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(*N*-*tert*-Butylimido)chlorido[dimethyl-(1*H*-pyrrol-2-ylmethyl)amine-κ^2N,N']-bis(pyridine-κN)titanium(IV) toluene solvate

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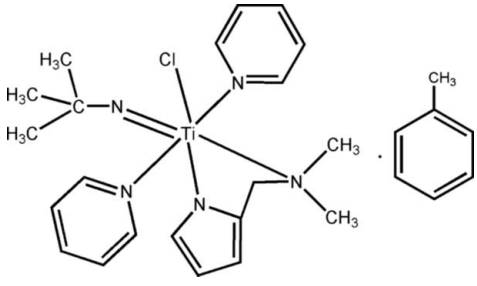
Key indicators: single-crystal X-ray study; T = 153 K; mean σ(C–C) = 0.006 Å; R factor = 0.073; wR factor = 0.138; data-to-parameter ratio = 16.2.

The title compound, $[Ti(C_4H_9N)Cl(C_5H_5N)_2(C_7H_{11}N_2)]\cdot C_7H_8$, has a mononuclear structure featuring a six-coordinate distorted octahedral titanium(IV) centre. The central Ti^{IV} ion is coordinated by two N atoms from a dimethyl(1*H*-pyrrol-2-ylmethyl)amine molecule, two N atoms from two pyridine molecules, one N atom from a *N*-*tert*-butylimido ligand and one Cl atom.

Related literature

For related literature, see: Wigley (1994); Mountford (1997).



Experimental

Crystal data

$[Ti(C_4H_9N)Cl(C_5H_5N)_2(C_7H_{11}N_2)]\cdot C_7H_8$
M_r = 527.98
Monoclinic, $P2_1/n$
a = 10.5646 (14) Å
b = 15.605 (2) Å
c = 17.432 (2) Å
β = 95.507 (4)°
V = 2860.6 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.42 mm^{-1}
T = 153 (2) K
0.30 × 0.20 × 0.15 mm

Data collection

Rigaku Mercury diffractometer
Absorption correction: multi-scan (*REQAB*; Jacobson, 1998)
T_{min} = 0.745, T_{max} = 0.939
27774 measured reflections
5227 independent reflections
3910 reflections with $I > 2\sigma(I)$
R_{int} = 0.088

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.073
$wR(F^2)$ = 0.138
S = 1.14
5227 reflections
323 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.37 e Å^{-3}
$\Delta\rho_{min}$ = −0.35 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ti—N3	1.687 (3)	Ti—N5	2.267 (3)
Ti—N1	2.071 (3)	Ti—Cl	2.4195 (11)
Ti—N4	2.245 (3)	Ti—N2	2.508 (3)
N3—Ti—N1	100.75 (13)	N1—Ti—Cl	156.85 (9)
N3—Ti—N4	90.05 (13)	N4—Ti—Cl	92.66 (8)
N1—Ti—N4	91.54 (11)	N5—Ti—Cl	89.01 (8)
N3—Ti—N5	92.98 (13)	N3—Ti—N2	170.61 (13)
N1—Ti—N5	85.59 (11)	N1—Ti—N2	74.53 (10)
N4—Ti—N5	176.17 (11)	N4—Ti—N2	82.05 (10)
N3—Ti—Cl	102.00 (11)	N5—Ti—N2	94.72 (10)

Data collection: *CrystalClear* (Rigaku/MSC, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalStructure* (Rigaku/MSC, 2005); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2001); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2268).

References

Bruker (2001). *SHELXTL*. Version 6.12. Bruker AXS Inc., Madison, Wisconsin, USA.
Jacobson, R. A. (1998). *REQAB*. Version 1.1. MSC, The Woodlands, Texas, USA.
Mountford, P. (1997). *J. Chem. Soc. Chem. Commun.* pp. 2127–2134.
Rigaku/MSC (2005). *CrystalClear* (Version 1.36) and *CrystalStructure* (Version 3.7.0). Rigaku/MSC Inc., The Woodlands, Texas, USA.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Wigley, D. E. (1994). *Prog. Inorg. Chem.* **42**, 239–482.

