

Crystal and Molecular Structure of Trichlorobis-(*NN'*-di-isopropylacetamidinato)tantalum(v)

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Crystals of the title compound are monoclinic, space group $P2_1/a$ with $a = 17.420(8)$, $b = 11.924(8)$, $c = 11.027(8)$ Å, $\beta = 95.88(8)^\circ$, $Z = 4$. The structure was solved by Patterson and Fourier methods from diffractometer data and refined by least-squares techniques to R 0.064 for 2003 independent reflections. The seven-coordinate monomer is a highly distorted pentagonal bipyramid with two chlorine atoms in axial positions [2.386(6), 2.370(6) Å], one chlorine [2.436(6)] and four nitrogens [2.098(14), 2.193(16), 2.180(20), and 2.058(16) Å] occupying the pentagonal girdle. There are many close contacts involving the isopropyl groups which cause considerable distortion to the polyhedron. The maximum deviation of a contributing atom from the least-squares plane of the $TaClN_4$ unit is 0.33 Å.

As part of a general study of seven-co-ordination,¹ we have been investigating the steric effects which distort the girdle of the D_{5h} pentagonal bipyramid (=PB). Previously, we reported the crystal structure² of $NbCl_3[NMeC(S)Me]_2$ which is a PB; in the co-ordination sphere, the two chlorine atoms occupy axial positions and the remaining five atoms in the girdle follow the sequence: Cl, N, S, S, N. This structure showed considerable distortions from ideal symmetry due to close contacts between the equatorial chlorine atom and adjacent methyl groups. We then prepared several compounds of general formula $Me_nMCl_{3-n}L_2$, [$M = Nb$ or Ta ; $n = 0$ or 1 ; $L =$ bidentate ligand] in which steric effects were potentially far greater than in $NbCl_3[NMeC(S)Me]_2$. There are several possible structures for this group of compounds the most likely being: (a) six-co-ordinate, with one of the potentially bidentate ligands unidentate, (b) seven-co-ordinate highly distorted PB, and (c) seven-co-ordinate with some other geometry. We report here the crystal and molecular structure of one of these compounds, trichlorobis-(*NN'*-di-isopropylacetamidinato)tantalum-(v), $TaCl_3[C_3H_7NC(Me)NC_3H_7]_2$, (I). The complex (I) was prepared during a study³ of the reactions between Me_nMCl_{5-n} ($M = Nb$ or Ta ; $n = 1-3$) and disubstituted carbodi-imides.

EXPERIMENTAL

Crystals of (I), prepared from Me_3TaCl_3 and *NN'*-di-isopropylcarbodi-imide,³ were obtained by slowly extracting the powdered material with pentane.

Crystal Data.— $C_{16}H_{34}Cl_3N_4Ta$, $M = 569.68$, Monoclinic, $a = 17.420(8)$, $b = 11.924(8)$, $c = 11.027(8)$ Å, $\beta = 95.88(8)^\circ$, $U = 2278.7$ Å³, $D_m = 1.63(2)$, $Z = 4$, $D_c = 1.66$, $F(000) = 1128$. Mo- $K\alpha$ radiation, $\lambda = 0.7107$ Å; $\mu(Mo-K\alpha) = 52.32$ cm⁻¹. Space group $P2_1/a$, from systematic absences: $h0l$, $h = 2n + 1$, and $0k0$, $k = 2n + 1$.

A crystal with dimensions *ca.* $0.1 \times 0.375 \times 0.23$ mm was mounted with the (011) planes perpendicular to the instrument axis of a General Electric XRD 5 apparatus which was used to measure diffraction intensities and cell dimensions. It was equipped with a manual goniostat, scintillation counter, and pulse-height discriminator. Zirconium-filtered molybdenum X -radiation was used. The stationary-crystal-stationary-counter method was used

¹ M. G. B. Drew and J. D. Wilkins, *J.C.S. Dalton*, 1973, 1830, and references therein.

² M. G. B. Drew and J. D. Wilkins, *J.C.S. Dalton*, 1974, 198.

³ J. D. Wilkins, *J. Organometallic Chem.*, in the press.

with a 4° take-off angle and a counting time of 10 s. Individual backgrounds were taken from plots of background as a function of 2θ . Several standard reflections, monitored during the course of the experiment, showed a regular decrease in intensity, and at the end of data collection, had *ca.* 0.7 of their former value. A linear correction factor was thus applied to all data. 3033 Independent reflections were measured with $2\theta < 45^\circ$. The standard deviation $\sigma(I)$ of the reflections was taken to be $[I + 2E + (0.03I^2)]^{1/2}$, where E is the estimated background of the reflection. 2005 Reflections with $I > \sigma(I)$ were used in subsequent calculations. An absorption correction was applied using program ABSORB;⁴ transmission factors varied between 0.48 and 0.34. No extinction correction was applied.

