Electronic Supplementary Information (ESI)

Effect of Ligand Substituents in Olefin Polymerisation by Half-Titanocenes Containing Monoanionic Iminoimidazolidide Ligands - MAO Catalyst Systems

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Electronic Supplementary Material (ESI) for Dalton Transactions

X-ray Structure Report for $CpTiCl_2[1,3\text{-}(2,6\text{-}Me_2C_6H_3)_2(CH_2N)_2C{=}N]$ (**1b**)

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Experimental

Data Collection

A yellow block crystal of $C_{24}H_{27}Cl_2N_3Ti$ having approximate dimensions of 0.450 x 0.450 x 0.120 mm was mounted on a glass fiber. All measurements were made on a Rigaku R-AXIS RAPID diffractometer using graphite monochromated Mo-Kα radiation.

The crystal-to-detector distance was 127.40 mm.

Cell constants and an orientation matrix for data collection corresponded to a primitive triclinic cell with dimensions:

a	=	8.2728(3) Å	α	=	80.281(1)^o
b	=	9.4651(4) Å	β	=	82.631(1)^o
c	=	16.8992(6) Å	γ	=	62.550(1)^o
V	=	1155.48(7) Å^3			

For Z = 2 and F.W. = 476.30, the calculated density is 1.369 g/cm^3. Based on a statistical analysis of intensity distribution, and the successful solution and refinement of the structure, the space group was determined to be:

P-1 (#2)

The data were collected at a temperature of -150 $\pm$ 1^{o}C to a maximum 2θ value of 54.9^o. A total of 74 oscillation images were collected. A sweep of data was done using ω scans from 130.0 to 190.0^o in 3.0^o step, at χ=45.0^o and ϕ = 0.0^o. The exposure rate was 70.0 [sec./^{o}]. A second sweep was performed using ω scans from 0.0 to 162.0^o in 3.0^o step, at χ=45.0^o and ϕ = 180.0^o. The exposure rate was 70.0 [sec./^{o}]. The crystal-to-detector distance was 127.40 mm. Readout was performed in the 0.100 mm pixel mode.

Data Reduction

Of the 11559 reflections that were collected, 5250 were unique (R_{int} = 0.0284); equivalent reflections were merged.

The linear absorption coefficient, μ, for Mo-Kα radiation is 6.173 cm^{-1}. An empirical absorption correction was applied which resulted in transmission factors ranging from 0.693 to 0.929. The data were corrected for Lorentz and polarization effects.

Structure Solution and Refinement

The structure was solved by direct methods[1] and expanded using Fourier techniques. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were refined using the riding model. The final cycle of full-matrix least-squares refinement[2] on F^2 was based on 5250 observed reflections and 295 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R1 = \Sigma \, ||Fo| - |Fc|| \, / \, \Sigma \, |Fo| = 0.0333$$

$$wR2 = [\Sigma \, (w \, (Fo^2 - Fc^2)^2) / \Sigma \, w(Fo^2)^2]^{1/2} = 0.0997$$

The standard deviation of an observation of unit weight[3] was 1.11. Unit weights were used. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.38 and -0.52 $e^-/Å^3$, respectively.

Neutral atom scattering factors were taken from Cromer and Waber[4]. Anomalous dispersion effects were included in Fcalc[5]; the values for Δf' and Δf'' were those of Creagh and McAuley[6]. The values for the mass attenuation coefficients are those of Creagh and Hubbell[7]. All calculations were performed using the CrystalStructure[8] crystallographic software package except for refinement, which was performed using SHELXL-97[9].

References

(1) SIR2008: M.C. Burla, R. Caliandro, M. Camalli, B. Carrozzini, G.L. Cascarano, L. De Caro, C. Giacovazzo, G. Polidori, D. Siliqi, R. Spagna (2007)

(2) Least Squares function minimized: (SHELXL97)

$\Sigma w(F_o^2-F_c^2)^2$ where w = Least Squares weights.

(3) Standard deviation of an observation of unit weight:

$[\Sigma w(F_o^2-F_c^2)^2/(N_o-N_v)]^{1/2}$

where: N_o = number of observations
N_v = number of variables

(4) Cromer, D. T. & Waber, J. T.; "International Tables for X-ray Crystallography", Vol. IV, The Kynoch Press, Birmingham, England, Table 2.2 A (1974).

(5) Ibers, J. A. & Hamilton, W. C.; Acta Crystallogr., 17, 781 (1964).

(6) Creagh, D. C. & McAuley, W.J .; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.6.8, pages 219-222 (1992).

(7) Creagh, D. C. & Hubbell, J.H..; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.4.3, pages 200-206 (1992).

(8) CrystalStructure 4.0: Crystal Structure Analysis Package, Rigaku Corporation (2000-2010). Tokyo 196-8666, Japan.

(9) SHELX97: Sheldrick, G.M. (2008). Acta Cryst. A64, 112-122.

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	$C_{24}H_{27}Cl_2N_3Ti$
Formula Weight	476.30
Crystal Color, Habit	yellow, block
Crystal Dimensions	0.450 X 0.450 X 0.120 mm
Crystal System	triclinic
Lattice Type	Primitive
Lattice Parameters	a = 8.2728(3) Å b = 9.4651(4) Å c = 16.8992(6) Å α = 80.281(1) ^{o} β = 82.631(1) ^{o} γ = 62.550(1) ^{o} V = 1155.48(7) Å^3
Space Group	P-1 (#2)
Z value	2
D_{calc}	1.369 g/cm^3
F_{000}	496.00
μ(MoKα)	6.173 cm^{-1}

B. Intensity Measurements

Diffractometer	R-AXIS RAPID
Radiation	MoKα (λ = 0.71075 Å) graphite monochromated
Voltage, Current	50kV, 30mA
Temperature	-150.0^{o}C
Detector Aperture	280 x 256 mm
Data Images	74 exposures
ω oscillation Range (χ=45.0, ϕ=0.0)	130.0 - 190.0^{o}
Exposure Rate	70.0 sec./^{o}
ω oscillation Range (χ=45.0, ϕ=180.0)	0.0 - 162.0^{o}
Exposure Rate	70.0 sec./^{o}
Detector Position	127.40 mm
Pixel Size	0.100 mm
$2\theta_{max}$	54.9^{o}
No. of Reflections Measured	Total: 11559 Unique: 5250 (R_{int} = 0.0284)
Corrections	Lorentz-polarization Absorption (trans. factors: 0.693 - 0.929)

C. Structure Solution and Refinement

Structure Solution	Direct Methods
Refinement	Full-matrix least-squares on F^2
Function Minimized	Σ w $(Fo^2 - Fc^2)^2$
Least Squares Weights	$w = 1/ [\sigma^2(Fo^2) + (0.0452 \cdot P)^2 + 0.7158 \cdot P]$ where $P = (Max(Fo^2,0) + 2Fc^2)/3$
$2\theta_{max}$ cutoff	54.9^o
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (All reflections)	5250
No. Variables	295
Reflection/Parameter Ratio	17.80
Residuals: R1 (I>2.00σ(I))	0.0333
Residuals: R (All reflections)	0.0429
Residuals: wR2 (All reflections)	0.0997
Goodness of Fit Indicator	1.112
Max Shift/Error in Final Cycle	0.001
Maximum peak in Final Diff. Map	0.38 $e^-/Å^3$
Minimum peak in Final Diff. Map	-0.52 $e^-/Å^3$

Table 1. Atomic coordinates and B_{iso}/B_{eq}

atom	x	y	z	B_{eq}
Ti1	0.33380(4)	-0.13443(4)	0.77131(2)	1.349(7)
Cl1	0.62540(6)	-0.18322(6)	0.79403(3)	1.94(1)
Cl2	0.39779(7)	-0.30405(6)	0.67574(3)	2.33(1)
N1	0.2446(2)	0.0649(2)	0.71980(9)	1.43(3)
N2	0.1296(3)	0.3296(2)	0.75414(9)	1.63(3)
N3	0.0901(3)	0.3030(2)	0.63220(9)	1.65(3)
C1	0.3287(3)	-0.2709(3)	0.9046(2)	2.26(4)
C2	0.2280(3)	-0.1038(3)	0.9067(1)	2.13(4)
C3	0.0752(3)	-0.0482(3)	0.8615(2)	2.26(4)
C4	0.0833(3)	-0.1802(3)	0.8290(2)	2.34(4)
C5	0.2390(4)	-0.3172(3)	0.8572(2)	2.44(4)
C6	0.1605(3)	0.2212(2)	0.7033(1)	1.28(3)
C7	0.0566(3)	0.4940(3)	0.7126(1)	1.95(3)
C8	-0.0066(3)	0.4765(3)	0.6351(2)	2.15(4)
C9	0.1961(3)	0.2914(2)	0.8329(1)	1.60(3)
C10	0.3817(3)	0.2392(3)	0.8413(1)	1.86(3)
C11	0.4419(3)	0.2036(3)	0.9190(2)	2.48(4)
C12	0.3198(4)	0.2235(3)	0.9853(2)	2.77(4)
C13	0.1371(4)	0.2809(3)	0.9757(2)	2.68(4)
C14	0.0697(3)	0.3163(3)	0.8991(1)	2.05(4)
C15	0.5124(3)	0.2255(3)	0.7688(2)	2.37(4)
C16	-0.1317(3)	0.3817(3)	0.8901(2)	2.86(4)
C17	0.0999(3)	0.2376(2)	0.5601(1)	1.53(3)
C18	-0.0521(3)	0.2265(3)	0.5411(2)	2.12(4)
C19	-0.0455(4)	0.1783(3)	0.4666(2)	2.77(4)
C20	0.1064(4)	0.1429(3)	0.4144(2)	2.90(5)
C21	0.2560(4)	0.1547(3)	0.4348(2)	2.60(4)
C22	0.2545(3)	0.2044(3)	0.5079(1)	1.92(3)
C23	-0.2166(4)	0.2639(4)	0.5989(2)	3.45(5)
C24	0.4088(3)	0.2306(3)	0.5292(2)	2.95(4)

$B_{eq} = 8/3\ \pi^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos\gamma + 2U_{13}(aa^*cc^*)\cos\beta + 2U_{23}(bb^*cc^*)\cos\alpha)$

Table 2. Atomic coordinates and B_{iso} involving hydrogen atoms

atom	x	y	z	B_{iso}
H1	0.4451	-0.3404	0.9267	3.22
H2	0.2638	-0.0361	0.9290	2.90
H3	-0.0162	0.0580	0.8536	3.19
H4	0.0066	-0.1738	0.7965	2.61
H5	0.2832	-0.4228	0.8449	3.26
H6	0.1521	0.5313	0.7009	2.34
H7	-0.0465	0.5695	0.7445	2.34
H8	-0.1405	0.5157	0.6379	2.58
H9	0.0289	0.5355	0.5877	2.58
H10	0.5678	0.1652	0.9264	2.97
H11	0.3628	0.1973	1.0378	3.33
H12	0.0547	0.2969	1.0218	3.22
H13	0.4703	0.3293	0.7349	2.84
H14	0.5176	0.1445	0.7379	2.84
H15	0.6341	0.1936	0.7863	2.84
H16	-0.1511	0.3516	0.8408	3.43
H17	-0.1898	0.4989	0.8873	3.43
H18	-0.1854	0.3372	0.9365	3.43
H19	-0.1473	0.1698	0.4517	3.32
H20	0.1084	0.1101	0.3641	3.48
H21	0.3606	0.1287	0.3985	3.12
H22	-0.2818	0.2052	0.5889	4.13
H23	-0.2977	0.3795	0.5914	4.13
H24	-0.1774	0.2315	0.6542	4.13
H25	0.3663	0.3451	0.5317	3.54
H26	0.5093	0.1939	0.4881	3.54
H27	0.4514	0.1699	0.5816	3.54

Table 3. Anisotropic displacement parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Ti1	0.0197(2)	0.0169(2)	0.0134(2)	-0.0074(2)	-0.0020(1)	-0.0003(1)
Cl1	0.0186(3)	0.0287(3)	0.0219(3)	-0.0066(2)	-0.0047(2)	-0.0011(2)
Cl2	0.0422(3)	0.0295(3)	0.0210(3)	-0.0186(3)	0.0012(2)	-0.0091(2)
N1	0.0205(8)	0.0195(8)	0.0138(7)	-0.0087(7)	-0.0039(6)	0.0003(6)
N2	0.0286(9)	0.0144(7)	0.0147(8)	-0.0061(7)	-0.0049(6)	0.0009(6)
N3	0.0284(9)	0.0157(8)	0.0152(8)	-0.0066(7)	-0.0072(6)	0.0018(6)
C1	0.034(1)	0.030(1)	0.0167(9)	-0.013(1)	-0.0003(8)	0.0053(8)
C2	0.036(2)	0.031(1)	0.0163(9)	-0.018(1)	0.0048(8)	-0.0047(8)
C3	0.027(1)	0.027(1)	0.026(1)	-0.0094(9)	0.0062(8)	-0.0017(8)
C4	0.030(1)	0.045(2)	0.024(1)	-0.027(1)	0.0018(8)	-0.0006(9)
C5	0.046(2)	0.028(1)	0.024(1)	-0.024(1)	0.0089(9)	-0.0038(8)
C6	0.0148(8)	0.0202(9)	0.0134(8)	-0.0081(7)	-0.0016(6)	0.0003(7)
C7	0.034(1)	0.0160(9)	0.0197(9)	-0.0073(8)	-0.0049(8)	0.0010(7)
C8	0.037(1)	0.0165(9)	0.022(1)	-0.0069(9)	-0.0084(8)	0.0019(7)
C9	0.030(1)	0.0172(9)	0.0144(9)	-0.0105(8)	-0.0033(7)	-0.0013(7)
C10	0.029(1)	0.0210(9)	0.0205(9)	-0.0111(8)	-0.0038(8)	-0.0003(7)
C11	0.039(2)	0.031(1)	0.027(1)	-0.017(1)	-0.0129(9)	-0.0010(9)
C12	0.056(2)	0.033(2)	0.018(1)	-0.019(1)	-0.013(1)	-0.0014(8)
C13	0.053(2)	0.029(1)	0.016(1)	-0.016(1)	0.0050(9)	-0.0040(8)
C14	0.034(1)	0.020(1)	0.020(1)	-0.0103(9)	0.0015(8)	-0.0009(7)
C15	0.027(1)	0.032(1)	0.030(1)	-0.0138(9)	-0.0013(8)	0.0001(9)
C16	0.032(2)	0.032(2)	0.034(2)	-0.009(1)	0.0066(9)	-0.0007(9)
C17	0.0258(9)	0.0181(9)	0.0126(8)	-0.0090(8)	-0.0061(7)	0.0029(7)
C18	0.032(1)	0.026(1)	0.025(1)	-0.0161(9)	-0.0060(8)	0.0029(8)
C19	0.052(2)	0.033(2)	0.031(2)	-0.026(1)	-0.020(1)	0.0039(9)
C20	0.069(2)	0.025(1)	0.018(1)	-0.022(1)	-0.010(1)	0.0008(8)
C21	0.046(2)	0.020(1)	0.021(1)	-0.007(1)	0.0045(9)	0.0005(8)
C22	0.025(1)	0.0190(9)	0.021(1)	-0.0054(8)	-0.0032(8)	0.0037(7)
C23	0.036(2)	0.052(2)	0.051(2)	-0.028(2)	0.004(1)	-0.008(2)
C24	0.025(1)	0.039(2)	0.041(2)	-0.012(1)	-0.0008(9)	0.001(1)

The general temperature factor expression: $\exp(-2\pi^2(a^{*2}U_{11}h^2 + b^{*2}U_{22}k^2 + c^{*2}U_{33}l^2 + 2a^*b^*U_{12}hk + 2a^*c^*U_{13}hl + 2b^*c^*U_{23}kl))$

Table 4. Bond lengths (Å)

atom	atom	distance	atom	atom	distance
Ti1	Cl1	2.3088(7)	Ti1	Cl2	2.3106(7)
Ti1	N1	1.7860(15)	Ti1	C1	2.406(2)
Ti1	C2	2.3586(19)	Ti1	C3	2.354(2)
Ti1	C4	2.367(3)	Ti1	C5	2.414(3)
N1	C6	1.308(3)	N2	C6	1.362(3)
N2	C7	1.468(3)	N2	C9	1.430(3)
N3	C6	1.353(3)	N3	C8	1.465(3)
N3	C17	1.434(3)	C1	C2	1.412(3)
C1	C5	1.398(4)	C2	C3	1.399(4)
C3	C4	1.419(4)	C4	C5	1.406(3)
C7	C8	1.531(4)	C9	C10	1.398(3)
C9	C14	1.400(3)	C10	C11	1.397(3)
C10	C15	1.507(3)	C11	C12	1.385(3)
C12	C13	1.372(4)	C13	C14	1.403(3)
C14	C16	1.505(4)	C17	C18	1.391(4)
C17	C22	1.395(3)	C18	C19	1.395(4)
C18	C23	1.504(4)	C19	C20	1.377(4)
C20	C21	1.382(5)	C21	C22	1.391(4)
C22	C24	1.504(4)			