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supplementary materials

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(*N-tert*-Butylimido)chlorido[dimethyl(1*H*-pyrrol-2-ylmethyl)amine-κ^2*N*,*N*']bis(pyridine-κ*N*)titanium(IV) toluene solvate

D.-J. Gong, H. Wei and Y.-H. Li

Comment

Over the last two decades, the chemistry of titanium–imido complexes has received considerable attention (Wigley, 1994). It has been shown that these complexes can be utilized in a wide variety of stoichiometric and sometimes catalytic coupling reactions with unsaturated substrates. A general entry point to new titanium–imido chemistry is gained *via* the readily prepared synthons [Ti(NR)Cl2(py)3] (*R* = ^{t}Bu or Aryl) (Mountford, 1997).

The title compound, (I), was prepared by combining equimolar amounts of Ti(N^{t}Bu)Cl$_2$py$_3$ with Lidap [*N*,*N*-dimethyl-(1-Li-pyrrol-2-yl)methanamine] in toluene. It possesses a mononuclear structure featuring a six-coordinate octahedral titanium(IV) centre.

Experimental

To a near-frozen solution of Ti(N^{t}Bu)Cl$_2$py$_3$ (Mountford, 1997) (1.234 g, 2.9 mmol) in toluene (10 ml), Lidap [*N*,*N*-dimethyl-(1-Li-pyrrol-2-yl)methanamine] (376.1 mg, 2.9 mmol) in toluene (10 ml) was added dropwise. After stirring at room temperature for 12 h, the resulting solution was filtered to remove solids. Volatiles were removed from the solution under reduced pressure to yield the orange crude product, which was washed with toluene and filtered to remove a trace amount of LiCl, then recrystallized from toluene (yield 920 mg, 60%).

Refinement

All H atoms were positioned geometrically and refined using a riding model (including free rotation about the ethanol C—C bond), with C—H = 0.95–0.99 Å and with U_{iso}(H) = 1.2 (1.5 for methyl groups) times U_{eq}(C).

Figures

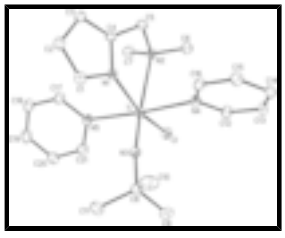


Fig. 1. The molecular structure of (I), with atom labels and 50% probability displacement ellipsoids.

(*N*-*tert*-Butylimido)chlorido[dimethyl(1*H*-pyrrol-2-ylmethyl)amine- κ^2N,N']bis(pyridine-κ*N*)titanium(IV) toluene solvate

Crystal data

$[Ti(C_4H_9N)Cl(C_5H_5N)_2(C_7H_{11}N_2)]\cdot C_7H_8$	F_{000} = 1120
M_r = 527.98	D_x = 1.226 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71070 Å
Hall symbol: -P 2yn	Cell parameters from 7469 reflections
a = 10.5646 (14) Å	θ = 3.2–25.3º
b = 15.605 (2) Å	μ = 0.42 mm^{-1}
c = 17.432 (2) Å	T = 153 (2) K
β = 95.507 (4)º	Block, orange-yellow
V = 2860.6 (6) Å^3	0.30 × 0.20 × 0.15 mm
Z = 4	

Data collection

Rigaku Mercury diffractometer	5227 independent reflections
Radiation source: fine-focus sealed tube	3910 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.088
Detector resolution: 7.31 pixels mm^{-1}	θ_{max} = 25.4º
T = 153(2) K	θ_{min} = 3.2º
ω scans	h = −12→12
Absorption correction: multi-scan (REQAB; Jacobson, 1998)	k = −18→18
T_{min} = 0.745, T_{max} = 0.939	l = −20→20
27774 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.073	H-atom parameters constrained
$wR(F^2)$ = 0.138	$w = 1/[\sigma^2(F_o^2) + (0.0334P)^2 + 3.8455P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.14	$(\Delta/\sigma)_{max}$ = 0.002
5227 reflections	$\Delta\rho_{max}$ = 0.37 e Å^{-3}
323 parameters	$\Delta\rho_{min}$ = −0.35 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