Structure Determination.—The positions of the tantalum atom were determined from the Patterson function. Fourier syntheses were then calculated to determine the positions of the remaining atoms. The structure was refined by full-matrix least squares. The weighting scheme, chosen to give average values of $w\Delta^2$ for groups of reflections independent of the value of F_o and $\sin \theta/\lambda$, was $\sqrt{w} = 1$ for $F_o < 140$ and $\sqrt{w} = 140/F_o$ for $F_o > 140$. Calculations were made on a CDC 7600 computer at the University of London Computer Centre⁴ and with some of our own programs on an I.C.L. 1904S at this university. Atomic scattering factors for tantalum, chlorine, nitrogen, and carbon were taken from ref. 5 together with corrections for the real and imaginary part of the anomalous dispersion for tantalum and chlorine. Hydrogen scattering factors were taken from ref. 6. The anisotropic thermal parameter was defined as $\exp -2\pi^2 \sum_i \sum_j U_{ij} h_i h_j b_i b_j$ ($i, j = 1, 2, 3$), b_i being the i th reciprocal cell dimension, and the isotropic thermal parameter as $\exp -8\pi^2 U \sin^2 \theta / \lambda^2$. The non-hydrogen atoms were refined anisotropically to R 0.064 for the 2005 significant reflections. (Refinement with the tantalum atom anisotropic and the other atoms isotropic gave R 0.074.) The four non-methyl hydrogen-atom positions were calculated assuming sp^3 carbon; these corresponded to positive regions in the difference Fourier map and were included in the structure-factor calculations, with thermal parameters equivalent to those of the atom to which they were bonded, but were not refined. Methyl hydrogen atoms were not unambiguously discernible in the difference Fourier map and

⁴ 'X-RAY' system of programs, ed. J. M. Stewart, University of Maryland, Technical Report, TR 192, June 1972.

⁵ 'International Tables for X-Ray Crystallography,' vol. III, Kynoch Press, Birmingham, 1965.

⁶ R. F. Stewart, E. R. Davidson, and W. T. Simpson, *J. Chem. Phys.*, 1965, **42**, 3175.

were not included. In the final cycle of refinement, no shift was $>0.08 \sigma$. The 1028 reflections given zero weight showed no large discrepancies. The final difference Fourier map showed no significant peaks, the maximum value found being 0.3 of the height of a carbon atom. Final positional co-ordinates and thermal parameters, together with their standard deviations, are in Tables 1 and 2. Molecular dimensions are in Table 3. Observed and calculated structure factors are listed in Supplementary Publication No. SUP 20948 (13 pp., 1 microfiche).*

TABLE 1

Atomic co-ordinates ($\times 10^4$), with estimated standard deviations in parentheses *

Atom	<i>x</i>	<i>y</i>	<i>z</i>
Ta	1623(1)	0997(1)	2540(1)
Cl(1)	1184(3)	1665(5)	0549(5)
Cl(2)	2118(4)	0468(6)	4540(6)
Cl(3)	2631(4)	2417(5)	2590(6)
N(1A)	0442(8)	0877(16)	2757(17)
N(2A)	1088(8)	2404(12)	3416(16)
N(1B)	1513(8)	-0709(13)	2304(12)
N(2B)	2454(11)	0083(17)	1569(18)
C(1A)	0390(10)	1875(20)	3340(18)
C(2A)	-0339(13)	2335(24)	3854(24)
C(1B)	2124(11)	-0901(22)	1664(18)
C(2B)	2426(14)	-2027(17)	1208(23)
C(11A)	-0225(11)	0322(20)	2092(20)
C(12A)	-0722(12)	1061(22)	1215(22)
C(13A)	-0749(14)	-0299(23)	2957(25)
C(21A)	1327(12)	3372(22)	4121(21)
C(22A)	1189(16)	3253(28)	5549(24)
C(23A)	1009(18)	4400(23)	3545(28)
C(11B)	1103(12)	-1622(17)	2899(21)
C(12B)	1643(15)	-2339(20)	3789(24)
C(13B)	0607(16)	-2436(25)	1994(24)
C(21B)	3118(12)	0377(19)	0878(24)
C(22B)	2957(17)	0051(25)	-0447(25)
C(23B)	3900(15)	0027(27)	1488(29)
H(11A)	-0004	-0358	1557
H(21A)	1940	3471	4077
H(11B)	0681	-1235	3419
H(21B)	3218	1275	0941

* Hydrogen atoms not refined and given isotropic thermal parameters equivalent to those of the atoms to which they were bonded.

