Supporting Information

for the

Communication entitled

Chemoselective Deprotonations of a Cationic Zirconium Primary Amido Complex to Either a Neutral Zirconium Terminal Imido or A Non-interconverting Tautomer

authored by

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Experimental details including single crystal X-ray analyses of compounds **2a**, **5** and **6**

(41 pages).

Experimental

Manipulations were performed under an inert atmosphere of dinitrogen using standard Schlenk techniques or a Vacuum Atmospheres glovebox. Dry, oxygen free solvents were employed throughout. Diethyl ether (Et_2O) and pentane were distilled from sodium/benzophenone (with a few milliliters of tetraglyme being added to the pot in the case of pentane). Chlorobenzene was distilled from CaH_2. Benzene-d_6 was vacuum transferred from NaK (1:1 by wt) prior to being used for NMR spectroscopy. Chlorobenzene-d_5 was vacuum transferred from CaH_2 prior to being used for NMR spectroscopy. ^{1}H-NMR spectra were recorded at 400 MHz and ^{13}C-NMR spectra were recorded at 100 MHz using benzene-d_6 or chlorobenzene-d_5 as solvent. Compound **3** was obtained by the published procedure.[6] $[Ph_3C][B(C_6F_5)_4]$ was purchased from Strem, $EtNP(NMe_2)_2NP(NMe_2)_3$ was purchased from Aldrich, and both were used as received. Elemental analyses were performed by Midwest Microlab.

Preparation of compound 4. To a solution of 0.21 g (0.44 mmol) of **3** in 10 mL of Et_2O, 9.6 μL (0.92 mmol) of *tert*-butylamine was added. The reaction mixture was then kept at ambient temperature during overnight, whereupon it was evaporated to dryness. The residue was taken up into small (c.a. 10 mL) amount of pentane, passed through a small pad of Celite and crystallized at -30°C to give 0.12 g (67% yield) of **3** as white crystalline material. ^{1}H NMR (benzene-d_6, 25°C): δ 1.14, 1.21, (d, 6 H, J = 6.8 Hz, CHMe_2); 1.37 (s, 18 H, CMe_3); 1.64(s, 3 H, CMe); 1.97 (s, 15 H, C_5Me_5); 3.60 (sept, 2 H, J = 6.8 Hz, CHMe$_2$); 4.21 (s, 2 H, NH). ^{13}C {^{1}H} NMR (benzene-d_6, 25°C): δ 12.0 (C_5Me_5); 17.2 (CMe); 24.5, 24.6 (CHMe_2); 34.5 (CCH_3); 47.5

(*CH*Me_2); 54.6 (*C*CH_3); 117.0 (*C*$_5Me_5$); 174.0 (*C*Me). Anal. Calcd for $C_{26}H_{52}N_4Zr$: C, 61.0; H, 10.16; N, 10.94. Found: C, 60.69; H, 10.16; N, 10.38.

Preparation of compound 2a. To a solution of 0.044 g (0.093 mmol) of **4** in 10 mL of chlorobenzene, 0.086 g (0.093 mmol) of $[Ph_3C][B(C_6F_5)_4]$ was added. The reaction mixture was then kept at ambient temperature for 5 min, whereupon 30 mL of pentane was added. The mother liquor was decanted and the oily residue was redissolved in 3 mL of chlorobenzene and layered with 5 mL of pentane. Slow diffusion crystallization at -30°C gave 0.093 g (91% yield) of **2a** as yellow crystalline material. ^{1}H NMR (chlorobenzene-d_5, 25°C): δ 0.68, 0.83 (d, 6 H, J = 6.8 Hz, CH*Me*$_2$); 1.37 (s, 9 H, *CMe*$_3$); 1.79 (s, 15 H, C$_5$*Me*$_5$); 1.81 (s, 3 H, C*Me*); 3.45 (sept, 2 H, J = 6.8 Hz, *CH*Me_2); 4.74 (s, 1 H, NH). ^{13}C {^{1}H} NMR (chlorobenzene-d_5, 25°C): δ 11.3 (C$_5$*Me*$_5$); 12.9 (C*Me*); 24.0, 24.3 (CH*Me*$_2$); 31.7 (C*CH*$_3$); 49.5 (*CH*Me_2); 57.2 (*C*CH_3); 125.5 (*C*$_5Me_5$); 126.0-134.0 (*C*$_6F_5$) 175.2 (*C*Me). Anal. Calcd for $C_{46}H_{42}BF_{20}N_3Zr$: C, 49.38; H, 3.78; N, 3.76. Found: C, 49.17; H, 3.75; N, 3.79.

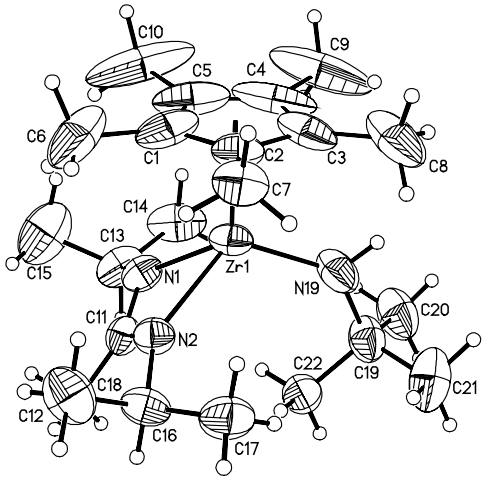
Preparation of compound 5. To a THF solution (5 mL) of 0.148 g (0.13 mmol) **2a**, 0.024 g (0.13 mmol) of $NaN(SiMe_3)_2$ was added at ambient temperature. After standing for 5 minutes all the volatiles were removed in *vacuo*. The residue was extracted 3 times by 3 mL of pentane, filtered through Celite and combined extracts were stripped to dryness and recrystallized from c.a. 3 mL of pentane yielding 0.036 g (78%) of **5** as red crystals. ^{1}H NMR (C_6D_6, 25°C): δ 1.11 (s, 9 H, *CMe*$_3$); 1.17, 1.25 (d, 6 H, J = 6.0 Hz, CH*Me*$_2$); 1.87 (s, 15 H, C$_5$*Me*$_5$); 3.39 (s, 2 H, C*H*$_2$); 3.69 (sept, 2 H, J = 6.0 Hz, *CH*Me_2); 4.74 (s, 1 H, NH). ^{13}C {^{1}H} NMR (C_6D_6, 25°C): δ 11.2 (C$_5$*Me*$_5$); 24.4, 26.1 (CH*Me*$_2$); 33.7 (C*CH*$_3$); 48.4 (*CH*Me_2); 55.3 (C*CH*$_2$); 56.5 (*C*CH_3); 120.2 (*C*$_5Me_5$); 146.5 (*C*CH_2). Anal. Calcd for $C_{22}H_{41}N_3Zr$: C, 60.02; H, 9.42; N, 9.56. Found: C, 59.55; H, 9.27; N, 9.41.

Preparation of compound 6. To a THF solution (5 mL) of 0.05 g (0.045 mmol) **2a**, 15 μl (0.045 mmol) of P_2-Et phosphazene base was added at ambient temperature. After standing for 5 minutes all the volatiles were removed *in vacuo*. The residue was extracted 3 times by 3 mL of pentane, filtered through Celite and combined extracts were stripped to dryness yielding 0.019 g (94%) of **6** as a crude material. Due to the high solubility of both the protonated P_2-Et phosphazene base and 5, analytically pure material could not be isolated and therefore elemental analysis of **6** was not possible. However, ^{1}H NMR confirmed that HP_2Et appeared to be the only contaminant and therefore, **6** was used as is for further studies. Single crystals of **6** could be obtained through slow diffusion and subjected to single crystal X-ray analysis ^{1}H NMR (C_6D_6, 25°C): δ 0.96, 1.11 (d, 6 H, J = 6.0 Hz, CH*Me*$_2$); 1.27 (s, 9 H, *CMe*$_3$); 1.52 (s, 3 H, C*Me*); 1.69 (s, 15 H, C_5*Me*$_5$); 3.42 (sept, 2 H, J = 6.0 Hz, *CH*Me$_2$). ^{13}C {^{1}H} NMR (C_6D_6, 25°C): δ 11.7 (C_5*Me*$_5$); 25.1, 26.0 (CH*Me*$_2$); 34.5 (C*Me*); 35.1 (C*CH*$_3$); 47.7 (*CH*Me$_2$); 61.0 (*C*CH$_3$); 118.1 (*C*$_5$Me$_5$); 165.8 (*C*Me).

Preparation of compound 7. To a solution of 0.04 g (0.036 mmol) of **2a** in 2 mL of chlorobenzene, 4 μl (0.036 mmol) of ^{t}BuNH$_2$ was added. The reaction mixture was then kept at ambient temperature for 5 min, whereupon it was layered with 5 mL of pentane. Slow diffusion crystallization at -30°C gave 0.041 g (97% yield) of **7** as yellow crystalline material. ^{1}H NMR (chlorobenzene-d_5, 25°C): δ 1.1, 1.16 (s, 9 H, *CMe*$_3$); 1.10 (d, 12 H, J = 6.0 Hz, CH*Me*$_2$); 1.82 (s, 15 H, C_5*Me*$_5$); 1.89 (s, 3 H, C*Me*); 2.23 (s, 2 H NH$_2$); 3.39 (sept, 2 H, J = 6.0 Hz, *CH*Me$_2$); 5.55 (s, 1 H, NH). ^{13}C {^{1}H} NMR (chlorobenzene-d_5, 25°C): δ 10.5 (C_5*Me*$_5$); 16.6 (C*Me*); 22.2, 23.0 (CH*Me*$_2$); 29.5, 31.8 (C*CH*$_3$); 47.4 (*CH*Me$_2$); 57.7 (*C*CH$_3$); 121.3 (*C*$_5$Me$_5$); 124.0-150.0 (*C*$_6$F$_5$) 178.8 (*C*Me). Anal. Calcd for $C_{50}H_{53}BF_{20}N_4Zr$: C, 50.38; H, 4.48; N, 4.70. Found: C, 50.33; H, 4.46; N, 4.64.

Crystalographic analysis of compound 2a

A yellow cut block with approximate orthogonal dimensions 0.493 x 0.169 x 0.162mm^3 was placed and optically centered on the Bruker SMART CCD system at –100°C. The initial unit cell was indexed using a least-squares analysis of a random set of reflections collected from three series of 0.3° wide ω-scans, 10 seconds per frame, and 25 frames per series that were well distributed in reciprocal space. Data frames were collected [MoKα] with 0.3° wide ω-scans, 14 seconds per frame and 606 frames per series. Five ω-data series were collected at varying φ angles (φ=0°, 72°, 144°, 216°, 288°), with a partial repeat of the first series, 200 frames, for decay purposes. The crystal to detector distance was 4.831cm, thus providing a complete sphere of data to $2\theta_{max}$=55.06°. A total of 38961 reflections were collected and corrected for Lorentz and polarization effects and absorption using Blessing's method as incorporated into the program SADABS[1,2] with 10984 unique.



Structural determination and Refinement:

All crystallographic calculations were performed on a Personal computer (PC) with a Pentium 3.06GHz processor and 512MB of extended memory. The SHELXTL[3] program package was implemented to determine the probable space group and set up the initial files. System symmetry, lack of systematic absences and intensity statistics indicated the centrosymmetric triclinic space group P-1 (no. 2). The structure was determined by direct methods with the successful location of parts of two molecules within the asymmetric unit using the program XS[4]. A unique molecule of interest and its counter-ion was located. The structure was refined with XL[5]. The 38961 data collected were truncated to $2\theta_{max}$=55.00° resulting in 38934 data that were further merged during least-squares refinement to 10973 unique data [R(int)=0.0275]. A series of least-squares difference-Fourier cycles were required to locate the remaining non-hydrogen. All non-hydrogen atoms were refined anisotropically. All hydrogen atoms were initially placed in calculated positions except for the hydrogen atom attached to N(19) which was located directly from a difference-Fourier map. During the final convergence stages all of the hydrogen atoms were allowed to refine freely but for those attached to the pentamethylcyclopentadienyl ligand and these remained idealized due to the large amount of libration within that ligand. A centroid was also calculated for the pentamethylcyclopentadienyl ligand. The final structure was refined to convergence [$\Delta/\sigma \leq 0.001$] with R(F)=5.44%, wR(F^2)=11.65%, GOF=1.045 for all 10973 unique reflections [R(F)=4.01%, wR(F^2)=10.82% for those 8706 data with Fo > 4σ(Fo)]. The final difference-Fourier map was featureless indicating that the structure is both correct and complete.

