Synthesis, Characterization, and Marked Polymerization Selectivity Characteristics of Binuclear Phenoxyiminato Organozirconium Catalysts.

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Supporting Information

Materials and Methods. All manipulations of air-sensitive materials were performed with rigorous exclusion of oxygen and moisture in flamed Schlenk-type glassware on a dual manifold Schlenk line, or interfaced to a high-vacuum line (10^{-5} Torr), or in a nitrogen-filled Vacuum Atmospheres glove box with a high capacity recirculator (<1 ppm O_2). Argon and ethylene (Matheson, polymerization grade) were purified by passage through a supported MnO oxygen-removal column and an activated Davison 4A molecular sieve column. Ether solvents were purified by distillation from Na/K alloy/benzophenone ketyl. Hydrocarbon solvents were distilled under nitrogen from Na/K alloy. All solvents for high-vacuum line manipulations were stored *in vacuo* over Na/K alloy in Teflon-valve sealed bulbs. Deuterated solvents were obtained from Cambridge

Isotope Laboratories (all ≥ 99 atom %D), were freeze pump- thaw degassed, dried over Na/K alloy and were stored in resealable flasks. Other non-halogenated solvents were dried over Na/K alloy, and halogenated solvents were distilled from CaH_2 and stored over activated Davison 4A molecular sieves. The reagents 2,6-di-*iso*-propylaniline, and chlorotrimethylsilane were purchased from Aldrich and used as received**.** The comonomer 1-hexene (Aldrich) was dried over CaH_2. The reagents $LiCH_2TMS$ and $ZrCl_4$ (Aldrich) were sublimed before use. Methylaluminoxane (MAO, obtained as a 10% toluene solution from Aldrich) was dried under high vacuum for 24 h to remove excess volatile aluminum alkyls before use. The reagents $ZrCl_4(THF)_2$[1] and $Zr\{3\text{-}^tBu\text{-}2\text{-}(O)C_6H_3CH{=}N(2,6\text{-}^iPr_2C_6H_3)\}Cl_3(THF)$[2] (**FI-Zr1**) were prepared according to literature procedures, however, for the latter, heating at reflux for 16 h was required for complete reaction.

Physical and Analytical Measurements. NMR spectra were recorded on Varian UNITYInova-500 (FT, 500 MHz, 1H; 125 MHz, 13C) or UNITYInova-400 (FT, 400 MHz, 1H; 100 MHz, 13C). Chemical shifts for ^1H and ^{13}C spectra were referenced using internal solvent resonances and are reported relative to tetramethylsilane. NMR experiments on air-sensitive samples were conducted in Teflon valve-sealed sample tubes (J.Young). Elemental analyses were performed by Midwest Microlab, Indianapolis, Indiana. ^{13}C NMR assays of polymer microstructure were conducted in mixture (1:1 in volume) of 1,2,4-trichlorobezene (containing 0.1 M $Cr(acac)_3$ solution) and 1,1,2,2-tetrachloroethane-d_2 at 130°C. Signals were assigned according to the literature for polyethylene and ethylene + α-olefin copolymers.[3] Melting temperatures of polymers were measured by DSC (DSC 2920, TA Instruments, Inc.) from the second scan with a heating rate of 10 °C/min. GPC measurements were performed on a Polymer Laboratories PL-GPC 220 instrument using 1,2,4-trichlorobenzene solvent (stabilized with 125 ppm BHT) at 150 °C. A set of three PLgel 10 μm mixed columns was used. Samples were prepared at 160 °C. Molecular weights were determined by GPC using narrow polystyrene standards and are not corrected. Laser desorption mass spectra were obtained with a Perseptive BioSystems time-of-flight MALDI mass spectrometer using a dithranol matrix.

Synthesis of 2,7-di(2,6-diisopropylphenyl)imino-1,8-dihydroxynaphthalene (1). Under N_2, the reagent 2,7-diformyl-1,8-dihydroxynaphthalene (2.15 g, 9.93 mmol) was dissolved in 50 mL dry dichloromethane. Activated molecular sieves (2 g) were then added, followed by formic acid (5 drops), and 2,6-di-*iso*-propylaniline (8.0 mL, 42 mmol). The reaction was heated under reflux for 3 h during which time the 2,7-diformyl-1,8-dihydroxynaphthalene gradually dissolved, and the reaction mixture color changed from yellow to dark red. The reaction mixture was then cooled and filtered. The volatiles were removed from the filtrate *in vacuo*, and the resulting red oily solid was washed with hexanes (3 x 15 mL) and collected by filtration to give a pure orange solid. X-ray quality single crystals were obtained by slow evaporation of a dichloromethane solution. Yield, 4.5 g (86%).

^{1}H NMR ($CDCl_3$, 23°C, 499.447 MHz): δ 14.56 (s, OH), 13.48 (s, OH), 8.75 (s), 8.34 (s), 7.66 (d, J = 11.3 Hz), 6.78-7.35 (m), 3.08 (d, $C\mathit{H}Me_2$), 1.20 (d, $CH\mathit{Me}_2$).

Anal. Found: C, 80.84; H, 7.90; N, 5.25. Calcd. for $C_{36}H_{42}N_2O_2$: C, 80.86; H, 7.92; N, 5.24.

Synthesis of 2,7-Di(2,6-diisopropylphenyl)imino-1,8-di(trimethylsiloxy)naphthalene (2). The reagents 2,7-di(2,6-diisopropylphenyl)imino-1,8-dihydroxynapthalene (589 mg, 1.10 mmol) and $LiCH_2TMS$ (228.2 mg, 2.424 mmol) were combined in a reaction flask in the glovebox. On the vacuum line, dry THF (25 mL) was added at -78°C and the mixture was stirred for 30 min, followed by stirring for 30 min at 0°C. The volatiles were next removed *in vacuo*, and the resulting solid was dissolved in 20 mL dry dichloromethane. Next, chlorotrimethylsilane (0.35 mL, 2.7 mmol) was added with stirring, and the resulting solution became hazy over a period of 4.5 h. The reaction mixture was then filtered, and volatiles were removed from the filtrate *in vacuo*. Pure orange product was obtained by washing the resulting orange/brown solid with cold pentane (5 mL). X-ray quality single crystals were obtained by cooling a saturated pentane solution at -10°C. Yield, 598 mg (80%).

^{1}H NMR ($CDCl_3$, 23°C, 499.447MHz): δ 8.762 (s, 2H, *H*C=N), 8.475 (d, 2H, $^3J_{H\text{-}H}$ = 8.457 Hz, naph C3), 7.639 (d, 2H, $^3J_{H\text{-}H}$ = 8.641 Hz, naph C4), 7.297 (d, 2H, $^3J_{H\text{-}H}$ = 7.537 Hz, dipp *p*), 7.231 (t, 4H, $^3J_{H\text{-}H}$ = 7.354 Hz, dipp *m*), 3.152 (sept, 4H, $^3J_{H\text{-}H}$ = 6.802 Hz, C*H*Me$_2$), 1.307 (s, 24H, CH*Me*$_2$), 0.227 (s, 18H, Si*Me*$_3$). ^{13}C NMR ($CDCl_3$, 23°C, 100.745 MHz) δ 158.447, 154.442, 150.080, 140.512, 138.032, 125.771, 124.647, 124.214, 123.351, 123.267, 122.460, 28.111, 24.214, 23.564, 0.387.

Anal. Found: C, 74.26; H, 8.60; N, 4.15. Calcd. for $C_{36}H_{42}N_2O_2$: C, 74.28; H, 8.61; N, 4.13.

