Table 7. Torsion Angles(°) (continued)

atom	atom	atom	atom	angle	atom	atom	atom	atom	angle
C(20)	C(19)	C(18)	C(23)	-173.9(3)	C(20)	C(21)	Ti(2)	C(22)	116.4(3)
C(20)	C(21)	C(22)	C(27)	170.1(3)	C(21)	Ti(2)	N(2)	C(28)	-35.8(5)
C(21)	Ti(2)	N(2)	C(29)	144.9(2)	C(21)	Ti(2)	C(18)	C(22)	36.8(2)
C(21)	Ti(2)	C(18)	C(23)	161.2(4)	C(21)	Ti(2)	C(19)	C(24)	-159.5(4)
C(21)	Ti(2)	C(20)	C(25)	-122.8(5)	C(21)	Ti(2)	C(22)	C(27)	120.2(5)
C(21)	C(20)	Ti(2)	C(22)	-36.7(2)	C(21)	C(20)	C(19)	C(24)	-173.2(4)
C(21)	C(22)	C(18)	C(23)	173.6(4)	C(22)	Ti(2)	N(2)	C(28)	-16.4(5)
C(22)	Ti(2)	N(2)	C(29)	164.3(2)	C(22)	Ti(2)	C(18)	C(23)	124.4(5)
C(22)	Ti(2)	C(19)	C(24)	159.6(4)	C(22)	Ti(2)	C(20)	C(25)	-159.5(4)
C(22)	Ti(2)	C(21)	C(26)	-121.9(4)	C(22)	C(18)	C(19)	C(24)	173.0(4)
C(22)	C(21)	C(20)	C(25)	-177.4(4)	C(23)	C(18)	C(19)	C(24)	0.0(6)
C(23)	C(18)	C(22)	C(27)	3.4(6)	C(24)	C(19)	C(20)	C(25)	4.1(6)
C(25)	C(20)	C(21)	C(26)	-3.8(6)	C(26)	C(21)	C(22)	C(27)	-3.7(6)
C(28)	N(2)	C(29)	C(30)	64.3(4)	C(28)	N(2)	C(29)	C(34)	-63.7(5)
C(29)	C(30)	C(31)	C(32)	54.9(5)	C(29)	C(34)	C(33)	C(32)	-56.8(5)
C(30)	C(29)	C(34)	C(33)	56.4(5)	C(30)	C(31)	C(32)	C(33)	-56.2(5)
C(31)	C(30)	C(29)	C(34)	-55.4(4)	C(31)	C(32)	C(33)	C(34)	57.2(5)

Table 8. Non-bonded Contacts out to 3.60 $\AA$

atom	atom	distance	ADC	atom	atom	distance	ADC
C(2)	C(25)	3.454(6)	55702	C(3)	C(25)	3.550(6)	55702
C(8)	C(21)	3.453(6)	65702				

The ADC (atom designator code) specifies the position of an atom in a crystal. The 5-digit number shown in the table is a composite of three one-digit numbers and one two-digit number: TA (first digit) + TB (second digit) + TC (third digit) + SN (last two digits). TA, TB and TC are the crystal lattice translation digits along cell edges a, b and c. A translation digit of 5 indicates the origin unit cell. If TA = 4, this indicates a translation of one unit cell length along the a-axis in the negative direction. Each translation digit can range in value from 1 to 9 and thus ± 4 lattice translations from the origin (TA=5, TB=5, TC=5) can be represented.

The SN, or symmetry operator number, refers to the number of the symmetry operator used to generate the coordinates of the target atom. A list of symmetry operators relevant to this structure are given below.

For a given intermolecular contact, the first atom (origin atom) is located in the origin unit cell and its position can be generated using the identity operator (SN=1). Thus, the ADC for an origin atom is always 55501. The position of the second atom (target atom) can be generated using the ADC and the coordinates of the atom in the parameter table. For example, an ADC of 47502 refers to the target atom moved through symmetry operator two, then translated -1 cell translations along the a axis, +2 cell translations along the b axis, and 0 cell translations along the c axis.

An ADC of 1 indicates an intermolecular contact between two fragments (eg. cation and anion) that reside in the same asymmetric unit.

Symmetry Operators:

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

X-ray Structure Report

for

$(1,3\text{-}Me_2C_5H_3)TiCl_2[N(Me)Cy]$ (**4**)

Measured on Saturday, February 9, 2002

Experimental

Data Collection

An orange platelet crystal of $C_{14}H_{23}Cl_2NTi$ having approximate dimensions of 0.80 x 0.67 x 0.33 mm was mounted on a glass fiber. All measurements were made on a Rigaku RAXIS-RAPID Imaging Plate diffractometer with graphite monochromated Mo-Kα radiation.

Indexing was performed from 3 oscillations which were exposed for 1.0 minutes. The camera radius was 127.40 mm. Readout was performed in the 0.200 mm pixel mode.

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

a = 13.8739(6) $\AA$
b = 6.7380(1) $\AA$ β = 108.786(1)°
c = 18.0293(2) $\AA$
V = 1595.63(7) $\AA^3$

For Z = 4 and F.W. = 324.15, the calculated density is 1.35 g/cm^3. The systematic absences of:

h0l: $l \neq 2n$
0k0: $k \neq 2n$

uniquely determine the space group to be:

$P2_1/c$ (#14)

The data were collected at a temperature of -30 $\pm$ 1°C to a maximum 2θ value of 54.9°. A total of 44 images, corresponding to 220.0 ° oscillation angles, were collected with 2 different goniometer settings. Exposure time was 1.00 minutes per degree. The camera radius was 127.40 mm. Readout was performed in the 0.200 mm pixel mode. Data were processed by the PROCESS-AUTO program package.

Data Reduction

Of the 14011 reflections which were collected, 3569 were unique (R_{int} = 0.049).

The linear absorption coefficient, μ, for Mo-Kα radiation is 8.5 cm^{-1}. A symmetry-related absorption correction using the program ABSCOR[1] was applied which resulted in transmission factors ranging from 0.19 to 0.75. The data were corrected for Lorentz and polarization effects.

Structure Solution and Refinement

The structure was solved by direct methods and expanded using Fourier techniques[3]. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were included but not refined. The final cycle of full-matrix least-squares refinement[4] was based on 3569 observed reflections (I > -10.00σ(I)) and 163 variable parameters and converged (largest parameter shift was 0.00 times its esd) with unweighted and weighted agreement factors of:

$$R = \Sigma(Fo^2 - Fc^2)/\Sigma Fo^2 = 0.086$$

$$R_w = \sqrt{\Sigma w(Fo^2 - Fc^2)^2/\Sigma w(Fo^2)^2} = 0.157$$

$$R1 = \Sigma||Fo| - |Fc||/\Sigma|Fo| = 0.049 \quad for \;\; I > 2.0\sigma(I) \;\; data$$

The standard deviation of an observation of unit weight[5] was 1.86. The weighting scheme was based on counting statistics and included a factor (p = 0.050) to downweight the intense reflections. Plots of $\Sigma w(Fo^2 - Fc^2)^2$ versus Fo^2, reflection order in data collection, sin θ/λ and various classes of indices showed no unusual trends. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.42 and -0.63 $e^-/\AA^3$, respectively.

