136	0.4412(2)	0.2527(2)	0.19712(13)	0.0535(12)
O2	0.1720(2)	-0.1053(2)	0.23400(15)	0.0418(12)
O3	0.0119(2)	0.1035(2)	0.23756(13)	0.0356(11)
C1	0.2315(3)	0.1491(3)	0.2578(2)	0.0345(15)
C2	0.2865(3)	0.0475(3)	0.2769(3)	0.0432(18)
C3	0.3492(3)	0.0005(4)	0.2274(3)	0.046(2)
C4	0.3492(4)	0.0484(4)	0.1576(3)	0.054(2)
C5	0.2876(4)	0.1446(4)	0.1383(3)	0.0441(18)
C6	0.2308(3)	0.1939(3)	0.1872(2)	0.0381(15)
C10	0.1857(3)	0.2039(3)	0.3111(2)	0.0371(13)
C21	0.1616(3)	-0.2009(3)	0.2622(2)	0.0382(15)
C22	0.2364(4)	-0.2703(3)	0.3029(2)	0.0445(17)
C23	0.2171(4)	-0.3643(4)	0.3362(2)	0.052(2)
C24	0.1298(4)	-0.3853(4)	0.3254(2)	0.055(2)
C25	0.0595(4)	-0.3189(4)	0.2807(2)	0.0500(19)
C26	0.0751(4)	-0.2242(3)	0.2479(2)	0.0412(17)
C31	-0.0920(3)	0.1394(3)	0.2610(2)	0.0356(15)
C32	-0.1325(3)	0.0930(4)	0.3282(2)	0.0419(17)
C33	-0.2404(4)	0.1274(4)	0.3507(2)	0.057(2)
C34	-0.3026(4)	0.2050(4)	0.3058(3)	0.058(2)
C35	-0.2625(3)	0.2509(4)	0.2398(2)	0.0495(18)
C36	-0.1539(3)	0.2181(3)	0.2161(2)	0.0410(15)
C40	0.1328(4)	0.0637(4)	0.0955(2)	0.0450(18)
C41	0.2256(4)	0.0175(4)	0.0508(2)	0.0460(18)
C42	0.2646(4)	-0.0865(4)	0.0648(3)	0.056(2)
C43	0.3504(5)	-0.1309(5)	0.0238(3)	0.073(2)
C44	0.4002(5)	-0.0710(5)	-0.0332(3)	0.080(3)
C45	0.3662(5)	0.0293(5)	-0.0471(3)	0.075(3)
C46	0.2774(4)	0.0763(4)	-0.0059(3)	0.061(2)

Positional Parameters and Their Estimated Standard Deviations (cont.)

for $Zr(O\text{-}2,6Ph\text{-}3,5Me\text{-}Ph)_2(CH_2Ph)(C_6H_5B(C_6F_5)_3)\cdot 2C_6$

Atom	x	y	z	U(Å^2)
C111	0.2357(3)	0.2921(3)	0.3937(2)	0.0378(15)
C112	0.1312(4)	0.3385(3)	0.4139(2)	0.0472(18)
C113	0.0940(4)	0.3669(4)	0.4729(3)	0.057(2)
C114	0.1643(6)	0.3521(5)	0.5180(3)	0.073(3)
C115	0.2681(5)	0.3094(4)	0.5021(3)	0.063(2)
C116	0.3014(4)	0.2816(4)	0.4413(2)	0.0476(18)
C121	0.2705(3)	0.3713(3)	0.2617(2)	0.0360(13)
C122	0.3488(3)	0.4163(3)	0.2564(2)	0.0426(18)
C123	0.3569(4)	0.5073(4)	0.2125(3)	0.054(2)
C124	0.2786(5)	0.5601(4)	0.1701(3)	0.060(2)
C125	0.1985(4)	0.5204(4)	0.1745(2)	0.0514(19)
C126	0.1943(4)	0.4293(3)	0.2190(2)	0.0431(18)
C131	0.3863(3)	0.1802(3)	0.3161(2)	0.0390(17)
C132	0.4134(4)	0.0968(4)	0.3690(2)	0.0474(18)
C133	0.5065(4)	0.0211(4)	0.3664(3)	0.064(2)
C134	0.5737(4)	0.0266(4)	0.3067(4)	0.065(3)
C135	0.5494(4)	0.1050(4)	0.2509(3)	0.055(2)
C136	0.4588(3)	0.1799(3)	0.2561(2)	0.0422(17)
C221	0.3352(4)	-0.2488(3)	0.3077(3)	0.056(2)
C222	0.3515(5)	-0.2274(4)	0.3643(3)	0.077(2)
C223	0.4473(7)	-0.2109(6)	0.3691(5)	0.100(4)
C224	0.5273(6)	-0.2209(5)	0.3167(6)	0.097(4)
C225	0.5162(5)	-0.2438(5)	0.2610(5)	0.095(3)
C226	0.4193(4)	-0.2567(4)	0.2550(4)	0.069(2)
C231	0.2924(5)	-0.4426(4)	0.3830(3)	0.074(3)
C251	-0.0328(4)	-0.3465(4)	0.2684(3)	0.064(2)
C261	0.0058(3)	-0.1512(3)	0.1970(2)	0.0402(18)
C262	0.0184(4)	-0.1611(4)	0.1307(2)	0.0505(18)
C263	-0.0448(4)	-0.0943(4)	0.0820(3)	0.058(2)
C264	-0.1212(4)	-0.0180(4)	0.0988(3)	0.063(2)
C265	-0.1359(4)	-0.0051(4)	0.1646(3)	0.054(2)
C266	-0.0728(3)	-0.0711(4)	0.2126(3)	0.0476(19)
C321	-0.0590(4)	0.0122(4)	0.3737(2)	0.0496(18)
C322	0.0186(4)	0.0339(5)	0.3947(3)	0.062(2)
C323	0.0911(5)	-0.0373(7)	0.4358(3)	0.104(3)
C324	0.0820(8)	-0.1330(9)	0.4554(5)	0.143(5)
C325	0.0063(10)	-0.1574(6)	0.4341(4)	0.128(5)
C326	-0.0662(6)	-0.0854(5)	0.3944(3)	0.085(3)
C331	-0.2902(4)	0.0830(6)	0.4232(3)	0.084(3)
C351	-0.3332(4)	0.3377(5)	0.1952(3)	0.075(2)
C361	-0.1041(3)	0.2638(3)	0.1467(2)	0.0426(17)
C362	-0.1281(4)	0.2545(4)	0.0870(3)	0.058(2)
C363	-0.0803(6)	0.2987(4)	0.0221(3)	0.077(3)
C364	-0.0116(5)	0.3521(5)	0.0163(3)	0.082(3)
C365	0.0119(5)	0.3644(5)	0.0746(4)	0.082(3)

