Zirconium and Titanium Propylene Polymerization Precatalysts Supported by a Fluxional C_2-Symmetric *Bis*(anilide)pyridine Ligand

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Contribution from the Arnold and Mabel Beckman Laboratories of Chemical Synthesis, California Institute of Technology, Pasadena, California 91125. *Received xxxxxxx xx, 2011.*
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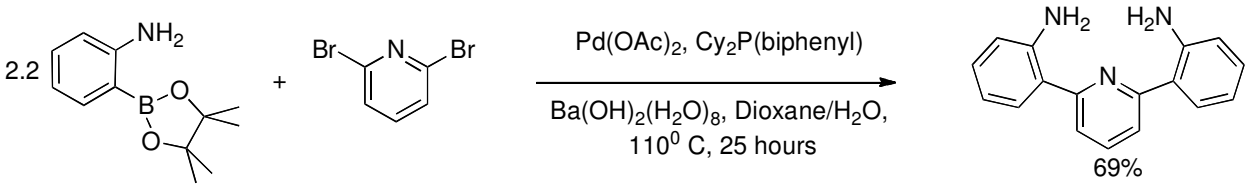
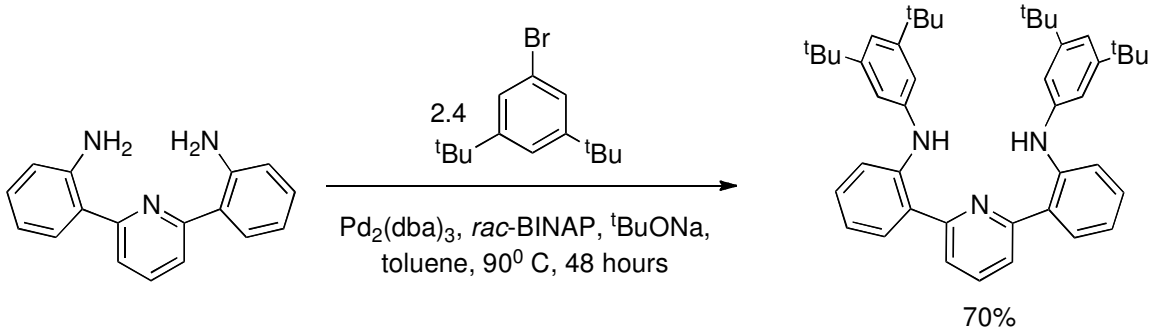
Supporting Information

Contents

Additional Experimental Details



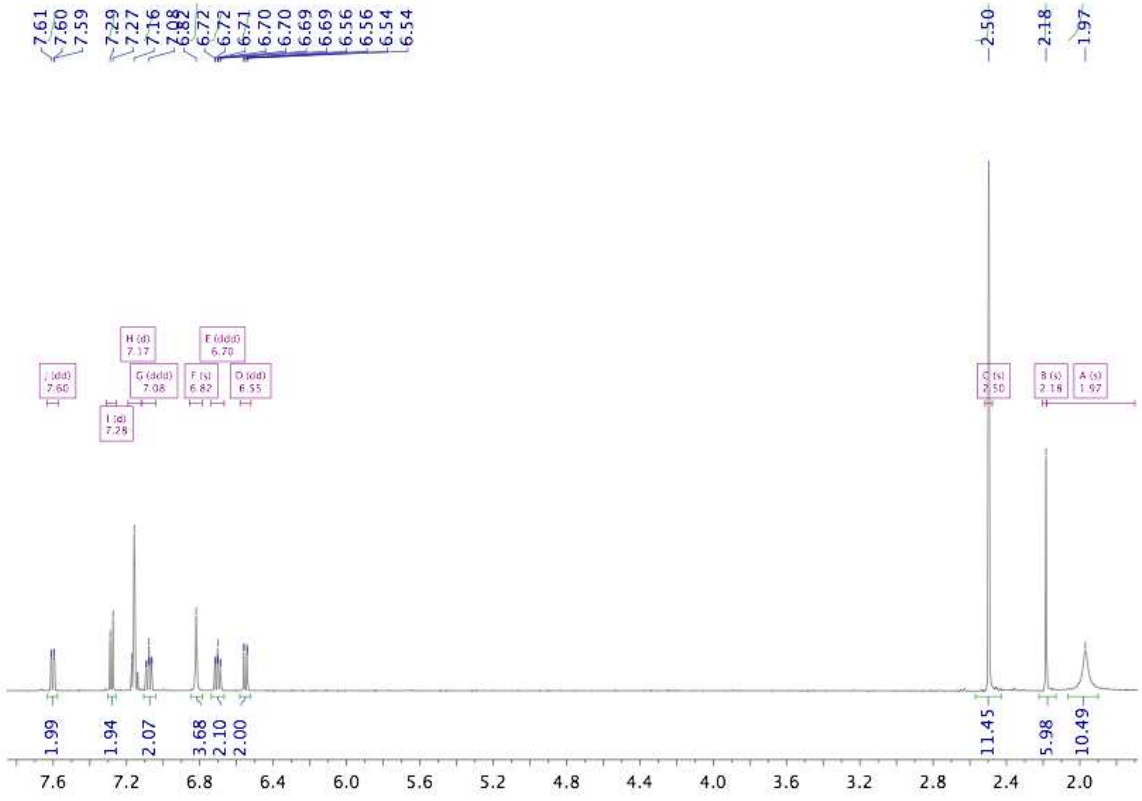
Improved synthesis of the ligand precursor : The preparation of the precursor may be accomplished as reported previously, with the following differences: Use of the commercially available, borylated aniline 2-aminobenzeneboronic acid pinacol ester, rather than generating it in situ via 2-bromoaniline, triethylamine, and pinacolborane. All the reagents may be added at once, and the period of heating at 80 °C for 2.5 hours may be omitted. The yield increases to 69%, more than double the previously reported 33%.



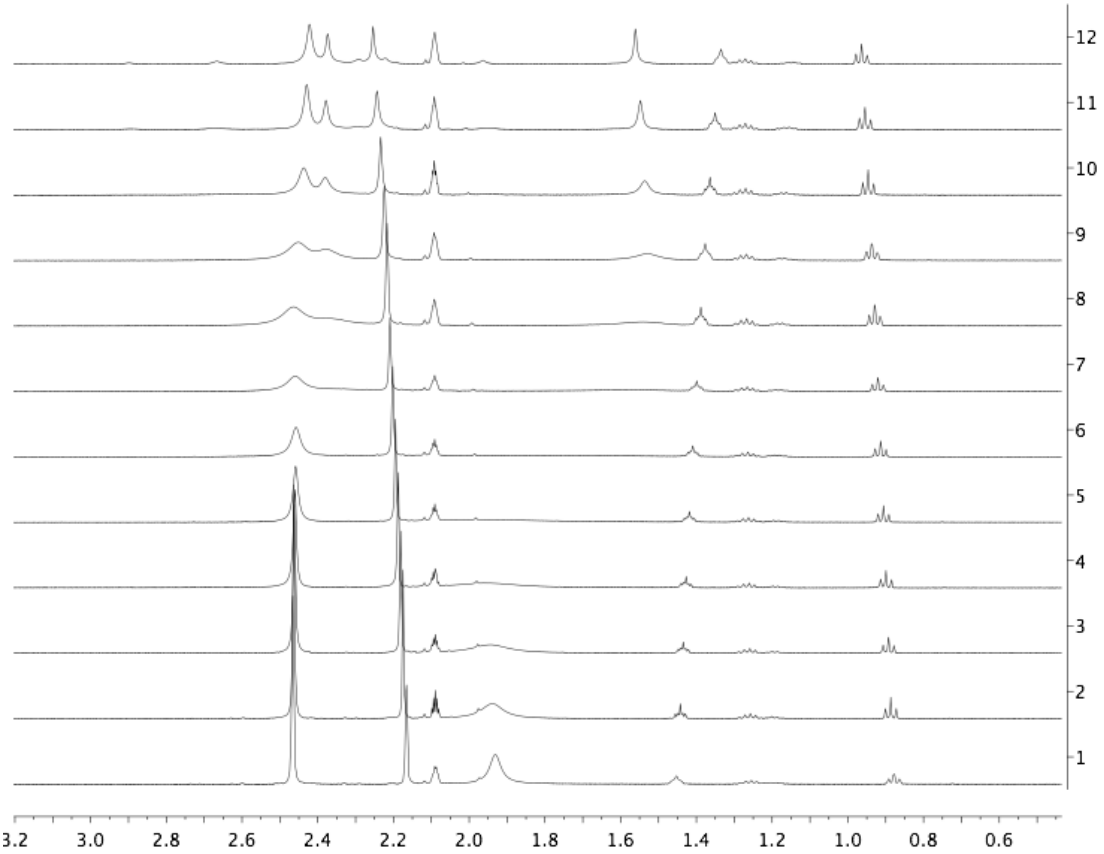
Synthesis of (^{tBu}NNN)H_2. The bis(aniline) ligand precursor (500 mg, 1.914 mmol), 3,5-di-*t*butyl-bromobenzene (1262 mg, 4.688 mmol), $Pd_2(dba)_3$ (87.6 mg, 95.7 μmol), BINAP (128.7 mg, 229.5 μmol) and sodium *t*-butoxide (552 mg, 5.744 mmol) were dissolved in 15 mL of toluene. The mixture was stirred for 48 h at 90 °C before cooling it to room temperature and adding 20 mL of water. After separation with dichloromethane, the volatiles were removed in vacuo from the organic extract. The obtained orange oil was passed through a silica gel column by a 20:1 mixture of dichloromethane and ethyl acetate and then was triturated from cold methanol and collected by filtration. 854 mg of the pale-yellow solid (^{tBu}NNN)H_2 were obtained after a single wash (70% yield). ^{1}H

NMR ($CDCl_3$): δ 1.21 (s, 36H, C(CH_3)$_3$), 6.91 (t, *J* = 7.4 Hz, 2H), 6.97 (s, 2H), 7.04 (s, 4H), 7.28 (t, *J* = 7.8 Hz, 2H), 7.52 (d, *J* = 8.3 Hz,2H), 7.64 (d, *J* = 7.9 Hz,4H), 7.89 (t, *J* = 7.9 Hz, 1H, Py-*H*), 9.78 (s, 2H,N*H*). NMR ($CDCl_3$): δ 31.5 (CH_3), 34.9 (C(CH_3)$_3$), 114.5, 115.8, 116.0, 118.9, 120.4, 124.6, 129.9, 130.4, 138.4, 141.4, 143.2, 151.8, 157.2. X-ray quality crystals may be obtained by slow evaporation of hexanes or acetonitrile.

Complex 2
^{1}H (C_6D_6)

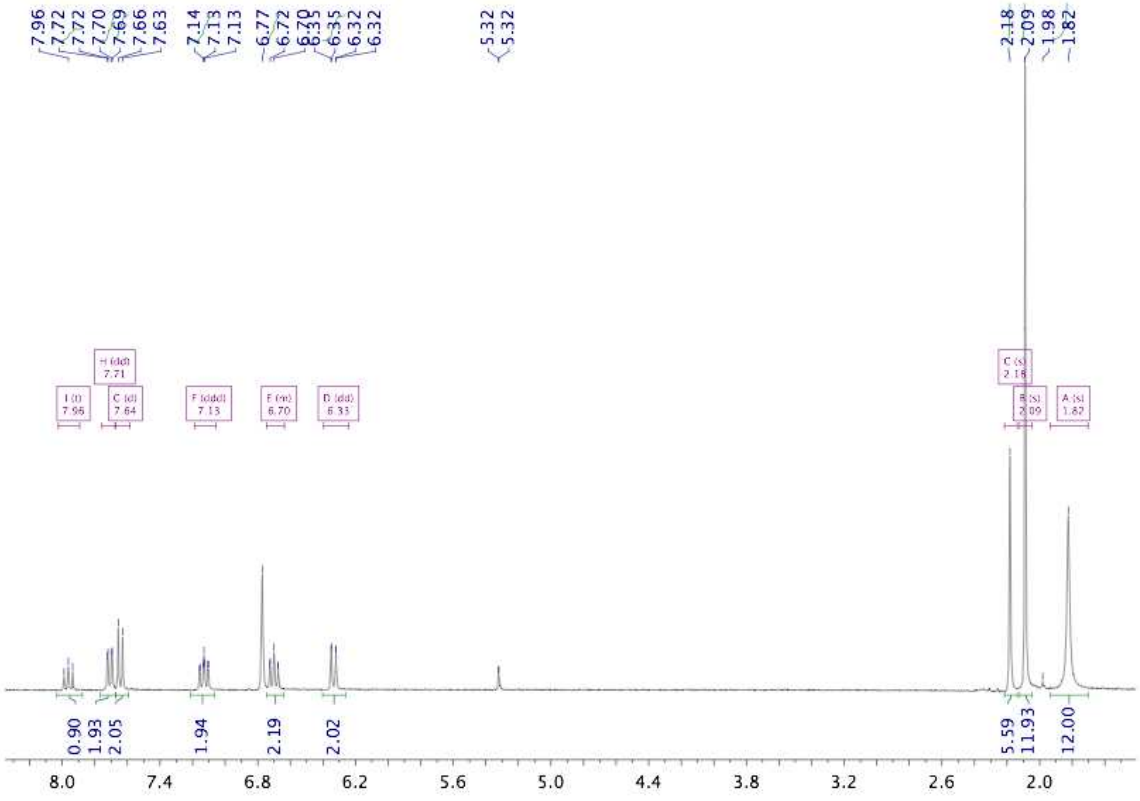


Complex 2
^{1}H Variable Temperature (d_8-toluene):

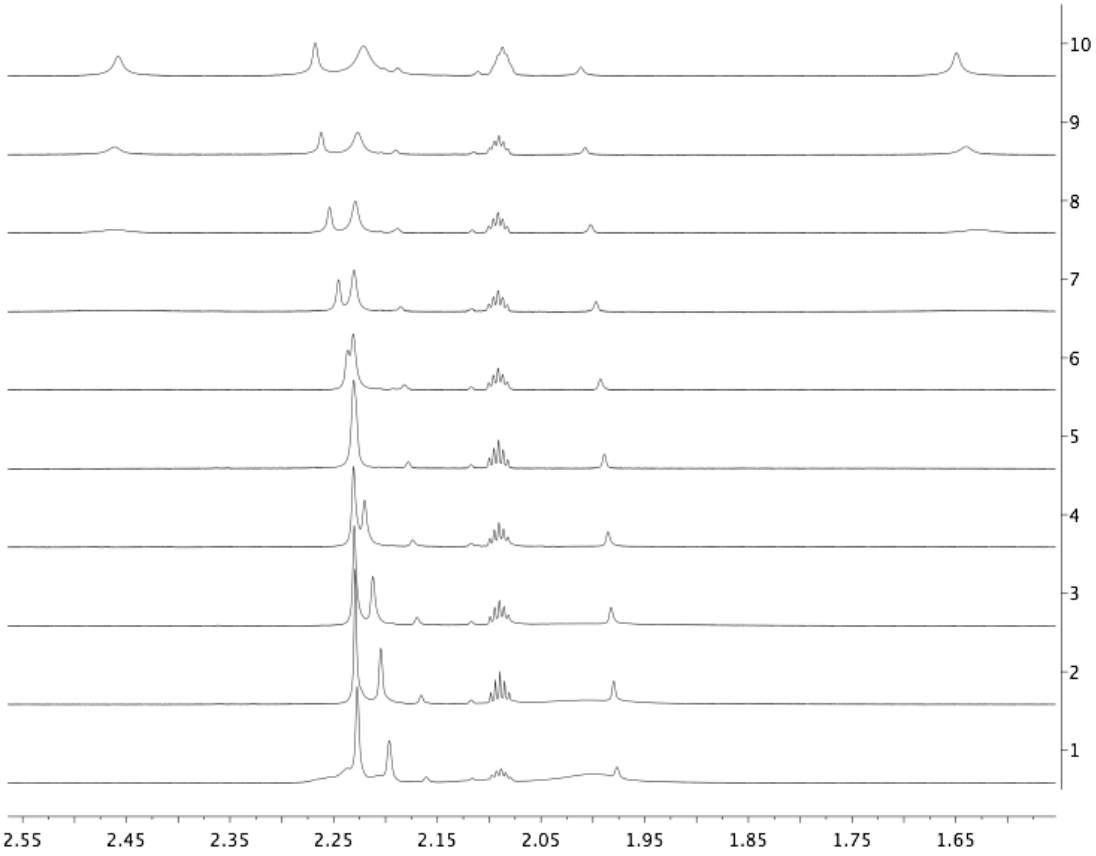


Note: VT spectrum of the alkyl region from 25 °C (1) to -80 °C (12), showing coalescence of the *ortho*-methyl groups of the mesitylene. Alkyl multiplets at ~1.4 and 0.9 ppm are pentane from recrystallization.

Complex 3
^{1}H (CD_2Cl_2)

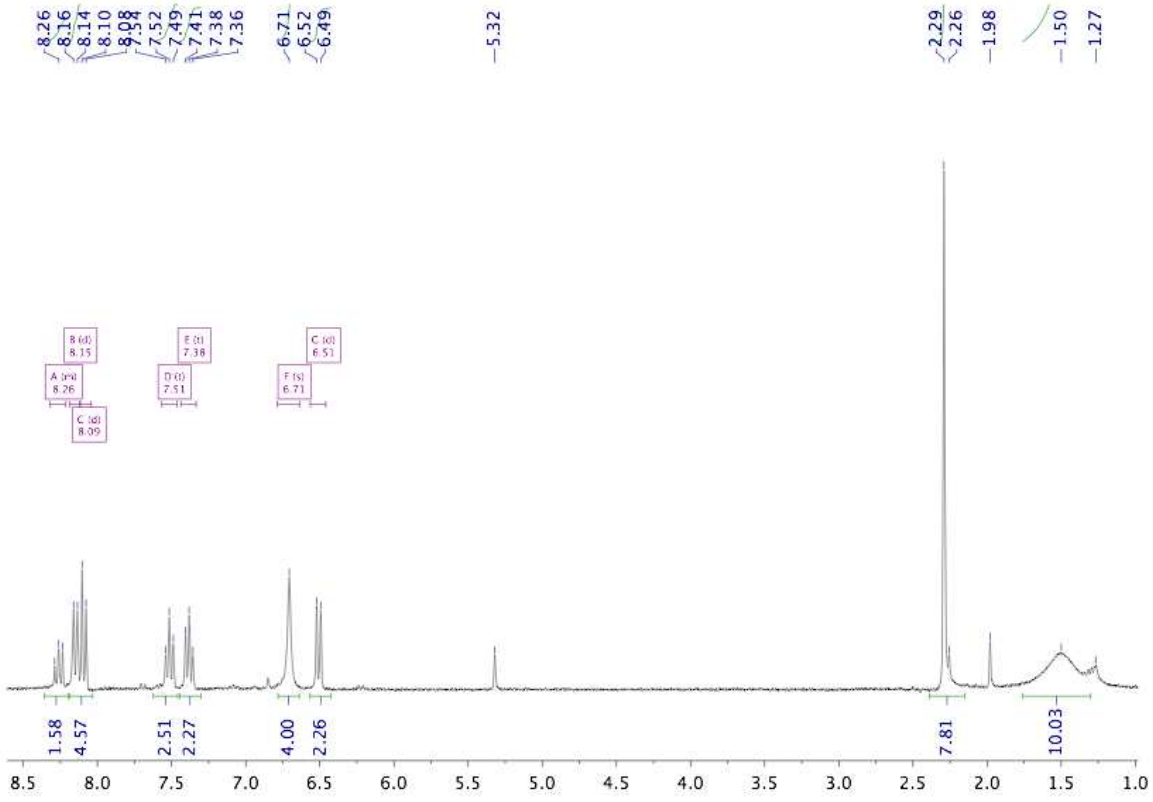


Complex 3
^{1}H Variable Temperature (d_8-toluene):



Note: VT spectrum of the alkyl region from 0 °C (1) to -90 °C (10), showing coalescence of the *ortho*-methyl groups of the mesitylene.

Complex 4
^{1}H (CD_2Cl_2)



Complex 4
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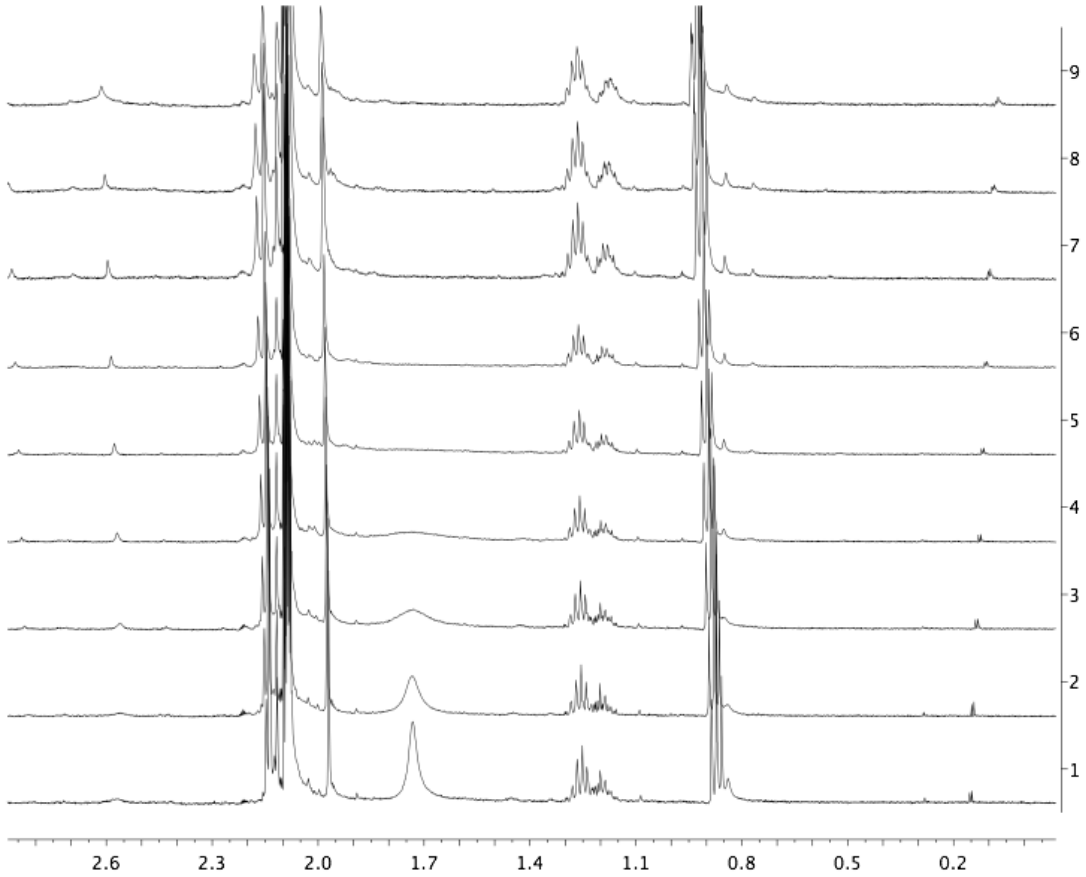
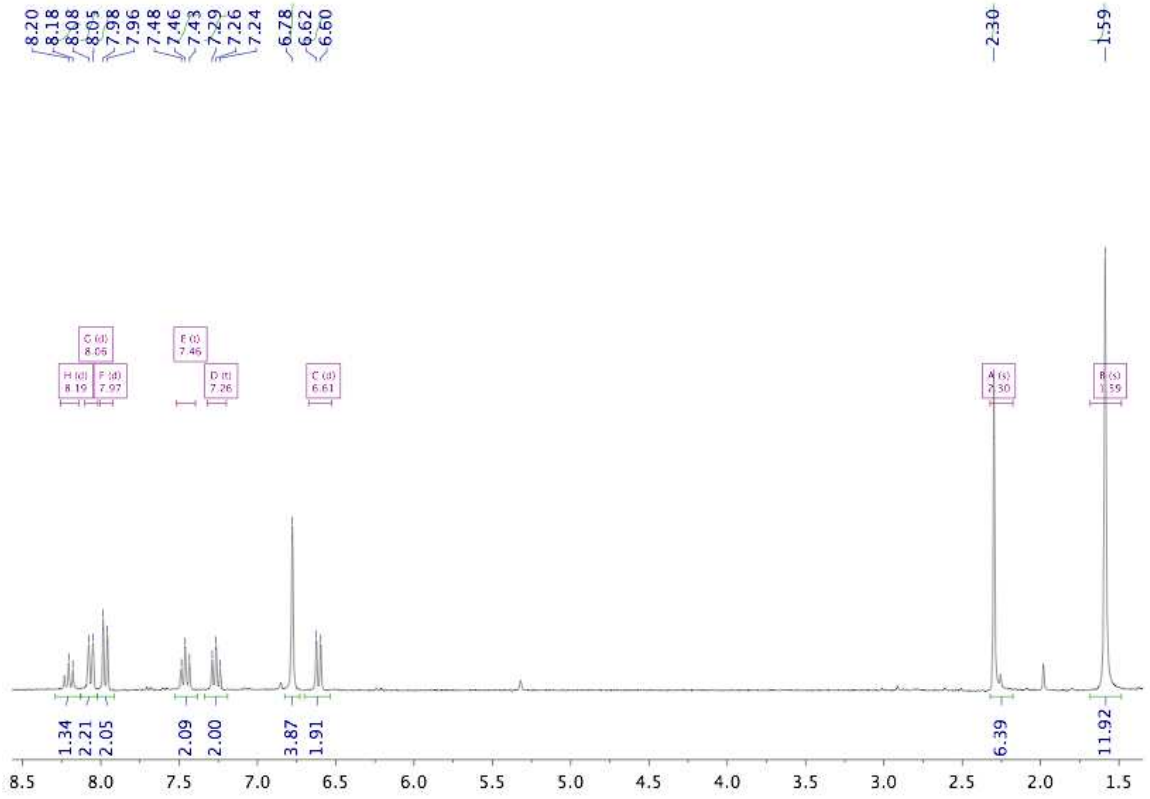
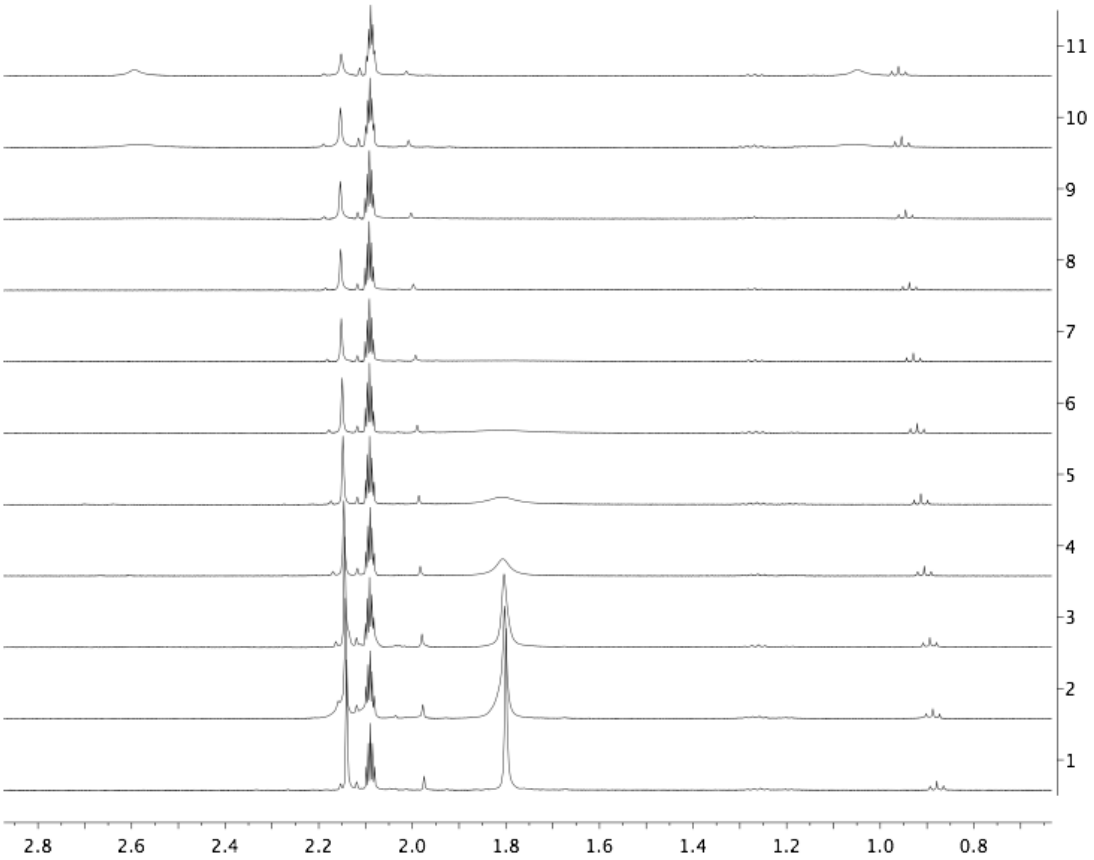


Note: VT spectrum of the alkyl region from 35 °C (1) to -50 °C (9), showing coalescence of the *ortho*-methyl groups of the mesitylene. The broad peaks at 2.6 and 0.9 ppm in spectrum 9 are the inequivalent methyl groups.

Complex 5
^{1}H (CD_2Cl_2)

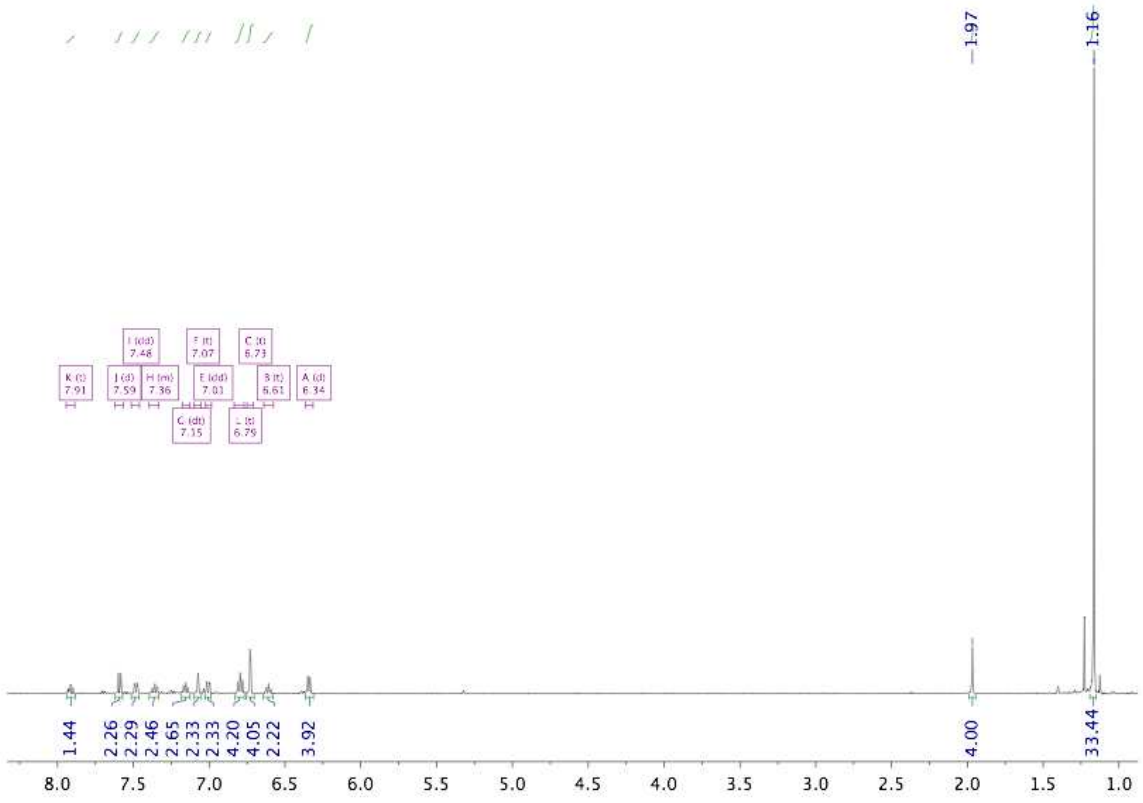


Complex 5
^{1}H Variable Temperature (d_8-toluene):

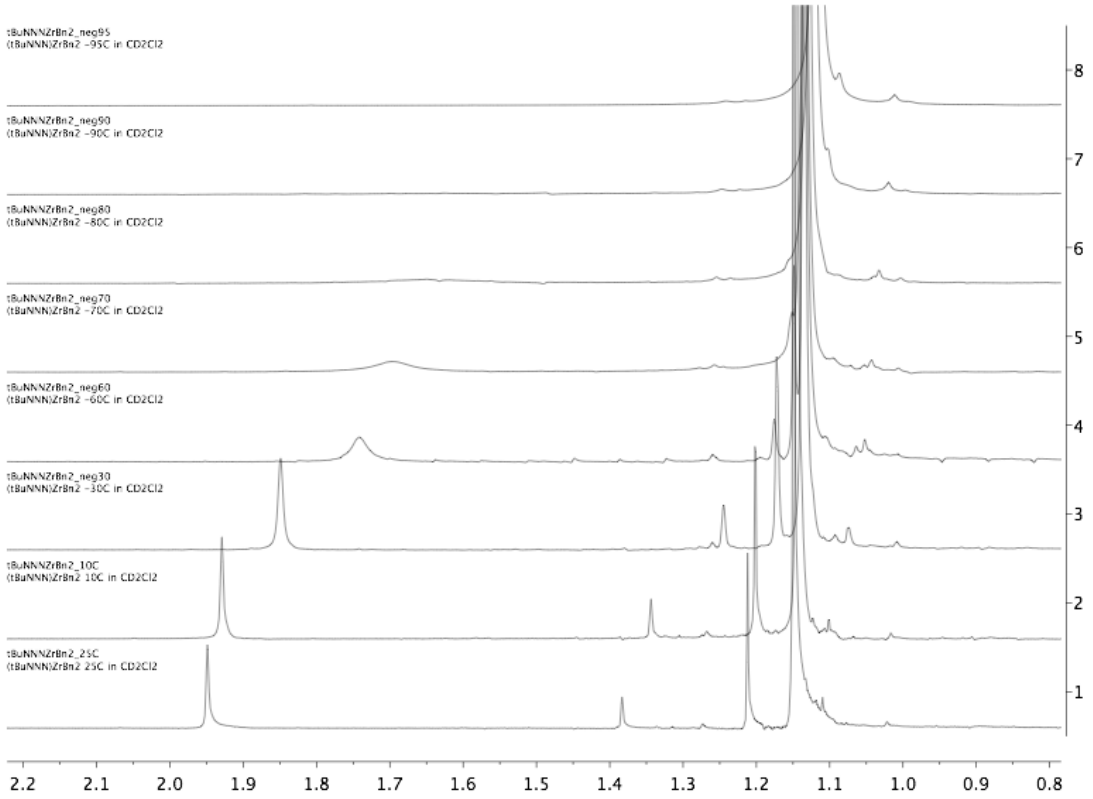


Note: VT spectrum of the alkyl region from 25 °C (1) to -90 °C (11), showing coalescence of the *ortho*-methyl groups of the mesitylene. Alkyl multiplets at ~0.9 ppm are pentane from recrystallization.

Complex 7
^{1}H (CD_2Cl_2)



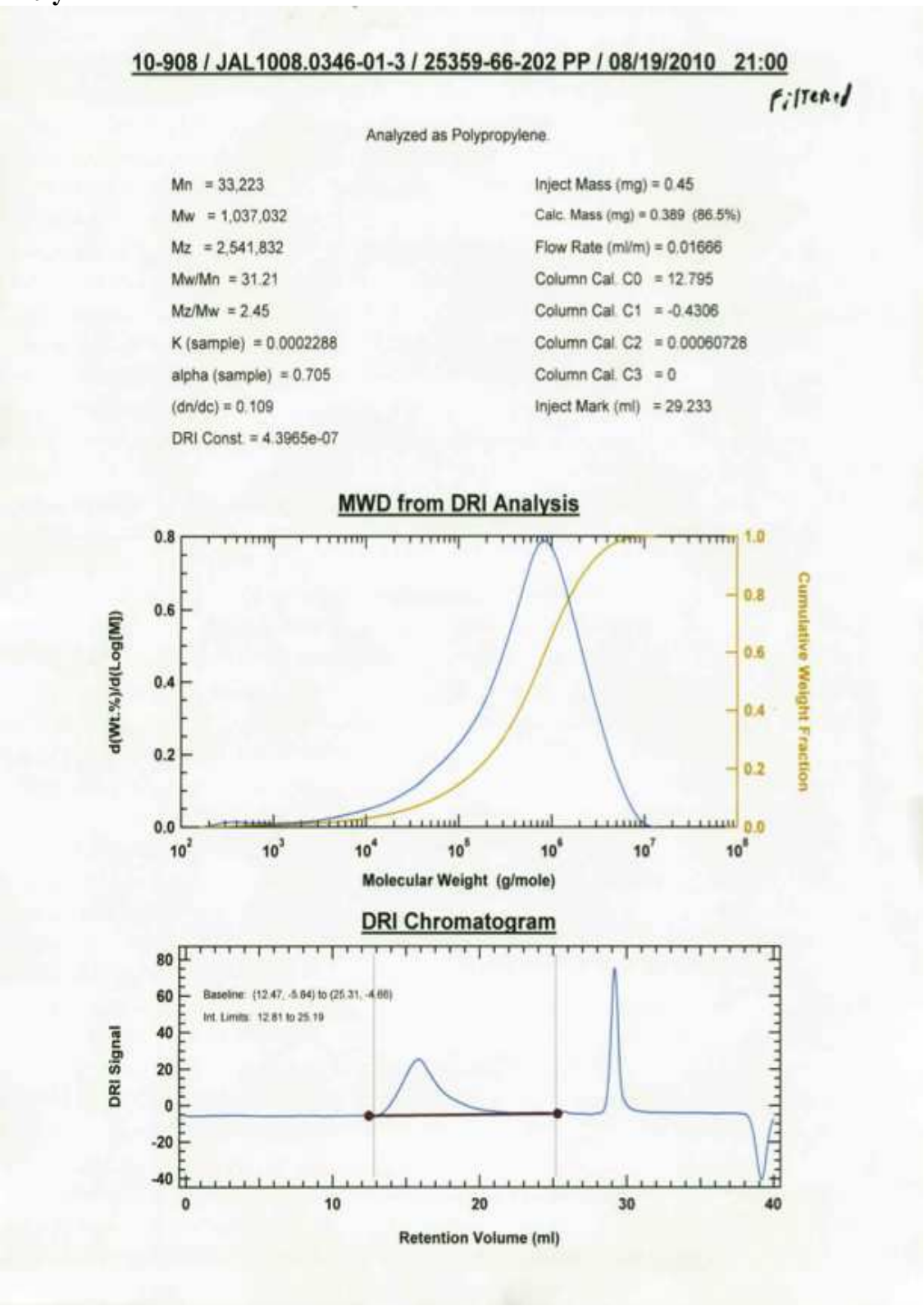
Complex 7
^{1}H Variable Temperature (CD_2Cl_2):



Note: Closeup of alkyl region of spectrum. The benzylic CH_2 resonance at 1.94 ppm broadens out as the temperature is decreased to -95 °C. Sample contained a free ligand impurity at 1.2 ppm.

GPC Data - entry #'s correlate to the Table 2 in the text

Entry 4



Entry 5

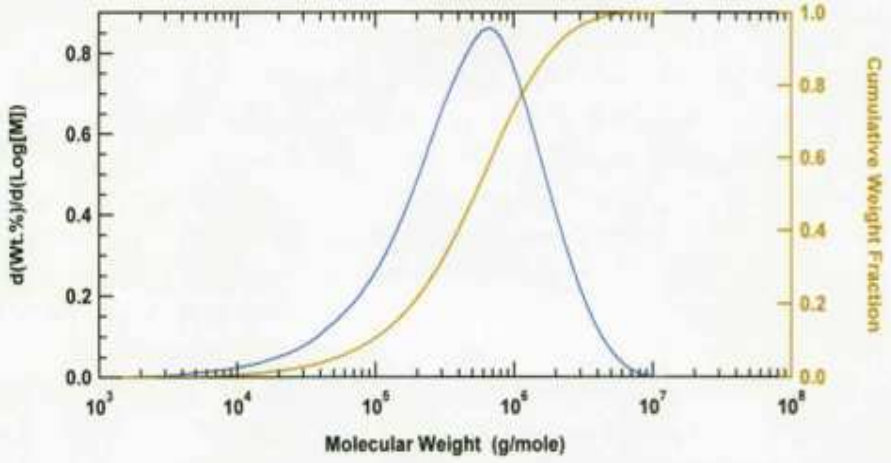
10-906 / JAL1008.0346-01-1 / 25359-66-202 PP / 08/19/2010 19:35

Filtered.