TABLE 2

Anisotropic thermal parameters ($\times 10^3$), with estimated standard deviations in parentheses

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Ta	29.7(4)	39.7(5)	40.8(5)	-0.6(5)	13.9(3)	-2.5(6)
Cl(1)	50(3)	64(4)	44(3)	-0.4(3)	13(3)	07(3)
Cl(2)	66(4)	71(4)	51(4)	17(3)	-03(3)	-02(3)
Cl(3)	50(3)	61(4)	88(5)	-16(3)	30(3)	-24(3)
N(1A)	12(7)	59(12)	86(13)	05(8)	12(7)	-06(11)
N(2A)	27(9)	21(8)	71(12)	06(7)	-02(8)	-09(8)
N(1B)	22(8)	47(10)	43(10)	14(7)	14(7)	22(8)
N(2B)	57(12)	63(14)	63(14)	00(11)	23(10)	19(11)
C(1A)	23(10)	72(15)	40(12)	14(11)	11(9)	-31(11)
C(2A)	46(14)	92(21)	77(18)	16(14)	22(14)	-40(16)
C(1B)	36(11)	65(15)	41(12)	-07(13)	01(9)	27(13)
C(2B)	80(17)	16(11)	82(18)	07(11)	45(14)	-16(12)
C(11A)	30(11)	61(15)	53(14)	00(11)	12(10)	10(12)
C(12A)	36(12)	59(14)	81(17)	-01(13)	00(11)	-12(15)
C(13A)	58(15)	66(17)	84(19)	-16(14)	24(14)	15(16)
C(21A)	32(11)	76(18)	55(15)	-10(12)	13(11)	-25(14)
C(22A)	81(20)	123(27)	53(16)	-15(19)	35(15)	-21(17)
C(23A)	93(22)	56(17)	87(21)	10(16)	-03(17)	12(15)
C(11B)	42(12)	27(11)	67(15)	-01(11)	26(11)	12(11)
C(12B)	77(18)	50(16)	76(18)	36(14)	-02(14)	30(14)
C(13B)	77(19)	88(22)	63(17)	01(16)	37(15)	15(16)
C(21B)	39(13)	40(13)	92(15)	01(11)	44(13)	12(13)
C(22B)	94(20)	81(20)	67(19)	-09(17)	54(17)	-10(16)
C(23B)	49(16)	96(23)	112(25)	10(16)	22(16)	-06(20)

TABLE 3

Molecular dimensions

(a) Distances (Å)			
Ta-Cl(1)	2.386(6)	Ta-Cl(2)	2.370(6)
Ta-Cl(3)	2.436(6)		
	(A)	(B)	
Ta-N(1)	2.098(14)	2.058(15)	
Ta-N(2)	2.193(16)	2.180(20)	
N(1)-C(1)	1.36(3)	1.36(3)	
N(2)-C(1)	1.36(3)	1.32(3)	
C(1)-C(2)	1.54(3)	1.55(3)	
N(1)-C(11)	1.47(3)	1.49(3)	
N(2)-C(21)	1.43(3)	1.49(3)	
C(11)-C(12)	1.51(3)	1.55(3)	
C(11)-C(13)	1.57(4)	1.58(3)	
C(21)-C(22)	1.62(4)	1.51(4)	
C(21)-C(23)	1.46(4)	1.52(3)	
	(b) Angles (°)		
Cl(1)-Ta-Cl(2)	175.3(2)	Cl(1)-Ta-Cl(3)	87.2(2)
Cl(2)-Ta-Cl(3)	88.3(2)		
	(A)	(B)	
Cl(1)-Ta-N(1)	84.4(5)	101.4(4)	
Cl(1)-Ta-N(2)	92.2(4)	83.2(5)	
Cl(2)-Ta-N(1)	98.2(2)	82.8(5)	
Cl(2)-Ta-N(2)	85.6(5)	97.3(5)	
Cl(3)-Ta-N(1)	139.2(5)	138.4(4)	
Cl(3)-Ta-N(2)	78.2(4)	81.1(4)	
N(1)-Ta-N(2)	62.4(6)	60.1(7)	
N(1)-Ta-N(2)	136.9(7)	140.7(6)	
N(1A)-Ta-N(1B)	82.4(6)	N(2A)-Ta-N(2B)	159.0(7)
Ta-N(1)-C(1)	96.2(11)	99.4(14)	
Ta-N(1)-C(11)	135.2(14)	135.2(13)	
C(1)-N(1)-C(11)	122.8(16)	122.9(17)	
Ta-N(2)-C(1)	92.0(12)	95.1(14)	
Ta-N(2)-C(21)	138.0(12)	136.3(15)	
C(1)-N(2)-C(21)	128.1(17)	128.3(20)	
N(1)-C(1)-C(2)	125.6(18)	128.9(21)	
N(2)-C(1)-C(2)	125.0(20)	125.6(19)	
N(1)-C(1)-N(2)	109.4(16)	105.4(20)	
N(1)-C(11)-C(12)	115.5(19)	113.3(17)	
N(1)-C(11)-C(13)	113.0(18)	115.2(19)	
C(12)-C(11)-C(13)	109.4(17)	108.6(18)	
N(2)-C(21)-C(22)	113.1(21)	111.0(19)	
N(2)-C(21)-C(23)	111.3(19)	114.7(21)	
C(22)-C(21)-C(23)	114.0(23)	115.3(22)	