Positional Parameters and Their Estimated Standard Deviations (cont.)

for $Zr(O\text{-}2,6Ph\text{-}3,5Me\text{-}Ph)_2(CH_2Ph)(C_6H_5B(C_6F_5)_3)\cdot 2C_6$

Atom	x	y	z	U(Å^2)
C366	-0.0344(4)	0.3209(4)	0.1396(3)	0.057(2)
B10	0.2703(4)	0.2633(4)	0.3196(3)	0.0368(18)
H2	0.282(4)	0.012(3)	0.332(3)	0.0560(18)*
H3	0.385(4)	-0.072(4)	0.226(3)	0.0600(18)*
H4	0.391(4)	0.021(4)	0.120(3)	0.0700(18)*
H5	0.284(4)	0.170(4)	0.093(3)	0.0570(18)*
H6	0.182(4)	0.254(3)	0.176(2)	0.0490(18)*

Starred atoms were refined isotropically

$U_{eq} = (1/3)\Sigma_i\Sigma_j\ U_{ij}a^*_i a^*_j \mathbf{a}_i\cdot\mathbf{a}_j$

Positional Parameters and Their Estimated Standard Deviations

for $Zr(O\text{-}2,6Ph\text{-}3,5Me\text{-}Ph)_2(CH_2Ph)(C_6H_5B(C_6F_5)_3)\cdot 2C_6$

Atom	x	y	z	U(Å²)
H24	0.1172(4)	-0.4468(4)	0.3491(2)	0.071
H34	-0.3743(4)	0.2270(4)	0.3208(3)	0.075
H10A	0.1695(3)	0.1579(3)	0.3550(2)	0.048
H10B	0.1200(3)	0.2519(3)	0.2983(2)	0.048
H222	0.2969(5)	-0.2238(4)	0.4008(3)	0.100
H223	0.4556(7)	-0.1937(6)	0.4069(5)	0.130
H224	0.5919(6)	-0.2116(5)	0.3195(6)	0.126
H225	0.5731(5)	-0.2512(5)	0.2262(5)	0.124
H226	0.4113(4)	-0.2706(4)	0.2157(4)	0.090
H23A	0.2706(18)	-0.5032(9)	0.3980(16)	0.097
H23B	0.292(2)	-0.4210(13)	0.4224(10)	0.097
H23C	0.3628(8)	-0.454(2)	0.3583(7)	0.097
H25A	-0.0260(15)	-0.344(3)	0.2205(5)	0.083
H25B	-0.0976(5)	-0.3007(16)	0.2804(17)	0.083
H25C	-0.0337(17)	-0.4123(10)	0.2964(14)	0.083
H262	0.0706(4)	-0.2138(4)	0.1186(2)	0.065
H263	-0.0346(4)	-0.1020(4)	0.0377(3)	0.075
H264	-0.1643(4)	0.0263(4)	0.0663(3)	0.081
H265	-0.1883(4)	0.0479(4)	0.1760(3)	0.070
H266	-0.0828(3)	-0.0622(4)	0.2565(3)	0.062
H322	0.0233(4)	0.0992(5)	0.3809(3)	0.081
H323	0.1438(5)	-0.0209(7)	0.4495(3)	0.136
H324	0.1285(8)	-0.1823(9)	0.4838(5)	0.186
H325	0.0036(10)	-0.2232(6)	0.4465(4)	0.167
H326	-0.1195(6)	-0.1019(5)	0.3815(3)	0.110
H33A	-0.278(3)	0.0129(8)	0.4308(8)	0.109
H33B	-0.3651(7)	0.113(2)	0.4281(7)	0.109
H33C	-0.259(2)	0.095(3)	0.4565(3)	0.109
H35A	-0.361(2)	0.3149(6)	0.1656(14)	0.097
H35B	-0.2931(9)	0.3841(14)	0.1675(15)	0.097
H35C	-0.3909(18)	0.3695(18)	0.2239(3)	0.097
H362	-0.1763(4)	0.2186(4)	0.0904(3)	0.076
H363	-0.0962(6)	0.2912(4)	-0.0174(3)	0.099
H364	0.0203(5)	0.3807(5)	-0.0271(3)	0.106
H365	0.0588(5)	0.4021(5)	0.0702(4)	0.107
H366	-0.0187(4)	0.3299(4)	0.1787(3)	0.074
H40A	0.0725(4)	0.0413(4)	0.0938(2)	0.059
H40B	0.1159(4)	0.1349(4)	0.0752(2)	0.059

Hydrogens included in calculation of structure factors but not refined
$B_{iso}(H)=1.3*B_{iso}(C)$

Anisotropic Temperature Factor Coefficients - U's

for $Zr(O\text{-}2,6Ph\text{-}3,5Me\text{-}Ph)_2(CH_2Ph)(C_6H_5B(C_6F_5)_3)\cdot 2C_6$