Synthesis of {1,7-$(O)_2C_{10}H_4$-2,7-[CH=N(2,6-*i*$Pr_2C_6H_3$)]$_2$}$Zr_2Cl_6(THF)_2$ (FI^2-Zr_2).

Under N_2, the reagent 2,7-di(2,6-diisopropylphenyl)imino-1,8-di(trimethylsiloxy)naphthalene (484 mg, 0.713 mmol) in 20 mL dry dichloromethane was added dropwise with stirring to a solution of $ZrCl_4(THF)_2$ (538 mg, 1.43 mmol) in 20 mL dry dichloromethane at -78°C. The solution was then allowed to warm to room temperature and stirred for 1 h before heating to reflux for 24 h. The volatiles were then removed *in vacuo*, and the residue was redissolved in 25 mL dichloromethane and filtered. Next, 20 mL dry toluene was added to the filtrate, and the resulting pure orange product was isolated by filtration. Attempts at isolating single crystals of FI^2-Zr_2 using standard methods gave powders. Yield, 762 mg (79%).

^{1}H NMR (CD_2Cl_2, 23°C, 499.447MHz): δ 12.953 (s, 2H, *H*C=N), 8.162 (d, 2H, $^3J_{H-H}$=14.668 Hz, naph C3), 7.620 (d, 2H, $^3J_{H-H}$ = 8.149 Hz, naph C4), 7.501 (t, 2H, $^3J_{H-H}$ = 7.335 Hz, dipp *p*), 7.330 (m, 4H, $^3J_{H-H}$ = 8.964 Hz, dipp *m*), 3.193 (sept, 4H, $^3J_{H-H}$ = 6.520 Hz, C*H*Me2), 1.286 (s, 24H, $^3J_{H-H}$ = 6.519 Hz, CH*Me2*). ^{13}C NMR (CD_2Cl_2, 23°C, 100.745 MHz): δ 171.281, 167.466, 147.524, 144.200, 142.764, 136.481, 134.094, 130.865, 124.656, 119.707, 110.889, 29.032, 23.436.

Anal. Found: C, 49.68; H, 5.48; N, 2.65. Calcd. for $C_{36}H_{42}N_2O_2$: C, 49.29; H, 5.26; N, 2.61.

MALDI-TOF-MS: m/z 1074.2 (parent ion calcd 1070.04), 921.9 (-2 THF calcd 925.93), 731.8 (-2 THF and -$ZrCl_3$ calcd 730.13).

Ethylene Homopolymerization Experiments. On the high vacuum line (10^{-5} Torr), ethylene polymerizations were carried out in 250 mL round-bottom three-neck flasks equipped with a large magnetic stirring bar, Morton indentations, and a thermocouple probe. In a typical experiment, a pre-assembled, degassed reactor containing toluene (50 mL) and MAO (1000 eq relative to Zr) was pre-saturated under of ethylene (1.0 atm) and equilibrated at the desired reaction temperature using an external bath. The catalytically-active species was freshly generated in toluene (4.0 mL) and MAO (500 equiv relative to metal). After an activation time (~30 min), the catalyst solution was then quickly injected into the rapidly stirred flask using a gas-tight syringe equipped with a flattened spraying needle. The temperature of the toluene solution was monitored in representative experiments using a thermocouple (OMEGA Type K thermocouple with a Model HH21 microprocessor thermometer). The reaction exotherm temperature rise was invariably less than 5°C during these polymerizations. After a measured time interval, the polymerization was quenched by the addition of 2% acidified methanol (15 mL). More methanol (100 mL) was then added, and the polymer was collected by filtration, washed with methanol and 10% aqueous HCl, and dried on the high vacuum line overnight to a constant weight.

Ethylene + Olefin Copolymerization Experiments. On the high vacuum line (10^{-5} Torr), ethylene polymerizations were carried out in 250 mL round-bottom three-neck flasks equipped with a large magnetic stirring bar, Morton indentations, and a thermocouple probe. In a typical experiment, 10 mL of comonomer was vacuum-transferred into a graduated flask at –78°C, then the comonomer was injected into a pre-assembled, degassed reactor containing toluene (50 mL) and MAO (500 equiv relative to Zr). The mixture was pre-saturated under of ethylene (1.0 atm) and equilibrated at the desired reaction temperature using an external bath. The catalytically-active species was freshly generated in toluene (4.0 mL) and MAO (500 eq relative to metal). After an activation time (~30 min), the catalyst solution was then quickly injected into the rapidly stirred flask using a gas-tight syringe equipped with a flattened spraying needle. The temperature of the toluene solution was monitored in representative experiments using a thermocouple (OMEGA Type K thermocouple with a Model HH21 microprocessor thermometer). The reaction exotherm temperature rise was invariably less than 5°C during these polymerizations. After a measured time interval, the polymerization was quenched by the addition of 2% acidified methanol (15 mL). More methanol (100 mL) was then added, and the polymer was collected by filtration, washed with methanol and 10% aqueous HCl, and dried on the high vacuum line overnight to a constant weight.

Crystal Structures of 1 and 2.

A. 2,7-Di(2,6-diisopropylphenyl)imino-1,8-dihydroxynaphthalene (1). A summary of crystal structure data for compound **1** is presented in Table S1. Selected bond distances and angles for **1** are summarized in Table S2. Figure S1 shows the solid state structure of the binucleating ligand precursor **1**. The asymmetric crystal structure reveals that O(2) actually is in essence deprotonated by the proximate imine functionality N(2). A proton was located in the difference map between O(1) and O(2) in a pseudo-chelating fashion which causes the anilino group to assume a *trans* geometry due to the steric demand of the *iso*propyl groups. The crystal structure explains the asymmetry and room temperature broadness of the ^{1}H NMR spectrum.

B. 2,7-Di(2,6-diisopropylphenyl)imino-1,8-di(trimethylsiloxy)naphthalene (2). A summary of crystal structure data for compound **2** is presented in Table S1. Selected bond distances and angles for **2** are summarized in Table S3. Figure S2 shows the solid state structure of **2**. The crystal structure reveals the symmetric nature of the molecule, and as expected, the bulky trimethylsiloxy groups have close nonbonded contacts with the di-*iso*propylanilino groups.

References

[1] Manzer, L. E. *Inorg. Synth.* **1982**, *21*, 136.

[2] Pennington, D. A.; Clegg, W.; Coles,S. J.; Harrington, R. W.; Hursthouse, M. B.; Hughes, D. L.; Light, M. E.; Schormann, M.; Bochmann, M.; Lancaster, S. J. *Dalton Trans.* **2005**, *3*, 561-571.

[3] Pooter, M. De.; Smith, P. B.; Dohrer, K. K.; Bennett, K. F.; Meadows, M. D.; Smith, C. G.; Schouwenaars, H. P.; Geerards, R. A. *J. App. Polym. Sci.* **1991**, *42*, 399-408.

Figure Captions

Figure S1. ORTEP plot and atom numbering scheme for 2,7-di(2,6-di-isopropylphenyl)imino-1,8-dihydroxynaphthalene (**1**). Thermal ellipsoids are drawn at the 50% probability level.

Figure S2. ORTEP plot and numbering scheme for 2,7-di(2,6-diisopropylphenyl)imino-1,8-di(trimethylsiloxy)naphthalene (**2**). Thermal ellipsoids are drawn at the 50% probability level.

Figure S3. ^{1}H NMR spectrum ($CDCl_3$, 500 MHz) of 2,7-di(2,6-diisopropylphenyl)imino-1,8-dihydroxynaphthalene (**1**).