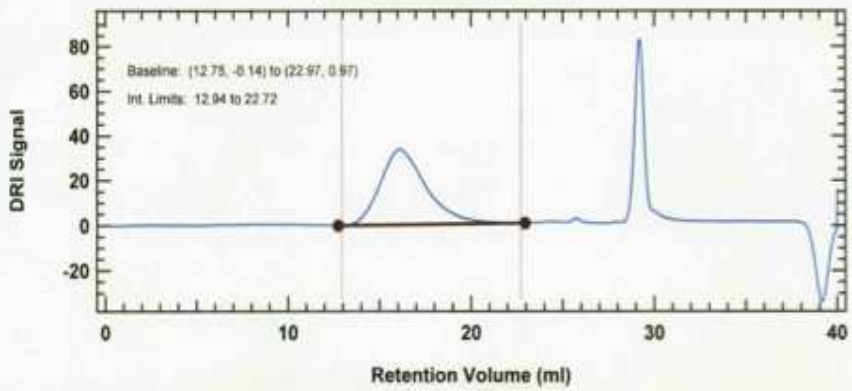
Analyzed as Polypropylene

Mn = 167,356	Inject Mass (mg) = 0.45
Mw = 821,242	Calc. Mass (mg) = 0.391 (86.8%)
Mz = 1,871,715	Flow Rate (ml/m) = 0.01666
Mw/Mn = 4.91	Column Cal. C0 = 12.795
Mz/Mw = 2.28	Column Cal. C1 = -0.4306
K (sample) = 0.0002288	Column Cal. C2 = 0.00060728
alpha (sample) = 0.705	Column Cal. C3 = 0
(dn/dc) = 0.109	Inject Mark (ml) = 29.233
DRI Const. = 4.3965e-07	

MWD from DRI Analysis



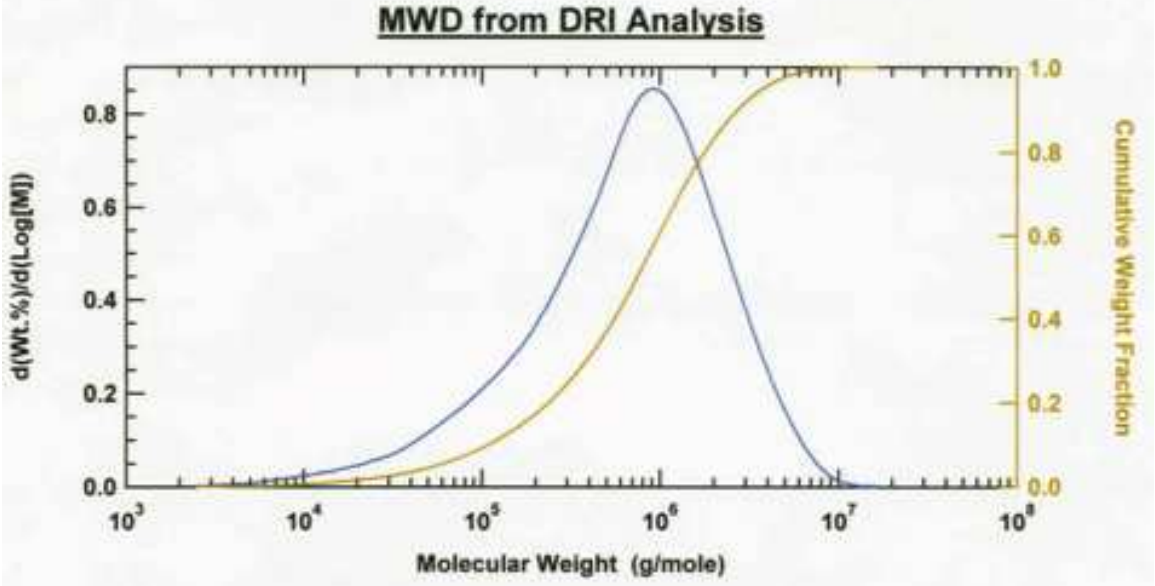
DRI Chromatogram



Entry 6

Analyzed as Polypropylene.

Mn = 206,247	Inject Mass (mg) = 0.45
Mw = 1,151,884	Calc. Mass (mg) = 0.288 (64%)
Mz = 2,610,625	Flow Rate (ml/m) = 0.01666
Mw/Mn = 5.58	Column Cal. C0 = 12.795
Mz/Mw = 2.27	Column Cal. C1 = -0.4306
K (sample) = 0.0002288	Column Cal. C2 = 0.00060728
alpha (sample) = 0.705	Column Cal. C3 = 0
(dn/dc) = 0.109	Inject Mark (ml) = 29.233
DRI Const. = 4.3965e-07	



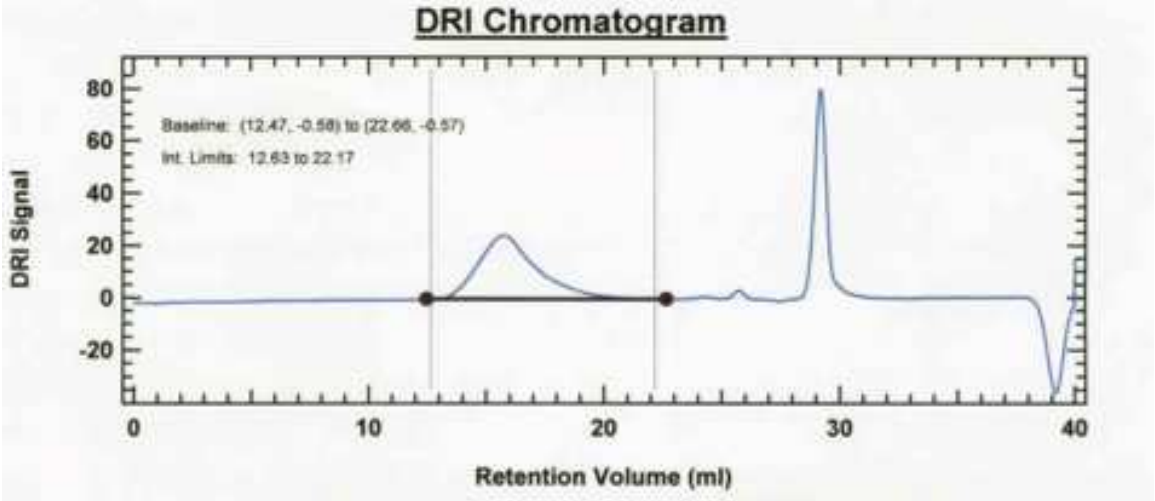


Table S1. Crystal and refinement data for complexes **2-7**.

	2	**3**	**4**
Empirical formula	$C_{39}H_{45}N_5Ti \bullet C_4H_8O$	$C_{39}H_{45}N_5Zr$	$C_{35}H_{33}N_3Cl_2Ti \bullet C_4H_8O$
Formula weight	703.80	675.02	686.55
T (K)	100(2)	100(2)	100(2)
a, Å	32.3018(14)	16.6663(7)	36.2843(15)
b, Å	14.9286(7)	11.7383(5)	11.6553(5)
c, Å	16.9824(8)	18.2430(7)	16.9710(8)
α, deg			
β, deg	110.525(2)	104.524(2)	109.665(2)
γ, deg			
Volume, $Å^3$	7669.4(6)	3454.9(2)	6758.5(5)
Z	8	4	8
Crystal system	Monoclinic	Monoclinic	Monoclinic
Space group	C 2/*c*	P 2_1/*c*	C2/*c*
d_{calc}, g/cm^3	1.219	1.298	1.349
θ range, deg	1.83 to 30.50	2.08 to 33.50	1.85 to 33.19
μ, mm^{-1}	0.263	0.352	0.448
Abs. Correction	None	None	None
GOF	2.491	1.774	1.938
R_1 ,[a] wR_2 [b] [I>2σ(I)]	R1 = 0.0677, *w*R2 = 0.1063	R1 = 0.0325, *w*R2 = 0.0505	R1 = 0.0420, *w*R2 = 0.0523

[a] $R_1 = \sum||F_o| - |F_c||/\sum|F_o|$. [b] $wR_2 = [\sum[w(F_o^2-F_c^2)^2]/\sum[w(F_o^2)^2]]^{1/2}$.

	5	**6**	**7**
Empirical formula	$C_{35}H_{33}N_3Cl_2Zr \bullet 0.75(C_7H_8)$	$C_{39}H_{41}N_3OCl_2Zr$	$C_{59}H_{67}N_3Zr \bullet C_5H_{12}$
Formula weight	726.86	729.87	981.52
T (K)	100(2)	100(2)	100(2)
a, Å	17.1199(7)	11.7169(5)	10.5191(4)
b, Å	23.1437(10)	12.7754(6)	12.8102(5)
c, Å	34.8624(15)	13.0786(6)	20.6372(8)
α, deg		61.415(2)	96.952(2)
β, deg		85.745(3)	90.511(2)
γ, deg		85.899(3)	98.726(2)
Volume, Å^3	13813.1(10)	1712.89(13)	2727.45(18)
Z	16	2	2
Crystal system	Orthorhombic	Triclinic	Triclinic
Space group	$P2_12_12_1$	P-1	P-1
d_{calc}, g/cm^3	1.398	1.415	1.195
θ range, deg	1.48 to 36.20	1.74 to 39.30	1.79 to 27.45
μ, mm^{-1}	0.506	0.513	0.243
Abs. Correction	None	Empirical, Twinabs, Multi-scan	None
GOF	1.589	1.957	1.925
R_1,[a] wR_2[b] [I>2σ(I)]	R1 = 0.0345, wR2 = 0.0543	R1 = 0.0381, wR2 = 0.0678	R1 = 0.0352, wR2 = 0.0492

[a] $R_1 = \sum||F_o| - |F_c||/\sum|F_o|$. [b] $wR_2 = [\sum[w(F_o^2-F_c^2)^2]/\sum[w(F_o^2)^2]]^{1/2}$.

CALIFORNIA INSTITUTE OF TECHNOLOGY

BECKMAN INSTITUTE
X-RAY CRYSTALLOGRAPHY LABORATORY

Date 16 November 2010

Crystal Structure Analysis of:

IAT47

(Complex 2)

For Investigator: Ian Tonks ext. 6576

Advisor: J. E. Bercaw ext. 6577

By Michael W. Day 116 Beckman ext. 2734
e-mail: mikeday@caltech.edu

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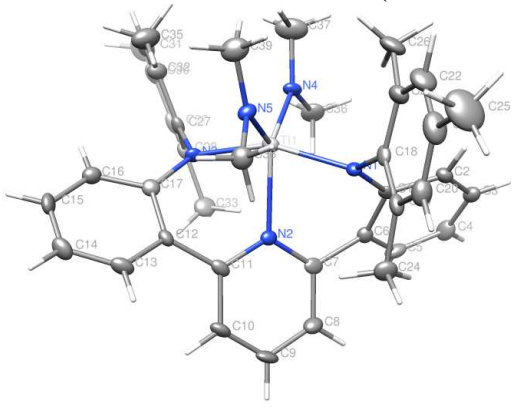


IAT47

Note: The crystallographic data have been deposited in the Cambridge Database (CCDC). The deposition number is 800977.

Table 1. Crystal data and structure refinement for IAT47 (CCDC 800979).

Empirical formula	$C_{39}H_{45}N_5Ti \bullet C_4H_8O$
Formula weight	703.80
Crystallization Solvent	THF/pentane
Crystal Habit	Blade
Crystal size	0.22 x 0.14 x 0.06 mm^3
Crystal color	Orange

Data Collection

Type of diffractometer	Bruker KAPPA APEX II	
Wavelength	0.71073 Å MoKα	
Data Collection Temperature	100(2) K	
θ range for 7821 reflections used in lattice determination	2.40 to 22.40°	
Unit cell dimensions	a = 32.3018(14) Å b = 14.9286(7) Å c = 16.9824(8) Å	α= 90° β= 110.525(2)° γ = 90°
Volume	7669.4(6) $Å^3$	
Z	8	
Crystal system	Monoclinic	
Space group	C 2/*c*	
Density (calculated)	1.219 Mg/m^3	
F(000)	3008	
Data collection program	Bruker APEX2 v2009.7-0	
θ range for data collection	1.83 to 30.50°	
Completeness to θ = 30.50°	93.3 %	
Index ranges	$-45 \le h \le 44$, $-21 \le k \le 20$, $-24 \le l \le 23$	
Data collection scan type	ω scans; 8 settings	
Data reduction program	Bruker SAINT-Plus v7.66A	
Reflections collected	63929	
Independent reflections	10914 [R_{int}= 0.0797]	
Absorption coefficient	0.263 mm^{-1}	
Absorption correction	None	
Max. and min. transmission	0.9844 and 0.9445	

Table 1 (cont.)

Structure solution and Refinement

Structure solution program	SHELXS-97 (Sheldrick, 2008)
Primary solution method	Direct methods
Secondary solution method	Difference Fourier map
Hydrogen placement	Geometric positions
Structure refinement program	SHELXL-97 (Sheldrick, 2008)
Refinement method	Full matrix least-squares on F^2
Data / restraints / parameters	10914 / 38 / 461
Treatment of hydrogen atoms	Riding
Goodness-of-fit on F^2	2.491
Final R indices [$I>2\sigma(I)$, 6025 reflections]	R1 = 0.0677, *w*R2 = 0.1063
R indices (all data)	R1 = 0.1333, *w*R2 = 0.1088
Type of weighting scheme used	Sigma
Weighting scheme used	$w=1/\sigma^2(Fo^2)$
Max shift/error	0.001
Average shift/error	0.000
Largest diff. peak and hole	1.602 and -0.747 e.Å^{-3}

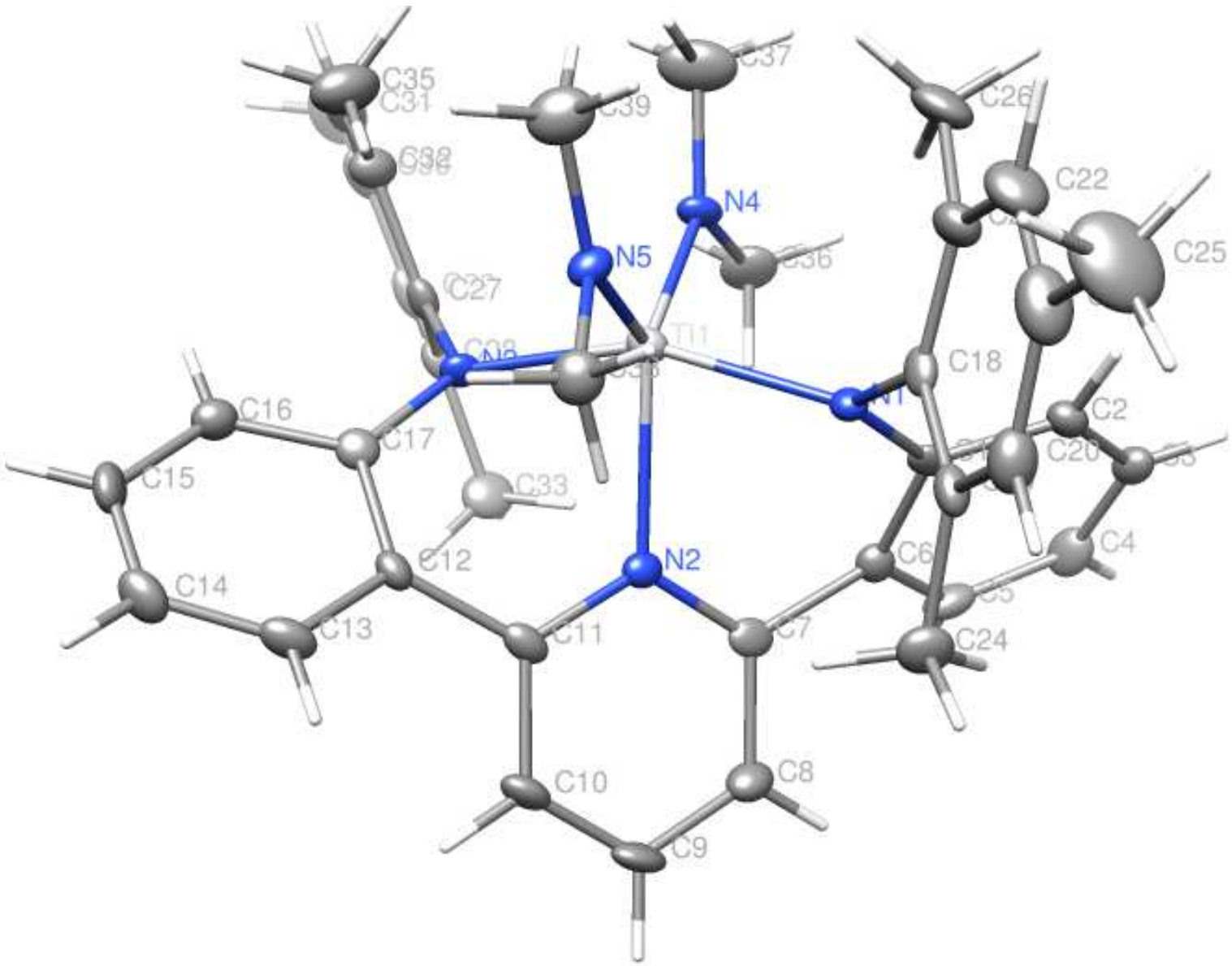
Special Refinement Details

Crystals were mounted on a glass fiber using Paratone oil then placed on the diffractometer under a nitrogen stream at 100K.

The crystal contains a solvent of crystallization, modeled as THF. Weak distance and angle restraints were applied and the ADP's were restrained toward isotropic behavior.

Refinement of F^2 against ALL reflections. The weighted R-factor (*w*R) and goodness of fit (S) are based on F^2, conventional R-factors (R) are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R-factors based on ALL data will be even larger.

All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.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C31
C39
C37
C26
C22
C25
N4
N5
C27
C36
Ti1
C18
C16
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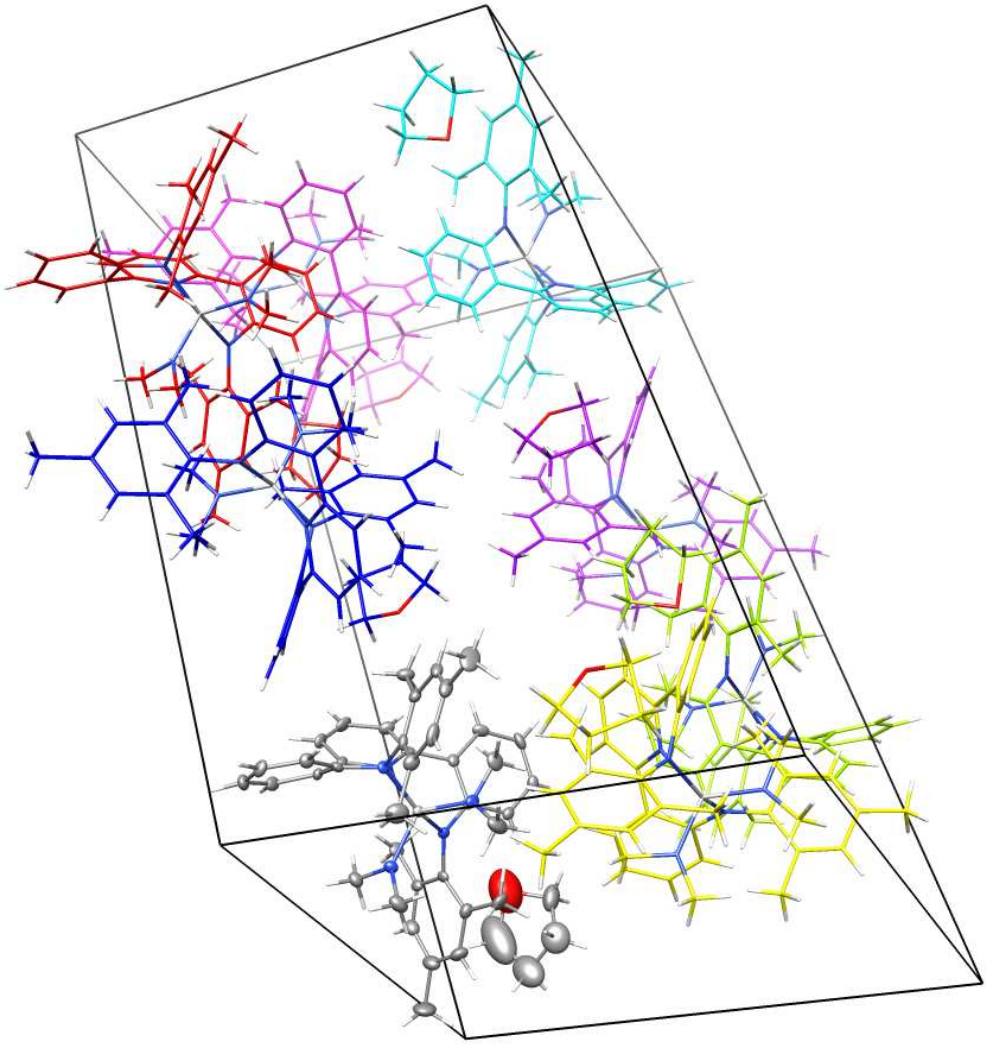
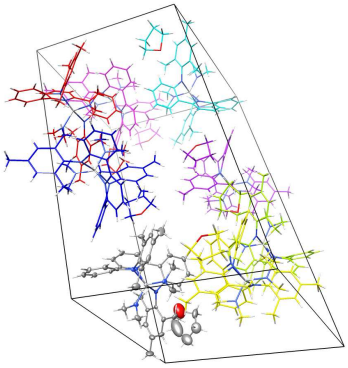
C13
C11
C7
C24
C10
C8
C9





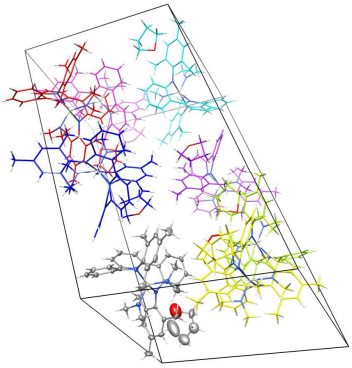


Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for IAT47 (CCDC 800979). U(eq) is defined as the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U_{eq}
Ti(1)	1259(1)	5582(1)	2188(1)	15(1)
N(1)	1346(1)	4209(2)	2120(2)	14(1)
N(2)	1827(1)	5521(2)	1779(2)	14(1)
N(3)	1428(1)	6927(2)	2215(2)	14(1)
N(4)	1260(1)	5641(2)	3299(2)	17(1)
N(5)	690(1)	5578(2)	1373(2)	17(1)
C(1)	1735(1)	3865(2)	2666(2)	14(1)
C(2)	1760(1)	3092(2)	3165(2)	17(1)
C(3)	2148(1)	2810(2)	3761(2)	20(1)
C(4)	2538(1)	3277(3)	3896(2)	23(1)
C(5)	2538(1)	3981(3)	3382(2)	22(1)
C(6)	2146(1)	4282(2)	2752(2)	16(1)
C(7)	2159(1)	4922(2)	2100(2)	15(1)
C(8)	2502(1)	4835(2)	1769(2)	22(1)
C(9)	2480(1)	5347(3)	1079(2)	24(1)
C(10)	2145(1)	5953(2)	750(2)	20(1)
C(11)	1819(1)	6062(2)	1120(2)	16(1)
C(12)	1510(1)	6804(2)	853(2)	15(1)
C(13)	1391(1)	7126(3)	31(2)	22(1)
C(14)	1214(1)	7967(3)	-191(2)	24(1)
C(15)	1171(1)	8520(3)	435(2)	24(1)
C(16)	1266(1)	8208(3)	1244(2)	20(1)
C(17)	1403(1)	7320(3)	1464(2)	16(1)
C(18)	1017(1)	3581(2)	1657(2)	16(1)
C(19)	1065(1)	3166(2)	954(2)	18(1)
C(20)	725(1)	2640(2)	434(2)	25(1)
C(21)	335(1)	2501(3)	587(3)	33(1)
C(22)	308(1)	2876(3)	1313(3)	32(1)
C(23)	644(1)	3406(3)	1863(2)	22(1)
C(24)	1482(1)	3282(2)	763(2)	23(1)
C(25)	-40(1)	1938(3)	2(3)	54(1)
C(26)	608(1)	3755(3)	2671(2)	33(1)
C(27)	1498(1)	7518(2)	2912(2)	18(1)
C(28)	1933(1)	7700(2)	3442(2)	19(1)
C(29)	2006(1)	8240(2)	4149(2)	24(1)
C(30)	1666(1)	8633(3)	4340(2)	27(1)
C(31)	1240(1)	8460(3)	3803(2)	28(1)
C(32)	1143(1)	7909(3)	3093(2)	24(1)
C(33)	2318(1)	7313(2)	3256(2)	26(1)
C(34)	1748(1)	9225(3)	5097(2)	44(1)
C(35)	668(1)	7756(3)	2523(2)	38(1)
C(36)	1690(1)	5559(3)	3932(2)	28(1)
C(37)	926(1)	5762(3)	3667(2)	39(1)
C(38)	674(1)	5316(2)	526(2)	23(1)
C(39)	246(1)	5715(3)	1379(2)	31(1)

C(52)	855(2)	743(4)	2669(3)	80(2)
C(53)	1348(1)	617(3)	2792(3)	57(1)
O(54)	1438(2)	882(4)	1986(3)	189(2)
C(55)	980(2)	446(5)	1413(4)	179(4)
C(56)	607(2)	329(4)	1794(4)	102(2)

Table 3. Selected bond lengths [Å] and angles [°] for IAT47 (CCDC 800979).

Ti(1)-N(5)	1.872(2)	N(5)-Ti(1)-N(4)	113.40(11)
Ti(1)-N(4)	1.887(3)	N(5)-Ti(1)-N(1)	94.16(12)
Ti(1)-N(1)	2.077(3)	N(4)-Ti(1)-N(1)	98.54(12)
Ti(1)-N(3)	2.077(3)	N(5)-Ti(1)-N(3)	101.65(12)
Ti(1)-N(2)	2.181(2)	N(4)-Ti(1)-N(3)	91.39(12)
		N(1)-Ti(1)-N(3)	156.23(9)
		N(5)-Ti(1)-N(2)	118.80(11)
		N(4)-Ti(1)-N(2)	127.81(10)
		N(1)-Ti(1)-N(2)	78.17(10)
		N(3)-Ti(1)-N(2)	78.68(11)

Table 4. Bond lengths [Å] and angles [°] for IAT47 (CCDC 800979).

Ti(1)-N(5)	1.872(2)	C(52)-C(56)	1.547(6)
Ti(1)-N(4)	1.887(3)	C(52)-C(53)	1.541(4)
Ti(1)-N(1)	2.077(3)	C(53)-O(54)	1.548(5)
Ti(1)-N(3)	2.077(3)	O(54)-C(55)	1.597(6)
Ti(1)-N(2)	2.181(2)	C(55)-C(56)	1.566(5)
N(1)-C(1)	1.373(4)		
N(1)-C(18)	1.429(4)	N(5)-Ti(1)-N(4)	113.40(11)
N(2)-C(7)	1.355(4)	N(5)-Ti(1)-N(1)	94.16(12)
N(2)-C(11)	1.374(4)	N(4)-Ti(1)-N(1)	98.54(12)
N(3)-C(17)	1.381(4)	N(5)-Ti(1)-N(3)	101.65(12)
N(3)-C(27)	1.429(4)	N(4)-Ti(1)-N(3)	91.39(12)
N(4)-C(36)	1.432(3)	N(1)-Ti(1)-N(3)	156.23(9)
N(4)-C(37)	1.435(3)	N(5)-Ti(1)-N(2)	118.80(11)
N(5)-C(39)	1.454(3)	N(4)-Ti(1)-N(2)	127.81(10)
N(5)-C(38)	1.472(4)	N(1)-Ti(1)-N(2)	78.17(10)
C(1)-C(2)	1.418(4)	N(3)-Ti(1)-N(2)	78.68(11)
C(1)-C(6)	1.425(4)	C(1)-N(1)-C(18)	117.0(3)
C(2)-C(3)	1.372(4)	C(1)-N(1)-Ti(1)	116.2(2)
C(3)-C(4)	1.389(4)	C(18)-N(1)-Ti(1)	126.1(2)
C(4)-C(5)	1.365(5)	C(7)-N(2)-C(11)	120.2(3)
C(5)-C(6)	1.415(4)	C(7)-N(2)-Ti(1)	122.4(2)
C(6)-C(7)	1.474(4)	C(11)-N(2)-Ti(1)	117.1(2)
C(7)-C(8)	1.413(4)	C(17)-N(3)-C(27)	116.4(3)
C(8)-C(9)	1.379(4)	C(17)-N(3)-Ti(1)	117.4(2)
C(9)-C(10)	1.370(4)	C(27)-N(3)-Ti(1)	125.4(2)
C(10)-C(11)	1.413(4)	C(36)-N(4)-C(37)	111.3(3)
C(11)-C(12)	1.453(4)	C(36)-N(4)-Ti(1)	114.1(2)
C(12)-C(13)	1.397(4)	C(37)-N(4)-Ti(1)	134.6(2)
C(12)-C(17)	1.427(5)	C(39)-N(5)-C(38)	110.3(2)
C(13)-C(14)	1.376(5)	C(39)-N(5)-Ti(1)	135.1(2)
C(14)-C(15)	1.391(5)	C(38)-N(5)-Ti(1)	114.36(19)
C(15)-C(16)	1.379(4)	N(1)-C(1)-C(2)	123.7(3)
C(16)-C(17)	1.406(5)	N(1)-C(1)-C(6)	120.3(3)
C(18)-C(23)	1.391(4)	C(2)-C(1)-C(6)	116.0(3)
C(18)-C(19)	1.401(5)	C(3)-C(2)-C(1)	122.4(3)
C(19)-C(20)	1.387(4)	C(2)-C(3)-C(4)	120.7(4)
C(19)-C(24)	1.501(4)	C(5)-C(4)-C(3)	118.9(3)
C(20)-C(21)	1.387(4)	C(4)-C(5)-C(6)	121.8(3)
C(21)-C(22)	1.385(5)	C(5)-C(6)-C(1)	119.6(3)
C(21)-C(25)	1.521(5)	C(5)-C(6)-C(7)	121.3(3)
C(22)-C(23)	1.403(5)	C(1)-C(6)-C(7)	118.5(3)
C(23)-C(26)	1.509(5)	N(2)-C(7)-C(8)	121.3(3)
C(27)-C(28)	1.404(4)	N(2)-C(7)-C(6)	120.1(3)
C(27)-C(32)	1.416(4)	C(8)-C(7)-C(6)	118.3(3)
C(28)-C(29)	1.396(5)	C(9)-C(8)-C(7)	118.3(3)
C(28)-C(33)	1.503(4)	C(10)-C(9)-C(8)	120.7(3)
C(29)-C(30)	1.380(5)	C(9)-C(10)-C(11)	120.0(3)
C(30)-C(31)	1.384(5)	N(2)-C(11)-C(10)	119.3(3)
C(30)-C(34)	1.504(5)	N(2)-C(11)-C(12)	121.2(3)
C(31)-C(32)	1.401(5)	C(10)-C(11)-C(12)	119.2(3)
C(32)-C(35)	1.516(4)	C(13)-C(12)-C(17)	119.2(3)

C(13)-C(12)-C(11)	119.5(3)
C(17)-C(12)-C(11)	119.9(3)
C(14)-C(13)-C(12)	122.0(3)
C(13)-C(14)-C(15)	118.4(4)
C(16)-C(15)-C(14)	120.9(4)
C(15)-C(16)-C(17)	121.5(4)
N(3)-C(17)-C(16)	123.8(3)
N(3)-C(17)-C(12)	119.5(3)
C(16)-C(17)-C(12)	116.7(3)
C(23)-C(18)-C(19)	119.7(3)
C(23)-C(18)-N(1)	121.8(3)
C(19)-C(18)-N(1)	118.4(3)
C(20)-C(19)-C(18)	119.6(3)
C(20)-C(19)-C(24)	119.9(3)
C(18)-C(19)-C(24)	120.6(3)
C(21)-C(20)-C(19)	122.2(4)
C(22)-C(21)-C(20)	116.9(4)
C(22)-C(21)-C(25)	121.3(4)
C(20)-C(21)-C(25)	121.8(4)
C(21)-C(22)-C(23)	122.9(4)
C(18)-C(23)-C(22)	118.4(4)
C(18)-C(23)-C(26)	120.9(3)
C(22)-C(23)-C(26)	120.7(3)
C(28)-C(27)-C(32)	119.1(3)
C(28)-C(27)-N(3)	119.0(3)
C(32)-C(27)-N(3)	121.9(3)
C(29)-C(28)-C(27)	119.5(3)
C(29)-C(28)-C(33)	120.0(3)
C(27)-C(28)-C(33)	120.5(3)
C(30)-C(29)-C(28)	122.7(4)
C(31)-C(30)-C(29)	117.1(4)
C(31)-C(30)-C(34)	120.6(4)
C(29)-C(30)-C(34)	122.4(4)
C(30)-C(31)-C(32)	123.2(4)
C(31)-C(32)-C(27)	118.4(4)
C(31)-C(32)-C(35)	120.6(3)
C(27)-C(32)-C(35)	121.0(3)
C(56)-C(52)-C(53)	104.2(3)
O(54)-C(53)-C(52)	110.7(4)
C(55)-O(54)-C(53)	90.8(4)
C(56)-C(55)-O(54)	118.4(5)
C(52)-C(56)-C(55)	97.9(3)

Table 5. Anisotropic displacement parameters ($Å^2 x 10^4$) for IAT47 (CCDC 800979). The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Ti(1)	137(3)	187(4)	119(3)	3(3)	47(3)	-7(3)
N(1)	108(14)	150(20)	156(17)	15(14)	56(13)	11(13)
N(2)	136(14)	140(20)	151(16)	-30(15)	54(12)	-19(14)
N(3)	166(15)	160(20)	111(17)	-31(14)	65(14)	1(13)
N(4)	182(15)	200(20)	156(17)	-44(15)	106(13)	-22(15)
N(5)	128(14)	180(20)	185(17)	-9(16)	44(13)	24(15)
C(1)	179(19)	180(30)	100(20)	-62(17)	83(16)	-29(17)
C(2)	193(19)	180(30)	170(20)	-3(18)	94(17)	-15(17)
C(3)	240(20)	210(30)	170(20)	38(18)	104(18)	65(18)
C(4)	270(20)	270(30)	120(20)	25(19)	20(18)	84(19)
C(5)	129(19)	270(30)	250(20)	-76(19)	67(17)	18(17)
C(6)	189(18)	150(30)	126(19)	-17(17)	58(16)	3(17)
C(7)	147(18)	160(20)	150(20)	-72(17)	40(16)	-66(17)
C(8)	172(19)	210(30)	290(20)	-22(19)	112(18)	-4(17)
C(9)	270(20)	260(30)	280(20)	-50(19)	219(19)	-70(18)
C(10)	260(20)	240(30)	160(20)	-14(18)	144(18)	-46(18)
C(11)	200(20)	180(30)	110(20)	-28(17)	65(17)	-69(17)
C(12)	148(18)	160(20)	140(20)	39(18)	50(16)	-18(16)
C(13)	240(20)	290(30)	140(20)	-34(19)	96(18)	-58(19)
C(14)	260(20)	300(30)	140(20)	60(20)	49(18)	-26(19)
C(15)	250(20)	160(30)	310(30)	100(20)	110(20)	26(18)
C(16)	200(20)	220(30)	200(20)	-9(19)	88(18)	7(18)
C(17)	114(18)	210(30)	130(20)	-11(18)	9(16)	-22(17)
C(18)	122(18)	130(20)	160(20)	59(17)	-9(16)	19(16)
C(19)	154(19)	130(20)	220(20)	47(18)	29(17)	-14(17)
C(20)	290(20)	200(30)	220(20)	-1(19)	41(19)	-3(19)
C(21)	280(20)	270(30)	350(30)	30(20)	0(20)	-70(20)
C(22)	190(20)	350(30)	410(30)	50(20)	110(20)	-60(20)
C(23)	220(20)	240(30)	240(20)	60(20)	118(19)	6(18)
C(24)	270(20)	260(30)	170(20)	-69(19)	92(18)	-4(18)
C(25)	390(30)	610(40)	480(30)	-50(30)	0(20)	-270(30)
C(26)	260(20)	410(30)	370(30)	90(20)	200(20)	-60(20)
C(27)	280(20)	150(30)	140(20)	45(17)	95(18)	6(18)
C(28)	210(20)	180(30)	180(20)	26(18)	71(18)	-49(17)
C(29)	350(20)	190(30)	160(20)	1(19)	60(19)	-110(20)
C(30)	440(30)	210(30)	200(20)	-25(19)	150(20)	-80(20)
C(31)	480(30)	220(30)	270(30)	50(20)	280(20)	60(20)
C(32)	240(20)	250(30)	270(20)	0(20)	131(19)	2(19)
C(33)	190(20)	290(30)	290(30)	-40(20)	60(19)	-63(18)
C(34)	790(30)	300(30)	300(30)	-180(20)	270(20)	-130(30)
C(35)	320(20)	430(30)	450(30)	-90(20)	190(20)	10(20)
C(36)	340(20)	360(30)	190(20)	-50(20)	138(18)	40(20)
C(37)	280(20)	660(40)	220(20)	-100(20)	71(19)	-60(20)
C(38)	230(20)	260(30)	180(20)	1(18)	35(17)	8(17)
C(39)	230(20)	420(30)	230(20)	-30(20)	32(18)	30(20)

C(52)	960(40)	530(40)	950(50)	-10(30)	380(40)	250(30)
C(53)	870(40)	480(40)	450(30)	70(30)	360(30)	-10(30)
O(54)	2190(50)	2090(60)	1260(40)	190(40)	470(40)	80(50)
C(55)	2840(80)	860(60)	1180(60)	30(50)	100(60)	370(60)
C(56)	1200(50)	540(50)	1090(50)	40(40)	90(40)	100(40)

CALIFORNIA INSTITUTE OF TECHNOLOGY

BECKMAN INSTITUTE

X-RAY CRYSTALLOGRAPHY LABORATORY

Date 16 November 2010

Crystal Structure Analysis of:

IAT48

(Complex 3)

For Investigator: Ian Tonks ext. 6576

Advisor: J. E. Bercaw ext. 6577

By Michael W. Day 116 Beckman ext. 2734

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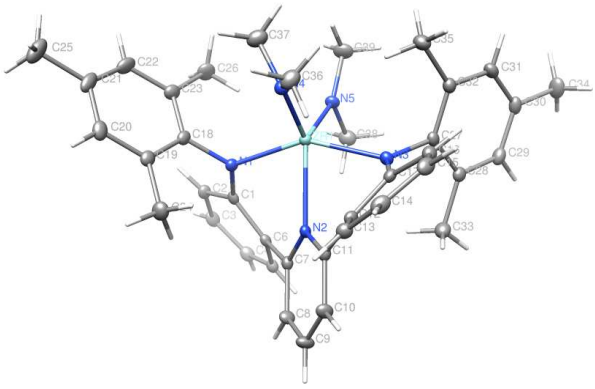
Contents



IAT48

Note: The crystallographic data have been deposited in the Cambridge Database (CCDC). The deposition number is 800978.

Table 1. Crystal data and structure refinement for IAT48 (CCDC 800978).