Name	U(1,1)	U(2,2)	U(3,3)	U(1,2)	U(1,3)	U(2,3)
Zr	0.0328(3)	0.0340(3)	0.0354(3)	-0.0093(2)	-0.0050(2)	-0.0093(2)
F112	0.044(2)	0.067(2)	0.074(2)	-0.0055(14)	-0.0058(14)	-0.035(2)
F113	0.073(2)	0.093(3)	0.094(3)	-0.013(2)	0.024(2)	-0.052(2)
F114	0.141(4)	0.143(4)	0.054(2)	-0.024(3)	0.008(2)	-0.056(2)
F115	0.125(3)	0.109(3)	0.063(2)	-0.020(2)	-0.040(2)	-0.033(2)
F116	0.056(2)	0.068(2)	0.060(2)	-0.0101(14)	-0.0232(14)	-0.0222(15)
F122	0.0433(15)	0.049(2)	0.071(2)	-0.0177(12)	-0.0080(14)	-0.0185(14)
F123	0.072(2)	0.053(2)	0.105(3)	-0.036(2)	0.013(2)	-0.021(2)
F124	0.114(3)	0.040(2)	0.087(2)	-0.022(2)	0.007(2)	0.012(2)
F125	0.101(2)	0.055(2)	0.064(2)	0.002(2)	-0.034(2)	0.000(2)
F126	0.055(2)	0.045(2)	0.068(2)	-0.0057(13)	-0.0287(14)	-0.0120(14)
F132	0.072(2)	0.047(2)	0.054(2)	-0.0166(14)	-0.0164(15)	0.0019(13)
F133	0.088(2)	0.050(2)	0.106(3)	0.002(2)	-0.053(2)	-0.001(2)
F134	0.053(2)	0.072(2)	0.162(4)	0.022(2)	-0.032(2)	-0.044(2)
F135	0.049(2)	0.099(3)	0.100(3)	-0.006(2)	0.011(2)	-0.049(2)
F136	0.049(2)	0.061(2)	0.047(2)	-0.0151(13)	0.0044(12)	-0.0160(14)
O2	0.047(2)	0.036(2)	0.045(2)	-0.0133(14)	-0.0122(14)	-0.0080(14)
O3	0.040(2)	0.037(2)	0.0296(15)	-0.0093(13)	-0.0010(13)	-0.0116(13)
C1	0.030(2)	0.033(2)	0.043(3)	-0.010(2)	-0.009(2)	-0.009(2)
C2	0.041(3)	0.040(3)	0.058(3)	-0.010(2)	-0.021(2)	-0.017(2)
C3	0.033(2)	0.040(3)	0.077(4)	-0.007(2)	-0.015(2)	-0.028(3)
C4	0.032(3)	0.067(4)	0.074(4)	-0.021(3)	0.004(2)	-0.034(3)
C5	0.042(3)	0.057(3)	0.042(3)	-0.027(2)	0.000(2)	-0.015(2)
C6	0.039(2)	0.034(2)	0.046(3)	-0.014(2)	-0.008(2)	-0.011(2)
C10	0.037(2)	0.037(2)	0.039(2)	-0.013(2)	-0.004(2)	-0.010(2)
C21	0.051(3)	0.029(2)	0.035(2)	-0.012(2)	-0.005(2)	-0.008(2)
C22	0.056(3)	0.033(2)	0.047(3)	-0.011(2)	-0.009(2)	-0.013(2)
C23	0.076(4)	0.041(3)	0.040(3)	-0.012(3)	-0.011(2)	-0.011(2)
C24	0.088(4)	0.042(3)	0.041(3)	-0.029(3)	-0.006(3)	-0.010(2)
C25	0.065(3)	0.050(3)	0.042(3)	-0.028(3)	0.006(2)	-0.018(2)
C26	0.050(3)	0.045(3)	0.038(2)	-0.019(2)	-0.002(2)	-0.020(2)
C31	0.029(2)	0.044(3)	0.037(2)	-0.012(2)	-0.001(2)	-0.015(2)
C32	0.043(3)	0.055(3)	0.036(2)	-0.022(2)	-0.003(2)	-0.016(2)
C33	0.043(3)	0.100(4)	0.033(3)	-0.032(3)	0.005(2)	-0.020(3)
C34	0.027(2)	0.095(4)	0.051(3)	-0.013(3)	0.002(2)	-0.027(3)
C35	0.031(2)	0.062(3)	0.053(3)	-0.005(2)	-0.004(2)	-0.020(3)
C36	0.038(2)	0.051(3)	0.039(2)	-0.014(2)	-0.009(2)	-0.014(2)
C40	0.047(3)	0.049(3)	0.042(3)	-0.013(2)	-0.007(2)	-0.015(2)
C41	0.049(3)	0.062(3)	0.037(3)	-0.025(2)	-0.002(2)	-0.019(2)
C42	0.052(3)	0.066(4)	0.060(3)	-0.013(3)	-0.004(3)	-0.036(3)
C43	0.063(4)	0.081(4)	0.076(4)	-0.021(3)	0.012(3)	-0.036(3)
C44	0.069(4)	0.090(5)	0.082(4)	-0.020(4)	0.014(3)	-0.040(4)
C45	0.072(4)	0.101(5)	0.062(4)	-0.040(4)	0.014(3)	-0.033(4)
C46	0.054(3)	0.088(4)	0.049(3)	-0.028(3)	0.000(3)	-0.023(3)

Anisotropic Temperature Factor Coefficients - U's (Continued)

for $Zr(O\text{-}2,6Ph\text{-}3,5Me\text{-}Ph)_2(CH_2Ph)(C_6H_5B(C_6F_5)_3)\cdot 2C_6$