The function minimized during the full-matrix least-squares refinement was $\Sigma w(Fo^2-Fc^2)$ where $w=1/[\sigma^2(Fo^2)+(0.0677*P)^2+0.5202*P]$ and $P=(\max(Fo^2,0)+2*Fc^2)/3$. An empirical correction for extinction was also attempted but found to be negative and therefore not applied.

Table 1. Crystal data and structure refinement for **2a**.

Empirical formula	C46 H42 B F20 N3 Zr	
Formula weight	1118.86	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system	Triclinic	
Space group	P-1	
Unit cell dimensions	a = 12.3139(4) Å	α= 91.7770(10)°.
	b = 14.1247(4) Å	β= 104.1750(10)°.
	c = 14.3103(5) Å	γ = 96.6060(10)°.
Volume	2392.66(13) $Å^3$	
Z	2	
Density (calculated)	1.553 Mg/m^3	
Absorption coefficient	0.344 mm^{-1}	
F(000)	1128	
Crystal size	0.49 x 0.17 x 0.16 mm^3	
Theta range for data collection	1.96 to 27.50°.	
Index ranges	-15<=h<=15, -18<=k<=18, -18<=l<=18	
Reflections collected	38934	
Independent reflections	10973 [R(int) = 0.0275]	
Completeness to theta = 27.50°	99.8 %	
Absorption correction	Empirical, SADABS (multi-scan)	
Max. and min. transmission	0.9463 and 0.8485	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	10973 / 3 / 771	
Goodness-of-fit on F^2	1.045	
Final R indices [I>2sigma(I)]	R1 = 0.0401, wR2 = 0.1082 [8706 Data]	
R indices (all data)	R1 = 0.0544, wR2 = 0.1165	
Largest diff. peak and hole	0.565 and -0.462 $e.Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **2a**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Zr(1)	9443(1)	2762(1)	2675(1)	43(1)
N(1)	8165(2)	1586(1)	2057(1)	45(1)
N(2)	7741(2)	3084(1)	2158(1)	43(1)
CNT1	10808(1)	3286(1)	2021(1)	0
C(1)	10141(3)	3320(2)	1273(2)	68(1)
C(2)	10516(2)	4067(2)	1994(2)	48(1)
C(3)	11314(2)	3744(2)	2770(2)	65(1)
C(4)	11403(3)	2777(2)	2491(4)	97(1)
C(5)	10667(3)	2523(2)	1577(3)	87(1)
C(6)	9310(4)	3364(3)	307(2)	97(1)
C(7)	10228(2)	5073(2)	1915(2)	56(1)
C(8)	11969(2)	4339(3)	3642(3)	93(1)
C(9)	12170(4)	2132(4)	3085(6)	159(3)
C(10)	10593(6)	1603(2)	989(5)	168(3)
C(11)	7349(2)	2147(2)	2012(1)	41(1)
C(12)	6130(2)	1764(2)	1870(2)	57(1)
C(13)	7933(3)	534(2)	1987(2)	59(1)
C(14)	8970(3)	139(2)	2559(3)	75(1)
C(15)	7605(6)	157(3)	929(3)	101(2)
C(16)	7015(2)	3831(2)	2209(2)	51(1)
C(17)	7656(3)	4606(2)	2953(2)	62(1)
C(18)	6624(3)	4231(3)	1224(3)	75(1)
N(19)	10016(2)	2766(2)	4118(2)	61(1)
C(19)	9480(2)	2532(2)	4918(2)	58(1)
C(20)	10022(3)	1734(3)	5472(3)	74(1)
C(21)	9620(4)	3422(3)	5582(2)	86(1)
C(22)	8222(2)	2208(2)	4496(2)	55(1)
C(31)	6136(2)	2035(1)	7734(1)	35(1)
C(32)	7244(2)	2392(2)	7758(2)	42(1)
C(33)	8152(2)	1891(2)	8064(2)	51(1)
C(34)	7985(2)	992(2)	8367(2)	56(1)

C(35)	6905(2)	584(2)	8345(2)	48(1)
C(36)	6027(2)	1108(1)	8028(1)	38(1)
C(37)	4111(2)	2536(1)	7979(1)	34(1)
C(38)	4335(2)	2394(1)	8958(1)	37(1)
C(39)	3582(2)	2508(2)	9523(1)	41(1)
C(40)	2547(2)	2786(2)	9113(2)	43(1)
C(41)	2279(2)	2944(1)	8148(2)	40(1)
C(42)	3049(2)	2820(1)	7616(1)	35(1)
C(43)	4488(2)	1912(1)	6239(1)	36(1)
C(44)	3558(2)	1217(2)	6013(1)	43(1)
C(45)	3244(2)	639(2)	5171(2)	54(1)
C(46)	3879(2)	724(2)	4506(2)	59(1)
C(47)	4812(2)	1393(2)	4689(2)	55(1)
C(48)	5091(2)	1967(2)	5534(2)	44(1)
C(49)	5253(2)	3705(1)	7160(2)	44(1)
C(50)	5866(2)	4288(2)	7953(2)	56(1)
C(51)	6012(3)	5281(2)	7965(3)	82(1)
C(52)	5504(3)	5718(2)	7159(4)	89(1)
C(53)	4873(3)	5176(2)	6360(3)	76(1)
C(54)	4777(2)	4188(2)	6374(2)	55(1)
B(31)	5001(2)	2540(2)	7277(2)	34(1)
F(32)	7495(1)	3269(1)	7465(1)	54(1)
F(33)	9203(1)	2287(1)	8066(1)	73(1)
F(34)	8872(1)	498(1)	8673(1)	84(1)
F(35)	6727(1)	-311(1)	8610(1)	64(1)
F(36)	4982(1)	659(1)	7995(1)	45(1)
F(38)	5334(1)	2133(1)	9440(1)	47(1)
F(39)	3854(1)	2338(1)	10463(1)	57(1)
F(40)	1814(1)	2907(1)	9647(1)	60(1)
F(41)	1275(1)	3221(1)	7737(1)	54(1)
F(42)	2729(1)	3009(1)	6673(1)	43(1)
F(44)	2892(1)	1051(1)	6627(1)	51(1)
F(45)	2307(2)	-3(1)	4988(1)	79(1)
F(46)	3586(2)	167(1)	3683(1)	88(1)
F(47)	5450(2)	1493(1)	4048(1)	80(1)
F(48)	6027(1)	2608(1)	5676(1)	56(1)

F(50)	6348(1)	3903(1)	8781(1)	67(1)
F(51)	6633(2)	5792(1)	8765(2)	114(1)
F(52)	5613(2)	6681(1)	7176(2)	130(1)
F(53)	4344(2)	5603(1)	5585(2)	104(1)
F(54)	4126(1)	3724(1)	5551(1)	62(1)

Table 3. Bond lengths [Å] and angles [°] for **2a**

Zr(1)-N(19)	2.012(2)	C(7)-H(7A)	0.9600
Zr(1)-N(2)	2.1516(17)	C(7)-H(7B)	0.9600
Zr(1)-N(1)	2.1549(18)	C(7)-H(7C)	0.9600
Zr(1)-CNT1	2.1850(11)	C(8)-H(8A)	0.9600
Zr(1)-C(5)	2.468(3)	C(8)-H(8B)	0.9600
Zr(1)-C(1)	2.483(2)	C(8)-H(8C)	0.9600
Zr(1)-C(4)	2.488(2)	C(9)-H(9A)	0.9600
Zr(1)-C(2)	2.505(2)	C(9)-H(9B)	0.9600
Zr(1)-C(3)	2.520(2)	C(9)-H(9C)	0.9600
N(1)-C(11)	1.341(3)	C(10)-H(10A)	0.9600
N(1)-C(13)	1.476(3)	C(10)-H(10B)	0.9600
N(2)-C(11)	1.347(3)	C(10)-H(10C)	0.9600
N(2)-C(16)	1.470(3)	C(11)-C(12)	1.498(3)
C(1)-C(5)	1.391(4)	C(12)-H(12A)	0.92(5)
C(1)-C(2)	1.406(4)	C(12)-H(12B)	1.05(4)
C(1)-C(6)	1.514(5)	C(12)-H(12C)	0.84(5)
C(2)-C(3)	1.417(3)	C(13)-C(14)	1.514(4)
C(2)-C(7)	1.505(3)	C(13)-C(15)	1.529(5)
C(3)-C(4)	1.435(5)	C(13)-H(13)	1.03(3)
C(3)-C(8)	1.483(5)	C(14)-H(14A)	0.96(3)
C(4)-C(5)	1.406(6)	C(14)-H(14B)	0.97(3)
C(4)-C(9)	1.515(5)	C(14)-H(14C)	1.00(3)
C(5)-C(10)	1.508(5)	C(15)-H(15A)	0.87(4)
C(6)-H(6A)	0.9600	C(15)-H(15B)	0.89(4)
C(6)-H(6B)	0.9600	C(15)-H(15C)	1.04(4)
C(6)-H(6C)	0.9600	C(16)-C(17)	1.508(4)

C(16)-C(18)	1.523(4)	C(37)-B(31)	1.657(3)
C(16)-H(16)	0.98(3)	C(38)-F(38)	1.353(2)
C(17)-H(17A)	1.00(3)	C(38)-C(39)	1.390(3)
C(17)-H(17B)	0.95(3)	C(39)-F(39)	1.342(2)
C(17)-H(17C)	0.98(3)	C(39)-C(40)	1.373(3)
C(18)-H(18A)	1.05(3)	C(40)-F(40)	1.340(2)
C(18)-H(18B)	1.02(3)	C(40)-C(41)	1.370(3)
C(18)-H(18C)	0.90(4)	C(41)-F(41)	1.342(2)
N(19)-C(19)	1.482(3)	C(41)-C(42)	1.376(3)
N(19)-H(19)	0.85(3)	C(42)-F(42)	1.355(2)
C(19)-C(21)	1.519(4)	C(43)-C(44)	1.385(3)
C(19)-C(20)	1.522(4)	C(43)-C(48)	1.391(3)
C(19)-C(22)	1.528(3)	C(43)-B(31)	1.650(3)
C(20)-H(20A)	1.03(4)	C(44)-F(44)	1.350(2)
C(20)-H(20B)	0.88(3)	C(44)-C(45)	1.381(3)
C(20)-H(20C)	1.07(3)	C(45)-F(45)	1.348(3)
C(21)-H(21A)	1.06(3)	C(45)-C(46)	1.372(4)
C(21)-H(21B)	1.02(4)	C(46)-F(46)	1.343(3)
C(21)-H(21C)	1.01(4)	C(46)-C(47)	1.367(4)
C(22)-H(22A)	0.96(3)	C(47)-F(47)	1.347(3)
C(22)-H(22B)	0.93(3)	C(47)-C(48)	1.381(3)
C(22)-H(22C)	1.01(3)	C(48)-F(48)	1.351(3)
C(31)-C(36)	1.390(3)	C(49)-C(54)	1.377(3)
C(31)-C(32)	1.390(3)	C(49)-C(50)	1.384(3)
C(31)-B(31)	1.644(3)	C(49)-B(31)	1.659(3)
C(32)-F(32)	1.347(2)	C(50)-F(50)	1.349(3)
C(32)-C(33)	1.380(3)	C(50)-C(51)	1.393(4)
C(33)-F(33)	1.348(3)	C(51)-F(51)	1.345(4)
C(33)-C(34)	1.364(4)	C(51)-C(52)	1.371(5)
C(34)-F(34)	1.354(3)	C(52)-F(52)	1.350(3)
C(34)-C(35)	1.380(4)	C(52)-C(53)	1.368(5)
C(35)-F(35)	1.340(3)	C(53)-F(53)	1.340(4)
C(35)-C(36)	1.373(3)	C(53)-C(54)	1.387(3)
C(36)-F(36)	1.357(2)	C(54)-F(54)	1.352(3)
C(37)-C(38)	1.385(3)	N(19)-Zr(1)-N(2)	115.69(8)
C(37)-C(42)	1.394(3)	N(19)-Zr(1)-N(1)	113.88(8)