Table 5. Bond lengths involving hydrogens (Å)

atom	atom	distance	atom	atom	distance
C1	H1	0.962	C2	H2	0.962
C3	H3	0.938	C4	H4	0.866
C5	H5	0.943	C7	H6	0.990
C7	H7	0.990	C8	H8	0.990
C8	H9	0.990	C11	H10	0.950
C12	H11	0.950	C13	H12	0.950
C15	H13	0.980	C15	H14	0.980
C15	H15	0.980	C16	H16	0.980
C16	H17	0.980	C16	H18	0.980
C19	H19	0.950	C20	H20	0.950
C21	H21	0.950	C23	H22	0.980
C23	H23	0.980	C23	H24	0.980
C24	H25	0.980	C24	H26	0.980
C24	H27	0.980			

Table 6. Bond angles (°)

atom	atom	atom	angle	atom	atom	atom	angle
Cl1	Ti1	Cl2	100.27(3)	Cl1	Ti1	N1	101.84(7)
Cl1	Ti1	C1	86.21(7)	Cl1	Ti1	C2	93.72(7)
Cl1	Ti1	C3	127.25(7)	Cl1	Ti1	C4	143.13(6)
Cl1	Ti1	C5	112.22(6)	Cl2	Ti1	N1	106.80(6)
Cl2	Ti1	C1	110.72(7)	Cl2	Ti1	C2	141.55(7)
Cl2	Ti1	C3	126.19(8)	Cl2	Ti1	C4	92.07(7)
Cl2	Ti1	C5	84.69(6)	N1	Ti1	C1	139.47(7)
N1	Ti1	C2	105.02(7)	N1	Ti1	C3	88.45(7)
N1	Ti1	C4	107.62(8)	N1	Ti1	C5	141.56(8)
C1	Ti1	C2	34.46(7)	C1	Ti1	C3	57.06(7)
C1	Ti1	C4	56.96(9)	C1	Ti1	C5	33.73(10)
C2	Ti1	C3	34.54(8)	C2	Ti1	C4	57.74(10)
C2	Ti1	C5	56.89(9)	C3	Ti1	C4	34.99(10)
C3	Ti1	C5	57.02(8)	C4	Ti1	C5	34.20(7)
Ti1	N1	C6	163.23(14)	C6	N2	C7	111.70(15)
C6	N2	C9	125.44(14)	C7	N2	C9	121.42(18)
C6	N3	C8	112.08(17)	C6	N3	C17	127.34(15)
C8	N3	C17	120.57(14)	Ti1	C1	C2	70.95(11)
Ti1	C1	C5	73.45(13)	C2	C1	C5	108.03(19)
Ti1	C2	C1	74.60(12)	Ti1	C2	C3	72.55(12)
C1	C2	C3	108.0(3)	Ti1	C3	C2	72.92(12)
Ti1	C3	C4	73.00(13)	C2	C3	C4	108.11(18)
Ti1	C4	C3	72.01(15)	Ti1	C4	C5	74.73(16)
C3	C4	C5	107.3(3)	Ti1	C5	C1	72.82(15)
Ti1	C5	C4	71.08(15)	C1	C5	C4	108.5(3)
N1	C6	N2	126.13(16)	N1	C6	N3	125.64(19)
N2	C6	N3	108.23(15)	N2	C7	C8	102.13(18)
N3	C8	C7	102.52(15)	N2	C9	C10	119.42(16)
N2	C9	C14	118.23(18)	C10	C9	C14	122.25(18)
C9	C10	C11	117.95(18)	C9	C10	C15	121.15(18)
C11	C10	C15	120.9(2)	C10	C11	C12	120.6(3)
C11	C12	C13	120.5(2)	C12	C13	C14	121.2(2)
C9	C14	C13	117.4(2)	C9	C14	C16	122.49(19)
C13	C14	C16	120.10(19)	N3	C17	C18	118.71(17)
N3	C17	C22	118.2(2)	C18	C17	C22	122.72(19)
C17	C18	C19	117.4(2)	C17	C18	C23	121.1(3)
C19	C18	C23	121.5(3)	C18	C19	C20	121.0(3)

Table 6. Bond angles (°) (continued)

atom	atom	atom	angle	atom	atom	atom	angle
C19	C20	C21	120.6(3)	C20	C21	C22	120.4(2)
C17	C22	C21	117.9(3)	C17	C22	C24	120.3(2)
C21	C22	C24	121.8(2)				

Table 7. Bond angles involving hydrogens (°)

atom	atom	atom	angle	atom	atom	atom	angle
Ti1	C1	H1	116.5	C2	C1	H1	126.8
C5	C1	H1	124.9	Ti1	C2	H2	112.8
C1	C2	H2	126.9	C3	C2	H2	124.7
Ti1	C3	H3	119.5	C2	C3	H3	126.1
C4	C3	H3	125.8	Ti1	C4	H4	117.2
C3	C4	H4	124.8	C5	C4	H4	127.9
Ti1	C5	H5	119.3	C1	C5	H5	123.8
C4	C5	H5	127.6	N2	C7	H6	111.3
N2	C7	H7	111.3	C8	C7	H6	111.3
C8	C7	H7	111.3	H6	C7	H7	109.2
N3	C8	H8	111.3	N3	C8	H9	111.3
C7	C8	H8	111.3	C7	C8	H9	111.3
H8	C8	H9	109.2	C10	C11	H10	119.7
C12	C11	H10	119.7	C11	C12	H11	119.7
C13	C12	H11	119.7	C12	C13	H12	119.4
C14	C13	H12	119.4	C10	C15	H13	109.5
C10	C15	H14	109.5	C10	C15	H15	109.5
H13	C15	H14	109.5	H13	C15	H15	109.5
H14	C15	H15	109.5	C14	C16	H16	109.5
C14	C16	H17	109.5	C14	C16	H18	109.5
H16	C16	H17	109.5	H16	C16	H18	109.5
H17	C16	H18	109.5	C18	C19	H19	119.5
C20	C19	H19	119.5	C19	C20	H20	119.7
C21	C20	H20	119.7	C20	C21	H21	119.8
C22	C21	H21	119.8	C18	C23	H22	109.5
C18	C23	H23	109.5	C18	C23	H24	109.5
H22	C23	H23	109.5	H22	C23	H24	109.5
H23	C23	H24	109.5	C22	C24	H25	109.5
C22	C24	H26	109.5	C22	C24	H27	109.5
H25	C24	H26	109.5	H25	C24	H27	109.5
H26	C24	H27	109.5				

Table 8. Torsion Angles(°)
(Those having bond angles > 160 or < 20 degrees are excluded.)

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
Cl1	Ti1	C1	C2	102.21(11)	Cl1	Ti1	C1	C5	-141.42(7)
Cl1	Ti1	C2	C1	-77.77(10)	Cl1	Ti1	C2	C3	167.26(9)
Cl1	Ti1	C3	C2	-16.05(14)	Cl1	Ti1	C3	C4	-131.77(7)
Cl1	Ti1	C4	C3	81.64(11)	Cl1	Ti1	C4	C5	-32.79(17)
Cl1	Ti1	C5	C1	42.23(9)	Cl1	Ti1	C5	C4	159.45(7)
Cl2	Ti1	C1	C2	-158.24(8)	Cl2	Ti1	C1	C5	-41.86(9)
Cl2	Ti1	C2	C1	33.90(16)	Cl2	Ti1	C2	C3	-81.07(10)
Cl2	Ti1	C3	C2	130.44(9)	Cl2	Ti1	C3	C4	14.73(10)
Cl2	Ti1	C4	C3	-168.15(7)	Cl2	Ti1	C4	C5	77.42(10)
Cl2	Ti1	C5	C1	141.18(8)	Cl2	Ti1	C5	C4	-101.60(9)
N1	Ti1	C1	C2	-1.6(2)	N1	Ti1	C1	C5	114.76(14)
N1	Ti1	C2	C1	178.91(11)	N1	Ti1	C2	C3	63.94(13)
N1	Ti1	C3	C2	-119.78(12)	N1	Ti1	C3	C4	124.50(10)
N1	Ti1	C4	C3	-59.80(11)	N1	Ti1	C4	C5	-174.23(10)
N1	Ti1	C5	C1	-108.35(14)	N1	Ti1	C5	C4	8.87(19)
C1	Ti1	C2	C1	0.00(12)	C1	Ti1	C2	C3	-115.0(2)
C2	Ti1	C1	C2	-0.00(12)	C2	Ti1	C1	C5	116.37(18)
C1	Ti1	C3	C2	37.67(10)	C1	Ti1	C3	C4	-78.04(11)
C3	Ti1	C1	C2	-37.76(11)	C3	Ti1	C1	C5	78.61(12)
C1	Ti1	C4	C3	78.34(11)	C1	Ti1	C4	C5	-36.09(9)
C4	Ti1	C1	C2	-79.78(11)	C4	Ti1	C1	C5	36.60(9)
C1	Ti1	C5	C1	0.00(8)	C1	Ti1	C5	C4	117.22(15)
C5	Ti1	C1	C2	-116.37(16)	C5	Ti1	C1	C5	0.00(9)
C2	Ti1	C3	C2	0.00(11)	C2	Ti1	C3	C4	-115.72(17)
C3	Ti1	C2	C1	115.0(2)	C3	Ti1	C2	C3	0.00(12)
C2	Ti1	C4	C3	37.16(8)	C2	Ti1	C4	C5	-77.27(11)
C4	Ti1	C2	C1	77.31(12)	C4	Ti1	C2	C3	-37.66(9)
C2	Ti1	C5	C1	-37.24(8)	C2	Ti1	C5	C4	79.98(10)
C5	Ti1	C2	C1	36.43(10)	C5	Ti1	C2	C3	-78.54(12)
C3	Ti1	C4	C3	0.00(8)	C3	Ti1	C4	C5	-114.43(16)
C4	Ti1	C3	C2	115.72(15)	C4	Ti1	C3	C4	0.00(9)
C3	Ti1	C5	C1	-78.72(11)	C3	Ti1	C5	C4	38.50(9)
C5	Ti1	C3	C2	78.13(11)	C5	Ti1	C3	C4	-37.59(9)
C4	Ti1	C5	C1	-117.22(17)	C4	Ti1	C5	C4	0.00(12)
C5	Ti1	C4	C3	114.43(18)	C5	Ti1	C4	C5	0.00(11)
C6	N2	C7	C8	16.5(2)	C7	N2	C6	N1	171.36(18)
C7	N2	C6	N3	-8.3(3)	C6	N2	C9	C10	72.0(3)

Table 8. Torsion angles (°) (continued)

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
C6	N2	C9	C14	-111.4(2)	C9	N2	C6	N1	5.0(4)
C9	N2	C6	N3	-174.62(18)	C7	N2	C9	C10	-93.1(2)
C7	N2	C9	C14	83.4(3)	C9	N2	C7	C8	-176.54(15)
C6	N3	C8	C7	14.2(3)	C8	N3	C6	N1	175.95(18)
C8	N3	C6	N2	-4.4(3)	C6	N3	C17	C18	101.7(2)
C6	N3	C17	C22	-85.2(3)	C17	N3	C6	N1	-3.2(4)
C17	N3	C6	N2	176.43(18)	C8	N3	C17	C18	-77.4(3)
C8	N3	C17	C22	95.7(2)	C17	N3	C8	C7	-166.56(17)
Ti1	C1	C2	Ti1	0.0	Ti1	C1	C2	C3	65.39(12)
Ti1	C1	C5	Ti1	0.0	Ti1	C1	C5	C4	-62.52(13)
C2	C1	C5	Ti1	62.94(16)	C2	C1	C5	C4	0.4(3)
C5	C1	C2	Ti1	-64.57(16)	C5	C1	C2	C3	0.8(3)
Ti1	C2	C3	Ti1	0.000(10)	Ti1	C2	C3	C4	65.02(12)
C1	C2	C3	Ti1	-66.75(15)	C1	C2	C3	C4	-1.7(3)
Ti1	C3	C4	Ti1	0.0	Ti1	C3	C4	C5	66.95(13)
C2	C3	C4	Ti1	-64.97(16)	C2	C3	C4	C5	2.0(3)
Ti1	C4	C5	Ti1	0.0	Ti1	C4	C5	C1	63.64(14)
C3	C4	C5	Ti1	-65.12(17)	C3	C4	C5	C1	-1.5(3)
N2	C7	C8	N3	-17.35(19)	N2	C9	C10	C11	179.76(17)
N2	C9	C10	C15	1.1(3)	N2	C9	C14	C13	-178.88(17)
N2	C9	C14	C16	-0.2(3)	C10	C9	C14	C13	-2.4(3)
C10	C9	C14	C16	176.27(18)	C14	C9	C10	C11	3.3(3)
C14	C9	C10	C15	-175.29(18)	C9	C10	C11	C12	-1.6(4)
C15	C10	C11	C12	177.0(2)	C10	C11	C12	C13	-0.9(4)
C11	C12	C13	C14	1.9(4)	C12	C13	C14	C9	-0.2(4)
C12	C13	C14	C16	-179.0(2)	N3	C17	C18	C19	173.45(14)
N3	C17	C18	C23	-7.0(3)	N3	C17	C22	C21	-174.34(13)
N3	C17	C22	C24	2.5(3)	C18	C17	C22	C21	-1.5(3)
C18	C17	C22	C24	175.32(15)	C22	C17	C18	C19	0.7(3)
C22	C17	C18	C23	-179.76(15)	C17	C18	C19	C20	0.2(3)
C23	C18	C19	C20	-179.39(18)	C18	C19	C20	C21	-0.1(3)
C19	C20	C21	C22	-0.8(3)	C20	C21	C22	C17	1.5(3)
C20	C21	C22	C24	-175.25(16)					

Table 9. Intramolecular contacts less than 3.60 Å

atom	atom	distance	atom	atom	distance
Cl1	C15	3.497(3)	N1	C9	2.966(3)
N1	C10	3.418(4)	N1	C15	3.449(4)
N1	C17	2.987(3)	N2	C15	2.881(3)
N2	C16	2.885(3)	N2	C17	3.597(3)
N3	C9	3.582(3)	N3	C23	2.862(4)
N3	C24	2.831(3)	C2	C14	3.552(3)
C3	C14	3.591(4)	C6	C10	3.214(3)
C6	C14	3.473(3)	C6	C15	3.263(4)
C6	C18	3.424(3)	C6	C22	3.307(3)
C6	C24	3.379(3)	C7	C10	3.395(3)
C7	C14	3.302(3)	C7	C15	3.584(3)
C7	C16	3.437(3)	C8	C18	3.242(4)
C8	C22	3.387(3)	C8	C23	3.369(5)
C8	C24	3.590(3)	C9	C12	2.758(3)
C10	C13	2.793(3)	C11	C14	2.801(4)
C17	C20	2.744(3)	C18	C21	2.798(3)
C19	C22	2.788(4)			

Table 10. Intramolecular contacts less than 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
Ti1	H14	3.546	Cl1	H1	3.086
Cl1	H2	3.402	Cl1	H14	2.826
Cl1	H15	3.581	Cl2	H4	3.411
Cl2	H5	3.087	N1	H2	3.507
N1	H3	2.933	N1	H14	2.754
N1	H24	3.351	N1	H27	3.021
N2	H3	3.431	N2	H8	2.878
N2	H9	3.108	N2	H13	2.795
N2	H14	2.863	N2	H16	2.523
N2	H17	3.226	N3	H6	2.882
N3	H7	3.112	N3	H23	3.090
N3	H24	2.564	N3	H25	2.808
N3	H27	2.737	C1	H3	3.180
C1	H4	3.116	C2	H4	3.111
C2	H5	3.176	C3	H1	3.203
C3	H5	3.193	C3	H16	3.337
C4	H1	3.198	C4	H2	3.202
C5	H2	3.202	C5	H3	3.181
C6	H3	3.283	C6	H6	2.897
C6	H7	3.091	C6	H8	2.908
C6	H9	3.074	C6	H13	3.295
C6	H14	2.814	C6	H16	3.173
C6	H24	2.971	C6	H25	3.480
C6	H27	2.872	C7	H13	3.082
C7	H16	3.108	C7	H17	3.361
C8	H23	3.148	C8	H24	3.183
C8	H25	3.151	C9	H2	3.081
C9	H3	3.354	C9	H6	2.828
C9	H7	2.785	C9	H10	3.257
C9	H12	3.257	C9	H13	2.758
C9	H14	2.799	C9	H15	3.321
C9	H16	2.648	C9	H17	2.995
C9	H18	3.285	C10	H2	3.257
C10	H6	3.312	C10	H11	3.271
C11	H2	3.199	C11	H12	3.247
C11	H13	3.157	C11	H14	3.136
C11	H15	2.565	C12	H2	3.018