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 Hubbel[9]. All calculations were performed using the teXsan[10] crystallographic software package of Molecular Structure Corporation.

References

(1) ABSCOR: Higashi T. (1995). Program for Absorption Correction, Rigaku Corporation, Tokyo, Japan.

(3) DIRDIF94: Beurskens, P.T., Admiraal, G., Beurskens, G., Bosman, W.P., de Gelder, R., Israel, R. and Smits, J.M.M. (1994). The DIRDIF-94 program system, Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.

(4) Least-Squares:

Function minimized: $\Sigma w(Fo^2 - Fc^2)^2$

where w = $\frac{1}{\sigma^2(Fo^2)} = [\sigma_c^2(Fo^2) + (p(Max(Fo^2, 0) + 2Fc^2)/3)^2]^{-1}$

$\sigma_c(Fo^2)$ = e.s.d. based on counting statistics

p = p-factor

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

$$\sqrt{\Sigma w(|Fo| - |Fc|)^2/(No - Nv)}$$

where: No = number of observations

Nv = number of variables

(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) teXsan: Crystal Structure Analysis Package, Molecular Structure Corporation (1985 & 1999).

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	$C_{14}H_{23}Cl_2NTi$
Formula Weight	324.15
Crystal Color, Habit	orange, platelet
Crystal Dimensions	0.80 X 0.67 X 0.33 mm
Crystal System	monoclinic
Lattice Type	Primitive
No. of Reflections Used for Unit Cell Determination (2θ range)	12956 (5.9 - 54.9°)
Indexing Images	3 oscillations at 1.0 minutes
Camera Radius	127.40 mm
Lattice Parameters	a = 13.8739(6) $\AA$ b = 6.7380(1) $\AA$ c = 18.0293(2) $\AA$ β = 108.786(1)° V = 1595.63(7) $\AA^3$
Space Group	$P2_1/c$ (#14)
Z value	4
D_{calc}	1.349 g/cm^3
F_{000}	680.00
μ(MoKα)	8.55 cm^{-1}

B. Intensity Measurements

Diffractometer	Rigaku RAXIS-RAPID Imaging Plate
Radiation	MoKα (λ = 0.71069 $\AA$) graphite monochromated

Temperature	-30.0 °C
Voltage, Current	50 kV, 30 mA
Collimator Size	0.8 mm
Detector Aperture	270.0 mm x 256.0 mm
Data Images	44 exposures at 1.0 minutes per degree
Oscillation Range (ϕ=0.0°,χ=45.0°)	ω 130.0 - 190.0° with 5.0° step
Oscillation Range (ϕ=180.0°,χ=45.0°)	ω 0.0 - 160.0° with 5.0° step
Camera Radius	127.40 mm
Pixel Size	0.200 mm
$2\theta_{max}$	54.9°
No. of Reflections Measured	Total: 14011 Unique: 3569 (R_{int} = 0.049)
Corrections	Lorentz-polarization Absorption (trans. factors: 0.1873 - 0.7542)

C. Structure Solution and Refinement

Structure Solution	Direct Methods
Refinement	Full-matrix least-squares
Function Minimized	$\Sigma w(Fo^2 - Fc^2)^2$
Least Squares Weights	$w = \frac{1}{\sigma^2(Fo^2)}$
p-factor	0.0500
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (I>-10.00σ(I))	3569
No. Variables	163
Reflection/Parameter Ratio	21.90
Residuals: R; Rw	0.086 ; 0.157
Residuals: R1	0.049

No. of Reflections to calc R1	2890
Goodness of Fit Indicator	1.86
Max Shift/Error in Final Cycle	0.001
Maximum peak in Final Diff. Map	0.42 $e^-/\AA^3$
Minimum peak in Final Diff. Map	-0.63 $e^-/\AA^3$

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

atom	x	y	z	B_{eq}	occ
Ti(1)	0.19023(3)	0.14336(7)	-0.32649(3)	2.06(1)	1.0000
Cl(1)	0.28907(6)	0.3496(1)	-0.23165(5)	3.30(2)	1.0000
Cl(2)	0.11900(6)	0.3562(1)	-0.42778(5)	3.61(2)	1.0000
N(1)	0.2803(2)	-0.0114(3)	-0.3590(1)	2.14(4)	1.0000
C(1)	0.1327(2)	0.0173(5)	-0.2252(2)	3.18(6)	0.9979
C(2)	0.1553(2)	-0.1407(4)	-0.2673(2)	3.13(6)	0.9858
C(3)	0.0860(2)	-0.1337(4)	-0.3443(2)	2.94(6)	0.9652
C(4)	0.0207(2)	0.0273(5)	-0.3507(2)	3.05(6)	0.9838
C(5)	0.0524(2)	0.1242(5)	-0.2773(2)	2.97(6)	0.9685
C(6)	0.1805(3)	0.0588(7)	-0.1403(2)	5.17(10)	1.0000
C(7)	-0.0688(2)	0.0797(6)	-0.4179(2)	4.78(8)	1.0000
C(8)	0.2989(2)	-0.2185(4)	-0.3720(2)	3.06(6)	1.0000
C(9)	0.3486(2)	0.1303(4)	-0.3795(1)	2.00(5)	0.9895
C(10)	0.4573(2)	0.1189(4)	-0.3244(2)	2.66(5)	1.0000
C(11)	0.5229(2)	0.2747(5)	-0.3455(2)	3.21(6)	1.0000
C(12)	0.5195(3)	0.2531(5)	-0.4295(2)	3.64(7)	0.9919
C(13)	0.4113(3)	0.2676(5)	-0.4840(2)	3.68(7)	1.0000
C(14)	0.3434(2)	0.1134(5)	-0.4646(2)	3.06(6)	1.0000
H(1)	0.2025	-0.2462	-0.2452	3.8062	1.0000
H(2)	0.0799	-0.2341	-0.3832	3.6020	1.0000
H(3)	0.0197	0.2353	-0.2625	3.5680	1.0000
H(4)	0.2336	-0.0321	-0.1171	6.4138	1.0000
H(5)	0.2071	0.1910	-0.1325	6.4138	1.0000
H(6)	0.1302	0.0482	-0.1140	6.4138	1.0000

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

atom	x	y	z	B_{eq}	occ
H(7)	-0.0991	0.1986	-0.4077	5.8358	1.0000
H(8)	-0.0480	0.1020	-0.4637	5.8358	1.0000
H(9)	-0.1172	-0.0242	-0.4294	5.8358	1.0000
H(10)	0.2514	-0.3006	-0.3581	3.7193	1.0000
H(11)	0.2909	-0.2397	-0.4262	3.7193	1.0000
H(12)	0.3660	-0.2546	-0.3414	3.7193	1.0000
H(13)	0.3241	0.2594	-0.3733	2.4468	1.0000
H(14)	0.4584	0.1382	-0.2717	3.3101	1.0000
H(15)	0.4845	-0.0103	-0.3282	3.3101	1.0000
H(16)	0.4985	0.4035	-0.3386	3.9136	1.0000
H(17)	0.5917	0.2626	-0.3119	3.9136	1.0000
H(18)	0.5604	0.3540	-0.4421	4.6712	1.0000
H(19)	0.5470	0.1265	-0.4364	4.6712	1.0000
H(20)	0.3849	0.3972	-0.4790	4.4162	1.0000
H(21)	0.4096	0.2520	-0.5372	4.4162	1.0000
H(22)	0.3653	-0.0169	-0.4742	3.8214	1.0000
H(23)	0.2743	0.1296	-0.4983	3.8214	1.0000