N(2)-Zr(1)-N(1)	62.87(7)	C(13)-N(1)-Zr(1)	142.87(16)
N(19)-Zr(1)-CNT1	110.49(8)	C(11)-N(2)-C(16)	123.19(18)
N(2)-Zr(1)-CNT1	122.85(6)	C(11)-N(2)-Zr(1)	90.95(12)
N(1)-Zr(1)-CNT1	123.80(7)	C(16)-N(2)-Zr(1)	142.26(15)
N(19)-Zr(1)-C(5)	121.93(13)	C(5)-C(1)-C(2)	109.4(3)
N(2)-Zr(1)-C(5)	122.22(13)	C(5)-C(1)-C(6)	124.5(3)
N(1)-Zr(1)-C(5)	96.12(11)	C(2)-C(1)-C(6)	126.1(3)
CNT1-Zr(1)-C(5)	29.25(7)	C(5)-C(1)-Zr(1)	73.09(15)
N(19)-Zr(1)-C(1)	138.37(10)	C(2)-C(1)-Zr(1)	74.48(13)
N(2)-Zr(1)-C(1)	96.91(9)	C(6)-C(1)-Zr(1)	119.6(2)
N(1)-Zr(1)-C(1)	103.52(8)	C(1)-C(2)-C(3)	108.4(2)
CNT1-Zr(1)-C(1)	28.50(8)	C(1)-C(2)-C(7)	126.1(2)
C(5)-Zr(1)-C(1)	32.63(10)	C(3)-C(2)-C(7)	125.2(2)
N(19)-Zr(1)-C(4)	90.24(14)	C(1)-C(2)-Zr(1)	72.79(13)
N(2)-Zr(1)-C(4)	150.73(11)	C(3)-C(2)-Zr(1)	74.19(12)
N(1)-Zr(1)-C(4)	120.60(12)	C(7)-C(2)-Zr(1)	124.28(15)
CNT1-Zr(1)-C(4)	28.57(6)	C(2)-C(3)-C(4)	105.8(3)
C(5)-Zr(1)-C(4)	32.94(13)	C(2)-C(3)-C(8)	125.2(3)
C(1)-Zr(1)-C(4)	53.87(13)	C(4)-C(3)-C(8)	128.8(3)
N(19)-Zr(1)-C(2)	111.39(9)	C(2)-C(3)-Zr(1)	73.04(12)
N(2)-Zr(1)-C(2)	101.12(7)	C(4)-C(3)-Zr(1)	72.15(14)
N(1)-Zr(1)-C(2)	134.49(7)	C(8)-C(3)-Zr(1)	123.4(2)
CNT1-Zr(1)-C(2)	28.56(5)	C(5)-C(4)-C(3)	109.1(3)
C(5)-Zr(1)-C(2)	54.64(8)	C(5)-C(4)-C(9)	125.4(5)
C(1)-Zr(1)-C(2)	32.74(8)	C(3)-C(4)-C(9)	125.4(5)
C(4)-Zr(1)-C(2)	54.22(8)	C(5)-C(4)-Zr(1)	72.75(16)
N(19)-Zr(1)-C(3)	84.07(9)	C(3)-C(4)-Zr(1)	74.55(13)
N(2)-Zr(1)-C(3)	131.15(7)	C(9)-C(4)-Zr(1)	119.2(2)
N(1)-Zr(1)-C(3)	151.33(9)	C(1)-C(5)-C(4)	107.3(3)
CNT1-Zr(1)-C(3)	29.00(8)	C(1)-C(5)-C(10)	126.7(5)
C(5)-Zr(1)-C(3)	55.28(12)	C(4)-C(5)-C(10)	125.5(4)
C(1)-Zr(1)-C(3)	54.46(10)	C(1)-C(5)-Zr(1)	74.28(14)
C(4)-Zr(1)-C(3)	33.30(11)	C(4)-C(5)-Zr(1)	74.31(17)
C(2)-Zr(1)-C(3)	32.77(8)	C(10)-C(5)-Zr(1)	123.4(2)
C(11)-N(1)-C(13)	122.4(2)	C(1)-C(6)-H(6A)	109.5
C(11)-N(1)-Zr(1)	90.98(13)	C(1)-C(6)-H(6B)	109.5

H(6A)-C(6)-H(6B)	109.5	H(12B)-C(12)-H(12C)	103(3)
C(1)-C(6)-H(6C)	109.5	N(1)-C(13)-C(14)	108.4(2)
H(6A)-C(6)-H(6C)	109.5	N(1)-C(13)-C(15)	110.4(2)
H(6B)-C(6)-H(6C)	109.5	C(14)-C(13)-C(15)	111.9(3)
C(2)-C(7)-H(7A)	109.5	N(1)-C(13)-H(13)	106.8(14)
C(2)-C(7)-H(7B)	109.5	C(14)-C(13)-H(13)	108.8(14)
H(7A)-C(7)-H(7B)	109.5	C(15)-C(13)-H(13)	110.3(14)
C(2)-C(7)-H(7C)	109.5	C(13)-C(14)-H(14A)	112.0(18)
H(7A)-C(7)-H(7C)	109.5	C(13)-C(14)-H(14B)	111.2(19)
H(7B)-C(7)-H(7C)	109.5	H(14A)-C(14)-H(14B)	105(3)
C(3)-C(8)-H(8A)	109.5	C(13)-C(14)-H(14C)	106.9(18)
C(3)-C(8)-H(8B)	109.5	H(14A)-C(14)-H(14C)	108(3)
H(8A)-C(8)-H(8B)	109.5	H(14B)-C(14)-H(14C)	114(3)
C(3)-C(8)-H(8C)	109.5	C(13)-C(15)-H(15A)	108(3)
H(8A)-C(8)-H(8C)	109.5	C(13)-C(15)-H(15B)	100(3)
H(8B)-C(8)-H(8C)	109.5	H(15A)-C(15)-H(15B)	110(4)
C(4)-C(9)-H(9A)	109.5	C(13)-C(15)-H(15C)	100(2)
C(4)-C(9)-H(9B)	109.5	H(15A)-C(15)-H(15C)	129(4)
H(9A)-C(9)-H(9B)	109.5	H(15B)-C(15)-H(15C)	107(4)
C(4)-C(9)-H(9C)	109.5	N(2)-C(16)-C(17)	109.1(2)
H(9A)-C(9)-H(9C)	109.5	N(2)-C(16)-C(18)	110.6(2)
H(9B)-C(9)-H(9C)	109.5	C(17)-C(16)-C(18)	111.1(2)
C(5)-C(10)-H(10A)	109.5	N(2)-C(16)-H(16)	110.1(15)
C(5)-C(10)-H(10B)	109.5	C(17)-C(16)-H(16)	107.8(15)
H(10A)-C(10)-H(10B)	109.5	C(18)-C(16)-H(16)	108.2(14)
C(5)-C(10)-H(10C)	109.5	C(16)-C(17)-H(17A)	111.4(16)
H(10A)-C(10)-H(10C)	109.5	C(16)-C(17)-H(17B)	110.1(18)
H(10B)-C(10)-H(10C)	109.5	H(17A)-C(17)-H(17B)	111(2)
N(1)-C(11)-N(2)	113.36(19)	C(16)-C(17)-H(17C)	106.6(16)
N(1)-C(11)-C(12)	123.1(2)	H(17A)-C(17)-H(17C)	107(2)
N(2)-C(11)-C(12)	123.4(2)	H(17B)-C(17)-H(17C)	111(2)
C(11)-C(12)-H(12A)	114(3)	C(16)-C(18)-H(18A)	108.7(16)
C(11)-C(12)-H(12B)	104(2)	C(16)-C(18)-H(18B)	111.4(18)
H(12A)-C(12)-H(12B)	102(3)	H(18A)-C(18)-H(18B)	110(2)
C(11)-C(12)-H(12C)	112(3)	C(16)-C(18)-H(18C)	107(3)
H(12A)-C(12)-H(12C)	119(4)	H(18A)-C(18)-H(18C)	113(3)

H(18B)-C(18)-H(18C)	108(3)	C(34)-C(33)-C(32)	119.8(2)
C(19)-N(19)-Zr(1)	134.25(16)	F(34)-C(34)-C(33)	120.1(2)
C(19)-N(19)-H(19)	106(2)	F(34)-C(34)-C(35)	120.2(2)
Zr(1)-N(19)-H(19)	120(2)	C(33)-C(34)-C(35)	119.7(2)
N(19)-C(19)-C(21)	109.0(2)	F(35)-C(35)-C(36)	121.4(2)
N(19)-C(19)-C(20)	109.9(3)	F(35)-C(35)-C(34)	120.1(2)
C(21)-C(19)-C(20)	110.5(3)	C(36)-C(35)-C(34)	118.4(2)
N(19)-C(19)-C(22)	108.98(19)	F(36)-C(36)-C(35)	115.78(19)
C(21)-C(19)-C(22)	109.1(3)	F(36)-C(36)-C(31)	119.08(16)
C(20)-C(19)-C(22)	109.4(2)	C(35)-C(36)-C(31)	125.13(19)
C(19)-C(20)-H(20A)	110(2)	C(38)-C(37)-C(42)	112.86(16)
C(19)-C(20)-H(20B)	113(2)	C(38)-C(37)-B(31)	127.43(16)
H(20A)-C(20)-H(20B)	108(3)	C(42)-C(37)-B(31)	119.20(16)
C(19)-C(20)-H(20C)	109.5(14)	F(38)-C(38)-C(37)	121.28(16)
H(20A)-C(20)-H(20C)	113(3)	F(38)-C(38)-C(39)	114.66(16)
H(20B)-C(20)-H(20C)	103(3)	C(37)-C(38)-C(39)	124.06(17)
C(19)-C(21)-H(21A)	114.3(16)	F(39)-C(39)-C(40)	119.69(18)
C(19)-C(21)-H(21B)	107(2)	F(39)-C(39)-C(38)	120.50(18)
H(21A)-C(21)-H(21B)	104(3)	C(40)-C(39)-C(38)	119.80(18)
C(19)-C(21)-H(21C)	109(2)	F(40)-C(40)-C(41)	120.50(18)
H(21A)-C(21)-H(21C)	112(3)	F(40)-C(40)-C(39)	120.66(18)
H(21B)-C(21)-H(21C)	111(3)	C(41)-C(40)-C(39)	118.83(18)
C(19)-C(22)-H(22A)	107.5(17)	F(41)-C(41)-C(40)	119.65(17)
C(19)-C(22)-H(22B)	109.4(15)	F(41)-C(41)-C(42)	120.91(18)
H(22A)-C(22)-H(22B)	114(2)	C(40)-C(41)-C(42)	119.44(18)
C(19)-C(22)-H(22C)	112.0(14)	F(42)-C(42)-C(41)	115.75(16)
H(22A)-C(22)-H(22C)	109(2)	F(42)-C(42)-C(37)	119.24(16)
H(22B)-C(22)-H(22C)	105(2)	C(41)-C(42)-C(37)	125.00(17)
C(36)-C(31)-C(32)	113.17(17)	C(44)-C(43)-C(48)	113.23(18)
C(36)-C(31)-B(31)	119.80(17)	C(44)-C(43)-B(31)	126.69(17)
C(32)-C(31)-B(31)	126.54(18)	C(48)-C(43)-B(31)	119.57(18)
F(32)-C(32)-C(33)	115.23(19)	F(44)-C(44)-C(45)	114.8(2)
F(32)-C(32)-C(31)	120.95(18)	F(44)-C(44)-C(43)	121.18(18)
C(33)-C(32)-C(31)	123.8(2)	C(45)-C(44)-C(43)	124.0(2)
F(33)-C(33)-C(34)	119.9(2)	F(45)-C(45)-C(46)	119.5(2)
F(33)-C(33)-C(32)	120.3(2)	F(45)-C(45)-C(44)	120.6(2)

C(46)-C(45)-C(44)	119.9(2)
F(46)-C(46)-C(47)	120.4(3)
F(46)-C(46)-C(45)	120.7(3)
C(47)-C(46)-C(45)	118.9(2)
F(47)-C(47)-C(46)	120.1(2)
F(47)-C(47)-C(48)	120.4(3)
C(46)-C(47)-C(48)	119.5(2)
F(48)-C(48)-C(47)	116.6(2)
F(48)-C(48)-C(43)	118.95(18)
C(47)-C(48)-C(43)	124.4(2)
C(54)-C(49)-C(50)	114.4(2)
C(54)-C(49)-B(31)	125.9(2)
C(50)-C(49)-B(31)	119.1(2)
F(50)-C(50)-C(49)	120.2(2)
F(50)-C(50)-C(51)	116.1(3)
C(49)-C(50)-C(51)	123.6(3)
F(51)-C(51)-C(52)	121.4(3)
F(51)-C(51)-C(50)	119.6(4)
C(52)-C(51)-C(50)	119.0(3)
F(52)-C(52)-C(53)	120.9(4)
F(52)-C(52)-C(51)	119.3(4)
C(53)-C(52)-C(51)	119.8(2)
F(53)-C(53)-C(52)	119.8(3)
F(53)-C(53)-C(54)	121.1(4)
C(52)-C(53)-C(54)	119.1(3)
F(54)-C(54)-C(49)	121.7(2)
F(54)-C(54)-C(53)	114.2(3)
C(49)-C(54)-C(53)	124.0(3)
C(31)-B(31)-C(43)	101.33(14)
C(31)-B(31)-C(37)	113.96(15)
C(43)-B(31)-C(37)	114.27(15)
C(31)-B(31)-C(49)	114.17(16)
C(43)-B(31)-C(49)	113.65(16)
C(37)-B(31)-C(49)	100.12(14)