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

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

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($Å^2$)

	x	*y*	*z*	U_{iso}*/U_{eq}
Ti	0.28648 (6)	0.78065 (4)	0.61619 (4)	0.02125 (18)
Cl	0.20129 (9)	0.77167 (6)	0.73991 (5)	0.0287 (2)
N1	0.4229 (3)	0.77268 (18)	0.53870 (16)	0.0208 (6)
N2	0.4963 (3)	0.78443 (19)	0.69666 (16)	0.0229 (7)
N3	0.1535 (3)	0.79291 (19)	0.55539 (17)	0.0249 (7)
N4	0.3083 (3)	0.92353 (19)	0.62425 (17)	0.0237 (7)
N5	0.2788 (3)	0.63571 (19)	0.60822 (17)	0.0235 (7)
C1	0.4128 (4)	0.7420 (2)	0.4639 (2)	0.0269 (9)
H1	0.3355	0.7387	0.4313	0.032*
C2	0.5283 (4)	0.7173 (2)	0.4432 (2)	0.0286 (9)
H2	0.5459	0.6947	0.3948	0.034*
C3	0.6175 (4)	0.7320 (2)	0.5086 (2)	0.0272 (9)
H3	0.7063	0.7207	0.5122	0.033*
C4	0.5508 (3)	0.7656 (2)	0.5649 (2)	0.0224 (8)
C5	0.5915 (3)	0.8026 (2)	0.6417 (2)	0.0270 (9)
H5A	0.6746	0.7780	0.6615	0.032*
H5B	0.6021	0.8654	0.6369	0.032*
C6	0.5096 (4)	0.8476 (3)	0.7600 (2)	0.0323 (9)
H6A	0.5942	0.8424	0.7880	0.048*
H6B	0.4446	0.8369	0.7953	0.048*
H6C	0.4988	0.9056	0.7387	0.048*
C7	0.5217 (4)	0.6992 (2)	0.7319 (2)	0.0326 (10)
H7A	0.6038	0.7002	0.7631	0.049*
H7B	0.5237	0.6561	0.6912	0.049*
H7C	0.4543	0.6850	0.7646	0.049*
C8	0.0369 (3)	0.8149 (2)	0.5080 (2)	0.0290 (9)
C9	−0.0585 (4)	0.8480 (3)	0.5615 (3)	0.0391 (11)
H9A	−0.0748	0.8034	0.5989	0.059*
H9B	−0.1383	0.8630	0.5310	0.059*
H9C	−0.0238	0.8989	0.5889	0.059*
C10	0.0665 (4)	0.8847 (3)	0.4507 (3)	0.0457 (12)
H10A	0.0970	0.9361	0.4790	0.069*
H10B	−0.0108	0.8985	0.4173	0.069*

H10C	0.1323	0.8641	0.4192	0.069*
C11	−0.0137 (4)	0.7350 (3)	0.4650 (2)	0.0393 (10)
H11A	0.0518	0.7120	0.4346	0.059*
H11B	−0.0894	0.7499	0.4307	0.059*
H11C	−0.0361	0.6918	0.5022	0.059*
C12	0.2603 (4)	0.9699 (2)	0.6796 (2)	0.0281 (9)
H12	0.2242	0.9405	0.7199	0.034*
C13	0.2613 (4)	1.0579 (2)	0.6802 (2)	0.0304 (9)
H13	0.2262	1.0883	0.7203	0.037*
C14	0.3131 (4)	1.1015 (2)	0.6226 (2)	0.0306 (9)
H14	0.3157	1.1624	0.6226	0.037*
C15	0.3614 (4)	1.0553 (2)	0.5648 (2)	0.0302 (9)
H15	0.3969	1.0837	0.5237	0.036*
C16	0.3574 (3)	0.9673 (2)	0.5676 (2)	0.0270 (9)
H16	0.3909	0.9358	0.5275	0.032*
C17	0.3656 (4)	0.5853 (2)	0.5789 (2)	0.0287 (9)
H17	0.4418	0.6111	0.5653	0.034*
C18	0.3502 (4)	0.4982 (2)	0.5674 (2)	0.0335 (10)
H18	0.4155	0.4649	0.5481	0.040*
C19	0.2380 (4)	0.4605 (3)	0.5845 (2)	0.0382 (10)
H19	0.2244	0.4008	0.5770	0.046*
C20	0.1462 (4)	0.5112 (3)	0.6126 (2)	0.0361 (10)
H20	0.0679	0.4871	0.6246	0.043*
C21	0.1701 (4)	0.5976 (2)	0.6231 (2)	0.0307 (9)
H21	0.1058	0.6320	0.6421	0.037*
C22	0.2942 (4)	0.5083 (3)	0.2703 (2)	0.0376 (10)
C23	0.2439 (4)	0.4756 (3)	0.2002 (3)	0.0394 (11)
H23	0.2702	0.4990	0.1540	0.047*
C24	0.1569 (4)	0.4101 (3)	0.1958 (3)	0.0536 (13)
H24	0.1227	0.3894	0.1469	0.064*
C25	0.1191 (5)	0.3744 (3)	0.2618 (4)	0.0651 (16)
H25	0.0588	0.3291	0.2588	0.078*
C26	0.1692 (5)	0.4047 (4)	0.3321 (4)	0.0639 (16)
H26	0.1446	0.3797	0.3781	0.077*
C27	0.2549 (4)	0.4711 (3)	0.3363 (3)	0.0490 (12)
H27	0.2879	0.4921	0.3854	0.059*
C28	0.3885 (5)	0.5808 (3)	0.2746 (3)	0.0656 (15)
H28A	0.3721	0.6174	0.2291	0.098*
H28B	0.4749	0.5575	0.2764	0.098*
H28C	0.3798	0.6146	0.3212	0.098*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Ti	0.0233 (3)	0.0179 (3)	0.0231 (4)	0.0003 (3)	0.0051 (3)	0.0004 (3)
Cl	0.0340 (5)	0.0250 (5)	0.0285 (5)	−0.0003 (4)	0.0115 (4)	0.0026 (4)
N1	0.0216 (15)	0.0168 (15)	0.0245 (16)	0.0016 (13)	0.0041 (13)	0.0002 (12)
N2	0.0256 (16)	0.0201 (16)	0.0235 (16)	−0.0006 (14)	0.0047 (13)	−0.0001 (13)