Table 10. Intramolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
C13	H2	2.900	C13	H10	3.247
C13	H16	3.278	C13	H17	2.941
C13	H18	2.620	C14	H2	2.943
C14	H3	3.074	C14	H7	3.146
C14	H11	3.271	C15	H6	3.221
C15	H10	2.682	C15	H27	3.428
C16	H3	2.911	C16	H7	2.991
C16	H12	2.667	C17	H8	2.878
C17	H9	2.713	C17	H19	3.242
C17	H21	3.250	C17	H22	3.279
C17	H23	2.936	C17	H24	2.636
C17	H25	2.785	C17	H26	3.308
C17	H27	2.734	C18	H8	3.173
C18	H9	3.513	C18	H20	3.266
C19	H21	3.250	C19	H22	2.615
C19	H23	2.998	C19	H24	3.261
C21	H19	3.250	C21	H25	3.108
C21	H26	2.580	C21	H27	3.186
C22	H9	3.242	C22	H20	3.262
C23	H8	2.920	C23	H19	2.687
C24	H9	3.315	C24	H21	2.695
H1	H2	2.566	H1	H5	2.474
H2	H3	2.501	H2	H11	3.516
H2	H12	3.367	H3	H4	2.463
H3	H16	2.453	H3	H18	2.872
H4	H5	2.513	H6	H8	2.839
H6	H9	2.268	H6	H13	2.508
H6	H14	3.527	H6	H25	3.457
H7	H8	2.267	H7	H9	2.683
H7	H16	2.793	H7	H17	2.670
H8	H16	3.524	H8	H23	2.474
H8	H24	2.809	H9	H25	2.682
H10	H11	2.327	H10	H13	3.370
H10	H14	3.315	H10	H15	2.359
H11	H12	2.310	H12	H16	3.538
H12	H17	3.010	H12	H18	2.454
H13	H27	3.265	H14	H27	2.715

Table 10. Intramolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H16	H24	3.591	H19	H20	2.316
H19	H22	2.445	H19	H23	3.100
H19	H24	3.525	H20	H21	2.325
H21	H25	3.307	H21	H26	2.383
H21	H27	3.400			

Table 11. Intermolecular contacts less than 3.60 Å

atom	atom	distance	atom	atom	distance
C7	C20[1]	3.495(3)	C8	C20[1]	3.282(3)
C8	C21[1]	3.229(3)	C8	C22[1]	3.501(3)
C15	C23[2]	3.485(4)	C18	C19[3]	3.555(4)
C19	C18[3]	3.555(4)	C19	C19[3]	3.134(4)
C19	C20[3]	3.554(4)	C20	C7[1]	3.495(3)
C20	C8[1]	3.282(3)	C20	C19[3]	3.554(4)
C20	C23[3]	3.566(4)	C21	C8[1]	3.229(3)
C21	C24[4]	3.424(3)	C22	C8[1]	3.501(3)
C23	C15[5]	3.485(4)	C23	C20[3]	3.566(4)
C24	C21[4]	3.424(3)			

Symmetry Operators:

(1) -X,-Y+1,-Z+1
(2) X+1,Y,Z
(3) -X,-Y,-Z+1
(4) -X+1,-Y,-Z+1
(5) X-1,Y,Z

Table 12. Intermolecular contacts less than 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
Ti1	H21[1]	3.584	Cl1	H4[2]	3.201
Cl1	H7[3]	2.787	Cl1	H11[4]	2.834
Cl1	H17[3]	2.941	Cl1	H20[1]	3.426
Cl1	H21[1]	3.203	Cl2	H6[5]	3.036
Cl2	H8[3]	3.412	Cl2	H13[5]	3.235
Cl2	H19[6]	2.888	Cl2	H21[1]	3.168
Cl2	H23[3]	3.304	Cl2	H26[1]	2.926
N1	H21[1]	3.474	C1	H10[4]	3.503
C1	H11[4]	3.232	C1	H12[7]	3.357
C1	H17[3]	3.552	C1	H18[7]	2.883
C2	H10[4]	3.302	C2	H11[4]	3.300
C2	H12[7]	3.569	C2	H18[7]	3.244
C3	H12[7]	3.287	C3	H15[8]	3.560
C4	H6[5]	3.565	C4	H7[5]	3.561
C4	H12[7]	2.892	C5	H6[5]	3.502
C5	H12[7]	2.924	C5	H18[7]	3.442
C7	H4[9]	3.496	C7	H5[9]	3.488
C7	H20[10]	3.417	C8	H21[10]	3.533
C9	H5[9]	3.147	C10	H5[9]	2.925
C11	H1[4]	3.497	C11	H2[4]	3.422
C11	H5[9]	3.232	C11	H11[4]	3.355
C12	H1[4]	3.240	C12	H2[4]	3.449
C12	H10[4]	3.461	C12	H17[11]	3.376
C13	H12[11]	3.561	C13	H17[11]	3.552
C14	H5[9]	3.592	C15	H5[9]	3.367
C15	H22[2]	3.278	C15	H24[2]	3.027
C16	H1[12]	3.326	C17	H9[10]	2.957
C18	H9[10]	2.912	C18	H19[6]	3.317
C18	H20[6]	3.520	C19	H6[10]	3.483
C19	H9[10]	2.769	C19	H19[6]	3.081
C19	H22[6]	3.581	C19	H26[8]	3.591
C20	H6[10]	3.293	C20	H7[10]	3.391
C20	H8[10]	3.334	C20	H9[10]	2.711
C20	H19[6]	3.317	C20	H22[6]	2.933
C20	H27[1]	3.487	C21	H8[10]	2.901
C21	H9[10]	2.789	C21	H22[6]	3.403
C21	H26[1]	3.106	C21	H27[1]	2.938

Table 12. Intermolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
C22	H8[10]	3.173	C22	H9[10]	2.910
C22	H21[1]	3.597	C22	H26[1]	3.346
C23	H13[8]	3.138	C23	H14[8]	3.421
C23	H15[8]	3.323	C23	H20[6]	3.194
C23	H25[8]	3.472	C23	H26[8]	3.462
C23	H27[8]	3.312	C24	H19[2]	3.550
C24	H21[1]	3.148	C24	H22[2]	2.764
H1	C11[4]	3.497	H1	C12[4]	3.240
H1	C16[3]	3.326	H1	H1[13]	3.408
H1	H10[4]	3.174	H1	H11[4]	2.686
H1	H17[3]	2.733	H1	H18[3]	3.169
H1	H18[7]	2.941	H2	C11[4]	3.422
H2	C12[4]	3.449	H2	H10[4]	2.775
H2	H11[4]	2.827	H2	H18[7]	3.585
H3	H10[8]	3.241	H3	H15[8]	2.870
H4	Cl1[8]	3.201	H4	C7[5]	3.496
H4	H6[5]	3.126	H4	H7[5]	2.945
H4	H12[7]	3.159	H4	H15[8]	3.416
H4	H20[6]	2.865	H5	C7[5]	3.488
H5	C9[5]	3.147	H5	C10[5]	2.925
H5	C11[5]	3.232	H5	C14[5]	3.592
H5	C15[5]	3.367	H5	H6[5]	2.958
H5	H7[5]	3.426	H5	H12[7]	3.245
H5	H13[5]	2.957	H6	Cl2[9]	3.036
H6	C4[9]	3.565	H6	C5[9]	3.502
H6	C19[10]	3.483	H6	C20[10]	3.293
H6	H4[9]	3.126	H6	H5[9]	2.958
H6	H19[10]	3.481	H6	H20[10]	3.159
H7	Cl1[12]	2.787	H7	C4[9]	3.561
H7	C20[10]	3.391	H7	H4[9]	2.945
H7	H5[9]	3.426	H7	H20[10]	3.123
H8	Cl2[12]	3.412	H8	C20[10]	3.334
H8	C21[10]	2.901	H8	C22[10]	3.173
H8	H21[10]	2.994	H8	H25[10]	3.357
H9	C17[10]	2.957	H9	C18[10]	2.912
H9	C19[10]	2.769	H9	C20[10]	2.711
H9	C21[10]	2.789	H9	C22[10]	2.910

Table 12. Intermolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H9	$H9^{10}$	3.284	H9	$H19^{10}$	3.302
H9	$H20^{10}$	3.222	H9	$H21^{10}$	3.340
H10	$C1^{4}$	3.503	H10	$C2^{4}$	3.302
H10	$C12^{4}$	3.461	H10	$H1^{4}$	3.174
H10	$H2^{4}$	2.775	H10	$H3^{2}$	3.241
H10	$H11^{4}$	3.169	H10	$H16^{2}$	3.559
H10	$H18^{2}$	3.177	H11	$Cl1^{4}$	2.834
H11	$C1^{4}$	3.232	H11	$C2^{4}$	3.300
H11	$C11^{4}$	3.355	H11	$H1^{4}$	2.686
H11	$H2^{4}$	2.827	H11	$H10^{4}$	3.169
H11	$H17^{11}$	2.980	H12	$C1^{7}$	3.357
H12	$C2^{7}$	3.569	H12	$C3^{7}$	3.287
H12	$C4^{7}$	2.892	H12	$C5^{7}$	2.924
H12	$C13^{11}$	3.561	H12	$H4^{7}$	3.159
H12	$H5^{7}$	3.245	H12	$H12^{11}$	3.486
H12	$H17^{11}$	3.282	H13	$Cl2^{9}$	3.235
H13	$C23^{2}$	3.138	H13	$H5^{9}$	2.957
H13	$H22^{2}$	3.022	H13	$H23^{2}$	3.008
H13	$H24^{2}$	2.851	H14	$C23^{2}$	3.421
H14	$H20^{1}$	3.392	H14	$H21^{1}$	3.468
H14	$H22^{2}$	2.957	H14	$H24^{2}$	3.107
H15	$C3^{2}$	3.560	H15	$C23^{2}$	3.323
H15	$H3^{2}$	2.870	H15	$H4^{2}$	3.416
H15	$H16^{2}$	3.094	H15	$H22^{2}$	3.314
H15	$H23^{2}$	3.570	H15	$H24^{2}$	2.627
H16	$H10^{8}$	3.559	H16	$H15^{8}$	3.094
H17	$Cl1^{12}$	2.941	H17	$C1^{12}$	3.552
H17	$C12^{11}$	3.376	H17	$C13^{11}$	3.552
H17	$H1^{12}$	2.733	H17	$H11^{11}$	2.980
H17	$H12^{11}$	3.282	H18	$C1^{7}$	2.883
H18	$C2^{7}$	3.244	H18	$C5^{7}$	3.442
H18	$H1^{12}$	3.169	H18	$H1^{7}$	2.941
H18	$H2^{7}$	3.585	H18	$H10^{8}$	3.177
H19	$Cl2^{6}$	2.888	H19	$C18^{6}$	3.317
H19	$C19^{6}$	3.081	H19	$C20^{6}$	3.317
H19	$C24^{8}$	3.550	H19	$H6^{10}$	3.481
H19	$H9^{10}$	3.302	H19	$H19^{6}$	3.311

Table 12. Intermolecular contacts less than 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H19	H26[8]	2.740	H20	Cl1[1]	3.426
H20	C7[10]	3.417	H20	C18[6]	3.520
H20	C23[6]	3.194	H20	H4[6]	2.865
H20	H6[10]	3.159	H20	H7[10]	3.123
H20	H9[10]	3.222	H20	H14[1]	3.392
H20	H22[6]	2.673	H20	H24[6]	3.077
H20	H27[1]	3.502	H21	Ti1[1]	3.584
H21	Cl1[1]	3.203	H21	Cl2[1]	3.168
H21	N1[1]	3.474	H21	C8[10]	3.533
H21	C22[1]	3.597	H21	C24[1]	3.148
H21	H8[10]	2.994	H21	H9[10]	3.340
H21	H14[1]	3.468	H21	H22[6]	3.478
H21	H26[1]	3.111	H21	H27[1]	2.506
H22	C15[8]	3.278	H22	C19[6]	3.581
H22	C20[6]	2.933	H22	C21[6]	3.403
H22	C24[8]	2.764	H22	H13[8]	3.022
H22	H14[8]	2.957	H22	H15[8]	3.314
H22	H20[6]	2.673	H22	H21[6]	3.478
H22	H25[8]	2.810	H22	H26[8]	2.624
H22	H27[8]	2.401	H23	Cl2[12]	3.304
H23	H13[8]	3.008	H23	H15[8]	3.570
H23	H25[8]	3.243	H23	H25[10]	2.924
H23	H27[8]	3.503	H24	C15[8]	3.027
H24	H13[8]	2.851	H24	H14[8]	3.107
H24	H15[8]	2.627	H24	H20[6]	3.077
H25	C23[2]	3.472	H25	H8[10]	3.357
H25	H22[2]	2.810	H25	H23[2]	3.243
H25	H23[10]	2.924	H26	Cl2[1]	2.926
H26	C19[2]	3.591	H26	C21[1]	3.106
H26	C22[1]	3.346	H26	C23[2]	3.462
H26	H19[2]	2.740	H26	H21[1]	3.111
H26	H22[2]	2.624	H27	C20[1]	3.487
H27	C21[1]	2.938	H27	C23[2]	3.312
H27	H20[1]	3.502	H27	H21[1]	2.506
H27	H22[2]	2.401	H27	H23[2]	3.503

Symmetry Operators:

(1) -X+1,-Y,-Z+1
(2) X+1,Y,Z
(3) X+1,Y-1,Z
(4) -X+1,-Y,-Z+2
(5) X,Y-1,Z
(6) -X,-Y,-Z+1
(7) -X,-Y,-Z+2
(8) X-1,Y,Z
(9) X,Y+1,Z
(10) -X,-Y+1,-Z+1
(11) -X,-Y+1,-Z+2
(12) X-1,Y+1,Z
(13) -X+1,-Y-1,-Z+2

Electronic Supplementary Material (ESI) for Dalton Transactions

X-ray Structure Report for $CpTiCl_2[1,3\text{-}Cy_2(CH_2N)_2C{=}N]$ (**1c**)

September 7, 2010

Experimental

Data Collection

A yellow block crystal of $C_{20}H_{31}Cl_2N_3Ti$ having approximate dimensions of 0.32 x 0.20 x 0.18 mm was mounted on a glass fiber. All measurements were made on a Rigaku RAXIS RAPID imaging plate area detector with graphite monochromated Mo-Kα radiation.

Indexing was performed from 3 oscillations that were exposed for 60 seconds. The crystal-to-detector distance was 127.40 mm.

Cell constants and an orientation matrix for data collection corresponded to a primitive orthorhombic cell with dimensions:

a = 9.0816(3) Å
b = 12.7130(5) Å
c = 18.7394(7) Å
V = 2163.54(13) Å^3

For Z = 4 and F.W. = 432.29, the calculated density is 1.327 g/cm^3. The systematic absences of:

h00: h ± 2n
0k0: k ± 2n
00l: l ± 2n

uniquely determine the space group to be:

$P2_12_12_1$ (#19)

The data were collected at a temperature of -150 ± 1°C to a maximum 2θ value of 55.0°. A total of 111 oscillation images were collected. A sweep of data was done using ω scans from 130.0 to 190.0° in 2.0° step, at χ=45.0° and ϕ = 0.0°. The exposure rate was 90.0 [sec./°]. A second sweep was performed using ω scans from 0.0 to 162.0° in 2.0° step, at χ=45.0° and ϕ = 180.0°. The exposure rate was 90.0 [sec./°]. The crystal-to-detector distance was 127.40 mm. Readout was performed in the 0.100 mm pixel mode.

Data Reduction

Of the 21288 reflections that were collected, 4941 were unique (R_{int} = 0.040); equivalent reflections were merged.

The linear absorption coefficient, μ, for Mo-Kα radiation is 6.513 cm^{-1}. An empirical absorption correction was applied which resulted in transmission factors ranging from 0.693 to 0.889. The data were corrected for Lorentz and polarization effects.