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **2a**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Zr(1)	39(1)	43(1)	54(1)	12(1)	23(1)	8(1)
N(1)	58(1)	38(1)	44(1)	6(1)	22(1)	7(1)
N(2)	44(1)	40(1)	47(1)	6(1)	15(1)	10(1)
C(1)	96(2)	46(1)	82(2)	6(1)	66(2)	2(1)
C(2)	47(1)	42(1)	66(1)	13(1)	32(1)	7(1)
C(3)	34(1)	70(2)	100(2)	36(2)	29(1)	8(1)
C(4)	63(2)	70(2)	198(4)	73(3)	88(2)	38(2)
C(5)	106(3)	47(2)	147(3)	24(2)	100(3)	19(2)
C(6)	150(3)	89(3)	57(2)	-7(2)	52(2)	-25(2)
C(7)	57(1)	42(1)	75(2)	12(1)	27(1)	7(1)
C(8)	41(1)	120(3)	108(3)	45(2)	7(2)	-14(2)
C(9)	73(2)	126(3)	329(9)	147(5)	108(4)	62(3)
C(10)	281(7)	47(2)	269(6)	10(3)	246(6)	17(3)
C(11)	49(1)	44(1)	32(1)	7(1)	10(1)	5(1)
C(12)	49(1)	57(2)	55(1)	10(1)	2(1)	-2(1)
C(13)	84(2)	37(1)	65(2)	8(1)	36(1)	5(1)
C(14)	88(2)	51(2)	108(3)	30(2)	57(2)	25(2)
C(15)	160(5)	51(2)	87(3)	-17(2)	35(3)	-2(2)
C(16)	44(1)	44(1)	71(2)	11(1)	21(1)	13(1)
C(17)	64(2)	50(1)	80(2)	1(1)	29(1)	17(1)
C(18)	72(2)	68(2)	82(2)	22(2)	5(2)	27(2)
N(19)	38(1)	78(2)	63(1)	18(1)	7(1)	-5(1)
C(19)	54(1)	66(2)	45(1)	11(1)	3(1)	-5(1)
C(20)	62(2)	82(2)	67(2)	23(2)	-2(1)	-2(2)
C(21)	111(3)	77(2)	54(2)	2(2)	2(2)	-8(2)
C(22)	50(1)	71(2)	45(1)	11(1)	16(1)	1(1)
C(31)	34(1)	36(1)	33(1)	-5(1)	8(1)	4(1)
C(32)	38(1)	45(1)	42(1)	-7(1)	11(1)	1(1)
C(33)	33(1)	65(2)	52(1)	-11(1)	11(1)	4(1)
C(34)	45(1)	69(2)	53(1)	-7(1)	5(1)	25(1)
C(35)	58(1)	45(1)	41(1)	-2(1)	10(1)	19(1)

C(36)	39(1)	41(1)	34(1)	-3(1)	9(1)	5(1)
C(37)	35(1)	30(1)	36(1)	1(1)	11(1)	2(1)
C(38)	36(1)	38(1)	36(1)	0(1)	9(1)	6(1)
C(39)	45(1)	46(1)	33(1)	2(1)	13(1)	7(1)
C(40)	40(1)	49(1)	45(1)	3(1)	22(1)	4(1)
C(41)	33(1)	41(1)	47(1)	6(1)	11(1)	6(1)
C(42)	35(1)	35(1)	36(1)	5(1)	11(1)	1(1)
C(43)	44(1)	35(1)	33(1)	4(1)	10(1)	12(1)
C(44)	52(1)	38(1)	37(1)	4(1)	7(1)	8(1)
C(45)	70(2)	38(1)	47(1)	-2(1)	-1(1)	8(1)
C(46)	83(2)	56(1)	36(1)	-9(1)	1(1)	26(1)
C(47)	69(2)	70(2)	35(1)	5(1)	17(1)	34(1)
C(48)	48(1)	52(1)	37(1)	5(1)	12(1)	17(1)
C(49)	49(1)	33(1)	60(1)	4(1)	33(1)	5(1)
C(50)	63(1)	40(1)	76(2)	-8(1)	41(1)	-3(1)
C(51)	88(2)	39(1)	135(3)	-27(2)	75(2)	-15(1)
C(52)	98(2)	32(1)	174(4)	16(2)	104(3)	13(1)
C(53)	88(2)	46(1)	130(3)	37(2)	83(2)	29(1)
C(54)	59(1)	43(1)	82(2)	20(1)	48(1)	17(1)
B(31)	35(1)	33(1)	34(1)	2(1)	12(1)	4(1)
F(32)	43(1)	51(1)	69(1)	0(1)	21(1)	-5(1)
F(33)	33(1)	99(1)	86(1)	-10(1)	17(1)	5(1)
F(34)	59(1)	94(1)	101(1)	4(1)	10(1)	44(1)
F(35)	78(1)	47(1)	68(1)	8(1)	11(1)	25(1)
F(36)	45(1)	37(1)	51(1)	6(1)	13(1)	3(1)
F(38)	42(1)	64(1)	34(1)	2(1)	7(1)	18(1)
F(39)	61(1)	83(1)	33(1)	8(1)	17(1)	21(1)
F(40)	49(1)	87(1)	57(1)	9(1)	31(1)	16(1)
F(41)	36(1)	71(1)	61(1)	15(1)	16(1)	17(1)
F(42)	41(1)	52(1)	37(1)	12(1)	10(1)	10(1)
F(44)	55(1)	46(1)	49(1)	2(1)	13(1)	-9(1)
F(45)	93(1)	53(1)	71(1)	-14(1)	-3(1)	-16(1)
F(46)	126(2)	81(1)	47(1)	-27(1)	-1(1)	32(1)
F(47)	90(1)	118(1)	45(1)	-3(1)	32(1)	41(1)
F(48)	51(1)	74(1)	51(1)	7(1)	27(1)	11(1)
F(50)	72(1)	61(1)	63(1)	-24(1)	22(1)	-12(1)

F(51)	117(2)	59(1)	170(2)	-59(1)	77(2)	-33(1)
F(52)	145(2)	29(1)	268(3)	16(1)	151(2)	10(1)
F(53)	124(2)	77(1)	163(2)	80(1)	103(2)	57(1)
F(54)	67(1)	70(1)	63(1)	32(1)	31(1)	27(1)

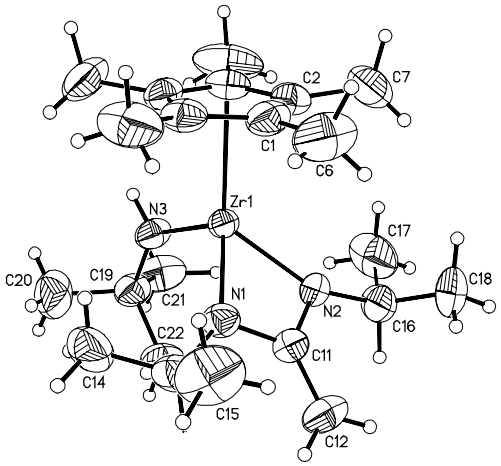
Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for **2a**.

	x	y	z	U(eq)
H(6A)	9694	3335	-199	250(30)
H(6B)	8984	3950	293	160(20)
H(6C)	8723	2834	214	129(15)
H(7A)	10788	5457	1680	69(8)
H(7B)	10211	5331	2539	86(10)
H(7C)	9501	5072	1474	96(11)
H(8A)	12521	4791	3470	131(15)
H(8B)	12344	3937	4114	90(10)
H(8C)	11467	4675	3907	99(12)
H(9A)	11733	1545	3161	128(15)
H(9B)	12525	2444	3709	120(18)
H(9C)	12738	2000	2762	112(13)
H(10A)	10830	1111	1412	180(20)
H(10B)	11074	1692	554	139(15)
H(10C)	9828	1420	625	250(40)
H(12A)	5990(40)	1400(40)	2360(40)	148(17)
H(12B)	5970(30)	1250(30)	1280(30)	112(12)
H(12C)	5700(40)	2170(30)	1660(30)	122(15)
H(13)	7280(20)	359(17)	2303(18)	53(7)
H(14A)	9620(30)	330(20)	2320(20)	76(9)
H(14B)	8870(30)	-550(30)	2500(20)	84(9)
H(14C)	9120(30)	420(20)	3240(20)	77(9)
H(15A)	6970(40)	360(30)	650(30)	106(17)

H(15B)	7490(30)	-470(30)	1000(30)	112(13)
H(15C)	8380(40)	290(30)	770(30)	108(14)
H(16)	6340(20)	3564(18)	2414(18)	53(7)
H(17A)	7930(20)	4350(20)	3600(20)	64(8)
H(17B)	7200(30)	5100(20)	2990(20)	75(9)
H(17C)	8330(20)	4853(19)	2750(20)	66(8)
H(18A)	7330(30)	4530(20)	1010(20)	67(8)
H(18B)	6110(30)	4740(20)	1240(20)	83(9)
H(18C)	6230(40)	3740(30)	820(30)	114(15)
H(19)	10700(30)	2980(20)	4390(20)	81(10)
H(20A)	9680(30)	1600(30)	6050(30)	100(11)
H(20B)	10750(30)	1870(20)	5690(20)	77(9)
H(20C)	9930(20)	1120(20)	4990(20)	60(7)
H(21A)	9320(20)	4020(20)	5230(20)	70(8)
H(21B)	10470(30)	3630(30)	5830(30)	106(12)
H(21C)	9270(40)	3260(30)	6130(30)	123(14)
H(22A)	7910(20)	2720(20)	4140(20)	68(8)
H(22B)	7890(20)	2030(18)	4992(19)	52(6)
H(22C)	8090(20)	1615(18)	4048(19)	51(7)

Crystallographic analysis of compound 5

An orange, cut block, with approximate orthogonal dimensions 0.405 x 0.287 x 0.230mm^3 was placed and optically centered on the Bruker SMART CCD system at –100°C. The initial unit cell was indexed using a least-squares analysis of a random set of reflections collected from three series of 0.3° wide ω-scans, 10 seconds per frame, and 25 frames per series that were well distributed in reciprocal space. Data frames were collected [MoKα] with 0.3° wide ω-scans, 14 seconds per frame and 606 frames per series. Five data series were collected at varying φ angles (φ=0°, 72°, 144°, 216°, 288°), including a partial repeat of the first series, 200 frames, for decay purposes. The crystal to detector distance was 4.767cm, thus providing a complete sphere of data to $2\theta_{max}$=55.12°. A total of 81069 reflections were collected and corrected for Lorentz and polarization effects and absorption using Blessing's method as incorporated into the program SADABS[1,2] with 6282 unique.



Structural determination and Refinement:

All crystallographic calculations were performed on a Personal computer (PC) with a Pentium 3.06GHz processor and 512MB of extended memory. The SHELXTL[3] program package was implemented to determine the probable space group and set up the initial files. System symmetry, systematic absences and intensity statistics indicated the centrosymmetric orthorhombic space group Pbca (no. 61). The structure was determined by direct methods with the successful location of nearly the entire main molecule using the program XS[4]. The structure was refined with XL[5]. The 81069 data collected were merged based upon identical indices yielding 45186 data [R(int)=0.0175] that were further truncated to $2\theta_{max}$=55.0° yielding 42889 that were further merged during least-squares refinement to 5680 unique data [R(int)=0.0184]. A series of least-squares difference-Fourier cycles were required to locate the remaining non-hydrogen atoms. All non-hydrogen atoms were refined anisotropically. The hydrogen atom on N(3) was located directly from a difference-Fourier map while the remaining hydrogen atoms were calculated. Many hydrogen atoms were allowed to refine freely during the final refinement stages. A centroid was calculated for the pentamethylcyclopentadienyl group. The final structure was refined to convergence [$\Delta/\sigma \leq 0.001$] with R(F)=4.01%, wR(F^2)=9.73%, GOF=1.120 for all 5680 unique reflections [R(F)=3.30%, wR(F^2)=9.09% for those 4921 data with Fo > 4σ(Fo)]. The final difference-Fourier map was featureless indicating that the structure is both correct and complete.