Empirical formula	$C_{39}H_{45}N_5Zr$
Formula weight	675.02
Crystallization Solvent	Benzene/pentane
Crystal Habit	Block
Crystal size	0.19 x 0.17 x 0.16 mm^3
Crystal color	Yellow

Data Collection

Type of diffractometer	Bruker KAPPA APEX II	
Wavelength	0.71073 Å MoKα	
Data Collection Temperature	100(2) K	
θ range for 9934 reflections used in lattice determination	2.28 to 33.30°	
Unit cell dimensions	a = 16.6663(7) Å b = 11.7383(5) Å c = 18.2430(7) Å	α= 90° β= 104.524(2)° γ = 90°
Volume	3454.9(2) $Å^3$	
Z	4	
Crystal system	Monoclinic	
Space group	P $2_1/c$	
Density (calculated)	1.298 Mg/m^3	
F(000)	1416	
Data collection program	Bruker APEX2 v2009.7-0	
θ range for data collection	2.08 to 33.50°	
Completeness to θ = 33.50°	99.4 %	
Index ranges	$-23 \le h \le 25$, $-18 \le k \le 18$, $-28 \le l \le 27$	
Data collection scan type	ω scans; 11 settings	
Data reduction program	Bruker SAINT-Plus v7.66A	
Reflections collected	94053	
Independent reflections	13473 [R_{int}= 0.0519]	
Absorption coefficient	0.352 mm^{-1}	
Absorption correction	None	
Max. and min. transmission	0.9458 and 0.9360	

Table 1 (cont.)

Structure solution and Refinement

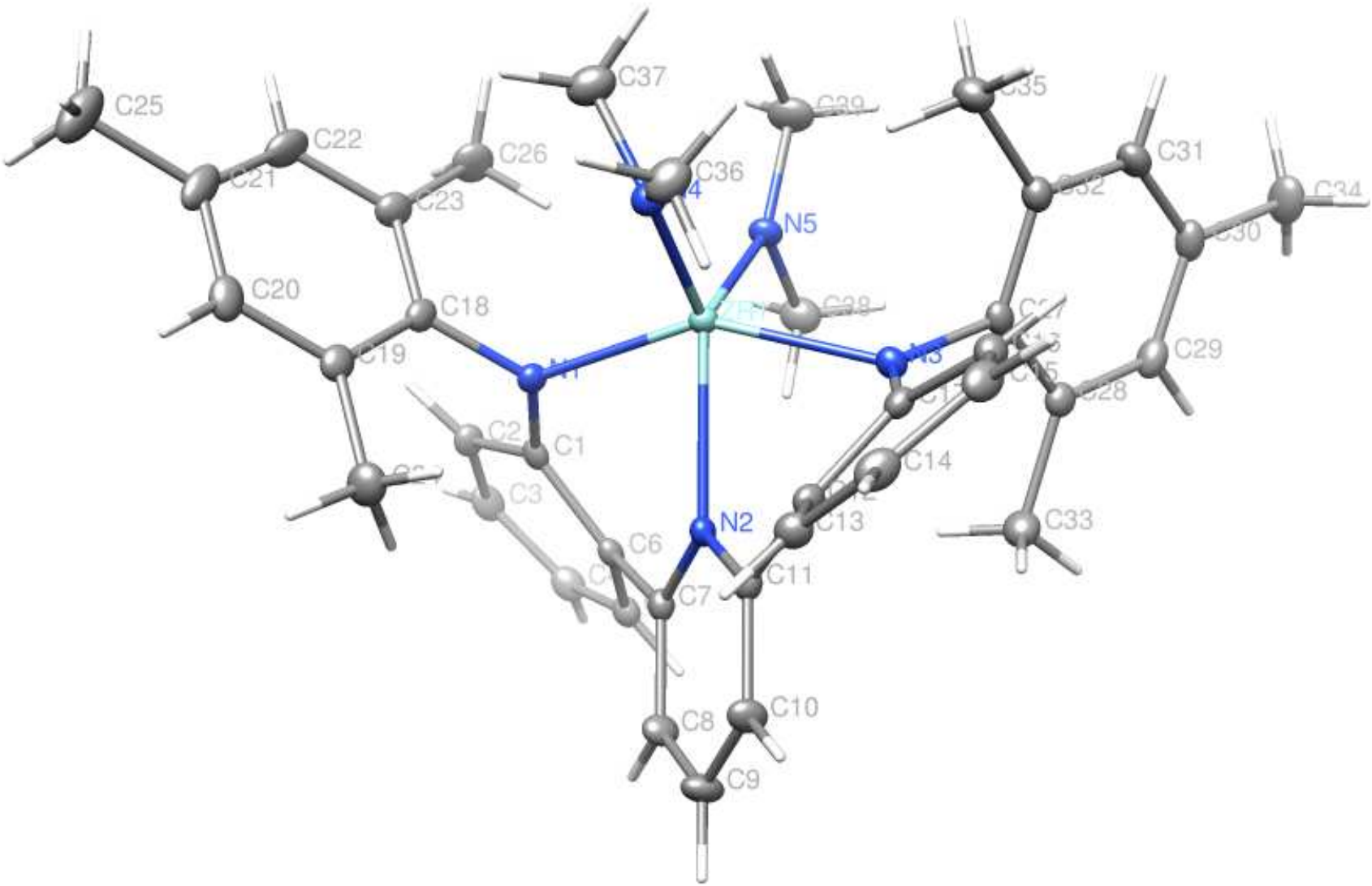
Structure solution program	SHELXS-97 (Sheldrick, 2008)
Primary solution method	Direct methods
Secondary solution method	Difference Fourier map
Hydrogen placement	Geometric positions
Structure refinement program	SHELXL-97 (Sheldrick, 2008)
Refinement method	Full matrix least-squares on F^2
Data / restraints / parameters	13473 / 0 / 416
Treatment of hydrogen atoms	Riding
Goodness-of-fit on F^2	1.774
Final R indices [$I>2\sigma(I)$, 10319 reflections]	R1 = 0.0325, *w*R2 = 0.0505
R indices (all data)	R1 = 0.0491, *w*R2 = 0.0515
Type of weighting scheme used	Sigma
Weighting scheme used	$w=1/\sigma^2(Fo^2)$
Max shift/error	0.002
Average shift/error	0.000
Largest diff. peak and hole	1.201 and -0.984 e.Å^{-3}

Special Refinement Details

Crystals were mounted on a glass fiber using Paratone oil then placed on the diffractometer under a nitrogen stream at 100K.

Refinement of F^2 against ALL reflections. The weighted R-factor (*w*R) and goodness of fit (S) are based on F^2, conventional R-factors (R) are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R-factors based on ALL data will be even larger.

All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.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C23
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C36
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C18
C38
C19
C29
C28
C2
C1
C14
C3
C33
N2
C13
C6
C11
C7
C10
C8
C9

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for IAT48 (CCDC 800978). U(eq) is defined as the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U_{eq}
Zr(1)	2624(1)	3896(1)	7977(1)	11(1)
N(1)	2329(1)	5721(1)	7784(1)	12(1)
N(2)	1192(1)	3963(1)	7678(1)	11(1)
N(3)	2250(1)	2116(1)	7844(1)	12(1)
N(4)	3317(1)	3856(1)	9062(1)	15(1)
N(5)	3334(1)	3725(1)	7230(1)	14(1)
C(1)	1850(1)	6084(1)	7092(1)	12(1)
C(2)	2024(1)	7078(1)	6717(1)	15(1)
C(3)	1603(1)	7316(1)	5982(1)	18(1)
C(4)	972(1)	6608(1)	5584(1)	17(1)
C(5)	728(1)	5709(1)	5965(1)	14(1)
C(6)	1132(1)	5455(1)	6717(1)	12(1)
C(7)	727(1)	4654(1)	7137(1)	12(1)
C(8)	-136(1)	4670(1)	6999(1)	16(1)
C(9)	-524(1)	3984(1)	7422(1)	19(1)
C(10)	-51(1)	3286(1)	7971(1)	16(1)
C(11)	808(1)	3278(1)	8088(1)	12(1)
C(12)	1301(1)	2459(1)	8629(1)	12(1)
C(13)	1072(1)	2236(1)	9308(1)	15(1)
C(14)	1380(1)	1316(1)	9752(1)	18(1)
C(15)	1901(1)	554(1)	9506(1)	18(1)
C(16)	2155(1)	761(1)	8852(1)	15(1)
C(17)	1913(1)	1760(1)	8426(1)	13(1)
C(18)	2771(1)	6571(1)	8292(1)	14(1)
C(19)	2401(1)	6972(1)	8855(1)	15(1)
C(20)	2828(1)	7737(1)	9391(1)	19(1)
C(21)	3620(1)	8120(1)	9396(1)	23(1)
C(22)	3965(1)	7734(1)	8825(1)	24(1)
C(23)	3557(1)	6972(1)	8267(1)	19(1)
C(24)	1556(1)	6542(1)	8887(1)	22(1)
C(25)	4079(1)	8907(1)	10012(1)	34(1)
C(26)	3976(1)	6602(1)	7661(1)	26(1)
C(27)	2558(1)	1283(1)	7410(1)	13(1)
C(28)	2035(1)	960(1)	6710(1)	15(1)
C(29)	2341(1)	235(1)	6238(1)	18(1)
C(30)	3144(1)	-194(1)	6446(1)	18(1)
C(31)	3642(1)	120(1)	7145(1)	19(1)
C(32)	3370(1)	860(1)	7635(1)	15(1)
C(33)	1162(1)	1410(1)	6461(1)	21(1)
C(34)	3466(1)	-988(1)	5931(1)	26(1)
C(35)	3960(1)	1178(1)	8375(1)	21(1)
C(36)	3290(1)	3270(1)	9762(1)	22(1)
C(37)	4116(1)	4430(1)	9152(1)	24(1)
C(38)	2816(1)	3729(1)	6461(1)	22(1)
C(39)	4193(1)	3513(1)	7242(1)	25(1)

Table 3. Selected bond lengths [Å] and angles [°] for IAT48 (CCDC 800978).

Zr(1)-N(5)	2.0258(10)	N(5)-Zr(1)-N(4)	111.63(4)
Zr(1)-N(4)	2.0265(10)	N(5)-Zr(1)-N(3)	91.69(4)
Zr(1)-N(3)	2.1761(11)	N(4)-Zr(1)-N(3)	99.47(4)
Zr(1)-N(1)	2.2074(11)	N(5)-Zr(1)-N(1)	97.67(4)
Zr(1)-N(2)	2.3127(10)	N(4)-Zr(1)-N(1)	103.18(4)
		N(3)-Zr(1)-N(1)	150.17(4)
		N(5)-Zr(1)-N(2)	125.94(4)
		N(4)-Zr(1)-N(2)	122.20(4)
		N(3)-Zr(1)-N(2)	75.87(4)
		N(1)-Zr(1)-N(2)	75.71(4)

Table 4. Bond lengths [Å] and angles [°] for IAT48 (CCDC 800978).

Zr(1)-N(5)	2.0258(10)		
Zr(1)-N(4)	2.0265(10)	N(5)-Zr(1)-N(4)	111.63(4)
Zr(1)-N(3)	2.1761(11)	N(5)-Zr(1)-N(3)	91.69(4)
Zr(1)-N(1)	2.2074(11)	N(4)-Zr(1)-N(3)	99.47(4)
Zr(1)-N(2)	2.3127(10)	N(5)-Zr(1)-N(1)	97.67(4)
N(1)-C(1)	1.3794(16)	N(4)-Zr(1)-N(1)	103.18(4)
N(1)-C(18)	1.4337(16)	N(3)-Zr(1)-N(1)	150.17(4)
N(2)-C(11)	1.3633(15)	N(5)-Zr(1)-N(2)	125.94(4)
N(2)-C(7)	1.3598(16)	N(4)-Zr(1)-N(2)	122.20(4)
N(3)-C(17)	1.3838(15)	N(3)-Zr(1)-N(2)	75.87(4)
N(3)-C(27)	1.4324(15)	N(1)-Zr(1)-N(2)	75.71(4)
N(4)-C(37)	1.4631(16)	C(1)-N(1)-C(18)	117.53(11)
N(4)-C(36)	1.4624(15)	C(1)-N(1)-Zr(1)	120.03(9)
N(5)-C(39)	1.4473(16)	C(18)-N(1)-Zr(1)	120.94(8)
N(5)-C(38)	1.4518(16)	C(11)-N(2)-C(7)	119.30(10)
C(1)-C(2)	1.4186(17)	C(11)-N(2)-Zr(1)	116.72(8)
C(1)-C(6)	1.4257(17)	C(7)-N(2)-Zr(1)	123.96(8)
C(2)-C(3)	1.3774(18)	C(17)-N(3)-C(27)	119.21(10)
C(3)-C(4)	1.3934(19)	C(17)-N(3)-Zr(1)	111.37(8)
C(4)-C(5)	1.3799(17)	C(27)-N(3)-Zr(1)	125.78(8)
C(5)-C(6)	1.4003(17)	C(37)-N(4)-C(36)	110.20(11)
C(6)-C(7)	1.4792(16)	C(37)-N(4)-Zr(1)	112.26(8)
C(7)-C(8)	1.3958(17)	C(36)-N(4)-Zr(1)	136.81(9)
C(8)-C(9)	1.3833(17)	C(39)-N(5)-C(38)	111.18(10)
C(9)-C(10)	1.3783(18)	C(39)-N(5)-Zr(1)	138.50(9)
C(10)-C(11)	1.3932(17)	C(38)-N(5)-Zr(1)	110.10(8)
C(11)-C(12)	1.4721(17)	N(1)-C(1)-C(2)	123.57(12)
C(12)-C(13)	1.4076(16)	N(1)-C(1)-C(6)	120.20(12)
C(12)-C(17)	1.4289(17)	C(2)-C(1)-C(6)	116.23(12)
C(13)-C(14)	1.3710(18)	C(3)-C(2)-C(1)	121.42(13)
C(14)-C(15)	1.3969(18)	C(2)-C(3)-C(4)	121.32(13)
C(15)-C(16)	1.3835(17)	C(5)-C(4)-C(3)	118.21(12)
C(16)-C(17)	1.4091(17)	C(4)-C(5)-C(6)	121.75(13)
C(18)-C(23)	1.4032(18)	C(5)-C(6)-C(1)	120.01(11)
C(18)-C(19)	1.4071(17)	C(5)-C(6)-C(7)	118.24(12)
C(19)-C(20)	1.3859(18)	C(1)-C(6)-C(7)	121.11(11)
C(19)-C(24)	1.5105(18)	N(2)-C(7)-C(8)	120.85(11)
C(20)-C(21)	1.3923(19)	N(2)-C(7)-C(6)	120.25(11)
C(21)-C(22)	1.3856(19)	C(8)-C(7)-C(6)	118.79(12)
C(21)-C(25)	1.5072(19)	C(9)-C(8)-C(7)	119.80(12)
C(22)-C(23)	1.3973(19)	C(8)-C(9)-C(10)	119.21(12)
C(23)-C(26)	1.5120(18)	C(9)-C(10)-C(11)	119.66(12)
C(27)-C(32)	1.4023(17)	N(2)-C(11)-C(10)	121.16(12)
C(27)-C(28)	1.4053(17)	N(2)-C(11)-C(12)	119.57(11)
C(28)-C(29)	1.3935(17)	C(10)-C(11)-C(12)	119.14(11)
C(28)-C(33)	1.5079(18)	C(13)-C(12)-C(17)	119.38(12)
C(29)-C(30)	1.3915(19)	C(13)-C(12)-C(11)	118.81(11)
C(30)-C(31)	1.3853(19)	C(17)-C(12)-C(11)	121.03(11)
C(30)-C(34)	1.5147(17)	C(14)-C(13)-C(12)	121.52(12)
C(31)-C(32)	1.4012(17)	C(13)-C(14)-C(15)	118.99(12)
C(32)-C(35)	1.5050(17)	C(16)-C(15)-C(14)	120.95(12)

C(15)-C(16)-C(17)	121.01(12)
N(3)-C(17)-C(16)	124.05(12)
N(3)-C(17)-C(12)	118.65(11)
C(16)-C(17)-C(12)	117.26(11)
C(23)-C(18)-C(19)	119.71(12)
C(23)-C(18)-N(1)	123.09(11)
C(19)-C(18)-N(1)	117.14(11)
C(20)-C(19)-C(18)	119.40(13)
C(20)-C(19)-C(24)	120.55(12)
C(18)-C(19)-C(24)	120.02(12)
C(19)-C(20)-C(21)	122.11(13)
C(22)-C(21)-C(20)	117.50(13)
C(22)-C(21)-C(25)	122.12(14)
C(20)-C(21)-C(25)	120.38(13)
C(21)-C(22)-C(23)	122.66(13)
C(18)-C(23)-C(22)	118.58(13)
C(18)-C(23)-C(26)	122.21(12)
C(22)-C(23)-C(26)	119.21(13)
C(32)-C(27)-C(28)	120.18(11)
C(32)-C(27)-N(3)	122.27(12)
C(28)-C(27)-N(3)	117.37(11)
C(29)-C(28)-C(27)	119.13(12)
C(29)-C(28)-C(33)	120.15(12)
C(27)-C(28)-C(33)	120.71(11)
C(30)-C(29)-C(28)	121.86(13)
C(31)-C(30)-C(29)	117.95(12)
C(31)-C(30)-C(34)	120.69(13)
C(29)-C(30)-C(34)	121.36(13)
C(30)-C(31)-C(32)	122.39(13)
C(31)-C(32)-C(27)	118.47(12)
C(31)-C(32)-C(35)	118.83(12)
C(27)-C(32)-C(35)	122.70(11)

Table 5. Anisotropic displacement parameters ($Å^2 x\ 10^4$) for IAT48 (CCDC 800978). 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)	97(1)	118(1)	121(1)	-5(1)	33(1)	-4(1)
N(1)	116(5)	120(5)	144(5)	-9(4)	41(4)	-14(4)
N(2)	115(5)	90(5)	131(5)	-9(4)	42(4)	0(5)
N(3)	130(6)	129(6)	122(5)	-1(4)	48(5)	12(5)
N(4)	151(5)	163(6)	142(5)	-7(5)	27(4)	-11(5)
N(5)	110(5)	170(6)	162(5)	-5(5)	54(4)	8(5)
C(1)	134(6)	114(6)	149(6)	-20(6)	76(5)	13(6)
C(2)	155(7)	129(7)	189(7)	-10(5)	76(6)	-24(5)
C(3)	213(8)	146(7)	202(7)	36(6)	118(6)	9(6)
C(4)	202(7)	190(7)	137(7)	21(6)	80(6)	34(6)
C(5)	150(7)	137(7)	155(6)	-19(5)	59(5)	14(5)
C(6)	123(6)	95(6)	153(6)	2(5)	65(5)	13(5)
C(7)	145(7)	106(6)	126(6)	-17(5)	45(5)	1(5)
C(8)	138(7)	150(7)	187(7)	40(5)	35(6)	20(6)
C(9)	106(6)	207(7)	259(7)	37(6)	61(6)	14(6)
C(10)	137(7)	170(7)	200(7)	38(6)	83(6)	-7(6)
C(11)	139(6)	112(6)	128(6)	-15(5)	48(5)	-9(5)
C(12)	117(6)	110(6)	146(6)	8(5)	31(5)	-18(5)
C(13)	141(7)	171(7)	158(7)	-10(5)	53(6)	-12(6)
C(14)	184(7)	208(8)	133(6)	21(5)	41(5)	-51(6)
C(15)	186(7)	149(7)	179(7)	53(6)	-2(6)	-18(6)
C(16)	148(7)	125(7)	170(7)	4(5)	23(6)	0(5)
C(17)	122(6)	118(6)	135(6)	-16(5)	11(5)	-28(5)
C(18)	152(7)	103(6)	156(6)	16(5)	20(5)	-15(5)
C(19)	178(7)	126(7)	151(7)	20(5)	28(6)	-3(6)
C(20)	281(8)	138(7)	155(7)	9(5)	38(6)	16(6)
C(21)	294(9)	137(7)	195(7)	25(6)	-43(7)	-53(6)
C(22)	194(8)	196(8)	298(8)	27(6)	8(7)	-80(6)
C(23)	164(7)	157(7)	246(8)	15(6)	49(6)	-26(6)
C(24)	236(8)	243(8)	213(7)	-58(6)	105(7)	-13(7)
C(25)	429(10)	249(8)	265(8)	-16(8)	-38(7)	-125(9)
C(26)	192(8)	244(8)	359(9)	-22(7)	128(7)	-70(7)
C(27)	149(6)	96(7)	150(6)	9(5)	62(5)	-3(5)
C(28)	164(7)	119(7)	162(6)	10(5)	42(5)	-12(6)
C(29)	234(8)	148(7)	155(7)	-21(5)	44(6)	-38(6)
C(30)	226(8)	126(7)	207(7)	-24(6)	109(6)	-24(6)
C(31)	154(7)	152(7)	265(8)	2(6)	79(6)	22(6)
C(32)	164(7)	124(7)	170(7)	-7(5)	44(6)	-2(5)
C(33)	190(7)	199(8)	218(7)	-23(6)	7(6)	-1(6)
C(34)	312(9)	219(8)	315(8)	-78(7)	178(7)	-25(7)
C(35)	172(7)	219(8)	232(7)	-12(7)	19(6)	38(7)
C(36)	233(8)	257(8)	158(7)	-11(6)	31(6)	-51(7)
C(37)	196(8)	310(9)	197(7)	-3(6)	38(6)	-44(7)
C(38)	203(7)	266(9)	190(7)	-11(6)	61(6)	23(7)
C(39)	160(7)	337(9)	282(8)	-32(7)	90(7)	15(7)

CALIFORNIA INSTITUTE OF TECHNOLOGY

BECKMAN INSTITUTE

X-RAY CRYSTALLOGRAPHY LABORATORY

Date 1 August 2008

Crystal Structure Analysis of:

DYT14

(Complex 4)

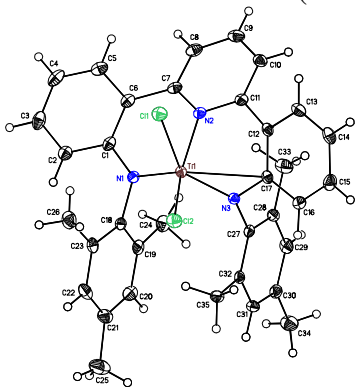
For Investigator: Daniel Tofan ext. 6576

Advisor: J. E. Bercaw ext. 6577

By Michael W. Day 116 Beckman ext. 2734

e-mail: mikeday@caltech.edu

Contents



DTY14

Note: The crystallographic data have been deposited in the Cambridge Database (CCDC). The deposition number is 697201

Table 1. Crystal data and structure refinement for DYT14 (CCDC 697201).

Empirical formula	$C_{35}H_{33}N_3Cl_2Ti \bullet C_4H_8O$
Formula weight	686.55
Crystallization Solvent	THF
Crystal Habit	Blade
Crystal size	0.36 x 0.11 x 0.05 mm^3
Crystal color	Red-brown

Data Collection

Type of diffractometer	Bruker KAPPA APEX II
Wavelength	0.71073 Å MoKα
Data Collection Temperature	100(2) K
θ range for 9047 reflections used in lattice determination	2.52 to 33.08°
Unit cell dimensions	a = 36.2843(15) Å b = 11.6553(5) Å β= 109.665(2)° c = 16.9710(8) Å
Volume	6758.5(5) $Å^3$
Z	8
Crystal system	Monoclinic
Space group	C2/*c*
Density (calculated)	1.349 Mg/m^3
F(000)	2880
Data collection program	Bruker APEX2 v2.1-0
θ range for data collection	1.85 to 33.19°
Completeness to θ = 33.19°	94.4 %
Index ranges	$-55 \le h \le 55$, $-14 \le k \le 17$, $-26 \le l \le 23$
Data collection scan type	ω scans; 17 settings
Data reduction program	Bruker SAINT-Plus v7.34A
Reflections collected	99635
Independent reflections	12205 [R_{int}= 0.0527]
Absorption coefficient	0.448 mm^{-1}
Absorption correction	None
Max. and min. transmission	0.9780 and 0.8555

Table 1 (cont.)

Structure solution and Refinement

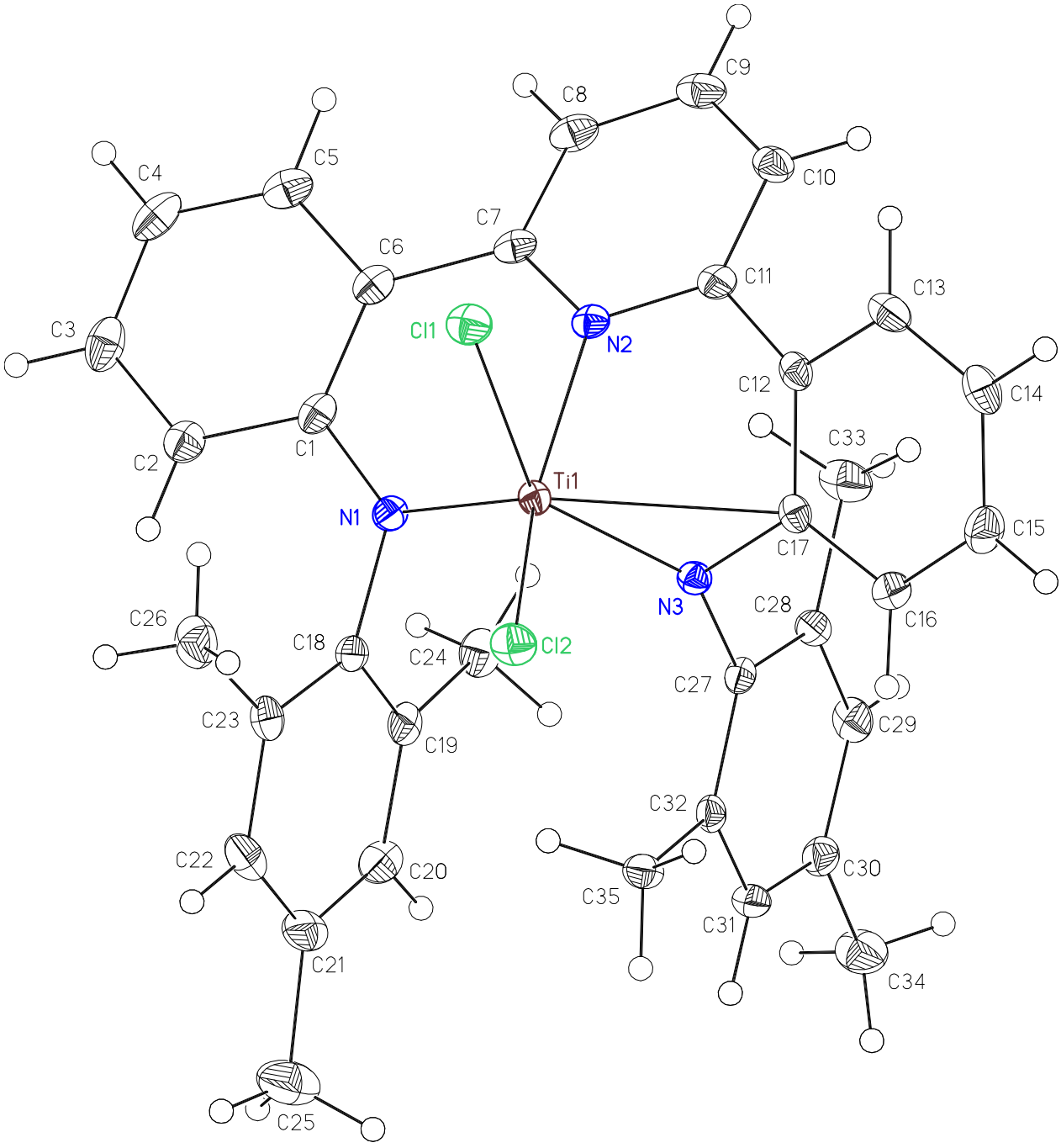
Structure solution program	SHELXS-97 (Sheldrick, 2008)
Primary solution method	Direct methods
Secondary solution method	Difference Fourier map
Hydrogen placement	Difference Fourier map
Structure refinement program	SHELXL-97 (Sheldrick, 2008)
Refinement method	Full matrix least-squares on F^2
Data / restraints / parameters	12205 / 0 / 579
Treatment of hydrogen atoms	Unrestrained
Goodness-of-fit on F^2	1.938
Final R indices [$I>2\sigma(I)$, 8505 reflections]	R1 = 0.0420, *w*R2 = 0.0523
R indices (all data)	R1 = 0.0703, *w*R2 = 0.0533
Type of weighting scheme used	Sigma
Weighting scheme used	$w=1/\sigma^2(Fo^2)$
Max shift/error	0.003
Average shift/error	0.000
Largest diff. peak and hole	1.441 and -0.740 e.Å^{-3}

Special Refinement Details

Crystals were mounted on a glass fiber using Paratone oil then placed on the diffractometer under a nitrogen stream at 100K.

Refinement of F^2 against ALL reflections. The weighted R-factor (*w*R) and goodness of fit (S) are based on F^2, conventional R-factors (R) are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R-factors based on ALL data will be even larger.

All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.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C5
C4
C10
C7
C6
C11
C13
C3
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N2
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C17
C15
N3
C28
C16
C26
C18
C24
Cl2
C27
C23
C19
C29
C32
C22
C20
C30
C35
C31
C21
C34
C25

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for DYT14 (CCDC 697201). U(eq) is defined as the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U_{eq}
Ti(1)	6225(1)	2198(1)	673(1)	12(1)
Cl(1)	6272(1)	200(1)	460(1)	18(1)
Cl(2)	6504(1)	2438(1)	-369(1)	18(1)
N(1)	5682(1)	2522(1)	298(1)	13(1)
N(2)	6141(1)	1517(1)	1765(1)	12(1)
N(3)	6504(1)	3418(1)	1394(1)	12(1)
C(1)	5370(1)	1953(1)	451(1)	14(1)
C(2)	4992(1)	2046(1)	-140(1)	17(1)
C(3)	4681(1)	1441(1)	-51(1)	21(1)
C(4)	4740(1)	734(1)	631(1)	22(1)
C(5)	5104(1)	663(1)	1228(1)	19(1)
C(6)	5429(1)	1274(1)	1176(1)	14(1)
C(7)	5803(1)	1185(1)	1875(1)	14(1)
C(8)	5820(1)	754(1)	2656(1)	18(1)
C(9)	6173(1)	570(1)	3276(1)	19(1)
C(10)	6515(1)	852(1)	3133(1)	16(1)
C(11)	6490(1)	1356(1)	2377(1)	13(1)
C(12)	6846(1)	1779(1)	2228(1)	13(1)
C(13)	7202(1)	1188(1)	2592(1)	18(1)
C(14)	7551(1)	1645(1)	2605(1)	20(1)
C(15)	7560(1)	2721(1)	2250(1)	21(1)
C(16)	7218(1)	3305(1)	1865(1)	17(1)
C(17)	6856(1)	2845(1)	1837(1)	12(1)
C(18)	5594(1)	3514(1)	-247(1)	13(1)
C(19)	5557(1)	4586(1)	103(1)	15(1)
C(20)	5518(1)	5550(1)	-398(1)	18(1)
C(21)	5505(1)	5486(1)	-1220(1)	21(1)
C(22)	5514(1)	4410(1)	-1566(1)	21(1)
C(23)	5555(1)	3413(1)	-1092(1)	16(1)
C(24)	5542(1)	4696(1)	971(1)	19(1)
C(25)	5486(1)	6571(2)	-1721(1)	33(1)
C(26)	5547(1)	2261(1)	-1509(1)	24(1)
C(27)	6476(1)	4617(1)	1596(1)	12(1)
C(28)	6460(1)	4894(1)	2387(1)	14(1)
C(29)	6416(1)	6039(1)	2575(1)	17(1)
C(30)	6384(1)	6909(1)	1999(1)	16(1)
C(31)	6410(1)	6614(1)	1230(1)	15(1)
C(32)	6460(1)	5482(1)	1012(1)	13(1)
C(33)	6473(1)	3995(1)	3029(1)	22(1)
C(34)	6324(1)	8134(1)	2217(1)	25(1)
C(35)	6507(1)	5246(1)	184(1)	16(1)
O(41)	7533(1)	5037(1)	10642(1)	24(1)
C(42)	7506(1)	6046(1)	10150(1)	28(1)
C(43)	7697(1)	5743(2)	9517(1)	52(1)
C(44)	7571(1)	4503(2)	9330(1)	58(1)
C(45)	7555(1)	4052(2)	10144(1)	38(1)

Table 3. Selected bond lengths [Å] and angles [°] for DYT14 (CCDC 697201).

Ti(1)-N(1)	1.8915(10)	N(1)-Ti(1)-N(3)	109.40(4)
Ti(1)-N(3)	1.9263(10)	N(1)-Ti(1)-N(2)	86.02(4)
Ti(1)-N(2)	2.1292(10)	N(3)-Ti(1)-N(2)	85.46(4)
Ti(1)-Cl(2)	2.3323(4)	N(1)-Ti(1)-Cl(2)	112.88(3)
Ti(1)-Cl(1)	2.3717(4)	N(3)-Ti(1)-Cl(2)	97.08(3)
Ti(1)-C(17)	2.5844(12)	N(2)-Ti(1)-Cl(2)	158.47(3)
		N(1)-Ti(1)-Cl(1)	105.39(3)
		N(3)-Ti(1)-Cl(1)	140.50(3)
		N(2)-Ti(1)-Cl(1)	78.80(3)
		Cl(2)-Ti(1)-Cl(1)	85.972(13)
		N(1)-Ti(1)-C(17)	138.95(4)
		N(3)-Ti(1)-C(17)	32.58(4)
		N(2)-Ti(1)-C(17)	78.07(4)
		Cl(2)-Ti(1)-C(17)	92.55(3)
		Cl(1)-Ti(1)-C(17)	108.16(3)

Table 4. Bond lengths [Å] and angles [°] for DYT14 (CCDC 697201).

Ti(1)-N(1)	1.8915(10)	C(23)-C(26)	1.5140(19)
Ti(1)-N(3)	1.9263(10)	C(24)-H(24A)	1.013(13)
Ti(1)-N(2)	2.1292(10)	C(24)-H(24B)	0.945(13)
Ti(1)-Cl(2)	2.3323(4)	C(24)-H(24C)	0.966(14)
Ti(1)-Cl(1)	2.3717(4)	C(25)-H(25A)	0.975(14)
Ti(1)-C(17)	2.5844(12)	C(25)-H(25B)	0.937(15)
N(1)-C(1)	1.4101(15)	C(25)-H(25C)	0.972(15)
N(1)-C(18)	1.4470(15)	C(26)-H(26A)	0.909(19)
N(2)-C(11)	1.3542(15)	C(26)-H(26B)	0.870(19)
N(2)-C(7)	1.3573(15)	C(26)-H(26C)	0.88(2)
N(3)-C(17)	1.4141(15)	C(27)-C(32)	1.4010(16)
N(3)-C(27)	1.4505(14)	C(27)-C(28)	1.4021(17)
C(1)-C(2)	1.4036(17)	C(28)-C(29)	1.3938(17)
C(1)-C(6)	1.4165(18)	C(28)-C(33)	1.5007(18)
C(2)-C(3)	1.3818(18)	C(29)-C(30)	1.3862(18)
C(2)-H(2)	0.947(11)	C(29)-H(29)	0.925(11)
C(3)-C(4)	1.379(2)	C(30)-C(31)	1.3838(18)
C(3)-H(3)	0.931(13)	C(30)-C(34)	1.5093(18)
C(4)-C(5)	1.3702(19)	C(31)-C(32)	1.3979(16)
C(4)-H(4)	0.897(12)	C(31)-H(31)	0.941(11)
C(5)-C(6)	1.4054(17)	C(32)-C(35)	1.4965(18)
C(5)-H(5)	0.969(12)	C(33)-H(33A)	1.007(13)
C(6)-C(7)	1.4766(17)	C(33)-H(33B)	0.946(13)
C(7)-C(8)	1.3997(18)	C(33)-H(33C)	0.964(14)
C(8)-C(9)	1.3753(19)	C(34)-H(34A)	0.972(14)
C(8)-H(8)	0.949(12)	C(34)-H(34B)	0.935(14)
C(9)-C(10)	1.3808(19)	C(34)-H(34C)	0.944(14)
C(9)-H(9)	0.913(12)	C(35)-H(35A)	0.954(13)
C(10)-C(11)	1.3861(18)	C(35)-H(35B)	0.961(13)
C(10)-H(10)	0.920(12)	C(35)-H(35C)	0.994(12)
C(11)-C(12)	1.4805(17)	O(41)-C(42)	1.4264(16)
C(12)-C(13)	1.4082(17)	O(41)-C(45)	1.4426(17)
C(12)-C(17)	1.4157(16)	C(42)-C(43)	1.505(2)
C(13)-C(14)	1.3696(18)	C(42)-H(42A)	0.994(14)
C(13)-H(13)	0.937(12)	C(42)-H(42B)	1.012(12)
C(14)-C(15)	1.3958(18)	C(43)-C(44)	1.516(3)
C(14)-H(14)	0.951(11)	C(43)-H(43A)	1.010(17)
C(15)-C(16)	1.3731(18)	C(43)-H(43B)	0.975(17)
C(15)-H(15)	0.930(12)	C(44)-C(45)	1.498(3)
C(16)-C(17)	1.4023(17)	C(44)-H(44A)	0.984(19)
C(16)-H(16)	0.955(11)	C(44)-H(44B)	0.943(16)
C(18)-C(23)	1.3978(17)	C(45)-H(45A)	1.033(17)
C(18)-C(19)	1.4093(16)	C(45)-H(45B)	0.997(15)
C(19)-C(20)	1.3869(17)		
C(19)-C(24)	1.4990(19)	N(1)-Ti(1)-N(3)	109.40(4)
C(20)-C(21)	1.3816(19)	N(1)-Ti(1)-N(2)	86.02(4)
C(20)-H(20)	0.933(12)	N(3)-Ti(1)-N(2)	85.46(4)
C(21)-C(22)	1.3896(19)	N(1)-Ti(1)-Cl(2)	112.88(3)
C(21)-C(25)	1.511(2)	N(3)-Ti(1)-Cl(2)	97.08(3)
C(22)-C(23)	1.3919(18)	N(2)-Ti(1)-Cl(2)	158.47(3)
C(22)-H(22)	0.932(12)	N(1)-Ti(1)-Cl(1)	105.39(3)