N3	0.0225 (16)	0.0243 (17)	0.0282 (17)	0.0018 (14)	0.0035 (13)	0.0005 (13)
N4	0.0266 (17)	0.0210 (16)	0.0233 (17)	0.0009 (13)	0.0023 (14)	0.0003 (13)
N5	0.0257 (17)	0.0225 (16)	0.0226 (17)	0.0028 (14)	0.0033 (13)	0.0010 (13)
C1	0.035 (2)	0.023 (2)	0.023 (2)	−0.0019 (17)	0.0052 (17)	0.0020 (15)
C2	0.035 (2)	0.022 (2)	0.030 (2)	−0.0048 (18)	0.0130 (17)	−0.0021 (16)
C3	0.025 (2)	0.023 (2)	0.035 (2)	0.0018 (17)	0.0074 (17)	0.0014 (17)
C4	0.0185 (17)	0.0201 (19)	0.029 (2)	0.0002 (15)	0.0052 (15)	0.0052 (15)
C5	0.0227 (19)	0.030 (2)	0.029 (2)	−0.0041 (17)	0.0039 (16)	−0.0007 (16)
C6	0.033 (2)	0.039 (2)	0.025 (2)	−0.0010 (19)	0.0017 (18)	−0.0051 (18)
C7	0.031 (2)	0.033 (2)	0.033 (2)	0.0028 (18)	0.0013 (18)	0.0073 (17)
C8	0.027 (2)	0.029 (2)	0.031 (2)	−0.0002 (17)	0.0038 (17)	0.0015 (17)
C9	0.026 (2)	0.043 (3)	0.048 (3)	0.006 (2)	0.0004 (19)	−0.004 (2)
C10	0.037 (3)	0.051 (3)	0.048 (3)	−0.002 (2)	−0.004 (2)	0.018 (2)
C11	0.035 (2)	0.046 (3)	0.036 (2)	−0.006 (2)	−0.0017 (19)	−0.010 (2)
C12	0.036 (2)	0.022 (2)	0.027 (2)	0.0023 (17)	0.0084 (17)	0.0025 (16)
C13	0.041 (2)	0.0199 (19)	0.031 (2)	0.0055 (18)	0.0044 (18)	−0.0031 (17)
C14	0.034 (2)	0.0186 (19)	0.038 (2)	0.0013 (17)	−0.0021 (19)	−0.0008 (17)
C15	0.034 (2)	0.022 (2)	0.036 (2)	−0.0012 (18)	0.0088 (18)	0.0077 (17)
C16	0.029 (2)	0.025 (2)	0.028 (2)	0.0031 (17)	0.0097 (17)	0.0016 (16)
C17	0.034 (2)	0.020 (2)	0.033 (2)	0.0014 (17)	0.0088 (18)	0.0026 (16)
C18	0.041 (2)	0.022 (2)	0.038 (2)	0.0041 (19)	0.0082 (19)	0.0003 (17)
C19	0.050 (3)	0.021 (2)	0.044 (3)	−0.003 (2)	0.011 (2)	−0.0039 (18)
C20	0.037 (2)	0.029 (2)	0.043 (3)	−0.0065 (19)	0.008 (2)	−0.0006 (19)
C21	0.034 (2)	0.024 (2)	0.035 (2)	−0.0006 (18)	0.0067 (18)	0.0039 (17)
C22	0.039 (2)	0.037 (2)	0.037 (3)	0.012 (2)	0.001 (2)	0.0002 (19)
C23	0.038 (2)	0.040 (3)	0.041 (3)	0.009 (2)	0.005 (2)	−0.005 (2)
C24	0.039 (3)	0.054 (3)	0.065 (4)	0.014 (3)	−0.005 (3)	−0.017 (3)
C25	0.032 (3)	0.049 (3)	0.116 (5)	0.003 (2)	0.014 (3)	0.000 (3)
C26	0.045 (3)	0.074 (4)	0.077 (4)	0.016 (3)	0.024 (3)	0.030 (3)
C27	0.043 (3)	0.062 (3)	0.041 (3)	0.016 (3)	0.000 (2)	0.003 (2)
C28	0.066 (4)	0.060 (4)	0.070 (4)	−0.011 (3)	0.000 (3)	−0.008 (3)