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

Table 2. Anisotropic Displacement Parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Ti(1)	0.0211(2)	0.0195(3)	0.0379(3)	-0.0030(2)	0.0097(2)	-0.0045(2)
Cl(1)	0.0416(4)	0.0370(4)	0.0458(4)	-0.0156(3)	0.0125(3)	-0.0139(3)
Cl(2)	0.0393(4)	0.0328(4)	0.0557(5)	0.0049(3)	0.0022(3)	0.0052(3)
N(1)	0.0242(10)	0.0207(10)	0.039(1)	-0.0017(8)	0.0135(9)	-0.0006(9)
C(1)	0.035(1)	0.044(2)	0.046(2)	-0.013(1)	0.019(1)	-0.005(1)
C(2)	0.033(1)	0.032(1)	0.062(2)	-0.005(1)	0.026(1)	0.005(1)
C(3)	0.033(1)	0.028(1)	0.059(2)	-0.015(1)	0.027(1)	-0.018(1)
C(4)	0.022(1)	0.041(2)	0.055(2)	-0.011(1)	0.014(1)	-0.016(1)
C(5)	0.028(1)	0.037(2)	0.054(2)	-0.007(1)	0.022(1)	-0.017(1)
C(6)	0.069(2)	0.086(3)	0.044(2)	-0.023(2)	0.021(2)	0.000(2)
C(7)	0.028(1)	0.075(3)	0.068(2)	-0.010(2)	0.001(1)	-0.021(2)
C(8)	0.042(2)	0.022(1)	0.060(2)	0.001(1)	0.027(1)	-0.003(1)
C(9)	0.022(1)	0.023(1)	0.031(1)	-0.0018(9)	0.0091(9)	0.001(1)
C(10)	0.024(1)	0.040(2)	0.036(1)	0.000(1)	0.0078(10)	0.009(1)
C(11)	0.026(1)	0.046(2)	0.047(2)	-0.009(1)	0.008(1)	0.001(1)
C(12)	0.044(2)	0.049(2)	0.055(2)	-0.011(1)	0.028(1)	0.004(2)
C(13)	0.055(2)	0.054(2)	0.032(1)	-0.014(2)	0.017(1)	0.006(1)
C(14)	0.039(1)	0.046(2)	0.031(1)	-0.011(1)	0.010(1)	0.000(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 3. Bond Lengths($\AA$)

atom	atom	distance	atom	atom	distance
Ti(1)	Cl(1)	2.2858(8)	Ti(1)	Cl(2)	2.2816(9)
Ti(1)	N(1)	1.861(2)	Ti(1)	C(1)	2.373(3)
Ti(1)	C(2)	2.317(3)	Ti(1)	C(3)	2.319(3)
Ti(1)	C(4)	2.382(3)	Ti(1)	C(5)	2.356(3)
N(1)	C(8)	1.451(3)	N(1)	C(9)	1.475(3)
C(1)	C(2)	1.400(4)	C(1)	C(5)	1.402(4)
C(1)	C(6)	1.486(5)	C(2)	C(3)	1.412(5)
C(3)	C(4)	1.395(4)	C(4)	C(5)	1.413(4)
C(4)	C(7)	1.472(4)	C(9)	C(10)	1.518(3)
C(9)	C(14)	1.516(4)	C(10)	C(11)	1.516(4)
C(11)	C(12)	1.507(4)	C(12)	C(13)	1.509(5)
C(13)	C(14)	1.517(4)			

Table 4. Bond Lengths($\AA$)

atom	atom	distance	atom	atom	distance
C(2)	H(1)	0.96	C(3)	H(2)	0.96
C(5)	H(3)	0.96	C(6)	H(4)	0.95
C(6)	H(5)	0.96	C(6)	H(6)	0.97
C(7)	H(7)	0.95	C(7)	H(8)	0.97
C(7)	H(9)	0.95	C(8)	H(10)	0.95
C(8)	H(11)	0.96	C(8)	H(12)	0.95
C(9)	H(13)	0.95	C(10)	H(14)	0.95
C(10)	H(15)	0.96	C(11)	H(16)	0.95
C(11)	H(17)	0.96	C(12)	H(18)	0.96
C(12)	H(19)	0.96	C(13)	H(20)	0.96
C(13)	H(21)	0.96	C(14)	H(22)	0.96
C(14)	H(23)	0.96			

Table 5. Bond Angles(°)

atom	atom	atom	angle	atom	atom	atom	angle
Cl(1)	Ti(1)	Cl(2)	102.56(4)	Cl(1)	Ti(1)	N(1)	105.93(7)
Cl(1)	Ti(1)	C(1)	84.92(8)	Cl(1)	Ti(1)	C(2)	108.76(9)
Cl(1)	Ti(1)	C(3)	142.18(8)	Cl(1)	Ti(1)	C(4)	130.96(8)
Cl(1)	Ti(1)	C(5)	96.63(8)	Cl(2)	Ti(1)	N(1)	105.18(7)
Cl(2)	Ti(1)	C(1)	132.41(8)	Cl(2)	Ti(1)	C(2)	141.89(9)
Cl(2)	Ti(1)	C(3)	107.77(9)	Cl(2)	Ti(1)	C(4)	84.96(9)
Cl(2)	Ti(1)	C(5)	98.11(9)	N(1)	Ti(1)	C(1)	117.9(1)
N(1)	Ti(1)	C(2)	86.76(10)	N(1)	Ti(1)	C(3)	87.53(10)
N(1)	Ti(1)	C(4)	118.81(10)	N(1)	Ti(1)	C(5)	142.78(10)
C(1)	Ti(1)	C(2)	34.7(1)	C(1)	Ti(1)	C(3)	58.0(1)
C(1)	Ti(1)	C(4)	57.7(1)	C(1)	Ti(1)	C(5)	34.5(1)
C(2)	Ti(1)	C(3)	35.5(1)	C(2)	Ti(1)	C(4)	58.2(1)
C(2)	Ti(1)	C(5)	57.7(1)	C(3)	Ti(1)	C(4)	34.5(1)
C(3)	Ti(1)	C(5)	57.52(10)	C(4)	Ti(1)	C(5)	34.7(1)
Ti(1)	N(1)	C(8)	139.6(2)	Ti(1)	N(1)	C(9)	105.5(2)
C(8)	N(1)	C(9)	114.8(2)	Ti(1)	C(1)	C(2)	70.5(2)
Ti(1)	C(1)	C(5)	72.1(2)	Ti(1)	C(1)	C(6)	125.2(2)
C(2)	C(1)	C(5)	107.2(3)	C(2)	C(1)	C(6)	126.7(3)
C(5)	C(1)	C(6)	126.0(3)	Ti(1)	C(2)	C(1)	74.8(2)
Ti(1)	C(2)	C(3)	72.3(2)	C(1)	C(2)	C(3)	107.9(3)
Ti(1)	C(3)	C(2)	72.2(2)	Ti(1)	C(3)	C(4)	75.2(2)
C(2)	C(3)	C(4)	109.0(3)	Ti(1)	C(4)	C(3)	70.3(2)
Ti(1)	C(4)	C(5)	71.7(2)	Ti(1)	C(4)	C(7)	126.4(2)
C(3)	C(4)	C(5)	106.5(3)	C(3)	C(4)	C(7)	127.4(3)