The function minimized during the full-matrix least-squares refinement was $\Sigma w(Fo^2-Fc^2)$ where $w=1/[\sigma^2(Fo^2)+(0.0523*P)^2+2.2682*P]$ and $P=(\max(Fo^2,0)+2*Fc^2)/3$. An empirical correction for extinction was also applied to the data in the form $(Fc^2,corr) = k[1 + 0.001 * x * Fc^2 * \lambda^3/\sin(2\theta)]^{(-1/4)}$ where k=0.21997 is the overall scale factor. The value determined for x was 0.00059(11).

Table 1. Crystal data and structure refinement for **5**.

Empirical formula	C22 H41 N3 Zr	
Formula weight	438.80	
Temperature	293(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	Pbca	
Unit cell dimensions	a = 16.3932(5) Å	α= 90°.
	b = 15.9442(5) Å	β= 90°.
	c = 18.8698(6) Å	γ = 90°.
Volume	4932.1(3) $Å^3$	
Z	8	
Density (calculated)	1.182 Mg/m^3	
Absorption coefficient	0.455 mm^{-1}	
F(000)	1872	
Crystal size	0.41 x 0.29 x 0.23 mm^3	
Theta range for data collection	2.08 to 27.50°.	
Index ranges	-21<=h<=21, -20<=k<=20, -24<=l<=24	
Reflections collected	42889	
Independent reflections	5680 [R(int) = 0.0184]	
Completeness to theta = 27.50°	100.0 %	
Absorption correction	Empirical, SADABS (multi-scan)	
Max. and min. transmission	0.9025 and 0.8371	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	5680 / 3 / 355	
Goodness-of-fit on F^2	1.120	
Final R indices [I>2sigma(I)]	R1 = 0.0330, wR2 = 0.0909 [4921 Data]	
R indices (all data)	R1 = 0.0401, wR2 = 0.0973	
Extinction coefficient	0.00059(11)	
Largest diff. peak and hole	0.668 and -0.477 $e.Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **5**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Zr(1)	9896(1)	2713(1)	1517(1)	31(1)
N(1)	9515(1)	3593(1)	2245(1)	41(1)
N(2)	8842(1)	3313(1)	1203(1)	42(1)
N(3)	10883(1)	3149(1)	958(1)	42(1)
CNT1	10022(1)	1327(1)	1691(1)	0
C(1)	9430(1)	1355(1)	2060(1)	54(1)
C(2)	9438(2)	1207(1)	1327(1)	54(1)
C(3)	10240(2)	1222(1)	1099(1)	50(1)
C(4)	10749(1)	1381(1)	1677(1)	46(1)
C(5)	10253(2)	1468(1)	2292(1)	49(1)
C(6)	8679(2)	1353(2)	2518(2)	94(1)
C(7)	8707(2)	965(2)	878(2)	97(1)
C(8)	10535(3)	1068(2)	346(2)	97(1)
C(9)	11666(2)	1396(2)	1667(2)	91(1)
C(10)	10525(3)	1545(2)	3036(2)	91(1)
C(11)	8840(1)	3823(1)	1818(1)	45(1)
C(12)	8306(2)	4445(2)	1962(2)	71(1)
C(13)	9684(2)	4063(2)	2889(1)	59(1)
C(14)	10570(2)	3966(2)	3073(2)	74(1)
C(15)	9141(3)	3786(3)	3498(2)	97(1)
C(16)	8221(2)	3449(2)	666(2)	64(1)
C(17)	8530(2)	3079(2)	-32(2)	83(1)
C(18)	7404(2)	3073(3)	891(3)	101(1)
C(19)	11139(1)	3962(1)	664(1)	47(1)
C(20)	11996(2)	4164(2)	909(3)	86(1)
C(21)	11120(2)	3915(2)	-148(2)	71(1)
C(22)	10552(2)	4638(1)	904(2)	56(1)

Table 3. Bond lengths [Å] and angles [°] for **5**.

Zr(1)-N(3)	2.0533(16)	C(9)-H(9A)	0.9600
Zr(1)-N(1)	2.0596(15)	C(9)-H(9B)	0.9600
Zr(1)-N(2)	2.0631(15)	C(9)-H(9C)	0.9600
Zr(1)-CNT1	2.2442(10)	C(10)-H(10A)	0.9600
Zr(1)-C(1)	2.514(2)	C(10)-H(10B)	0.9600
Zr(1)-C(5)	2.5339(19)	C(10)-H(10C)	0.9600
Zr(1)-C(2)	2.541(2)	C(11)-C(12)	1.351(3)
Zr(1)-C(4)	2.5610(19)	C(12)-H(12A)	0.98(3)
Zr(1)-C(3)	2.568(2)	C(12)-H(12B)	0.95(3)
N(1)-C(11)	1.417(3)	C(13)-C(14)	1.502(4)
N(1)-C(13)	1.455(3)	C(13)-C(15)	1.519(5)
N(2)-C(11)	1.416(3)	C(13)-H(13)	0.94(3)
N(2)-C(16)	1.453(3)	C(14)-H(14A)	0.90(4)
N(3)-C(19)	1.471(2)	C(14)-H(14B)	0.97(3)
N(3)-H(3)	0.88(3)	C(14)-H(14C)	1.00(3)
C(1)-C(2)	1.403(4)	C(15)-H(15A)	0.94(4)
C(1)-C(5)	1.430(3)	C(15)-H(15B)	0.97(5)
C(1)-C(6)	1.504(3)	C(15)-H(15C)	1.00(3)
C(2)-C(3)	1.384(3)	C(16)-C(18)	1.528(5)
C(2)-C(7)	1.518(4)	C(16)-C(17)	1.530(4)
C(3)-C(4)	1.397(3)	C(16)-H(16)	1.04(3)
C(3)-C(8)	1.520(3)	C(17)-H(17A)	0.9600
C(4)-C(5)	1.423(3)	C(17)-H(17B)	0.9600
C(4)-C(9)	1.503(3)	C(17)-H(17C)	0.9600
C(5)-C(10)	1.477(3)	C(18)-H(18A)	0.99(4)
C(6)-H(6A)	0.9600	C(18)-H(18B)	0.84(4)
C(6)-H(6B)	0.9600	C(18)-H(18C)	1.04(5)
C(6)-H(6C)	0.9600	C(19)-C(20)	1.514(4)
C(7)-H(7A)	0.9600	C(19)-C(22)	1.514(3)
C(7)-H(7B)	0.9600	C(19)-C(21)	1.534(4)
C(7)-H(7C)	0.9600	C(20)-H(20A)	0.95(3)
C(8)-H(8A)	0.9600	C(20)-H(20B)	0.97(4)
C(8)-H(8B)	0.9600	C(20)-H(20C)	0.92(4)
C(8)-H(8C)	0.9600	C(21)-H(21A)	0.93(3)

C(21)-H(21B)	0.96(4)	N(2)-Zr(1)-C(3)	121.64(7)
C(21)-H(21C)	1.11(4)	CNT1-Zr(1)-C(3)	27.48(5)
C(22)-H(22A)	0.95(3)	C(1)-Zr(1)-C(3)	52.75(7)
C(22)-H(22B)	0.98(3)	C(5)-Zr(1)-C(3)	53.23(7)
C(22)-H(22C)	0.90(3)	C(2)-Zr(1)-C(3)	31.43(8)
		C(4)-Zr(1)-C(3)	31.61(7)
N(3)-Zr(1)-N(1)	110.59(7)	C(11)-N(1)-C(13)	119.36(17)
N(3)-Zr(1)-N(2)	110.83(7)	C(11)-N(1)-Zr(1)	91.97(11)
N(1)-Zr(1)-N(2)	67.75(6)	C(13)-N(1)-Zr(1)	148.21(15)
N(3)-Zr(1)-CNT1	109.63(5)	C(11)-N(2)-C(16)	118.97(18)
N(1)-Zr(1)-CNT1	126.97(5)	C(11)-N(2)-Zr(1)	91.86(11)
N(2)-Zr(1)-CNT1	125.14(5)	C(16)-N(2)-Zr(1)	148.97(16)
N(3)-Zr(1)-C(1)	137.77(6)	C(19)-N(3)-Zr(1)	135.78(13)
N(1)-Zr(1)-C(1)	102.87(7)	C(19)-N(3)-H(3)	108.6(16)
N(2)-Zr(1)-C(1)	105.16(7)	Zr(1)-N(3)-H(3)	115.6(16)
CNT1-Zr(1)-C(1)	28.38(5)	C(2)-C(1)-C(5)	108.32(19)
N(3)-Zr(1)-C(5)	112.33(7)	C(2)-C(1)-C(6)	125.0(3)
N(1)-Zr(1)-C(5)	102.67(7)	C(5)-C(1)-C(6)	126.6(3)
N(2)-Zr(1)-C(5)	136.27(7)	C(2)-C(1)-Zr(1)	74.96(12)
CNT1-Zr(1)-C(5)	28.69(5)	C(5)-C(1)-Zr(1)	74.32(11)
C(1)-Zr(1)-C(5)	32.91(8)	C(6)-C(1)-Zr(1)	118.98(17)
N(3)-Zr(1)-C(2)	118.68(8)	C(3)-C(2)-C(1)	108.2(2)
N(1)-Zr(1)-C(2)	130.37(8)	C(3)-C(2)-C(7)	125.5(3)
N(2)-Zr(1)-C(2)	98.60(7)	C(1)-C(2)-C(7)	125.9(3)
CNT1-Zr(1)-C(2)	28.01(5)	C(3)-C(2)-Zr(1)	75.35(12)
C(1)-Zr(1)-C(2)	32.22(8)	C(1)-C(2)-Zr(1)	72.82(12)
C(5)-Zr(1)-C(2)	53.81(7)	C(7)-C(2)-Zr(1)	123.57(16)
N(3)-Zr(1)-C(4)	84.91(6)	C(2)-C(3)-C(4)	109.1(2)
N(1)-Zr(1)-C(4)	130.71(7)	C(2)-C(3)-C(8)	126.2(3)
N(2)-Zr(1)-C(4)	151.15(7)	C(4)-C(3)-C(8)	124.7(3)
CNT1-Zr(1)-C(4)	27.81(4)	C(2)-C(3)-Zr(1)	73.22(12)
C(1)-Zr(1)-C(4)	53.38(7)	C(4)-C(3)-Zr(1)	73.93(11)
C(5)-Zr(1)-C(4)	32.44(7)	C(8)-C(3)-Zr(1)	120.46(16)
C(2)-Zr(1)-C(4)	52.71(7)	C(3)-C(4)-C(5)	108.28(19)
N(3)-Zr(1)-C(3)	88.97(7)	C(3)-C(4)-C(9)	126.2(3)
N(1)-Zr(1)-C(3)	154.45(7)	C(5)-C(4)-C(9)	125.4(3)