Geometric parameters (Å, °)

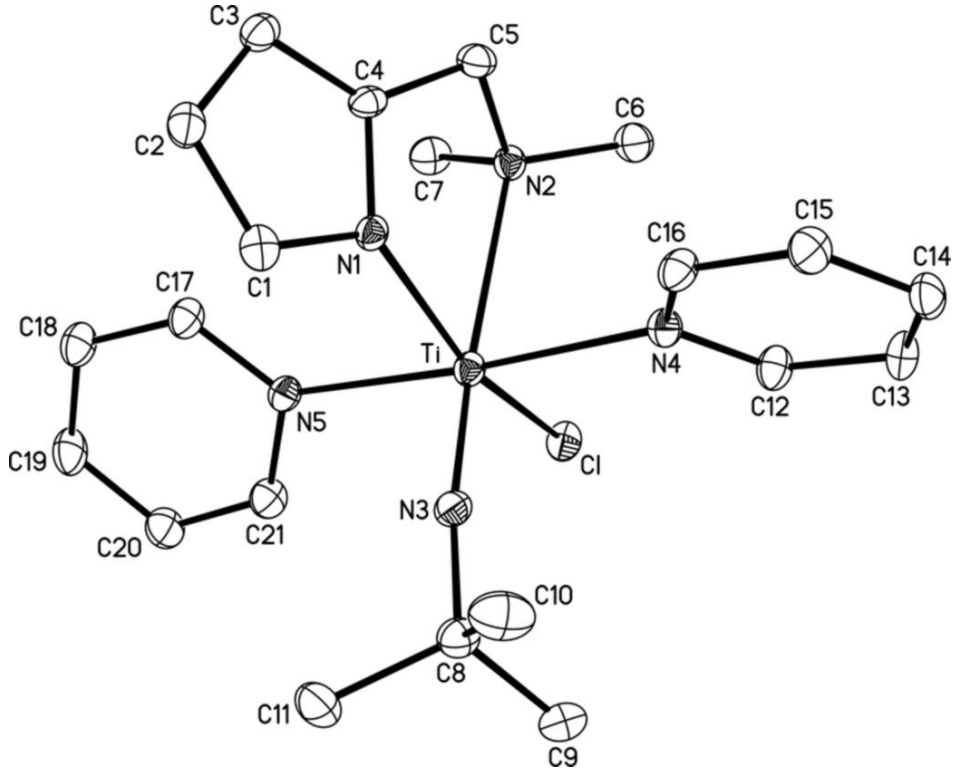
Ti—N3	1.687 (3)	C10—H10B	0.9800
Ti—N1	2.071 (3)	C10—H10C	0.9800
Ti—N4	2.245 (3)	C11—H11A	0.9800
Ti—N5	2.267 (3)	C11—H11B	0.9800
Ti—Cl	2.4195 (11)	C11—H11C	0.9800
Ti—N2	2.508 (3)	C12—C13	1.374 (5)
N1—C1	1.383 (4)	C12—H12	0.9500
N1—C4	1.389 (4)	C13—C14	1.370 (5)
N2—C6	1.477 (5)	C13—H13	0.9500
N2—C7	1.478 (5)	C14—C15	1.377 (5)
N2—C5	1.482 (4)	C14—H14	0.9500
N3—C8	1.457 (5)	C15—C16	1.375 (5)
N4—C12	1.343 (5)	C15—H15	0.9500
N4—C16	1.345 (4)	C16—H16	0.9500
N5—C21	1.341 (5)	C17—C18	1.381 (5)

