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## Comment

Titanium tetrahalides are strong Lewis acids and form Lewis acid–Lewis base adducts with electron-pair donors (Emeléus & Rao, 1958; Rao, 1960; Mazo *et al.*, 1987; Hensen, Lemke & Näther, 1997; Hensen, Pickel *et al.*, 1997). Thus, titanium tetrachloride forms complexes with 3,4-dimethylpyridine (3,4-dimepy), 4-methylpyridine (4-mepy) and *N*-methylimidazole (NMI) in a 1:2 ratio. The structures of (I), (II) and (III) have been determined as part of our studies on the structures and properties of adducts of titanium tetrahalides and tertiary amines.

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## Addition compounds of titanium tetrachloride with aromatic nitrogen bases

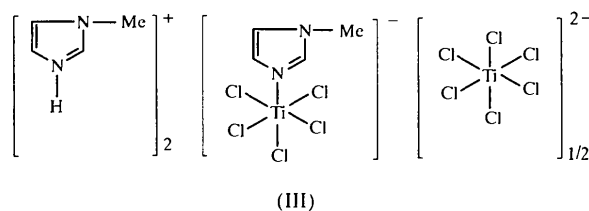
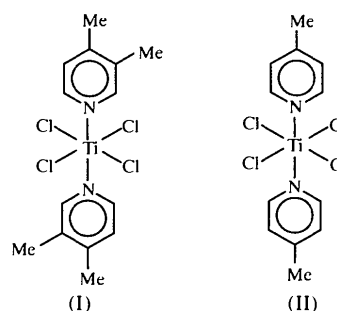
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### Abstract

Three reaction products of an addition reaction of titanium tetrachloride with aromatic nitrogen bases are presented. *trans*-Tetrachlorobis(3,4-dimethylpyridine)-titanium, [TiCl<sub>4</sub>(C<sub>7</sub>H<sub>9</sub>N)<sub>2</sub>], (I), and *trans*-tetrachlorobis(4-methylpyridine)titanium, [TiCl<sub>4</sub>(C<sub>6</sub>H<sub>7</sub>N)<sub>2</sub>], (II), are both neutral complexes in which the central Ti atom is bonded to four Cl and two N atoms of two 3,4-dimethylpyridine and 4-methylpyridine ligands, respectively, resulting in a slightly distorted octahedral environment around the Ti atom. The methylpyridine ligands occupy axial positions, with the four chloro ligands in the equatorial plane. The ionic structure of bis(1-methylimidazolium) hexachlorotitanate(IV) pentachloro(*N*-methylimidazole-*N*<sup>3</sup>)-titanate(IV), (C<sub>4</sub>H<sub>7</sub>N<sub>2</sub>)<sub>2</sub>[TiCl<sub>5</sub>(C<sub>4</sub>H<sub>6</sub>N<sub>2</sub>)] [TiCl<sub>6</sub>]<sub>0.5</sub>, (III), consists of a pentachloro(*N*-methylimidazole-*N*<sup>3</sup>)titanate anion and half a [TiCl<sub>6</sub>]<sup>2-</sup> complex anion, in both of which the Ti atom appears in a nearly perfect octahedral coordination sphere with two *N*-methylimidazolium cations as counter-ions.



Compounds (I) and (II) belong to the symmetry point group  $C_{2h}$ , with the Ti atom on a special position of site symmetry  $2/m$  and the pyridine moiety located on a crystallographic mirror plane. Only the Cl atoms and two H atoms of each methyl group occupy a general position. The Ti centre appears in a nearly ideal octahedral environment, where the two methylpyridine ligands occupy axial positions and the four chloro ligands lie in the equatorial plane. There are only minor deviations from perfect octahedral coordination. The planes of the methylpyridine rings almost exactly bisect the Cl–Ti–Cl angle. A least-squares fit of (I) and (II), fitting all non-H atoms excluding the methyl groups (r.m.s. deviation of 0.014 Å), shows that these two structures do not show any significant difference. The only comparable structure to (I) and (II) retrieved from the Cambridge Structural Database (Version 5.12 of April 1998; Allen & Kennard, 1993), is TiCl<sub>4</sub>(py)<sub>2</sub> (Mazo *et al.*, 1987). The geometrical parameters of all three structures are in excellent agreement. Thus, the conclusion can be drawn that the methyl groups in (I) and (II) have no influence on the geometry of the rest of the molecule.