C(3)-C(4)-Zr(1)	74.46(11)	C(5)-C(10)-H(10C)	109.5
C(5)-C(4)-Zr(1)	72.73(11)	H(10A)-C(10)-H(10C)	109.5
C(9)-C(4)-Zr(1)	122.05(16)	H(10B)-C(10)-H(10C)	109.5
C(4)-C(5)-C(1)	106.08(19)	C(12)-C(11)-N(2)	126.0(2)
C(4)-C(5)-C(10)	127.6(3)	C(12)-C(11)-N(1)	125.5(2)
C(1)-C(5)-C(10)	125.9(3)	N(2)-C(11)-N(1)	108.39(15)
C(4)-C(5)-Zr(1)	74.83(11)	C(11)-C(12)-H(12A)	121.5(17)
C(1)-C(5)-Zr(1)	72.77(11)	C(11)-C(12)-H(12B)	121(2)
C(10)-C(5)-Zr(1)	123.53(16)	H(12A)-C(12)-H(12B)	117(3)
C(1)-C(6)-H(6A)	109.5	N(1)-C(13)-C(14)	108.9(2)
C(1)-C(6)-H(6B)	109.5	N(1)-C(13)-C(15)	111.8(3)
H(6A)-C(6)-H(6B)	109.5	C(14)-C(13)-C(15)	111.2(3)
C(1)-C(6)-H(6C)	109.5	N(1)-C(13)-H(13)	107.3(16)
H(6A)-C(6)-H(6C)	109.5	C(14)-C(13)-H(13)	110.5(17)
H(6B)-C(6)-H(6C)	109.5	C(15)-C(13)-H(13)	107.1(16)
C(2)-C(7)-H(7A)	109.5	C(13)-C(14)-H(14A)	110(2)
C(2)-C(7)-H(7B)	109.5	C(13)-C(14)-H(14B)	109(2)
H(7A)-C(7)-H(7B)	109.5	H(14A)-C(14)-H(14B)	111(3)
C(2)-C(7)-H(7C)	109.5	C(13)-C(14)-H(14C)	113.9(18)
H(7A)-C(7)-H(7C)	109.5	H(14A)-C(14)-H(14C)	106(3)
H(7B)-C(7)-H(7C)	109.5	H(14B)-C(14)-H(14C)	108(3)
C(3)-C(8)-H(8A)	109.5	C(13)-C(15)-H(15A)	110(3)
C(3)-C(8)-H(8B)	109.5	C(13)-C(15)-H(15B)	109(3)
H(8A)-C(8)-H(8B)	109.5	H(15A)-C(15)-H(15B)	107(4)
C(3)-C(8)-H(8C)	109.5	C(13)-C(15)-H(15C)	110.8(19)
H(8A)-C(8)-H(8C)	109.5	H(15A)-C(15)-H(15C)	111(4)
H(8B)-C(8)-H(8C)	109.5	H(15B)-C(15)-H(15C)	110(3)
C(4)-C(9)-H(9A)	109.5	N(2)-C(16)-C(18)	111.2(3)
C(4)-C(9)-H(9B)	109.5	N(2)-C(16)-C(17)	108.2(2)
H(9A)-C(9)-H(9B)	109.5	C(18)-C(16)-C(17)	112.3(3)
C(4)-C(9)-H(9C)	109.5	N(2)-C(16)-H(16)	109.1(15)
H(9A)-C(9)-H(9C)	109.5	C(18)-C(16)-H(16)	104.9(15)
H(9B)-C(9)-H(9C)	109.5	C(17)-C(16)-H(16)	111.2(15)
C(5)-C(10)-H(10A)	109.5	C(16)-C(17)-H(17A)	109.5
C(5)-C(10)-H(10B)	109.5	C(16)-C(17)-H(17B)	109.5
H(10A)-C(10)-H(10B)	109.5	H(17A)-C(17)-H(17B)	109.5

C(16)-C(17)-H(17C)	109.5
H(17A)-C(17)-H(17C)	109.5
H(17B)-C(17)-H(17C)	109.5
C(16)-C(18)-H(18A)	109(2)
C(16)-C(18)-H(18B)	116(3)
H(18A)-C(18)-H(18B)	108(4)
C(16)-C(18)-H(18C)	112(3)
H(18A)-C(18)-H(18C)	113(3)
H(18B)-C(18)-H(18C)	99(4)
N(3)-C(19)-C(20)	109.7(2)
N(3)-C(19)-C(22)	109.46(17)
C(20)-C(19)-C(22)	110.4(3)
N(3)-C(19)-C(21)	109.04(19)
C(20)-C(19)-C(21)	109.5(3)
C(22)-C(19)-C(21)	108.7(2)
C(19)-C(20)-H(20A)	111.1(19)
C(19)-C(20)-H(20B)	113(2)
H(20A)-C(20)-H(20B)	105(3)
C(19)-C(20)-H(20C)	112(2)
H(20A)-C(20)-H(20C)	106(3)
H(20B)-C(20)-H(20C)	109(3)
C(19)-C(21)-H(21A)	106.6(18)
C(19)-C(21)-H(21B)	109(2)
H(21A)-C(21)-H(21B)	106(3)
C(19)-C(21)-H(21C)	108.9(19)
H(21A)-C(21)-H(21C)	114(3)
H(21B)-C(21)-H(21C)	111(3)
C(19)-C(22)-H(22A)	112(2)
C(19)-C(22)-H(22B)	110.3(16)
H(22A)-C(22)-H(22B)	110(3)
C(19)-C(22)-H(22C)	110.6(19)
H(22A)-C(22)-H(22C)	105(3)
H(22B)-C(22)-H(22C)	109(2)

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **5**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Zr(1)	32(1)	29(1)	33(1)	0(1)	0(1)	2(1)
N(1)	45(1)	40(1)	38(1)	-5(1)	2(1)	4(1)
N(2)	34(1)	40(1)	52(1)	-2(1)	-10(1)	3(1)
N(3)	39(1)	35(1)	53(1)	8(1)	9(1)	6(1)
C(1)	47(1)	38(1)	79(2)	14(1)	20(1)	4(1)
C(2)	59(1)	31(1)	70(1)	6(1)	-9(1)	-3(1)
C(3)	70(1)	30(1)	49(1)	0(1)	5(1)	3(1)
C(4)	41(1)	33(1)	62(1)	9(1)	7(1)	5(1)
C(5)	72(1)	33(1)	41(1)	8(1)	-3(1)	4(1)
C(6)	84(2)	69(2)	130(3)	25(2)	62(2)	9(2)
C(7)	98(3)	49(1)	145(3)	11(2)	-60(2)	-17(2)
C(8)	181(4)	51(2)	60(2)	-10(1)	36(2)	8(2)
C(9)	41(1)	63(2)	170(4)	44(2)	9(2)	12(1)
C(10)	163(4)	56(2)	55(2)	9(1)	-37(2)	5(2)
C(11)	40(1)	39(1)	56(1)	-3(1)	1(1)	4(1)
C(12)	60(2)	61(2)	90(2)	-16(1)	-3(1)	25(1)
C(13)	83(2)	49(1)	44(1)	-13(1)	-5(1)	11(1)
C(14)	92(2)	60(2)	68(2)	-12(1)	-32(2)	-3(2)
C(15)	124(4)	118(3)	48(2)	-13(2)	23(2)	22(3)
C(16)	58(1)	48(1)	86(2)	2(1)	-36(1)	5(1)
C(17)	118(3)	71(2)	61(2)	3(1)	-44(2)	-10(2)
C(18)	43(2)	90(2)	169(4)	-15(3)	-39(2)	2(2)
C(19)	44(1)	38(1)	60(1)	11(1)	8(1)	0(1)
C(20)	53(2)	77(2)	127(3)	24(2)	-8(2)	-19(1)
C(21)	95(2)	58(2)	62(2)	16(1)	26(2)	13(2)
C(22)	67(2)	36(1)	65(2)	7(1)	10(1)	1(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for **5**.

	x	y	z	U(eq)
H(3)	11243(16)	2768(15)	835(13)	52(7)
H(6A)	8264	1691	2300	143(17)
H(6B)	8810	1579	2976	78(10)
H(6C)	8485	789	2571	149(17)
H(7A)	8637	367	891	108(12)
H(7B)	8796	1141	398	190(30)
H(7C)	8227	1233	1061	149(19)
H(8A)	10992	1427	247	220(30)
H(8B)	10102	1188	19	210(30)
H(8C)	10698	493	297	162(18)
H(9A)	11860	1783	2019	127(16)
H(9B)	11852	1570	1208	148(18)
H(9C)	11871	845	1769	144(16)
H(10A)	10574	996	3241	116(13)
H(10B)	10133	1866	3298	170(30)
H(10C)	11045	1822	3050	46(6)
H(12A)	8342(18)	4774(17)	2402(15)	72(8)
H(12B)	7870(20)	4570(20)	1649(16)	83(10)
H(13)	9559(16)	4630(17)	2796(13)	60(7)
H(14A)	10690(20)	3420(20)	3132(18)	91(11)
H(14B)	10680(20)	4280(20)	3502(17)	86(11)
H(14C)	10949(19)	4170(20)	2697(17)	81(10)
H(15A)	8590(30)	3810(30)	3360(20)	117(16)
H(15B)	9210(30)	4180(30)	3890(20)	127(14)
H(15C)	9290(20)	3210(20)	3659(17)	79(10)
H(16)	8112(16)	4089(18)	619(14)	67(8)
H(17A)	9059	3304	-138	111(13)
H(17B)	8158	3220	-407	119(13)
H(17C)	8567	2480	10	69(8)
H(18A)	7450(20)	2460(20)	898(17)	98(12)
H(18B)	7000(30)	3210(30)	640(20)	122(14)

H(18C)	7210(30)	3320(30)	1370(30)	142(18)
H(20A)	12024(18)	4197(19)	1412(16)	64(9)
H(20B)	12190(20)	4700(20)	738(19)	105(11)
H(20C)	12360(20)	3750(20)	780(19)	105(13)
H(21A)	11494(19)	3500(20)	-278(15)	72(8)
H(21B)	11320(20)	4430(20)	-340(17)	91(10)
H(21C)	10490(30)	3790(20)	-321(19)	106(12)
H(22A)	10007(18)	4520(20)	776(17)	71(9)
H(22B)	10712(16)	5182(17)	710(14)	64(7)
H(22C)	10545(18)	4675(18)	1382(16)	65(8)

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **5**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Zr(1)	32(1)	29(1)	33(1)	0(1)	0(1)	2(1)
N(1)	45(1)	40(1)	38(1)	-5(1)	2(1)	4(1)
N(2)	34(1)	40(1)	52(1)	-2(1)	-10(1)	3(1)
N(3)	39(1)	35(1)	53(1)	8(1)	9(1)	6(1)
C(1)	47(1)	38(1)	79(2)	14(1)	20(1)	4(1)
C(2)	59(1)	31(1)	70(1)	6(1)	-9(1)	-3(1)
C(3)	70(1)	30(1)	49(1)	0(1)	5(1)	3(1)
C(4)	41(1)	33(1)	62(1)	9(1)	7(1)	5(1)
C(5)	72(1)	33(1)	41(1)	8(1)	-3(1)	4(1)
C(6)	84(2)	69(2)	130(3)	25(2)	62(2)	9(2)
C(7)	98(3)	49(1)	145(3)	11(2)	-60(2)	-17(2)
C(8)	181(4)	51(2)	60(2)	-10(1)	36(2)	8(2)
C(9)	41(1)	63(2)	170(4)	44(2)	9(2)	12(1)
C(10)	163(4)	56(2)	55(2)	9(1)	-37(2)	5(2)
C(11)	40(1)	39(1)	56(1)	-3(1)	1(1)	4(1)
C(12)	60(2)	61(2)	90(2)	-16(1)	-3(1)	25(1)
C(13)	83(2)	49(1)	44(1)	-13(1)	-5(1)	11(1)
C(14)	92(2)	60(2)	68(2)	-12(1)	-32(2)	-3(2)

C(15)	124(4)	118(3)	48(2)	-13(2)	23(2)	22(3)
C(16)	58(1)	48(1)	86(2)	2(1)	-36(1)	5(1)
C(17)	118(3)	71(2)	61(2)	3(1)	-44(2)	-10(2)
C(18)	43(2)	90(2)	169(4)	-15(3)	-39(2)	2(2)
C(19)	44(1)	38(1)	60(1)	11(1)	8(1)	0(1)
C(20)	53(2)	77(2)	127(3)	24(2)	-8(2)	-19(1)
C(21)	95(2)	58(2)	62(2)	16(1)	26(2)	13(2)
C(22)	67(2)	36(1)	65(2)	7(1)	10(1)	1(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for **5**.

	x	y	z	U(eq)
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H(7B)	8796	1141	398	190(30)
H(7C)	8227	1233	1061	149(19)
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H(12A)	8342(18)	4774(17)	2402(15)	72(8)
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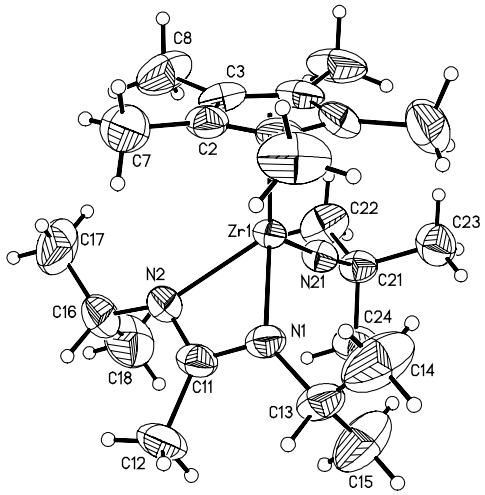
H(14A)	10690(20)	3420(20)	3132(18)	91(11)