N5—C17	1.346 (5)	C17—H17	0.9500
C1—C2	1.361 (5)	C18—C19	1.381 (6)
C1—H1	0.9500	C18—H18	0.9500
C2—C3	1.426 (5)	C19—C20	1.377 (6)
C2—H2	0.9500	C19—H19	0.9500
C3—C4	1.367 (5)	C20—C21	1.380 (5)
C3—H3	0.9500	C20—H20	0.9500
C4—C5	1.484 (5)	C21—H21	0.9500
C5—H5A	0.9900	C22—C23	1.383 (6)
C5—H5B	0.9900	C22—C27	1.388 (6)
C6—H6A	0.9800	C22—C28	1.504 (6)
C6—H6B	0.9800	C23—C24	1.372 (6)
C6—H6C	0.9800	C23—H23	0.9500
C7—H7A	0.9800	C24—C25	1.371 (7)
C7—H7B	0.9800	C24—H24	0.9500
C7—H7C	0.9800	C25—C26	1.373 (8)
C8—C11	1.524 (5)	C25—H25	0.9500
C8—C9	1.528 (5)	C26—C27	1.373 (7)
C8—C10	1.530 (5)	C26—H26	0.9500
C9—H9A	0.9800	C27—H27	0.9500
C9—H9B	0.9800	C28—H28A	0.9800
C9—H9C	0.9800	C28—H28B	0.9800
C10—H10A	0.9800	C28—H28C	0.9800
N3—Ti—N1	100.75 (13)	C8—C9—H9C	109.5
N3—Ti—N4	90.05 (13)	H9A—C9—H9C	109.5
N1—Ti—N4	91.54 (11)	H9B—C9—H9C	109.5
N3—Ti—N5	92.98 (13)	C8—C10—H10A	109.5
N1—Ti—N5	85.59 (11)	C8—C10—H10B	109.5
N4—Ti—N5	176.17 (11)	H10A—C10—H10B	109.5
N3—Ti—Cl	102.00 (11)	C8—C10—H10C	109.5
N1—Ti—Cl	156.85 (9)	H10A—C10—H10C	109.5
N4—Ti—Cl	92.66 (8)	H10B—C10—H10C	109.5
N5—Ti—Cl	89.01 (8)	C8—C11—H11A	109.5
N3—Ti—N2	170.61 (13)	C8—C11—H11B	109.5
N1—Ti—N2	74.53 (10)	H11A—C11—H11B	109.5
N4—Ti—N2	82.05 (10)	C8—C11—H11C	109.5
N5—Ti—N2	94.72 (10)	H11A—C11—H11C	109.5
Cl—Ti—N2	83.52 (7)	H11B—C11—H11C	109.5
C1—N1—C4	105.5 (3)	N4—C12—C13	122.8 (4)
C1—N1—Ti	129.8 (2)	N4—C12—H12	118.6
C4—N1—Ti	120.4 (2)	C13—C12—H12	118.6
C6—N2—C7	106.9 (3)	C14—C13—C12	119.6 (4)
C6—N2—C5	109.5 (3)	C14—C13—H13	120.2
C7—N2—C5	109.5 (3)	C12—C13—H13	120.2
C6—N2—Ti	116.7 (2)	C13—C14—C15	118.5 (3)
C7—N2—Ti	109.0 (2)	C13—C14—H14	120.7
C5—N2—Ti	105.1 (2)	C15—C14—H14	120.7
C8—N3—Ti	172.1 (3)	C16—C15—C14	118.9 (4)
C12—N4—C16	116.9 (3)	C16—C15—H15	120.6