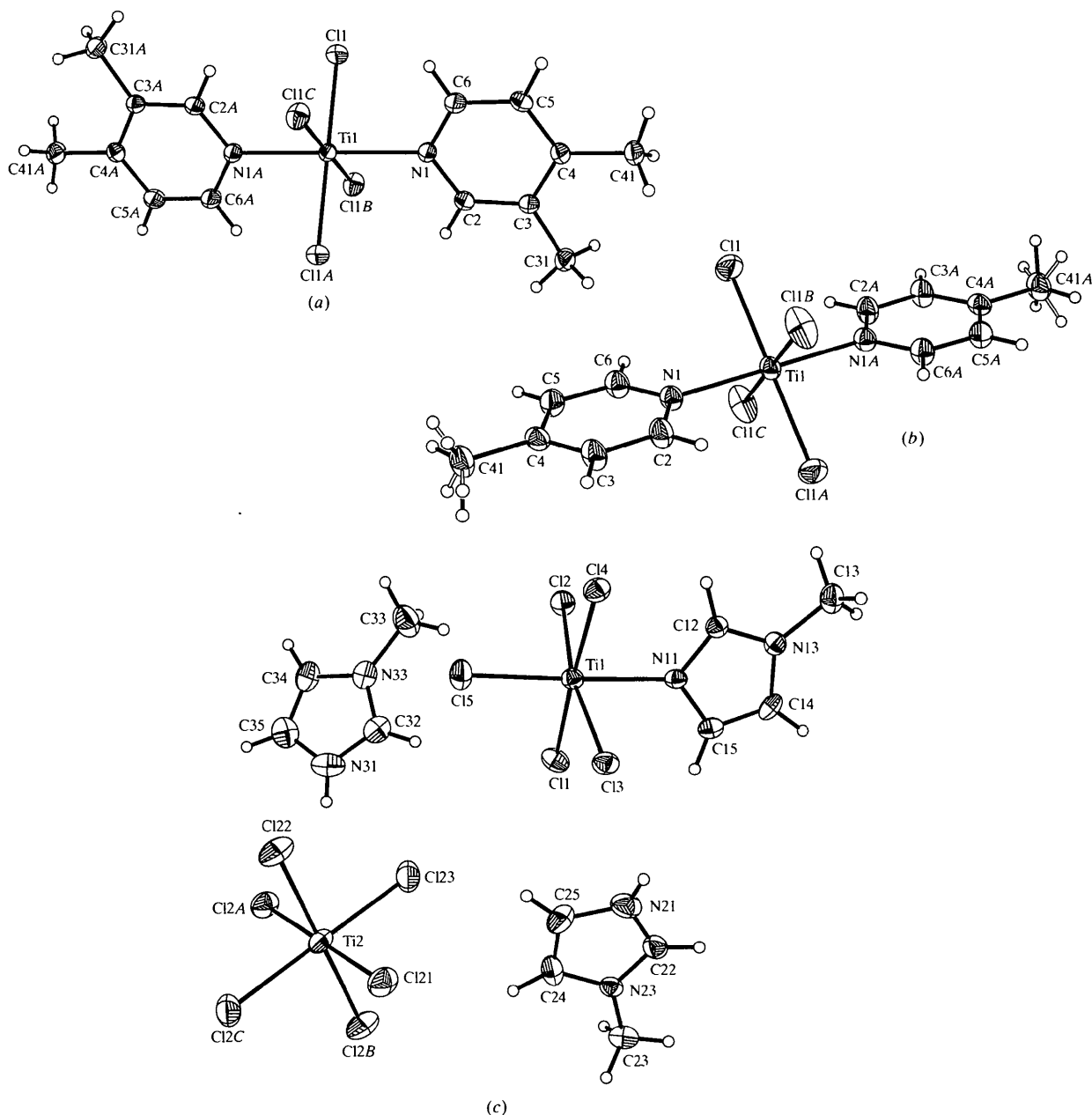


Fig. 1. Perspective view of (a) (I), (b) (II) and (c) (III) with the atomic numbering; displacement ellipsoids are at the 50% probability level.