N(3)-Ti(1)-Cl(1)	140.50(3)	C(14)-C(13)-H(13)	120.6(7)
N(2)-Ti(1)-Cl(1)	78.80(3)	C(12)-C(13)-H(13)	117.8(7)
Cl(2)-Ti(1)-Cl(1)	85.972(13)	C(13)-C(14)-C(15)	119.88(13)
N(1)-Ti(1)-C(17)	138.95(4)	C(13)-C(14)-H(14)	121.0(7)
N(3)-Ti(1)-C(17)	32.58(4)	C(15)-C(14)-H(14)	119.1(7)
N(2)-Ti(1)-C(17)	78.07(4)	C(16)-C(15)-C(14)	120.15(13)
Cl(2)-Ti(1)-C(17)	92.55(3)	C(16)-C(15)-H(15)	120.1(8)
Cl(1)-Ti(1)-C(17)	108.16(3)	C(14)-C(15)-H(15)	119.7(8)
C(1)-N(1)-C(18)	118.37(10)	C(15)-C(16)-C(17)	120.79(13)
C(1)-N(1)-Ti(1)	130.80(8)	C(15)-C(16)-H(16)	121.4(7)
C(18)-N(1)-Ti(1)	110.82(7)	C(17)-C(16)-H(16)	117.7(7)
C(11)-N(2)-C(7)	120.70(11)	C(16)-C(17)-N(3)	120.41(11)
C(11)-N(2)-Ti(1)	110.39(8)	C(16)-C(17)-C(12)	119.47(11)
C(7)-N(2)-Ti(1)	128.75(9)	N(3)-C(17)-C(12)	120.08(11)
C(17)-N(3)-C(27)	116.83(10)	C(16)-C(17)-Ti(1)	135.80(9)
C(17)-N(3)-Ti(1)	100.24(7)	N(3)-C(17)-Ti(1)	47.18(5)
C(27)-N(3)-Ti(1)	142.91(8)	C(12)-C(17)-Ti(1)	86.88(7)
C(2)-C(1)-N(1)	118.91(12)	C(23)-C(18)-C(19)	121.00(12)
C(2)-C(1)-C(6)	119.25(12)	C(23)-C(18)-N(1)	120.86(11)
N(1)-C(1)-C(6)	121.83(11)	C(19)-C(18)-N(1)	118.13(11)
C(3)-C(2)-C(1)	121.34(13)	C(20)-C(19)-C(18)	117.66(13)
C(3)-C(2)-H(2)	119.3(7)	C(20)-C(19)-C(24)	120.29(12)
C(1)-C(2)-H(2)	119.3(7)	C(18)-C(19)-C(24)	122.00(12)
C(4)-C(3)-C(2)	119.73(14)	C(21)-C(20)-C(19)	122.54(13)
C(4)-C(3)-H(3)	120.9(8)	C(21)-C(20)-H(20)	120.8(8)
C(2)-C(3)-H(3)	119.3(8)	C(19)-C(20)-H(20)	116.7(8)
C(5)-C(4)-C(3)	119.63(14)	C(20)-C(21)-C(22)	118.43(13)
C(5)-C(4)-H(4)	121.2(9)	C(20)-C(21)-C(25)	120.14(14)
C(3)-C(4)-H(4)	119.2(9)	C(22)-C(21)-C(25)	121.43(14)
C(4)-C(5)-C(6)	122.94(14)	C(21)-C(22)-C(23)	121.51(14)
C(4)-C(5)-H(5)	118.9(7)	C(21)-C(22)-H(22)	119.1(7)
C(6)-C(5)-H(5)	118.1(7)	C(23)-C(22)-H(22)	119.4(7)
C(5)-C(6)-C(1)	116.97(12)	C(22)-C(23)-C(18)	118.51(12)
C(5)-C(6)-C(7)	118.56(12)	C(22)-C(23)-C(26)	119.24(13)
C(1)-C(6)-C(7)	124.47(11)	C(18)-C(23)-C(26)	122.23(12)
N(2)-C(7)-C(8)	118.68(12)	C(19)-C(24)-H(24A)	111.5(8)
N(2)-C(7)-C(6)	119.91(12)	C(19)-C(24)-H(24B)	113.5(8)
C(8)-C(7)-C(6)	121.41(12)	H(24A)-C(24)-H(24B)	106.1(11)
C(9)-C(8)-C(7)	120.77(13)	C(19)-C(24)-H(24C)	110.0(9)
C(9)-C(8)-H(8)	120.0(8)	H(24A)-C(24)-H(24C)	107.3(10)
C(7)-C(8)-H(8)	119.2(8)	H(24B)-C(24)-H(24C)	108.1(11)
C(8)-C(9)-C(10)	119.38(14)	C(21)-C(25)-H(25A)	110.8(8)
C(8)-C(9)-H(9)	120.3(8)	C(21)-C(25)-H(25B)	109.6(9)
C(10)-C(9)-H(9)	120.3(8)	H(25A)-C(25)-H(25B)	106.3(12)
C(9)-C(10)-C(11)	118.76(13)	C(21)-C(25)-H(25C)	111.6(9)
C(9)-C(10)-H(10)	121.4(8)	H(25A)-C(25)-H(25C)	108.4(12)
C(11)-C(10)-H(10)	119.8(8)	H(25B)-C(25)-H(25C)	110.1(12)
N(2)-C(11)-C(10)	121.39(12)	C(23)-C(26)-H(26A)	114.0(11)
N(2)-C(11)-C(12)	117.90(11)	C(23)-C(26)-H(26B)	113.6(13)
C(10)-C(11)-C(12)	120.66(12)	H(26A)-C(26)-H(26B)	101.3(15)
C(13)-C(12)-C(17)	118.01(12)	C(23)-C(26)-H(26C)	112.5(13)
C(13)-C(12)-C(11)	119.02(11)	H(26A)-C(26)-H(26C)	106.2(16)
C(17)-C(12)-C(11)	122.33(11)	H(26B)-C(26)-H(26C)	108.4(16)
C(14)-C(13)-C(12)	121.58(13)	C(32)-C(27)-C(28)	120.43(11)

C(32)-C(27)-N(3)	121.11(11)	O(41)-C(45)-C(44)	106.72(15)
C(28)-C(27)-N(3)	118.45(11)	O(41)-C(45)-H(45A)	106.3(9)
C(29)-C(28)-C(27)	118.97(12)	C(44)-C(45)-H(45A)	112.7(10)
C(29)-C(28)-C(33)	118.76(12)	O(41)-C(45)-H(45B)	104.7(9)
C(27)-C(28)-C(33)	122.23(11)	C(44)-C(45)-H(45B)	113.6(9)
C(30)-C(29)-C(28)	121.89(13)	H(45A)-C(45)-H(45B)	112.0(13)
C(30)-C(29)-H(29)	119.9(7)		
C(28)-C(29)-H(29)	118.1(7)		
C(31)-C(30)-C(29)	117.87(12)		
C(31)-C(30)-C(34)	121.88(12)		
C(29)-C(30)-C(34)	120.25(13)		
C(30)-C(31)-C(32)	122.68(12)		
C(30)-C(31)-H(31)	119.9(7)		
C(32)-C(31)-H(31)	117.5(7)		
C(31)-C(32)-C(27)	118.09(12)		
C(31)-C(32)-C(35)	119.14(12)		
C(27)-C(32)-C(35)	122.74(11)		
C(28)-C(33)-H(33A)	110.3(8)		
C(28)-C(33)-H(33B)	110.6(8)		
H(33A)-C(33)-H(33B)	105.2(11)		
C(28)-C(33)-H(33C)	111.2(8)		
H(33A)-C(33)-H(33C)	110.1(11)		
H(33B)-C(33)-H(33C)	109.3(12)		
C(30)-C(34)-H(34A)	109.4(8)		
C(30)-C(34)-H(34B)	112.1(9)		
H(34A)-C(34)-H(34B)	107.1(12)		
C(30)-C(34)-H(34C)	111.4(8)		
H(34A)-C(34)-H(34C)	107.9(11)		
H(34B)-C(34)-H(34C)	108.7(12)		
C(32)-C(35)-H(35A)	110.5(8)		
C(32)-C(35)-H(35B)	109.2(8)		
H(35A)-C(35)-H(35B)	106.6(10)		
C(32)-C(35)-H(35C)	113.3(7)		
H(35A)-C(35)-H(35C)	110.3(10)		
H(35B)-C(35)-H(35C)	106.6(10)		
C(42)-O(41)-C(45)	108.71(12)		
O(41)-C(42)-C(43)	105.41(14)		
O(41)-C(42)-H(42A)	106.8(8)		
C(43)-C(42)-H(42A)	114.2(8)		
O(41)-C(42)-H(42B)	110.5(7)		
C(43)-C(42)-H(42B)	110.4(7)		
H(42A)-C(42)-H(42B)	109.3(10)		
C(42)-C(43)-C(44)	101.22(16)		
C(42)-C(43)-H(43A)	110.2(10)		
C(44)-C(43)-H(43A)	112.0(10)		
C(42)-C(43)-H(43B)	113.0(10)		
C(44)-C(43)-H(43B)	111.8(10)		
H(43A)-C(43)-H(43B)	108.5(13)		
C(45)-C(44)-C(43)	104.11(15)		
C(45)-C(44)-H(44A)	107.4(11)		
C(43)-C(44)-H(44A)	109.4(11)		
C(45)-C(44)-H(44B)	111.2(11)		
C(43)-C(44)-H(44B)	112.3(10)		
H(44A)-C(44)-H(44B)	112.0(15)		

Table 5. Anisotropic displacement parameters ($Å^2 x\ 10^4$) for DYT14 (CCDC 697201). 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}
Ti(1)	116(1)	121(1)	106(1)	-3(1)	31(1)	2(1)
Cl(1)	241(2)	140(2)	168(2)	-20(1)	85(1)	7(1)
Cl(2)	218(2)	198(2)	156(2)	-1(1)	99(1)	2(1)
N(1)	124(5)	119(6)	138(6)	-1(5)	44(4)	-4(4)
N(2)	159(6)	81(6)	130(6)	-20(4)	58(5)	2(4)
N(3)	121(5)	109(6)	116(6)	3(4)	39(5)	6(4)
C(1)	142(7)	103(7)	185(7)	-49(5)	68(6)	-1(5)
C(2)	162(7)	142(7)	205(8)	-15(6)	63(6)	11(6)
C(3)	133(7)	206(8)	280(9)	-80(7)	53(7)	-1(6)
C(4)	190(8)	188(8)	325(9)	-68(7)	145(7)	-64(6)
C(5)	244(8)	143(8)	222(8)	-27(6)	138(7)	-18(6)
C(6)	154(7)	104(7)	182(8)	-40(6)	86(6)	8(5)
C(7)	194(7)	71(7)	165(8)	-30(5)	93(6)	1(5)
C(8)	224(8)	144(7)	211(8)	-12(6)	137(7)	-21(6)
C(9)	307(9)	140(8)	156(8)	7(6)	115(7)	-9(6)
C(10)	223(8)	118(7)	141(7)	4(6)	42(6)	16(6)
C(11)	190(7)	70(7)	138(7)	-24(5)	53(6)	12(5)
C(12)	152(7)	138(7)	92(7)	-20(5)	27(5)	10(5)
C(13)	229(8)	165(8)	132(8)	22(6)	44(6)	34(6)
C(14)	152(8)	240(8)	172(8)	8(6)	19(6)	63(6)
C(15)	133(7)	240(8)	240(8)	-5(7)	49(6)	-23(6)
C(16)	172(7)	146(8)	184(8)	12(6)	39(6)	-13(6)
C(17)	136(6)	133(7)	95(6)	-24(5)	32(5)	11(5)
C(18)	88(6)	146(7)	155(7)	20(6)	26(5)	-4(5)
C(19)	67(6)	182(7)	187(8)	-3(6)	34(5)	11(5)
C(20)	138(7)	133(8)	280(9)	0(6)	66(6)	32(6)
C(21)	136(7)	210(8)	267(9)	82(7)	51(6)	45(6)
C(22)	169(7)	286(9)	150(8)	47(7)	37(6)	39(6)
C(23)	109(7)	202(8)	159(7)	-1(6)	16(6)	13(5)
C(24)	151(8)	211(9)	224(8)	-30(7)	65(6)	11(6)
C(25)	350(11)	275(10)	368(12)	133(8)	131(9)	75(8)
C(26)	263(9)	240(9)	184(8)	-52(7)	33(7)	-2(7)
C(27)	97(6)	110(7)	139(7)	-18(5)	27(5)	-3(5)
C(28)	148(7)	136(7)	130(7)	7(5)	36(5)	3(5)
C(29)	214(8)	186(8)	117(7)	-45(6)	64(6)	2(6)
C(30)	153(7)	125(7)	182(8)	-12(6)	31(6)	1(5)
C(31)	156(7)	131(7)	165(8)	35(6)	39(6)	2(5)
C(32)	90(6)	158(7)	117(7)	0(5)	19(5)	-11(5)
C(33)	379(10)	181(9)	140(8)	-16(6)	122(7)	20(7)
C(34)	358(10)	159(9)	218(9)	-11(7)	93(8)	22(7)
C(35)	167(7)	149(8)	155(7)	29(6)	61(6)	3(6)
O(41)	337(6)	182(6)	210(6)	-32(4)	86(5)	-15(4)
C(42)	266(9)	300(10)	260(10)	73(8)	73(8)	-2(7)
C(43)	415(13)	889(18)	336(12)	231(12)	219(10)	173(12)
C(44)	433(13)	952(19)	303(12)	-253(11)	56(10)	247(12)
C(45)	336(11)	350(11)	475(13)	-223(9)	171(10)	9(8)

Table 6. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^{2}x 10^3) for DYT14 (CCDC 697201).

	x	y	z	U_{iso}
H(2)	4951(3)	2527(9)	-611(7)	4(3)
H(3)	4436(4)	1498(10)	-465(9)	24(4)
H(4)	4537(4)	329(10)	677(8)	24(4)
H(5)	5140(4)	182(10)	1713(8)	18(4)
H(8)	5583(4)	588(9)	2755(8)	16(4)
H(9)	6182(3)	279(10)	3781(8)	15(4)
H(10)	6756(3)	736(9)	3534(8)	13(3)
H(13)	7193(3)	483(10)	2851(8)	17(4)
H(14)	7790(3)	1240(10)	2852(7)	14(3)
H(15)	7797(4)	3021(10)	2248(8)	17(4)
H(16)	7216(3)	4014(10)	1581(8)	13(3)
H(20)	5510(3)	6257(10)	-148(8)	19(4)
H(22)	5497(3)	4357(10)	-2125(8)	16(4)
H(24A)	5653(4)	5456(11)	1234(8)	29(4)
H(24B)	5686(4)	4123(11)	1342(9)	30(4)
H(24C)	5274(4)	4658(11)	957(9)	38(4)
H(25A)	5273(4)	7064(11)	-1693(9)	38(5)
H(25B)	5429(4)	6386(11)	-2287(10)	36(5)
H(25C)	5729(5)	7002(12)	-1518(10)	48(5)
H(26A)	5339(5)	2164(15)	-1986(12)	83(7)
H(26B)	5518(6)	1684(15)	-1210(13)	82(8)
H(26C)	5758(6)	2140(17)	-1646(13)	102(8)
H(29)	6395(3)	6207(9)	3091(8)	9(3)
H(31)	6396(3)	7188(9)	831(7)	7(3)
H(33A)	6261(4)	3409(11)	2789(9)	29(4)
H(33B)	6418(4)	4321(10)	3488(9)	26(4)
H(33C)	6725(4)	3623(11)	3227(9)	34(4)
H(34A)	6045(4)	8282(11)	2078(9)	34(5)
H(34B)	6418(4)	8661(11)	1916(9)	33(5)
H(34C)	6446(4)	8277(10)	2794(9)	29(4)
H(35A)	6752(4)	4882(10)	260(8)	23(4)
H(35B)	6511(4)	5962(11)	-95(8)	23(4)
H(35C)	6289(4)	4787(9)	-198(8)	16(3)
H(42A)	7640(4)	6669(11)	10542(9)	34(4)
H(42B)	7222(4)	6264(10)	9860(8)	18(4)
H(43A)	7991(5)	5819(14)	9773(11)	67(6)
H(43B)	7608(5)	6219(13)	9016(11)	56(5)
H(44A)	7303(6)	4479(15)	8924(12)	81(7)
H(44B)	7748(5)	4078(13)	9146(11)	62(6)
H(45A)	7307(5)	3571(14)	10074(11)	69(6)
H(45B)	7798(4)	3644(12)	10484(9)	41(5)

CALIFORNIA INSTITUTE OF TECHNOLOGY

BECKMAN INSTITUTE

X-RAY CRYSTALLOGRAPHY LABORATORY

Date 31 July 2008

Crystal Structure Analysis of:

DYT12

(Complex 5)

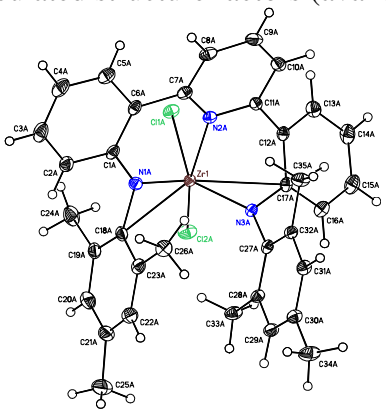
For Investigator: Daniel Tofan ext. 6576

Advisor: J. E. Bercaw ext. 6577

By Michael W. Day 116 Beckman ext. 2734

e-mail: mikeday@caltech.edu

Contents



DYT12

Note: The crystallographic data have been deposited in the Cambridge Database (CCDC). The deposition number is 697086.

Table 1. Crystal data and structure refinement for DYT12 (CCDC 697086).

Empirical formula	$C_{35}H_{33}N_3Cl_2Zr \bullet 0.75(C_7H_8)$
Formula weight	726.86
Crystallization Solvent	Toluene
Crystal Habit	Block
Crystal size	0.26 x 0.24 x 0.21 mm^3
Crystal color	Light yellow

Data Collection

Type of diffractometer	Bruker KAPPA APEX II
Wavelength	0.71073 Å MoKα
Data Collection Temperature	100(2) K
θ range for 9976 reflections used in lattice determination	2.42 to 36.16°
Unit cell dimensions	a = 17.1199(7) Å b = 23.1437(10) Å c = 34.8624(15) Å
Volume	13813.1(10) $Å^3$
Z	16
Crystal system	Orthorhombic
Space group	$P2_12_12_1$
Density (calculated)	1.398 Mg/m^3
F(000)	6008
Data collection program	Bruker APEX2 v2.1-0
θ range for data collection	1.48 to 36.20°
Completeness to θ = 36.20°	93.5 %
Index ranges	$-18 \le h \le 28$, $-37 \le k \le 36$, $-53 \le l \le 51$
Data collection scan type	ω scans; 14 settings
Data reduction program	Bruker SAINT-Plus v7.34A
Reflections collected	360236
Independent reflections	60649 [R_{int}= 0.0519]
Absorption coefficient	0.506 mm^{-1}
Absorption correction	None
Max. and min. transmission	0.9011 and 0.8796

Table 1 (cont.)

Structure solution and Refinement

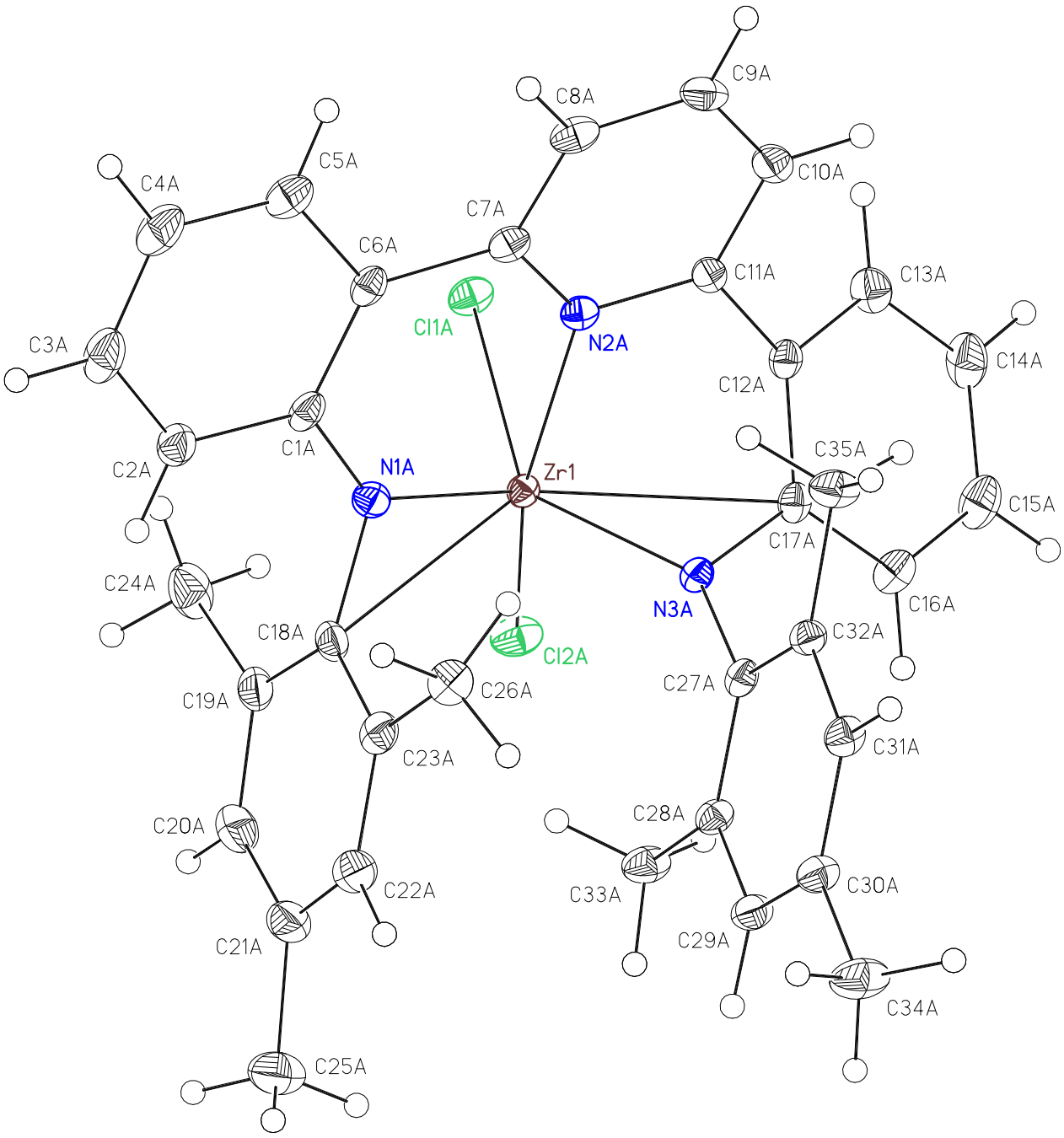
Structure solution program	SHELXS-97 (Sheldrick, 2008)
Primary solution method	Direct methods
Secondary solution method	Difference Fourier map
Hydrogen placement	Geometric positions
Structure refinement program	SHELXL-97 (Sheldrick, 2008)
Refinement method	Full matrix least-squares on F^2
Data / restraints / parameters	60649 / 0 / 1693
Treatment of hydrogen atoms	Riding
Goodness-of-fit on F^2	1.589
Final R indices [$I>2\sigma(I)$, 47805 reflections]	R1 = 0.0345, *w*R2 = 0.0543
R indices (all data)	R1 = 0.0527, *w*R2 = 0.0553
Type of weighting scheme used	Sigma
Weighting scheme used	$w=1/\sigma^2(Fo^2)$
Max shift/error	0.006
Average shift/error	0.000
Absolute structure determination	Possible racemic twin
Absolute structure parameter	0.487(10)
Largest diff. peak and hole	1.013 and -0.749 e.Å^{-3}

Special Refinement Details

Crystals were mounted on a glass fiber using Paratone oil then placed on the diffractometer under a nitrogen stream at 100K.

Refinement of F^2 against ALL reflections. The weighted R-factor (*w*R) and goodness of fit (S) are based on F^2, conventional R-factors (R) are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R-factors based on ALL data will be even larger.

All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.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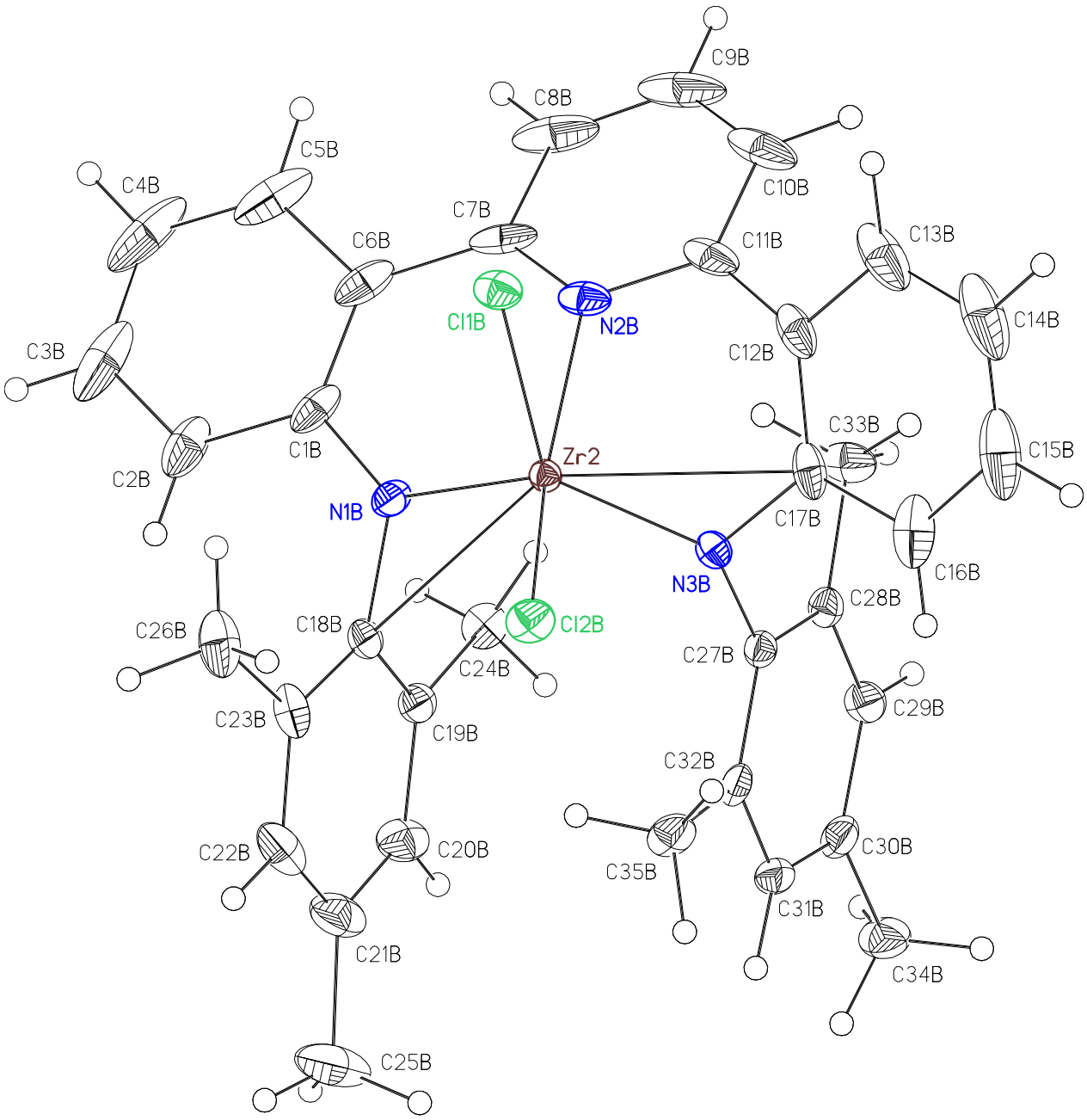
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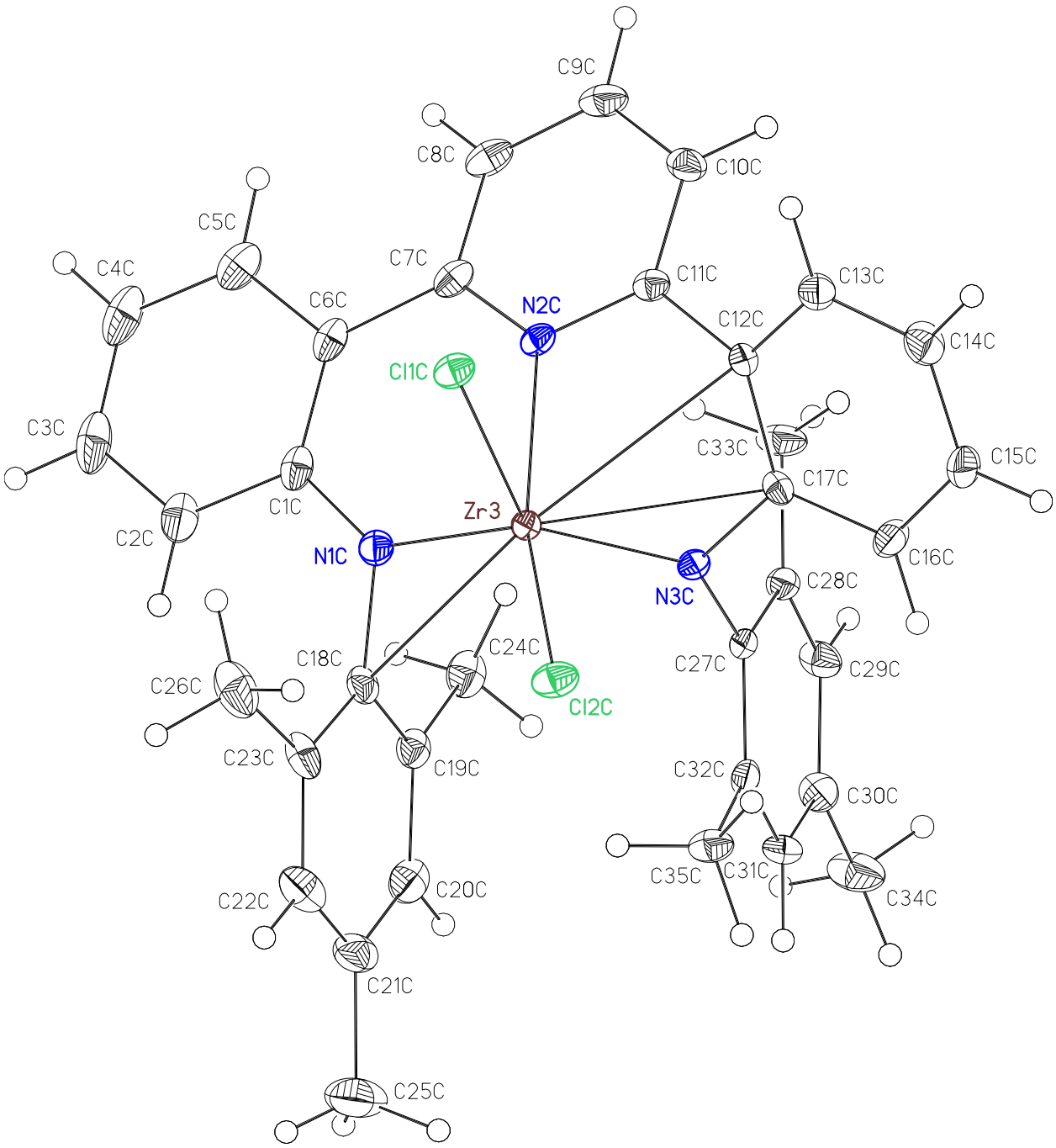
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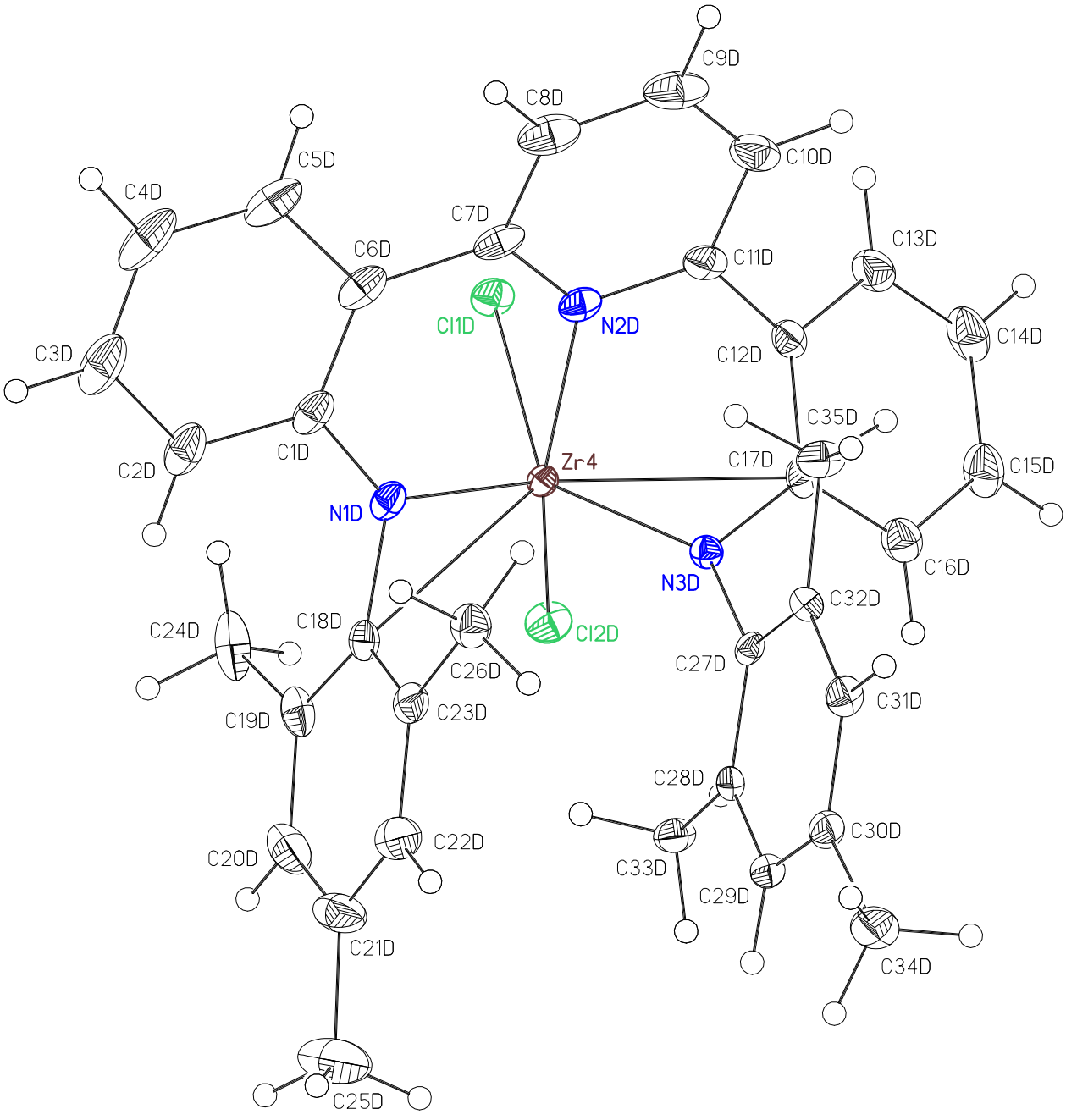
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C21D
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C25D

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for DYT12 (CCDC 697086). U(eq) is defined as the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U_{eq}
Zr(1)	6827(1)	-164(1)	8682(1)	11(1)
Cl(1A)	6575(1)	-1208(1)	8762(1)	16(1)
Cl(2A)	5487(1)	-1(1)	8916(1)	19(1)
N(1A)	7102(1)	-35(1)	8122(1)	14(1)
N(2A)	8059(1)	-517(1)	8663(1)	12(1)
N(3A)	7361(1)	480(1)	8998(1)	12(1)
C(1A)	7614(1)	-266(1)	7852(1)	14(1)
C(2A)	7463(1)	-186(1)	7459(1)	19(1)
C(3A)	7922(1)	-443(1)	7181(1)	24(1)
C(4A)	8541(1)	-790(1)	7289(1)	24(1)
C(5A)	8713(1)	-860(1)	7672(1)	20(1)
C(6A)	8272(1)	-595(1)	7968(1)	14(1)
C(7A)	8534(1)	-669(1)	8368(1)	14(1)
C(8A)	9281(1)	-886(1)	8455(1)	17(1)
C(9A)	9515(1)	-969(1)	8828(1)	18(1)
C(10A)	9014(1)	-824(1)	9123(1)	16(1)
C(11A)	8293(1)	-587(1)	9034(1)	12(1)
C(12A)	7772(1)	-378(1)	9347(1)	13(1)
C(13A)	7730(1)	-712(1)	9686(1)	18(1)
C(14A)	7403(1)	-490(1)	10016(1)	23(1)
C(15A)	7103(1)	73(1)	10018(1)	22(1)
C(16A)	7108(1)	395(1)	9689(1)	18(1)
C(17A)	7429(1)	176(1)	9347(1)	13(1)
C(18A)	6587(1)	432(1)	8022(1)	15(1)
C(19A)	5781(1)	328(1)	7971(1)	18(1)
C(20A)	5280(1)	796(1)	7935(1)	22(1)
C(21A)	5556(1)	1362(1)	7935(1)	23(1)
C(22A)	6363(1)	1452(1)	7955(1)	20(1)
C(23A)	6888(1)	1001(1)	8002(1)	16(1)
C(24A)	5459(1)	-281(1)	7948(1)	26(1)
C(25A)	5003(1)	1866(1)	7923(1)	32(1)
C(26A)	7753(1)	1115(1)	8022(1)	20(1)
C(27A)	7589(1)	1073(1)	8982(1)	13(1)
C(28A)	7037(1)	1516(1)	8933(1)	13(1)
C(29A)	7294(1)	2083(1)	8893(1)	16(1)
C(30A)	8085(1)	2226(1)	8906(1)	16(1)
C(31A)	8622(1)	1782(1)	8958(1)	16(1)
C(32A)	8393(1)	1210(1)	8996(1)	14(1)
C(33A)	6172(1)	1389(1)	8925(1)	19(1)
C(34A)	8354(1)	2842(1)	8867(1)	26(1)
C(35A)	9004(1)	744(1)	9033(1)	19(1)
Zr(2)	1782(1)	2756(1)	8717(1)	11(1)
Cl(1B)	1576(1)	3811(1)	8756(1)	20(1)
Cl(2B)	482(1)	2661(1)	8998(1)	21(1)
N(1B)	1834(1)	2507(1)	8158(1)	16(1)
N(2B)	3000(1)	3080(1)	8570(1)	19(1)

N(3B)	2394(1)	2158(1)	9034(1)	13(1)
C(1B)	2218(1)	2729(1)	7834(1)	22(1)
C(2B)	1904(1)	2604(1)	7470(1)	30(1)
C(3B)	2224(1)	2834(1)	7142(1)	46(1)
C(4B)	2861(2)	3199(1)	7168(1)	49(1)
C(5B)	3184(1)	3313(1)	7518(1)	39(1)
C(6B)	2895(1)	3070(1)	7864(1)	25(1)
C(7B)	3339(1)	3164(1)	8221(1)	25(1)
C(8B)	4122(1)	3340(1)	8212(1)	39(1)
C(9B)	4523(1)	3448(1)	8541(1)	44(1)
C(10B)	4164(1)	3377(1)	8893(1)	36(1)
C(11B)	3392(1)	3178(1)	8900(1)	23(1)
C(12B)	3002(1)	3047(1)	9269(1)	24(1)
C(13B)	3123(1)	3434(1)	9578(1)	38(1)
C(14B)	2924(1)	3289(1)	9945(1)	46(1)
C(15B)	2579(1)	2756(1)	10018(1)	44(1)
C(16B)	2419(1)	2376(1)	9723(1)	32(1)
C(17B)	2618(1)	2521(1)	9343(1)	20(1)
C(18B)	1402(1)	1977(1)	8115(1)	17(1)
C(19B)	1824(1)	1453(1)	8105(1)	17(1)
C(20B)	1407(1)	939(1)	8098(1)	24(1)
C(21B)	592(1)	928(1)	8096(1)	28(1)
C(22B)	196(1)	1446(1)	8082(1)	28(1)
C(23B)	581(1)	1976(1)	8087(1)	23(1)
C(24B)	2702(1)	1445(1)	8095(1)	20(1)
C(25B)	165(1)	356(1)	8114(1)	43(1)
C(26B)	114(1)	2526(1)	8044(1)	32(1)
C(27B)	2605(1)	1557(1)	9053(1)	13(1)
C(28B)	3404(1)	1404(1)	9038(1)	15(1)
C(29B)	3606(1)	826(1)	9039(1)	18(1)
C(30B)	3054(1)	387(1)	9040(1)	19(1)
C(31B)	2269(1)	547(1)	9053(1)	18(1)
C(32B)	2036(1)	1124(1)	9066(1)	16(1)
C(33B)	4033(1)	1853(1)	9007(1)	24(1)
C(34B)	3297(1)	-241(1)	9041(1)	25(1)
C(35B)	1176(1)	1265(1)	9096(1)	22(1)
Zr(3)	8174(1)	2723(1)	6275(1)	10(1)
Cl(1C)	8412(1)	3770(1)	6199(1)	15(1)
Cl(2C)	9481(1)	2574(1)	6002(1)	18(1)
N(1C)	8004(1)	2587(1)	6844(1)	14(1)
N(2C)	6948(1)	3070(1)	6344(1)	12(1)
N(3C)	7596(1)	2077(1)	5978(1)	11(1)
C(1C)	7515(1)	2805(1)	7134(1)	16(1)
C(2C)	7723(1)	2712(1)	7519(1)	21(1)
C(3C)	7293(1)	2941(1)	7817(1)	27(1)
C(4C)	6640(1)	3271(1)	7737(1)	29(1)
C(5C)	6422(1)	3360(1)	7360(1)	23(1)
C(6C)	6835(1)	3125(1)	7047(1)	16(1)
C(7C)	6521(1)	3221(1)	6659(1)	15(1)
C(8C)	5777(1)	3461(1)	6597(1)	20(1)
C(9C)	5498(1)	3559(1)	6231(1)	20(1)
C(10C)	5947(1)	3404(1)	5919(1)	17(1)
C(11C)	6669(1)	3153(1)	5985(1)	13(1)

C(12C)	7140(1)	2946(1)	5654(1)	13(1)
C(13C)	7137(1)	3284(1)	5317(1)	17(1)
C(14C)	7381(1)	3069(1)	4973(1)	21(1)
C(15C)	7636(1)	2494(1)	4951(1)	19(1)
C(16C)	7685(1)	2160(1)	5277(1)	17(1)
C(17C)	7457(1)	2382(1)	5635(1)	12(1)
C(18C)	8519(1)	2112(1)	6931(1)	14(1)
C(19C)	8208(1)	1549(1)	6962(1)	15(1)
C(20C)	8717(1)	1093(1)	7015(1)	21(1)
C(21C)	9523(1)	1174(1)	7041(1)	23(1)
C(22C)	9811(1)	1727(1)	7024(1)	24(1)
C(23C)	9327(1)	2206(1)	6974(1)	19(1)
C(24C)	7338(1)	1446(1)	6955(1)	20(1)
C(25C)	10055(1)	659(1)	7089(1)	36(1)
C(26C)	9671(1)	2811(1)	6982(1)	28(1)
C(27C)	7379(1)	1480(1)	6006(1)	11(1)
C(28C)	6579(1)	1343(1)	6040(1)	13(1)
C(29C)	6366(1)	775(1)	6101(1)	17(1)
C(30C)	6910(1)	334(1)	6135(1)	18(1)
C(31C)	7696(1)	477(1)	6089(1)	17(1)
C(32C)	7943(1)	1042(1)	6022(1)	14(1)
C(33C)	5965(1)	1806(1)	6023(1)	20(1)
C(34C)	6658(1)	-279(1)	6214(1)	27(1)
C(35C)	8804(1)	1163(1)	5964(1)	18(1)
Zr(4)	3217(1)	-178(1)	6323(1)	10(1)
Cl(1D)	3407(1)	-1233(1)	6290(1)	17(1)
Cl(2D)	4467(1)	-86(1)	5989(1)	19(1)
N(1D)	3178(1)	49(1)	6885(1)	15(1)
N(2D)	2002(1)	-503(1)	6468(1)	14(1)
N(3D)	2591(1)	433(1)	6023(1)	11(1)
C(1D)	2790(1)	-172(1)	7206(1)	18(1)
C(2D)	3101(1)	-59(1)	7572(1)	24(1)
C(3D)	2777(1)	-290(1)	7902(1)	30(1)
C(4D)	2128(1)	-645(1)	7869(1)	32(1)
C(5D)	1803(1)	-749(1)	7517(1)	27(1)
C(6D)	2100(1)	-508(1)	7173(1)	19(1)
C(7D)	1662(1)	-606(1)	6815(1)	18(1)
C(8D)	885(1)	-801(1)	6822(1)	26(1)
C(9D)	492(1)	-911(1)	6483(1)	28(1)
C(10D)	858(1)	-820(1)	6135(1)	23(1)
C(11D)	1615(1)	-602(1)	6135(1)	17(1)
C(12D)	2011(1)	-455(1)	5769(1)	16(1)
C(13D)	1930(1)	-834(1)	5453(1)	23(1)
C(14D)	2173(1)	-681(1)	5093(1)	26(1)
C(15D)	2508(1)	-137(1)	5032(1)	25(1)
C(16D)	2612(1)	235(1)	5333(1)	20(1)
C(17D)	2385(1)	83(1)	5707(1)	13(1)
C(18D)	3620(1)	579(1)	6925(1)	16(1)
C(19D)	4441(1)	568(1)	6932(1)	20(1)
C(20D)	4837(1)	1099(1)	6933(1)	26(1)
C(21D)	4443(1)	1620(1)	6941(1)	28(1)
C(22D)	3630(1)	1615(1)	6957(1)	23(1)
C(23D)	3208(1)	1104(1)	6952(1)	16(1)

C(24D)	4895(1)	14(1)	6952(1)	30(1)
C(25D)	4877(1)	2187(1)	6925(1)	43(1)
C(26D)	2331(1)	1118(1)	6981(1)	19(1)
C(27D)	2379(1)	1035(1)	6019(1)	11(1)
C(28D)	2951(1)	1469(1)	5997(1)	12(1)
C(29D)	2711(1)	2043(1)	6017(1)	16(1)
C(30D)	1927(1)	2199(1)	6046(1)	16(1)
C(31D)	1378(1)	1763(1)	6067(1)	16(1)
C(32D)	1586(1)	1183(1)	6058(1)	14(1)
C(33D)	3802(1)	1332(1)	5942(1)	17(1)
C(34D)	1682(1)	2826(1)	6038(1)	23(1)
C(35D)	965(1)	727(1)	6107(1)	19(1)
C(1)	4729(1)	9837(1)	10000(1)	44(1)
C(2)	4946(1)	9203(1)	9964(1)	30(1)
C(3)	4539(1)	8774(1)	10164(1)	33(1)
C(4)	4750(1)	8200(1)	10137(1)	35(1)
C(5)	5371(1)	8038(1)	9908(1)	34(1)
C(6)	5768(1)	8456(1)	9701(1)	30(1)
C(7)	5560(1)	9031(1)	9732(1)	26(1)
C(11)	-149(2)	3216(1)	-30(1)	79(1)
C(12)	109(1)	2599(1)	65(1)	41(1)
C(13)	681(1)	2504(1)	337(1)	48(1)
C(14)	935(1)	1950(1)	424(1)	42(1)
C(15)	621(1)	1483(1)	242(1)	42(1)
C(16)	49(1)	1573(1)	-36(1)	48(1)
C(17)	-196(1)	2132(1)	-119(1)	48(1)
C(21)	5774(1)	-128(1)	5216(1)	31(1)
C(22)	5294(1)	380(1)	5079(1)	26(1)
C(23)	5512(1)	941(1)	5158(1)	30(1)
C(24)	5096(1)	1407(1)	5017(1)	37(1)
C(25)	4441(1)	1313(1)	4790(1)	40(1)
C(26)	4206(1)	761(1)	4712(1)	39(1)
C(27)	4626(1)	292(1)	4853(1)	35(1)

Table 3. Selected bond lengths [Å] and angles [°] for DYT12 (CCDC 697086).