Name	U(1,1)	U(2,2)	U(3,3)	U(1,2)	U(1,3)	U(2,3)
C111	0.042(3)	0.031(2)	0.036(2)	-0.011(2)	-0.003(2)	-0.003(2)
C112	0.051(3)	0.046(3)	0.043(3)	-0.012(2)	-0.002(2)	-0.013(2)
C113	0.060(3)	0.055(3)	0.053(3)	-0.017(3)	0.011(3)	-0.019(3)
C114	0.097(5)	0.080(4)	0.041(3)	-0.026(4)	0.010(3)	-0.026(3)
C115	0.092(4)	0.069(4)	0.033(3)	-0.027(3)	-0.018(3)	-0.007(3)
C116	0.052(3)	0.047(3)	0.045(3)	-0.015(2)	-0.012(2)	-0.008(2)
C121	0.039(2)	0.034(2)	0.035(2)	-0.008(2)	-0.003(2)	-0.012(2)
C122	0.042(3)	0.044(3)	0.041(3)	-0.007(2)	-0.003(2)	-0.016(2)
C123	0.059(3)	0.039(3)	0.064(3)	-0.024(3)	0.021(3)	-0.022(3)
C124	0.081(4)	0.036(3)	0.046(3)	-0.005(3)	0.005(3)	-0.006(2)
C125	0.064(3)	0.041(3)	0.042(3)	-0.003(3)	-0.014(3)	-0.007(2)
C126	0.049(3)	0.041(3)	0.042(3)	-0.011(2)	-0.008(2)	-0.014(2)
C131	0.037(2)	0.041(3)	0.044(3)	-0.014(2)	-0.012(2)	-0.010(2)
C132	0.046(3)	0.046(3)	0.050(3)	-0.012(2)	-0.007(2)	-0.012(2)
C133	0.062(3)	0.038(3)	0.092(4)	-0.004(3)	-0.046(3)	-0.004(3)
C134	0.037(3)	0.060(4)	0.101(5)	0.007(3)	-0.015(3)	-0.041(4)
C135	0.036(3)	0.065(4)	0.072(4)	-0.011(3)	0.000(3)	-0.034(3)
C136	0.034(2)	0.044(3)	0.054(3)	-0.011(2)	-0.008(2)	-0.018(2)
C221	0.058(3)	0.032(3)	0.073(4)	-0.004(2)	-0.023(3)	-0.007(2)
C222	0.091(4)	0.074(4)	0.071(4)	-0.023(3)	-0.043(3)	-0.004(3)
C223	0.106(6)	0.095(6)	0.108(6)	-0.029(5)	-0.070(5)	0.000(5)
C224	0.074(5)	0.057(4)	0.160(8)	-0.009(4)	-0.061(6)	-0.007(5)
C225	0.052(4)	0.053(4)	0.166(8)	0.006(3)	-0.009(4)	-0.034(5)
C226	0.057(3)	0.044(3)	0.108(5)	-0.001(3)	-0.014(3)	-0.031(3)
C231	0.101(5)	0.045(3)	0.069(4)	-0.013(3)	-0.032(3)	0.003(3)
C251	0.081(4)	0.069(4)	0.052(3)	-0.039(3)	-0.001(3)	-0.016(3)
C261	0.042(3)	0.041(3)	0.044(3)	-0.020(2)	-0.003(2)	-0.013(2)
C262	0.055(3)	0.056(3)	0.049(3)	-0.015(2)	-0.010(2)	-0.023(2)
C263	0.061(3)	0.063(3)	0.055(3)	-0.011(3)	-0.022(3)	-0.019(3)
C264	0.057(3)	0.070(4)	0.068(4)	-0.013(3)	-0.030(3)	-0.017(3)
C265	0.039(3)	0.057(3)	0.068(4)	-0.010(2)	-0.004(2)	-0.023(3)
C266	0.039(3)	0.056(3)	0.057(3)	-0.017(2)	0.001(2)	-0.028(3)
C321	0.051(3)	0.060(3)	0.036(3)	-0.022(2)	0.005(2)	-0.010(2)
C322	0.045(3)	0.084(4)	0.047(3)	-0.025(3)	-0.009(2)	0.006(3)
C323	0.065(4)	0.149(7)	0.061(4)	-0.028(4)	-0.017(3)	0.028(5)
C324	0.108(7)	0.144(10)	0.078(6)	0.022(6)	0.000(5)	0.045(6)
C325	0.185(10)	0.079(5)	0.079(6)	-0.036(7)	0.001(6)	0.024(5)
C326	0.125(6)	0.074(4)	0.052(3)	-0.042(4)	-0.010(4)	0.003(3)
C331	0.052(3)	0.151(6)	0.044(3)	-0.044(4)	0.005(3)	-0.011(4)
C351	0.037(3)	0.087(4)	0.083(4)	-0.003(3)	-0.007(3)	-0.013(3)
C361	0.038(2)	0.039(3)	0.044(3)	-0.004(2)	-0.011(2)	-0.004(2)
C362	0.062(3)	0.052(3)	0.050(3)	-0.006(3)	-0.019(3)	-0.001(3)
C363	0.103(5)	0.063(4)	0.045(3)	-0.004(4)	-0.016(3)	-0.002(3)
C364	0.087(5)	0.072(4)	0.051(4)	-0.005(4)	-0.004(3)	0.012(3)
C365	0.058(4)	0.074(4)	0.087(5)	-0.025(3)	-0.006(3)	0.022(4)

Anisotropic Temperature Factor Coefficients - U's (Continued)

for $Zr(O\text{-}2,6Ph\text{-}3,5Me\text{-}Ph)_2(CH_2Ph)(C_6H_5B(C_6F_5)_3)\cdot 2C_6$

Name	U(1,1)	U(2,2)	U(3,3)	U(1,2)	U(1,3)	U(2,3)
C366	0.048(3)	0.059(3)	0.057(3)	-0.018(3)	-0.008(2)	-0.002(3)
B10	0.033(3)	0.034(3)	0.040(3)	-0.009(2)	-0.002(2)	-0.007(2)

The form of the anisotropic temperature factor is:

$\exp[-2\pi (h^2a^{*2}U(1,1) + k^2b^{*2}U(2,2) + l^2c^{*2}U(3,3) + 2hka^*b^*U(1,2) + 2hla^*c^*U(1,3)$

$+ 2klb^*c\ U(2,3))]$ where a^*, b^*, and c^* are reciprocal lattice constants.

Table of Bond Distances in Angstroms

for $Zr(O\text{-}2,6Ph\text{-}3,5Me\text{-}Ph)_2(CH_2Ph)(C_6H_5B(C_6F_5)_3)\cdot 2C_6$