Table 5. Bond Angles(°) (continued)

atom	atom	atom	angle	atom	atom	atom	angle
C(5)	C(4)	C(7)	126.0(3)	Ti(1)	C(5)	C(1)	73.4(2)
Ti(1)	C(5)	C(4)	73.7(2)	C(1)	C(5)	C(4)	109.3(3)
N(1)	C(9)	C(10)	112.7(2)	N(1)	C(9)	C(14)	112.3(2)
C(10)	C(9)	C(14)	111.9(2)	C(9)	C(10)	C(11)	110.5(2)
C(10)	C(11)	C(12)	111.0(3)	C(11)	C(12)	C(13)	110.6(3)
C(12)	C(13)	C(14)	111.5(3)	C(9)	C(14)	C(13)	110.5(2)

Table 6. Bond Angles(°)

atom	atom	atom	angle	atom	atom	atom	angle
Ti(1)	C(2)	H(1)	125.8	C(1)	C(2)	H(1)	125.4
C(3)	C(2)	H(1)	126.0	Ti(1)	C(3)	H(2)	125.0
C(2)	C(3)	H(2)	124.6	C(4)	C(3)	H(2)	125.9
Ti(1)	C(5)	H(3)	125.2	C(1)	C(5)	H(3)	124.2
C(4)	C(5)	H(3)	126.1	C(1)	C(6)	H(4)	111.1
C(1)	C(6)	H(5)	110.4	C(1)	C(6)	H(6)	109.8
H(4)	C(6)	H(5)	109.3	H(4)	C(6)	H(6)	108.6
H(5)	C(6)	H(6)	107.6	C(4)	C(7)	H(7)	110.5
C(4)	C(7)	H(8)	109.6	C(4)	C(7)	H(9)	110.7
H(7)	C(7)	H(8)	107.9	H(7)	C(7)	H(9)	109.9
H(8)	C(7)	H(9)	108.3	N(1)	C(8)	H(10)	110.0
N(1)	C(8)	H(11)	109.8	N(1)	C(8)	H(12)	110.2
H(10)	C(8)	H(11)	108.5	H(10)	C(8)	H(12)	109.3
H(11)	C(8)	H(12)	108.9	N(1)	C(9)	H(13)	106.1
C(10)	C(9)	H(13)	106.7	C(14)	C(9)	H(13)	106.5
C(9)	C(10)	H(14)	109.9	C(9)	C(10)	H(15)	109.2
C(11)	C(10)	H(14)	109.7	C(11)	C(10)	H(15)	109.2
H(14)	C(10)	H(15)	108.3	C(10)	C(11)	H(16)	109.3
C(10)	C(11)	H(17)	109.8	C(12)	C(11)	H(16)	108.7
C(12)	C(11)	H(17)	109.3	H(16)	C(11)	H(17)	108.7
C(11)	C(12)	H(18)	109.9	C(11)	C(12)	H(19)	109.4
C(13)	C(12)	H(18)	109.9	C(13)	C(12)	H(19)	109.1
H(18)	C(12)	H(19)	108.0	C(12)	C(13)	H(20)	109.0
C(12)	C(13)	H(21)	110.1	C(14)	C(13)	H(20)	108.4

Table 6. Bond Angles(°) (continued)

atom	atom	atom	angle	atom	atom	atom	angle
C(14)	C(13)	H(21)	109.8	H(20)	C(13)	H(21)	107.8
C(9)	C(14)	H(22)	109.6	C(9)	C(14)	H(23)	110.0
C(13)	C(14)	H(22)	109.1	C(13)	C(14)	H(23)	110.1
H(22)	C(14)	H(23)	107.4				

Table 7. Torsion Angles(°)

atom	atom	atom	atom	angle	atom	atom	atom	atom	angle
Ti(1)	N(1)	C(9)	C(10)	112.8(2)	Ti(1)	N(1)	C(9)	C(14)	-119.6(2)
Ti(1)	C(1)	C(2)	C(3)	-65.3(2)	Ti(1)	C(1)	C(5)	C(4)	65.4(2)
Ti(1)	C(2)	C(1)	C(5)	63.2(2)	Ti(1)	C(2)	C(1)	C(6)	-119.9(3)
Ti(1)	C(2)	C(3)	C(4)	-66.8(2)	Ti(1)	C(3)	C(2)	C(1)	66.9(2)
Ti(1)	C(3)	C(4)	C(5)	-62.9(2)	Ti(1)	C(3)	C(4)	C(7)	121.3(3)
Ti(1)	C(4)	C(3)	C(2)	64.8(2)	Ti(1)	C(4)	C(5)	C(1)	-65.3(2)
Ti(1)	C(5)	C(1)	C(2)	-62.1(2)	Ti(1)	C(5)	C(1)	C(6)	120.9(3)
Ti(1)	C(5)	C(4)	C(3)	62.0(2)	Ti(1)	C(5)	C(4)	C(7)	-122.1(3)
Cl(1)	Ti(1)	N(1)	C(8)	130.0(3)	Cl(1)	Ti(1)	N(1)	C(9)	-52.9(2)
Cl(1)	Ti(1)	C(1)	C(2)	-134.1(2)	Cl(1)	Ti(1)	C(1)	C(5)	109.5(2)
Cl(1)	Ti(1)	C(1)	C(6)	-12.3(3)	Cl(1)	Ti(1)	C(2)	C(1)	49.1(2)
Cl(1)	Ti(1)	C(2)	C(3)	164.0(1)	Cl(1)	Ti(1)	C(3)	C(2)	-25.2(2)
Cl(1)	Ti(1)	C(3)	C(4)	90.7(2)	Cl(1)	Ti(1)	C(4)	C(3)	-125.7(2)
Cl(1)	Ti(1)	C(4)	C(5)	-9.8(2)	Cl(1)	Ti(1)	C(4)	C(7)	111.8(3)
Cl(1)	Ti(1)	C(5)	C(1)	-70.9(2)	Cl(1)	Ti(1)	C(5)	C(4)	172.5(2)
Cl(2)	Ti(1)	N(1)	C(8)	-121.8(3)	Cl(2)	Ti(1)	N(1)	C(9)	55.2(2)
Cl(2)	Ti(1)	C(1)	C(2)	123.5(2)	Cl(2)	Ti(1)	C(1)	C(5)	7.1(2)
Cl(2)	Ti(1)	C(1)	C(6)	-114.7(3)	Cl(2)	Ti(1)	C(2)	C(1)	-94.5(2)
Cl(2)	Ti(1)	C(2)	C(3)	20.4(2)	Cl(2)	Ti(1)	C(3)	C(2)	-167.0(2)
Cl(2)	Ti(1)	C(3)	C(4)	-51.0(2)	Cl(2)	Ti(1)	C(4)	C(3)	132.0(2)
Cl(2)	Ti(1)	C(4)	C(5)	-112.1(2)	Cl(2)	Ti(1)	C(4)	C(7)	9.5(3)
Cl(2)	Ti(1)	C(5)	C(1)	-174.7(2)	Cl(2)	Ti(1)	C(5)	C(4)	68.8(2)
N(1)	Ti(1)	C(1)	C(2)	-28.7(2)	N(1)	Ti(1)	C(1)	C(5)	-145.1(2)
N(1)	Ti(1)	C(1)	C(6)	93.0(3)	N(1)	Ti(1)	C(2)	C(1)	154.8(2)