Zr(1)-N(1A)	2.0327(12)	Zr(4)-N(1D)	2.0295(12)
Zr(1)-N(3A)	2.0658(12)	Zr(4)-N(3D)	2.0587(12)
Zr(1)-N(2A)	2.2624(12)	Zr(4)-N(2D)	2.2700(12)
Zr(1)-Cl(2A)	2.4638(4)	Zr(4)-Cl(2D)	2.4439(4)
Zr(1)-Cl(1A)	2.4692(4)	Zr(4)-Cl(1D)	2.4658(4)
Zr(1)-C(17A)	2.6543(14)	Zr(4)-C(17D)	2.6467(14)
Zr(1)-C(18A)	2.7174(15)	Zr(4)-C(18D)	2.8203(15)
Zr(2)-N(1B)	2.0342(13)	Zr(3)-N(1C)	2.0325(13)
Zr(2)-N(3B)	2.0583(12)	Zr(3)-N(3C)	2.0719(12)
Zr(2)-N(2B)	2.2741(13)	Zr(3)-N(2C)	2.2612(12)
Zr(2)-Cl(2B)	2.4414(4)	Zr(3)-Cl(2C)	2.4546(4)
Zr(2)-Cl(1B)	2.4698(4)	Zr(3)-Cl(1C)	2.4706(4)
Zr(2)-C(17B)	2.6646(16)	Zr(3)-C(17C)	2.6652(15)
Zr(2)-C(18B)	2.8437(15)	Zr(3)-C(18C)	2.7537(15)
		Zr(3)-C(12C)	2.8450(15)
N(1A)-Zr(1)-N(3A)	107.67(5)	N(1D)-Zr(4)-N(3D)	107.12(5)
N(1A)-Zr(1)-N(2A)	78.96(5)	N(1D)-Zr(4)-N(2D)	80.74(5)
N(3A)-Zr(1)-N(2A)	82.22(4)	N(3D)-Zr(4)-N(2D)	82.13(5)
N(1A)-Zr(1)-Cl(2A)	120.75(4)	N(1D)-Zr(4)-Cl(2D)	117.78(4)
N(3A)-Zr(1)-Cl(2A)	97.20(3)	N(3D)-Zr(4)-Cl(2D)	98.94(4)
N(2A)-Zr(1)-Cl(2A)	158.90(3)	N(2D)-Zr(4)-Cl(2D)	159.63(3)
N(1A)-Zr(1)-Cl(1A)	106.97(4)	N(1D)-Zr(4)-Cl(1D)	107.74(3)
N(3A)-Zr(1)-Cl(1A)	136.38(4)	N(3D)-Zr(4)-Cl(1D)	136.49(4)
N(2A)-Zr(1)-Cl(1A)	79.23(3)	N(2D)-Zr(4)-Cl(1D)	78.63(3)
Cl(2A)-Zr(1)-Cl(1A)	87.099(13)	Cl(2D)-Zr(4)-Cl(1D)	87.075(14)
N(1A)-Zr(1)-C(17A)	134.89(5)	N(1D)-Zr(4)-C(17D)	134.92(5)
N(3A)-Zr(1)-C(17A)	31.79(5)	N(3D)-Zr(4)-C(17D)	31.95(5)
N(2A)-Zr(1)-C(17A)	76.77(4)	N(2D)-Zr(4)-C(17D)	76.32(5)
Cl(2A)-Zr(1)-C(17A)	91.59(3)	Cl(2D)-Zr(4)-C(17D)	93.76(3)
Cl(1A)-Zr(1)-C(17A)	105.06(3)	Cl(1D)-Zr(4)-C(17D)	105.05(3)
N(1A)-Zr(1)-C(18A)	31.26(5)	N(1D)-Zr(4)-C(18D)	29.38(5)
N(3A)-Zr(1)-C(18A)	98.71(5)	N(3D)-Zr(4)-C(18D)	94.51(5)
N(2A)-Zr(1)-C(18A)	107.43(4)	N(2D)-Zr(4)-C(18D)	105.27(5)
Cl(2A)-Zr(1)-C(18A)	93.53(3)	Cl(2D)-Zr(4)-C(18D)	94.95(3)
Cl(1A)-Zr(1)-C(18A)	124.44(3)	Cl(1D)-Zr(4)-C(18D)	128.08(3)
C(17A)-Zr(1)-C(18A)	130.41(5)	C(17D)-Zr(4)-C(18D)	126.45(5)

N(1B)-Zr(2)-N(3B)	107.58(5)	N(1C)-Zr(3)-N(3C)	107.93(5)
N(1B)-Zr(2)-N(2B)	80.59(5)	N(1C)-Zr(3)-N(2C)	79.42(5)
N(3B)-Zr(2)-N(2B)	82.85(5)	N(3C)-Zr(3)-N(2C)	82.31(4)
N(1B)-Zr(2)-Cl(2B)	113.48(4)	N(1C)-Zr(3)-Cl(2C)	119.18(4)
N(3B)-Zr(2)-Cl(2B)	100.84(4)	N(3C)-Zr(3)-Cl(2C)	98.08(4)
N(2B)-Zr(2)-Cl(2B)	162.99(4)	N(2C)-Zr(3)-Cl(2C)	159.69(3)
N(1B)-Zr(2)-Cl(1B)	109.82(4)	N(1C)-Zr(3)-Cl(1C)	106.27(4)
N(3B)-Zr(2)-Cl(1B)	135.02(4)	N(3C)-Zr(3)-Cl(1C)	137.16(4)
N(2B)-Zr(2)-Cl(1B)	79.53(3)	N(2C)-Zr(3)-Cl(1C)	79.39(3)
Cl(2B)-Zr(2)-Cl(1B)	86.398(14)	Cl(2C)-Zr(3)-Cl(1C)	86.961(13)
N(1B)-Zr(2)-C(17B)	134.64(5)	N(1C)-Zr(3)-C(17C)	134.84(5)
N(3B)-Zr(2)-C(17B)	31.72(5)	N(3C)-Zr(3)-C(17C)	31.49(5)
N(2B)-Zr(2)-C(17B)	76.10(5)	N(2C)-Zr(3)-C(17C)	76.55(4)
Cl(2B)-Zr(2)-C(17B)	98.22(4)	Cl(2C)-Zr(3)-C(17C)	93.12(3)
Cl(1B)-Zr(2)-C(17B)	103.50(4)	Cl(1C)-Zr(3)-C(17C)	106.10(3)
N(1B)-Zr(2)-C(18B)	28.69(5)	N(1C)-Zr(3)-C(18C)	30.57(5)
N(3B)-Zr(2)-C(18B)	94.99(5)	N(3C)-Zr(3)-C(18C)	98.46(5)
N(2B)-Zr(2)-C(18B)	104.57(5)	N(2C)-Zr(3)-C(18C)	106.97(4)
Cl(2B)-Zr(2)-C(18B)	91.73(3)	Cl(2C)-Zr(3)-C(18C)	93.11(3)
Cl(1B)-Zr(2)-C(18B)	129.41(3)	Cl(1C)-Zr(3)-C(18C)	123.85(3)
C(17B)-Zr(2)-C(18B)	126.70(5)	C(17C)-Zr(3)-C(18C)	129.90(5)
		N(1C)-Zr(3)-C(12C)	133.03(5)
		N(3C)-Zr(3)-C(12C)	56.83(4)
		N(2C)-Zr(3)-C(12C)	55.88(4)
		Cl(2C)-Zr(3)-C(12C)	107.37(3)
		Cl(1C)-Zr(3)-C(12C)	80.96(3)
		C(17C)-Zr(3)-C(12C)	29.58(4)
		C(18C)-Zr(3)-C(12C)	149.21(4)

Table 4. Bond lengths [Å] and angles [°] for DYT12 (CCDC 697086).

Zr(1)-N(1A)	2.0327(12)	Zr(2)-N(2B)	2.2741(13)
Zr(1)-N(3A)	2.0658(12)	Zr(2)-Cl(2B)	2.4414(4)
Zr(1)-N(2A)	2.2624(12)	Zr(2)-Cl(1B)	2.4698(4)
Zr(1)-Cl(2A)	2.4638(4)	Zr(2)-C(17B)	2.6646(16)
Zr(1)-Cl(1A)	2.4692(4)	Zr(2)-C(18B)	2.8437(15)
Zr(1)-C(17A)	2.6543(14)	N(1B)-C(1B)	1.404(2)
Zr(1)-C(18A)	2.7174(15)	N(1B)-C(18B)	1.4408(19)
N(1A)-C(1A)	1.3912(19)	N(2B)-C(11B)	1.353(2)
N(1A)-C(18A)	1.4396(19)	N(2B)-C(7B)	1.361(2)
N(2A)-C(7A)	1.3575(19)	N(3B)-C(17B)	1.416(2)
N(2A)-C(11A)	1.3642(18)	N(3B)-C(27B)	1.4400(19)
N(3A)-C(17A)	1.4111(18)	C(1B)-C(6B)	1.406(2)
N(3A)-C(27A)	1.4274(18)	C(1B)-C(2B)	1.407(2)
C(1A)-C(2A)	1.407(2)	C(2B)-C(3B)	1.376(2)
C(1A)-C(6A)	1.418(2)	C(3B)-C(4B)	1.382(3)
C(2A)-C(3A)	1.383(2)	C(4B)-C(5B)	1.365(3)
C(3A)-C(4A)	1.382(2)	C(5B)-C(6B)	1.421(2)
C(4A)-C(5A)	1.377(2)	C(6B)-C(7B)	1.475(3)
C(5A)-C(6A)	1.416(2)	C(7B)-C(8B)	1.402(2)
C(6A)-C(7A)	1.477(2)	C(8B)-C(9B)	1.361(3)
C(7A)-C(8A)	1.406(2)	C(9B)-C(10B)	1.380(3)
C(8A)-C(9A)	1.374(2)	C(10B)-C(11B)	1.400(2)
C(9A)-C(10A)	1.383(2)	C(11B)-C(12B)	1.479(3)
C(10A)-C(11A)	1.385(2)	C(12B)-C(17B)	1.408(2)
C(11A)-C(12A)	1.488(2)	C(12B)-C(13B)	1.417(2)
C(12A)-C(17A)	1.410(2)	C(13B)-C(14B)	1.364(3)
C(12A)-C(13A)	1.414(2)	C(14B)-C(15B)	1.389(3)
C(13A)-C(14A)	1.377(2)	C(15B)-C(16B)	1.383(3)
C(14A)-C(15A)	1.401(2)	C(16B)-C(17B)	1.409(2)
C(15A)-C(16A)	1.367(2)	C(18B)-C(23B)	1.408(2)
C(16A)-C(17A)	1.409(2)	C(18B)-C(19B)	1.413(2)
C(18A)-C(19A)	1.411(2)	C(19B)-C(20B)	1.386(2)
C(18A)-C(23A)	1.415(2)	C(19B)-C(24B)	1.504(2)
C(19A)-C(20A)	1.388(2)	C(20B)-C(21B)	1.396(2)
C(19A)-C(24A)	1.516(2)	C(21B)-C(22B)	1.378(3)
C(20A)-C(21A)	1.391(2)	C(21B)-C(25B)	1.513(2)
C(21A)-C(22A)	1.399(2)	C(22B)-C(23B)	1.393(2)
C(21A)-C(25A)	1.504(2)	C(23B)-C(26B)	1.510(2)
C(22A)-C(23A)	1.387(2)	C(27B)-C(32B)	1.399(2)
C(23A)-C(26A)	1.507(2)	C(27B)-C(28B)	1.414(2)
C(27A)-C(28A)	1.404(2)	C(28B)-C(29B)	1.381(2)
C(27A)-C(32A)	1.414(2)	C(28B)-C(33B)	1.501(2)
C(28A)-C(29A)	1.392(2)	C(29B)-C(30B)	1.387(2)
C(28A)-C(33A)	1.510(2)	C(30B)-C(31B)	1.395(2)
C(29A)-C(30A)	1.395(2)	C(30B)-C(34B)	1.512(2)
C(30A)-C(31A)	1.389(2)	C(31B)-C(32B)	1.395(2)
C(30A)-C(34A)	1.504(2)	C(32B)-C(35B)	1.511(2)
C(31A)-C(32A)	1.386(2)	Zr(3)-N(1C)	2.0325(13)
C(32A)-C(35A)	1.507(2)	Zr(3)-N(3C)	2.0719(12)
Zr(2)-N(1B)	2.0342(13)	Zr(3)-N(2C)	2.2612(12)
Zr(2)-N(3B)	2.0583(12)	Zr(3)-Cl(2C)	2.4546(4)

Zr(3)-Cl(1C)	2.4706(4)	N(1D)-C(18D)	1.4484(19)
Zr(3)-C(17C)	2.6652(15)	N(2D)-C(11D)	1.358(2)
Zr(3)-C(18C)	2.7537(15)	N(2D)-C(7D)	1.3622(19)
Zr(3)-C(12C)	2.8450(15)	N(3D)-C(17D)	1.4130(18)
N(1C)-C(1C)	1.4038(19)	N(3D)-C(27D)	1.4403(18)
N(1C)-C(18C)	1.4410(19)	C(1D)-C(2D)	1.404(2)
N(2C)-C(11C)	1.3532(19)	C(1D)-C(6D)	1.419(2)
N(2C)-C(7C)	1.3630(18)	C(2D)-C(3D)	1.384(2)
N(3C)-C(17C)	1.4065(18)	C(3D)-C(4D)	1.386(3)
N(3C)-C(27C)	1.4339(19)	C(4D)-C(5D)	1.371(2)
C(1C)-C(2C)	1.405(2)	C(5D)-C(6D)	1.416(2)
C(1C)-C(6C)	1.413(2)	C(6D)-C(7D)	1.476(2)
C(2C)-C(3C)	1.380(2)	C(7D)-C(8D)	1.405(2)
C(3C)-C(4C)	1.382(2)	C(8D)-C(9D)	1.383(3)
C(4C)-C(5C)	1.382(2)	C(9D)-C(10D)	1.383(2)
C(5C)-C(6C)	1.410(2)	C(10D)-C(11D)	1.391(2)
C(6C)-C(7C)	1.473(2)	C(11D)-C(12D)	1.483(2)
C(7C)-C(8C)	1.405(2)	C(12D)-C(13D)	1.414(2)
C(8C)-C(9C)	1.382(2)	C(12D)-C(17D)	1.417(2)
C(9C)-C(10C)	1.380(2)	C(13D)-C(14D)	1.368(2)
C(10C)-C(11C)	1.386(2)	C(14D)-C(15D)	1.401(2)
C(11C)-C(12C)	1.488(2)	C(15D)-C(16D)	1.369(2)
C(12C)-C(13C)	1.410(2)	C(16D)-C(17D)	1.405(2)
C(12C)-C(17C)	1.417(2)	C(18D)-C(19D)	1.405(2)
C(13C)-C(14C)	1.364(2)	C(18D)-C(23D)	1.409(2)
C(14C)-C(15C)	1.402(2)	C(19D)-C(20D)	1.404(2)
C(15C)-C(16C)	1.375(2)	C(19D)-C(24D)	1.501(2)
C(16C)-C(17C)	1.406(2)	C(20D)-C(21D)	1.382(3)
C(18C)-C(23C)	1.409(2)	C(21D)-C(22D)	1.393(2)
C(18C)-C(19C)	1.411(2)	C(21D)-C(25D)	1.508(2)
C(19C)-C(20C)	1.382(2)	C(22D)-C(23D)	1.385(2)
C(19C)-C(24C)	1.510(2)	C(23D)-C(26D)	1.506(2)
C(20C)-C(21C)	1.394(2)	C(27D)-C(28D)	1.403(2)
C(21C)-C(22C)	1.373(2)	C(27D)-C(32D)	1.408(2)
C(21C)-C(25C)	1.510(2)	C(28D)-C(29D)	1.393(2)
C(22C)-C(23C)	1.395(2)	C(28D)-C(33D)	1.504(2)
C(23C)-C(26C)	1.519(2)	C(29D)-C(30D)	1.393(2)
C(27C)-C(32C)	1.401(2)	C(30D)-C(31D)	1.381(2)
C(27C)-C(28C)	1.410(2)	C(30D)-C(34D)	1.510(2)
C(28C)-C(29C)	1.381(2)	C(31D)-C(32D)	1.389(2)
C(28C)-C(33C)	1.503(2)	C(32D)-C(35D)	1.508(2)
C(29C)-C(30C)	1.388(2)	C(1)-C(2)	1.519(3)
C(30C)-C(31C)	1.394(2)	C(2)-C(7)	1.387(2)
C(30C)-C(34C)	1.507(2)	C(2)-C(3)	1.397(3)
C(31C)-C(32C)	1.394(2)	C(3)-C(4)	1.381(3)
C(32C)-C(35C)	1.514(2)	C(4)-C(5)	1.382(3)
Zr(4)-N(1D)	2.0295(12)	C(5)-C(6)	1.384(2)
Zr(4)-N(3D)	2.0587(12)	C(6)-C(7)	1.381(3)
Zr(4)-N(2D)	2.2700(12)	C(11)-C(12)	1.531(3)
Zr(4)-Cl(2D)	2.4439(4)	C(12)-C(17)	1.361(3)
Zr(4)-Cl(1D)	2.4658(4)	C(12)-C(13)	1.382(3)
Zr(4)-C(17D)	2.6467(14)	C(13)-C(14)	1.386(3)
Zr(4)-C(18D)	2.8203(15)	C(14)-C(15)	1.364(3)
N(1D)-C(1D)	1.4003(19)	C(15)-C(16)	1.392(3)

C(16)-C(17)	1.390(3)	C(9A)-C(10A)-C(11A)	118.81(15)
C(21)-C(22)	1.512(2)	N(2A)-C(11A)-C(10A)	121.41(14)
C(22)-C(23)	1.379(2)	N(2A)-C(11A)-C(12A)	118.69(13)
C(22)-C(27)	1.403(2)	C(10A)-C(11A)-C(12A)	119.84(13)
C(23)-C(24)	1.384(2)	C(17A)-C(12A)-C(13A)	118.37(14)
C(24)-C(25)	1.388(3)	C(17A)-C(12A)-C(11A)	122.99(13)
C(25)-C(26)	1.368(3)	C(13A)-C(12A)-C(11A)	117.75(13)
C(26)-C(27)	1.391(3)	C(14A)-C(13A)-C(12A)	121.02(15)
		C(13A)-C(14A)-C(15A)	119.97(15)
N(1A)-Zr(1)-N(3A)	107.67(5)	C(16A)-C(15A)-C(14A)	120.07(15)
N(1A)-Zr(1)-N(2A)	78.96(5)	C(15A)-C(16A)-C(17A)	121.04(15)
N(3A)-Zr(1)-N(2A)	82.22(4)	C(16A)-C(17A)-C(12A)	119.36(14)
N(1A)-Zr(1)-Cl(2A)	120.75(4)	C(16A)-C(17A)-N(3A)	121.19(14)
N(3A)-Zr(1)-Cl(2A)	97.20(3)	C(12A)-C(17A)-N(3A)	119.27(13)
N(2A)-Zr(1)-Cl(2A)	158.90(3)	C(16A)-C(17A)-Zr(1)	133.95(10)
N(1A)-Zr(1)-Cl(1A)	106.97(4)	C(12A)-C(17A)-Zr(1)	83.82(9)
N(3A)-Zr(1)-Cl(1A)	136.38(4)	N(3A)-C(17A)-Zr(1)	50.46(7)
N(2A)-Zr(1)-Cl(1A)	79.23(3)	C(19A)-C(18A)-C(23A)	120.59(14)
Cl(2A)-Zr(1)-Cl(1A)	87.099(13)	C(19A)-C(18A)-N(1A)	120.05(13)
N(1A)-Zr(1)-C(17A)	134.89(5)	C(23A)-C(18A)-N(1A)	119.20(14)
N(3A)-Zr(1)-C(17A)	31.79(5)	C(19A)-C(18A)-Zr(1)	99.58(10)
N(2A)-Zr(1)-C(17A)	76.77(4)	C(23A)-C(18A)-Zr(1)	117.25(10)
Cl(2A)-Zr(1)-C(17A)	91.59(3)	N(1A)-C(18A)-Zr(1)	47.11(7)
Cl(1A)-Zr(1)-C(17A)	105.06(3)	C(20A)-C(19A)-C(18A)	118.84(15)
N(1A)-Zr(1)-C(18A)	31.26(5)	C(20A)-C(19A)-C(24A)	119.73(14)
N(3A)-Zr(1)-C(18A)	98.71(5)	C(18A)-C(19A)-C(24A)	121.41(14)
N(2A)-Zr(1)-C(18A)	107.43(4)	C(19A)-C(20A)-C(21A)	121.58(16)
Cl(2A)-Zr(1)-C(18A)	93.53(3)	C(20A)-C(21A)-C(22A)	118.49(16)
Cl(1A)-Zr(1)-C(18A)	124.44(3)	C(20A)-C(21A)-C(25A)	121.03(16)
C(17A)-Zr(1)-C(18A)	130.41(5)	C(22A)-C(21A)-C(25A)	120.46(16)
C(1A)-N(1A)-C(18A)	120.69(12)	C(23A)-C(22A)-C(21A)	122.15(16)
C(1A)-N(1A)-Zr(1)	137.68(10)	C(22A)-C(23A)-C(18A)	118.06(15)
C(18A)-N(1A)-Zr(1)	101.63(9)	C(22A)-C(23A)-C(26A)	120.69(14)
C(7A)-N(2A)-C(11A)	120.79(13)	C(18A)-C(23A)-C(26A)	121.24(14)
C(7A)-N(2A)-Zr(1)	132.42(10)	C(28A)-C(27A)-C(32A)	119.71(14)
C(11A)-N(2A)-Zr(1)	106.75(9)	C(28A)-C(27A)-N(3A)	121.44(13)
C(17A)-N(3A)-C(27A)	119.40(12)	C(32A)-C(27A)-N(3A)	118.76(13)
C(17A)-N(3A)-Zr(1)	97.76(9)	C(29A)-C(28A)-C(27A)	119.14(14)
C(27A)-N(3A)-Zr(1)	142.57(10)	C(29A)-C(28A)-C(33A)	119.44(13)
N(1A)-C(1A)-C(2A)	119.51(14)	C(27A)-C(28A)-C(33A)	121.42(14)
N(1A)-C(1A)-C(6A)	120.93(13)	C(28A)-C(29A)-C(30A)	121.86(14)
C(2A)-C(1A)-C(6A)	119.54(14)	C(31A)-C(30A)-C(29A)	118.11(14)
C(3A)-C(2A)-C(1A)	121.48(15)	C(31A)-C(30A)-C(34A)	120.66(14)
C(4A)-C(3A)-C(2A)	119.57(15)	C(29A)-C(30A)-C(34A)	121.23(13)
C(5A)-C(4A)-C(3A)	119.89(15)	C(32A)-C(31A)-C(30A)	122.08(14)
C(4A)-C(5A)-C(6A)	122.61(16)	C(31A)-C(32A)-C(27A)	119.11(14)
C(5A)-C(6A)-C(1A)	116.75(14)	C(31A)-C(32A)-C(35A)	119.71(14)
C(5A)-C(6A)-C(7A)	118.32(14)	C(27A)-C(32A)-C(35A)	121.13(14)
C(1A)-C(6A)-C(7A)	124.92(13)	N(1B)-Zr(2)-N(3B)	107.58(5)
N(2A)-C(7A)-C(8A)	118.31(14)	N(1B)-Zr(2)-N(2B)	80.59(5)
N(2A)-C(7A)-C(6A)	120.28(13)	N(3B)-Zr(2)-N(2B)	82.85(5)
C(8A)-C(7A)-C(6A)	121.39(14)	N(1B)-Zr(2)-Cl(2B)	113.48(4)
C(9A)-C(8A)-C(7A)	121.26(14)	N(3B)-Zr(2)-Cl(2B)	100.84(4)
C(8A)-C(9A)-C(10A)	119.32(14)	N(2B)-Zr(2)-Cl(2B)	162.99(4)

N(1B)-Zr(2)-Cl(1B)	109.82(4)	C(16B)-C(17B)-Zr(2)	133.54(12)
N(3B)-Zr(2)-Cl(1B)	135.02(4)	N(3B)-C(17B)-Zr(2)	49.82(7)
N(2B)-Zr(2)-Cl(1B)	79.53(3)	C(23B)-C(18B)-C(19B)	120.49(15)
Cl(2B)-Zr(2)-Cl(1B)	86.398(14)	C(23B)-C(18B)-N(1B)	121.37(14)
N(1B)-Zr(2)-C(17B)	134.64(5)	C(19B)-C(18B)-N(1B)	118.14(14)
N(3B)-Zr(2)-C(17B)	31.72(5)	C(23B)-C(18B)-Zr(2)	106.26(11)
N(2B)-Zr(2)-C(17B)	76.10(5)	C(19B)-C(18B)-Zr(2)	116.41(10)
Cl(2B)-Zr(2)-C(17B)	98.22(4)	N(1B)-C(18B)-Zr(2)	42.68(7)
Cl(1B)-Zr(2)-C(17B)	103.50(4)	C(20B)-C(19B)-C(18B)	118.30(16)
N(1B)-Zr(2)-C(18B)	28.69(5)	C(20B)-C(19B)-C(24B)	120.26(15)
N(3B)-Zr(2)-C(18B)	94.99(5)	C(18B)-C(19B)-C(24B)	121.42(14)
N(2B)-Zr(2)-C(18B)	104.57(5)	C(19B)-C(20B)-C(21B)	122.00(16)
Cl(2B)-Zr(2)-C(18B)	91.73(3)	C(22B)-C(21B)-C(20B)	118.39(16)
Cl(1B)-Zr(2)-C(18B)	129.41(3)	C(22B)-C(21B)-C(25B)	121.69(17)
C(17B)-Zr(2)-C(18B)	126.70(5)	C(20B)-C(21B)-C(25B)	119.91(17)
C(1B)-N(1B)-C(18B)	117.80(13)	C(21B)-C(22B)-C(23B)	122.28(17)
C(1B)-N(1B)-Zr(2)	133.50(11)	C(22B)-C(23B)-C(18B)	118.28(16)
C(18B)-N(1B)-Zr(2)	108.63(9)	C(22B)-C(23B)-C(26B)	119.34(16)
C(11B)-N(2B)-C(7B)	121.65(15)	C(18B)-C(23B)-C(26B)	122.32(16)
C(11B)-N(2B)-Zr(2)	108.58(11)	C(32B)-C(27B)-C(28B)	119.72(14)
C(7B)-N(2B)-Zr(2)	129.76(12)	C(32B)-C(27B)-N(3B)	121.24(13)
C(17B)-N(3B)-C(27B)	118.04(13)	C(28B)-C(27B)-N(3B)	118.95(13)
C(17B)-N(3B)-Zr(2)	98.46(9)	C(29B)-C(28B)-C(27B)	119.05(14)
C(27B)-N(3B)-Zr(2)	143.37(10)	C(29B)-C(28B)-C(33B)	119.44(14)
N(1B)-C(1B)-C(6B)	122.07(16)	C(27B)-C(28B)-C(33B)	121.47(14)
N(1B)-C(1B)-C(2B)	118.22(16)	C(28B)-C(29B)-C(30B)	122.51(14)
C(6B)-C(1B)-C(2B)	119.71(16)	C(29B)-C(30B)-C(31B)	117.58(14)
C(3B)-C(2B)-C(1B)	121.3(2)	C(29B)-C(30B)-C(34B)	121.09(14)
C(2B)-C(3B)-C(4B)	119.7(2)	C(31B)-C(30B)-C(34B)	121.30(14)
C(5B)-C(4B)-C(3B)	119.79(19)	C(30B)-C(31B)-C(32B)	122.05(14)
C(4B)-C(5B)-C(6B)	122.8(2)	C(31B)-C(32B)-C(27B)	119.03(14)
C(1B)-C(6B)-C(5B)	116.55(18)	C(31B)-C(32B)-C(35B)	119.17(14)
C(1B)-C(6B)-C(7B)	124.75(15)	C(27B)-C(32B)-C(35B)	121.80(14)
C(5B)-C(6B)-C(7B)	118.62(17)	N(1C)-Zr(3)-N(3C)	107.93(5)
N(2B)-C(7B)-C(8B)	117.97(18)	N(1C)-Zr(3)-N(2C)	79.42(5)
N(2B)-C(7B)-C(6B)	120.90(15)	N(3C)-Zr(3)-N(2C)	82.31(4)
C(8B)-C(7B)-C(6B)	121.13(17)	N(1C)-Zr(3)-Cl(2C)	119.18(4)
C(9B)-C(8B)-C(7B)	121.1(2)	N(3C)-Zr(3)-Cl(2C)	98.08(4)
C(8B)-C(9B)-C(10B)	120.20(18)	N(2C)-Zr(3)-Cl(2C)	159.69(3)
C(9B)-C(10B)-C(11B)	118.4(2)	N(1C)-Zr(3)-Cl(1C)	106.27(4)
N(2B)-C(11B)-C(10B)	120.60(18)	N(3C)-Zr(3)-Cl(1C)	137.16(4)
N(2B)-C(11B)-C(12B)	118.66(15)	N(2C)-Zr(3)-Cl(1C)	79.39(3)
C(10B)-C(11B)-C(12B)	120.70(18)	Cl(2C)-Zr(3)-Cl(1C)	86.961(13)
C(17B)-C(12B)-C(13B)	118.29(18)	N(1C)-Zr(3)-C(17C)	134.84(5)
C(17B)-C(12B)-C(11B)	123.19(15)	N(3C)-Zr(3)-C(17C)	31.49(5)
C(13B)-C(12B)-C(11B)	117.81(17)	N(2C)-Zr(3)-C(17C)	76.55(4)
C(14B)-C(13B)-C(12B)	121.5(2)	Cl(2C)-Zr(3)-C(17C)	93.12(3)
C(13B)-C(14B)-C(15B)	119.73(18)	Cl(1C)-Zr(3)-C(17C)	106.10(3)
C(16B)-C(15B)-C(14B)	120.8(2)	N(1C)-Zr(3)-C(18C)	30.57(5)
C(15B)-C(16B)-C(17B)	120.0(2)	N(3C)-Zr(3)-C(18C)	98.46(5)
C(12B)-C(17B)-C(16B)	119.49(16)	N(2C)-Zr(3)-C(18C)	106.97(4)
C(12B)-C(17B)-N(3B)	119.96(15)	Cl(2C)-Zr(3)-C(18C)	93.11(3)
C(16B)-C(17B)-N(3B)	120.45(17)	Cl(1C)-Zr(3)-C(18C)	123.85(3)
C(12B)-C(17B)-Zr(2)	85.61(10)	C(17C)-Zr(3)-C(18C)	129.90(5)

N(1C)-Zr(3)-C(12C)	133.03(5)	C(23C)-C(18C)-Zr(3)	102.69(10)
N(3C)-Zr(3)-C(12C)	56.83(4)	C(19C)-C(18C)-Zr(3)	117.18(10)
N(2C)-Zr(3)-C(12C)	55.88(4)	N(1C)-C(18C)-Zr(3)	45.85(7)
Cl(2C)-Zr(3)-C(12C)	107.37(3)	C(20C)-C(19C)-C(18C)	118.54(15)
Cl(1C)-Zr(3)-C(12C)	80.96(3)	C(20C)-C(19C)-C(24C)	120.27(14)
C(17C)-Zr(3)-C(12C)	29.58(4)	C(18C)-C(19C)-C(24C)	121.13(14)
C(18C)-Zr(3)-C(12C)	149.21(4)	C(19C)-C(20C)-C(21C)	121.99(16)
C(1C)-N(1C)-C(18C)	119.26(12)	C(22C)-C(21C)-C(20C)	118.56(16)
C(1C)-N(1C)-Zr(3)	137.03(10)	C(22C)-C(21C)-C(25C)	121.58(17)
C(18C)-N(1C)-Zr(3)	103.58(9)	C(20C)-C(21C)-C(25C)	119.86(16)
C(11C)-N(2C)-C(7C)	121.19(13)	C(21C)-C(22C)-C(23C)	122.18(16)
C(11C)-N(2C)-Zr(3)	106.17(9)	C(22C)-C(23C)-C(18C)	118.31(15)
C(7C)-N(2C)-Zr(3)	132.52(10)	C(22C)-C(23C)-C(26C)	120.01(15)
C(17C)-N(3C)-C(27C)	119.83(12)	C(18C)-C(23C)-C(26C)	121.64(15)
C(17C)-N(3C)-Zr(3)	98.21(9)	C(32C)-C(27C)-C(28C)	120.19(14)
C(27C)-N(3C)-Zr(3)	141.76(10)	C(32C)-C(27C)-N(3C)	121.41(13)
N(1C)-C(1C)-C(2C)	118.67(15)	C(28C)-C(27C)-N(3C)	118.29(13)
N(1C)-C(1C)-C(6C)	121.72(13)	C(29C)-C(28C)-C(27C)	118.95(14)
C(2C)-C(1C)-C(6C)	119.60(14)	C(29C)-C(28C)-C(33C)	119.94(14)
C(3C)-C(2C)-C(1C)	121.68(17)	C(27C)-C(28C)-C(33C)	121.09(14)
C(2C)-C(3C)-C(4C)	119.54(16)	C(28C)-C(29C)-C(30C)	122.40(14)
C(3C)-C(4C)-C(5C)	119.45(16)	C(29C)-C(30C)-C(31C)	117.60(14)
C(4C)-C(5C)-C(6C)	122.93(17)	C(29C)-C(30C)-C(34C)	121.03(14)
C(5C)-C(6C)-C(1C)	116.72(15)	C(31C)-C(30C)-C(34C)	121.37(14)
C(5C)-C(6C)-C(7C)	118.04(15)	C(32C)-C(31C)-C(30C)	122.32(14)
C(1C)-C(6C)-C(7C)	125.22(14)	C(31C)-C(32C)-C(27C)	118.46(14)
N(2C)-C(7C)-C(8C)	117.68(14)	C(31C)-C(32C)-C(35C)	119.49(14)
N(2C)-C(7C)-C(6C)	120.27(14)	C(27C)-C(32C)-C(35C)	122.04(14)
C(8C)-C(7C)-C(6C)	122.05(14)	N(1D)-Zr(4)-N(3D)	107.12(5)
C(9C)-C(8C)-C(7C)	121.34(15)	N(1D)-Zr(4)-N(2D)	80.74(5)
C(10C)-C(9C)-C(8C)	119.50(15)	N(3D)-Zr(4)-N(2D)	82.13(5)
C(9C)-C(10C)-C(11C)	118.29(15)	N(1D)-Zr(4)-Cl(2D)	117.78(4)
N(2C)-C(11C)-C(10C)	121.96(14)	N(3D)-Zr(4)-Cl(2D)	98.94(4)
N(2C)-C(11C)-C(12C)	118.84(13)	N(2D)-Zr(4)-Cl(2D)	159.63(3)
C(10C)-C(11C)-C(12C)	119.15(14)	N(1D)-Zr(4)-Cl(1D)	107.74(3)
C(13C)-C(12C)-C(17C)	118.35(14)	N(3D)-Zr(4)-Cl(1D)	136.49(4)
C(13C)-C(12C)-C(11C)	117.84(13)	N(2D)-Zr(4)-Cl(1D)	78.63(3)
C(17C)-C(12C)-C(11C)	122.61(13)	Cl(2D)-Zr(4)-Cl(1D)	87.075(14)
C(13C)-C(12C)-Zr(3)	137.41(10)	N(1D)-Zr(4)-C(17D)	134.92(5)
C(17C)-C(12C)-Zr(3)	68.17(8)	N(3D)-Zr(4)-C(17D)	31.95(5)
C(11C)-C(12C)-Zr(3)	78.69(8)	N(2D)-Zr(4)-C(17D)	76.32(5)
C(14C)-C(13C)-C(12C)	121.87(15)	Cl(2D)-Zr(4)-C(17D)	93.76(3)
C(13C)-C(14C)-C(15C)	119.32(15)	Cl(1D)-Zr(4)-C(17D)	105.05(3)
C(16C)-C(15C)-C(14C)	120.49(15)	N(1D)-Zr(4)-C(18D)	29.38(5)
C(15C)-C(16C)-C(17C)	120.80(15)	N(3D)-Zr(4)-C(18D)	94.51(5)
C(16C)-C(17C)-N(3C)	121.65(14)	N(2D)-Zr(4)-C(18D)	105.27(5)
C(16C)-C(17C)-C(12C)	118.85(14)	Cl(2D)-Zr(4)-C(18D)	94.95(3)
N(3C)-C(17C)-C(12C)	119.28(13)	Cl(1D)-Zr(4)-C(18D)	128.08(3)
C(16C)-C(17C)-Zr(3)	136.34(11)	C(17D)-Zr(4)-C(18D)	126.45(5)
N(3C)-C(17C)-Zr(3)	50.30(7)	C(1D)-N(1D)-C(18D)	118.60(12)
C(12C)-C(17C)-Zr(3)	82.25(9)	C(1D)-N(1D)-Zr(4)	134.05(10)
C(23C)-C(18C)-C(19C)	120.31(15)	C(18D)-N(1D)-Zr(4)	107.19(9)
C(23C)-C(18C)-N(1C)	120.39(14)	C(11D)-N(2D)-C(7D)	121.38(13)
C(19C)-C(18C)-N(1C)	119.28(13)	C(11D)-N(2D)-Zr(4)	108.19(10)