C12—N4—Ti	122.4 (2)	C14—C15—H15	120.6
C16—N4—Ti	120.2 (2)	N4—C16—C15	123.3 (4)
C21—N5—C17	116.2 (3)	N4—C16—H16	118.4
C21—N5—Ti	117.2 (2)	C15—C16—H16	118.4
C17—N5—Ti	125.8 (2)	N5—C17—C18	123.7 (4)
C2—C1—N1	111.0 (3)	N5—C17—H17	118.1
C2—C1—H1	124.5	C18—C17—H17	118.1
N1—C1—H1	124.5	C19—C18—C17	118.6 (4)
C1—C2—C3	106.4 (3)	C19—C18—H18	120.7
C1—C2—H2	126.8	C17—C18—H18	120.7
C3—C2—H2	126.8	C20—C19—C18	118.7 (4)
C4—C3—C2	106.8 (3)	C20—C19—H19	120.6
C4—C3—H3	126.6	C18—C19—H19	120.6
C2—C3—H3	126.6	C19—C20—C21	118.8 (4)
C3—C4—N1	110.3 (3)	C19—C20—H20	120.6
C3—C4—C5	132.3 (3)	C21—C20—H20	120.6
N1—C4—C5	117.0 (3)	N5—C21—C20	123.9 (4)
N2—C5—C4	110.7 (3)	N5—C21—H21	118.1
N2—C5—H5A	109.5	C20—C21—H21	118.1
C4—C5—H5A	109.5	C23—C22—C27	117.3 (4)
N2—C5—H5B	109.5	C23—C22—C28	121.2 (4)
C4—C5—H5B	109.5	C27—C22—C28	121.4 (4)
H5A—C5—H5B	108.1	C24—C23—C22	121.5 (5)
N2—C6—H6A	109.5	C24—C23—H23	119.2
N2—C6—H6B	109.5	C22—C23—H23	119.2
H6A—C6—H6B	109.5	C25—C24—C23	120.2 (5)
N2—C6—H6C	109.5	C25—C24—H24	119.9
H6A—C6—H6C	109.5	C23—C24—H24	119.9
H6B—C6—H6C	109.5	C24—C25—C26	119.4 (5)
N2—C7—H7A	109.5	C24—C25—H25	120.3
N2—C7—H7B	109.5	C26—C25—H25	120.3
H7A—C7—H7B	109.5	C25—C26—C27	120.2 (5)
N2—C7—H7C	109.5	C25—C26—H26	119.9
H7A—C7—H7C	109.5	C27—C26—H26	119.9
H7B—C7—H7C	109.5	C26—C27—C22	121.3 (5)
N3—C8—C11	108.9 (3)	C26—C27—H27	119.4
N3—C8—C9	107.8 (3)	C22—C27—H27	119.4
C11—C8—C9	110.8 (3)	C22—C28—H28A	109.5
N3—C8—C10	108.9 (3)	C22—C28—H28B	109.5
C11—C8—C10	110.2 (3)	H28A—C28—H28B	109.5
C9—C8—C10	110.2 (3)	C22—C28—H28C	109.5
C8—C9—H9A	109.5	H28A—C28—H28C	109.5
C8—C9—H9B	109.5	H28B—C28—H28C	109.5
H9A—C9—H9B	109.5		
N3—Ti—N1—C1	−29.6 (3)	N1—Ti—N5—C17	15.3 (3)
N4—Ti—N1—C1	−120.0 (3)	N4—Ti—N5—C17	−26.3 (17)
N5—Ti—N1—C1	62.6 (3)	Cl—Ti—N5—C17	−142.1 (3)
Cl—Ti—N1—C1	139.6 (3)	N2—Ti—N5—C17	−58.7 (3)
N2—Ti—N1—C1	158.7 (3)	C4—N1—C1—C2	−0.4 (4)