The Ti atoms in the  $[\text{TiCl}_5(\text{NMI})]^-$  and  $[\text{TiCl}_6]^{2-}$  anions of (III) are also almost perfectly octahedrally coordinated, with two *N*-methylimidazolium cations as counter-ions. Compared with (I), where the aromatic ring almost perfectly bisects the equatorial Cl—Ti—Cl angle, the NMI ring in  $[\text{TiCl}_5(\text{NMI})]^-$  is in an asymmetric position between the equatorial Ti—Cl bonds. The Ti atom deviates by 0.1314(9) Å from the plane of the equatorial Cl atoms towards the axial Cl atom, so that all  $\text{Cl}_{\text{ax}}\text{—Ti—Cl}_{\text{eq}}$  angles are wider and all  $\text{N—Ti—Cl}_{\text{eq}}$  angles are smaller than

90°, except for  $\text{N11—Ti—Cl1}$  which is 90.29(9)°. One of the equatorial Ti—Cl bonds is slightly elongated, which is caused by two hydrogen bonds:  $\text{H21}\cdots\text{Cl3}$  2.684 Å and  $\text{N21—H21}\cdots\text{Cl3}$  135.9°, and  $\text{H21}\cdots\text{Cl3}^i$  2.841 Å and  $\text{N21—H21}\cdots\text{Cl3}^i$  123.5° [symmetry code: (i)  $-x+1, -y+1, -z$ ]. The other NMI solvent molecule forms two hydrogen bonds to the  $[\text{TiCl}_6]^{2-}$  ion:  $\text{H31}\cdots\text{Cl23}$  2.637 Å and  $\text{N31—H31}\cdots\text{Cl23}$  128.0°, and  $\text{H31}\cdots\text{Cl21}^{ii}$  2.495 Å and  $\text{N31—H31}\cdots\text{Cl21}^{ii}$  152.2° [symmetry code: (ii)  $-x+1, -y+2, -z+1$ ]. Fig. 2 shows the resulting hydrogen-bond pattern.

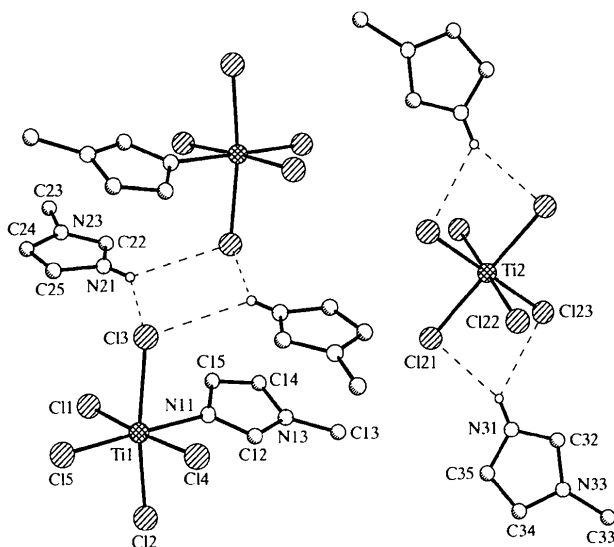


Fig. 2. Perspective view of (III) showing the hydrogen-bond pattern; only symmetry-independent atoms are labelled.

Our original aim was to determine the structure of the compound which is formed by titanium tetrachloride and NMI in a 1:2 ratio. However, even though all manipulations were carried out in a dry nitrogen environment, the reactants must have been affected by atmospheric humidity and the associated hydrolysis of  $\text{TiCl}_4(\text{NMI})_2$  must be the cause of the formation of (III).