C(7D)-N(2D)-Zr(4)	130.42(11)	C(28D)-C(27D)-N(3D)	121.18(13)
C(17D)-N(3D)-C(27D)	118.99(12)	C(32D)-C(27D)-N(3D)	118.48(13)
C(17D)-N(3D)-Zr(4)	97.61(9)	C(29D)-C(28D)-C(27D)	118.32(14)
C(27D)-N(3D)-Zr(4)	143.14(10)	C(29D)-C(28D)-C(33D)	119.56(13)
N(1D)-C(1D)-C(2D)	118.65(15)	C(27D)-C(28D)-C(33D)	122.09(14)
N(1D)-C(1D)-C(6D)	121.94(14)	C(28D)-C(29D)-C(30D)	122.32(14)
C(2D)-C(1D)-C(6D)	119.41(15)	C(31D)-C(30D)-C(29D)	118.06(14)
C(3D)-C(2D)-C(1D)	121.95(17)	C(31D)-C(30D)-C(34D)	120.84(14)
C(2D)-C(3D)-C(4D)	118.90(17)	C(29D)-C(30D)-C(34D)	121.05(14)
C(5D)-C(4D)-C(3D)	120.17(17)	C(30D)-C(31D)-C(32D)	122.03(15)
C(4D)-C(5D)-C(6D)	122.83(18)	C(31D)-C(32D)-C(27D)	118.97(14)
C(5D)-C(6D)-C(1D)	116.56(16)	C(31D)-C(32D)-C(35D)	119.59(14)
C(5D)-C(6D)-C(7D)	118.18(15)	C(27D)-C(32D)-C(35D)	121.39(14)
C(1D)-C(6D)-C(7D)	125.23(14)	C(7)-C(2)-C(3)	117.69(18)
N(2D)-C(7D)-C(8D)	118.50(16)	C(7)-C(2)-C(1)	120.74(17)
N(2D)-C(7D)-C(6D)	120.44(14)	C(3)-C(2)-C(1)	121.57(18)
C(8D)-C(7D)-C(6D)	121.04(15)	C(4)-C(3)-C(2)	121.33(18)
C(9D)-C(8D)-C(7D)	120.26(16)	C(3)-C(4)-C(5)	120.04(17)
C(8D)-C(9D)-C(10D)	120.16(16)	C(4)-C(5)-C(6)	119.34(18)
C(9D)-C(10D)-C(11D)	118.52(17)	C(7)-C(6)-C(5)	120.38(18)
N(2D)-C(11D)-C(10D)	121.07(16)	C(6)-C(7)-C(2)	121.18(17)
N(2D)-C(11D)-C(12D)	118.25(13)	C(17)-C(12)-C(13)	117.9(2)
C(10D)-C(11D)-C(12D)	120.65(15)	C(17)-C(12)-C(11)	121.9(2)
C(13D)-C(12D)-C(17D)	117.97(15)	C(13)-C(12)-C(11)	120.2(2)
C(13D)-C(12D)-C(11D)	118.79(14)	C(12)-C(13)-C(14)	121.4(2)
C(17D)-C(12D)-C(11D)	122.74(14)	C(15)-C(14)-C(13)	120.4(2)
C(14D)-C(13D)-C(12D)	121.68(16)	C(14)-C(15)-C(16)	118.9(2)
C(13D)-C(14D)-C(15D)	119.75(16)	C(17)-C(16)-C(15)	119.7(2)
C(16D)-C(15D)-C(14D)	120.15(16)	C(12)-C(17)-C(16)	121.7(2)
C(15D)-C(16D)-C(17D)	121.17(16)	C(23)-C(22)-C(27)	118.03(17)
C(16D)-C(17D)-N(3D)	120.84(14)	C(23)-C(22)-C(21)	121.39(16)
C(16D)-C(17D)-C(12D)	119.15(14)	C(27)-C(22)-C(21)	120.53(17)
N(3D)-C(17D)-C(12D)	119.74(13)	C(22)-C(23)-C(24)	121.52(19)
C(16D)-C(17D)-Zr(4)	131.38(10)	C(23)-C(24)-C(25)	119.8(2)
N(3D)-C(17D)-Zr(4)	50.44(7)	C(26)-C(25)-C(24)	119.80(19)
C(12D)-C(17D)-Zr(4)	85.32(9)	C(25)-C(26)-C(27)	120.36(19)
C(19D)-C(18D)-C(23D)	121.02(15)	C(26)-C(27)-C(22)	120.48(19)
C(19D)-C(18D)-N(1D)	120.61(14)		
C(23D)-C(18D)-N(1D)	118.36(14)		
C(19D)-C(18D)-Zr(4)	104.22(10)		
C(23D)-C(18D)-Zr(4)	117.54(10)		
N(1D)-C(18D)-Zr(4)	43.43(6)		
C(20D)-C(19D)-C(18D)	117.86(15)		
C(20D)-C(19D)-C(24D)	119.84(16)		
C(18D)-C(19D)-C(24D)	122.26(15)		
C(21D)-C(20D)-C(19D)	121.86(16)		
C(20D)-C(21D)-C(22D)	118.74(16)		
C(20D)-C(21D)-C(25D)	121.16(17)		
C(22D)-C(21D)-C(25D)	120.10(18)		
C(23D)-C(22D)-C(21D)	121.91(17)		
C(22D)-C(23D)-C(18D)	118.39(15)		
C(22D)-C(23D)-C(26D)	120.15(15)		
C(18D)-C(23D)-C(26D)	121.45(14)		
C(28D)-C(27D)-C(32D)	120.26(14)		

Symmetry transformations used to generate equivalent atoms:

Table 5. Anisotropic displacement parameters ($Å^2 x 10^4$) for DYT12 (CCDC 697086). The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Zr(1)	98(1)	106(1)	112(1)	-9(1)	2(1)	-7(1)
Cl(1A)	147(2)	121(2)	208(2)	-12(1)	26(2)	-25(1)
Cl(2A)	119(2)	169(2)	291(2)	-20(2)	39(2)	2(2)
N(1A)	172(6)	119(6)	135(6)	13(5)	-1(5)	2(5)
N(2A)	120(6)	88(5)	144(6)	10(5)	11(5)	-23(5)
N(3A)	129(6)	105(6)	119(6)	-14(5)	-2(5)	-9(5)
C(1A)	212(8)	99(7)	123(7)	-13(5)	21(6)	-35(6)
C(2A)	268(8)	156(8)	139(7)	2(6)	-10(6)	-4(7)
C(3A)	372(10)	215(9)	130(7)	-5(6)	28(7)	-65(8)
C(4A)	321(10)	229(9)	174(8)	-48(7)	87(7)	-15(7)
C(5A)	222(8)	182(8)	202(8)	-31(6)	60(7)	-2(6)
C(6A)	176(8)	115(6)	134(7)	-16(5)	18(6)	-40(6)
C(7A)	159(8)	91(7)	175(8)	3(6)	18(6)	-29(6)
C(8A)	135(8)	142(8)	226(8)	-17(6)	66(6)	5(6)
C(9A)	153(8)	118(7)	274(9)	9(6)	-28(7)	14(6)
C(10A)	179(8)	114(7)	195(8)	25(6)	-34(7)	-27(6)
C(11A)	134(7)	90(6)	139(7)	16(5)	-20(6)	-32(6)
C(12A)	131(7)	133(7)	125(7)	-7(5)	-7(6)	-28(6)
C(13A)	196(8)	181(8)	171(8)	24(6)	-10(7)	-31(6)
C(14A)	309(10)	236(9)	137(8)	36(7)	1(7)	-77(7)
C(15A)	267(9)	254(9)	148(8)	-35(6)	50(7)	-43(7)
C(16A)	202(8)	173(8)	172(8)	-28(6)	26(6)	-17(6)
C(17A)	124(7)	134(7)	130(7)	-6(6)	-7(5)	-46(6)
C(18A)	171(7)	162(7)	108(7)	12(6)	-24(6)	-1(6)
C(19A)	238(8)	170(8)	139(7)	3(6)	-46(6)	-13(6)
C(20A)	187(8)	259(9)	223(9)	3(7)	-56(7)	6(7)
C(21A)	286(9)	234(9)	164(8)	14(7)	-14(7)	83(7)
C(22A)	274(9)	179(8)	143(8)	30(6)	28(7)	17(7)
C(23A)	229(8)	158(7)	108(7)	8(5)	31(7)	-10(7)
C(24A)	246(9)	252(9)	279(9)	31(7)	-98(7)	-82(7)
C(25A)	361(11)	287(10)	308(11)	5(8)	-12(9)	141(8)
C(26A)	200(8)	190(8)	194(8)	7(7)	25(7)	-21(7)
C(27A)	146(7)	116(7)	118(7)	-15(5)	4(6)	-26(6)
C(28A)	112(7)	132(7)	154(7)	-25(6)	19(6)	3(6)
C(29A)	159(8)	128(7)	208(8)	-25(6)	-11(6)	12(6)
C(30A)	152(7)	136(7)	200(8)	-15(6)	7(6)	0(6)
C(31A)	137(7)	141(8)	197(8)	-14(6)	-2(6)	-35(6)
C(32A)	132(7)	134(7)	154(7)	-19(6)	-6(6)	11(6)
C(33A)	133(7)	157(8)	276(9)	-27(7)	10(7)	17(6)
C(34A)	195(8)	131(7)	441(11)	-10(7)	-6(8)	-23(6)
C(35A)	143(8)	125(8)	311(9)	16(7)	-15(7)	-21(6)
Zr(2)	102(1)	118(1)	111(1)	0(1)	-1(1)	-2(1)
Cl(1B)	167(2)	130(2)	289(2)	-12(2)	-8(2)	16(1)
Cl(2B)	131(2)	242(2)	247(2)	26(2)	50(2)	1(2)
N(1B)	221(7)	127(6)	136(6)	11(5)	12(6)	-11(6)
N(2B)	148(7)	100(6)	328(8)	-8(6)	14(6)	4(5)

N(3B)	115(6)	144(6)	141(6)	-16(5)	-5(5)	8(5)
C(1B)	383(10)	116(8)	172(8)	33(6)	93(7)	85(7)
C(2B)	551(13)	207(8)	155(8)	13(6)	38(9)	117(9)
C(3B)	883(18)	308(11)	192(9)	78(8)	124(11)	281(12)
C(4B)	859(18)	294(11)	327(12)	164(9)	375(12)	254(12)
C(5B)	513(13)	206(9)	444(12)	132(8)	316(11)	125(9)
C(6B)	355(10)	130(8)	266(9)	54(7)	153(8)	74(7)
C(7B)	199(9)	83(7)	457(12)	22(7)	135(8)	-3(6)
C(8B)	243(10)	192(9)	745(17)	70(10)	251(11)	-18(8)
C(9B)	127(9)	193(10)	1000(20)	68(11)	71(11)	-41(8)
C(10B)	193(9)	118(8)	779(17)	-69(9)	-171(10)	6(7)
C(11B)	151(8)	95(7)	446(11)	-58(7)	-73(8)	17(6)
C(12B)	214(9)	204(9)	301(10)	-81(7)	-146(8)	90(7)
C(13B)	375(11)	259(10)	503(13)	-209(9)	-254(10)	143(9)
C(14B)	508(13)	474(13)	404(13)	-290(11)	-258(11)	262(11)
C(15B)	446(12)	682(17)	183(10)	-145(10)	-88(9)	280(12)
C(16B)	332(10)	457(12)	186(9)	-22(9)	-30(8)	169(9)
C(17B)	194(8)	253(9)	160(8)	-66(7)	-62(7)	112(7)
C(18B)	224(9)	163(8)	116(7)	-28(6)	-27(6)	-21(6)
C(19B)	223(8)	154(7)	127(7)	-16(6)	-2(7)	-10(7)
C(20B)	316(10)	171(8)	227(9)	-59(7)	41(8)	-25(7)
C(21B)	326(10)	275(10)	252(10)	-94(8)	30(8)	-106(8)
C(22B)	212(9)	383(11)	252(10)	-81(8)	-35(8)	-64(8)
C(23B)	214(9)	298(10)	167(8)	-37(7)	-59(7)	30(8)
C(24B)	201(8)	196(8)	206(9)	-7(7)	27(7)	31(7)
C(25B)	466(13)	393(12)	424(13)	-132(10)	126(10)	-260(10)
C(26B)	295(10)	398(11)	260(10)	-25(8)	-95(8)	111(9)
C(27B)	136(7)	152(8)	113(7)	11(6)	-2(6)	-1(6)
C(28B)	139(7)	160(8)	159(7)	13(6)	-19(6)	21(6)
C(29B)	127(7)	193(8)	210(8)	8(7)	-4(6)	9(6)
C(30B)	243(9)	158(7)	176(8)	45(6)	-23(7)	19(6)
C(31B)	158(8)	196(8)	199(8)	57(6)	-10(6)	-43(6)
C(32B)	149(7)	202(8)	124(7)	59(6)	-7(6)	17(6)
C(33B)	109(8)	179(8)	418(11)	14(7)	4(7)	16(6)
C(34B)	240(9)	156(8)	360(9)	35(7)	-32(8)	4(7)
C(35B)	129(8)	265(9)	277(9)	92(7)	38(7)	-15(7)
Zr(3)	100(1)	97(1)	99(1)	-2(1)	-2(1)	0(1)
Cl(1C)	154(2)	110(2)	194(2)	-2(1)	21(2)	-10(1)
Cl(2C)	122(2)	168(2)	255(2)	-11(2)	38(2)	12(2)
N(1C)	188(7)	106(6)	135(6)	7(5)	-15(5)	-2(5)
N(2C)	114(6)	97(5)	153(6)	-23(5)	13(5)	-9(5)
N(3C)	117(6)	91(6)	119(6)	0(5)	3(5)	1(5)
C(1C)	231(8)	117(7)	119(7)	-3(6)	14(6)	-61(6)
C(2C)	311(9)	175(8)	154(8)	-4(6)	-4(7)	-50(7)
C(3C)	429(11)	251(9)	120(8)	-18(7)	25(8)	-118(8)
C(4C)	397(11)	285(10)	178(8)	-83(7)	97(8)	-59(8)
C(5C)	257(9)	216(9)	220(9)	-49(7)	70(7)	-11(7)
C(6C)	208(8)	124(7)	132(7)	-20(5)	48(7)	-51(6)
C(7C)	165(8)	90(7)	181(8)	-12(6)	48(6)	-34(6)
C(8C)	180(8)	164(8)	239(9)	-25(7)	88(7)	18(6)
C(9C)	130(7)	162(7)	309(10)	-16(7)	9(7)	35(6)
C(10C)	168(8)	119(7)	217(8)	13(6)	-50(7)	8(6)
C(11C)	117(7)	102(7)	168(7)	-1(5)	4(6)	-7(6)

C(12C)	128(7)	132(7)	121(7)	-16(6)	-33(6)	8(6)
C(13C)	200(8)	135(7)	161(8)	9(6)	-20(6)	5(6)
C(14C)	276(9)	194(8)	146(8)	45(6)	-9(7)	-3(7)
C(15C)	262(9)	214(8)	102(7)	-21(6)	3(6)	35(7)
C(16C)	223(8)	136(7)	152(8)	-32(6)	15(6)	12(6)
C(17C)	120(7)	124(7)	118(7)	13(6)	-24(6)	-16(6)
C(18C)	178(8)	159(7)	93(7)	21(6)	-15(6)	-3(6)
C(19C)	193(8)	157(7)	109(7)	10(5)	10(6)	-9(7)
C(20C)	320(10)	159(8)	140(8)	25(6)	27(7)	-2(7)
C(21C)	254(9)	258(9)	164(8)	47(7)	7(7)	78(8)
C(22C)	197(9)	322(10)	205(9)	61(7)	-21(7)	20(7)
C(23C)	207(8)	222(9)	142(7)	42(6)	-54(6)	-46(7)
C(24C)	255(9)	194(8)	150(8)	10(6)	27(7)	-39(7)
C(25C)	387(11)	360(11)	327(11)	92(9)	34(9)	192(9)
C(26C)	295(10)	275(10)	276(10)	49(8)	-114(8)	-112(8)
C(27C)	136(7)	97(7)	107(7)	-19(5)	-20(6)	10(6)
C(28C)	131(8)	109(7)	162(8)	-15(5)	-24(6)	0(6)
C(29C)	120(7)	153(8)	245(9)	16(6)	-41(6)	-23(6)
C(30C)	202(8)	117(7)	210(8)	14(6)	-40(7)	-33(6)
C(31C)	182(8)	131(7)	186(8)	15(6)	-31(6)	50(6)
C(32C)	169(8)	130(7)	111(7)	-29(5)	-12(6)	-5(6)
C(33C)	111(8)	150(8)	352(10)	10(7)	-29(7)	21(6)
C(34C)	206(8)	148(7)	456(11)	56(7)	-78(8)	-24(6)
C(35C)	132(8)	160(8)	245(9)	-4(7)	17(7)	29(6)
Zr(4)	107(1)	99(1)	101(1)	4(1)	4(1)	7(1)
Cl(1D)	169(2)	113(2)	225(2)	0(2)	0(2)	27(1)
Cl(2D)	138(2)	206(2)	214(2)	12(2)	53(2)	6(2)
N(1D)	203(7)	128(6)	108(5)	11(4)	16(5)	17(6)
N(2D)	141(6)	91(6)	203(7)	17(5)	24(5)	26(5)
N(3D)	119(6)	112(6)	110(6)	-8(5)	0(5)	2(5)
C(1D)	271(9)	115(7)	152(7)	14(6)	31(6)	60(6)
C(2D)	392(10)	187(8)	134(7)	5(6)	14(7)	67(8)
C(3D)	515(12)	234(9)	152(8)	18(7)	57(8)	110(9)
C(4D)	521(13)	251(10)	200(9)	89(7)	178(9)	134(9)
C(5D)	367(10)	161(8)	278(9)	68(7)	128(9)	65(8)
C(6D)	257(9)	124(7)	194(8)	26(6)	83(7)	68(7)
C(7D)	200(8)	96(7)	249(8)	33(6)	82(7)	29(6)
C(8D)	211(9)	203(9)	380(11)	41(8)	127(8)	-2(7)
C(9D)	165(9)	191(9)	495(12)	38(8)	78(9)	-19(7)
C(10D)	201(8)	131(8)	355(10)	-7(7)	-51(8)	-13(6)
C(11D)	145(8)	87(7)	270(9)	6(6)	-22(7)	25(6)
C(12D)	157(8)	116(7)	193(8)	-10(6)	-59(6)	26(6)
C(13D)	269(9)	139(7)	283(9)	-25(7)	-94(8)	-1(7)
C(14D)	325(10)	227(9)	225(9)	-102(7)	-86(8)	55(8)
C(15D)	310(9)	277(10)	151(8)	-50(7)	-25(7)	29(8)
C(16D)	205(8)	196(8)	184(8)	-26(7)	-16(6)	11(7)
C(17D)	119(7)	138(7)	147(7)	-11(6)	-33(6)	47(6)
C(18D)	201(8)	193(8)	75(7)	-5(6)	-9(6)	-18(7)
C(19D)	199(8)	280(9)	127(7)	-24(7)	-33(7)	15(7)
C(20D)	199(9)	408(11)	174(9)	-44(8)	1(7)	-84(8)
C(21D)	353(11)	297(10)	183(9)	-60(7)	53(8)	-164(8)
C(22D)	312(10)	205(9)	166(8)	-26(7)	33(7)	-41(7)
C(23D)	220(8)	171(7)	101(7)	-9(5)	5(7)	-4(7)

C(24D)	262(9)	468(12)	182(9)	-34(8)	-76(7)	124(9)
C(25D)	509(13)	401(12)	386(12)	-117(10)	136(10)	-238(11)
C(26D)	207(9)	195(8)	174(8)	-16(6)	-4(7)	17(7)
C(27D)	164(8)	81(7)	86(7)	1(5)	-5(6)	18(6)
C(28D)	133(7)	146(7)	96(7)	12(5)	-22(6)	-17(6)
C(29D)	191(8)	124(7)	149(8)	13(6)	-29(6)	-21(6)
C(30D)	198(8)	121(7)	160(7)	3(6)	-34(6)	12(6)
C(31D)	163(8)	136(8)	178(8)	-10(6)	-16(6)	42(6)
C(32D)	125(7)	142(7)	139(7)	0(6)	-18(6)	-13(6)
C(33D)	154(8)	147(8)	212(8)	28(6)	4(7)	-21(6)
C(34D)	245(9)	126(7)	329(10)	-11(6)	-56(8)	27(7)
C(35D)	133(8)	144(8)	306(9)	25(7)	11(7)	15(6)
C(1)	411(12)	441(13)	475(13)	-86(11)	28(10)	15(10)
C(2)	230(9)	437(12)	218(9)	-31(8)	-38(8)	-61(8)
C(3)	255(9)	546(13)	201(9)	-20(9)	4(8)	-113(9)
C(4)	285(10)	504(13)	258(10)	158(9)	-56(8)	-169(9)
C(5)	267(10)	319(11)	431(12)	144(9)	-78(9)	-37(8)
C(6)	181(9)	391(11)	330(11)	134(9)	-18(8)	-2(8)
C(7)	210(8)	334(10)	249(9)	86(8)	-26(7)	-48(8)
C(11)	1130(20)	810(20)	441(16)	182(14)	115(16)	616(18)
C(12)	493(13)	531(14)	215(10)	45(9)	60(9)	172(11)
C(13)	738(16)	416(13)	299(11)	-64(9)	-61(11)	54(12)
C(14)	437(12)	461(13)	372(12)	52(10)	-89(10)	5(10)
C(15)	408(12)	410(13)	429(13)	66(10)	150(10)	-27(10)
C(16)	511(14)	610(16)	308(12)	-36(11)	99(11)	-232(12)
C(17)	320(11)	841(19)	274(11)	172(12)	-8(9)	-38(12)
C(21)	358(10)	297(10)	276(9)	73(8)	52(8)	21(8)
C(22)	235(9)	364(10)	180(8)	93(7)	69(7)	70(8)
C(23)	354(10)	394(11)	159(8)	-14(8)	32(8)	89(9)
C(24)	502(13)	371(11)	238(10)	-13(9)	89(9)	160(10)
C(25)	366(11)	541(14)	281(11)	158(10)	95(9)	203(10)
C(26)	243(10)	575(15)	350(11)	216(11)	22(9)	24(10)
C(27)	301(10)	417(12)	334(10)	165(9)	33(8)	-49(9)

CALIFORNIA INSTITUTE OF TECHNOLOGY

BECKMAN INSTITUTE

X-RAY CRYSTALLOGRAPHY LABORATORY

Date 12 March 2009

Crystal Structure Analysis of:

DYT13

(Complex 6)

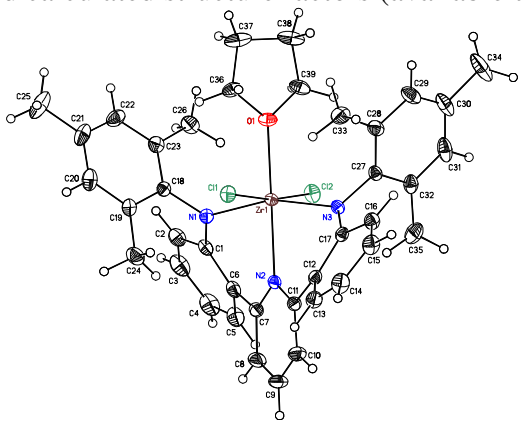
For Investigator: Daniel Tofan ext. 6576

Advisor: J. E. Bercaw ext. 6577

By Michael W. Day 116 Beckman ext. 2734

e-mail: mikeday@caltech.edu

Contents



DYT13

Note: The crystallographic data have been deposited in the Cambridge Database (CCDC). The deposition number is 723743.

Table 1. Crystal data and structure refinement for DYT13 (CCDC 723743).

Empirical formula	$C_{39}H_{41}N_3OCl_2Zr$
Formula weight	729.87
Crystallization Solvent	THF
Crystal Habit	Block
Crystal size	0.26 x 0.22 x 0.15 mm^3
Crystal color	Yellow/orange

Data Collection

Type of diffractometer	Bruker KAPPA APEX II	
Wavelength	0.71073 Å MoKα	
Data Collection Temperature	100(2) K	
θ range for 9916 reflections used in lattice determination	2.43 to 38.89°	
Unit cell dimensions	a = 11.7169(5) Å b = 12.7754(6) Å c = 13.0786(6) Å	α= 61.415(2)° β= 85.745(3)° γ = 85.899(3)°
Volume	1712.89(13) $Å^3$	
Z	2	
Crystal system	Triclinic	
Space group	P-1	
Density (calculated)	1.415 Mg/m^3	
F(000)	756	
Data collection program	Bruker APEX2 v2.1-0	
θ range for data collection	1.74 to 39.30°	
Completeness to θ = 39.30°	87.1 %	
Index ranges	$-20 \le h \le 20$, $-19 \le k \le 22$, $0 \le l \le 23$	
Data collection scan type	ω scans; 16 settings	
Data reduction program	Bruker SAINT-Plus v7.34A	
Reflections collected	17760	
Independent reflections	17760 [R_{int}= 0.0000]	
Absorption coefficient	0.513 mm^{-1}	
Absorption correction	Empirical, Twinabs, multi-scan	
Max. and min. transmission	0.7477 and 0.6054	

Table 1 (cont.)

Structure solution and Refinement

Structure solution program	SHELXS-97 (Sheldrick, 2008)
Primary solution method	Direct methods
Secondary solution method	Difference Fourier map
Hydrogen placement	Geometric positions
Structure refinement program	SHELXL-97 (Sheldrick, 2008)
Refinement method	Full matrix least-squares on F^2
Data / restraints / parameters	17760 / 0 / 422
Treatment of hydrogen atoms	Riding
Goodness-of-fit on F^2	1.957
Final R indices [$I>2\sigma(I)$, 14403 reflections]	R1 = 0.0381, *w*R2 = 0.0678
R indices (all data)	R1 = 0.0540, *w*R2 = 0.0693
Type of weighting scheme used	Sigma
Weighting scheme used	$w=1/\sigma^2(Fo^2)$
Max shift/error	0.003
Average shift/error	0.000
Largest diff. peak and hole	1.171 and -0.920 e.Å^{-3}

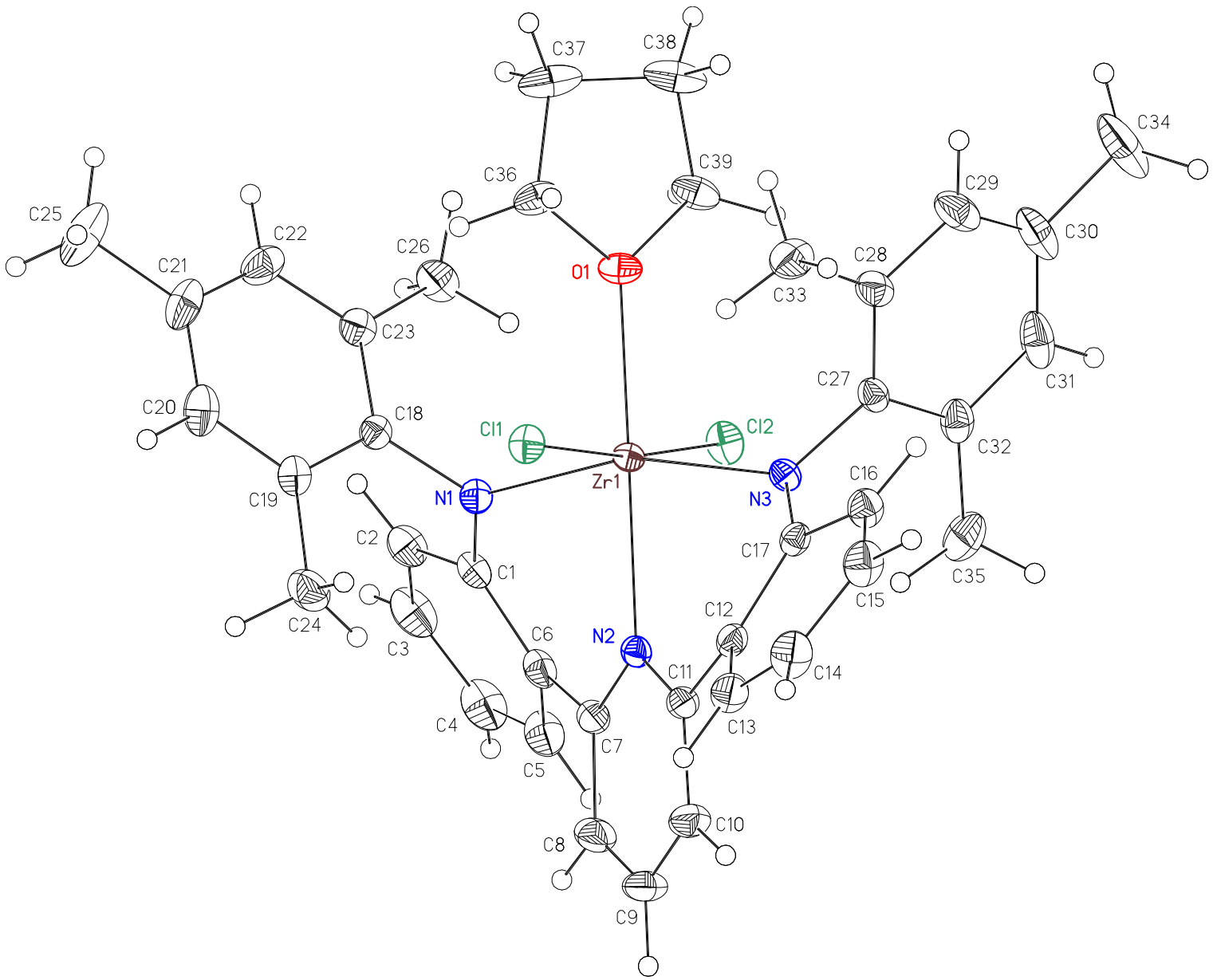
Special Refinement Details

Crystals were mounted on a glass fiber using Paratone oil then placed on the diffractometer under a nitrogen stream at 100K.

The crystal is a twin. Reflections from the multiple crystals were combined in a file by the thresholding routine. Input to Cellnow, the twin relationship between the two major components (related to each other by a two-fold rotation along the real a-axis) resulted in a multiple matrix file for use in data integration. The structure was solved by direct methods using data frome the major domain and refined against an HKLF5 type file compiled with Twinabs.

Refinement of F^2 against ALL reflections. The weighted R-factor (*w*R) and goodness of fit (S) are based on F^2, conventional R-factors (R) are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R-factors based on ALL data will be even larger.

All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.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C38
C34
C39
C36
C29
C25
C22
C26
O1
C28
C30
C21
C33
C23
C27
C31
C20
C18
Cl1
Cl2
C32
Zr1
C16
C19
N1
N3
C2
C17
C1
C35
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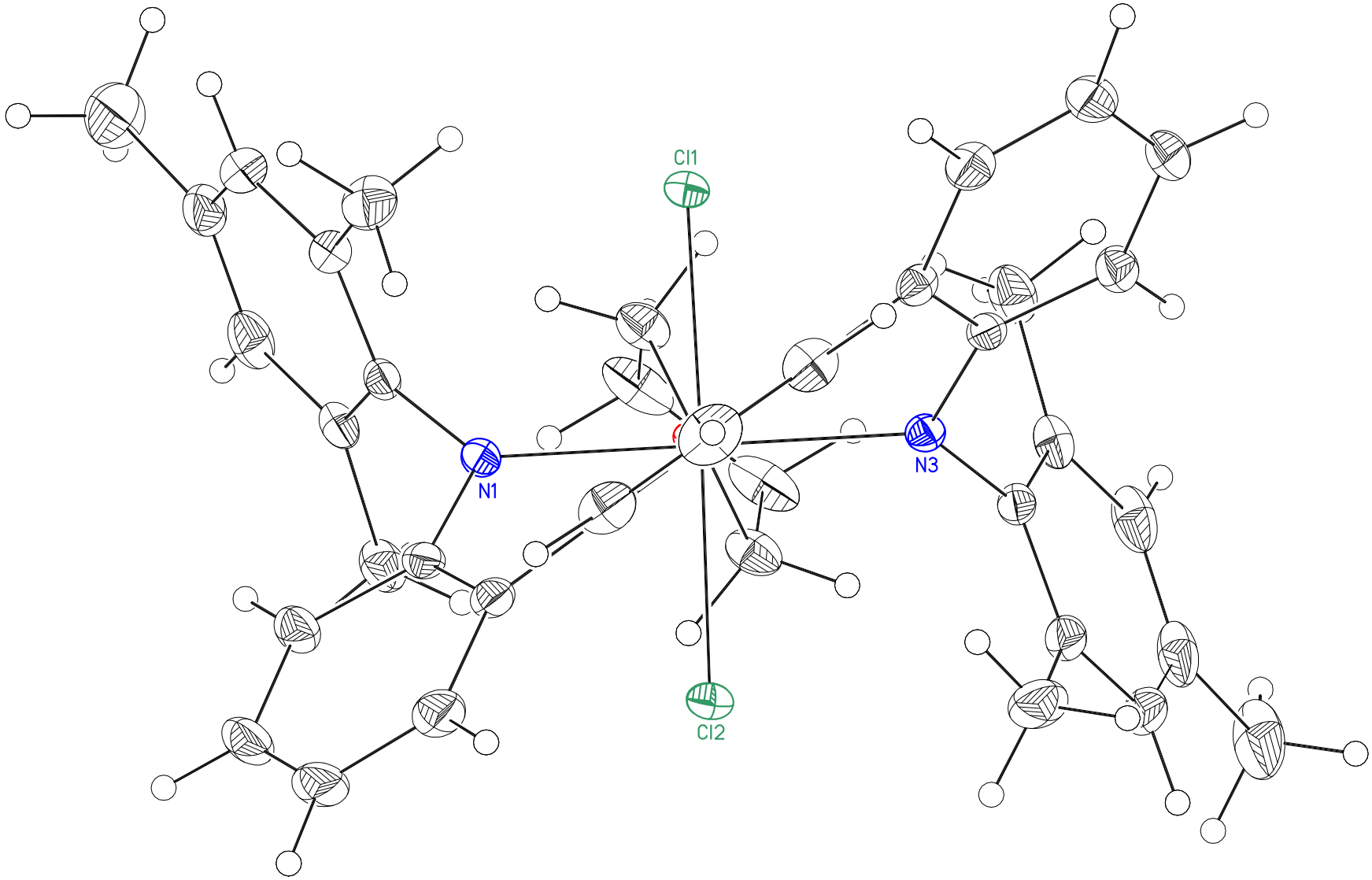
C4
C13
C7
C5
C10
C8
C9


Cl1
N1
N3
Cl2

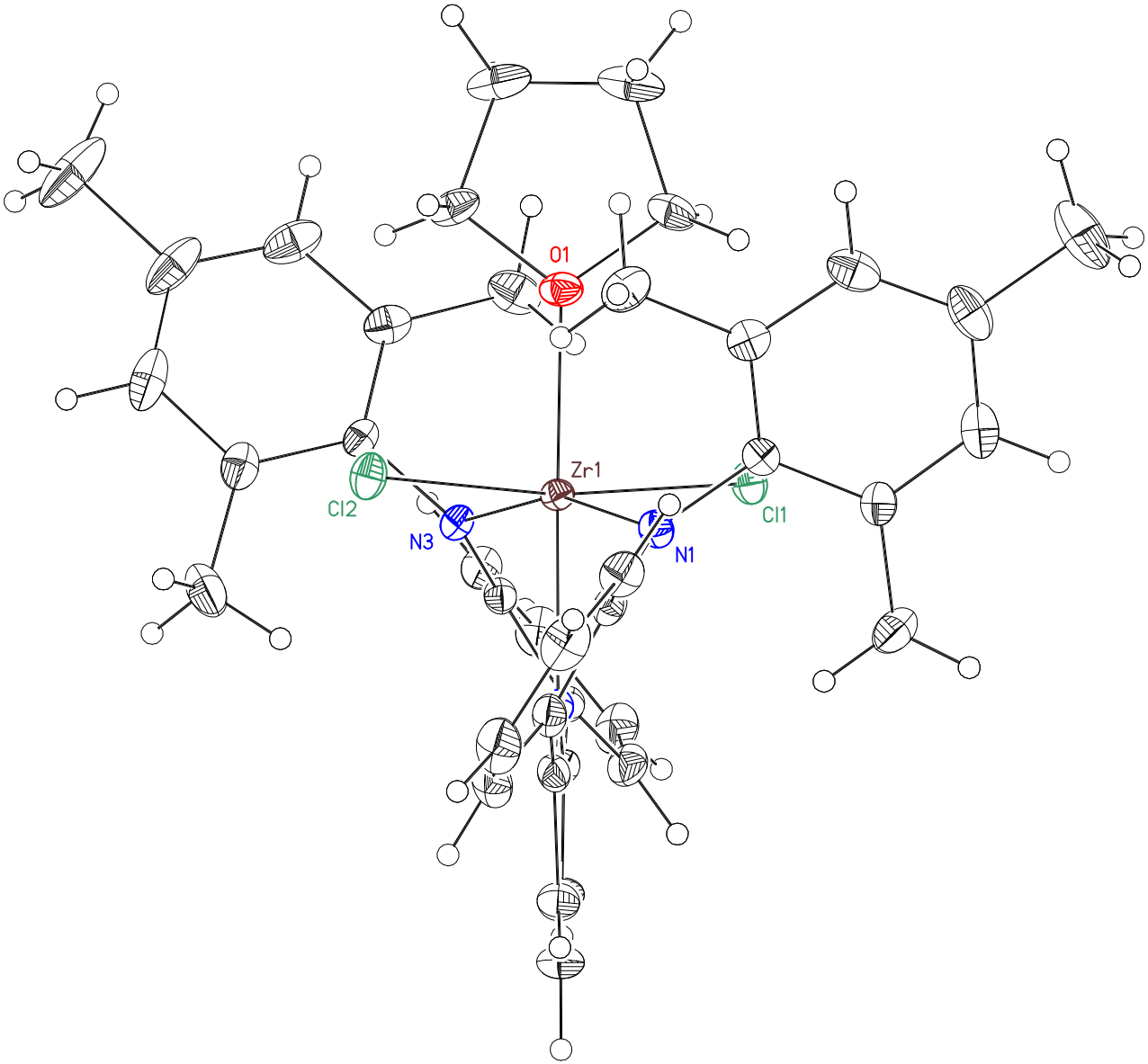

O1
Zr1
Cl2
Cl1
N3
N1

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for DYT13 (CCDC 723743). U(eq) is defined as the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U_{eq}
Zr(1)	2552(1)	2832(1)	7065(1)	13(1)
Cl(1)	4521(1)	3297(1)	7158(1)	18(1)
Cl(2)	556(1)	2647(1)	6755(1)	20(1)
O(1)	2485(1)	4571(1)	5408(1)	19(1)
N(1)	1976(1)	3437(1)	8286(1)	15(1)
N(2)	2683(1)	1043(1)	8768(1)	15(1)
N(3)	3165(1)	1692(1)	6334(1)	15(1)
C(1)	1075(1)	2903(1)	9104(1)	17(1)
C(2)	236(1)	3606(1)	9359(1)	23(1)
C(3)	-719(1)	3098(2)	10076(1)	29(1)
C(4)	-848(1)	1884(2)	10576(1)	30(1)
C(5)	-8(1)	1176(1)	10397(1)	27(1)
C(6)	989(1)	1655(1)	9679(1)	19(1)
C(7)	1920(1)	803(1)	9698(1)	18(1)
C(8)	2037(1)	-272(1)	10740(1)	26(1)
C(9)	2870(1)	-1117(1)	10821(1)	29(1)
C(10)	3612(1)	-886(1)	9884(1)	24(1)
C(11)	3532(1)	215(1)	8861(1)	17(1)
C(12)	4391(1)	379(1)	7912(1)	17(1)
C(13)	5471(1)	-208(1)	8233(1)	23(1)
C(14)	6254(1)	-322(1)	7450(1)	27(1)
C(15)	5938(1)	111(1)	6308(1)	25(1)
C(16)	4903(1)	720(1)	5955(1)	21(1)
C(17)	4132(1)	946(1)	6715(1)	15(1)
C(18)	2336(1)	4545(1)	8160(1)	16(1)
C(19)	3242(1)	4515(1)	8827(1)	19(1)
C(20)	3650(1)	5587(1)	8645(1)	27(1)
C(21)	3197(1)	6685(1)	7840(1)	28(1)
C(22)	2266(1)	6695(1)	7233(1)	26(1)
C(23)	1823(1)	5645(1)	7378(1)	19(1)
C(24)	3771(1)	3349(1)	9710(1)	28(1)
C(25)	3703(2)	7824(2)	7639(2)	45(1)
C(26)	797(1)	5723(1)	6711(1)	25(1)
C(27)	2772(1)	1943(1)	5219(1)	16(1)
C(28)	3291(1)	2817(1)	4171(1)	21(1)
C(29)	2827(1)	3086(1)	3118(1)	29(1)
C(30)	1879(1)	2543(2)	3061(1)	33(1)
C(31)	1410(1)	1654(2)	4097(1)	30(1)
C(32)	1846(1)	1342(1)	5173(1)	22(1)
C(33)	4344(1)	3434(1)	4158(1)	27(1)
C(34)	1348(2)	2893(2)	1914(1)	50(1)
C(35)	1322(1)	335(1)	6267(1)	31(1)
C(36)	3224(1)	5591(1)	5082(1)	22(1)
C(37)	2685(1)	6615(1)	4036(1)	32(1)
C(38)	2202(1)	5993(1)	3433(1)	31(1)
C(39)	1698(1)	4904(1)	4457(1)	23(1)

Table 3. Selected bond lengths [Å] and angles [°] for DYT13 (CCDC 723743).