Table 7. Torsion Angles(°) (continued)

atom	atom	atom	atom	angle	atom	atom	atom	atom	angle
N(1)	Ti(1)	C(2)	C(3)	-90.3(2)	N(1)	Ti(1)	C(3)	C(2)	87.9(2)
N(1)	Ti(1)	C(3)	C(4)	-156.2(2)	N(1)	Ti(1)	C(4)	C(3)	27.5(2)
N(1)	Ti(1)	C(4)	C(5)	143.3(2)	N(1)	Ti(1)	C(4)	C(7)	-95.1(3)
N(1)	Ti(1)	C(5)	C(1)	56.7(2)	N(1)	Ti(1)	C(5)	C(4)	-59.9(3)
N(1)	C(9)	C(10)	C(11)	-177.0(2)	N(1)	C(9)	C(14)	C(13)	177.5(2)
C(1)	Ti(1)	N(1)	C(8)	37.3(3)	C(1)	Ti(1)	N(1)	C(9)	-145.6(2)
C(1)	Ti(1)	C(2)	C(3)	114.9(2)	C(1)	Ti(1)	C(3)	C(2)	-37.5(2)
C(1)	Ti(1)	C(3)	C(4)	78.4(2)	C(1)	Ti(1)	C(4)	C(3)	-79.1(2)
C(1)	Ti(1)	C(4)	C(5)	36.8(2)	C(1)	Ti(1)	C(4)	C(7)	158.4(3)
C(1)	Ti(1)	C(5)	C(4)	-116.5(3)	C(1)	C(2)	Ti(1)	C(3)	-114.9(2)
C(1)	C(2)	Ti(1)	C(4)	-78.1(2)	C(1)	C(2)	Ti(1)	C(5)	-36.9(2)
C(1)	C(2)	C(3)	C(4)	0.1(3)	C(1)	C(5)	Ti(1)	C(2)	37.1(2)
C(1)	C(5)	Ti(1)	C(3)	79.4(2)	C(1)	C(5)	Ti(1)	C(4)	116.5(3)
C(1)	C(5)	C(4)	C(3)	-3.2(3)	C(1)	C(5)	C(4)	C(7)	172.6(3)
C(2)	Ti(1)	N(1)	C(8)	21.4(3)	C(2)	Ti(1)	N(1)	C(9)	-161.5(2)
C(2)	Ti(1)	C(1)	C(5)	-116.4(3)	C(2)	Ti(1)	C(1)	C(6)	121.7(4)
C(2)	Ti(1)	C(3)	C(4)	116.0(3)	C(2)	Ti(1)	C(4)	C(3)	-37.9(2)
C(2)	Ti(1)	C(4)	C(5)	78.0(2)	C(2)	Ti(1)	C(4)	C(7)	-160.4(3)
C(2)	Ti(1)	C(5)	C(4)	-79.4(2)	C(2)	C(1)	Ti(1)	C(3)	38.4(2)
C(2)	C(1)	Ti(1)	C(4)	79.4(2)	C(2)	C(1)	Ti(1)	C(5)	116.4(3)
C(2)	C(1)	C(5)	C(4)	3.3(3)	C(2)	C(3)	Ti(1)	C(4)	-116.0(3)
C(2)	C(3)	Ti(1)	C(5)	-78.6(2)	C(2)	C(3)	C(4)	C(5)	1.9(3)
C(2)	C(3)	C(4)	C(7)	-173.9(3)	C(3)	Ti(1)	N(1)	C(8)	-14.1(3)
C(3)	Ti(1)	N(1)	C(9)	163.0(2)	C(3)	Ti(1)	C(1)	C(5)	-78.0(2)

Table 7. Torsion Angles(°) (continued)

atom	atom	atom	atom	angle	atom	atom	atom	atom	angle
C(3)	Ti(1)	C(1)	C(6)	160.1(4)	C(3)	Ti(1)	C(4)	C(5)	115.9(3)
C(3)	Ti(1)	C(4)	C(7)	-122.5(4)	C(3)	Ti(1)	C(5)	C(4)	-37.2(2)
C(3)	C(2)	Ti(1)	C(4)	36.8(2)	C(3)	C(2)	Ti(1)	C(5)	78.0(2)
C(3)	C(2)	C(1)	C(5)	-2.1(3)	C(3)	C(2)	C(1)	C(6)	174.8(3)
C(3)	C(4)	Ti(1)	C(5)	-115.9(3)	C(4)	Ti(1)	N(1)	C(8)	-29.2(3)
C(4)	Ti(1)	N(1)	C(9)	147.8(2)	C(4)	Ti(1)	C(1)	C(5)	-37.0(2)
C(4)	Ti(1)	C(1)	C(6)	-158.9(4)	C(4)	C(3)	Ti(1)	C(5)	37.4(2)
C(4)	C(5)	C(1)	C(6)	-173.6(3)	C(5)	Ti(1)	N(1)	C(8)	5.0(4)
C(5)	Ti(1)	N(1)	C(9)	-178.0(2)	C(5)	Ti(1)	C(1)	C(6)	-121.9(4)
C(5)	Ti(1)	C(4)	C(7)	121.6(4)	C(8)	N(1)	C(9)	C(10)	-69.3(3)
C(8)	N(1)	C(9)	C(14)	58.3(3)	C(9)	C(10)	C(11)	C(12)	-56.5(3)
C(9)	C(14)	C(13)	C(12)	55.3(4)	C(10)	C(9)	C(14)	C(13)	-54.5(3)
C(10)	C(11)	C(12)	C(13)	57.6(4)	C(11)	C(10)	C(9)	C(14)	55.2(3)
C(11)	C(12)	C(13)	C(14)	-57.1(4)					

Table 8. Non-bonded Contacts out to 3.60 $\AA$

atom	atom	distance	ADC	atom	atom	distance	ADC
C(2)	C(5)	3.599(4)	54402				

The ADC (atom designator code) specifies the position of an atom in a crystal. The 5-digit number shown in the table is a composite of three one-digit numbers and one two-digit number: TA (first digit) + TB (second digit) + TC (third digit) + SN (last two digits). TA, TB and TC are the crystal lattice translation digits along cell edges a, b and c. A translation digit of 5 indicates the origin unit cell. If TA = 4, this indicates a translation of one unit cell length along the a-axis in the negative direction. Each translation digit can range in value from 1 to 9 and thus ± 4 lattice translations from the origin (TA=5, TB=5, TC=5) can be represented.

The SN, or symmetry operator number, refers to the number of the symmetry operator used to generate the coordinates of the target atom. A list of symmetry operators relevant to this structure are given below.