Atom 1	Atom 2	Distance	Atom 1	Atom 2	Distance
Zr	O(2)	1.893(3)	C(25)	C(26)	1.400(6)
Zr	O(3)	1.900(3)	C(25)	C(251)	1.502(7)
Zr	C(40)	2.230(4)	C(26)	C(261)	1.483(6)
Zr	C(2)	2.653(4)	C(31)	C(36)	1.393(6)
Zr	C(3)	2.662(4)	C(31)	C(32)	1.405(6)
Zr	C(4)	2.681(5)	C(32)	C(33)	1.401(7)
Zr	C(5)	2.694(4)	C(32)	C(321)	1.495(7)
Zr	C(6)	2.764(4)	C(33)	C(34)	1.388(7)
Zr	C(1)	2.821(4)	C(33)	C(331)	1.522(7)
F(112)	C(112)	1.364(5)	C(34)	C(35)	1.383(7)
F(113)	C(113)	1.351(6)	C(35)	C(36)	1.411(6)
F(114)	C(114)	1.345(6)	C(35)	C(351)	1.503(7)
F(115)	C(115)	1.343(6)	C(36)	C(361)	1.481(6)
F(116)	C(116)	1.363(5)	C(40)	C(41)	1.495(6)
F(122)	C(122)	1.368(5)	C(41)	C(46)	1.402(7)
F(123)	C(123)	1.340(6)	C(41)	C(42)	1.412(7)
F(124)	C(124)	1.341(6)	C(42)	C(43)	1.388(7)
F(125)	C(125)	1.364(5)	C(43)	C(44)	1.401(9)
F(126)	C(126)	1.355(5)	C(44)	C(45)	1.358(9)
F(132)	C(132)	1.370(5)	C(45)	C(46)	1.429(8)
F(133)	C(133)	1.344(6)	C(111)	C(112)	1.390(6)
F(134)	C(134)	1.356(6)	C(111)	C(116)	1.394(6)
F(135)	C(135)	1.352(6)	C(111)	B(10)	1.646(7)
F(136)	C(136)	1.349(5)	C(112)	C(113)	1.355(7)
O(2)	C(21)	1.380(5)	C(113)	C(114)	1.384(8)
O(3)	C(31)	1.365(5)	C(114)	C(115)	1.351(8)
C(1)	C(6)	1.394(6)	C(115)	C(116)	1.381(7)
C(1)	C(2)	1.418(6)	C(121)	C(122)	1.369(6)
C(1)	C(10)	1.482(6)	C(121)	C(126)	1.384(6)
C(2)	H(2)	1.09(5)	C(121)	B(10)	1.657(6)
C(2)	C(3)	1.394(7)	C(122)	C(123)	1.379(6)
C(3)	H(3)	1.03(5)	C(123)	C(124)	1.388(8)
C(3)	C(4)	1.386(7)	C(124)	C(125)	1.342(8)
C(4)	H(4)	0.97(6)	C(125)	C(126)	1.372(7)
C(4)	C(5)	1.383(7)	C(131)	C(132)	1.369(6)
C(5)	H(5)	0.89(5)	C(131)	C(136)	1.396(6)
C(5)	C(6)	1.383(7)	C(131)	B(10)	1.658(6)
C(6)	H(6)	0.92(5)	C(132)	C(133)	1.398(7)
C(10)	B(10)	1.691(6)	C(133)	C(134)	1.353(8)
C(21)	C(22)	1.391(6)	C(134)	C(135)	1.359(8)
C(21)	C(26)	1.412(6)	C(135)	C(136)	1.375(7)
C(22)	C(23)	1.411(7)	C(221)	C(222)	1.375(8)
C(22)	C(221)	1.477(7)	C(221)	C(226)	1.388(8)
C(23)	C(24)	1.371(7)	C(222)	C(223)	1.403(9)
C(23)	C(231)	1.506(7)	C(223)	C(224)	1.354(11)
C(24)	C(25)	1.387(7)	C(224)	C(225)	1.344(11)

Bond Distances (cont.)

Atom 1	Atom 2	Distance	Atom 1	Atom 2	Distance
C(225)	C(226)	1.400(9)	C(323)	C(324)	1.373(14)
C(261)	C(262)	1.390(6)	C(324)	C(325)	1.363(14)
C(261)	C(266)	1.391(6)	C(325)	C(326)	1.375(11)
C(262)	C(263)	1.384(7)	C(361)	C(366)	1.392(7)
C(263)	C(264)	1.347(7)	C(361)	C(362)	1.392(7)
C(264)	C(265)	1.392(7)	C(362)	C(363)	1.396(8)
C(265)	C(266)	1.370(7)	C(363)	C(364)	1.345(9)
C(321)	C(322)	1.359(7)	C(364)	C(365)	1.381(10)
C(321)	C(326)	1.391(8)	C(365)	C(366)	1.388(8)
C(322)	C(323)	1.386(8)			

Numbers in parentheses are estimated standard deviations in the least significant digits.

Table of Bond Angles in Degrees

for $Zr(O\text{-}2,6Ph\text{-}3,5Me\text{-}Ph)_2(CH_2Ph)(C_6H_5B(C_6F_5)_3)\cdot 2C_6$