The only structures, also retrieved from the Cambridge Structural Database, which are comparable to  $[\text{TiCl}_5(\text{NMI})]^-$  are the  $[\text{TiCl}_5(\text{thf})]^-$  anion (Sobota *et al.*, 1984) and the  $[\text{TiCl}_5(4\text{-mepy})]^-$  anion (Hensen, Pickel *et al.*, 1997). In both of these structures, the axial ring bisects the equatorial  $\text{Cl}-\text{Ti}-\text{Cl}$  angles and the Ti atoms are displaced from the equatorial plane towards the axial Cl atom. In  $\text{TiCl}_5(\text{thf})$ , all equatorial  $\text{Ti}-\text{Cl}$  bonds are in the same range, 2.296–2.318 Å, whereas the axial  $\text{Ti}-\text{Cl}$  bond (2.249 Å) is significantly shorter. In  $\text{TiCl}_5(4\text{-mepy})$ , two of the equatorial  $\text{Ti}-\text{Cl}$  bonds are significantly longer than the others, which are in the same range as the axial  $\text{Ti}-\text{Cl}$  bond, an effect which is also caused by  $\text{Cl}\cdots\text{H}$  contacts.

## Experimental

All manipulations were carried out in a dry nitrogen atmosphere in a standard glove box or using standard Schlenk techniques. 4-Methylpyridine, 3,4-dimethylpyridine and *N*-methylimidazole were distilled from  $\text{CaH}_2$  prior to use. Chloroform was dried through a molecular sieve (0.4 Å).  $\text{CDCl}_3$  was purchased from Deutero and dried through a molecular sieve (0.4 Å). NMR chemical shifts are reported in p.p.m. versus TMS. C, H and N analysis was obtained using a Heraeus CHN-Rapid. The  $^1\text{H}$  NMR spectra were obtained using an AM 250 MHz NMR spectrometer (Bruker). For the synthesis of  $[\text{TiCl}_4(\text{C}_7\text{H}_9\text{N})_2]$ , titanium tetrachloride (5.7 mM, 0.62 ml) was dissolved in 25 ml chloroform and 3,4-dimethyl-

pyridine (11.43 mM, 1.28 ml) was added dropwise to the stirred solution, whereupon a yellow solid started to precipitate. The suspension was filtered under nitrogen, washed with chloroform and dried *in vacuo*. Subsequently, the yellow solid was transferred to a dried flask, evacuated ( $10^{-6}$  mbar) and heated to 443 K (decomposition occurred above 443 K). After 3 d, single crystals were obtained. Analysis measured (calculated) in %: C 40.85 (41.62), H 4.49 (4.46), N 6.70 (6.93), Cl 34.54 (35.10).  $^1\text{H}$  NMR:  $\delta$  = 2.50 (*d*, 12H,  $\text{CH}_3$ ), 7.60 (*s*, 2H, H5), 8.50 (*s*, 4H, H2,6).  $[\text{TiCl}_4(\text{C}_6\text{H}_7\text{N})_2]$  was synthesized as above using titanium tetrachloride (4.97 mM, 0.54 ml) and 4-methylpyridine (10 mM, 0.97 ml). A yellow solid resulted which was evacuated ( $10^{-6}$  mbar) and heated at 403 K (decomposition occurred above 428 K). After 3 d, single crystals were obtained. Analysis measured (calculated) in %: C 37.99 (38.30), H 3.85 (3.72), N 7.29 (7.45), Cl 37.76 (37.72).  $^1\text{H}$  NMR:  $\delta$  = 2.70 (*s*, 6H,  $\text{CH}_3$ ), 7.75 (*d*, 4H, H3,5), 8.65 (*d*, 4H, H2,6).  $(\text{C}_4\text{H}_7\text{N}_2)_2[\text{TiCl}_5(\text{C}_4\text{H}_6\text{N}_2)][\text{TiCl}_6]$  was synthesized as for  $[\text{TiCl}_4(\text{C}_7\text{H}_9\text{N})_2]$  using  $\text{TiCl}_4$  (5.57 mM, 0.61 ml) and *N*-methylimidazole (11.14 mM, 0.88 ml) yielding an orange solid. Single crystals were obtained at  $10^{-6}$  mbar and a temperature of *ca* 443 K (decomposition occurred above 448 K).