Zr(1)-N(1)	2.1327(10)	N(1)-Zr(1)-N(3)	161.79(3)
Zr(1)-N(3)	2.1515(11)	N(1)-Zr(1)-O(1)	99.74(4)
Zr(1)-O(1)	2.2432(7)	N(3)-Zr(1)-O(1)	98.47(4)
Zr(1)-N(2)	2.3103(9)	N(1)-Zr(1)-N(2)	80.79(4)
Zr(1)-Cl(1)	2.4497(3)	N(3)-Zr(1)-N(2)	80.99(4)
Zr(1)-Cl(2)	2.4495(3)	O(1)-Zr(1)-N(2)	178.15(3)
		N(1)-Zr(1)-Cl(1)	90.43(3)
		N(3)-Zr(1)-Cl(1)	90.48(3)
		O(1)-Zr(1)-Cl(1)	86.01(2)
		N(2)-Zr(1)-Cl(1)	92.22(3)
		N(1)-Zr(1)-Cl(2)	89.56(3)
		N(3)-Zr(1)-Cl(2)	91.97(3)
		O(1)-Zr(1)-Cl(2)	86.26(2)
		N(2)-Zr(1)-Cl(2)	95.52(3)

Table 4. Bond lengths [Å] and angles [°] for DYT13 (CCDC 723743).

Zr(1)-N(1)	2.1327(10)	C(37)-C(38)	1.518(2)
Zr(1)-N(3)	2.1515(11)	C(38)-C(39)	1.5147(16)
Zr(1)-O(1)	2.2432(7)		
Zr(1)-N(2)	2.3103(9)	N(1)-Zr(1)-N(3)	161.79(3)
Zr(1)-Cl(1)	2.4497(3)	N(1)-Zr(1)-O(1)	99.74(4)
Zr(1)-Cl(2)	2.4495(3)	N(3)-Zr(1)-O(1)	98.47(4)
O(1)-C(36)	1.4843(16)	N(1)-Zr(1)-N(2)	80.79(4)
O(1)-C(39)	1.4785(15)	N(3)-Zr(1)-N(2)	80.99(4)
N(1)-C(1)	1.4006(13)	O(1)-Zr(1)-N(2)	178.15(3)
N(1)-C(18)	1.4355(17)	N(1)-Zr(1)-Cl(1)	90.43(3)
N(2)-C(7)	1.3760(14)	N(3)-Zr(1)-Cl(1)	90.48(3)
N(2)-C(11)	1.3663(17)	O(1)-Zr(1)-Cl(1)	86.01(2)
N(3)-C(17)	1.3848(16)	N(2)-Zr(1)-Cl(1)	92.22(3)
N(3)-C(27)	1.4408(14)	N(1)-Zr(1)-Cl(2)	89.56(3)
C(1)-C(2)	1.4122(18)	N(3)-Zr(1)-Cl(2)	91.97(3)
C(1)-C(6)	1.4078(19)	O(1)-Zr(1)-Cl(2)	86.26(2)
C(2)-C(3)	1.3847(16)	N(2)-Zr(1)-Cl(2)	95.52(3)
C(3)-C(4)	1.380(2)	Cl(1)-Zr(1)-Cl(2)	172.164(10)
C(4)-C(5)	1.372(2)	C(36)-O(1)-C(39)	108.24(8)
C(5)-C(6)	1.4174(15)	C(36)-O(1)-Zr(1)	125.63(7)
C(6)-C(7)	1.478(2)	C(39)-O(1)-Zr(1)	126.13(7)
C(7)-C(8)	1.4043(16)	C(1)-N(1)-C(18)	116.27(10)
C(8)-C(9)	1.373(2)	C(1)-N(1)-Zr(1)	121.56(9)
C(9)-C(10)	1.3700(19)	C(18)-N(1)-Zr(1)	121.08(6)
C(10)-C(11)	1.4056(15)	C(7)-N(2)-C(11)	119.41(9)
C(11)-C(12)	1.4807(16)	C(7)-N(2)-Zr(1)	120.70(8)
C(12)-C(17)	1.4219(15)	C(11)-N(2)-Zr(1)	119.88(7)
C(12)-C(13)	1.4079(18)	C(17)-N(3)-C(27)	116.57(10)
C(13)-C(14)	1.3765(19)	C(17)-N(3)-Zr(1)	122.44(8)
C(14)-C(15)	1.3916(19)	C(27)-N(3)-Zr(1)	118.67(8)
C(15)-C(16)	1.3763(19)	N(1)-C(1)-C(2)	120.67(12)
C(16)-C(17)	1.4103(16)	N(1)-C(1)-C(6)	120.31(12)
C(18)-C(19)	1.4091(18)	C(2)-C(1)-C(6)	119.01(10)
C(18)-C(23)	1.4068(17)	C(3)-C(2)-C(1)	120.97(14)
C(19)-C(20)	1.387(2)	C(2)-C(3)-C(4)	120.04(14)
C(19)-C(24)	1.5056(19)	C(5)-C(4)-C(3)	119.83(12)
C(20)-C(21)	1.386(2)	C(4)-C(5)-C(6)	122.03(14)
C(21)-C(22)	1.391(2)	C(5)-C(6)-C(1)	117.75(13)
C(21)-C(25)	1.508(2)	C(5)-C(6)-C(7)	117.36(12)
C(22)-C(23)	1.395(2)	C(1)-C(6)-C(7)	124.46(10)
C(23)-C(26)	1.5078(19)	N(2)-C(7)-C(8)	119.91(13)
C(27)-C(32)	1.3944(18)	N(2)-C(7)-C(6)	122.95(10)
C(27)-C(28)	1.4154(15)	C(8)-C(7)-C(6)	117.09(11)
C(28)-C(29)	1.3931(18)	C(9)-C(8)-C(7)	120.57(12)
C(28)-C(33)	1.505(2)	C(10)-C(9)-C(8)	119.25(11)
C(29)-C(30)	1.377(2)	C(9)-C(10)-C(11)	120.18(14)
C(30)-C(31)	1.390(2)	N(2)-C(11)-C(10)	120.56(11)
C(30)-C(34)	1.5150(19)	N(2)-C(11)-C(12)	123.78(9)
C(31)-C(32)	1.3923(19)	C(10)-C(11)-C(12)	115.63(12)
C(32)-C(35)	1.5166(18)	C(17)-C(12)-C(13)	118.35(11)
C(36)-C(37)	1.5100(17)	C(17)-C(12)-C(11)	123.51(11)

C(13)-C(12)-C(11)	117.49(10)
C(14)-C(13)-C(12)	122.75(11)
C(15)-C(14)-C(13)	118.35(13)
C(14)-C(15)-C(16)	120.39(13)
C(15)-C(16)-C(17)	122.14(11)
N(3)-C(17)-C(12)	120.12(10)
N(3)-C(17)-C(16)	122.68(10)
C(12)-C(17)-C(16)	117.20(11)
C(19)-C(18)-C(23)	119.92(12)
C(19)-C(18)-N(1)	118.49(11)
C(23)-C(18)-N(1)	121.58(11)
C(20)-C(19)-C(18)	118.70(12)
C(20)-C(19)-C(24)	120.23(12)
C(18)-C(19)-C(24)	121.06(12)
C(21)-C(20)-C(19)	122.56(14)
C(20)-C(21)-C(22)	117.83(14)
C(20)-C(21)-C(25)	120.55(15)
C(22)-C(21)-C(25)	121.62(15)
C(21)-C(22)-C(23)	121.98(13)
C(22)-C(23)-C(18)	118.85(12)
C(22)-C(23)-C(26)	119.11(12)
C(18)-C(23)-C(26)	122.03(12)
C(32)-C(27)-C(28)	119.65(11)
C(32)-C(27)-N(3)	119.49(9)
C(28)-C(27)-N(3)	120.83(11)
C(29)-C(28)-C(27)	118.58(13)
C(29)-C(28)-C(33)	119.21(11)
C(27)-C(28)-C(33)	122.18(11)
C(30)-C(29)-C(28)	122.49(12)
C(29)-C(30)-C(31)	117.89(13)
C(29)-C(30)-C(34)	121.81(14)
C(31)-C(30)-C(34)	120.30(16)
C(30)-C(31)-C(32)	121.98(14)
C(31)-C(32)-C(27)	119.28(11)
C(31)-C(32)-C(35)	119.37(13)
C(27)-C(32)-C(35)	121.33(12)
O(1)-C(36)-C(37)	104.91(11)
C(36)-C(37)-C(38)	102.56(12)
C(39)-C(38)-C(37)	101.78(11)
O(1)-C(39)-C(38)	104.87(11)

Table 5. Anisotropic displacement parameters ($Å^2 x 10^4$) for DYT13 (CCDC 723743). The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Zr(1)	110(1)	136(1)	117(1)	-50(1)	4(1)	-5(1)
Cl(1)	125(1)	212(1)	215(1)	-111(1)	15(1)	-31(1)
Cl(2)	126(1)	273(2)	246(1)	-147(1)	-22(1)	0(1)
O(1)	145(4)	185(5)	165(3)	-27(3)	3(3)	-6(3)
N(1)	140(4)	177(5)	156(3)	-86(3)	27(3)	-33(4)
N(2)	154(5)	158(5)	135(3)	-66(3)	-14(3)	-35(4)
N(3)	134(4)	167(5)	138(3)	-74(3)	-23(3)	4(3)
C(1)	131(5)	243(6)	141(4)	-98(4)	2(4)	-27(4)
C(2)	194(6)	304(7)	196(4)	-133(4)	33(4)	-13(5)
C(3)	180(6)	470(9)	243(5)	-202(6)	43(5)	-3(6)
C(4)	193(6)	476(10)	265(5)	-201(6)	92(5)	-126(6)
C(5)	251(7)	361(8)	229(5)	-158(5)	88(5)	-156(6)
C(6)	179(6)	269(7)	159(4)	-119(4)	34(4)	-83(5)
C(7)	194(6)	197(6)	149(4)	-74(4)	12(4)	-81(5)
C(8)	307(7)	254(7)	155(4)	-50(4)	12(5)	-106(6)
C(9)	405(8)	200(7)	182(5)	-6(4)	-51(5)	-66(6)
C(10)	294(7)	174(6)	216(5)	-47(4)	-73(5)	13(5)
C(11)	186(6)	156(6)	167(4)	-65(4)	-58(4)	-11(4)
C(12)	181(6)	136(5)	192(4)	-83(4)	-52(4)	14(4)
C(13)	232(6)	200(6)	267(5)	-114(5)	-101(5)	53(5)
C(14)	196(6)	277(8)	372(6)	-185(5)	-105(5)	89(5)
C(15)	209(6)	266(7)	318(6)	-179(5)	-19(5)	57(5)
C(16)	201(6)	216(6)	226(5)	-124(4)	-24(4)	25(5)
C(17)	139(5)	137(5)	187(4)	-84(4)	-29(4)	6(4)
C(18)	153(5)	167(5)	167(4)	-93(4)	25(4)	-9(4)
C(19)	176(6)	221(6)	217(5)	-131(4)	-9(4)	-10(5)
C(20)	210(7)	325(8)	360(6)	-237(5)	-12(5)	-30(6)
C(21)	251(7)	251(7)	402(7)	-225(6)	89(6)	-62(5)
C(22)	285(7)	171(6)	286(5)	-103(5)	55(5)	21(5)
C(23)	176(6)	211(6)	196(4)	-111(4)	11(4)	31(4)
C(24)	292(7)	310(8)	230(5)	-124(5)	-96(5)	42(6)
C(25)	424(10)	302(9)	726(11)	-332(8)	88(9)	-103(8)
C(26)	231(6)	284(7)	234(5)	-134(5)	-45(5)	84(5)
C(27)	160(5)	181(6)	157(4)	-95(4)	-38(4)	31(4)
C(28)	242(6)	212(6)	167(4)	-95(4)	8(4)	43(5)
C(29)	357(8)	328(8)	169(4)	-122(5)	-26(5)	123(6)
C(30)	348(8)	476(10)	259(5)	-267(6)	-151(5)	228(7)
C(31)	223(7)	438(9)	395(7)	-322(6)	-125(6)	87(6)
C(32)	195(6)	245(7)	271(5)	-173(5)	-51(5)	42(5)
C(33)	294(7)	239(7)	242(5)	-105(5)	98(5)	-41(5)
C(34)	528(11)	770(15)	341(7)	-401(8)	-249(7)	337(10)
C(35)	270(7)	259(8)	430(7)	-178(6)	-16(6)	-78(6)
C(36)	198(6)	167(6)	235(5)	-45(4)	51(4)	-29(5)
C(37)	225(7)	199(7)	311(6)	33(5)	82(5)	39(5)
C(38)	207(6)	340(8)	183(5)	24(5)	16(5)	70(5)
C(39)	174(6)	276(7)	168(4)	-48(4)	-34(4)	52(5)

CALIFORNIA INSTITUTE OF TECHNOLOGY

BECKMAN INSTITUTE

X-RAY CRYSTALLOGRAPHY LABORATORY

Date 16 November 2010

Crystal Structure Analysis of:

IAT65

(Complex 7)

For Investigator: Ian Tonks ext. 6576

Advisor: J. E. Bercaw ext. 6577

By Michael W. Day 116 Beckman ext. 2734

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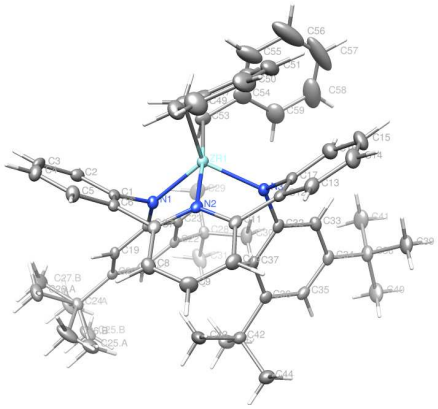
Contents



IAT65

Note: The crystallographic data have been deposited in the Cambridge Database (CCDC). The deposition number is 800979.

Table 1. Crystal data and structure refinement for IAT65 (CCDC 800979).

Empirical formula	$C_{59}H_{67}N_3Zr \bullet C_5H_{12}$
Formula weight	981.52
Crystallization Solvent	Pentane
Crystal Habit	Block
Crystal size	0.19 x 0.18 x 0.11 mm^3
Crystal color	Yellow

Data Collection

Type of diffractometer	Bruker KAPPA APEX II	
Wavelength	0.71073 Å MoKα	
Data Collection Temperature	100(2) K	
θ range for 9856 reflections used in lattice determination	2.17 to 27.29°	
Unit cell dimensions	a = 10.5191(4) Å b = 12.8102(5) Å c = 20.6372(8) Å	α= 96.952(2)° β= 90.511(2)° γ = 98.726(2)°
Volume	2727.45(18) $Å^3$	
Z	2	
Crystal system	Triclinic	
Space group	P-1	
Density (calculated)	1.195 Mg/m^3	
F(000)	1048	
θ range for data collection	1.79 to 27.45°	
Completeness to θ = 27.45°	99.0 %	
Index ranges	$-13 \le h \le 13$, $-16 \le k \le 16$, $-26 \le l \le 26$	
Data collection scan type	ω scans; 17 settings	
Reflections collected	93111	
Independent reflections	12342 [R_{int}= 0.0489]	
Absorption coefficient	0.243 mm^{-1}	
Absorption correction	None	
Max. and min. transmission	0.9738 and 0.9553	

Table 1 (cont.)

Structure solution and Refinement

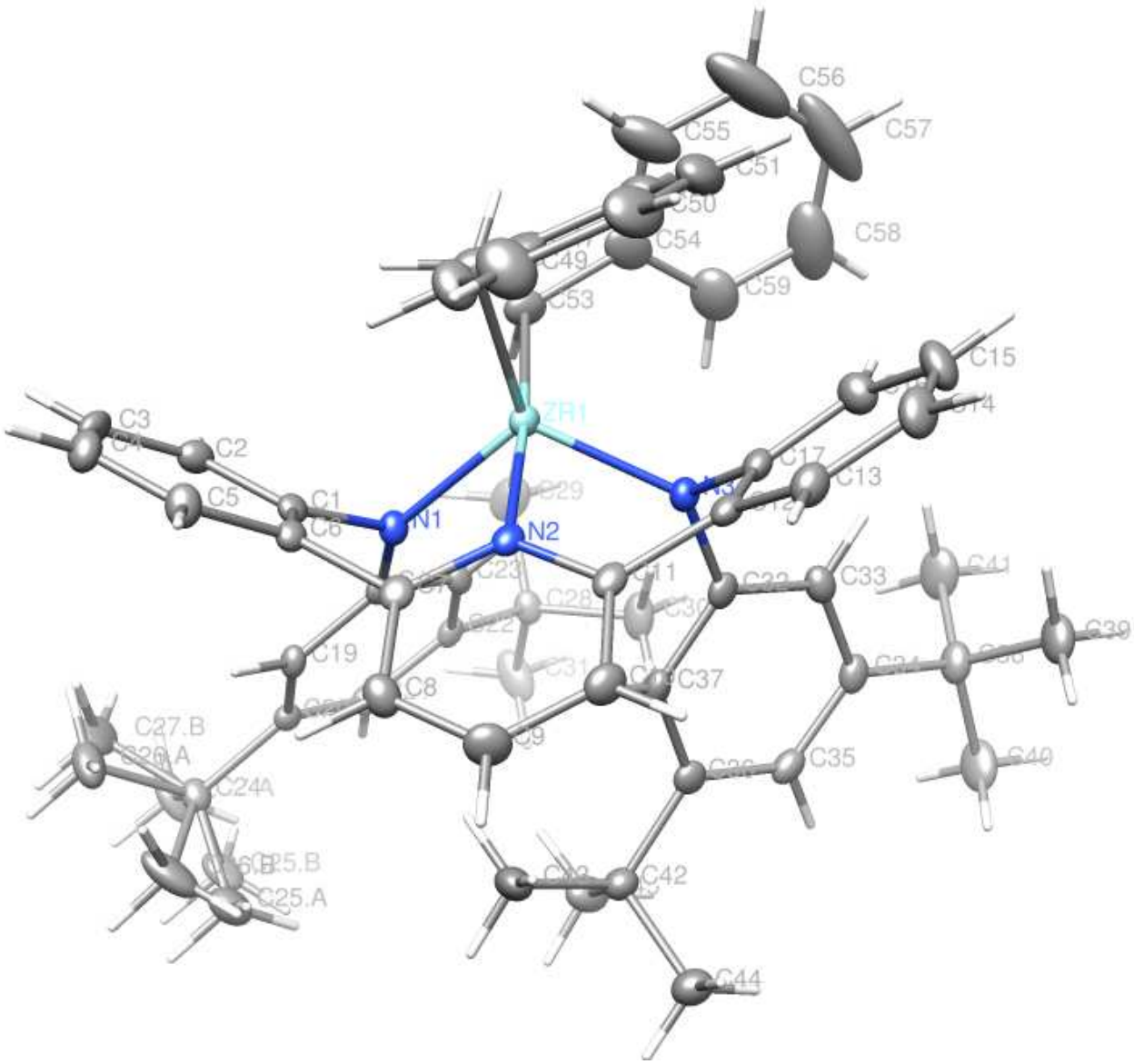
Structure solution program	SHELXS-97 (Sheldrick, 2008)
Primary solution method	Direct methods
Secondary solution method	Difference Fourier map
Hydrogen placement	Geometric positions
Structure refinement program	SHELXL-97 (Sheldrick, 2008)
Refinement method	Full matrix least-squares on F^2
Data / restraints / parameters	12342 / 0 / 658
Treatment of hydrogen atoms	Riding
Goodness-of-fit on F^2	1.925
Final R indices [$I>2\sigma(I)$, 10227 reflections]	R1 = 0.0352, *w*R2 = 0.0492
R indices (all data)	R1 = 0.0466, *w*R2 = 0.0499
Type of weighting scheme used	Sigma
Weighting scheme used	$w=1/\sigma^2(Fo^2)$
Max shift/error	0.001
Average shift/error	0.000
Largest diff. peak and hole	0.633 and -0.734 e.Å^{-3}

Special Refinement Details

Crystals were mounted on a glass fiber using Paratone oil then placed on the diffractometer under a nitrogen stream at 100K.

Refinement of F^2 against ALL reflections. The weighted R-factor (*w*R) and goodness of fit (S) are based on F^2, conventional R-factors (R) are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R-factors based on ALL data will be even larger.

All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.


C56
C57
C55
C51
C50
C58
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C49
C59
C53
C15
C14
ZR1
C3
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C17
C13
C5
C1
N1
N2
C6
C29
C11
C33
C41
C39
C19
C8
C37
C27.B
C24A
C35
C40
C25.B
C25.A
C42
C44

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for IAT65 (CCDC 800979). U(eq) is defined as the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U_{eq}	Occ
Zr(1)	2382(1)	2506(1)	8270(1)	14(1)	1
N(1)	2731(1)	4026(1)	8782(1)	14(1)	1
N(2)	205(1)	2683(1)	8508(1)	13(1)	1
N(3)	1642(1)	2418(1)	7349(1)	15(1)	1
C(1)	2332(2)	3938(1)	9436(1)	14(1)	1
C(2)	3232(2)	4125(1)	9954(1)	18(1)	1
C(3)	2887(2)	3976(1)	10583(1)	21(1)	1
C(4)	1605(2)	3624(1)	10707(1)	22(1)	1
C(5)	701(2)	3437(1)	10206(1)	20(1)	1
C(6)	1034(2)	3588(1)	9560(1)	14(1)	1
C(7)	-3(2)	3340(1)	9048(1)	14(1)	1
C(8)	-1161(2)	3717(1)	9135(1)	19(1)	1
C(9)	-2127(2)	3400(1)	8666(1)	22(1)	1
C(10)	-1938(2)	2695(1)	8127(1)	20(1)	1
C(11)	-760(2)	2338(1)	8058(1)	15(1)	1
C(12)	-545(2)	1521(1)	7514(1)	16(1)	1
C(13)	-1544(2)	680(1)	7326(1)	22(1)	1
C(14)	-1376(2)	-135(1)	6851(1)	27(1)	1
C(15)	-201(2)	-146(1)	6558(1)	29(1)	1
C(16)	799(2)	686(1)	6730(1)	24(1)	1
C(17)	633(2)	1532(1)	7191(1)	16(1)	1
C(18)	3461(2)	5003(1)	8652(1)	14(1)	1
C(19)	3441(2)	5962(1)	9047(1)	14(1)	1
C(20)	4136(2)	6913(1)	8895(1)	14(1)	1
C(21)	4871(2)	6878(1)	8334(1)	15(1)	1
C(22)	4916(2)	5939(1)	7928(1)	14(1)	1
C(23)	4189(2)	5008(1)	8092(1)	15(1)	1
C(24)	4061(2)	7957(1)	9327(1)	17(1)	1
C(25)	2960(3)	8430(2)	9068(2)	33(1)	0.642(3)
C(26)	3836(4)	7788(2)	10043(1)	33(1)	0.642(3)
C(27)	5325(3)	8736(2)	9318(2)	34(1)	0.642(3)
C(25B)	4491(6)	8962(4)	8978(3)	30(2)	0.358(3)
C(26B)	2636(5)	8048(4)	9521(3)	39(2)	0.358(3)
C(27B)	4888(7)	8035(4)	9933(3)	44(2)	0.358(3)
C(28)	5789(2)	5854(1)	7336(1)	16(1)	1
C(29)	6857(2)	5228(1)	7500(1)	33(1)	1
C(30)	5042(2)	5261(1)	6731(1)	26(1)	1
C(31)	6415(2)	6948(1)	7171(1)	33(1)	1
C(32)	1714(2)	3241(1)	6923(1)	15(1)	1
C(33)	2050(2)	3054(1)	6278(1)	18(1)	1
C(34)	2091(2)	3852(1)	5866(1)	18(1)	1
C(35)	1806(2)	4832(1)	6132(1)	18(1)	1
C(36)	1490(2)	5058(1)	6783(1)	17(1)	1
C(37)	1444(2)	4238(1)	7174(1)	16(1)	1
C(38)	2464(2)	3610(1)	5152(1)	22(1)	1
C(39)	1529(2)	2659(1)	4812(1)	29(1)	1
C(40)	2393(2)	4562(1)	4769(1)	32(1)	1

C(41)	3838(2)	3350(1)	5128(1)	34(1)	1
C(42)	1180(2)	6161(1)	7044(1)	20(1)	1
C(43)	797(2)	6222(1)	7760(1)	30(1)	1
C(44)	71(2)	6417(1)	6634(1)	26(1)	1
C(45)	2363(2)	7016(1)	6992(1)	30(1)	1
C(46)	2030(2)	1120(1)	8862(1)	24(1)	1
C(47)	792(2)	372(1)	8808(1)	22(1)	1
C(48)	-197(2)	501(1)	9243(1)	29(1)	1
C(49)	-1352(2)	-192(1)	9176(1)	36(1)	1
C(50)	-1552(2)	-1019(1)	8676(1)	35(1)	1
C(51)	-588(2)	-1156(1)	8240(1)	33(1)	1
C(52)	567(2)	-470(1)	8307(1)	27(1)	1
C(53)	4522(2)	2298(1)	8135(1)	22(1)	1
C(54)	4691(2)	1475(1)	7601(1)	28(1)	1
C(55)	4661(2)	411(2)	7700(1)	48(1)	1
C(56)	4753(2)	-375(2)	7182(2)	74(1)	1
C(57)	4905(2)	-128(2)	6554(2)	82(1)	1
C(58)	4957(2)	917(2)	6450(1)	65(1)	1
C(59)	4843(2)	1696(2)	6959(1)	40(1)	1
C(61)	8152(2)	3269(1)	6251(1)	49(1)	1
C(62)	8251(2)	2199(2)	5859(1)	45(1)	1
C(63)	7721(2)	2139(2)	5168(1)	51(1)	1
C(64)	7906(2)	1111(2)	4760(1)	54(1)	1
C(65)	7206(2)	108(2)	4979(1)	64(1)	1

Table 3. Selected bond lengths [Å] and angles [°] for IAT65 (CCDC 800979).

Zr(1)-N(3)	2.0312(13)	N(3)-Zr(1)-N(1)	115.23(5)
Zr(1)-N(1)	2.0791(13)	N(3)-Zr(1)-C(46)	122.10(6)
Zr(1)-C(46)	2.2616(14)	N(1)-Zr(1)-C(46)	117.27(6)
Zr(1)-C(53)	2.3215(16)	N(3)-Zr(1)-C(53)	104.94(6)
Zr(1)-N(2)	2.3850(13)	N(1)-Zr(1)-C(53)	96.62(6)
Zr(1)-C(1)	2.8494(16)	C(46)-Zr(1)-C(53)	91.72(6)
		N(3)-Zr(1)-N(2)	80.03(5)
		N(1)-Zr(1)-N(2)	82.37(5)
		C(46)-Zr(1)-N(2)	84.28(5)
		C(53)-Zr(1)-N(2)	174.82(6)
		N(3)-Zr(1)-C(1)	134.40(5)
		N(1)-Zr(1)-C(1)	28.62(4)
		C(46)-Zr(1)-C(1)	89.59(5)
		C(53)-Zr(1)-C(1)	105.92(6)
		N(2)-Zr(1)-C(1)	70.84(5)

Table 4. Bond lengths [Å] and angles [°] for IAT65 (CCDC 800979).

Zr(1)-N(3)	2.0312(13)	C(34)-C(38)	1.535(2)
Zr(1)-N(1)	2.0791(13)	C(35)-C(36)	1.393(2)
Zr(1)-C(46)	2.2616(14)	C(36)-C(37)	1.3962(19)
Zr(1)-C(53)	2.3215(16)	C(36)-C(42)	1.533(2)
Zr(1)-N(2)	2.3850(13)	C(38)-C(41)	1.532(2)
Zr(1)-C(1)	2.8494(16)	C(38)-C(39)	1.536(2)
N(1)-C(18)	1.4220(17)	C(38)-C(40)	1.542(2)
N(1)-C(1)	1.4287(19)	C(42)-C(44)	1.533(2)
N(2)-C(11)	1.354(2)	C(42)-C(43)	1.532(2)
N(2)-C(7)	1.355(2)	C(42)-C(45)	1.542(2)
N(3)-C(17)	1.4352(19)	C(46)-C(47)	1.490(2)
N(3)-C(32)	1.4466(18)	C(47)-C(52)	1.392(2)
C(1)-C(2)	1.397(2)	C(47)-C(48)	1.396(2)
C(1)-C(6)	1.407(2)	C(48)-C(49)	1.386(2)
C(2)-C(3)	1.377(2)	C(49)-C(50)	1.377(3)
C(3)-C(4)	1.392(2)	C(50)-C(51)	1.379(2)
C(4)-C(5)	1.373(2)	C(51)-C(52)	1.382(2)
C(5)-C(6)	1.409(2)	C(53)-C(54)	1.462(2)
C(6)-C(7)	1.484(2)	C(54)-C(59)	1.393(3)
C(7)-C(8)	1.381(2)	C(54)-C(55)	1.399(2)
C(8)-C(9)	1.378(2)	C(55)-C(56)	1.391(3)
C(9)-C(10)	1.382(2)	C(56)-C(57)	1.375(3)
C(10)-C(11)	1.387(2)	C(57)-C(58)	1.374(3)
C(11)-C(12)	1.483(2)	C(58)-C(59)	1.377(3)
C(12)-C(13)	1.401(2)	C(61)-C(62)	1.523(3)
C(12)-C(17)	1.410(2)	C(62)-C(63)	1.513(3)
C(13)-C(14)	1.375(2)	C(63)-C(64)	1.514(3)
C(14)-C(15)	1.382(2)	C(64)-C(65)	1.503(2)
C(15)-C(16)	1.389(2)		
C(16)-C(17)	1.387(2)	N(3)-Zr(1)-N(1)	115.23(5)
C(18)-C(23)	1.392(2)	N(3)-Zr(1)-C(46)	122.10(6)
C(18)-C(19)	1.393(2)	N(1)-Zr(1)-C(46)	117.27(6)
C(19)-C(20)	1.3934(19)	N(3)-Zr(1)-C(53)	104.94(6)
C(20)-C(21)	1.398(2)	N(1)-Zr(1)-C(53)	96.62(6)
C(20)-C(24)	1.529(2)	C(46)-Zr(1)-C(53)	91.72(6)
C(21)-C(22)	1.387(2)	N(3)-Zr(1)-N(2)	80.03(5)
C(22)-C(23)	1.3963(19)	N(1)-Zr(1)-N(2)	82.37(5)
C(22)-C(28)	1.538(2)	C(46)-Zr(1)-N(2)	84.28(5)
C(24)-C(25)	1.508(3)	C(53)-Zr(1)-N(2)	174.82(6)
C(24)-C(26)	1.535(3)	N(3)-Zr(1)-C(1)	134.40(5)
C(24)-C(27B)	1.503(6)	N(1)-Zr(1)-C(1)	28.62(4)
C(24)-C(27)	1.539(3)	C(46)-Zr(1)-C(1)	89.59(5)
C(24)-C(25B)	1.560(5)	C(53)-Zr(1)-C(1)	105.92(6)
C(24)-C(26B)	1.572(5)	N(2)-Zr(1)-C(1)	70.84(5)
C(28)-C(30)	1.525(2)	C(18)-N(1)-C(1)	117.95(13)
C(28)-C(29)	1.534(2)	C(18)-N(1)-Zr(1)	133.52(10)
C(28)-C(31)	1.5339(19)	C(1)-N(1)-Zr(1)	107.18(9)
C(32)-C(33)	1.382(2)	C(11)-N(2)-C(7)	119.67(15)
C(32)-C(37)	1.389(2)	C(11)-N(2)-Zr(1)	121.50(11)
C(33)-C(34)	1.404(2)	C(7)-N(2)-Zr(1)	117.32(11)
C(34)-C(35)	1.385(2)	C(17)-N(3)-C(32)	115.51(13)

C(17)-N(3)-Zr(1)	112.93(9)	C(27B)-C(24)-C(20)	110.0(2)
C(32)-N(3)-Zr(1)	128.95(10)	C(27)-C(24)-C(20)	110.92(16)
C(2)-C(1)-C(6)	118.91(15)	C(25)-C(24)-C(25B)	66.0(2)
C(2)-C(1)-N(1)	120.68(15)	C(26)-C(24)-C(25B)	133.4(3)
C(6)-C(1)-N(1)	120.26(15)	C(27B)-C(24)-C(25B)	108.4(3)
C(2)-C(1)-Zr(1)	126.65(11)	C(27)-C(24)-C(25B)	45.9(2)
C(6)-C(1)-Zr(1)	94.98(10)	C(20)-C(24)-C(25B)	113.3(2)
N(1)-C(1)-Zr(1)	44.20(7)	C(25)-C(24)-C(26B)	43.3(2)
C(3)-C(2)-C(1)	121.97(16)	C(26)-C(24)-C(26B)	68.5(3)
C(2)-C(3)-C(4)	119.25(17)	C(27B)-C(24)-C(26B)	109.6(4)
C(5)-C(4)-C(3)	119.94(16)	C(27)-C(24)-C(26B)	135.8(2)
C(4)-C(5)-C(6)	121.66(16)	C(20)-C(24)-C(26B)	111.3(2)
C(1)-C(6)-C(5)	118.27(16)	C(25B)-C(24)-C(26B)	104.1(3)
C(1)-C(6)-C(7)	123.83(14)	C(30)-C(28)-C(29)	108.38(14)
C(5)-C(6)-C(7)	117.87(15)	C(30)-C(28)-C(31)	108.13(13)
N(2)-C(7)-C(8)	121.34(16)	C(29)-C(28)-C(31)	108.54(14)
N(2)-C(7)-C(6)	117.23(15)	C(30)-C(28)-C(22)	111.28(13)
C(8)-C(7)-C(6)	121.34(16)	C(29)-C(28)-C(22)	108.07(13)
C(9)-C(8)-C(7)	119.08(17)	C(31)-C(28)-C(22)	112.34(14)
C(8)-C(9)-C(10)	119.76(17)	C(33)-C(32)-C(37)	119.95(14)
C(9)-C(10)-C(11)	119.27(17)	C(33)-C(32)-N(3)	121.29(15)
N(2)-C(11)-C(10)	120.75(16)	C(37)-C(32)-N(3)	118.75(14)
N(2)-C(11)-C(12)	118.04(15)	C(32)-C(33)-C(34)	120.76(16)
C(10)-C(11)-C(12)	121.11(16)	C(35)-C(34)-C(33)	117.60(16)
C(13)-C(12)-C(17)	118.51(16)	C(35)-C(34)-C(38)	123.27(14)
C(13)-C(12)-C(11)	118.43(15)	C(33)-C(34)-C(38)	119.12(15)
C(17)-C(12)-C(11)	123.02(15)	C(34)-C(35)-C(36)	123.27(15)
C(14)-C(13)-C(12)	121.10(17)	C(35)-C(36)-C(37)	117.25(16)
C(13)-C(14)-C(15)	120.19(17)	C(35)-C(36)-C(42)	120.46(14)
C(14)-C(15)-C(16)	119.76(18)	C(37)-C(36)-C(42)	122.28(15)
C(17)-C(16)-C(15)	120.81(17)	C(32)-C(37)-C(36)	121.14(15)
C(16)-C(17)-C(12)	119.51(15)	C(41)-C(38)-C(34)	109.66(15)
C(16)-C(17)-N(3)	121.15(15)	C(41)-C(38)-C(39)	109.61(15)
C(12)-C(17)-N(3)	119.34(15)	C(34)-C(38)-C(39)	109.73(13)
C(23)-C(18)-C(19)	118.61(14)	C(41)-C(38)-C(40)	108.77(14)
C(23)-C(18)-N(1)	118.50(15)	C(34)-C(38)-C(40)	111.59(15)
C(19)-C(18)-N(1)	122.84(14)	C(39)-C(38)-C(40)	107.44(15)
C(20)-C(19)-C(18)	121.48(14)	C(36)-C(42)-C(44)	109.41(14)
C(19)-C(20)-C(21)	118.00(15)	C(36)-C(42)-C(43)	111.93(13)
C(19)-C(20)-C(24)	120.18(14)	C(44)-C(42)-C(43)	108.82(15)
C(21)-C(20)-C(24)	121.81(13)	C(36)-C(42)-C(45)	110.12(14)
C(22)-C(21)-C(20)	122.28(14)	C(44)-C(42)-C(45)	108.43(13)
C(21)-C(22)-C(23)	117.88(15)	C(43)-C(42)-C(45)	108.05(15)
C(21)-C(22)-C(28)	123.74(14)	C(47)-C(46)-Zr(1)	121.77(12)
C(23)-C(22)-C(28)	118.23(15)	C(52)-C(47)-C(48)	117.40(17)
C(18)-C(23)-C(22)	121.73(16)	C(52)-C(47)-C(46)	120.86(17)
C(25)-C(24)-C(26)	109.5(2)	C(48)-C(47)-C(46)	121.72(17)
C(25)-C(24)-C(27B)	140.5(3)	C(49)-C(48)-C(47)	120.84(18)
C(26)-C(24)-C(27B)	43.9(3)	C(50)-C(49)-C(48)	120.67(19)
C(25)-C(24)-C(27)	110.21(19)	C(49)-C(50)-C(51)	119.32(18)
C(26)-C(24)-C(27)	106.6(2)	C(50)-C(51)-C(52)	120.16(19)
C(27B)-C(24)-C(27)	66.6(3)	C(51)-C(52)-C(47)	121.60(18)
C(25)-C(24)-C(20)	107.64(18)	C(54)-C(53)-Zr(1)	112.62(12)
C(26)-C(24)-C(20)	111.98(15)	C(59)-C(54)-C(55)	115.96(19)