Atom 1	Atom 2	Atom 3	Angle	Atom 1	Atom 2	Atom 3	Angle
O(2)	Zr	O(3)	113.63(12)	C(3)	C(2)	C(1)	121.0(4)
O(2)	Zr	C(40)	99.4(2)	H(2)	C(2)	Zr	119(2)
O(3)	Zr	C(40)	99.55(14)	C(3)	C(2)	Zr	75.1(2)
O(2)	Zr	C(2)	98.62(13)	C(1)	C(2)	Zr	81.7(2)
O(3)	Zr	C(2)	108.22(13)	H(3)	C(3)	C(4)	101(3)
C(40)	Zr	C(2)	137.1(2)	H(3)	C(3)	C(2)	136(3)
O(2)	Zr	C(3)	86.36(14)	C(4)	C(3)	C(2)	120.7(5)
O(3)	Zr	C(3)	138.43(14)	H(3)	C(3)	Zr	106(3)
C(40)	Zr	C(3)	113.2(2)	C(4)	C(3)	Zr	75.8(3)
C(2)	Zr	C(3)	30.42(15)	C(2)	C(3)	Zr	74.4(2)
O(2)	Zr	C(4)	100.34(15)	H(4)	C(4)	C(5)	116(3)
O(3)	Zr	C(4)	144.36(15)	H(4)	C(4)	C(3)	126(3)
C(40)	Zr	C(4)	84.7(2)	C(5)	C(4)	C(3)	118.4(5)
C(2)	Zr	C(4)	53.9(2)	H(4)	C(4)	Zr	123(3)
C(3)	Zr	C(4)	30.1(2)	C(5)	C(4)	Zr	75.6(3)
O(2)	Zr	C(5)	129.67(14)	C(3)	C(4)	Zr	74.2(3)
O(3)	Zr	C(5)	116.52(14)	H(5)	C(5)	C(4)	115(3)
C(40)	Zr	C(5)	76.6(2)	H(5)	C(5)	C(6)	123(3)
C(2)	Zr	C(5)	62.0(2)	C(4)	C(5)	C(6)	121.2(5)
C(3)	Zr	C(5)	52.7(2)	H(5)	C(5)	Zr	115(3)
C(4)	Zr	C(5)	29.8(2)	C(4)	C(5)	Zr	74.6(3)
O(2)	Zr	C(6)	147.36(13)	C(6)	C(5)	Zr	78.1(3)
O(3)	Zr	C(6)	91.88(13)	H(6)	C(6)	C(5)	123(3)
C(40)	Zr	C(6)	95.8(2)	H(6)	C(6)	C(1)	114(3)
C(2)	Zr	C(6)	52.23(14)	C(5)	C(6)	C(1)	122.0(4)
C(3)	Zr	C(6)	61.07(14)	H(6)	C(6)	Zr	114(3)
C(4)	Zr	C(6)	52.5(2)	C(5)	C(6)	Zr	72.5(3)
C(5)	Zr	C(6)	29.33(14)	C(1)	C(6)	Zr	77.8(2)
O(2)	Zr	C(1)	127.58(12)	C(1)	C(10)	B(10)	111.4(3)
O(3)	Zr	C(1)	87.75(12)	O(2)	C(21)	C(22)	119.1(4)
C(40)	Zr	C(1)	124.7(2)	O(2)	C(21)	C(26)	118.1(4)
C(2)	Zr	C(1)	29.83(13)	C(22)	C(21)	C(26)	122.7(4)
C(3)	Zr	C(1)	52.94(13)	C(21)	C(22)	C(23)	117.2(4)
C(4)	Zr	C(1)	62.04(15)	C(21)	C(22)	C(221)	121.5(4)
C(5)	Zr	C(1)	52.21(14)	C(23)	C(22)	C(221)	121.2(4)
C(6)	Zr	C(1)	28.89(12)	C(24)	C(23)	C(22)	119.9(4)
C(21)	O(2)	Zr	165.3(3)	C(24)	C(23)	C(231)	119.6(5)
C(31)	O(3)	Zr	170.5(3)	C(22)	C(23)	C(231)	120.6(5)
C(6)	C(1)	C(2)	116.1(4)	C(23)	C(24)	C(25)	123.0(5)
C(6)	C(1)	C(10)	123.3(4)	C(24)	C(25)	C(26)	118.5(4)
C(2)	C(1)	C(10)	120.4(4)	C(24)	C(25)	C(251)	121.2(4)
C(6)	C(1)	Zr	73.3(2)	C(26)	C(25)	C(251)	120.3(5)
C(2)	C(1)	Zr	68.5(2)	C(25)	C(26)	C(21)	118.3(4)
C(10)	C(1)	Zr	132.0(3)	C(25)	C(26)	C(261)	121.3(4)
H(2)	C(2)	C(3)	123(3)	C(21)	C(26)	C(261)	120.4(4)
H(2)	C(2)	C(1)	116(3)	O(3)	C(31)	C(36)	118.2(4)

Bond Angles (cont.)

Atom 1	Atom 2	Atom 3	Angle	Atom 1	Atom 2	Atom 3	Angle
O(3)	C(31)	C(32)	118.6(4)	F(122)	C(122)	C(123)	114.7(4)
C(36)	C(31)	C(32)	123.1(4)	C(121)	C(122)	C(123)	126.0(4)
C(33)	C(32)	C(31)	118.2(4)	F(123)	C(123)	C(122)	121.4(5)
C(33)	C(32)	C(321)	122.5(4)	F(123)	C(123)	C(124)	120.6(4)
C(31)	C(32)	C(321)	119.2(4)	C(122)	C(123)	C(124)	118.0(5)
C(34)	C(33)	C(32)	118.8(4)	F(124)	C(124)	C(125)	122.3(5)
C(34)	C(33)	C(331)	119.5(5)	F(124)	C(124)	C(123)	119.3(5)
C(32)	C(33)	C(331)	121.7(5)	C(125)	C(124)	C(123)	118.4(4)
C(35)	C(34)	C(33)	122.9(4)	C(124)	C(125)	F(125)	119.1(4)
C(34)	C(35)	C(36)	119.4(4)	C(124)	C(125)	C(126)	121.2(5)
C(34)	C(35)	C(351)	120.1(4)	F(125)	C(125)	C(126)	119.7(5)
C(36)	C(35)	C(351)	120.5(4)	F(126)	C(126)	C(125)	114.7(4)
C(31)	C(36)	C(35)	117.6(4)	F(126)	C(126)	C(121)	121.4(4)
C(31)	C(36)	C(361)	119.5(4)	C(125)	C(126)	C(121)	123.8(4)
C(35)	C(36)	C(361)	123.0(4)	C(132)	C(131)	C(136)	112.9(4)
C(41)	C(40)	Zr	117.4(3)	C(132)	C(131)	B(10)	122.7(4)
C(46)	C(41)	C(42)	117.9(5)	C(136)	C(131)	B(10)	123.8(4)
C(46)	C(41)	C(40)	121.0(5)	C(131)	C(132)	F(132)	119.3(4)
C(42)	C(41)	C(40)	121.1(4)	C(131)	C(132)	C(133)	124.8(5)
C(43)	C(42)	C(41)	122.1(5)	F(132)	C(132)	C(133)	115.9(4)
C(42)	C(43)	C(44)	119.1(6)	F(133)	C(133)	C(134)	121.7(5)
C(45)	C(44)	C(43)	120.2(6)	F(133)	C(133)	C(132)	119.5(6)
C(44)	C(45)	C(46)	121.4(5)	C(134)	C(133)	C(132)	118.8(5)
C(41)	C(46)	C(45)	119.3(5)	C(133)	C(134)	F(134)	119.4(6)
C(112)	C(111)	C(116)	111.1(4)	C(133)	C(134)	C(135)	119.6(5)
C(112)	C(111)	B(10)	120.9(4)	F(134)	C(134)	C(135)	121.0(6)
C(116)	C(111)	B(10)	127.8(4)	F(135)	C(135)	C(134)	120.0(5)
C(113)	C(112)	F(112)	115.5(4)	F(135)	C(135)	C(136)	120.0(5)
C(113)	C(112)	C(111)	126.1(5)	C(134)	C(135)	C(136)	119.9(5)
F(112)	C(112)	C(111)	118.4(4)	F(136)	C(136)	C(135)	114.9(4)
F(113)	C(113)	C(112)	121.4(5)	F(136)	C(136)	C(131)	121.1(4)
F(113)	C(113)	C(114)	119.5(5)	C(135)	C(136)	C(131)	123.9(5)
C(112)	C(113)	C(114)	119.1(5)	C(222)	C(221)	C(226)	117.3(5)
F(114)	C(114)	C(115)	120.6(6)	C(222)	C(221)	C(22)	123.2(5)
F(114)	C(114)	C(113)	120.4(6)	C(226)	C(221)	C(22)	119.3(5)
C(115)	C(114)	C(113)	119.0(5)	C(221)	C(222)	C(223)	122.5(7)
F(115)	C(115)	C(114)	120.4(5)	C(224)	C(223)	C(222)	117.8(7)
F(115)	C(115)	C(116)	120.3(5)	C(225)	C(224)	C(223)	122.0(7)
C(114)	C(115)	C(116)	119.3(5)	C(224)	C(225)	C(226)	120.2(8)
F(116)	C(116)	C(115)	114.9(4)	C(221)	C(226)	C(225)	120.1(7)
F(116)	C(116)	C(111)	119.8(4)	C(262)	C(261)	C(266)	117.3(4)
C(115)	C(116)	C(111)	125.3(5)	C(262)	C(261)	C(26)	120.0(4)
C(122)	C(121)	C(126)	112.5(4)	C(266)	C(261)	C(26)	122.7(4)
C(122)	C(121)	B(10)	119.6(4)	C(263)	C(262)	C(261)	121.5(5)
C(126)	C(121)	B(10)	127.7(4)	C(264)	C(263)	C(262)	119.9(5)
F(122)	C(122)	C(121)	119.3(4)	C(263)	C(264)	C(265)	120.4(5)