Structure Solution and Refinement

The structure was solved by direct methods[1] and expanded using Fourier techniques[2]. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were refined using the riding model. The final cycle of full-matrix least-squares refinement[3] on F^2 was based on 4941 observed reflections and 237 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R1 = \Sigma \; ||Fo| - |Fc|| \; / \; \Sigma \; |Fo| = 0.0352$$

$$wR2 = [\; \Sigma \; (\; w \; (Fo^2 - Fc^2)^2 \;) / \; \Sigma \; w(Fo^2)^2]^{1/2} = 0.0854$$

The standard deviation of an observation of unit weight[4] was 1.07. Unit weights were used. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.24 and -0.42 $e^-/Å^3$, respectively. The absolute structure was deduced based on Flack parameter, 0.01(2), refined using 2141 Friedel pairs.[5]

Neutral atom scattering factors were taken from Cromer and Waber[6]. Anomalous dispersion effects were included in Fcalc[7]; the values for Δf' and Δf'' were those of Creagh and McAuley[8]. The values for the mass attenuation coefficients are those of Creagh and Hubbell[9]. All calculations were performed using the CrystalStructure[10] crystallographic software package except for refinement, which was performed using SHELXL-97[11].

References

(1) SIR92: Altomare, A., Cascarano, G., Giacovazzo, C., Guagliardi, A., Burla, M., Polidori, G., and Camalli, M. (1994) J. Appl. Cryst., 27, 435.

(2) DIRDIF99: Beurskens, P.T., Admiraal, G., Beurskens, G., Bosman, W.P., de Gelder, R., Israel, R. and Smits, J.M.M.(1999). The DIRDIF-99 program system, Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.

(3) Least Squares function minimized: (SHELXL97)

$\Sigma w(F_o^2-F_c^2)^2$ where w = Least Squares weights.

(4) Standard deviation of an observation of unit weight:

$[\Sigma w(F_o^2-F_c^2)^2/(N_o-N_v)]^{1/2}$

where: N_o = number of observations
N_v = number of variables

(5) Flack, H. D. (1983), Acta Cryst. A39, 876-881.

(6) Cromer, D. T. & Waber, J. T.; "International Tables for X-ray Crystallography", Vol. IV, The Kynoch Press, Birmingham, England, Table 2.2 A (1974).

(7) Ibers, J. A. & Hamilton, W. C.; Acta Crystallogr., 17, 781 (1964).

(8) Creagh, D. C. & McAuley, W.J .; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.6.8, pages 219-222 (1992).

(9) Creagh, D. C. & Hubbell, J.H..; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.4.3, pages 200-206 (1992).

(10) CrystalStructure 3.8: Crystal Structure Analysis Package, Rigaku and Rigaku Americas (2000-2007). 9009 New Trails Dr. The Woodlands TX 77381 USA.

(11) SHELX97: Sheldrick, G.M. (1997).

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	$C_{20}H_{31}Cl_2N_3Ti$
Formula Weight	432.29
Crystal Color, Habit	yellow, block
Crystal Dimensions	0.32 X 0.20 X 0.18 mm
Crystal System	orthorhombic
Lattice Type	Primitive
Indexing Images	3 oscillations @ 60.0 seconds
Detector Position	127.40 mm
Pixel Size	0.100 mm
Lattice Parameters	a = 9.0816(3) Å b = 12.7130(5) Å c = 18.7394(7) Å V = 2163.54(13) Å^3
Space Group	$P2_12_12_1$ (#19)
Z value	4
D_{calc}	1.327 g/cm^3
F_{000}	912.00
μ(MoKα)	6.513 cm^{-1}

B. Intensity Measurements

Diffractometer	Rigaku RAXIS-RAPID
Radiation	MoKα (λ = 0.71075 Å) graphite monochromated
Detector Aperture	280 mm x 256 mm
Data Images	111 exposures
ω oscillation Range (χ=45.0, ϕ=0.0)	130.0 - 190.0^{o}
Exposure Rate	90.0 sec./^{o}
ω oscillation Range (χ=45.0, ϕ=180.0)	0.0 - 162.0^{o}
Exposure Rate	90.0 sec./^{o}
Detector Position	127.40 mm
Pixel Size	0.100 mm
$2\theta_{max}$	55.0^{o}
No. of Reflections Measured	Total: 21288 Unique: 4941 (R_{int} = 0.040) Friedel pairs: 2141
Corrections	Lorentz-polarization Absorption (trans. factors: 0.693 - 0.889)

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SIR92)
Refinement	Full-matrix least-squares on F^2
Function Minimized	Σ w $(Fo^2 - Fc^2)^2$
Least Squares Weights	$w = 1/ [\sigma^2(Fo^2) + (0.0348 \cdot P)^2 + 1.0996 \cdot P]$ where $P = (Max(Fo^2,0) + 2Fc^2)/3$
$2\theta_{max}$ cutoff	55.0^o
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (All reflections)	4941
No. Variables	237
Reflection/Parameter Ratio	20.85
Residuals: R1 (I>2.00σ(I))	0.0352
Residuals: R (All reflections)	0.0372
Residuals: wR2 (All reflections)	0.0854
Goodness of Fit Indicator	1.072
Flack Parameter (Friedel pairs = 2141)	0.01(2)
Max Shift/Error in Final Cycle	0.001
Maximum peak in Final Diff. Map	0.24 $e^-/Å^3$
Minimum peak in Final Diff. Map	-0.42 $e^-/Å^3$

Table 1. Atomic coordinates and B_{iso}/B_{eq}

atom	x	y	z	B_{eq}
Ti(1)	0.02906(4)	0.13329(3)	0.65777(2)	1.932(7)
Cl(1)	-0.09192(6)	0.28576(5)	0.62537(4)	2.910(11)
Cl(2)	-0.11695(6)	-0.00230(5)	0.61501(3)	2.701(11)
N(1)	0.18247(18)	0.13264(15)	0.59995(9)	1.91(2)
N(2)	0.3724(2)	0.04788(14)	0.53619(11)	2.09(3)
N(3)	0.4294(2)	0.19438(14)	0.59483(11)	2.17(3)
C(1)	-0.0330(4)	0.0699(4)	0.77412(17)	6.89(13)
C(2)	0.1114(4)	0.0353(2)	0.75703(16)	4.33(6)
C(3)	0.1977(3)	0.1240(2)	0.75266(15)	3.91(5)
C(4)	0.1134(5)	0.2102(2)	0.76329(17)	5.32(8)
C(5)	-0.0271(5)	0.1804(4)	0.77759(18)	6.92(12)
C(6)	0.3211(2)	0.12635(18)	0.57821(11)	1.90(3)
C(7)	0.5331(2)	0.05600(18)	0.52943(15)	2.67(4)
C(8)	0.5627(2)	0.16968(18)	0.55268(15)	2.64(4)
C(9)	0.2897(2)	-0.04944(16)	0.52497(12)	1.87(3)
C(10)	0.3209(2)	-0.13061(19)	0.58293(12)	2.31(3)
C(11)	0.2292(2)	-0.23017(18)	0.57034(12)	2.48(3)
C(12)	0.2541(2)	-0.27420(17)	0.49599(13)	2.40(3)
C(13)	0.2231(2)	-0.19205(18)	0.43927(13)	2.52(4)
C(14)	0.3179(2)	-0.09360(18)	0.45045(12)	2.21(3)
C(15)	0.4015(2)	0.29920(16)	0.62453(12)	1.97(3)
C(16)	0.4969(2)	0.32094(18)	0.68968(12)	2.36(3)
C(17)	0.4546(2)	0.42728(18)	0.72290(13)	2.56(4)
C(18)	0.4649(2)	0.51540(18)	0.66819(13)	2.75(4)
C(19)	0.3754(3)	0.49148(18)	0.60173(14)	2.82(4)
C(20)	0.4188(2)	0.38582(17)	0.56928(13)	2.41(3)

$B_{eq} = 8/3\ \pi^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos\gamma + 2U_{13}(aa^*cc^*)\cos\beta + 2U_{23}(bb^*cc^*)\cos\alpha)$

Table 2. Atomic coordinates and B $_{iso}$ involving hydrogens/B_{eq}

atom	x	y	z	B_{eq}
H(1)	-0.1230	0.0243	0.7831	8.81
H(2)	0.1285	-0.0426	0.7531	2.37
H(3)	0.3095	0.1267	0.7398	2.37
H(4)	0.1680	0.2878	0.7555	2.37
H(5)	-0.1150	0.2327	0.7887	7.93
H(6)	0.5651	0.0439	0.4796	3.20
H(7)	0.5836	0.0052	0.5611	3.20
H(8)	0.6528	0.1747	0.5822	3.16
H(9)	0.5723	0.2170	0.5110	3.16
H(10)	0.1827	-0.0314	0.5279	2.24
H(11)	0.2966	-0.1006	0.6302	2.78
H(12)	0.4269	-0.1488	0.5827	2.78
H(13)	0.2562	-0.2840	0.6062	2.98
H(14)	0.1235	-0.2135	0.5765	2.98
H(15)	0.3573	-0.2984	0.4916	2.88
H(16)	0.1889	-0.3356	0.4885	2.88
H(17)	0.1177	-0.1723	0.4409	3.02
H(18)	0.2437	-0.2221	0.3916	3.02
H(19)	0.4234	-0.1118	0.4452	2.66
H(20)	0.2930	-0.0400	0.4140	2.66
H(21)	0.2967	0.3004	0.6408	2.36
H(22)	0.6019	0.3221	0.6755	2.83
H(23)	0.4832	0.2642	0.7253	2.83
H(24)	0.3528	0.4234	0.7416	3.08
H(25)	0.5212	0.4427	0.7634	3.08
H(26)	0.4289	0.5816	0.6898	3.30
H(27)	0.5693	0.5258	0.6547	3.30
H(28)	0.3912	0.5478	0.5661	3.38
H(29)	0.2695	0.4904	0.6141	3.38
H(30)	0.3554	0.3706	0.5275	2.89
H(31)	0.5222	0.3887	0.5528	2.89

$B_{eq} = 8/3\ \pi^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos\gamma + 2U_{13}(aa^*cc^*)\cos\beta + 2U_{23}(bb^*cc^*)\cos\alpha)$

Table 3. Anisotropic displacement parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Ti(1)	0.01435(16)	0.03095(19)	0.02809(19)	-0.00162(16)	0.00203(15)	-0.00526(16)
Cl(1)	0.0228(2)	0.0317(2)	0.0561(3)	0.0041(2)	0.0013(2)	-0.0074(2)
Cl(2)	0.0251(2)	0.0339(2)	0.0437(3)	-0.0089(2)	-0.0021(2)	-0.0038(2)
N(1)	0.0162(7)	0.0274(8)	0.0291(9)	-0.0006(7)	0.0021(7)	-0.0051(8)
N(2)	0.0144(8)	0.0223(8)	0.0427(10)	-0.0003(6)	0.0051(7)	-0.0066(7)
N(3)	0.0152(8)	0.0246(8)	0.0425(10)	-0.0018(6)	0.0055(7)	-0.0081(8)
C(1)	0.064(2)	0.171(4)	0.0266(14)	-0.069(3)	0.0020(16)	0.022(2)
C(2)	0.088(2)	0.0455(15)	0.0308(13)	-0.0088(17)	-0.0044(16)	0.0076(11)
C(3)	0.0391(13)	0.073(2)	0.0364(13)	-0.0126(15)	-0.0139(12)	0.0154(14)
C(4)	0.110(3)	0.0555(19)	0.0366(15)	-0.010(2)	-0.024(2)	-0.0090(13)
C(5)	0.090(3)	0.144(4)	0.0287(15)	0.049(3)	0.0133(19)	-0.016(2)
C(6)	0.0172(9)	0.0256(10)	0.0295(10)	0.0007(8)	0.0005(8)	-0.0018(8)
C(7)	0.0174(10)	0.0285(11)	0.0553(15)	-0.0026(9)	0.0100(11)	-0.0123(10)
C(8)	0.0141(10)	0.0301(11)	0.0560(15)	-0.0017(8)	0.0061(10)	-0.0088(10)
C(9)	0.0197(9)	0.0222(10)	0.0290(11)	-0.0019(8)	0.0024(8)	-0.0020(8)
C(10)	0.0294(11)	0.0321(11)	0.0264(10)	-0.0008(10)	-0.0002(9)	-0.0010(9)
C(11)	0.0338(12)	0.0280(11)	0.0326(11)	0.0007(10)	0.0062(10)	0.0029(9)
C(12)	0.0314(11)	0.0225(10)	0.0372(11)	-0.0018(9)	0.0039(10)	-0.0028(9)
C(13)	0.0342(12)	0.0318(11)	0.0296(11)	-0.0042(10)	0.0009(10)	-0.0034(9)
C(14)	0.0279(10)	0.0287(11)	0.0276(11)	-0.0030(9)	0.0038(9)	-0.0014(8)
C(15)	0.0161(9)	0.0233(10)	0.0353(11)	0.0009(7)	0.0017(9)	-0.0053(8)
C(16)	0.0258(11)	0.0321(11)	0.0318(11)	-0.0003(8)	-0.0025(9)	0.0008(9)
C(17)	0.0280(12)	0.0351(12)	0.0343(11)	-0.0022(10)	-0.0010(10)	-0.0056(9)
C(18)	0.0330(11)	0.0291(11)	0.0425(12)	-0.0008(10)	-0.0042(11)	-0.0049(9)
C(19)	0.0384(12)	0.0270(11)	0.0417(13)	0.0049(10)	-0.0058(11)	-0.0016(10)
C(20)	0.0287(10)	0.0280(11)	0.0349(11)	0.0013(9)	-0.0028(9)	-0.0006(9)

The general temperature factor expression: $\exp(-2\pi^2(a^{*2}U_{11}h^2 + b^{*2}U_{22}k^2 + c^{*2}U_{33}l^2 + 2a^*b^*U_{12}hk + 2a^*c^*U_{13}hl + 2b^*c^*U_{23}kl))$

Table 4. Bond lengths (Å)

atom	atom	distance	atom	atom	distance
Ti(1)	Cl(1)	2.3093(7)	Ti(1)	Cl(2)	2.3177(7)
Ti(1)	N(1)	1.7650(16)	Ti(1)	C(1)	2.392(3)
Ti(1)	C(2)	2.360(3)	Ti(1)	C(3)	2.350(2)
Ti(1)	C(4)	2.335(3)	Ti(1)	C(5)	2.379(3)
N(1)	C(6)	1.325(2)	N(2)	C(6)	1.354(2)
N(2)	C(7)	1.468(2)	N(2)	C(9)	1.462(2)
N(3)	C(6)	1.346(2)	N(3)	C(8)	1.480(3)
N(3)	C(15)	1.466(2)	C(1)	C(2)	1.420(5)
C(1)	C(5)	1.407(8)	C(2)	C(3)	1.376(4)
C(3)	C(4)	1.351(5)	C(4)	C(5)	1.358(7)
C(7)	C(8)	1.533(3)	C(9)	C(10)	1.525(3)
C(9)	C(14)	1.527(3)	C(10)	C(11)	1.534(3)
C(11)	C(12)	1.518(3)	C(12)	C(13)	1.517(3)
C(13)	C(14)	1.534(3)	C(15)	C(16)	1.522(3)
C(15)	C(20)	1.520(3)	C(16)	C(17)	1.537(3)
C(17)	C(18)	1.521(3)	C(18)	C(19)	1.518(3)
C(19)	C(20)	1.526(3)			

Table 5. Bond lengths involving hydrogens (Å)

atom	atom	distance	atom	atom	distance
C(1)	H(1)	1.016	C(2)	H(2)	1.006
C(3)	H(3)	1.044	C(4)	H(4)	1.114
C(5)	H(5)	1.059	C(7)	H(6)	0.990
C(7)	H(7)	0.990	C(8)	H(8)	0.990
C(8)	H(9)	0.990	C(9)	H(10)	1.000
C(10)	H(11)	0.990	C(10)	H(12)	0.990
C(11)	H(13)	0.990	C(11)	H(14)	0.990
C(12)	H(15)	0.990	C(12)	H(16)	0.990
C(13)	H(17)	0.990	C(13)	H(18)	0.990
C(14)	H(19)	0.990	C(14)	H(20)	0.990
C(15)	H(21)	1.000	C(16)	H(22)	0.990
C(16)	H(23)	0.990	C(17)	H(24)	0.990
C(17)	H(25)	0.990	C(18)	H(26)	0.990
C(18)	H(27)	0.990	C(19)	H(28)	0.990
C(19)	H(29)	0.990	C(20)	H(30)	0.990
C(20)	H(31)	0.990			