H(14B)	10680(20)	4280(20)	3502(17)	86(11)
H(14C)	10949(19)	4170(20)	2697(17)	81(10)
H(15A)	8590(30)	3810(30)	3360(20)	117(16)
H(15B)	9210(30)	4180(30)	3890(20)	127(14)
H(15C)	9290(20)	3210(20)	3659(17)	79(10)
H(16)	8112(16)	4089(18)	619(14)	67(8)
H(17A)	9059	3304	-138	111(13)
H(17B)	8158	3220	-407	119(13)
H(17C)	8567	2480	10	69(8)
H(18A)	7450(20)	2460(20)	898(17)	98(12)
H(18B)	7000(30)	3210(30)	640(20)	122(14)
H(18C)	7210(30)	3320(30)	1370(30)	142(18)
H(20A)	12024(18)	4197(19)	1412(16)	64(9)
H(20B)	12190(20)	4700(20)	738(19)	105(11)
H(20C)	12360(20)	3750(20)	780(19)	105(13)
H(21A)	11494(19)	3500(20)	-278(15)	72(8)
H(21B)	11320(20)	4430(20)	-340(17)	91(10)
H(21C)	10490(30)	3790(20)	-321(19)	106(12)
H(22A)	10007(18)	4520(20)	776(17)	71(9)
H(22B)	10712(16)	5182(17)	710(14)	64(7)
H(22C)	10545(18)	4675(18)	1382(16)	65(8)

Crystallographic analysis of compound 6

An orange rod with approximate orthogonal dimensions 0.409 x 0.128 x 0.117mm^3 was placed and optically centered on the Bruker SMART CCD system at –100°C. The initial unit cell was indexed using a least-squares analysis of a random set of reflections collected from three series of 0.3° wide ω-scans, 10 seconds per frame, and 25 frames per series that were well distributed in reciprocal space. Data frames were collected [MoKα] with 0.3° wide ω-scans, 14 seconds per frame and 606 frames per series. Five data series were collected at varying φ angles (φ=0°, 72°, 144°, 216°, 288°), and additionally a partial repeat of the first series, 200 frames, for decay purposes. The crystal to detector distance was 4.990cm, thus providing a complete sphere of data to $2\theta_{max}$=55.04°. A total of 38948 reflections were collected and corrected for Lorentz and polarization effects and absorption using Blessing's method as incorporated into the program SADABS[1,2] with 627 unique.



Structural determination and Refinement:

All crystallographic calculations were performed on a Personal computer (PC) with a Pentium 3.06GHz processor and 512MB of extended memory. The SHELXTL[3] program package was implemented to determine the probable space group and set up the initial files. System symmetry, systematic absences and intensity statistics indicated the non-centrosymmetric orthorhombic space group $Pna2_1$ (no. 33) or the centrosymmetric orthorhombic space group Pnma (no. 62). The structure was determined by direct methods with the successful location of a majority of the non-hydrogen atoms comprising two molecules using the program XS[4] in $Pna2_1$. All attempts in Pnma proved fruitless. The structure was refined with XL[5]. The 38948 data collected were merged based upon identical indices yielding 22203 reflections [R(int)=0.0256] that were then truncated to $2\theta_{max}$=55.00°, 21108 reflections, and finally all symmetry equivalents were merged during least-squares refinement yielding 5666 unique data [R(int)=0.0265]. A series of least-squares difference-Fourier cycles were required to locate the remaining non-hydrogen atoms. All full occupancy non-hydrogen atoms were refined anisotropically. Hydrogen atoms were placed in calculated positions throughout the final refinement stages. The absolute structure parameter, Flack(x)[6], was found to be 0.29(4) indicating racemic twinning that was further refined indicating that both enantiomorphs were present and that the correct enantiomorph has been chosen. A centroid was calculated for the pentamethylcyclopentadienyl group. The final structure was refined to convergence [$\Delta/\sigma \leq 0.001$] with R(F)=3.54%, wR(F^2)=7.86%, GOF=1.043 for all 5666 unique reflections [R(F)=2.93%, wR(F^2)=7.54% for those 5038 data with Fo > 4σ(Fo)]. The final difference-Fourier map was featureless indicating that the structure is both correct and complete.

The function minimized during the full-matrix least-squares refinement was $\Sigma w(Fo^2-Fc^2)$ where $w=1/[\sigma^2(Fo^2)+(0.0493*P)^2+0.2984*P]$ and $P=(\max(Fo^2,0)+2*Fc^2)/3$. An empirical correction for extinction was also attempted but found to be negative and therefore not applied.

Table 1. Crystal data and structure refinement for **6**.

Empirical formula	C22 H41 N3 Zr	
Formula weight	438.80	
Temperature	173(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	Pna2(1)	
Unit cell dimensions	a = 17.2874(9) Å	α= 90°.
	b = 9.0107(5) Å	β= 90°.
	c = 15.7778(9) Å	γ = 90°.
Volume	2457.7(2) $Å^3$	
Z	4	
Density (calculated)	1.186 Mg/m^3	
Absorption coefficient	0.457 mm^{-1}	
F(000)	936	
Crystal size	0.41 x 0.13 x 0.12 mm^3	
Theta range for data collection	2.36 to 27.50°.	
Index ranges	-22<=h<=22, -11<=k<=11, -20<=l<=20	
Reflections collected	21108	
Independent reflections	5666 [R(int) = 0.0265]	
Completeness to theta = 27.50°	100.0 %	
Absorption correction	Empirical, SADABS (multi-scan)	
Max. and min. transmission	0.9485 and 0.8352	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	5666 / 4 / 272	
Goodness-of-fit on F^2	1.043	
Final R indices [I>2sigma(I)]	R1 = 0.0293, wR2 = 0.0754 [5038 Data]	
R indices (all data)	R1 = 0.0354, wR2 = 0.0786	
Absolute structure parameter	0.29(4)	
Largest diff. peak and hole	0.802 and -0.294 $e.Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **6**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Zr(1)	9250(1)	9049(1)	8451(1)	33(1)
N(1)	10103(1)	10806(2)	8106(2)	43(1)
N(2)	8990(2)	10569(3)	7371(2)	48(1)
CNT1	9517(1)	6629(1)	8201	0
C(1)	10171(2)	6893(3)	8037(2)	53(1)
C(2)	9575(2)	6819(3)	7452(2)	47(1)
C(3)	8886(2)	6489(3)	7905(2)	51(1)
C(4)	9072(2)	6328(3)	8757(2)	54(1)
C(5)	9880(2)	6613(3)	8854(2)	57(1)
C(6)	11016(2)	7063(4)	7804(3)	88(1)
C(7)	9655(3)	6981(4)	6505(2)	78(1)
C(8)	8096(3)	6217(5)	7509(4)	86(1)
C(9)	8530(3)	5818(3)	9452(3)	83(1)
C(10)	10325(3)	6566(5)	9673(3)	91(1)
C(11)	9610(2)	11407(3)	7548(2)	46(1)
C(12)	9733(2)	12930(3)	7165(2)	70(1)
C(13)	10738(1)	11659(3)	8496(4)	56(1)
C(14)	11352(2)	10599(5)	8777(5)	117(2)
C(15)	10464(3)	12604(6)	9183(4)	107(2)
C(16)	8335(2)	11107(4)	6875(3)	65(1)
C(17)	7946(6)	9843(11)	6429(7)	92(3)
C(18)	7783(7)	11892(11)	7456(9)	90(3)
C(16A)	8335(2)	11107(4)	6875(3)	65(1)
C(17A)	8386(8)	10498(14)	6010(6)	102(3)
C(18A)	7579(7)	10780(20)	7341(11)	164(8)
C(21)	8251(2)	10082(3)	10130(2)	45(1)
N(21)	8699(1)	9693(2)	9378(2)	43(1)
C(22)	7429(2)	9474(4)	10049(3)	72(1)
C(23)	8642(2)	9440(4)	10921(2)	66(1)
C(24)	8229(2)	11771(3)	10204(2)	63(1)

Table 3. Bond lengths [Å] and angles [°] for **6**.

Zr(1)-N(21)	1.839(2)	C(9)-H(9C)	0.9800
Zr(1)-N(2)	2.232(2)	C(10)-H(10A)	0.9800
Zr(1)-N(1)	2.232(2)	C(10)-H(10B)	0.9800
Zr(1)-CNT1	2.2637(11)	C(10)-H(10C)	0.9800
Zr(1)-C(4)	2.518(3)	C(11)-C(12)	1.515(4)
Zr(1)-C(5)	2.531(3)	C(12)-H(12A)	0.9800
Zr(1)-C(3)	2.541(3)	C(12)-H(12B)	0.9800
Zr(1)-C(1)	2.595(3)	C(12)-H(12C)	0.9800
Zr(1)-C(2)	2.615(2)	C(13)-C(15)	1.457(6)
N(1)-C(11)	1.340(4)	C(13)-C(14)	1.495(6)
N(1)-C(13)	1.474(4)	C(13)-H(13)	1.0000
N(2)-C(11)	1.340(4)	C(14)-H(14A)	0.9800
N(2)-C(16)	1.460(4)	C(14)-H(14B)	0.9800
C(1)-C(2)	1.385(4)	C(14)-H(14C)	0.9800
C(1)-C(5)	1.406(4)	C(15)-H(15A)	0.9800
C(1)-C(6)	1.515(4)	C(15)-H(15B)	0.9800
C(2)-C(3)	1.421(4)	C(15)-H(15C)	0.9800
C(2)-C(7)	1.507(4)	C(16)-C(17)	1.498(10)
C(3)-C(4)	1.391(5)	C(16)-C(18)	1.500(12)
C(3)-C(8)	1.521(5)	C(16)-H(16)	1.0000
C(4)-C(5)	1.428(5)	C(17)-H(17A)	0.9800
C(4)-C(9)	1.514(5)	C(17)-H(17B)	0.9800
C(5)-C(10)	1.504(5)	C(17)-H(17C)	0.9800
C(6)-H(6A)	0.9800	C(18)-H(18A)	0.9800
C(6)-H(6B)	0.9800	C(18)-H(18B)	0.9800
C(6)-H(6C)	0.9800	C(18)-H(18C)	0.9800
C(7)-H(7A)	0.9800	C(17A)-H(17D)	0.9800
C(7)-H(7B)	0.9800	C(17A)-H(17E)	0.9800
C(7)-H(7C)	0.9800	C(17A)-H(17F)	0.9800
C(8)-H(8A)	0.9800	C(18A)-H(18D)	0.9800
C(8)-H(8B)	0.9800	C(18A)-H(18E)	0.9800
C(8)-H(8C)	0.9800	C(18A)-H(18F)	0.9800
C(9)-H(9A)	0.9800	C(21)-N(21)	1.460(3)
C(9)-H(9B)	0.9800	C(21)-C(24)	1.526(4)