Bond Angles (cont.)

Atom 1	Atom 2	Atom 3	Angle	Atom 1	Atom 2	Atom 3	Angle
C(266)	C(265)	C(264)	119.6(5)	C(362)	C(361)	C(36)	121.6(4)
C(265)	C(266)	C(261)	121.4(5)	C(361)	C(362)	C(363)	120.5(6)
C(322)	C(321)	C(326)	118.7(5)	C(364)	C(363)	C(362)	120.6(6)
C(322)	C(321)	C(32)	119.4(5)	C(363)	C(364)	C(365)	120.2(6)
C(326)	C(321)	C(32)	121.9(5)	C(364)	C(365)	C(366)	120.3(6)
C(321)	C(322)	C(323)	122.7(6)	C(365)	C(366)	C(361)	120.3(6)
C(324)	C(323)	C(322)	117.2(8)	C(111)	B(10)	C(121)	103.6(3)
C(325)	C(324)	C(323)	121.4(8)	C(111)	B(10)	C(131)	112.9(4)
C(324)	C(325)	C(326)	120.5(9)	C(121)	B(10)	C(131)	113.0(3)
C(325)	C(326)	C(321)	119.4(7)	C(111)	B(10)	C(10)	109.8(3)
C(366)	C(361)	C(362)	118.2(5)	C(121)	B(10)	C(10)	115.3(4)
C(366)	C(361)	C(36)	120.2(4)	C(131)	B(10)	C(10)	102.6(3)

Numbers in parentheses are estimated standard deviations in the least significant digits.

$[Ti(OC_6H_3Ph_2\text{-}2,6)_2(CH_2Ph)][\eta^6\text{-}C_6H_5CH_2B(C_6F_5)_3]$ **(4a).**