Table 6. Bond angles (°)

atom	atom	atom	angle	atom	atom	atom	angle
Cl(1)	Ti(1)	Cl(2)	105.14(2)	Cl(1)	Ti(1)	N(1)	102.60(6)
Cl(1)	Ti(1)	C(1)	114.22(12)	Cl(1)	Ti(1)	C(2)	143.21(8)
Cl(1)	Ti(1)	C(3)	123.44(8)	Cl(1)	Ti(1)	C(4)	91.56(9)
Cl(1)	Ti(1)	C(5)	86.27(13)	Cl(2)	Ti(1)	N(1)	103.64(6)
Cl(2)	Ti(1)	C(1)	85.97(11)	Cl(2)	Ti(1)	C(2)	93.52(8)
Cl(2)	Ti(1)	C(3)	126.68(8)	Cl(2)	Ti(1)	C(4)	142.36(9)
Cl(2)	Ti(1)	C(5)	113.00(13)	N(1)	Ti(1)	C(1)	138.08(12)
N(1)	Ti(1)	C(2)	103.37(11)	N(1)	Ti(1)	C(3)	87.12(8)
N(1)	Ti(1)	C(4)	105.24(13)	N(1)	Ti(1)	C(5)	138.58(14)
C(1)	Ti(1)	C(2)	34.77(14)	C(1)	Ti(1)	C(3)	56.43(12)
C(1)	Ti(1)	C(4)	56.39(15)	C(1)	Ti(1)	C(5)	34.29(19)
C(2)	Ti(1)	C(3)	33.97(12)	C(2)	Ti(1)	C(4)	56.60(11)
C(2)	Ti(1)	C(5)	57.11(15)	C(3)	Ti(1)	C(4)	33.52(13)
C(3)	Ti(1)	C(5)	55.82(14)	C(4)	Ti(1)	C(5)	33.48(17)
Ti(1)	N(1)	C(6)	159.82(15)	C(6)	N(2)	C(7)	109.90(17)
C(6)	N(2)	C(9)	122.05(18)	C(7)	N(2)	C(9)	123.86(17)
C(6)	N(3)	C(8)	109.76(18)	C(6)	N(3)	C(15)	123.08(17)
C(8)	N(3)	C(15)	122.46(17)	Ti(1)	C(1)	C(2)	71.39(18)
Ti(1)	C(1)	C(5)	72.4(2)	C(2)	C(1)	C(5)	106.5(3)
Ti(1)	C(2)	C(1)	73.8(2)	Ti(1)	C(2)	C(3)	72.61(17)
C(1)	C(2)	C(3)	106.6(3)	Ti(1)	C(3)	C(2)	73.42(18)
Ti(1)	C(3)	C(4)	72.6(2)	C(2)	C(3)	C(4)	109.4(3)
Ti(1)	C(4)	C(3)	73.84(18)	Ti(1)	C(4)	C(5)	75.0(2)
C(3)	C(4)	C(5)	109.6(3)	Ti(1)	C(5)	C(1)	73.3(2)
Ti(1)	C(5)	C(4)	71.5(2)	C(1)	C(5)	C(4)	107.8(4)
N(1)	C(6)	N(2)	123.37(19)	N(1)	C(6)	N(3)	125.7(2)
N(2)	C(6)	N(3)	110.91(17)	N(2)	C(7)	C(8)	102.49(17)
N(3)	C(8)	C(7)	102.00(17)	N(2)	C(9)	C(10)	112.02(18)
N(2)	C(9)	C(14)	110.89(18)	C(10)	C(9)	C(14)	111.81(17)
C(9)	C(10)	C(11)	110.37(18)	C(10)	C(11)	C(12)	111.37(19)
C(11)	C(12)	C(13)	111.19(18)	C(12)	C(13)	C(14)	111.21(19)
C(9)	C(14)	C(13)	109.31(18)	N(3)	C(15)	C(16)	111.80(17)
N(3)	C(15)	C(20)	112.47(18)	C(16)	C(15)	C(20)	110.87(17)
C(15)	C(16)	C(17)	110.02(18)	C(16)	C(17)	C(18)	111.06(19)
C(17)	C(18)	C(19)	111.86(19)	C(18)	C(19)	C(20)	111.4(2)
C(15)	C(20)	C(19)	109.87(19)				

Table 7. Bond angles involving hydrogens (°)

atom	atom	atom	angle	atom	atom	atom	angle
Ti(1)	C(1)	H(1)	122.2	C(2)	C(1)	H(1)	127.1
C(5)	C(1)	H(1)	126.4	Ti(1)	C(2)	H(2)	120.7
C(1)	C(2)	H(2)	117.6	C(3)	C(2)	H(2)	135.7
Ti(1)	C(3)	H(3)	117.2	C(2)	C(3)	H(3)	126.5
C(4)	C(3)	H(3)	124.0	Ti(1)	C(4)	H(4)	114.0
C(3)	C(4)	H(4)	116.5	C(5)	C(4)	H(4)	133.7
Ti(1)	C(5)	H(5)	120.4	C(1)	C(5)	H(5)	127.4
C(4)	C(5)	H(5)	124.8	N(2)	C(7)	H(6)	111.3
N(2)	C(7)	H(7)	111.3	C(8)	C(7)	H(6)	111.3
C(8)	C(7)	H(7)	111.3	H(6)	C(7)	H(7)	109.2
N(3)	C(8)	H(8)	111.4	N(3)	C(8)	H(9)	111.4
C(7)	C(8)	H(8)	111.4	C(7)	C(8)	H(9)	111.4
H(8)	C(8)	H(9)	109.2	N(2)	C(9)	H(10)	107.3
C(10)	C(9)	H(10)	107.3	C(14)	C(9)	H(10)	107.3
C(9)	C(10)	H(11)	109.6	C(9)	C(10)	H(12)	109.6
C(11)	C(10)	H(11)	109.6	C(11)	C(10)	H(12)	109.6
H(11)	C(10)	H(12)	108.1	C(10)	C(11)	H(13)	109.4
C(10)	C(11)	H(14)	109.4	C(12)	C(11)	H(13)	109.4
C(12)	C(11)	H(14)	109.3	H(13)	C(11)	H(14)	108.0
C(11)	C(12)	H(15)	109.4	C(11)	C(12)	H(16)	109.4
C(13)	C(12)	H(15)	109.4	C(13)	C(12)	H(16)	109.4
H(15)	C(12)	H(16)	108.0	C(12)	C(13)	H(17)	109.4
C(12)	C(13)	H(18)	109.4	C(14)	C(13)	H(17)	109.4
C(14)	C(13)	H(18)	109.4	H(17)	C(13)	H(18)	108.0
C(9)	C(14)	H(19)	109.8	C(9)	C(14)	H(20)	109.8
C(13)	C(14)	H(19)	109.8	C(13)	C(14)	H(20)	109.8
H(19)	C(14)	H(20)	108.3	N(3)	C(15)	H(21)	107.1
C(16)	C(15)	H(21)	107.1	C(20)	C(15)	H(21)	107.1
C(15)	C(16)	H(22)	109.7	C(15)	C(16)	H(23)	109.7
C(17)	C(16)	H(22)	109.7	C(17)	C(16)	H(23)	109.7
H(22)	C(16)	H(23)	108.2	C(16)	C(17)	H(24)	109.4
C(16)	C(17)	H(25)	109.4	C(18)	C(17)	H(24)	109.4
C(18)	C(17)	H(25)	109.4	H(24)	C(17)	H(25)	108.0
C(17)	C(18)	H(26)	109.3	C(17)	C(18)	H(27)	109.2
C(19)	C(18)	H(26)	109.2	C(19)	C(18)	H(27)	109.2
H(26)	C(18)	H(27)	107.9	C(18)	C(19)	H(28)	109.3
C(18)	C(19)	H(29)	109.3	C(20)	C(19)	H(28)	109.3

Table 7. Bond angles involving hydrogens (°) (continued)

atom	atom	atom	angle	atom	atom	atom	angle
C(20)	C(19)	H(29)	109.3	H(28)	C(19)	H(29)	108.0
C(15)	C(20)	H(30)	109.7	C(15)	C(20)	H(31)	109.7
C(19)	C(20)	H(30)	109.7	C(19)	C(20)	H(31)	109.7
H(30)	C(20)	H(31)	108.2				

Table 8. Torsion Angles(°)

atom1	atom2	atom3	atom4	angle
Cl(1)	Ti(1)	N(1)	C(6)	130.1(4)
Cl(1)	Ti(1)	C(1)	C(5)	-38.2(2)
Cl(1)	Ti(1)	C(2)	C(3)	-70.2(2)
Cl(1)	Ti(1)	C(3)	C(4)	20.3(2)
Cl(1)	Ti(1)	C(4)	C(5)	80.8(2)
Cl(1)	Ti(1)	C(5)	C(4)	-98.5(2)
Cl(2)	Ti(1)	C(1)	C(2)	102.1(2)
Cl(2)	Ti(1)	C(2)	C(1)	-77.8(2)
Cl(2)	Ti(1)	C(3)	C(2)	-14.4(2)
Cl(2)	Ti(1)	C(4)	C(3)	79.3(2)
Cl(2)	Ti(1)	C(5)	C(1)	40.7(2)
N(1)	Ti(1)	C(1)	C(2)	-3.9(3)
C(1)	Ti(1)	N(1)	C(6)	-21.3(5)
N(1)	Ti(1)	C(2)	C(3)	63.57(19)
N(1)	Ti(1)	C(3)	C(2)	-119.3(2)
C(3)	Ti(1)	N(1)	C(6)	6.5(5)
N(1)	Ti(1)	C(4)	C(5)	-175.7(2)
N(1)	Ti(1)	C(5)	C(1)	-109.5(2)
C(5)	Ti(1)	N(1)	C(6)	31.4(5)
C(2)	Ti(1)	C(1)	C(5)	114.9(3)
C(1)	Ti(1)	C(3)	C(4)	-78.4(2)
C(3)	Ti(1)	C(1)	C(5)	77.1(2)
C(1)	Ti(1)	C(4)	C(5)	-37.5(2)
C(4)	Ti(1)	C(1)	C(5)	36.6(2)
C(5)	Ti(1)	C(1)	C(2)	-114.9(3)
C(3)	Ti(1)	C(2)	C(1)	113.7(3)
C(2)	Ti(1)	C(4)	C(5)	-79.5(3)
C(4)	Ti(1)	C(2)	C(3)	-36.0(2)
C(2)	Ti(1)	C(5)	C(4)	77.9(2)
C(5)	Ti(1)	C(2)	C(3)	-76.3(2)
C(4)	Ti(1)	C(3)	C(2)	117.2(3)
C(3)	Ti(1)	C(5)	C(4)	36.9(2)
C(5)	Ti(1)	C(3)	C(4)	-36.8(2)
C(5)	Ti(1)	C(4)	C(3)	116.0(3)
Ti(1)	N(1)	C(6)	N(3)	-65.4(5)
C(7)	N(2)	C(6)	N(1)	-172.7(2)
C(6)	N(2)	C(9)	C(10)	-86.3(2)
Cl(1)	Ti(1)	C(1)	C(2)	-153.12(19)
Cl(1)	Ti(1)	C(2)	C(1)	43.5(3)
Cl(1)	Ti(1)	C(3)	C(2)	137.52(17)
Cl(1)	Ti(1)	C(4)	C(3)	-163.1(2)
Cl(1)	Ti(1)	C(5)	C(1)	145.6(2)
Cl(2)	Ti(1)	N(1)	C(6)	-120.6(4)
Cl(2)	Ti(1)	C(1)	C(5)	-143.0(2)
Cl(2)	Ti(1)	C(2)	C(3)	168.49(18)
Cl(2)	Ti(1)	C(3)	C(4)	-131.6(2)
Cl(2)	Ti(1)	C(4)	C(5)	-36.8(3)
Cl(2)	Ti(1)	C(5)	C(4)	156.6(2)
N(1)	Ti(1)	C(1)	C(5)	111.0(3)
N(1)	Ti(1)	C(2)	C(1)	177.3(2)
C(2)	Ti(1)	N(1)	C(6)	-23.6(5)
N(1)	Ti(1)	C(3)	C(4)	123.5(2)
N(1)	Ti(1)	C(4)	C(3)	-59.6(2)
C(4)	Ti(1)	N(1)	C(6)	35.0(5)
N(1)	Ti(1)	C(5)	C(4)	6.3(4)
C(1)	Ti(1)	C(2)	C(3)	-113.7(3)
C(1)	Ti(1)	C(3)	C(2)	38.8(2)
C(3)	Ti(1)	C(1)	C(2)	-37.9(2)
C(1)	Ti(1)	C(4)	C(3)	78.5(2)
C(4)	Ti(1)	C(1)	C(2)	-78.4(2)
C(1)	Ti(1)	C(5)	C(4)	115.9(4)
C(2)	Ti(1)	C(3)	C(4)	-117.2(3)
C(2)	Ti(1)	C(4)	C(3)	36.5(2)
C(4)	Ti(1)	C(2)	C(1)	77.7(2)
C(2)	Ti(1)	C(5)	C(1)	-38.0(2)
C(5)	Ti(1)	C(2)	C(1)	37.5(2)
C(3)	Ti(1)	C(4)	C(5)	-116.0(3)
C(3)	Ti(1)	C(5)	C(1)	-79.0(2)
C(5)	Ti(1)	C(3)	C(2)	80.4(2)
C(4)	Ti(1)	C(5)	C(1)	-115.9(4)
Ti(1)	N(1)	C(6)	N(2)	113.7(4)
C(6)	N(2)	C(7)	C(8)	-18.0(2)
C(7)	N(2)	C(6)	N(3)	6.6(2)
C(6)	N(2)	C(9)	C(14)	148.0(2)

Table 8. Torsion angles (°) (continued)

atom1	atom2	atom3	atom4	angle
C(9)	N(2)	C(6)	N(1)	-14.9(3)
C(7)	N(2)	C(9)	C(10)	68.3(2)
C(9)	N(2)	C(7)	C(8)	-175.3(2)
C(8)	N(3)	C(6)	N(1)	-172.1(2)
C(6)	N(3)	C(15)	C(16)	131.5(2)
C(15)	N(3)	C(6)	N(1)	-16.0(3)
C(8)	N(3)	C(15)	C(16)	-75.3(2)
C(15)	N(3)	C(8)	C(7)	-175.41(19)
Ti(1)	C(1)	C(5)	C(4)	-63.6(2)
C(2)	C(1)	C(5)	C(4)	0.0(3)
C(5)	C(1)	C(2)	C(3)	1.4(3)
C(1)	C(2)	C(3)	Ti(1)	-66.6(2)
Ti(1)	C(3)	C(4)	C(5)	67.1(2)
C(2)	C(3)	C(4)	C(5)	2.4(3)
C(3)	C(4)	C(5)	Ti(1)	-66.4(2)
N(2)	C(7)	C(8)	N(3)	21.3(2)
N(2)	C(9)	C(14)	C(13)	-176.92(17)
C(14)	C(9)	C(10)	C(11)	-56.3(2)
C(10)	C(11)	C(12)	C(13)	-55.5(2)
C(12)	C(13)	C(14)	C(9)	-57.2(2)
N(3)	C(15)	C(20)	C(19)	175.08(18)
C(20)	C(15)	C(16)	C(17)	58.6(2)
C(16)	C(17)	C(18)	C(19)	54.0(2)
C(18)	C(19)	C(20)	C(15)	56.5(2)
C(9)	N(2)	C(6)	N(3)	164.36(19)
C(7)	N(2)	C(9)	C(14)	-57.4(2)
C(6)	N(3)	C(8)	C(7)	-19.1(2)
C(8)	N(3)	C(6)	N(2)	8.6(2)
C(6)	N(3)	C(15)	C(20)	-103.0(2)
C(15)	N(3)	C(6)	N(2)	164.79(19)
C(8)	N(3)	C(15)	C(20)	50.2(2)
Ti(1)	C(1)	C(2)	C(3)	65.7(2)
C(2)	C(1)	C(5)	Ti(1)	63.7(2)
C(5)	C(1)	C(2)	Ti(1)	-64.3(2)
Ti(1)	C(2)	C(3)	C(4)	64.2(2)
C(1)	C(2)	C(3)	C(4)	-2.4(3)
C(2)	C(3)	C(4)	Ti(1)	-64.7(2)
Ti(1)	C(4)	C(5)	C(1)	64.9(2)
C(3)	C(4)	C(5)	C(1)	-1.5(3)
N(2)	C(9)	C(10)	C(11)	178.49(17)
C(10)	C(9)	C(14)	C(13)	57.3(2)
C(9)	C(10)	C(11)	C(12)	54.8(2)
C(11)	C(12)	C(13)	C(14)	57.0(2)
N(3)	C(15)	C(16)	C(17)	-175.04(17)
C(16)	C(15)	C(20)	C(19)	-58.9(2)
C(15)	C(16)	C(17)	C(18)	-55.7(2)
C(17)	C(18)	C(19)	C(20)	-54.7(2)

The sign is positive if when looking from atom 2 to atom 3 a clock-wise motion of atom 1 would superimpose it on atom 4.