Figure S4. ^{1}H NMR spectrum ($CDCl_3$, 400 MHz) of 2,7-di(2,6-diisopropylphenyl)imino-1,8-dihydroxynaphthalene (**1**) at -33°C.

Figure S5. ^{1}H NMR spectrum ($CDCl_3$, 500 MHz) of 2,7-di(2,6-diisopropylphenyl)imino-1,8-di(trimethylsiloxy)naphthalene (**2**).

Figure S6. ^{13}C NMR spectrum ($CDCl_3$, 100 MHz) of 2,7-di(2,6-diisopropylphenyl)imino-1,8-di(trimethylsiloxy)naphthalene (**2**).

Figure S7. ^{1}H NMR spectrum (CD_2Cl_2, 500 MHz) of $\{1,7\text{-}(O)_2C_{10}H_4\text{-}2,7\text{-}[CH{=}N(2,6\text{-}i\text{Pr}_2C_6H_3)]_2\}Zr_2Cl_6(THF)_2$ (**FI^2-Zr_2**).

Figure S8. ^{13}C NMR spectrum (CD_2Cl_2, 100 MHz) of $\{1,7\text{-}(O)_2C_{10}H_4\text{-}2,7\text{-}[CH{=}N(2,6\text{-}i\text{Pr}_2C_6H_3)]_2\}Zr_2Cl_6(THF)_2$ (**FI^2-Zr_2**).

Figure 9. MALDI mass spectrum of $\{1,7\text{-}(O)_2C_{10}H_4\text{-}2,7\text{-}[CH{=}N(2,6\text{-}i\text{Pr}_2C_6H_3)]_2\}Zr_2Cl_6(THF)_2$ (**FI^2-Zr_2**) with dithranol as the supporting matrix.

Figure S1.

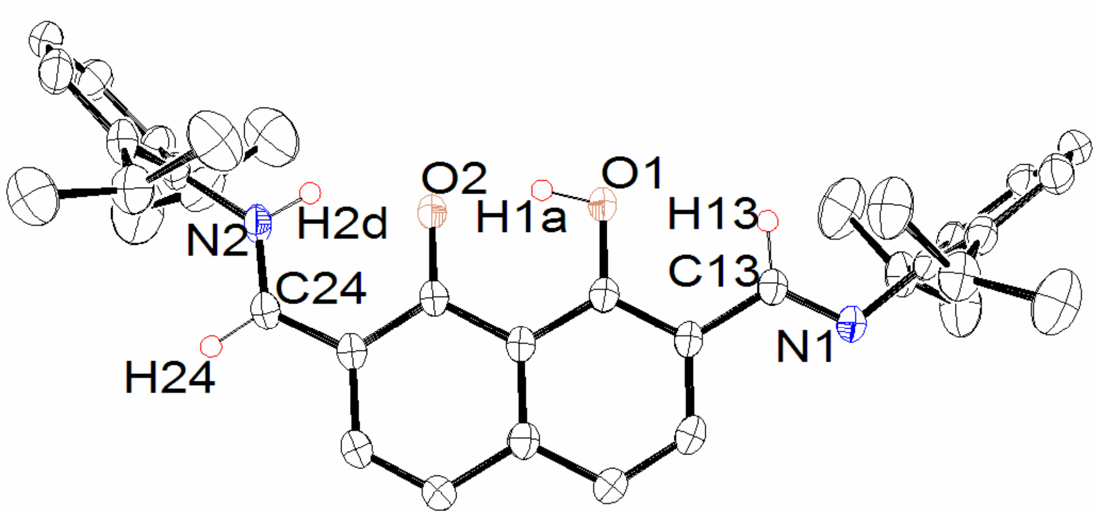


Figure S2.

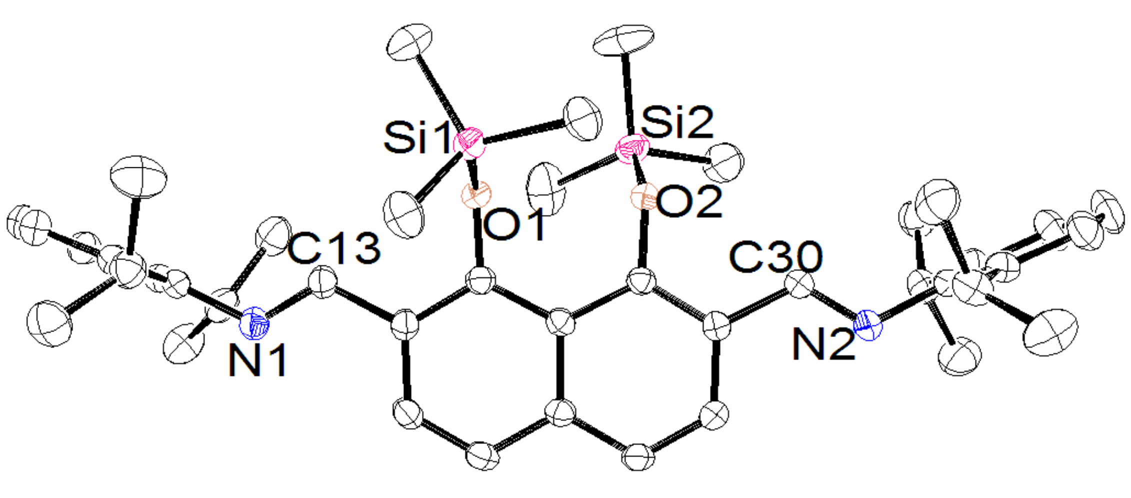
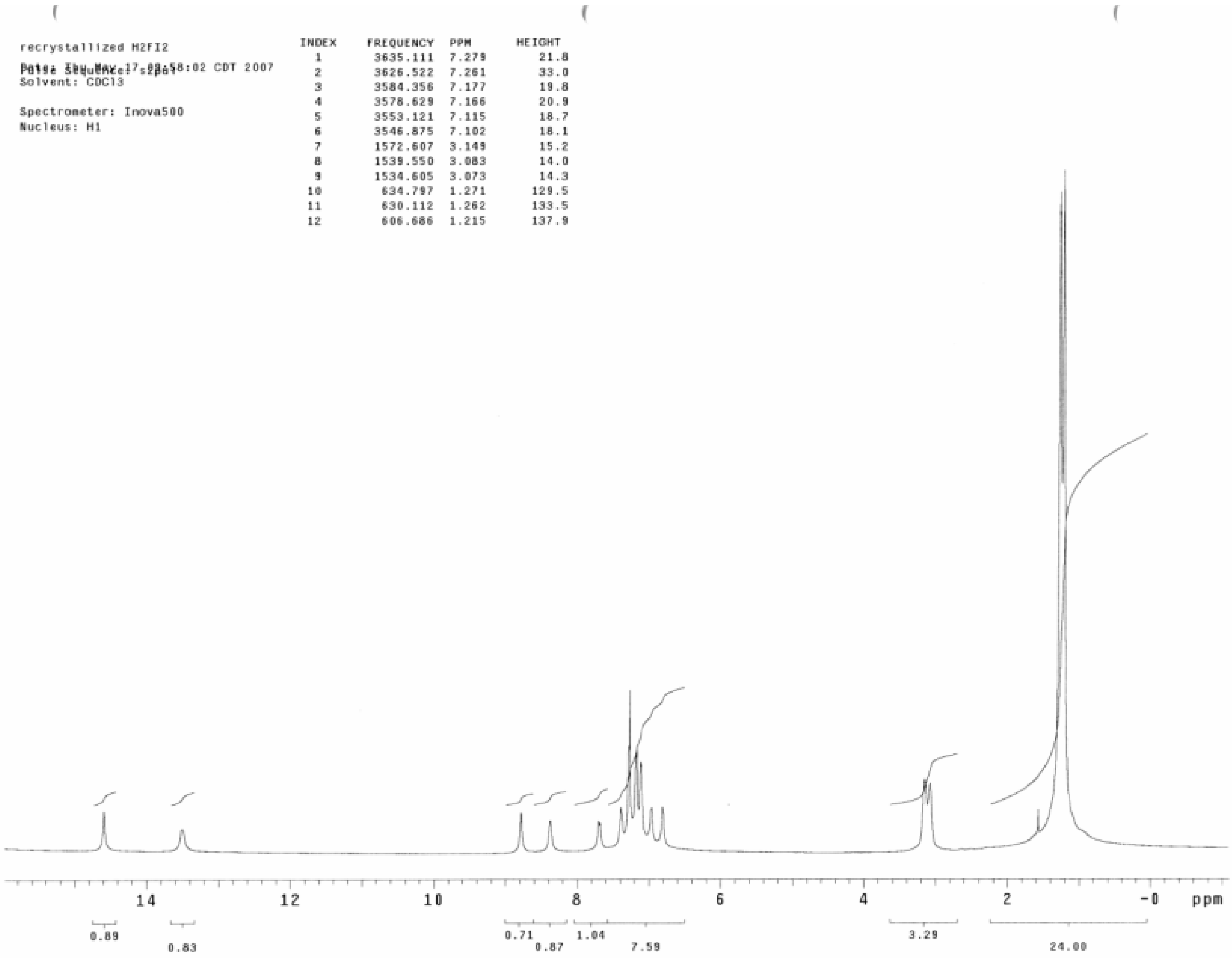