## Compound (I)

### Crystal data

$[\text{TiCl}_4(\text{C}_7\text{H}_9\text{N})_2]$

$M_r$  = 404.00

Orthorhombic

*Cmca*

$a$  = 7.3215 (1) Å

$b$  = 17.3709 (2) Å

$c$  = 13.3435 (2) Å

$V$  = 1697.04 (4) Å<sup>3</sup>

$Z$  = 4

$D_x$  = 1.581 Mg m<sup>-3</sup>

$D_m$  not measured

Mo  $K\alpha$  radiation

$\lambda$  = 0.71073 Å

Cell parameters from 7292

reflections

$\theta$  = 1–25°

$\mu$  = 1.127 mm<sup>-1</sup>

$T$  = 173 K

Block

0.35 × 0.20 × 0.20 mm

Yellow

### Data collection

Siemens CCD three-circle diffractometer

$\omega$  scans

Absorption correction:

empirical (SADABS;

Sheldrick, 1996)

$T_{\min}$  = 0.694,  $T_{\max}$  = 0.806

11 422 measured reflections

940 independent reflections

843 reflections with

$I > 2\sigma(I)$

$R_{\text{int}}$  = 0.038

$\theta_{\max}$  = 26.39°

$h$  = -9 → 8

$k$  = -21 → 20

$l$  = -16 → 16

170 standard reflections

frequency: 600 min

intensity decay: none

### Refinement

Refinement on  $F^2$

$R[F^2 > 2\sigma(F^2)]$  = 0.029

$wR(F^2)$  = 0.068

$S$  = 1.108

940 reflections

62 parameters

H atoms treated by a

mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0285P)^2 + 3.0256P]$

where  $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.280 \text{ e \AA}^{-3}$

$\Delta\rho_{\min} = -0.286 \text{ e \AA}^{-3}$

Extinction correction: none

Scattering factors from

*International Tables for Crystallography* (Vol. C)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters ( $\text{\AA}^2$ ) for (I)
$$U_{\text{eq}} = (1/3)\sum_i \sum_j U^{ij} a'_i a'_j$$

	x	y	z	$U_{\text{eq}}$
Ti1	1/2	1/2	0	0.01721 (18)
Cl1	0.27823 (7)	0.42275 (3)	0.06869 (3)	0.02623 (16)
N1	1/2	0.57009 (12)	0.13672 (16)	0.0190 (5)
C2	1/2	0.64805 (15)	0.13156 (19)	0.0193 (5)
C3	1/2	0.69581 (15)	0.21512 (19)	0.0185 (5)
C31	1/2	0.78211 (15)	0.2007 (2)	0.0268 (6)
C4	1/2	0.66169 (15)	0.31075 (19)	0.0191 (5)
C41	1/2	0.70950 (17)	0.4048 (2)	0.0267 (6)
C5	1/2	0.58158 (16)	0.3157 (2)	0.0236 (6)
C6	1/2	0.53797 (16)	0.22916 (19)	0.0226 (6)

Table 3. Fractional atomic coordinates and equivalent isotropic displacement parameters ( $\text{\AA}^2$ ) for (II)
$$U_{\text{eq}} = (1/3)\sum_i \sum_j U^{ij} a'_i a'_j$$

	x	y	z	$U_{\text{eq}}$
Ti1	0	1/2	1/2	0.0214 (3)
Cl1	0.22593 (17)	0.33137 (14)	0.43608 (7)	0.0443 (4)
N1	0	0.6505 (5)	0.3707 (3)	0.0232 (10)
C2	0	0.8186 (7)	0.3744 (4)	0.0294 (12)
C3	0	0.9169 (7)	0.2939 (4)	0.0291 (13)
C4	0	0.8441 (7)	0.2048 (4)	0.0251 (12)
C41	0	0.9468 (7)	0.1151 (4)	0.0334 (13)
C5	0	0.6714 (7)	0.2012 (4)	0.0292 (12)
C6	0	0.5805 (7)	0.2842 (4)	0.0280 (12)

Table 2. Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ ) for (I)