C(59)-C(54)-C(53)	122.09(17)
C(55)-C(54)-C(53)	121.91(19)
C(56)-C(55)-C(54)	121.4(2)
C(57)-C(56)-C(55)	121.1(2)
C(56)-C(57)-C(58)	118.3(3)
C(57)-C(58)-C(59)	121.0(3)
C(58)-C(59)-C(54)	122.3(2)
C(63)-C(62)-C(61)	111.34(17)
C(64)-C(63)-C(62)	111.74(18)
C(65)-C(64)-C(63)	115.84(19)

Table 5. Anisotropic displacement parameters ($Å^2 x 10^4$) for IAT65 (CCDC 800979). The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2a^{*2}U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Zr(1)	135(1)	143(1)	144(1)	20(1)	-10(1)	7(1)
N(1)	140(8)	150(7)	124(8)	22(6)	19(6)	-15(6)
N(2)	151(8)	120(7)	131(8)	51(6)	-4(6)	-8(6)
N(3)	156(8)	156(7)	128(8)	17(6)	-1(6)	2(6)
C(1)	176(9)	99(9)	142(9)	4(7)	-2(8)	11(7)
C(2)	162(9)	153(9)	210(10)	14(8)	-14(8)	18(7)
C(3)	263(11)	197(10)	164(10)	17(8)	-84(8)	51(8)
C(4)	322(11)	222(10)	134(10)	47(8)	16(9)	27(9)
C(5)	212(10)	208(10)	162(10)	26(8)	35(8)	-10(8)
C(6)	171(9)	109(9)	131(9)	18(7)	-2(8)	4(7)
C(7)	158(9)	128(9)	140(10)	61(8)	25(8)	-19(7)
C(8)	179(10)	189(9)	182(10)	7(8)	41(8)	-2(8)
C(9)	135(10)	262(11)	279(12)	38(9)	27(8)	42(8)
C(10)	154(9)	215(10)	219(11)	48(8)	-29(8)	-7(8)
C(11)	159(9)	140(9)	143(10)	64(8)	-9(8)	-21(7)
C(12)	189(10)	144(9)	146(10)	49(8)	-43(8)	15(7)
C(13)	223(10)	220(10)	195(10)	43(8)	-32(8)	-8(8)
C(14)	328(12)	206(10)	242(11)	10(9)	-57(9)	-68(9)
C(15)	434(13)	186(10)	224(11)	-43(9)	15(10)	-1(9)
C(16)	291(11)	218(10)	203(11)	24(9)	34(9)	36(9)
C(17)	214(10)	150(9)	129(9)	33(8)	-40(8)	19(8)
C(18)	104(9)	158(9)	137(9)	21(8)	-22(7)	4(7)
C(19)	126(9)	188(9)	98(9)	11(7)	17(7)	18(7)
C(20)	130(9)	154(9)	127(9)	16(7)	-14(7)	33(7)
C(21)	138(9)	157(9)	162(10)	53(8)	-4(8)	-19(7)
C(22)	113(9)	195(9)	113(9)	24(8)	-13(7)	14(7)
C(23)	142(9)	159(9)	139(9)	-16(8)	-11(7)	9(7)
C(24)	208(10)	151(9)	157(10)	18(8)	38(8)	5(8)
C(25)	400(20)	257(19)	350(20)	-34(16)	18(17)	133(16)
C(26)	580(30)	207(18)	188(18)	-1(14)	74(18)	32(17)
C(27)	370(20)	225(18)	350(20)	-117(16)	115(17)	-74(15)
C(25B)	440(40)	140(30)	320(40)	10(30)	120(30)	60(30)
C(26B)	400(40)	180(30)	580(50)	-40(30)	220(40)	50(30)
C(27B)	820(60)	150(30)	340(40)	-80(30)	-150(40)	150(40)
C(28)	143(9)	189(9)	148(10)	3(8)	33(8)	-6(7)
C(29)	226(11)	541(13)	245(12)	76(10)	71(9)	118(10)
C(30)	228(11)	366(11)	157(10)	-5(9)	52(9)	-29(9)
C(31)	406(13)	276(11)	263(12)	-18(9)	198(10)	-75(9)
C(32)	101(9)	197(9)	135(9)	26(8)	-18(7)	-19(7)
C(33)	150(9)	189(9)	172(10)	-14(8)	12(8)	1(7)
C(34)	137(9)	230(10)	140(10)	17(8)	-6(8)	-25(8)
C(35)	152(9)	216(10)	170(10)	69(8)	-28(8)	-12(8)
C(36)	113(9)	200(10)	178(10)	19(8)	-25(8)	11(7)
C(37)	130(9)	237(10)	110(9)	18(8)	1(7)	24(7)
C(38)	233(10)	257(10)	140(10)	20(8)	21(8)	-18(8)
C(39)	370(12)	320(11)	155(11)	9(9)	17(9)	-13(9)
C(40)	426(13)	352(11)	154(11)	49(9)	80(10)	-33(10)

C(41)	303(12)	497(13)	205(11)	-2(10)	89(10)	42(10)
C(42)	211(10)	209(10)	189(10)	34(8)	-17(8)	58(8)
C(43)	435(13)	270(11)	226(12)	-11(9)	-11(10)	195(10)
C(44)	243(11)	287(11)	260(11)	57(9)	-10(9)	83(9)
C(45)	288(12)	218(10)	388(13)	-11(9)	-73(10)	52(9)
C(46)	291(11)	199(10)	258(11)	77(9)	-28(9)	58(8)
C(47)	308(11)	149(10)	233(11)	105(9)	-45(9)	53(8)
C(48)	401(13)	176(10)	257(12)	20(9)	27(10)	-29(9)
C(49)	369(13)	288(12)	405(14)	40(10)	86(11)	-22(10)
C(50)	349(13)	186(11)	480(15)	28(10)	-86(11)	-41(9)
C(51)	435(14)	174(11)	365(13)	-28(9)	-124(11)	78(10)
C(52)	359(12)	209(10)	276(12)	50(9)	-19(10)	110(9)
C(53)	156(10)	225(10)	295(12)	53(9)	-34(9)	20(8)
C(54)	102(9)	294(11)	420(14)	1(10)	-14(9)	19(8)
C(55)	357(13)	290(12)	800(20)	18(13)	133(13)	102(10)
C(56)	445(17)	352(15)	1350(30)	-188(18)	243(19)	83(12)
C(57)	368(16)	740(20)	1170(30)	-650(20)	107(18)	36(15)
C(58)	391(15)	990(20)	500(18)	-291(16)	17(13)	164(16)
C(59)	286(12)	504(14)	417(15)	-36(12)	55(11)	120(11)
C(61)	609(17)	391(13)	470(16)	61(12)	-44(13)	51(12)
C(62)	396(14)	592(15)	358(14)	114(12)	17(11)	47(12)
C(63)	494(16)	604(16)	425(16)	71(13)	12(13)	27(13)
C(64)	474(16)	565(16)	546(17)	-79(14)	-13(13)	50(13)
C(65)	770(20)	604(17)	513(18)	-52(14)	-1(15)	56(15)

CALIFORNIA INSTITUTE OF TECHNOLOGY

BECKMAN INSTITUTE

X-RAY CRYSTALLOGRAPHY LABORATORY

Date 13 September 2010

Crystal Structure Analysis of:

ECW18

(^{t}BuNNN)-H_2

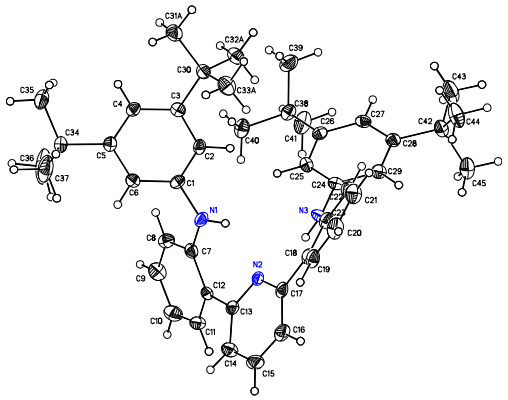
For Investigator: Edward Weintrob ext. 6576

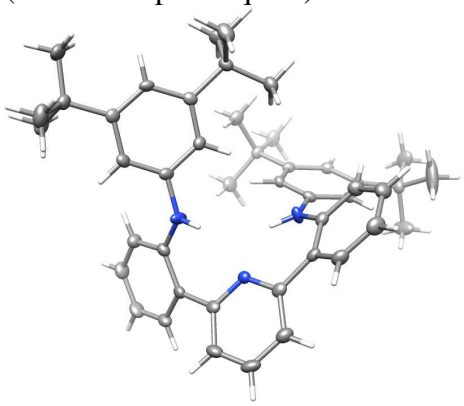
Advisor: J. E. Bercaw ext. 6577

By Michael W. Day 116 Beckman ext. 2734

e-mail: mikeday@caltech.edu

Contents

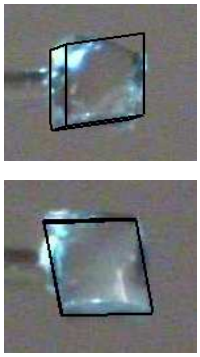




ECW18

Note: The crystallographic data have been deposited in the Cambridge Database (CCDC). The deposition number is 793155.

Table 1. Crystal data and structure refinement for ECW18 (CCDC 793155).



Empirical formula	$C_{45}H_{55}N_3 \bullet C_6H_{12}$
Formula weight	722.08
Crystallization Solvent	Hexanes
Crystal Habit	Block
Crystal size	0.19 x 0.17 x 0.16 mm^3
Crystal color	Colorless

Data Collection

Type of diffractometer	Bruker KAPPA APEX II	
Wavelength	0.71073 Å MoKα	
Data Collection Temperature	100(2) K	
θ range for 9933 reflections used in lattice determination	2.39 to 25.70°	
Unit cell dimensions	a = 13.2349(5) Å b = 17.6560(8) Å c = 19.0978(8) Å	α= 90° β= 102.355(2)° γ = 90°
Volume	4359.3(3) $Å^3$	
Z	4	
Crystal system	Monoclinic	
Space group	P $2_1/c$	
Density (calculated)	1.100 Mg/m^3	
F(000)	1576	
θ range for data collection	1.95 to 27.52°	
Completeness to θ = 27.52°	99.8 %	
Index ranges	$-17 \le h \le 16$, $-22 \le k \le 22$, $-24 \le l \le 24$	
Data collection scan type	ω scans; 8 settings	
Reflections collected	63493	
Independent reflections	10009 [R_{int}= 0.0554]	
Absorption coefficient	0.063 mm^{-1}	
Absorption correction	None	
Max. and min. transmission	0.9900 and 0.9881	

Table 1 (cont.)

Structure solution and Refinement

Structure solution program	SHELXS-97 (Sheldrick, 2008)
Primary solution method	Direct methods
Secondary solution method	Difference Fourier map
Hydrogen placement	Geometric positions
Structure refinement program	SHELXL-97 (Sheldrick, 2008)
Refinement method	Full matrix least-squares on F^2
Data / restraints / parameters	10009 / 0 / 537
Treatment of hydrogen atoms	Riding
Goodness-of-fit on F^2	2.513
Final R indices [$I>2\sigma(I)$, 6629 reflections]	R1 = 0.0641, *w*R2 = 0.0867
R indices (all data)	R1 = 0.0986, *w*R2 = 0.0886
Type of weighting scheme used	Sigma
Weighting scheme used	$w=1/\sigma^2(Fo^2)$
Max shift/error	0.000
Average shift/error	0.000
Largest diff. peak and hole	1.093 and -0.828 e.Å^{-3}

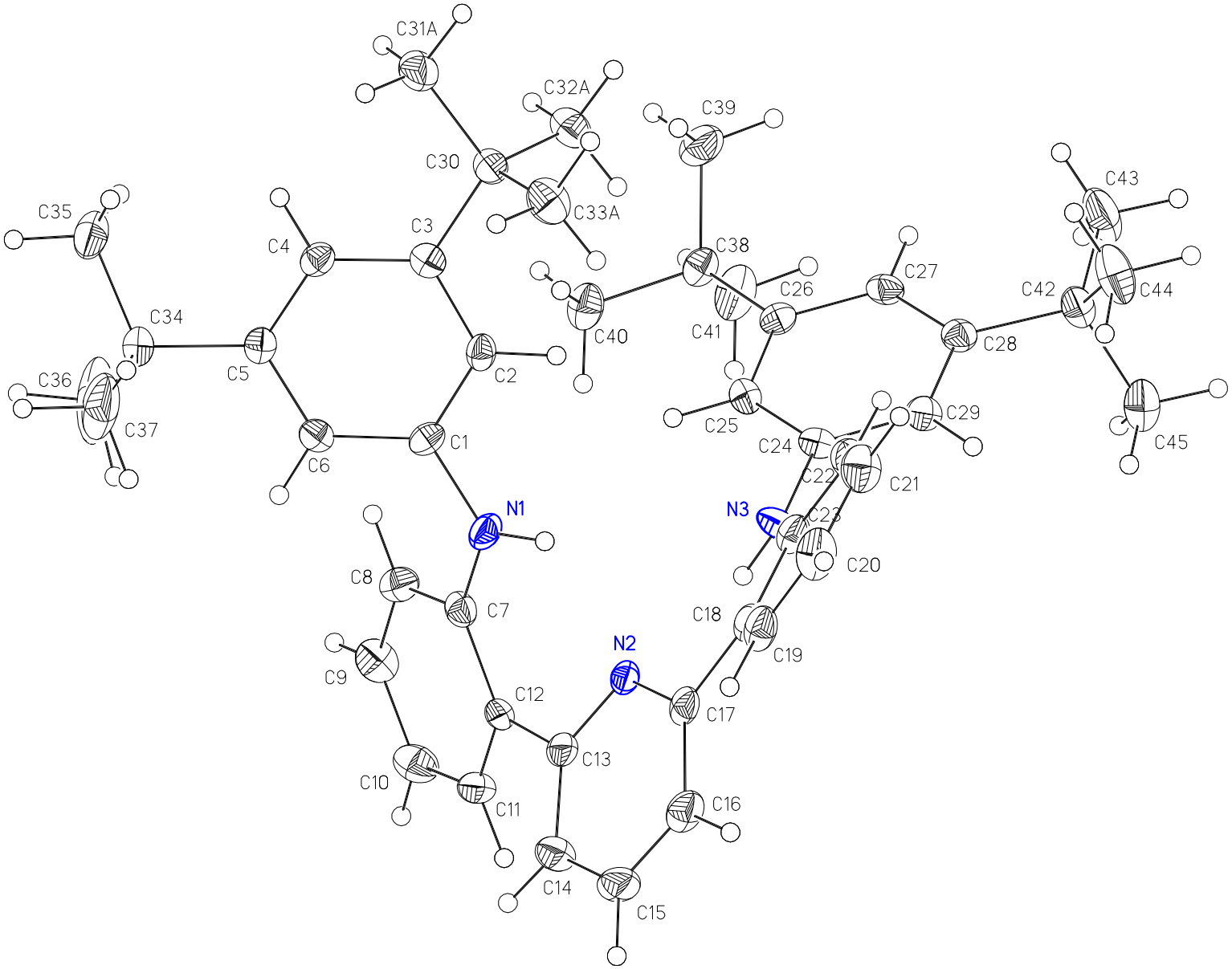
Special Refinement Details

Crystals were mounted on a glass fiber using Paratone oil then placed on the diffractometer under a nitrogen stream at 100K.

One of the t-butyl groups is disordered by rotation around the tertiary C-C bond as is common for t-butyl groups. Additional disorder occurs in the solvent of crystallization, methylcylcopentane, with the methyl group disordered over two carbon sites.

Refinement of F^2 against ALL reflections. The weighted R-factor (*w*R) and goodness of fit (S) are based on F^2, conventional R-factors (R) are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R-factors based on ALL data will be even larger.

All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.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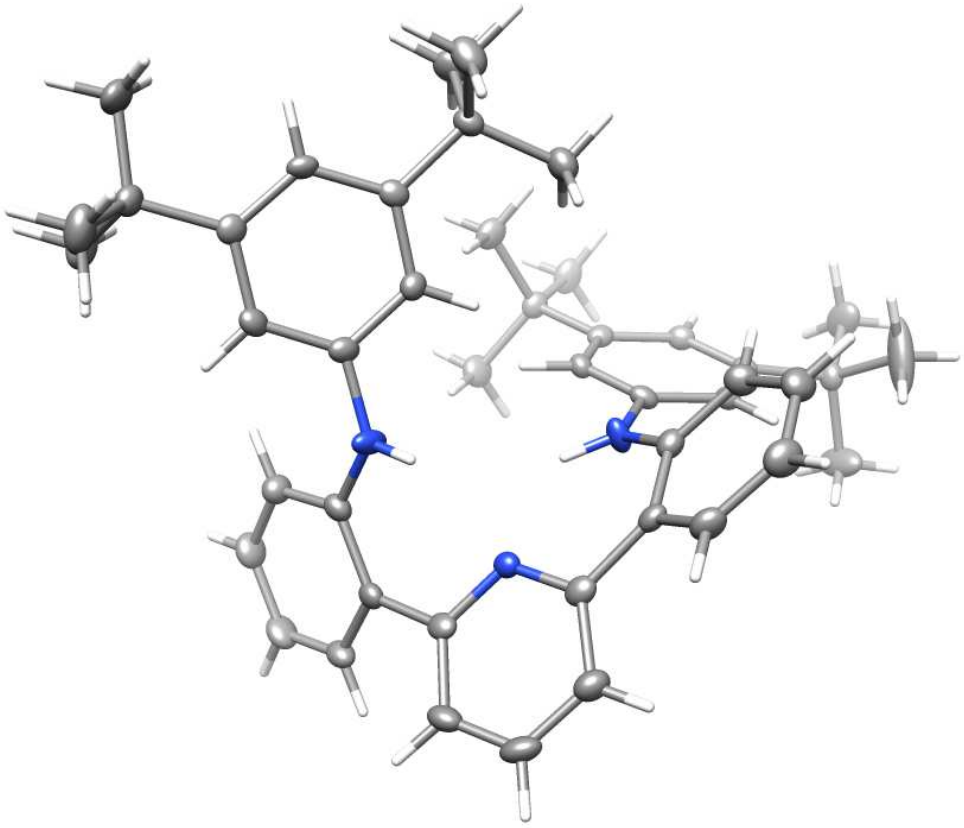

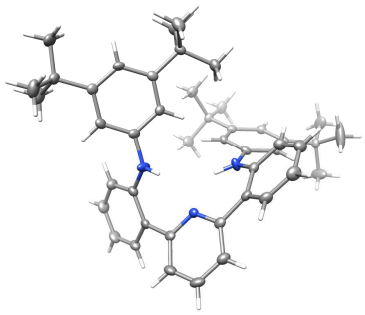

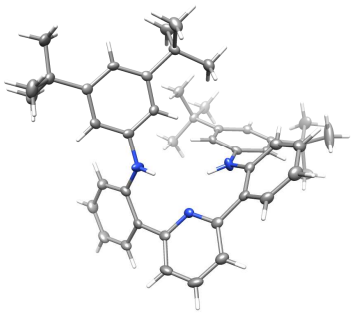

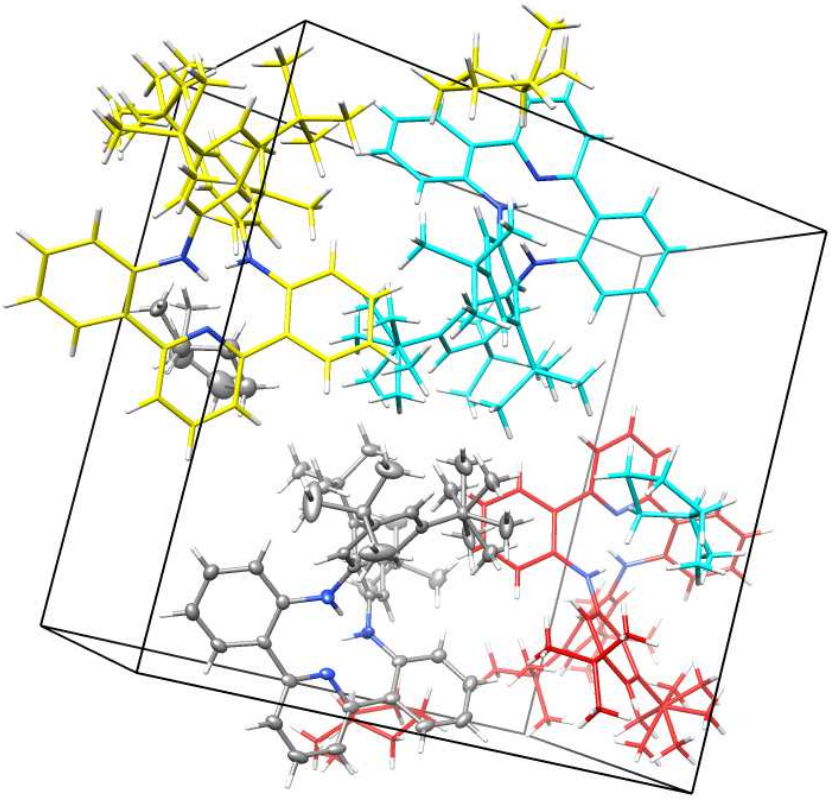

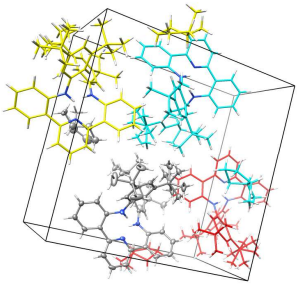

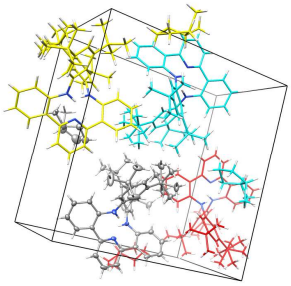

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for ECW18 (CCDC 793155). U(eq) is defined as the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U_{eq}	Occ
N(1)	3974(1)	8271(1)	4141(1)	22(1)	1
N(2)	3363(1)	9602(1)	4650(1)	19(1)	1
N(3)	2370(1)	8733(1)	5492(1)	25(1)	1
C(1)	4677(1)	7661(1)	4308(1)	18(1)	1
C(2)	4719(1)	7273(1)	4946(1)	18(1)	1
C(3)	5436(2)	6695(1)	5167(1)	18(1)	1
C(4)	6113(1)	6524(1)	4722(1)	19(1)	1
C(5)	6104(2)	6910(1)	4085(1)	18(1)	1
C(6)	5372(1)	7482(1)	3879(1)	18(1)	1
C(7)	3521(2)	8552(1)	3468(1)	20(1)	1
C(8)	3363(2)	8084(1)	2864(1)	24(1)	1
C(9)	2885(2)	8353(1)	2194(1)	28(1)	1
C(10)	2544(2)	9099(1)	2112(1)	29(1)	1
C(11)	2696(2)	9559(1)	2705(1)	25(1)	1
C(12)	3192(2)	9318(1)	3388(1)	19(1)	1
C(13)	3352(2)	9866(1)	3987(1)	21(1)	1
C(14)	3500(2)	10632(1)	3869(1)	27(1)	1
C(15)	3600(2)	11128(1)	4437(1)	31(1)	1
C(16)	3585(2)	10860(1)	5114(1)	27(1)	1
C(17)	3496(2)	10079(1)	5214(1)	21(1)	1
C(18)	3558(2)	9757(1)	5939(1)	20(1)	1
C(19)	4210(2)	10096(1)	6522(1)	26(1)	1
C(20)	4336(2)	9830(1)	7213(1)	30(1)	1
C(21)	3793(2)	9191(1)	7334(1)	30(1)	1
C(22)	3141(2)	8833(1)	6771(1)	26(1)	1
C(23)	3011(2)	9095(1)	6067(1)	22(1)	1
C(24)	1772(2)	8074(1)	5509(1)	19(1)	1
C(25)	1853(1)	7499(1)	5024(1)	19(1)	1
C(26)	1253(1)	6847(1)	4986(1)	18(1)	1
C(27)	575(1)	6789(1)	5453(1)	19(1)	1
C(28)	470(2)	7356(1)	5936(1)	20(1)	1
C(29)	1081(2)	8007(1)	5957(1)	21(1)	1
C(30)	5452(2)	6275(1)	5869(1)	20(1)	1
C(31A)	6273(2)	5639(2)	6012(1)	30(1)	0.849(3)
C(32A)	4393(2)	5904(2)	5835(1)	27(1)	0.849(3)
C(33A)	5693(2)	6833(2)	6488(1)	31(1)	0.849(3)
C(31B)	6545(11)	6330(10)	6405(8)	44(5)	0.151(3)
C(32B)	5142(13)	5441(9)	5736(8)	43(5)	0.151(3)
C(33B)	4651(12)	6629(9)	6323(8)	39(5)	0.151(3)
C(34)	6925(2)	6765(1)	3642(1)	22(1)	1
C(35)	7413(2)	5985(1)	3780(1)	44(1)	1
C(36)	6456(2)	6806(2)	2840(1)	79(1)	1
C(37)	7762(2)	7364(1)	3841(2)	59(1)	1
C(38)	1318(2)	6211(1)	4453(1)	21(1)	1
C(39)	1588(2)	5459(1)	4857(1)	32(1)	1
C(40)	2134(1)	6362(1)	4005(1)	29(1)	1
C(41)	260(1)	6124(1)	3931(1)	34(1)	1

C(42)	-307(2)	7315(1)	6430(1)	25(1)	1
C(43)	-857(2)	6548(1)	6377(1)	42(1)	1
C(44)	250(2)	7437(1)	7213(1)	40(1)	1
C(45)	-1133(2)	7936(1)	6206(1)	42(1)	1
C(51)	1385(2)	4424(1)	1794(1)	45(1)	1
C(52)	1090(2)	4984(1)	2341(1)	49(1)	1
C(53)	-141(2)	4988(2)	2108(2)	82(1)	1
C(54)	-426(2)	4637(2)	1296(2)	69(1)	1
C(55)	633(2)	4593(2)	1085(1)	45(1)	1
C(56A)	639(2)	4039(2)	498(1)	50(1)	0.886(3)
C(56B)	-745(12)	3769(10)	1281(10)	21(5)	0.114(3)

Table 3. Bond lengths [Å] and angles [°] for ECW18 (CCDC 793155).

N(1)-C(7)	1.388(2)	C(38)-C(41)	1.543(3)
N(1)-C(1)	1.414(2)	C(42)-C(43)	1.530(3)
N(2)-C(13)	1.348(2)	C(42)-C(44)	1.535(3)
N(2)-C(17)	1.350(2)	C(42)-C(45)	1.544(3)
N(3)-C(23)	1.391(2)	C(51)-C(55)	1.529(3)
N(3)-C(24)	1.413(2)	C(51)-C(52)	1.548(3)
C(1)-C(2)	1.389(3)	C(52)-C(53)	1.595(3)
C(1)-C(6)	1.392(2)	C(53)-C(54)	1.638(4)
C(2)-C(3)	1.396(3)	C(54)-C(56B)	1.590(17)
C(3)-C(4)	1.393(3)	C(54)-C(55)	1.541(3)
C(3)-C(30)	1.529(3)	C(55)-C(56A)	1.489(3)
C(4)-C(5)	1.391(3)		
C(5)-C(6)	1.397(3)	C(7)-N(1)-C(1)	127.91(18)
C(5)-C(34)	1.534(3)	C(13)-N(2)-C(17)	120.47(19)
C(7)-C(8)	1.398(3)	C(23)-N(3)-C(24)	127.63(18)
C(7)-C(12)	1.417(3)	C(2)-C(1)-C(6)	119.75(19)
C(8)-C(9)	1.386(3)	C(2)-C(1)-N(1)	118.37(18)
C(9)-C(10)	1.390(3)	C(6)-C(1)-N(1)	121.71(19)
C(10)-C(11)	1.373(3)	C(1)-C(2)-C(3)	121.53(19)
C(11)-C(12)	1.396(3)	C(4)-C(3)-C(2)	117.3(2)
C(12)-C(13)	1.479(3)	C(4)-C(3)-C(30)	122.72(19)
C(13)-C(14)	1.391(3)	C(2)-C(3)-C(30)	120.00(19)
C(14)-C(15)	1.379(3)	C(5)-C(4)-C(3)	122.7(2)
C(15)-C(16)	1.381(3)	C(4)-C(5)-C(6)	118.43(19)
C(16)-C(17)	1.399(3)	C(4)-C(5)-C(34)	121.66(19)
C(17)-C(18)	1.483(3)	C(6)-C(5)-C(34)	119.74(19)
C(18)-C(19)	1.390(3)	C(1)-C(6)-C(5)	120.3(2)
C(18)-C(23)	1.422(3)	N(1)-C(7)-C(8)	120.7(2)
C(19)-C(20)	1.376(3)	N(1)-C(7)-C(12)	120.2(2)
C(20)-C(21)	1.384(3)	C(8)-C(7)-C(12)	119.1(2)
C(21)-C(22)	1.381(3)	C(9)-C(8)-C(7)	121.1(2)
C(22)-C(23)	1.398(3)	C(8)-C(9)-C(10)	120.2(2)
C(24)-C(29)	1.385(3)	C(11)-C(10)-C(9)	118.7(2)
C(24)-C(25)	1.393(3)	C(10)-C(11)-C(12)	123.2(2)
C(25)-C(26)	1.393(3)	C(11)-C(12)-C(7)	117.7(2)
C(26)-C(27)	1.397(3)	C(11)-C(12)-C(13)	118.93(19)
C(26)-C(38)	1.530(3)	C(7)-C(12)-C(13)	123.4(2)
C(27)-C(28)	1.388(3)	N(2)-C(13)-C(14)	121.0(2)
C(28)-C(29)	1.399(3)	N(2)-C(13)-C(12)	118.10(19)
C(28)-C(42)	1.539(3)	C(14)-C(13)-C(12)	120.9(2)
C(30)-C(33A)	1.520(3)	C(15)-C(14)-C(13)	118.9(2)
C(30)-C(32B)	1.536(15)	C(14)-C(15)-C(16)	120.0(2)
C(30)-C(32A)	1.536(3)	C(15)-C(16)-C(17)	119.0(2)
C(30)-C(31A)	1.546(3)	N(2)-C(17)-C(16)	120.4(2)
C(30)-C(31B)	1.586(15)	N(2)-C(17)-C(18)	118.45(19)
C(30)-C(33B)	1.631(14)	C(16)-C(17)-C(18)	121.1(2)
C(34)-C(35)	1.520(3)	C(19)-C(18)-C(23)	117.9(2)
C(34)-C(37)	1.520(3)	C(19)-C(18)-C(17)	118.87(19)
C(34)-C(36)	1.527(3)	C(23)-C(18)-C(17)	123.18(19)
C(38)-C(40)	1.538(2)	C(20)-C(19)-C(18)	123.2(2)
C(38)-C(39)	1.538(3)	C(19)-C(20)-C(21)	118.6(2)

C(22)-C(21)-C(20)	120.2(2)	C(44)-C(42)-C(45)	109.46(19)
C(21)-C(22)-C(23)	121.7(2)	C(28)-C(42)-C(45)	108.84(18)
N(3)-C(23)-C(22)	122.41(19)	C(55)-C(51)-C(52)	105.2(2)
N(3)-C(23)-C(18)	119.2(2)	C(51)-C(52)-C(53)	101.8(2)
C(22)-C(23)-C(18)	118.3(2)	C(52)-C(53)-C(54)	106.1(2)
C(29)-C(24)-C(25)	120.24(19)	C(56B)-C(54)-C(55)	101.5(6)
C(29)-C(24)-N(3)	122.17(19)	C(56B)-C(54)-C(53)	112.8(7)
C(25)-C(24)-N(3)	117.49(18)	C(55)-C(54)-C(53)	103.2(2)
C(26)-C(25)-C(24)	120.82(19)	C(56A)-C(55)-C(51)	115.6(2)
C(25)-C(26)-C(27)	117.6(2)	C(56A)-C(55)-C(54)	112.4(2)
C(25)-C(26)-C(38)	121.92(19)	C(51)-C(55)-C(54)	103.5(2)
C(27)-C(26)-C(38)	120.45(19)		
C(28)-C(27)-C(26)	122.7(2)		
C(27)-C(28)-C(29)	118.16(19)		
C(27)-C(28)-C(42)	123.18(19)		
C(29)-C(28)-C(42)	118.63(19)		
C(24)-C(29)-C(28)	120.4(2)		
C(33A)-C(30)-C(3)	109.25(19)		
C(33A)-C(30)-C(32B)	139.0(6)		
C(3)-C(30)-C(32B)	111.5(6)		
C(33A)-C(30)-C(32A)	110.32(19)		
C(3)-C(30)-C(32A)	109.19(18)		
C(32B)-C(30)-C(32A)	51.3(6)		
C(33A)-C(30)-C(31A)	108.1(2)		
C(3)-C(30)-C(31A)	112.62(18)		
C(32B)-C(30)-C(31A)	59.0(6)		
C(32A)-C(30)-C(31A)	107.4(2)		
C(33A)-C(30)-C(31B)	56.5(6)		
C(3)-C(30)-C(31B)	112.1(6)		
C(32B)-C(30)-C(31B)	109.9(9)		
C(32A)-C(30)-C(31B)	138.7(6)		
C(31A)-C(30)-C(31B)	54.6(6)		
C(33A)-C(30)-C(33B)	52.4(6)		
C(3)-C(30)-C(33B)	113.1(5)		
C(32B)-C(30)-C(33B)	105.9(8)		
C(32A)-C(30)-C(33B)	59.7(6)		
C(31A)-C(30)-C(33B)	134.1(5)		
C(31B)-C(30)-C(33B)	103.9(8)		
C(35)-C(34)-C(37)	109.08(18)		
C(35)-C(34)-C(36)	106.4(2)		
C(37)-C(34)-C(36)	109.6(2)		
C(35)-C(34)-C(5)	112.41(18)		
C(37)-C(34)-C(5)	108.10(18)		
C(36)-C(34)-C(5)	111.23(18)		
C(26)-C(38)-C(40)	112.91(17)		
C(26)-C(38)-C(39)	109.86(17)		
C(40)-C(38)-C(39)	108.26(17)		
C(26)-C(38)-C(41)	109.30(17)		
C(40)-C(38)-C(41)	107.81(18)		
C(39)-C(38)-C(41)	108.60(17)		
C(43)-C(42)-C(44)	108.1(2)		
C(43)-C(42)-C(28)	111.93(19)		
C(44)-C(42)-C(28)	110.26(17)		
C(43)-C(42)-C(45)	108.19(18)		

Table 4. Anisotropic displacement parameters ($Å^2 x 10^4$) for ECW18 (CCDC 793155). 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}
N(1)	279(11)	223(11)	177(11)	-13(9)	77(9)	94(9)
N(2)	167(10)	156(10)	257(11)	-18(9)	96(8)	3(8)
N(3)	364(12)	197(11)	172(11)	20(9)	41(9)	-107(9)
C(1)	183(12)	147(12)	191(13)	-40(10)	7(10)	6(9)
C(2)	181(12)	190(13)	188(13)	-53(10)	65(10)	-16(10)
C(3)	161(11)	192(13)	178(12)	-11(10)	6(10)	-40(10)
C(4)	161(12)	182(13)	233(13)	-10(10)	28(10)	11(9)
C(5)	167(12)	179(13)	191(12)	-52(10)	38(10)	-31(10)
C(6)	222(12)	164(12)	160(12)	-18(10)	40(10)	-32(10)
C(7)	164(12)	228(13)	205(13)	16(11)	56(10)	-15(10)
C(8)	233(13)	208(13)	264(14)	16(11)	37(11)	19(10)
C(9)	316(14)	300(15)	224(14)	-32(12)	42(11)	-63(11)
C(10)	306(14)	303(15)	248(14)	69(12)	25(11)	-14(12)
C(11)	261(13)	190(13)	318(15)	67(12)	95(11)	18(11)
C(12)	165(12)	176(13)	246(13)	41(11)	95(10)	-6(10)
C(13)	148(12)	195(13)	300(14)	28(11)	85(10)	22(10)
C(14)	278(14)	219(14)	320(15)	53(12)	95(11)	-19(11)
C(15)	271(14)	165(13)	500(18)	37(13)	131(13)	-34(11)
C(16)	243(13)	183(13)	410(16)	-94(12)	113(12)	-40(11)
C(17)	131(12)	197(13)	326(15)	-42(11)	94(10)	-18(10)
C(18)	185(12)	177(13)	272(14)	-60(11)	107(10)	5(10)
C(19)	192(13)	239(14)	370(16)	-112(12)	114(11)	-52(10)
C(20)	243(13)	371(16)	286(15)	-137(13)	34(11)	-51(12)
C(21)	292(14)	378(16)	223(14)	-48(12)	44(11)	3(12)
C(22)	266(13)	260(14)	259(14)	-22(12)	83(11)	-39(11)
C(23)	196(12)	198(13)	258(14)	-64(11)	66(11)	7(10)
C(24)	215(12)	167(13)	181(12)	21(10)	19(10)	-24(10)
C(25)	175(12)	223(13)	178(12)	4(10)	47(10)	-15(10)
C(26)	169(12)	177(13)	176(12)	1(10)	-9(10)	19(10)
C(27)	192(12)	146(12)	234(13)	16(10)	30(10)	-34(10)
C(28)	195(12)	208(13)	179(12)	30(10)	22(10)	14(10)
C(29)	255(13)	191(13)	168(12)	-18(10)	40(10)	-7(10)
C(30)	178(12)	235(14)	196(13)	28(11)	36(10)	2(10)
C(31A)	278(17)	370(20)	265(18)	106(15)	75(14)	60(14)
C(32A)	256(16)	323(19)	239(17)	62(14)	49(13)	-23(14)
C(33A)	356(19)	372(19)	194(16)	-20(14)	45(14)	-41(15)
C(31B)	430(110)	690(150)	190(100)	20(100)	40(80)	170(100)
C(32B)	640(140)	410(120)	290(100)	110(90)	190(100)	140(100)
C(33B)	400(110)	600(140)	210(90)	190(90)	150(80)	260(100)
C(34)	196(12)	251(14)	213(13)	-19(11)	76(10)	10(10)
C(35)	479(16)	312(16)	650(20)	-17(15)	382(15)	66(13)
C(36)	560(20)	1570(40)	278(17)	60(20)	197(15)	490(20)
C(37)	454(17)	390(18)	1070(30)	-287(18)	518(18)	-168(14)
C(38)	183(12)	208(13)	245(13)	-53(11)	31(10)	-2(10)
C(39)	320(14)	214(14)	432(17)	-46(12)	95(12)	24(11)
C(40)	273(13)	309(15)	303(15)	-83(12)	97(11)	0(11)
C(41)	251(13)	441(17)	314(15)	-141(13)	23(11)	12(12)

C(42)	255(13)	275(14)	242(14)	-12(11)	105(11)	-41(11)
C(43)	510(17)	398(17)	466(18)	-83(14)	327(14)	-178(14)
C(44)	386(15)	600(20)	248(15)	-41(14)	147(12)	-95(14)
C(45)	375(16)	498(19)	467(18)	39(15)	246(14)	73(13)
C(51)	457(17)	473(19)	442(18)	-6(15)	157(14)	14(14)
C(52)	549(19)	490(20)	436(18)	2(16)	119(15)	-72(15)
C(53)	640(20)	860(30)	990(30)	170(20)	250(20)	51(19)
C(54)	383(18)	970(30)	720(20)	20(20)	115(17)	81(18)
C(55)	420(17)	467(19)	433(18)	1(15)	35(14)	-28(14)
C(56A)	640(20)	450(20)	331(19)	-162(17)	-92(16)	186(17)

Table 5. Hydrogen bonds for ECW18 (CCDC 793155) [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
N(1)-H(1)...N(2)	0.88	2.06	2.730(2)	131.7
N(3)-H(3)...N(2)	0.88	2.08	2.750(2)	132.3