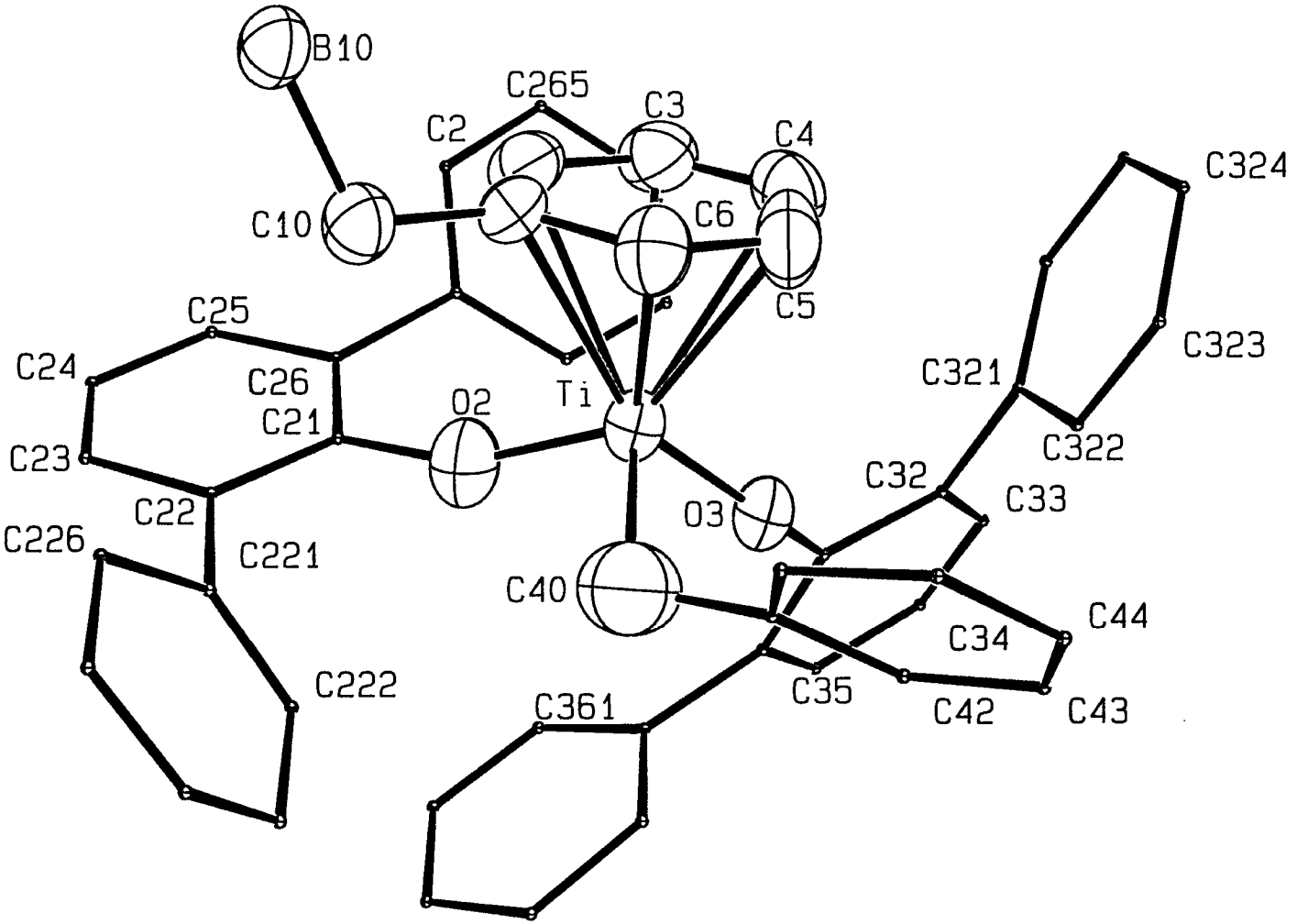


TABLE OF CONTENTS

EXPERIMENTAL

DATA COLLECTION

A red chunk of $C_{68}H_{40}TiBF_{15}O_2$ having approximate dimensions of 0.64 x 0.62 x 0.58 mm was mounted in a glass capillary in a random orientation. Preliminary examination and data collection were performed with Mo K_α radiation (λ = 0.71073 Å) on an Enraf-Nonius CAD4 computer controlled kappa axis diffractometer equipped with a graphite crystal, incident beam monochromator.

Cell constants and an orientation matrix for data collection were obtained from least-squares refinement, using the setting angles of 25 reflections in the range $16 < \theta < 18°$, measured by the computer controlled diagonal slit method of centering. The triclinic cell parameters and calculated volume are: a = 12.917(2), b = 13.045(2), c = 19.600(2)Å, α = 84.76(1), β = 77.58(1), γ = 61.87(1)°, V = 2844.0Å^3. For Z = 2 and F.W. = 1232.76 the calculated density is 1.44 g/cm^3. As a check on crystal quality, omega scans of several intense reflections were measured; the width at half-height was 0.98° with a take-off angle of 3.0° indicating poor crystal quality. The space group was determined by the program ABSEN(ref 1). There were no systematic absences; the space group was determined to be P1(# 2).

The data were collected at a temperature of 295 ± 1 K using the omega scan technique. The scan rate varied from 3 to 16°/min (in omega). The variable scan rate allows rapid data collection for intense reflections where a fast scan rate is used and assures good counting statistics for weak reflections where a slow scan rate is used. Data were collected to a maximum 2θ of 45.3°. The scan range (in deg.) was determined as a function of θ to correct for the separation of the Kα doublet(ref 2); the scan width was calculated as follows:

$$\omega \text{ scan width} = 0.98 + 0.500 \tan\theta$$

Moving-crystal moving-counter background counts were made by scanning an additional 25% above and below this range. Thus the ratio of peak counting time to background counting time was 2:1. The counter aperture was also adjusted as a function of θ. The horizontal aperture width ranged from 1.8 to 2.4 mm; the vertical aperture was set at 4.0 mm. The diameter of the incident beam collimator was 0.7 mm and the crystal to detector distance was 21cm. For intense reflections an attenuator was automatically inserted in front of the detector; the attenuator factor was 13.2.

DATA REDUCTION

A total of 7524 reflections were collected, of which 7524 were unique. As a check on crystal and electronic stability 3 representative reflections were measured every 83 min. The intensities of these standards remained constant within experimental error throughout data collection. No decay correction was applied.

Lorentz and polarization corrections were applied to the data. The linear absorption coefficient is 2.4 /cm for Mo K radiation. No absorption correction was made. Intensities of equivalent reflections were averaged. The agreement factor for the averaging was 1.7% based on intensity.

STRUCTURE SOLUTION AND REFINEMENT

The structure was solved using the structure solution program PATTY in DIRDIF92 (ref 3). The remaining atoms were located in succeeding difference Fourier syntheses. Hydrogen atoms were included in the refinement but restrained to ride on the atom to which they are bonded. The structure was refined in full-matrix least-squares where the function minimized was $\Sigma w(|Fo|^2-|Fc|^2)^2$ and the weight w is defined as $w=1/[\sigma^2(Fo^2)+(0.1033P)^2+6.4177P]$ where $P=(Fo^2+2Fc^2)/3$

Scattering factors were taken from the "International Tables for Crystallography" (ref 4). 7523 reflections were used in the refinements. However, only reflections with $F_o^2>2\sigma(F_o^2)$ were used in calculating R. The final cycle of refinement included 804 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R1 = \Sigma\ |Fo - Fc|\ /\ \Sigma\ Fo = 0.069$$

$$R2 = SQRT\ (\ \Sigma\ w\ (\ Fo^2 - Fc^2\ )^2\ /\ \Sigma\ w\ (Fo^2)^2\ ) = 0.178$$

The standard deviation of an observation of unit weight was 1.09. The highest peak in the final difference Fourier had a height of 1.16 e/A^3. The minimum negative peak had a height of -0.29 e/A^3.

Refinement was performed on a AlphaServer 2100 using SHELX-93 (ref 5). Crystallographic drawings were done using programs ORTEP (ref 6) and/or PLUTON (ref 7).

---

(1) P. C. McArdle, J. Appl. Cryst., 239, 306 (1996).
(2) "CAD4 Operations Manual", Enraf-Nonius, Delft, (1977)
(3) P. T. Beurskens, G. Admirall, G. Beurskens, W. P. Bosman S. Garcia-Granda, R. O. Gould, J. M. M. Smits, and C. Smykalla, The DIRDIF92 Program System, Technical Report. Crystallography Laboratory, Univ. of Nijmegen, The Netherlands, (1992)
(4) "International Tables for Crystallography", Vol. C, Kluwer Academic Publishers, Dordrecht, The Netherlands, (199)2, Tables 4.2.6.8 and 6.1.1.4
(5) G. M. Sheldrick, SHELXS93. A Program for Crystal Structure Refinement. Univ. of Gottingen, Germany, (1993)