Ti1—N1	2.193 (2)	Ti1—Cl1	2.2971 (5)
N1—Ti1—Cl1	89.56 (4)		
Cl1—Ti1—N1—C6	44.979 (13)		

Table 4. Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ ) for (II)

Ti1—N1	2.191 (4)	Ti1—Cl1	2.2976 (10)
N1—Ti1—Cl1	89.71 (8)		
Cl1—Ti1—N1—C6	44.95 (3)		

**Compound (II)***Crystal data*[TiCl<sub>4</sub>(C<sub>6</sub>H<sub>7</sub>N)<sub>2</sub>] $M_r = 375.95$ 

Orthorhombic

*Pmna* $a = 7.184$  (1)  $\text{\AA}$  $b = 8.014$  (2)  $\text{\AA}$  $c = 14.151$  (3)  $\text{\AA}$  $V = 814.7$  (3)  $\text{\AA}^3$  $Z = 2$  $D_x = 1.533$  Mg m<sup>-3</sup> $D_m$  not measuredMo  $K\alpha$  radiation $\lambda = 0.71073$   $\text{\AA}$ 

Cell parameters from 5612

reflections

 $\theta = 1-25^\circ$  $\mu = 1.168$  mm<sup>-1</sup> $T = 173$  K

Block

0.40 × 0.20 × 0.20 mm

Yellow

*Data collection*

Siemens CCD three-circle diffractometer

 $\omega$  scans

Absorption correction:

empirical (SADABS; Sheldrick, 1996)

 $T_{\text{min}} = 0.652$ ,  $T_{\text{max}} = 0.800$ 

10 329 measured reflections

892 independent reflections

780 reflections with

 $I > 2\sigma(I)$  $R_{\text{int}} = 0.051$  $\theta_{\text{max}} = 26.27^\circ$  $h = -8 \rightarrow 8$  $k = -9 \rightarrow 9$  $l = -17 \rightarrow 17$ 

141 standard reflections

frequency: 1080 min

intensity decay: none

*Refinement*Refinement on  $F^2$  $R[F^2 > 2\sigma(F^2)] = 0.047$  $wR(F^2) = 0.109$  $S = 1.310$ 

892 reflections

56 parameters

H atoms treated by a

mixture of independent and constrained refinement

 $w = 1/[\sigma^2(F_o^2) + (0.0118P)^2 + 2.7576P]$ where  $P = (F_o^2 + 2F_c^2)/3$  $(\Delta/\sigma)_{\text{max}} = 0.001$  $\Delta\rho_{\text{max}} = 0.280$  e  $\text{\AA}^{-3}$  $\Delta\rho_{\text{min}} = -0.627$  e  $\text{\AA}^{-3}$ 

Extinction correction: none

Scattering factors from

*International Tables for Crystallography* (Vol. C)**Compound (III)***Crystal data*(C<sub>4</sub>H<sub>7</sub>N<sub>2</sub>)<sub>2</sub>[TiCl<sub>5</sub>(C<sub>4</sub>H<sub>6</sub>N<sub>2</sub>)]-[TiCl<sub>6</sub>]<sub>0.5</sub> $M_r = 603.79$ 

Triclinic

*P1* $a = 7.8998$  (2)  $\text{\AA}$  $b = 9.9722$  (3)  $\text{\AA}$  $c = 16.0448$  (5)  $\text{\AA}$  $\alpha = 107.540$  (1) $^\circ$  $\beta = 93.784$  (1) $^\circ$  $\gamma = 99.551$  (1) $^\circ$  $V = 1179.43$  (6)  $\text{\AA}^3$  $Z = 2$  $D_x = 1.700$  Mg m<sup>-3</sup> $D_m$  not measuredMo  $K\alpha$  radiation $\lambda = 0.71073$   $\text{\AA}$ 

Cell parameters from 6449

reflections

 $\theta = 1-25^\circ$  $\mu = 1.438$  mm<sup>-1</sup> $T = 173$  K

Block

0.20 × 0.20 × 0.10 mm

Yellow

*Data collection*

Siemens CCD three-circle diffractometer

 $\omega$  scans

Absorption correction:

empirical (SADABS;