Table 9. Distances beyond the asymmetric unit out to 3.60 Å

atom	atom	distance	atom	atom	distance
C(5)	C(11)[1)]	3.575(4)	C(11)	C(5)[2)]	3.575(4)

Symmetry Operators:

(1) -X,Y+1/2,-Z+1/2+1

(2) -X,Y+1/2-1,-Z+1/2+1

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
Cl(1)	H(2)[1)]	3.171	Cl(1)	H(6)[2)]	3.255
Cl(1)	H(8)[3)]	2.833	Cl(1)	H(9)[2)]	2.959
Cl(1)	H(20)[2)]	3.477	Cl(1)	H(22)[3)]	2.971
Cl(1)	H(30)[2)]	3.520	Cl(2)	H(7)[3)]	2.903
Cl(2)	H(8)[3)]	3.132	Cl(2)	H(15)[4)]	3.235
Cl(2)	H(16)[4)]	3.334	Cl(2)	H(24)[5)]	3.564
Cl(2)	H(28)[2)]	3.444	Cl(2)	H(30)[2)]	3.163
N(1)	H(9)[2)]	2.996	N(1)	H(31)[2)]	3.222
C(1)	H(13)[1)]	3.548	C(1)	H(19)[6)]	3.399
C(1)	H(20)[6)]	3.431	C(1)	H(24)[5)]	3.463
C(1)	H(29)[5)]	3.166	C(2)	H(20)[6)]	3.068
C(2)	H(25)[7)]	3.558	C(2)	H(27)[7)]	3.340
C(3)	H(18)[6)]	2.935	C(3)	H(20)[6)]	3.209
C(3)	H(25)[7)]	3.452	C(3)	H(26)[7)]	3.598
C(3)	H(27)[7)]	3.009	C(4)	H(18)[6)]	2.736
C(5)	H(13)[1)]	3.045	C(5)	H(14)[1)]	3.172
C(5)	H(18)[6)]	3.387	C(5)	H(19)[6)]	3.393
C(6)	H(9)[2)]	3.444	C(7)	H(16)[8)]	3.157
C(7)	H(29)[9)]	3.492	C(7)	H(30)[9)]	3.253
C(8)	H(30)[9)]	3.096	C(9)	H(31)[2)]	3.493
C(10)	H(5)[5)]	3.507	C(10)	H(25)[7)]	3.349
C(11)	H(5)[5)]	2.876	C(11)	H(15)[4)]	3.590
C(11)	H(19)[4)]	3.440	C(11)	H(28)[10)]	3.183
C(12)	H(7)[4)]	3.489	C(12)	H(12)[4)]	3.459
C(12)	H(17)[8)]	3.572	C(12)	H(19)[4)]	3.512
C(12)	H(28)[10)]	2.897	C(13)	H(12)[4)]	3.391
C(13)	H(15)[4)]	3.567	C(13)	H(27)[2)]	3.085
C(13)	H(28)[2)]	3.529	C(14)	H(27)[2)]	3.118
C(17)	H(1)[1)]	3.257	C(17)	H(3)[11)]	3.392
C(17)	H(11)[11)]	3.578	C(18)	H(1)[1)]	3.239
C(18)	H(3)[11)]	3.028	C(18)	H(13)[12)]	3.384
C(18)	H(17)[9)]	3.176	C(18)	H(20)[9)]	3.369
C(19)	H(1)[1)]	3.177	C(19)	H(6)[2)]	3.236
C(19)	H(13)[12)]	3.054	C(19)	H(15)[12)]	3.379
C(19)	H(16)[12)]	3.494	C(19)	H(17)[9)]	3.281
C(20)	H(6)[2)]	3.457	C(20)	H(10)[9)]	3.534
H(1)	C(17)[5)]	3.257	H(1)	C(18)[5)]	3.239

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(1)	C(19)$^{5)}$	3.177	H(1)	H(4)$^{5)}$	3.119
H(1)	H(13)$^{1)}$	3.422	H(1)	H(21)$^{5)}$	3.553
H(1)	H(24)$^{5)}$	2.493	H(1)	H(26)$^{5)}$	2.917
H(1)	H(29)$^{5)}$	2.381	H(2)	Cl(1)$^{5)}$	3.171
H(2)	H(4)$^{5)}$	3.453	H(2)	H(5)$^{5)}$	2.965
H(2)	H(20)$^{6)}$	3.272	H(2)	H(22)$^{7)}$	3.277
H(2)	H(25)$^{7)}$	3.202	H(2)	H(27)$^{7)}$	3.358
H(3)	C(17)$^{7)}$	3.392	H(3)	C(18)$^{7)}$	3.028
H(3)	H(18)$^{6)}$	3.129	H(3)	H(20)$^{6)}$	3.570
H(3)	H(25)$^{7)}$	2.799	H(3)	H(26)$^{7)}$	2.777
H(3)	H(27)$^{7)}$	2.601	H(4)	H(1)$^{1)}$	3.119
H(4)	H(2)$^{1)}$	3.453	H(4)	H(18)$^{6)}$	2.800
H(5)	C(10)$^{1)}$	3.507	H(5)	C(11)$^{1)}$	2.876
H(5)	H(2)$^{1)}$	2.965	H(5)	H(11)$^{1)}$	3.086
H(5)	H(13)$^{1)}$	2.359	H(5)	H(14)$^{1)}$	2.617
H(5)	H(22)$^{3)}$	3.521	H(5)	H(26)$^{5)}$	3.461
H(6)	Cl(1)$^{9)}$	3.255	H(6)	C(19)$^{9)}$	3.236
H(6)	C(20)$^{9)}$	3.457	H(6)	H(16)$^{8)}$	2.938
H(6)	H(28)$^{9)}$	3.296	H(6)	H(29)$^{9)}$	2.592
H(6)	H(30)$^{9)}$	2.855	H(7)	Cl(2)$^{13)}$	2.903
H(7)	C(12)$^{8)}$	3.489	H(7)	H(16)$^{8)}$	2.535
H(7)	H(25)$^{7)}$	3.514	H(7)	H(30)$^{9)}$	3.369
H(8)	Cl(1)$^{13)}$	2.833	H(8)	Cl(2)$^{13)}$	3.132
H(8)	H(30)$^{9)}$	2.819	H(9)	Cl(1)$^{9)}$	2.959
H(9)	N(1)$^{9)}$	2.996	H(9)	C(6)$^{9)}$	3.444
H(9)	H(21)$^{9)}$	3.506	H(9)	H(30)$^{9)}$	2.893
H(10)	C(20)$^{2)}$	3.534	H(10)	H(27)$^{2)}$	3.573
H(10)	H(28)$^{2)}$	3.187	H(10)	H(31)$^{2)}$	2.775
H(11)	C(17)$^{7)}$	3.578	H(11)	H(5)$^{5)}$	3.086
H(11)	H(25)$^{7)}$	2.649	H(12)	C(12)$^{8)}$	3.459
H(12)	C(13)$^{8)}$	3.391	H(12)	H(16)$^{8)}$	2.735
H(12)	H(17)$^{8)}$	2.893	H(12)	H(18)$^{8)}$	3.347
H(12)	H(25)$^{7)}$	3.146	H(13)	C(1)$^{5)}$	3.548
H(13)	C(5)$^{5)}$	3.045	H(13)	C(18)$^{10)}$	3.384
H(13)	C(19)$^{10)}$	3.054	H(13)	H(1)$^{5)}$	3.422
H(13)	H(5)$^{5)}$	2.359	H(13)	H(17)$^{8)}$	3.445
H(13)	H(19)$^{4)}$	3.438	H(13)	H(26)$^{10)}$	2.799

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(13)	H(28)$^{10)}$	2.577	H(13)	H(29)$^{10)}$	2.875
H(14)	C(5)$^{5)}$	3.172	H(14)	H(5)$^{5)}$	2.617
H(14)	H(15)$^{4)}$	2.738	H(14)	H(18)$^{4)}$	3.595
H(14)	H(19)$^{4)}$	2.899	H(15)	Cl(2)$^{8)}$	3.235
H(15)	C(11)$^{8)}$	3.590	H(15)	C(13)$^{8)}$	3.567
H(15)	C(19)$^{10)}$	3.379	H(15)	H(14)$^{8)}$	2.738
H(15)	H(16)$^{8)}$	3.480	H(15)	H(17)$^{8)}$	2.707
H(15)	H(28)$^{10)}$	2.421	H(16)	Cl(2)$^{8)}$	3.334
H(16)	C(7)$^{4)}$	3.157	H(16)	C(19)$^{10)}$	3.494
H(16)	H(6)$^{4)}$	2.938	H(16)	H(7)$^{4)}$	2.535
H(16)	H(12)$^{4)}$	2.735	H(16)	H(15)$^{4)}$	3.480
H(16)	H(19)$^{4)}$	2.794	H(16)	H(28)$^{10)}$	2.772
H(16)	H(29)$^{10)}$	3.312	H(17)	C(12)$^{4)}$	3.572
H(17)	C(18)$^{2)}$	3.176	H(17)	C(19)$^{2)}$	3.281
H(17)	H(12)$^{4)}$	2.893	H(17)	H(13)$^{4)}$	3.445
H(17)	H(15)$^{4)}$	2.707	H(17)	H(26)$^{2)}$	3.205
H(17)	H(27)$^{2)}$	2.622	H(17)	H(28)$^{2)}$	2.599
H(18)	C(3)$^{14)}$	2.935	H(18)	C(4)$^{14)}$	2.736
H(18)	C(5)$^{14)}$	3.387	H(18)	H(3)$^{14)}$	3.129
H(18)	H(4)$^{14)}$	2.800	H(18)	H(12)$^{4)}$	3.347
H(18)	H(14)$^{8)}$	3.595	H(18)	H(27)$^{2)}$	3.080
H(19)	C(1)$^{14)}$	3.399	H(19)	C(5)$^{14)}$	3.393
H(19)	C(11)$^{8)}$	3.440	H(19)	C(12)$^{8)}$	3.512
H(19)	H(13)$^{8)}$	3.438	H(19)	H(14)$^{8)}$	2.899
H(19)	H(16)$^{8)}$	2.794	H(20)	Cl(1)$^{9)}$	3.477
H(20)	C(1)$^{14)}$	3.431	H(20)	C(2)$^{14)}$	3.068
H(20)	C(3)$^{14)}$	3.209	H(20)	C(18)$^{2)}$	3.369
H(20)	H(2)$^{14)}$	3.272	H(20)	H(3)$^{14)}$	3.570
H(20)	H(27)$^{2)}$	2.412	H(20)	H(31)$^{2)}$	3.184
H(21)	H(1)$^{1)}$	3.553	H(21)	H(9)$^{2)}$	3.506
H(22)	Cl(1)$^{13)}$	2.971	H(22)	H(2)$^{11)}$	3.277
H(22)	H(5)$^{13)}$	3.521	H(23)	H(26)$^{7)}$	2.925
H(24)	Cl(2)$^{1)}$	3.564	H(24)	C(1)$^{1)}$	3.463
H(24)	H(1)$^{1)}$	2.493	H(25)	C(2)$^{11)}$	3.558
H(25)	C(3)$^{11)}$	3.452	H(25)	C(10)$^{11)}$	3.349
H(25)	H(2)$^{11)}$	3.202	H(25)	H(3)$^{11)}$	2.799
H(25)	H(7)$^{11)}$	3.514	H(25)	H(11)$^{11)}$	2.649

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(25)	H(12)$^{11)}$	3.146	H(26)	C(3)$^{11)}$	3.598
H(26)	H(1)$^{1)}$	2.917	H(26)	H(3)$^{11)}$	2.777
H(26)	H(5)$^{1)}$	3.461	H(26)	H(13)$^{12)}$	2.799
H(26)	H(17)$^{9)}$	3.205	H(26)	H(23)$^{11)}$	2.925
H(27)	C(2)$^{11)}$	3.340	H(27)	C(3)$^{11)}$	3.009
H(27)	C(13)$^{9)}$	3.085	H(27)	C(14)$^{9)}$	3.118
H(27)	H(2)$^{11)}$	3.358	H(27)	H(3)$^{11)}$	2.601
H(27)	H(10)$^{9)}$	3.573	H(27)	H(17)$^{9)}$	2.622
H(27)	H(18)$^{9)}$	3.080	H(27)	H(20)$^{9)}$	2.412
H(28)	Cl(2)$^{9)}$	3.444	H(28)	C(11)$^{12)}$	3.183
H(28)	C(12)$^{12)}$	2.897	H(28)	C(13)$^{9)}$	3.529
H(28)	H(6)$^{2)}$	3.296	H(28)	H(10)$^{9)}$	3.187
H(28)	H(13)$^{12)}$	2.577	H(28)	H(15)$^{12)}$	2.421
H(28)	H(16)$^{12)}$	2.772	H(28)	H(17)$^{9)}$	2.599
H(29)	C(1)$^{1)}$	3.166	H(29)	C(7)$^{2)}$	3.492
H(29)	H(1)$^{1)}$	2.381	H(29)	H(6)$^{2)}$	2.592
H(29)	H(13)$^{12)}$	2.875	H(29)	H(16)$^{12)}$	3.312
H(30)	Cl(1)$^{9)}$	3.520	H(30)	Cl(2)$^{9)}$	3.163
H(30)	C(7)$^{2)}$	3.253	H(30)	C(8)$^{2)}$	3.096
H(30)	H(6)$^{2)}$	2.855	H(30)	H(7)$^{2)}$	3.369
H(30)	H(8)$^{2)}$	2.819	H(30)	H(9)$^{2)}$	2.893
H(31)	N(1)$^{9)}$	3.222	H(31)	C(9)$^{9)}$	3.493
H(31)	H(10)$^{9)}$	2.775	H(31)	H(20)$^{9)}$	3.184

Symmetry Operators:

(1) -X,Y+1/2,-Z+1/2+1
(2) X+1/2-1,-Y+1/2,-Z+1
(3) X-1,Y,Z
(4) X+1/2-1,-Y+1/2-1,-Z+1
(5) -X,Y+1/2-1,-Z+1/2+1
(6) -X+1/2,-Y,Z+1/2
(7) -X+1,Y+1/2-1,-Z+1/2+1
(8) X+1/2,-Y+1/2-1,-Z+1
(9) X+1/2,-Y+1/2,-Z+1
(10) X,Y-1,Z
(11) -X+1,Y+1/2,-Z+1/2+1
(12) X,Y+1,Z
(13) X+1,Y,Z
(14) -X+1/2,-Y,Z+1/2-1

Electronic Supplementary Material (ESI) for Dalton Transactions

X-ray Structure Report for $CpTiCl_2[1,3\text{-}^tBu_2(CH_2N)_2C{=}N]$ (**1d**)

August 27, 2010

Experimental

Data Collection

A yellow block crystal of $C_{16}H_{27}Cl_2N_3Ti$ having approximate dimensions of 0.48 x 0.16 x 0.16 mm was mounted on a glass fiber. All measurements were made on a Rigaku RAXIS RAPID imaging plate area detector with graphite monochromated Mo-Kα radiation.

Indexing was performed from 3 oscillations that were exposed for 60 seconds. The crystal-to-detector distance was 127.40 mm.

Cell constants and an orientation matrix for data collection corresponded to a primitive orthorhombic cell with dimensions:

a = 12.8021(3) Å
b = 17.5227(4) Å
c = 8.7440(2) Å
V = 1961.53(8) Å^3

For Z = 4 and F.W. = 380.22, the calculated density is 1.287 g/cm^3. Based on the systematic absences of:

0kl: k+l ± 2n
hk0: h ± 2n

packing considerations, a statistical analysis of intensity distribution, and the successful solution and refinement of the structure, the space group was determined to be:

Pnma (#62)

The data were collected at a temperature of -150 ± 1°C to a maximum 2θ value of 54.9°. A total of 74 oscillation images were collected. A sweep of data was done using ω scans from 130.0 to 190.0° in 3.0° step, at χ=45.0° and ϕ = 0.0°. The exposure rate was 50.0 [sec./°]. A second sweep was performed using ω scans from 0.0 to 162.0° in 3.0° step, at χ=45.0° and ϕ = 180.0°. The exposure rate was 50.0 [sec./°]. The crystal-to-detector distance was 127.40 mm. Readout was performed in the 0.100 mm pixel mode.

Data Reduction

Of the 18393 reflections that were collected, 2310 were unique (R_{int} = 0.021); equivalent reflections were merged.

The linear absorption coefficient, μ, for Mo-Kα radiation is 7.086 cm^{-1}. An empirical absorption correction was applied which resulted in transmission factors ranging from 0.736 to 0.893. The data were corrected for Lorentz and polarization effects.

Structure Solution and Refinement

The structure was solved by direct methods[1] and expanded using Fourier techniques[2]. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were refined using the riding model. The final cycle of full-matrix least-squares refinement[3] on F^2 was based on 2310 observed reflections and 107 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R1 = \Sigma \; ||Fo| - |Fc|| \; / \; \Sigma \; |Fo| = 0.0369$$

$$wR2 = [\; \Sigma \; (\; w \; (Fo^2 - Fc^2)^2 \;) / \; \Sigma \; w(Fo^2)^2]^{1/2} = 0.0993$$

The standard deviation of an observation of unit weight[4] was 1.07. Unit weights were used. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.65 and -0.53 $e^-/Å^3$, respectively.