Figure S3.



recrystallized H2FI2

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Solvent: CDCl3

Spectrometer: Inova500
Nucleus: H1

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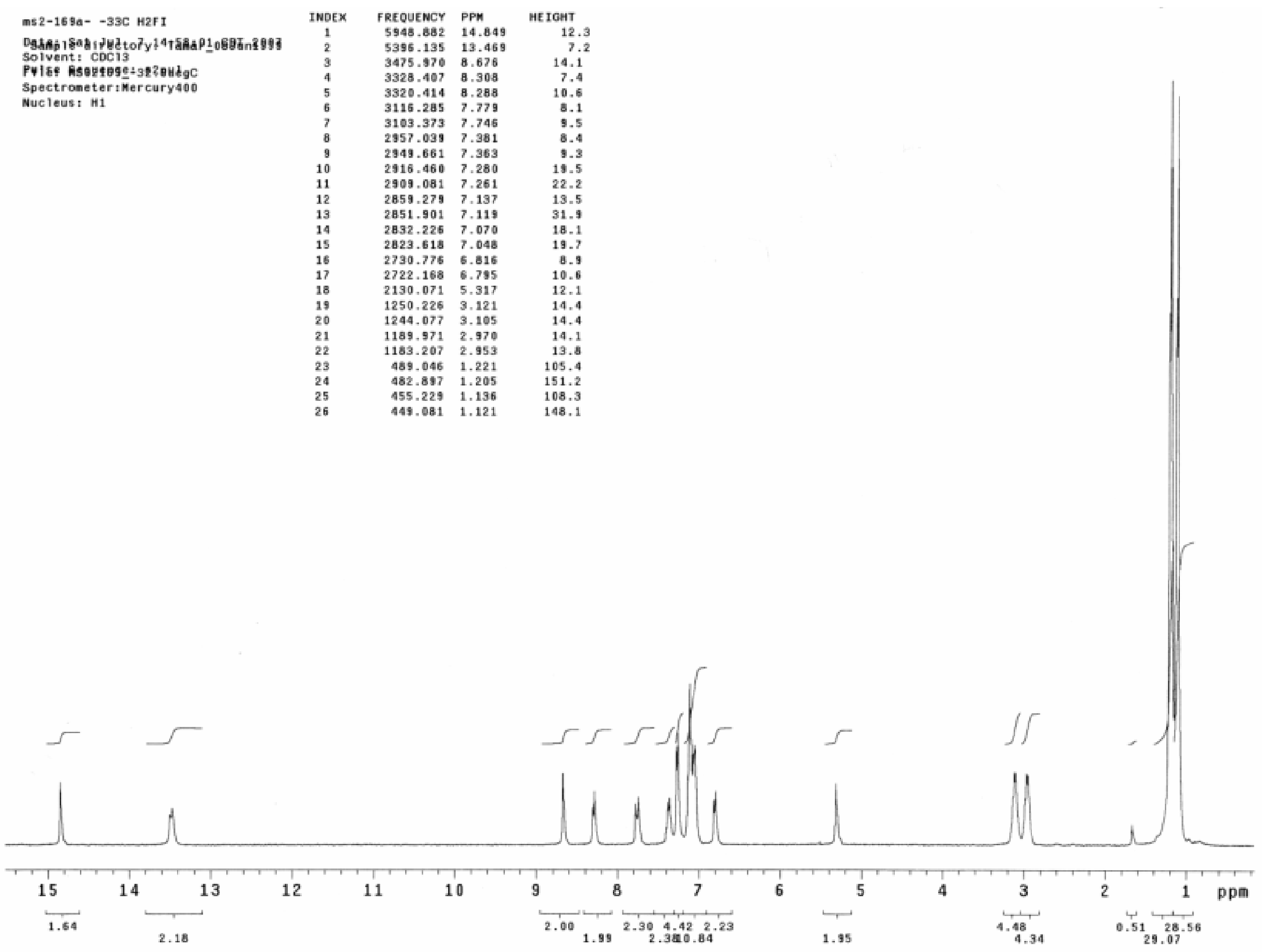
2	3626.522	7.261	33.0
3	3584.356	7.177	19.8
4	3578.629	7.166	20.9
5	3553.121	7.115	18.7
6	3546.875	7.102	18.1
7	1572.607	3.149	15.2
8	1539.550	3.083	14.0
9	1534.605	3.073	14.3
10	634.797	1.271	129.5
11	630.112	1.262	133.5
12	606.686	1.215	137.9

Figure S4.



ms2-169a- -33C H2FI

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Solvent: CDCl3
Pulse Sequence: s2pul / File: [illegible]degC
Spectrometer:Mercury400
Nucleus: H1