For a given intermolecular contact, the first atom (origin atom) is located in the origin unit cell and its position can be generated using the identity operator (SN=1). Thus, the ADC for an origin atom is always 55501. The position of the second atom (target atom) can be generated using the ADC and the coordinates of the atom in the parameter table. For example, an ADC of 47502 refers to the target atom moved through symmetry operator two, then translated -1 cell translations along the a axis, +2 cell translations along the b axis, and 0 cell translations along the c axis.

An ADC of 1 indicates an intermolecular contact between two fragments (eg. cation and anion) that reside in the same asymmetric unit.

Symmetry Operators:

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

X-ray Structure Report

for

$(1,3\text{-}Me_2C_5H_3)TiCl_2(NCy_2)$ (**5**)

Measured on Tuesday, January 29, 2002

Experimental

Data Collection

A red prism crystal of $C_{19}H_{31}Cl_2NTi$ having approximate dimensions of 0.80 x 0.80 x 0.50 mm was mounted on a glass fiber. All measurements were made on a Rigaku RAXIS-RAPID Imaging Plate diffractometer with graphite monochromated Mo-Kα radiation.

Indexing was performed from 3 oscillations which were exposed for 1.0 minutes. The camera radius was 127.40 mm. Readout was performed in the 0.200 mm pixel mode.

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

a = 11.0294(4) $\AA$
b = 12.5162(5) $\AA$
c = 14.6992(4) $\AA$
V = 2029.2(1) $\AA^3$

For Z = 4 and F.W. = 392.27, the calculated density is 1.28 g/cm^3. The systematic absences of:

h00: h $\neq$ 2n
0k0: k $\neq$ 2n
00l: l $\neq$ 2n

uniquely determine the space group to be:

$P2_12_12_1$ (#19)

The data were collected at a temperature of -30 $\pm$ 1°C to a maximum 2θ value of 55.0°. A total of 44 images, corresponding to 220.0 ° oscillation angles, were collected with 2 different goniometer settings. Exposure time was 1.00 minutes per degree. The camera radius was 127.40 mm. Readout was performed in the 0.200 mm pixel mode. Data were processed by the PROCESS-AUTO program package.

Data Reduction

Of the 18507 reflections which were collected, 2632 were unique (R_{int} = 0.050).

The linear absorption coefficient, μ, for Mo-Kα radiation is 6.8 cm^{-1}. A symmetry-related absorption correction using the program ABSCOR[1] was applied which resulted in transmission factors ranging from 0.35 to 0.71. The data were corrected for Lorentz and polarization effects.

Structure Solution and Refinement

The structure was solved by direct methods and expanded using Fourier techniques[3]. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were included but not refined. The final cycle of full-matrix least-squares refinement[4] was based on 2632 observed reflections (I > -10.00σ(I)) and 209 variable parameters and converged (largest parameter shift was 3.04 times its esd) with unweighted and weighted agreement factors of:

$$R = \Sigma(Fo^2 - Fc^2)/\Sigma Fo^2 = 0.108$$

$$R_w = \sqrt{\Sigma w(Fo^2 - Fc^2)^2/\Sigma w(Fo^2)^2} = 0.172$$

$$R1 = \Sigma||Fo| - |Fc||/\Sigma|Fo| = 0.059 \quad for \;\; I > 2.0\sigma(I) \;\; data$$

The standard deviation of an observation of unit weight[5] was 1.92. The weighting scheme was based on counting statistics and included a factor (p = 0.050) to downweight the intense reflections. Plots of $\Sigma w(Fo^2 - Fc^2)^2$ versus Fo^2, reflection order in data collection, sin θ/λ and various classes of indices showed no unusual trends. The maximum and minimum peaks on the final difference Fourier map corresponded to 0.62 and -0.51 $e^-/\mathring{A}^3$, respectively.

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 Hubbel[9]. All calculations were performed using the teXsan[10] crystallographic software package of Molecular Structure Corporation.

References

(1) ABSCOR: Higashi T. (1995). Program for Absorption Correction, Rigaku Corporation, Tokyo, Japan.

(3) DIRDIF94: Beurskens, P.T., Admiraal, G., Beurskens, G., Bosman, W.P., de Gelder, R., Israel, R. and Smits, J.M.M. (1994). The DIRDIF-94 program system, Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands.

(4) Least-Squares:

Function minimized: $\Sigma w(Fo^2 - Fc^2)^2$

where w = $\frac{1}{\sigma^2(Fo^2)} = [\sigma_c^2(Fo^2) + (p(Max(Fo^2, 0) + 2Fc^2)/3)^2]^{-1}$

$\sigma_c(Fo^2)$ = e.s.d. based on counting statistics

p = p-factor

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

$$\sqrt{\Sigma w(|Fo| - |Fc|)^2/(No - Nv)}$$

where: No = number of observations

Nv = number of variables

(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) teXsan: Crystal Structure Analysis Package, Molecular Structure Corporation (1985 & 1999).

EXPERIMENTAL DETAILS

A. Crystal Data

Empirical Formula	$C_{19}H_{31}Cl_2NTi$
Formula Weight	392.27
Crystal Color, Habit	red, prism
Crystal Dimensions	0.80 X 0.80 X 0.50 mm
Crystal System	orthorhombic
Lattice Type	Primitive
No. of Reflections Used for Unit Cell Determination (2θ range)	25900 (2.8 - 55.0°)
Indexing Images	3 oscillations at 1.0 minutes
Camera Radius	127.40 mm
Lattice Parameters	a = 11.0294(4) $\AA$ b = 12.5162(5) $\AA$ c = 14.6992(4) $\AA$ V = 2029.2(1) $\AA^3$
Space Group	$P2_12_12_1$ (#19)
Z value	4
D_{calc}	1.284 g/cm^3
F_{000}	832.00
μ(MoKα)	6.84 cm^{-1}

B. Intensity Measurements

Diffractometer	Rigaku RAXIS-RAPID Imaging Plate
Radiation	MoKα (λ = 0.71069 $\AA$) graphite monochromated

Temperature	-30.0 °C
Voltage, Current	50 kV, 30 mA
Collimator Size	0.8 mm
Detector Aperture	270.0 mm x 256.0 mm
Data Images	44 exposures at 1.0 minutes per degree
Oscillation Range (ϕ=0.0°,χ=45.0°)	ω 130.0 - 190.0° with 5.0° step
Oscillation Range (ϕ=180.0°,χ=45.0°)	ω 0.0 - 160.0° with 5.0° step
Camera Radius	127.40 mm
Pixel Size	0.200 mm
$2\theta_{max}$	55.0°
No. of Reflections Measured	Total: 18507 Unique: 2632 (R_{int} = 0.050)
Corrections	Lorentz-polarization Absorption (trans. factors: 0.3474 - 0.7102)

C. Structure Solution and Refinement

Structure Solution	Direct Methods
Refinement	Full-matrix least-squares
Function Minimized	$\Sigma w(Fo^2 - Fc^2)^2$
Least Squares Weights	$w = \frac{1}{\sigma^2(Fo^2)}$
p-factor	0.0500
Anomalous Dispersion	All non-hydrogen atoms
No. Observations (I>-10.00σ(I))	2632
No. Variables	209
Reflection/Parameter Ratio	12.59
Residuals: R; Rw	0.108 ; 0.172
Residuals: R1	0.059