Neutral atom scattering factors were taken from Cromer and Waber[5]. Anomalous dispersion effects were included in Fcalc[6]; the values for Δf' and Δf'' were those of Creagh and McAuley[7]. The values for the mass attenuation coefficients are those of Creagh and Hubbell[8]. All calculations were performed using the CrystalStructure[9] crystallographic software package except for refinement, which was performed using SHELXL-97[10].

References

(1) SIR92: Altomare, A., Cascarano, G., Giacovazzo, C., Guagliardi, A., Burla, M., Polidori, G., and Camalli, M. (1994) J. Appl. Cryst., 27, 435.

(2) DIRDIF99: Beurskens, P.T., Admiraal, G., Beurskens, G., Bosman, W.P., de Gelder, R., Israel, R. and Smits, J.M.M.(1999). The DIRDIF-99 program system, Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.

(3) Least Squares function minimized: (SHELXL97)

$\Sigma w(F_o^2-F_c^2)^2$ where w = Least Squares weights.

(4) Standard deviation of an observation of unit weight:

$[\Sigma w(F_o^2-F_c^2)^2/(N_o-N_v)]^{1/2}$

where: N_o = number of observations
N_v = number of variables

(5) Cromer, D. T. & Waber, J. T.; "International Tables for X-ray Crystallography", Vol. IV, The Kynoch Press, Birmingham, England, Table 2.2 A (1974).

(6) Ibers, J. A. & Hamilton, W. C.; Acta Crystallogr., 17, 781 (1964).

(7) Creagh, D. C. & McAuley, W.J .; "International Tables for Crystallography", Vol C, (A.J.C. Wilson, ed.), Kluwer Academic Publishers, Boston, Table 4.2.6.8, pages 219-222 (1992).

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(9) CrystalStructure 3.8: Crystal Structure Analysis Package, Rigaku and Rigaku Americas (2000-2007). 9009 New Trails Dr. The Woodlands TX 77381 USA.

(10) SHELX97: Sheldrick, G.M. (1997).

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	$C_{16}H_{27}Cl_2N_3Ti$
Formula Weight	380.22
Crystal Color, Habit	yellow, block
Crystal Dimensions	0.48 X 0.16 X 0.16 mm
Crystal System	orthorhombic
Lattice Type	Primitive
Indexing Images	3 oscillations @ 60.0 seconds
Detector Position	127.40 mm
Pixel Size	0.100 mm
Lattice Parameters	a = 12.8021(3) Å b = 17.5227(4) Å c = 8.7440(2) Å V = 1961.53(8) Å^3
Space Group	Pnma (#62)
Z value	4
D_{calc}	1.287 g/cm^3
F_{000}	800.00
μ(MoKα)	7.086 cm^{-1}

B. Intensity Measurements

Diffractometer	Rigaku RAXIS-RAPID
Radiation	MoKα (λ = 0.71075 Å) graphite monochromated
Detector Aperture	280 mm x 256 mm
Data Images	74 exposures
ω oscillation Range (χ=45.0, ϕ=0.0)	130.0 - 190.0^{o}
Exposure Rate	50.0 sec./^{o}
ω oscillation Range (χ=45.0, ϕ=180.0)	0.0 - 162.0^{o}
Exposure Rate	50.0 sec./^{o}
Detector Position	127.40 mm
Pixel Size	0.100 mm
$2\theta_{max}$	54.9^{o}
No. of Reflections Measured	Total: 18393 Unique: 2310 (R_{int} = 0.021)
Corrections	Lorentz-polarization Absorption (trans. factors: 0.736 - 0.893)

C. Structure Solution and Refinement

Structure Solution	Direct Methods (SIR92)
Refinement	Full-matrix least-squares on F^2
Function Minimized	Σ w $(Fo^2 - Fc^2)^2$
Least Squares Weights	$w = 1/ [\sigma^2(Fo^2) + (0.0496 \cdot P)^2 + 1.9236 \cdot P]$ where $P = (Max(Fo^2,0) + 2Fc^2)/3$
$2\theta_{max}$ cutoff	54.9^o
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (All reflections)	2310
No. Variables	107
Reflection/Parameter Ratio	21.59
Residuals: R1 (I>2.00σ(I))	0.0369
Residuals: R (All reflections)	0.0384
Residuals: wR2 (All reflections)	0.0993
Goodness of Fit Indicator	1.070
Max Shift/Error in Final Cycle	0.001
Maximum peak in Final Diff. Map	0.65 $e^-/Å^3$
Minimum peak in Final Diff. Map	-0.53 $e^-/Å^3$

Table 1. Atomic coordinates and B_{iso}/B_{eq} and occupancy

atom	x	y	z	B_{eq}	occ
Ti(1)	0.52564(3)	0.2500	0.67414(4)	1.472(10)	1/2
Cl(1)	0.63592(3)	0.35164(3)	0.62534(5)	2.264(11)	
N(1)	0.43722(15)	0.2500	0.5167(2)	1.55(3)	1/2
N(2)	0.31548(14)	0.18710(9)	0.3545(2)	2.83(3)	
C(1)	0.3860(3)	0.2500	0.8469(4)	4.72(9)	1/2
C(2)	0.4441(2)	0.31288(18)	0.8792(2)	5.23(7)	
C(3)	0.5396(2)	0.2889(2)	0.9368(2)	5.28(7)	
C(4)	0.36136(17)	0.2500	0.4144(3)	1.43(3)	1/2
C(5)	0.24340(14)	0.20718(10)	0.2338(2)	2.08(2)	
C(6)	0.35009(16)	0.10620(10)	0.3742(2)	2.78(3)	
C(7)	0.45925(19)	0.09597(12)	0.3072(2)	3.16(3)	
C(8)	0.34869(18)	0.08373(12)	0.5419(3)	3.57(4)	
C(9)	0.2725(2)	0.05498(13)	0.2897(5)	7.94(13)	

$B_{eq} = 8/3\ \pi^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos\gamma + 2U_{13}(aa^*cc^*)\cos\beta + 2U_{23}(bb^*cc^*)\cos\alpha)$

Table 2. Atomic coordinates and B_{iso} involving hydrogens/B_{eq} and occupancy

atom	x	y	z	B_{eq}	occ
H(1)	0.3097	0.2500	0.8005	1.80	1/2
H(2)	0.4081	0.3586	0.8444	1.80	
H(3)	0.6036	0.3232	0.9701	1.80	
H(4)	0.2669	0.1878	0.1379	2.54	
H(5)	0.1753	0.1878	0.2541	2.54	
H(6)	0.4792	0.0420	0.3127	3.79	
H(7)	0.5092	0.1267	0.3657	3.79	
H(8)	0.4594	0.1126	0.2002	3.79	
H(9)	0.3592	0.0285	0.5511	4.28	
H(10)	0.2812	0.0976	0.5869	4.28	
H(11)	0.4048	0.1105	0.5960	4.28	
H(12)	0.2938	0.0015	0.3005	9.52	
H(13)	0.2713	0.0688	0.1811	9.52	
H(14)	0.2026	0.0618	0.3333	9.52	

$$B_{eq} = 8/3\ \pi^2(U_{11}(aa^*)^2 + U_{22}(bb^*)^2 + U_{33}(cc^*)^2 + 2U_{12}(aa^*bb^*)\cos\gamma + 2U_{13}(aa^*cc^*)\cos\beta + 2U_{23}(bb^*cc^*)\cos\alpha)$$

Table 3. Anisotropic displacement parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Ti(1)	0.0130(2)	0.0304(2)	0.0126(2)	0.0000	-0.00106(14)	0.0000
Cl(1)	0.0242(2)	0.0326(2)	0.0292(2)	-0.00700(16)	-0.00150(16)	-0.00301(17)
N(1)	0.0190(9)	0.0229(9)	0.0171(8)	0.0000	-0.0040(7)	0.0000
N(2)	0.0389(9)	0.0174(7)	0.0512(10)	-0.0008(6)	-0.0304(8)	-0.0002(6)
C(1)	0.0269(14)	0.129(4)	0.0232(14)	0.0000	0.0102(12)	0.0000
C(2)	0.103(2)	0.0694(18)	0.0264(11)	0.0316(18)	0.0278(13)	-0.0027(11)
C(3)	0.0578(14)	0.126(2)	0.0169(9)	-0.0389(15)	0.0071(9)	-0.0204(11)
C(4)	0.0180(10)	0.0197(10)	0.0167(9)	0.0000	-0.0019(8)	0.0000
C(5)	0.0295(8)	0.0274(8)	0.0222(8)	-0.0038(7)	-0.0113(6)	-0.0008(6)
C(6)	0.0354(10)	0.0164(7)	0.0539(12)	-0.0015(7)	-0.0216(9)	0.0011(7)
C(7)	0.0619(14)	0.0286(9)	0.0296(9)	0.0072(9)	0.0061(9)	-0.0023(7)
C(8)	0.0389(10)	0.0283(9)	0.0683(15)	0.0028(8)	0.0203(10)	0.0204(10)
C(9)	0.098(2)	0.0192(10)	0.184(4)	-0.0016(12)	-0.102(2)	-0.0099(16)

The general temperature factor expression: $\exp(-2\pi^2(a^{*2}U_{11}h^2 + b^{*2}U_{22}k^2 + c^{*2}U_{33}l^2 + 2a^*b^*U_{12}hk + 2a^*c^*U_{13}hl + 2b^*c^*U_{23}kl))$

Table 4. Bond lengths (Å)

atom	atom	distance	atom	atom	distance
Ti(1)	Cl(1)	2.3124(5)	Ti(1)	Cl(1)[1)]	2.3124(5)
Ti(1)	N(1)	1.7823(18)	Ti(1)	C(1)	2.340(3)
Ti(1)	C(2)	2.350(2)	Ti(1)	C(2)[1)]	2.350(2)
Ti(1)	C(3)	2.403(2)	Ti(1)	C(3)[1)]	2.403(2)
N(1)	C(4)	1.320(3)	N(2)	C(4)	1.354(2)
N(2)	C(5)	1.446(2)	N(2)	C(6)	1.495(2)
C(1)	C(2)	1.359(3)	C(1)	C(2)[1)]	1.359(3)
C(2)	C(3)	1.387(4)	C(3)	C(3)[1)]	1.364(4)
C(5)	C(5)[1)]	1.501(2)	C(6)	C(7)	1.526(3)
C(6)	C(8)	1.519(3)	C(6)	C(9)	1.529(4)

Symmetry Operators:

(1) X,-Y+1/2,Z

Table 5. Bond lengths involving hydrogens (Å)

atom	atom	distance	atom	atom	distance
C(5)	H(4)	0.953	C(5)	H(5)	0.952
C(7)	H(6)	0.980	C(7)	H(7)	0.980
C(7)	H(8)	0.980	C(8)	H(9)	0.980
C(8)	H(10)	0.980	C(8)	H(11)	0.980
C(9)	H(12)	0.980	C(9)	H(13)	0.980
C(9)	H(14)	0.980			

Table 6. Bond angles (°)

atom	atom	atom	angle	atom	atom	atom	angle
Cl(1)	Ti(1)	Cl(1)[1)]	100.74(2)	Cl(1)	Ti(1)	N(1)	104.20(3)
Cl(1)	Ti(1)	C(1)	125.83(3)	Cl(1)	Ti(1)	C(2)	92.92(8)
Cl(1)	Ti(1)	C(2)[1)]	140.66(7)	Cl(1)	Ti(1)	C(3)	84.98(7)
Cl(1)	Ti(1)	C(3)[1)]	110.47(7)	Cl(1)[1)]	Ti(1)	N(1)	104.20(3)
Cl(1)[1)]	Ti(1)	C(1)	125.83(3)	Cl(1)[1)]	Ti(1)	C(2)	140.66(7)
Cl(1)[1)]	Ti(1)	C(2)[1)]	92.92(8)	Cl(1)[1)]	Ti(1)	C(3)	110.47(7)
Cl(1)[1)]	Ti(1)	C(3)[1)]	84.98(7)	N(1)	Ti(1)	C(1)	90.77(10)
N(1)	Ti(1)	C(2)	107.89(9)	N(1)	Ti(1)	C(2)[1)]	107.89(9)
N(1)	Ti(1)	C(3)	141.77(8)	N(1)	Ti(1)	C(3)[1)]	141.77(8)
C(1)	Ti(1)	C(2)	33.68(9)	C(1)	Ti(1)	C(2)[1)]	33.68(9)
C(1)	Ti(1)	C(3)	55.92(10)	C(1)	Ti(1)	C(3)[1)]	55.92(10)
C(2)	Ti(1)	C(2)[1)]	55.93(10)	C(2)	Ti(1)	C(3)	33.92(10)
C(2)	Ti(1)	C(3)[1)]	55.70(10)	C(2)[1)]	Ti(1)	C(3)	55.70(10)
C(2)[1)]	Ti(1)	C(3)[1)]	33.92(10)	C(3)	Ti(1)	C(3)[1)]	32.98(11)
Ti(1)	N(1)	C(4)	172.08(17)	C(4)	N(2)	C(5)	111.17(15)
C(4)	N(2)	C(6)	126.77(17)	C(5)	N(2)	C(6)	120.26(16)
Ti(1)	C(1)	C(2)	73.5(2)	Ti(1)	C(1)	C(2)[1)]	73.5(2)
C(2)	C(1)	C(2)[1)]	108.4(3)	Ti(1)	C(2)	C(1)	72.79(19)
Ti(1)	C(2)	C(3)	75.14(17)	C(1)	C(2)	C(3)	108.2(2)
Ti(1)	C(3)	C(2)	70.94(14)	Ti(1)	C(3)	C(3)[1)]	73.51(14)
C(2)	C(3)	C(3)[1)]	107.6(2)	N(1)	C(4)	N(2)	125.52(11)
N(1)	C(4)	N(2)[1)]	125.52(11)	N(2)	C(4)	N(2)[1)]	108.95(19)
N(2)	C(5)	C(5)[1)]	104.09(14)	N(2)	C(6)	C(7)	109.79(16)
N(2)	C(6)	C(8)	110.70(17)	N(2)	C(6)	C(9)	107.95(18)
C(7)	C(6)	C(8)	110.55(17)	C(7)	C(6)	C(9)	109.9(2)
C(8)	C(6)	C(9)	107.9(2)				

Symmetry Operators:

(1) X,-Y+1/2,Z

Table 7. Bond angles involving hydrogens (°)

atom	atom	atom	angle	atom	atom	atom	angle
Ti(1)	C(1)	H(1)	117.2	C(2)	C(1)	H(1)	125.8
C(2)[1)]	C(1)	H(1)	125.8	Ti(1)	C(2)	H(2)	111.0
C(1)	C(2)	H(2)	110.1	C(3)	C(2)	H(2)	141.3
Ti(1)	C(3)	H(3)	118.8	C(2)	C(3)	H(3)	127.7
C(3)[1)]	C(3)	H(3)	124.6	N(2)	C(5)	H(4)	110.7
N(2)	C(5)	H(5)	111.2	C(5)[1)]	C(5)	H(4)	110.9
C(5)[1)]	C(5)	H(5)	110.9	H(4)	C(5)	H(5)	109.0
C(6)	C(7)	H(6)	109.5	C(6)	C(7)	H(7)	109.5
C(6)	C(7)	H(8)	109.5	H(6)	C(7)	H(7)	109.5
H(6)	C(7)	H(8)	109.5	H(7)	C(7)	H(8)	109.5
C(6)	C(8)	H(9)	109.5	C(6)	C(8)	H(10)	109.5
C(6)	C(8)	H(11)	109.5	H(9)	C(8)	H(10)	109.5
H(9)	C(8)	H(11)	109.5	H(10)	C(8)	H(11)	109.5
C(6)	C(9)	H(12)	109.5	C(6)	C(9)	H(13)	109.5
C(6)	C(9)	H(14)	109.5	H(12)	C(9)	H(13)	109.5
H(12)	C(9)	H(14)	109.5	H(13)	C(9)	H(14)	109.5

Symmetry Operators:

(1) X,-Y+1/2,Z

Table 8. Torsion Angles(°)

atom1	atom2	atom3	atom4	angle	atom1	atom2	atom3	atom4	angle
Cl(1)	Ti(1)	N(1)	C(4)	-127.397(19)	Cl(1)	Ti(1)	C(1)	C(2)	-14.1(2)
Cl(1)	Ti(1)	C(1)	C(2)$^{1)}$	-129.54(14)	Cl(1)	Ti(1)	C(2)	C(1)	168.61(17)
Cl(1)	Ti(1)	C(2)	C(3)	-76.53(18)	Cl(1)	Ti(1)	C(2)$^{1)}$	C(3)$^{1)}$	-34.4(2)
Cl(1)	Ti(1)	C(2)$^{1)}$	C(1)	80.5(2)	Cl(1)	Ti(1)	C(3)	C(2)	102.85(18)
Cl(1)	Ti(1)	C(3)	C(3)$^{1)}$	-141.29(16)	Cl(1)	Ti(1)	C(3)$^{1)}$	C(2)$^{1)}$	157.54(16)
Cl(1)	Ti(1)	C(3)$^{1)}$	C(3)	41.68(17)	Cl(1)$^{1)}$	Ti(1)	N(1)	C(4)	127.397(19)
Cl(1)$^{1)}$	Ti(1)	C(1)	C(2)	129.54(14)	Cl(1)$^{1)}$	Ti(1)	C(1)	C(2)$^{1)}$	14.1(2)
Cl(1)$^{1)}$	Ti(1)	C(2)	C(1)	-80.5(2)	Cl(1)$^{1)}$	Ti(1)	C(2)	C(3)	34.4(2)
Cl(1)$^{1)}$	Ti(1)	C(2)$^{1)}$	C(3)$^{1)}$	76.53(18)	Cl(1)$^{1)}$	Ti(1)	C(2)$^{1)}$	C(1)	-168.61(17)
Cl(1)$^{1)}$	Ti(1)	C(3)	C(2)	-157.54(16)	Cl(1)$^{1)}$	Ti(1)	C(3)	C(3)$^{1)}$	-41.68(17)
Cl(1)$^{1)}$	Ti(1)	C(3)$^{1)}$	C(2)$^{1)}$	-102.85(18)	Cl(1)$^{1)}$	Ti(1)	C(3)$^{1)}$	C(3)	141.29(16)
N(1)	Ti(1)	C(1)	C(2)	-122.27(17)	N(1)	Ti(1)	C(1)	C(2)$^{1)}$	122.27(17)
N(1)	Ti(1)	C(2)	C(1)	62.68(18)	N(1)	Ti(1)	C(2)	C(3)	177.54(17)
C(2)	Ti(1)	N(1)	C(4)	-29.52(8)	N(1)	Ti(1)	C(2)$^{1)}$	C(3)$^{1)}$	-177.54(17)
N(1)	Ti(1)	C(2)$^{1)}$	C(1)	-62.68(18)	C(2)$^{1)}$	Ti(1)	N(1)	C(4)	29.52(8)
N(1)	Ti(1)	C(3)	C(2)	-3.8(2)	N(1)	Ti(1)	C(3)	C(3)$^{1)}$	112.07(17)
C(3)	Ti(1)	N(1)	C(4)	-27.30(13)	N(1)	Ti(1)	C(3)$^{1)}$	C(2)$^{1)}$	3.8(2)
N(1)	Ti(1)	C(3)$^{1)}$	C(3)	-112.07(17)	C(3)$^{1)}$	Ti(1)	N(1)	C(4)	27.30(13)
C(1)	Ti(1)	C(2)	C(3)	114.9(2)	C(2)	Ti(1)	C(1)	C(2)$^{1)}$	-115.5(2)
C(1)	Ti(1)	C(2)$^{1)}$	C(3)$^{1)}$	-114.9(2)	C(2)$^{1)}$	Ti(1)	C(1)	C(2)	115.5(2)
C(1)	Ti(1)	C(3)	C(2)	-37.41(16)	C(1)	Ti(1)	C(3)	C(3)$^{1)}$	78.45(16)
C(3)	Ti(1)	C(1)	C(2)	37.69(19)	C(3)	Ti(1)	C(1)	C(2)$^{1)}$	-77.8(2)
C(1)	Ti(1)	C(3)$^{1)}$	C(2)$^{1)}$	37.41(16)	C(1)	Ti(1)	C(3)$^{1)}$	C(3)	-78.45(16)
C(3)$^{1)}$	Ti(1)	C(1)	C(2)	77.8(2)	C(3)$^{1)}$	Ti(1)	C(1)	C(2)$^{1)}$	-37.69(19)
C(2)	Ti(1)	C(2)$^{1)}$	C(3)$^{1)}$	-77.7(2)	C(2)	Ti(1)	C(2)$^{1)}$	C(1)	37.19(18)
C(2)$^{1)}$	Ti(1)	C(2)	C(1)	-37.19(18)	C(2)$^{1)}$	Ti(1)	C(2)	C(3)	77.7(2)
C(2)	Ti(1)	C(3)	C(3)$^{1)}$	115.9(2)	C(3)	Ti(1)	C(2)	C(1)	-114.9(2)
C(2)	Ti(1)	C(3)$^{1)}$	C(2)$^{1)}$	78.4(2)	C(2)	Ti(1)	C(3)$^{1)}$	C(3)	-37.44(17)
C(3)$^{1)}$	Ti(1)	C(2)	C(1)	-78.49(19)	C(3)$^{1)}$	Ti(1)	C(2)	C(3)	36.37(18)
C(2)$^{1)}$	Ti(1)	C(3)	C(2)	-78.4(2)	C(2)$^{1)}$	Ti(1)	C(3)	C(3)$^{1)}$	37.44(17)
C(3)	Ti(1)	C(2)$^{1)}$	C(3)$^{1)}$	-36.37(18)	C(3)	Ti(1)	C(2)$^{1)}$	C(1)	78.49(19)
C(2)$^{1)}$	Ti(1)	C(3)$^{1)}$	C(3)	-115.9(2)	C(3)$^{1)}$	Ti(1)	C(2)$^{1)}$	C(1)	114.9(2)
C(3)	Ti(1)	C(3)$^{1)}$	C(2)$^{1)}$	115.9(2)	C(3)$^{1)}$	Ti(1)	C(3)	C(2)	-115.9(2)
Ti(1)	N(1)	C(4)	N(2)	-89.3(2)	Ti(1)	N(1)	C(4)	N(2)$^{1)}$	89.3(2)
C(4)	N(2)	C(5)	C(5)$^{1)}$	-4.4(2)	C(5)	N(2)	C(4)	N(1)	-173.7(2)
C(5)	N(2)	C(4)	N(2)$^{1)}$	7.4(2)	C(4)	N(2)	C(6)	C(7)	-61.8(2)
C(4)	N(2)	C(6)	C(8)	60.5(2)	C(4)	N(2)	C(6)	C(9)	178.3(2)

Table 8. Torsion angles (°) (continued)

atom1	atom2	atom3	atom4	angle
C(6)	N(2)	C(4)	N(1)	-9.1(3)
C(5)	N(2)	C(6)	C(7)	101.5(2)
C(5)	N(2)	C(6)	C(9)	-18.3(2)
Ti(1)	C(1)	C(2)	C(3)	-67.39(19)
C(2)	C(1)	C(2)[1)]	Ti(1)	-65.8(2)
C(2)[1)]	C(1)	C(2)	Ti(1)	65.8(2)
Ti(1)	C(2)	C(3)	C(3)[1)]	-64.86(17)
C(1)	C(2)	C(3)	C(3)[1)]	1.0(3)
C(2)	C(3)	C(3)[1)]	Ti(1)	63.17(17)
C(6)	N(2)	C(4)	N(2)[1)]	172.01(19)
C(5)	N(2)	C(6)	C(8)	-136.16(18)
C(6)	N(2)	C(5)	C(5)[1)]	-170.17(16)
Ti(1)	C(1)	C(2)[1)]	C(3)[1)]	67.39(19)
C(2)	C(1)	C(2)[1)]	C(3)[1)]	1.6(3)
C(2)[1)]	C(1)	C(2)	C(3)	-1.6(3)
C(1)	C(2)	C(3)	Ti(1)	65.8(2)
Ti(1)	C(3)	C(3)[1)]	C(2)[1)]	-63.17(17)

Symmetry Operators:

(1) X,-Y+1/2,Z

The sign is positive if when looking from atom 2 to atom 3 a clock-wise motion of atom 1 would superimpose it on atom 4.

Table 9. Distances beyond the asymmetric unit out to 3.60 Å

atom	atom	distance	atom	atom	distance
Cl(1)	C(5)[1)]	3.5797(18)	C(5)	Cl(1)[2)]	3.5797(18)

Symmetry Operators:

(1) X+1/2,-Y+1/2,-Z+1/2

(2) X+1/2-1,-Y+1/2,-Z+1/2

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens

atom	atom	distance	atom	atom	distance
Ti(1)	H(1)	2.977	Ti(1)	H(2)	2.846
Ti(1)	H(2)[1)]	2.846	Ti(1)	H(3)	3.056
Ti(1)	H(3)[1)]	3.056	Cl(1)	H(1)[2)]	2.923
Cl(1)	H(2)	3.491	Cl(1)	H(2)[3)]	3.497
Cl(1)	H(3)	3.083	Cl(1)	H(4)[4)]	2.931
Cl(1)	H(5)[4)]	3.426	Cl(1)	H(9)[5)]	3.463
Cl(1)	H(10)[2)]	3.253	Cl(1)	H(12)[5)]	2.851
Cl(1)	H(13)[4)]	3.482	N(1)	H(1)	2.970
N(1)	H(2)	3.460	N(1)	H(2)[1)]	3.460
N(2)	H(3)[6)]	3.121	C(1)	H(1)	1.058
C(1)	H(2)	1.924	C(1)	H(2)[1)]	1.924
C(1)	H(3)	3.251	C(1)	H(3)[1)]	3.251
C(1)	H(4)[7)]	3.160	C(1)	H(4)[8)]	3.160
C(2)	H(1)	2.156	C(2)	H(2)	0.973
C(2)	H(2)[1)]	3.055	C(2)	H(3)	2.199
C(2)	H(3)[1)]	3.239	C(2)	H(4)[8)]	3.203
C(2)	H(8)[8)]	3.102	C(3)	H(1)	3.247
C(3)	H(2)	2.231	C(3)	H(2)[1)]	3.189
C(3)	H(3)	1.057	C(3)	H(3)[1)]	2.149
C(3)	H(5)[2)]	3.239	C(3)	H(8)[8)]	3.055
C(4)	H(1)	3.440	C(5)	H(3)[6)]	3.193
C(5)	H(7)[9)]	3.425	C(7)	H(5)[10)]	3.245
C(7)	H(9)[11)]	3.420	C(7)	H(14)[10)]	3.402
C(8)	H(2)[1)]	2.932	C(8)	H(3)[6)]	3.538
C(8)	H(6)[11)]	3.366	C(8)	H(12)[12)]	3.267
C(8)	H(13)[12)]	3.314	C(9)	H(9)[13)]	3.055
C(9)	H(10)[13)]	3.282	H(1)	Ti(1)	2.977
H(1)	Cl(1)[6)]	2.923	H(1)	Cl(1)[14)]	2.923
H(1)	N(1)	2.970	H(1)	C(1)	1.058
H(1)	C(2)	2.156	H(1)	C(2)[1)]	2.156
H(1)	C(3)	3.247	H(1)	C(3)[1)]	3.247
H(1)	C(4)	3.440	H(1)	H(2)	2.314
H(1)	H(2)[1)]	2.314	H(1)	H(4)[7)]	3.193
H(1)	H(4)[8)]	3.193	H(1)	H(10)	3.279
H(1)	H(10)[1)]	3.279	H(1)	H(11)	3.264
H(1)	H(11)[1)]	3.264	H(2)	Ti(1)	2.846
H(2)	Cl(1)	3.491	H(2)	Cl(1)[14)]	3.497

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(2)	N(1)	3.460	H(2)	C(1)	1.924
H(2)	C(2)	0.973	H(2)	C(2)[1)]	3.055
H(2)	C(3)	2.231	H(2)	C(3)[1)]	3.189
H(2)	C(8)[1)]	2.932	H(2)	H(1)	2.314
H(2)	H(3)	2.803	H(2)	H(4)[8)]	3.243
H(2)	H(8)[8)]	3.220	H(2)	H(9)[1)]	3.299
H(2)	H(10)[1)]	2.881	H(2)	H(11)[1)]	2.239
H(3)	Ti(1)	3.056	H(3)	Cl(1)	3.083
H(3)	N(2)[2)]	3.121	H(3)	C(1)	3.251
H(3)	C(2)	2.199	H(3)	C(2)[1)]	3.239
H(3)	C(3)	1.057	H(3)	C(3)[1)]	2.149
H(3)	C(5)[2)]	3.193	H(3)	C(8)[2)]	3.538
H(3)	H(2)	2.803	H(3)	H(3)[1)]	2.565
H(3)	H(5)[2)]	2.588	H(3)	H(5)[3)]	3.506
H(3)	H(8)[8)]	2.953	H(3)	H(10)[2)]	2.710
H(3)	H(14)[2)]	2.936	H(4)	Cl(1)[15)]	2.931
H(4)	C(1)[16)]	3.160	H(4)	C(2)[17)]	3.203
H(4)	H(1)[16)]	3.193	H(4)	H(2)[17)]	3.243
H(4)	H(7)[9)]	3.468	H(5)	Cl(1)[15)]	3.426
H(5)	C(3)[6)]	3.239	H(5)	C(7)[9)]	3.245
H(5)	H(3)[6)]	2.588	H(5)	H(3)[14)]	3.506
H(5)	H(7)[9)]	2.601	H(5)	H(8)[9)]	3.088
H(6)	C(8)[11)]	3.366	H(6)	H(9)[11)]	2.689
H(6)	H(11)[11)]	3.161	H(6)	H(14)[10)]	3.151
H(7)	C(5)[10)]	3.425	H(7)	H(4)[10)]	3.468
H(7)	H(5)[10)]	2.601	H(7)	H(9)[11)]	3.280
H(7)	H(13)[10)]	3.528	H(7)	H(14)[10)]	3.232
H(8)	C(2)[17)]	3.102	H(8)	C(3)[17)]	3.055
H(8)	H(2)[17)]	3.220	H(8)	H(3)[17)]	2.953
H(8)	H(5)[10)]	3.088	H(8)	H(14)[10)]	3.251
H(9)	Cl(1)[18)]	3.463	H(9)	C(7)[11)]	3.420
H(9)	C(9)[12)]	3.055	H(9)	H(2)[1)]	3.299
H(9)	H(6)[11)]	2.689	H(9)	H(7)[11)]	3.280
H(9)	H(12)[12)]	2.978	H(9)	H(13)[12)]	2.643
H(9)	H(14)[12)]	3.037	H(10)	Cl(1)[6)]	3.253
H(10)	C(9)[12)]	3.282	H(10)	H(1)	3.279
H(10)	H(2)[1)]	2.881	H(10)	H(3)[6)]	2.710

Table 10. Distances beyond the asymmetric unit out to 3.60 Å involving hydrogens (continued)

atom	atom	distance	atom	atom	distance
H(10)	H(12)[12)]	2.726	H(10)	H(13)[12)]	3.103
H(10)	H(14)[12)]	3.535	H(11)	H(1)	3.264
H(11)	H(2)[1)]	2.239	H(11)	H(6)[11)]	3.161
H(12)	Cl(1)[18)]	2.851	H(12)	C(8)[13)]	3.267
H(12)	H(9)[13)]	2.978	H(12)	H(10)[13)]	2.726
H(13)	Cl(1)[15)]	3.482	H(13)	C(8)[13)]	3.314
H(13)	H(7)[9)]	3.528	H(13)	H(9)[13)]	2.643
H(13)	H(10)[13)]	3.103	H(14)	C(7)[9)]	3.402
H(14)	H(3)[6)]	2.936	H(14)	H(6)[9)]	3.151
H(14)	H(7)[9)]	3.232	H(14)	H(8)[9)]	3.251
H(14)	H(9)[13)]	3.037	H(14)	H(10)[13)]	3.535

Symmetry Operators:

(1) X,-Y+1/2,Z
(2) X+1/2,-Y+1/2,-Z+1/2+1
(3) X+1/2,Y,-Z+1/2+1
(4) X+1/2,-Y+1/2,-Z+1/2
(5) -X+1,Y+1/2,-Z+1
(6) X+1/2-1,-Y+1/2,-Z+1/2+1
(7) X,Y,Z+1
(8) X,-Y+1/2,Z+1
(9) X+1/2-1,Y,-Z+1/2
(10) X+1/2,Y,-Z+1/2
(11) -X+1,-Y,-Z+1
(12) -X+1/2,-Y,Z+1/2
(13) -X+1/2,-Y,Z+1/2-1
(14) X+1/2-1,Y,-Z+1/2+1
(15) X+1/2-1,-Y+1/2,-Z+1/2
(16) X,Y,Z-1
(17) X,-Y+1/2,Z-1
(18) -X+1,Y+1/2-1,-Z+1