Sheldrick, 1996)

 $T_{\text{min}} = 0.762$ ,  $T_{\text{max}} = 0.87$ 

12 610 measured reflections

4155 independent reflections

2938 reflections with

 $I > 2\sigma(I)$  $R_{\text{int}} = 0.055$  $\theta_{\text{max}} = 26.16^\circ$  $h = -9 \rightarrow 9$  $k = -12 \rightarrow 11$  $l = -19 \rightarrow 19$ 

162 standard reflections

frequency: 1000 min

intensity decay: none

*Refinement*Refinement on  $F^2$  $R[F^2 > 2\sigma(F^2)] = 0.046$  $wR(F^2) = 0.093$  $S = 1.039$ 

4155 reflections

253 parameters

H atoms treated by a

mixture of independent and constrained refinement

 $w = 1/[\sigma^2(F_o^2) + (0.0291P)^2 + 1.2599P]$ where  $P = (F_o^2 + 2F_c^2)/3$  $(\Delta/\sigma)_{\text{max}} < 0.001$  $\Delta\rho_{\text{max}} = 0.390$  e  $\text{\AA}^{-3}$  $\Delta\rho_{\text{min}} = -0.578$  e  $\text{\AA}^{-3}$ 

Extinction correction: none

Scattering factors from

*International Tables for Crystallography* (Vol. C)

Table 5. Fractional atomic coordinates and equivalent isotropic displacement parameters ( $\text{\AA}^2$ ) for (III)
$$U_{eq} = (1/3) \sum_i \sum_j U^{ij} a^i a^j \mathbf{a}_i \cdot \mathbf{a}_j$$

	x	y	z	$U_{eq}$
Ti1	0.20585 (9)	0.32310 (8)	0.13327 (5)	0.01949 (18)
Cl1	0.46430 (14)	0.39575 (11)	0.22445 (7)	0.0299 (3)
Cl2	0.15040 (14)	0.11212 (11)	0.16810 (7)	0.0264 (3)
Cl3	0.29291 (13)	0.51583 (10)	0.07747 (6)	0.0246 (2)
Cl4	-0.04572 (13)	0.25393 (11)	0.03555 (6)	0.0251 (2)
Cl5	0.06205 (14)	0.44744 (11)	0.24240 (7)	0.0300 (3)
N11	0.3307 (4)	0.2026 (3)	0.0272 (2)	0.0196 (8)
Cl2	0.2522 (5)	0.0907 (4)	-0.0405 (3)	0.0213 (9)
N13	0.3665 (4)	0.0386 (3)	-0.0942 (2)	0.0216 (8)
Cl3	0.3261 (6)	-0.0848 (5)	-0.1748 (3)	0.0315 (11)
Cl4	0.5265 (5)	0.1218 (5)	-0.0591 (3)	0.0266 (10)
Cl5	0.5043 (5)	0.2222 (4)	0.0153 (3)	0.0244 (10)
N21	0.7180 (5)	0.6515 (4)	0.1425 (2)	0.0345 (9)
C22	0.8874 (6)	0.6663 (4)	0.1409 (3)	0.0282 (10)
N23	0.9651 (4)	0.7598 (3)	0.2165 (2)	0.0219 (8)
C23	1.1510 (5)	0.8059 (5)	0.2407 (3)	0.0326 (11)
C24	0.8412 (6)	0.8036 (5)	0.2688 (3)	0.0332 (11)
C25	0.6864 (6)	0.7357 (5)	0.2223 (3)	0.0369 (12)
N31	0.3057 (5)	0.5901 (4)	0.5267 (3)	0.0396 (10)
C32	0.2710 (6)	0.4826 (5)	0.4514 (3)	0.0347 (11)
N33	0.1718 (5)	0.3729 (4)	0.4654 (2)	0.0299 (9)
C33	0.1040 (7)	0.2320 (5)	0.3993 (3)	0.0437 (13)
C34	0.1432 (6)	0.4129 (5)	0.5521 (3)	0.0343 (12)
C35	0.2276 (6)	0.5487 (5)	0.5901 (3)	0.0375 (12)
Ti2	1/2	1	1/2	0.0268 (3)
Cl21	0.49886 (15)	1.07407 (13)	0.37399 (7)	0.0359 (3)
Cl22	0.19956 (14)	0.93235 (14)	0.47824 (8)	0.0412 (3)
Cl23	0.52107 (17)	0.76923 (13)	0.41544 (8)	0.0422 (3)