No. of Reflections to calc R1	2437
Goodness of Fit Indicator	1.92
Max Shift/Error in Final Cycle	3.036
Maximum peak in Final Diff. Map	0.62 $e^-/\AA^3$
Minimum peak in Final Diff. Map	-0.51 $e^-/\AA^3$

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

atom	x	y	z	B_{eq}
Ti(1)	-0.14413(6)	-0.62988(5)	-0.66838(3)	3.49(1)
Cl(1)	-0.08756(9)	-0.46043(7)	-0.62310(6)	4.68(2)
Cl(2)	-0.3048(1)	-0.66151(9)	-0.57280(6)	5.17(2)
N(1)	-0.2098(2)	-0.6200(2)	-0.7856(2)	3.31(4)
C(1)	-0.072(1)	-0.7901(8)	-0.5986(8)	11.4(2)
C(2)	0.0081(7)	-0.7142(6)	-0.5811(4)	9.1(1)
C(3)	0.0640(4)	-0.6718(5)	-0.6652(7)	9.4(1)
C(4)	-0.0055(6)	-0.7489(6)	-0.7282(3)	8.3(1)
C(5)	-0.0753(8)	-0.8053(4)	-0.6757(7)	9.7(2)
C(6)	-0.150(1)	-0.8777(9)	-0.573(2)	37(1)
C(7)	0.1534(7)	-0.5965(9)	-0.662(2)	35(1)
C(8)	-0.2084(3)	-0.6575(3)	-0.8812(2)	3.92(6)
C(9)	-0.0906(4)	-0.6304(5)	-0.9298(2)	5.97(10)
C(10)	-0.1021(4)	-0.6663(6)	-1.0309(3)	7.0(1)
C(11)	-0.1358(6)	-0.7803(6)	-1.0396(3)	7.9(1)
C(12)	-0.2522(6)	-0.8051(4)	-0.9905(3)	6.7(1)
C(13)	-0.2448(5)	-0.7745(3)	-0.8895(3)	5.04(8)
C(14)	-0.3046(3)	-0.5353(2)	-0.7711(2)	3.17(5)
C(15)	-0.2813(4)	-0.4334(3)	-0.8259(2)	4.45(7)
C(16)	-0.3805(5)	-0.3510(3)	-0.8047(3)	5.45(8)
C(17)	-0.5061(5)	-0.3935(4)	-0.8208(4)	6.33(10)
C(18)	-0.5270(3)	-0.4929(4)	-0.7633(3)	5.30(9)
C(19)	-0.4338(3)	-0.5777(3)	-0.7838(2)	3.95(6)
H(1)	0.0313	-0.6923	-0.5216	11.1918

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

atom	x	y	z	B_{eq}
H(2)	0.0098	-0.7619	-0.7909	9.9630
H(3)	-0.1246	-0.8602	-0.6995	11.5091
H(4)	-0.1605	-0.8803	-0.5098	45.6037
H(5)	-0.2281	-0.8679	-0.6018	45.6037
H(6)	-0.1171	-0.9434	-0.5946	45.6037
H(7)	0.2205	-0.6240	-0.6298	42.3518
H(8)	0.1765	-0.5779	-0.7221	42.3518
H(9)	0.1229	-0.5348	-0.6317	42.3518
H(10)	-0.2701	-0.6181	-0.9115	4.7237
H(11)	-0.0769	-0.5555	-0.9272	7.1290
H(12)	-0.0252	-0.6669	-0.9019	7.1290
H(13)	-0.1627	-0.6240	-1.0597	8.3798
H(14)	-0.0266	-0.6552	-1.0604	8.3798
H(15)	-0.1458	-0.7969	-1.1022	9.5972
H(16)	-0.0728	-0.8228	-1.0147	9.5972
H(17)	-0.3158	-0.7663	-1.0182	8.0774
H(18)	-0.2672	-0.8794	-0.9953	8.0774
H(19)	-0.1858	-0.8178	-0.8599	6.0242
H(20)	-0.3215	-0.7851	-0.8618	6.0242
H(21)	-0.2984	-0.5152	-0.7090	3.8114
H(22)	-0.2043	-0.4048	-0.8098	5.3672
H(23)	-0.2819	-0.4497	-0.8890	5.3672
H(24)	-0.3741	-0.3305	-0.7427	6.5262
H(25)	-0.3690	-0.2904	-0.8425	6.5262

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

atom	x	y	z	B_{eq}
H(26)	-0.5641	-0.3405	-0.8045	7.6264
H(27)	-0.5155	-0.4111	-0.8832	7.6264
H(28)	-0.5218	-0.4743	-0.7007	6.3491
H(29)	-0.6055	-0.5206	-0.7758	6.3491
H(30)	-0.4440	-0.6007	-0.8448	4.7562
H(31)	-0.4463	-0.6364	-0.7438	4.7562

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

Table 2. Anisotropic Displacement Parameters

atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Ti(1)	0.0519(3)	0.0437(3)	0.0369(2)	0.0068(3)	-0.0038(2)	0.0009(2)
Cl(1)	0.0551(4)	0.0562(5)	0.0665(5)	-0.0089(4)	-0.0088(4)	-0.0056(4)
Cl(2)	0.0818(6)	0.0661(5)	0.0485(4)	-0.0153(6)	0.0115(4)	0.0101(4)
N(1)	0.046(1)	0.044(1)	0.0356(9)	0.003(1)	0.0009(9)	-0.001(1)
C(1)	0.151(7)	0.105(5)	0.179(6)	0.043(3)	-0.041(6)	0.075(5)
C(2)	0.141(4)	0.120(4)	0.085(2)	0.096(3)	-0.089(2)	-0.052(2)
C(3)	0.049(1)	0.074(3)	0.236(6)	0.021(2)	0.025(4)	0.043(4)
C(4)	0.123(4)	0.133(4)	0.060(2)	0.106(3)	0.021(2)	0.011(2)
C(5)	0.127(6)	0.050(2)	0.190(6)	0.032(3)	0.005(5)	0.020(4)
C(6)	0.123(7)	0.149(9)	1.17(6)	0.016(7)	0.06(2)	0.36(2)
C(7)	0.060(3)	0.127(6)	1.15(7)	0.007(3)	0.06(1)	0.22(2)
C(8)	0.051(2)	0.063(2)	0.035(1)	0.006(2)	-0.001(1)	-0.005(1)
C(9)	0.058(2)	0.124(4)	0.045(1)	-0.006(3)	0.009(2)	0.004(2)
C(10)	0.064(2)	0.158(4)	0.043(1)	0.021(3)	0.009(2)	0.001(2)
C(11)	0.115(3)	0.143(4)	0.044(2)	0.074(3)	-0.005(2)	-0.015(3)
C(12)	0.113(3)	0.081(3)	0.060(2)	0.025(3)	-0.024(2)	-0.025(2)
C(13)	0.080(3)	0.060(2)	0.052(1)	0.003(2)	-0.002(2)	-0.007(2)
C(14)	0.045(1)	0.037(1)	0.039(1)	0.000(1)	-0.002(1)	0.004(1)
C(15)	0.075(2)	0.045(2)	0.049(1)	-0.013(2)	-0.008(2)	0.014(1)
C(16)	0.101(2)	0.042(2)	0.064(2)	0.006(2)	-0.012(2)	0.005(2)
C(17)	0.090(2)	0.060(2)	0.090(3)	0.026(2)	-0.019(3)	-0.005(2)
C(18)	0.053(2)	0.070(2)	0.078(2)	0.018(2)	-0.006(2)	-0.011(2)
C(19)	0.048(1)	0.047(2)	0.055(2)	-0.003(1)	-0.001(1)	-0.004(2)