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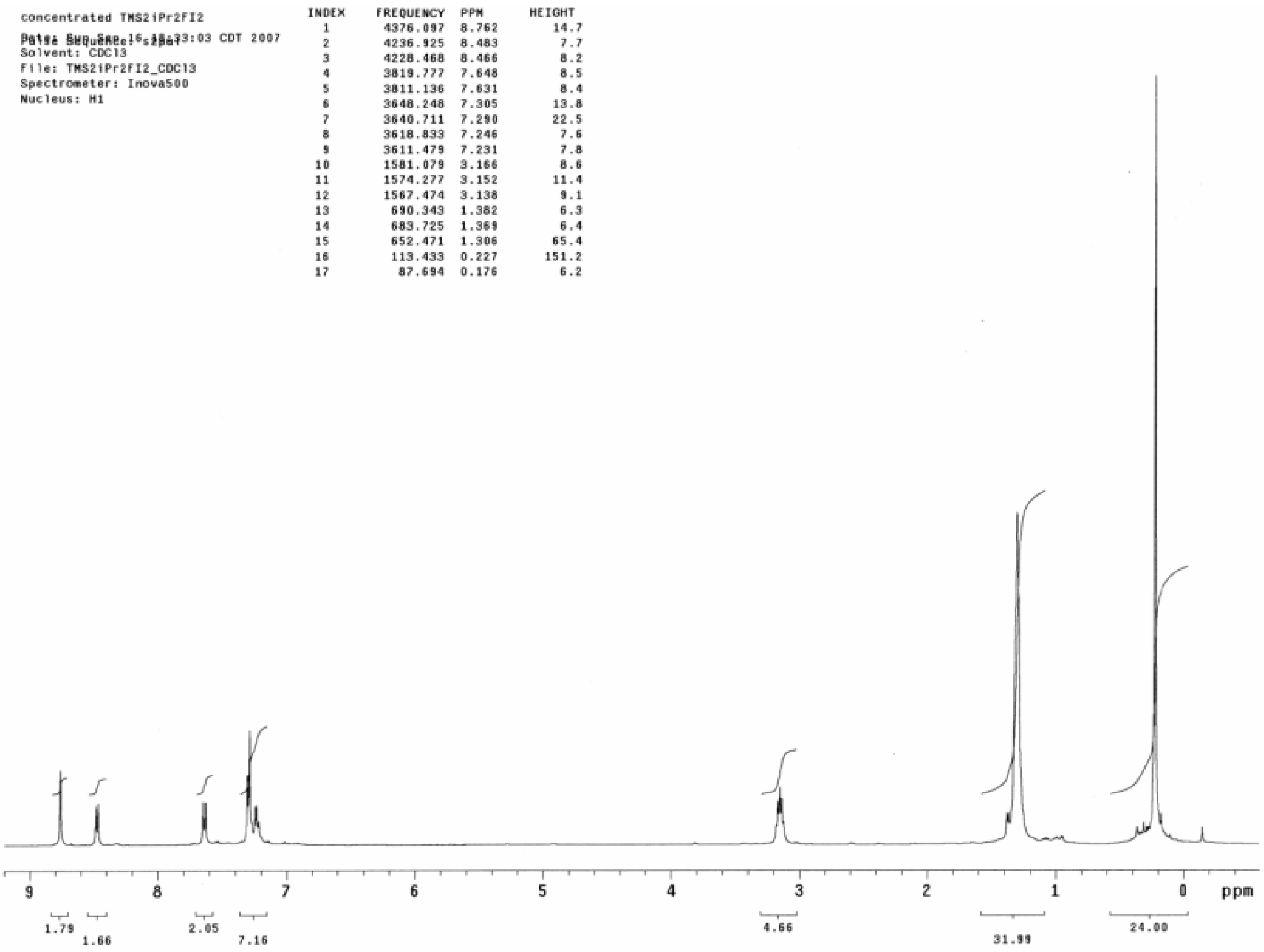
3	3475.970	8.676	14.1
4	3328.407	8.308	7.4
5	3320.414	8.288	10.6
6	3116.285	7.779	8.1
7	3103.373	7.746	9.5
8	2957.039	7.381	8.4
9	2949.661	7.363	9.3
10	2916.460	7.280	19.5
11	2909.081	7.261	22.2
12	2859.279	7.137	13.5
13	2851.901	7.119	31.9
14	2832.226	7.070	18.1
15	2823.618	7.048	19.7
16	2730.776	6.816	8.9
17	2722.168	6.795	10.6
18	2130.071	5.317	12.1
19	1250.226	3.121	14.4
20	1244.077	3.105	14.4
21	1189.971	2.970	14.1
22	1183.207	2.953	13.8
23	489.046	1.221	105.4
24	482.897	1.205	151.2
25	455.229	1.136	108.3
26	449.081	1.121	148.1

Figure S5.



concentrated TMS2iPr2FI2

Date: Sun Sep 16 [illegible]:33:03 CDT 2007
Pulse Sequence: s2pul
Solvent: CDCl3
File: TMS2iPr2FI2_CDCl3
Spectrometer: Inova500
Nucleus: H1

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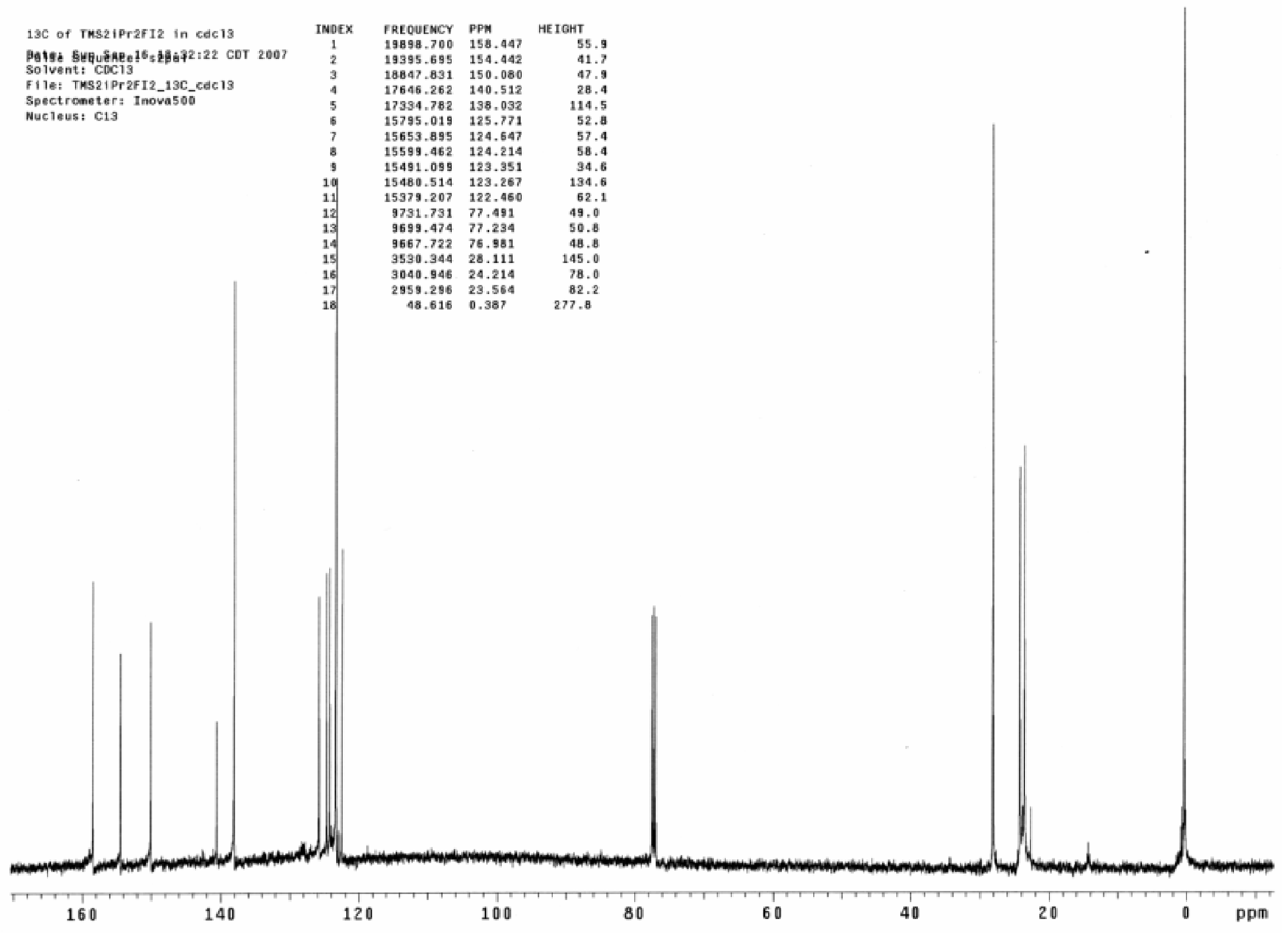
3	4228.468	8.466	8.2
4	3819.777	7.648	8.5
5	3811.136	7.631	8.4
6	3648.248	7.305	13.8
7	3640.711	7.290	22.5
8	3618.833	7.246	7.6
9	3611.479	7.231	7.8
10	1581.079	3.166	8.6
11	1574.277	3.152	11.4
12	1567.474	3.138	9.1
13	690.343	1.382	6.3
14	683.725	1.369	6.4
15	652.471	1.306	65.4
16	113.433	0.227	151.2
17	87.694	0.176	6.2

Figure S6.