C(21)-C(22)	1.528(5)	CNT1-Zr(1)-C(1)	27.12(6)
C(21)-C(23)	1.534(4)	C(4)-Zr(1)-C(1)	52.71(10)
C(22)-H(22A)	0.9800	C(5)-Zr(1)-C(1)	31.82(9)
C(22)-H(22B)	0.9800	C(3)-Zr(1)-C(1)	52.20(9)
C(22)-H(22C)	0.9800	N(21)-Zr(1)-C(2)	146.44(9)
C(23)-H(23A)	0.9800	N(2)-Zr(1)-C(2)	93.13(9)
C(23)-H(23B)	0.9800	N(1)-Zr(1)-C(2)	104.81(8)
C(23)-H(23C)	0.9800	CNT1-Zr(1)-C(2)	27.26(6)
C(24)-H(24A)	0.9800	C(4)-Zr(1)-C(2)	52.70(10)
C(24)-H(24B)	0.9800	C(5)-Zr(1)-C(2)	52.62(10)
C(24)-H(24C)	0.9800	C(3)-Zr(1)-C(2)	31.96(10)
		C(1)-Zr(1)-C(2)	30.83(10)
N(21)-Zr(1)-N(2)	108.00(9)	C(11)-N(1)-C(13)	122.5(2)
N(21)-Zr(1)-N(1)	108.22(9)	C(11)-N(1)-Zr(1)	91.51(16)
N(2)-Zr(1)-N(1)	60.73(9)	C(13)-N(1)-Zr(1)	139.4(3)
N(21)-Zr(1)-CNT1	123.23(8)	C(11)-N(2)-C(16)	123.0(3)
N(2)-Zr(1)-CNT1	119.96(8)	C(11)-N(2)-Zr(1)	91.51(16)
N(1)-Zr(1)-CNT1	120.42(7)	C(16)-N(2)-Zr(1)	140.3(2)
N(21)-Zr(1)-C(4)	95.24(10)	C(2)-C(1)-C(5)	109.7(3)
N(2)-Zr(1)-C(4)	136.04(11)	C(2)-C(1)-C(6)	124.1(4)
N(1)-Zr(1)-C(4)	144.93(10)	C(5)-C(1)-C(6)	125.8(3)
CNT1-Zr(1)-C(4)	28.38(7)	C(2)-C(1)-Zr(1)	75.40(15)
N(21)-Zr(1)-C(5)	107.25(10)	C(5)-C(1)-Zr(1)	71.62(18)
N(2)-Zr(1)-C(5)	144.16(10)	C(6)-C(1)-Zr(1)	125.3(2)
N(1)-Zr(1)-C(5)	113.09(10)	C(1)-C(2)-C(3)	107.3(3)
CNT1-Zr(1)-C(5)	28.45(7)	C(1)-C(2)-C(7)	126.1(3)
C(4)-Zr(1)-C(5)	32.85(11)	C(3)-C(2)-C(7)	126.6(3)
N(21)-Zr(1)-C(3)	115.33(10)	C(1)-C(2)-Zr(1)	73.77(16)
N(2)-Zr(1)-C(3)	104.38(10)	C(3)-C(2)-Zr(1)	71.16(15)
N(1)-Zr(1)-C(3)	136.45(9)	C(7)-C(2)-Zr(1)	122.90(19)
CNT1-Zr(1)-C(3)	28.00(7)	C(4)-C(3)-C(2)	108.3(3)
C(4)-Zr(1)-C(3)	31.92(11)	C(4)-C(3)-C(8)	126.0(4)
C(5)-Zr(1)-C(3)	53.47(10)	C(2)-C(3)-C(8)	125.5(3)
N(21)-Zr(1)-C(1)	138.94(10)	C(4)-C(3)-Zr(1)	73.1(2)
N(2)-Zr(1)-C(1)	113.01(10)	C(2)-C(3)-Zr(1)	76.88(15)
N(1)-Zr(1)-C(1)	93.68(9)	C(8)-C(3)-Zr(1)	120.5(2)

C(3)-C(4)-C(5)	108.1(3)	C(5)-C(10)-H(10A)	109.5
C(3)-C(4)-C(9)	126.1(4)	C(5)-C(10)-H(10B)	109.5
C(5)-C(4)-C(9)	125.6(4)	H(10A)-C(10)-H(10B)	109.5
C(3)-C(4)-Zr(1)	75.0(2)	C(5)-C(10)-H(10C)	109.5
C(5)-C(4)-Zr(1)	74.11(17)	H(10A)-C(10)-H(10C)	109.5
C(9)-C(4)-Zr(1)	120.7(2)	H(10B)-C(10)-H(10C)	109.5
C(1)-C(5)-C(4)	106.5(3)	N(1)-C(11)-N(2)	114.7(2)
C(1)-C(5)-C(10)	127.5(3)	N(1)-C(11)-C(12)	122.6(3)
C(4)-C(5)-C(10)	125.9(4)	N(2)-C(11)-C(12)	122.7(3)
C(1)-C(5)-Zr(1)	76.57(18)	C(11)-C(12)-H(12A)	109.5
C(4)-C(5)-Zr(1)	73.05(17)	C(11)-C(12)-H(12B)	109.5
C(10)-C(5)-Zr(1)	117.4(2)	H(12A)-C(12)-H(12B)	109.5
C(1)-C(6)-H(6A)	109.5	C(11)-C(12)-H(12C)	109.5
C(1)-C(6)-H(6B)	109.5	H(12A)-C(12)-H(12C)	109.5
H(6A)-C(6)-H(6B)	109.5	H(12B)-C(12)-H(12C)	109.5
C(1)-C(6)-H(6C)	109.5	C(15)-C(13)-N(1)	111.9(3)
H(6A)-C(6)-H(6C)	109.5	C(15)-C(13)-C(14)	112.6(5)
H(6B)-C(6)-H(6C)	109.5	N(1)-C(13)-C(14)	108.6(2)
C(2)-C(7)-H(7A)	109.5	C(15)-C(13)-H(13)	107.9
C(2)-C(7)-H(7B)	109.5	N(1)-C(13)-H(13)	107.9
H(7A)-C(7)-H(7B)	109.5	C(14)-C(13)-H(13)	107.9
C(2)-C(7)-H(7C)	109.5	C(13)-C(14)-H(14A)	109.5
H(7A)-C(7)-H(7C)	109.5	C(13)-C(14)-H(14B)	109.5
H(7B)-C(7)-H(7C)	109.5	H(14A)-C(14)-H(14B)	109.5
C(3)-C(8)-H(8A)	109.5	C(13)-C(14)-H(14C)	109.5
C(3)-C(8)-H(8B)	109.5	H(14A)-C(14)-H(14C)	109.5
H(8A)-C(8)-H(8B)	109.5	H(14B)-C(14)-H(14C)	109.5
C(3)-C(8)-H(8C)	109.5	C(13)-C(15)-H(15A)	109.5
H(8A)-C(8)-H(8C)	109.5	C(13)-C(15)-H(15B)	109.5
H(8B)-C(8)-H(8C)	109.5	H(15A)-C(15)-H(15B)	109.5
C(4)-C(9)-H(9A)	109.5	C(13)-C(15)-H(15C)	109.5
C(4)-C(9)-H(9B)	109.5	H(15A)-C(15)-H(15C)	109.5
H(9A)-C(9)-H(9B)	109.5	H(15B)-C(15)-H(15C)	109.5
C(4)-C(9)-H(9C)	109.5	N(2)-C(16)-C(17)	110.4(4)
H(9A)-C(9)-H(9C)	109.5	N(2)-C(16)-C(18)	108.8(5)
H(9B)-C(9)-H(9C)	109.5	C(17)-C(16)-C(18)	111.1(7)

N(2)-C(16)-H(16)	108.8
C(17)-C(16)-H(16)	108.8
C(18)-C(16)-H(16)	108.8
H(17D)-C(17A)-H(17E)	109.5
H(17D)-C(17A)-H(17F)	109.5
H(17E)-C(17A)-H(17F)	109.5
H(18D)-C(18A)-H(18E)	109.5
H(18D)-C(18A)-H(18F)	109.5
H(18E)-C(18A)-H(18F)	109.5
N(21)-C(21)-C(24)	108.4(2)
N(21)-C(21)-C(22)	109.8(2)
C(24)-C(21)-C(22)	109.9(3)
N(21)-C(21)-C(23)	109.7(2)
C(24)-C(21)-C(23)	108.9(2)
C(22)-C(21)-C(23)	110.0(3)
C(21)-N(21)-Zr(1)	175.52(18)
C(21)-C(22)-H(22A)	109.5
C(21)-C(22)-H(22B)	109.5
H(22A)-C(22)-H(22B)	109.5
C(21)-C(22)-H(22C)	109.5
H(22A)-C(22)-H(22C)	109.5
H(22B)-C(22)-H(22C)	109.5
C(21)-C(23)-H(23A)	109.5
C(21)-C(23)-H(23B)	109.5
H(23A)-C(23)-H(23B)	109.5
C(21)-C(23)-H(23C)	109.5
H(23A)-C(23)-H(23C)	109.5
H(23B)-C(23)-H(23C)	109.5
C(21)-C(24)-H(24A)	109.5
C(21)-C(24)-H(24B)	109.5
H(24A)-C(24)-H(24B)	109.5
C(21)-C(24)-H(24C)	109.5
H(24A)-C(24)-H(24C)	109.5
H(24B)-C(24)-H(24C)	109.5

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **6**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Zr(1)	36(1)	27(1)	36(1)	1(1)	1(1)	0(1)
N(1)	48(1)	36(1)	46(1)	-2(1)	4(1)	-9(1)
N(2)	60(1)	40(1)	44(1)	5(1)	-7(1)	3(1)
C(1)	48(2)	35(1)	75(2)	1(1)	5(1)	10(1)
C(2)	63(2)	31(1)	48(1)	-4(1)	9(1)	3(1)
C(3)	51(2)	31(1)	70(2)	-13(1)	5(2)	-7(1)
C(4)	68(2)	26(1)	67(2)	4(1)	17(1)	-3(1)
C(5)	74(2)	36(1)	61(2)	11(1)	-9(2)	12(1)
C(6)	50(2)	70(2)	143(4)	-5(2)	24(2)	6(2)
C(7)	117(3)	64(2)	53(2)	-6(2)	20(2)	5(2)
C(8)	66(2)	72(2)	120(4)	-31(2)	-20(2)	-11(2)
C(9)	120(4)	41(2)	89(3)	7(2)	49(3)	-13(2)
C(10)	115(3)	80(2)	78(3)	12(2)	-26(2)	28(2)
C(11)	63(2)	32(1)	43(1)	1(1)	13(1)	2(1)
C(12)	96(3)	42(1)	73(2)	17(1)	10(2)	-7(2)
C(13)	55(1)	51(1)	63(2)	-7(2)	5(2)	-20(1)
C(14)	59(2)	98(3)	194(7)	-50(3)	-38(3)	7(2)
C(15)	98(3)	110(3)	113(3)	-58(3)	-18(3)	4(3)
C(16)	76(2)	58(2)	61(2)	14(1)	-15(2)	13(2)
C(17)	88(6)	102(7)	86(6)	-14(5)	-48(5)	22(5)
C(18)	85(7)	86(5)	100(6)	6(6)	-8(5)	39(5)
C(16A)	76(2)	58(2)	61(2)	14(1)	-15(2)	13(2)
C(17A)	125(9)	100(7)	81(6)	14(5)	-41(6)	11(6)
C(18A)	57(6)	300(20)	133(12)	66(16)	-2(7)	48(11)
C(21)	49(1)	40(1)	48(1)	-3(1)	10(1)	-2(1)
N(21)	46(1)	37(1)	46(1)	3(1)	3(1)	-1(1)
C(22)	57(2)	77(2)	81(2)	-15(2)	21(2)	-14(2)
C(23)	89(2)	67(2)	43(2)	2(1)	10(2)	4(2)
C(24)	74(2)	45(2)	70(2)	-8(1)	16(2)	4(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for **6**.

	x	y	z	U(eq)
H(6A)	11076	7889	7406	132
H(6B)	11201	6145	7539	132
H(6C)	11319	7263	8317	132
H(7A)	9970	6164	6283	117
H(7B)	9907	7929	6374	117
H(7C)	9142	6955	6243	117
H(8A)	7697	6693	7856	129
H(8B)	7998	5147	7481	129
H(8C)	8086	6636	6936	129
H(9A)	8490	4734	9442	125
H(9B)	8017	6254	9362	125
H(9C)	8732	6138	10003	125
H(10A)	10800	7149	9615	137
H(10B)	10456	5535	9808	137
H(10C)	10007	6982	10129	137
H(12A)	9618	13692	7590	105
H(12B)	9388	13057	6677	105
H(12C)	10272	13027	6980	105
H(13)	10966	12314	8050	68
H(14A)	11152	9981	9239	176
H(14B)	11803	11156	8977	176
H(14C)	11502	9965	8301	176
H(15A)	10239	11987	9631	160
H(15B)	10069	13287	8965	160
H(15C)	10898	13175	9413	160
H(16)	8528	11828	6442	78
H(17A)	7701	9191	6847	138
H(17B)	8330	9280	6106	138
H(17C)	7551	10232	6042	138
H(18A)	8058	12677	7763	135

H(18B)	7567	11182	7862	135
H(18C)	7364	12332	7122	135
H(16A)	8385	12210	6831	78
H(17D)	8302	9423	6027	153
H(17E)	8900	10705	5776	153
H(17F)	7991	10961	5652	153
H(18D)	7565	9728	7502	246
H(18E)	7140	11000	6968	246
H(18F)	7547	11396	7851	246
H(22A)	7446	8387	10033	108
H(22B)	7193	9848	9526	108
H(22C)	7121	9797	10537	108
H(23A)	9153	9893	10993	100
H(23B)	8699	8364	10857	100
H(23C)	8323	9651	11421	100
H(24A)	7987	12192	9697	94
H(24B)	8758	12152	10259	94
H(24C)	7928	12052	10706	94