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 3. Bond Lengths($\AA$)

atom	atom	distance	atom	atom	distance
Ti(1)	Cl(1)	2.309(2)	Ti(1)	Cl(2)	2.296(2)
Ti(1)	N(1)	1.873(3)	Ti(1)	C(1)	2.39(1)
Ti(1)	C(2)	2.362(5)	Ti(1)	C(3)	2.356(6)
Ti(1)	C(4)	2.309(6)	Ti(1)	C(5)	2.326(8)
N(1)	C(8)	1.482(5)	N(1)	C(14)	1.505(6)
C(1)	C(2)	1.32(2)	C(1)	C(5)	1.15(2)
C(1)	C(6)	1.44(2)	C(2)	C(3)	1.48(1)
C(3)	C(4)	1.54(1)	C(3)	C(7)	1.36(2)
C(4)	C(5)	1.30(1)	C(8)	C(9)	1.521(7)
C(8)	C(13)	1.523(8)	C(9)	C(10)	1.558(8)
C(10)	C(11)	1.48(1)	C(11)	C(12)	1.50(1)
C(12)	C(13)	1.537(7)	C(14)	C(15)	1.529(6)
C(14)	C(19)	1.532(6)	C(15)	C(16)	1.535(9)
C(16)	C(17)	1.50(1)	C(17)	C(18)	1.52(1)
C(18)	C(19)	1.509(8)			

Table 4. Bond Lengths($\AA$)

atom	atom	distance	atom	atom	distance
C(2)	H(1)	0.95	C(4)	H(2)	0.95
C(5)	H(3)	0.94	C(6)	H(4)	0.94
C(6)	H(5)	0.97	C(6)	H(6)	0.95
C(7)	H(7)	0.95	C(7)	H(8)	0.94
C(7)	H(9)	0.95	C(8)	H(10)	0.95
C(9)	H(11)	0.95	C(9)	H(12)	0.95
C(10)	H(13)	0.95	C(10)	H(14)	0.95
C(11)	H(15)	0.95	C(11)	H(16)	0.95
C(12)	H(17)	0.94	C(12)	H(18)	0.95
C(13)	H(19)	0.95	C(13)	H(20)	0.95
C(14)	H(21)	0.95	C(15)	H(22)	0.95
C(15)	H(23)	0.95	C(16)	H(24)	0.95
C(16)	H(25)	0.95	C(17)	H(26)	0.95
C(17)	H(27)	0.95	C(18)	H(28)	0.95
C(18)	H(29)	0.95	C(19)	H(30)	0.95
C(19)	H(31)	0.95			

Table 5. Bond Angles(°)

atom	atom	atom	angle	atom	atom	atom	angle
Cl(1)	Ti(1)	Cl(2)	100.99(6)	Cl(1)	Ti(1)	N(1)	108.0(1)
Cl(1)	Ti(1)	C(1)	123.8(4)	Cl(1)	Ti(1)	C(2)	93.5(2)
Cl(1)	Ti(1)	C(3)	86.3(2)	Cl(1)	Ti(1)	C(4)	121.6(3)
Cl(1)	Ti(1)	C(5)	142.4(3)	Cl(2)	Ti(1)	N(1)	106.0(1)
Cl(2)	Ti(1)	C(1)	81.4(4)	Cl(2)	Ti(1)	C(2)	98.0(3)
Cl(2)	Ti(1)	C(3)	134.6(3)	Cl(2)	Ti(1)	C(4)	129.3(3)
Cl(2)	Ti(1)	C(5)	96.7(3)	N(1)	Ti(1)	C(1)	125.4(5)
N(1)	Ti(1)	C(2)	143.5(2)	N(1)	Ti(1)	C(3)	114.2(4)
N(1)	Ti(1)	C(4)	87.0(2)	N(1)	Ti(1)	C(5)	98.4(4)
C(1)	Ti(1)	C(2)	32.3(4)	C(1)	Ti(1)	C(3)	58.6(4)
C(1)	Ti(1)	C(4)	53.2(4)	C(1)	Ti(1)	C(5)	28.2(4)
C(2)	Ti(1)	C(3)	36.6(4)	C(2)	Ti(1)	C(4)	56.5(2)
C(2)	Ti(1)	C(5)	51.1(4)	C(3)	Ti(1)	C(4)	38.6(4)
C(3)	Ti(1)	C(5)	58.2(4)	C(4)	Ti(1)	C(5)	32.5(4)
Ti(1)	N(1)	C(8)	148.0(3)	Ti(1)	N(1)	C(14)	100.7(2)
C(8)	N(1)	C(14)	111.4(3)	Ti(1)	C(1)	C(2)	72.7(5)
Ti(1)	C(1)	C(5)	72.8(6)	Ti(1)	C(1)	C(6)	123(1)
C(2)	C(1)	C(5)	109(1)	C(2)	C(1)	C(6)	153(2)
C(5)	C(1)	C(6)	96(2)	Ti(1)	C(2)	C(1)	75.0(5)
Ti(1)	C(2)	C(3)	71.5(4)	C(1)	C(2)	C(3)	111.9(9)
Ti(1)	C(3)	C(2)	72.0(4)	Ti(1)	C(3)	C(4)	69.1(3)
Ti(1)	C(3)	C(7)	123.4(7)	C(2)	C(3)	C(4)	94.0(6)
C(2)	C(3)	C(7)	121(2)	C(4)	C(3)	C(7)	144(2)
Ti(1)	C(4)	C(3)	72.4(3)	Ti(1)	C(4)	C(5)	74.4(5)

Table 5. Bond Angles(°) (continued)

atom	atom	atom	angle	atom	atom	atom	angle
C(3)	C(4)	C(5)	106.2(8)	Ti(1)	C(5)	C(1)	79.0(8)
Ti(1)	C(5)	C(4)	73.0(4)	C(1)	C(5)	C(4)	118(1)
N(1)	C(8)	C(9)	112.6(4)	N(1)	C(8)	C(13)	112.2(4)
C(9)	C(8)	C(13)	113.7(5)	C(8)	C(9)	C(10)	108.3(5)
C(9)	C(10)	C(11)	112.4(6)	C(10)	C(11)	C(12)	111.8(6)
C(11)	C(12)	C(13)	111.5(7)	C(8)	C(13)	C(12)	109.3(5)
N(1)	C(14)	C(15)	113.4(4)	N(1)	C(14)	C(19)	112.6(3)
C(15)	C(14)	C(19)	112.4(4)	C(14)	C(15)	C(16)	109.5(4)
C(15)	C(16)	C(17)	112.8(5)	C(16)	C(17)	C(18)	110.0(5)
C(17)	C(18)	C(19)	111.2(5)	C(14)	C(19)	C(18)	111.5(4)