13C of TMS2iPr2FI2 in cdcl3

Date: Sun Sep 16 18:32:22 CDT 2007
Pulse Sequence: s2pul
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Spectrometer: Inova500
Nucleus: C13

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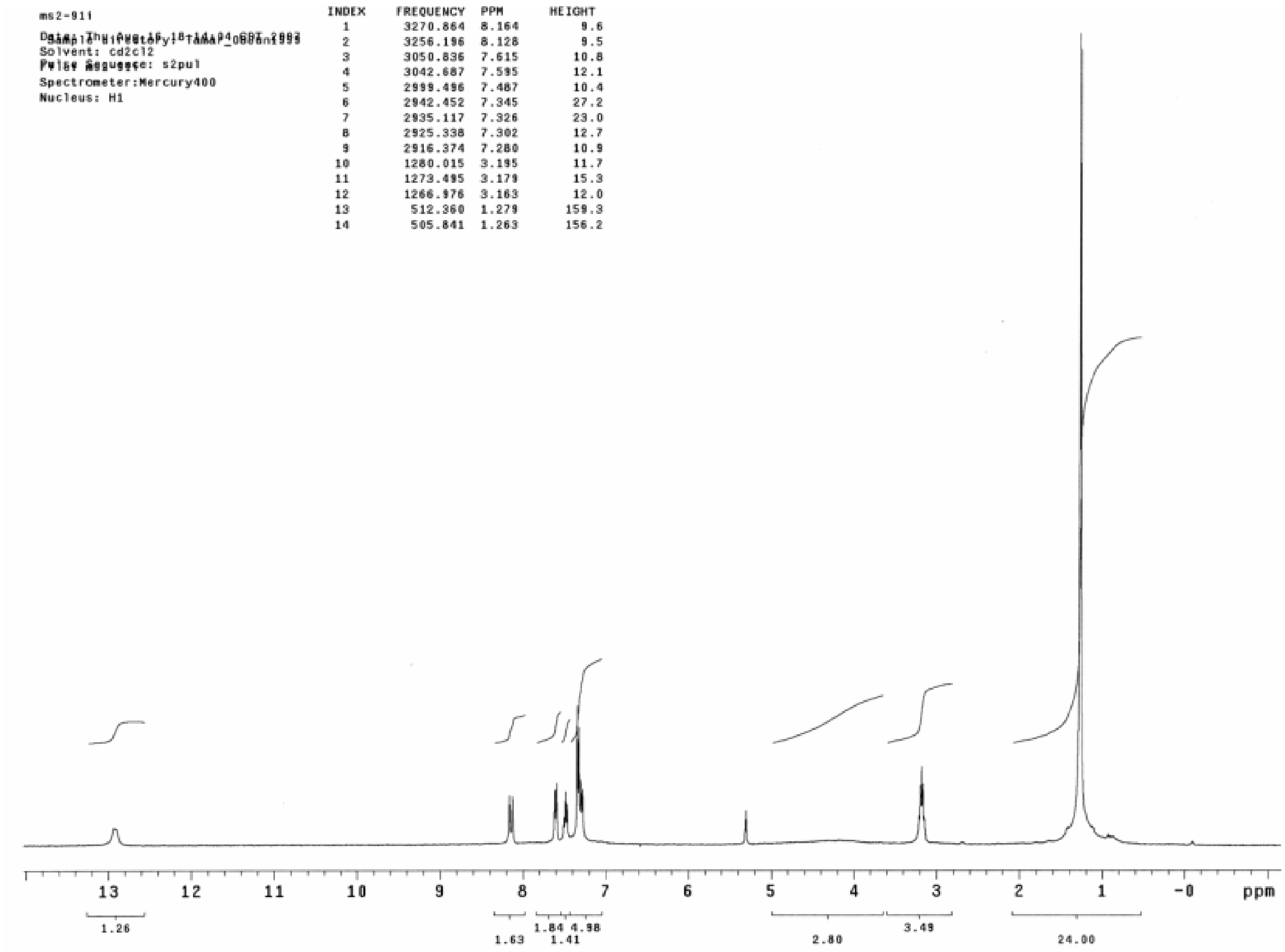
4	17646.262	140.512	28.4
5	17334.782	138.032	114.5
6	15795.019	125.771	52.8
7	15653.895	124.647	57.4
8	15599.462	124.214	58.4
9	15491.099	123.351	34.6
10	15480.514	123.267	134.6
11	15379.207	122.460	62.1
12	9731.731	77.491	49.0
13	9699.474	77.234	50.8
14	9667.722	76.981	48.8
15	3530.344	28.111	145.0
16	3040.946	24.214	78.0
17	2959.296	23.564	82.2
18	48.616	0.387	277.8

Figure S7.