N3—Ti—N1—C4	177.3 (3)	Ti—N1—C1—C2	−156.6 (3)
N4—Ti—N1—C4	86.9 (3)	N1—C1—C2—C3	0.6 (4)
N5—Ti—N1—C4	−90.5 (3)	C1—C2—C3—C4	−0.5 (4)
Cl—Ti—N1—C4	−13.5 (4)	C2—C3—C4—N1	0.3 (4)
N2—Ti—N1—C4	5.6 (2)	C2—C3—C4—C5	−172.2 (4)
N3—Ti—N2—C6	75.7 (8)	C1—N1—C4—C3	0.1 (4)
N1—Ti—N2—C6	136.6 (3)	Ti—N1—C4—C3	158.9 (2)
N4—Ti—N2—C6	42.7 (3)	C1—N1—C4—C5	173.8 (3)
N5—Ti—N2—C6	−139.3 (3)	Ti—N1—C4—C5	−27.3 (4)
Cl—Ti—N2—C6	−50.9 (2)	C6—N2—C5—C4	−157.6 (3)
N3—Ti—N2—C7	−163.1 (7)	C7—N2—C5—C4	85.6 (4)
N1—Ti—N2—C7	−102.3 (2)	Ti—N2—C5—C4	−31.5 (3)
N4—Ti—N2—C7	163.9 (2)	C3—C4—C5—N2	−147.3 (4)
N5—Ti—N2—C7	−18.2 (2)	N1—C4—C5—N2	40.7 (4)
Cl—Ti—N2—C7	70.3 (2)	Ti—N3—C8—C11	−167.5 (18)
N3—Ti—N2—C5	−45.8 (8)	Ti—N3—C8—C9	−47 (2)
N1—Ti—N2—C5	15.0 (2)	Ti—N3—C8—C10	72 (2)
N4—Ti—N2—C5	−78.8 (2)	C16—N4—C12—C13	−0.7 (5)
N5—Ti—N2—C5	99.1 (2)	Ti—N4—C12—C13	−172.8 (3)
Cl—Ti—N2—C5	−172.4 (2)	N4—C12—C13—C14	−0.1 (6)
N1—Ti—N3—C8	−117.7 (19)	C12—C13—C14—C15	0.9 (6)
N4—Ti—N3—C8	−26.1 (19)	C13—C14—C15—C16	−0.9 (6)
N5—Ti—N3—C8	156.2 (19)	C12—N4—C16—C15	0.8 (5)
Cl—Ti—N3—C8	66.6 (19)	Ti—N4—C16—C15	173.1 (3)
N2—Ti—N3—C8	−59 (2)	C14—C15—C16—N4	0.0 (6)
N3—Ti—N4—C12	92.7 (3)	C21—N5—C17—C18	−3.1 (5)
N1—Ti—N4—C12	−166.5 (3)	Ti—N5—C17—C18	−172.6 (3)
N5—Ti—N4—C12	−125.0 (15)	N5—C17—C18—C19	2.1 (6)
Cl—Ti—N4—C12	−9.3 (3)	C17—C18—C19—C20	−0.3 (6)
N2—Ti—N4—C12	−92.4 (3)	C18—C19—C20—C21	−0.4 (6)
N3—Ti—N4—C16	−79.1 (3)	C17—N5—C21—C20	2.3 (6)
N1—Ti—N4—C16	21.6 (3)	Ti—N5—C21—C20	172.7 (3)
N5—Ti—N4—C16	63.1 (17)	C19—C20—C21—N5	−0.7 (6)
Cl—Ti—N4—C16	178.9 (3)	C27—C22—C23—C24	1.1 (6)
N2—Ti—N4—C16	95.8 (3)	C28—C22—C23—C24	−179.3 (4)
N3—Ti—N5—C21	−53.5 (3)	C22—C23—C24—C25	−1.0 (7)
N1—Ti—N5—C21	−154.1 (3)	C23—C24—C25—C26	−0.1 (7)
N4—Ti—N5—C21	164.3 (15)	C24—C25—C26—C27	1.0 (7)
Cl—Ti—N5—C21	48.5 (3)	C25—C26—C27—C22	−0.9 (7)
N2—Ti—N5—C21	131.9 (3)	C23—C22—C27—C26	−0.1 (6)
N3—Ti—N5—C17	115.9 (3)	C28—C22—C27—C26	−179.7 (5)

Fig. 1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