Table 6. Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ ) for (III)

Ti1—N11	2.167 (3)	Ti1—Cl3	2.3767 (12)
Ti1—Cl1	2.3043 (13)	Ti2—Cl22	2.3297 (11)
Ti1—Cl5	2.3055 (12)	Ti2—Cl23	2.3323 (11)
Ti1—Cl4	2.3164 (12)	Ti2—Cl21	2.3523 (10)
Ti1—Cl2	2.3164 (12)		
N11—Ti1—Cl1	90.26 (9)	Cl5—Ti1—Cl2	94.78 (4)
N11—Ti1—Cl5	177.59 (10)	Cl4—Ti1—Cl2	91.59 (4)
Cl1—Ti1—Cl5	92.14 (5)	N11—Ti1—Cl3	84.28 (9)
N11—Ti1—Cl4	87.59 (9)	Cl1—Ti1—Cl3	88.38 (4)
Cl1—Ti1—Cl4	176.73 (5)	Cl5—Ti1—Cl3	95.98 (4)
Cl5—Ti1—Cl4	90.02 (4)	Cl4—Ti1—Cl3	88.96 (4)
N11—Ti1—Cl2	84.99 (9)	Cl2—Ti1—Cl3	169.22 (5)
Cl1—Ti1—Cl2	90.68 (4)		
Cl1—Ti1—N11—Cl2	155.7 (3)	Cl2—Ti1—N11—Cl2	65.1 (3)
Cl4—Ti1—N11—Cl2	-26.7 (3)	Cl3—Ti1—N11—Cl2	-115.9 (3)

The data collection nominally covered a sphere of reciprocal space by a combination of seven, for (I) and (II), and six, for (III), sets of exposures; each set had a different  $\varphi$  angle for the crystal and each exposure covered  $0.3^\circ$  in  $\omega$ . The crystal-to-detector distance was 6.0 cm. Coverage of the unique set was complete to at least  $26.4^\circ$  in  $\theta$ , for (I) and (II), and 90% complete to at least  $24.7^\circ$  in  $\theta$ , for (III). Crystal decay was monitored by repeating the initial frames at the end of data collection and analysing the duplicate reflections. All H atoms were located by difference Fourier synthesis and the H atoms bonded to carbon were refined with fixed individual displacement parameters [ $U(\text{H}) = 1.2U_{eq}(\text{C})$ ,  $U(\text{H}) = 1.2U_{eq}(\text{C})$  or  $U(\text{H}) = 1.2U_{eq}(\text{N})$ ] using a riding model with C—H(aromatic) = 0.95, C—H(methyl) = 0.98 or N—H = 0.88  $\text{\AA}$ , respectively. The methyl group of (II) is disordered; two different positions rotated from each other by  $60^\circ$  were found. These two conformations were refined with the same site-occupation factor. The methyl groups of the imidazole rings of (III) were allowed to rotate about their local threefold axis.

For all compounds, data collection: *SMART* (Siemens, 1995); cell refinement: *SMART*; data reduction: *SAINT* (Siemens, 1995); program(s) used to solve structures: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structures: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: SK1252). Services for accessing these data are described at the back of the journal.

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## catena-Poly[[bis( $\mu$ -2,6-dimethylpiperidino-*N:N*)dilithium]- $\mu$ -(*N,N,N',N'*-tetramethylethylenediamine-*N:N'*)]

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## Abstract

The title compound,  $[\text{Li}_2(\text{C}_7\text{H}_{14}\text{N})_2(\text{C}_6\text{H}_{16}\text{N}_2)]_n$ , is a chain polymer in which strictly planar  $\text{Li}_2\text{N}_2$  rings incorporating 2,6-dimethylpiperidino ligands are linked by