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ms2-911

[illegible]
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Pulse Sequence: s2pul
Spectrometer:Mercury400
Nucleus: H1
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  5        2999.496  7.487       10.4
  6        2942.452  7.345       27.2
  7        2935.117  7.326       23.0
  8        2925.338  7.302       12.7
  9        2916.374  7.280       10.9
 10        1280.015  3.195       11.7
 11        1273.495  3.179       15.3
 12        1266.976  3.163       12.0
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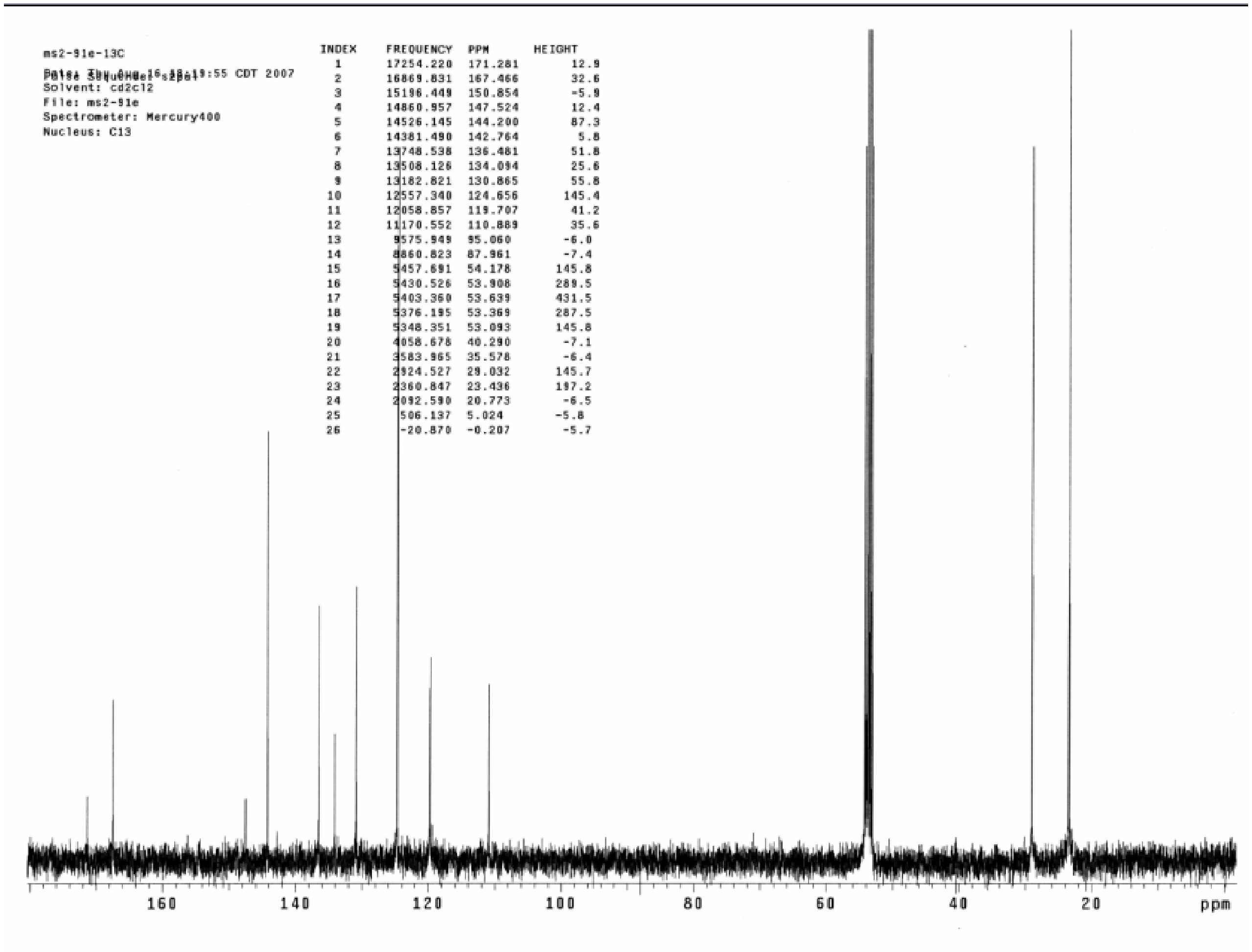
Figure S8.



ms2-91e-13C

Date: Thu Aug 16 18:19:55 CDT 2007
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File: ms2-91e
Spectrometer: Mercury400
Nucleus: C13

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4	14860.957	147.524	12.4
5	14526.145	144.200	87.3
6	14381.490	142.764	5.8
7	13748.538	136.481	51.8
8	13508.126	134.094	25.6
9	13182.821	130.865	55.8
10	12557.340	124.656	145.4
11	12058.857	119.707	41.2
12	11170.552	110.889	35.6
13	9575.949	95.060	-6.0
14	8860.823	87.961	-7.4
15	5457.691	54.178	145.8
16	5430.526	53.908	289.5
17	5403.360	53.639	431.5
18	5376.195	53.369	287.5
19	5348.351	53.093	145.8
20	4058.678	40.290	-7.1
21	3583.965	35.578	-6.4
22	2924.527	29.032	145.7
23	2360.847	23.436	197.2
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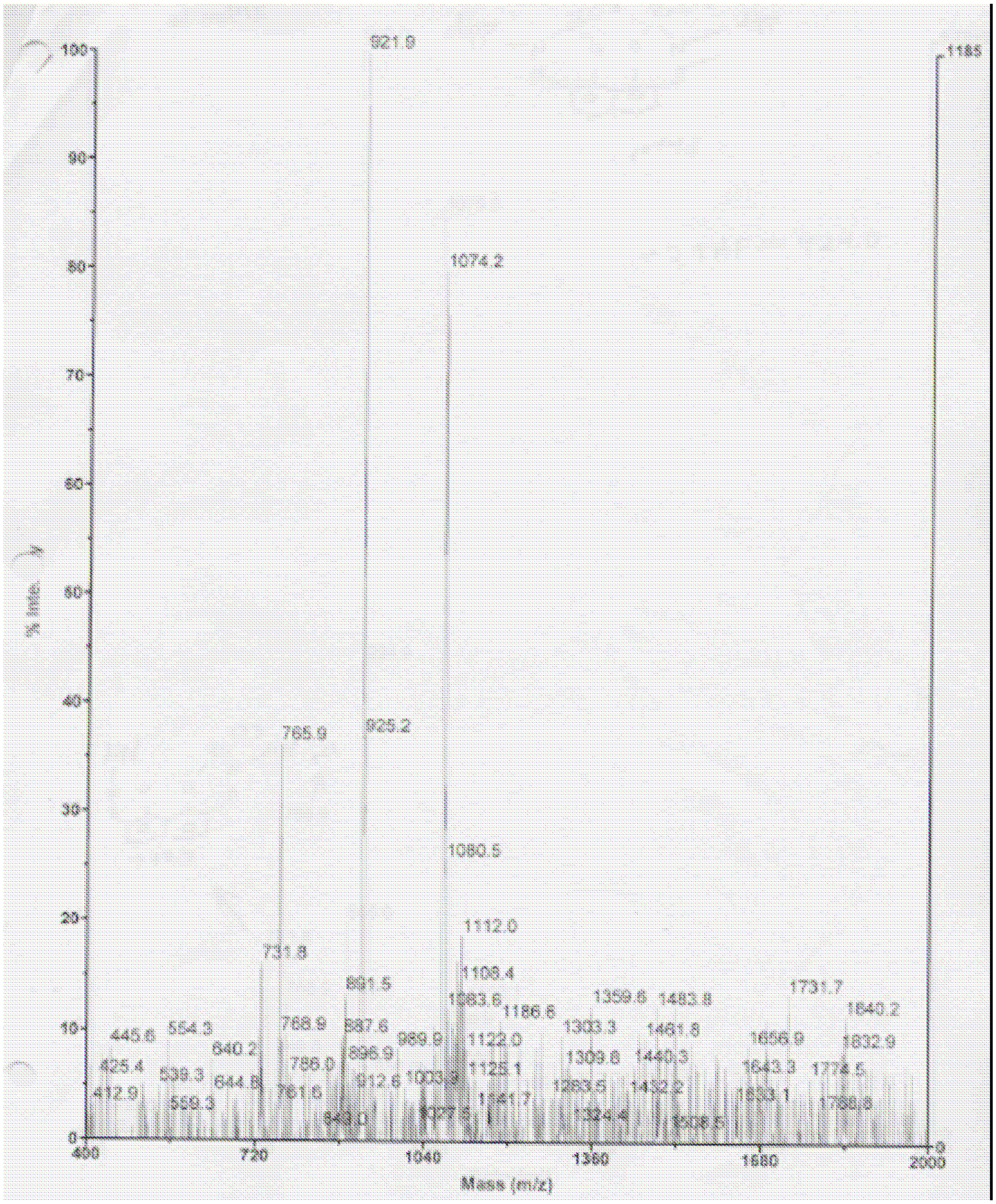
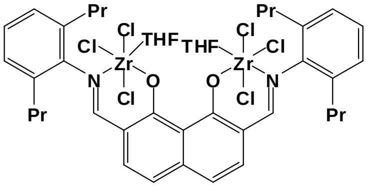
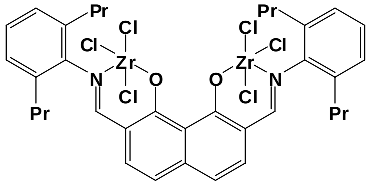
Figure S9.

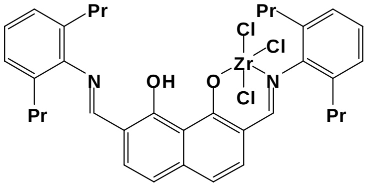




Chemical Formula: $C_{44}H_{54}Cl_6N_2O_4Zr_2$
Molecular Weight: 1070.08



Chemical Formula: $C_{36}H_{40}Cl_6N_2O_2Zr_2$
Exact Mass: 921.93



Chemical Formula: $C_{36}H_{41}Cl_3N_2O_2Zr$
Molecular Weight: 731.31

Table 1. Summary of the Crystal Structure Data for Complexes **1** and **2**.

Complex	**1**	**2**
Formula	$C_{36}H_{42}N_2O_2$	$C_{42}H_{58}N_2O_2Si_2$
Formula Weight	534.72	679.08
Crystal Dimensions (mm)	0.506 x 0.326 x 0.186	0.598 x 0.232 x .070
Crystal System	Monoclinic	Monoclinic
a, Å	10.0592(8)	26.914(6)
b, Å	23.0731(18)	9.765(2)
c, Å	13.9928(11)	16.289(4)
α, deg	90	90
β, deg	109.4350(10)	109.311(3)
γ, deg	90	90
V, $Å^3$	3062.6(4)	4040.1(15)
Space group	P2(1)/n	Cc
Z value	4	4
D_{calc}, mg/m^3	1.160	1.116
Temp K	153(2)	153(2)
μ, mm^{-1}	0.071	0.123
Radiation	MoKα	MoKα
2θ range, deg	1.77 to 28.91	1.60 to 28.78
Intensities (unique, (R_{int})	7436, 0.0299	9137, 0.0828
R	0.0616	0.0462
wR2	0.1614	0.1123

Table 2. Selected Bond Distances (Å) and Angles (deg) for compound **1**.

Distances

O(2)-C(20)	1.289(2)	N(2)-C(25)	1.438(2)
O(1)-C(14)	1.3472(19)	C(18)-C(17)	1.412(2)
C(20)-C(23)	1.436(2)	C(18)-C(21)	1.435(2)
C(20)-C(19)	1.449(2)	C(12)-N(1)	1.4286(19)
C(19)-C(18)	1.414(2)	C(22)-C(21)	1.353(2)
C(19)-C(14)	1.428(2)	C(22)-C(23)	1.426(2)
C(14)-C(15)	1.399(2)	N(1)-C(13)	1.279(2)
C(15)-C(16)	1.408(2)	C(16)-C(17)	1.372(2)
C(15)-C(13)	1.464(2)	C(23)-C(24)	1.409(2)
N(2)-C(24)	1.306(2)		

Angles

O(2)-C(20)-C(23)	121.84(13)	C(14)-C(15)-C(13)	119.49(14)
O(2)-C(20)-C(19)	120.39(13)	C(16)-C(15)-C(13)	121.77(13)
C(23)-C(20)-C(19)	117.76(14)	C(24)-N(2)-C(25)	128.79(14)
C(18)-C(19)-C(14)	119.48(13)	C(1)-C(12)-N(1)	120.36(14)
C(18)-C(19)-C(20)	120.29(13)	C(8)-C(12)-N(1)	117.93(14)
C(14)-C(19)-C(20)	120.23(14)	C(13)-N(1)-C(12)	116.33(13)
O(1)-C(14)-C(15)	119.10(14)	N(2)-C(24)-C(23)	122.35(14)
O(1)-C(14)-C(19)	120.50(13)	C(26)-C(25)-N(2)	118.65(14)
C(15)-C(14)-C(19)	120.40(14)	C(33)-C(25)-N(2)	117.90(14)
C(14)-C(15)-C(16)	118.74(14)	N(1)-C(13)-C(15)	122.81(15)

Table 3. Selected Bond Distances (Å) and Angles (deg) for compound **2**.

Distances

C(1)-N(1)	1.421(3)	C(22)-C(23)	1.412(3)
C(13)-N(1)	1.274(3)	C(23)-C(24)	1.367(3)
C(13)-C(14)	1.471(3)	C(24)-C(25)	1.415(3)
C(14)-C(17)	1.396(3)	C(25)-C(26)	1.386(3)
C(14)-C(15)	1.411(3)	C(25)-C(30)	1.470(3)
C(15)-C(16)	1.356(3)	C(26)-O(2)	1.361(2)
C(16)-C(22)	1.417(3)	C(27)-Si(2)	1.857(3)
C(17)-O(1)	1.370(2)	C(28)-Si(2)	1.843(3)
C(17)-C(21)	1.427(3)	C(29)-Si(2)	1.851(3)
C(18)-Si(1)	1.856(2)	C(30)-N(2)	1.262(3)
C(19)-Si(1)	1.846(2)	C(31)-N(2)	1.425(2)
C(20)-Si(1)	1.849(2)	O(1)-Si(1)	1.6822(15)
C(21)-C(22)	1.426(3)	O(2)-Si(2)	1.6765(15)
C(21)-C(26)	1.441(2)		

Angles

C(9)-C(1)-N(1)	117.48(17)	O(2)-C(26)-C(21)	120.45(17)
C(2)-C(1)-N(1)	120.93(18)	C(25)-C(26)-C(21)	120.67(16)
N(1)-C(13)-C(14)	120.58(18)	N(2)-C(30)-C(25)	121.31(17)
C(17)-C(14)-C(15)	119.24(17)	C(32)-C(31)-C(39)	121.03(17)
C(17)-C(14)-C(13)	121.30(17)	C(32)-C(31)-N(2)	119.48(18)
C(15)-C(14)-C(13)	119.44(17)	C(39)-C(31)-N(2)	119.21(17)
C(16)-C(15)-C(14)	121.30(18)	C(13)-N(1)-C(1)	121.56(18)
C(15)-C(16)-C(22)	120.59(18)	C(30)-N(2)-C(31)	120.28(17)
O(1)-C(17)-C(14)	118.46(17)	C(17)-O(1)-Si(1)	124.56(12)
O(1)-C(17)-C(21)	120.89(16)	C(26)-O(2)-Si(2)	127.82(12)
C(14)-C(17)-C(21)	120.64(17)	O(1)-Si(1)-C(19)	104.29(10)
C(22)-C(21)-C(17)	117.73(16)	O(1)-Si(1)-C(20)	111.42(9)
C(22)-C(21)-C(26)	117.54(17)	C(19)-Si(1)-C(20)	110.55(12)
C(17)-C(21)-C(26)	124.72(17)	O(1)-Si(1)-C(18)	109.54(9)
C(23)-C(22)-C(16)	120.16(17)	C(19)-Si(1)-C(18)	111.01(12)
C(23)-C(22)-C(21)	120.11(17)	C(20)-Si(1)-C(18)	109.92(11)
C(16)-C(22)-C(21)	119.73(18)	O(2)-Si(2)-C(28)	102.82(11)
C(24)-C(23)-C(22)	120.51(18)	O(2)-Si(2)-C(29)	109.12(11)
C(23)-C(24)-C(25)	121.04(18)	C(28)-Si(2)-C(29)	109.70(15)
C(26)-C(25)-C(24)	119.60(17)	O(2)-Si(2)-C(27)	111.30(11)
C(26)-C(25)-C(30)	121.15(16)	C(28)-Si(2)-C(27)	115.21(17)
C(24)-C(25)-C(30)	119.25(17)	C(29)-Si(2)-C(27)	108.49(14)
O(2)-C(26)-C(25)	118.88(16)		