DISCUSSION

The molecule (I) is shown in Figure 1 with the atomic numbering scheme. Both the *NN'*-di-isopropylacetamidinate ligands [labelled (A) and (B)] are bidentate, bonding to the metal atom *via* the two nitrogen atoms. The tantalum atom is thus seven-co-ordinate being bonded to three chlorine and four nitrogen atoms. The co-ordination sphere is a distorted pentagonal bipyramid with two chlorine atoms [Cl(1) and Cl(2)] in axial positions. The atoms in the girdle follow the sequence: Cl(3), N(2A), N(1A), N(1B), N(2B). In a PB, the ideal angle subtended by pairs of ligand atoms at the metal atom is 72° and while this is not unreasonable for atoms the size of the present five, the presence of the four isopropyl groups in the girdle make this ideal symmetric arrangement impossible.

Several distortions are necessary to fit these two bidentate ligands into the pentagonal girdle and these are best examined with the aid of Figure 1. The numbers listed in this Figure represent the distance of each atom (in units of 0.01 Å) from the least-squares plane of atoms Ta, N(1A), N(1B), N(2A), N(2B), Cl(3).

* See Notice to authors No. 7 in *J.C.S. Dalton*, 1973, Index issue.

The two four-membered rings are planar (Table 4). The planes are twisted by 10.7 and 15.5° out of the plane of the pentagonal girdle, in opposite directions such that N(1A) is 0.27 and N(1B) -0.33 Å from the girdle

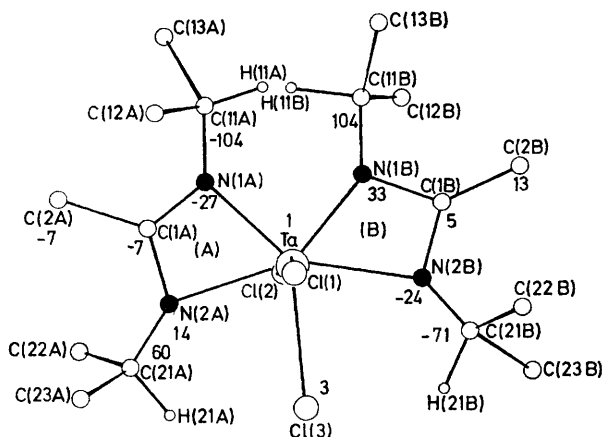


FIGURE 1 Structure of (I). Deviations of atoms ($\text{\AA} \times 10^2$) from the least-squares plane of the girdle are given

plane. These twists significantly increase the contacts between isopropyl groups on N(1A) and N(1B). The planes of the two four-membered rings intersect at

TABLE 4

Equations of least-squares planes for (I) in the form $Ax + By + Cz = D$, where x , y , and z are the crystallographic fractional co-ordinates of the atoms. Distances (\AA) of relevant atoms from each plane are given in square brackets

	A	B	C	D
Plane (1)				
Ta, Cl(3), N(1A), N(2A), N(1B), N(2B)	4.60	-3.47	9.79	2.87
[Ta 0.01, Cl(3) 0.03, N(1A) -0.27, N(2A) 0.14, N(2B) -0.24, N(1B) 0.33, C(1A) -0.07, C(2A) -0.07, C(1B) 0.05, C(2B) 0.13, C(11A) -1.04, C(21A) 0.60, C(21B) -0.71, C(11B) 1.04]				
Plane (2)				
Ta, N(1A), N(2A), C(1A)	2.46	-5.17	9.60	2.31
[Ta 0.01, N(1A) -0.01, N(2A) -0.01, C(1A) 0.02, C(2A) 0.09, C(11A) -0.53, C(21A) 0.22]				
Plane (3)				
Ta, N(1B), N(2B), C(1B)	8.48	-1.83	8.88	3.45
[Ta -0.00, N(2B) 0.01, N(1B) 0.01, C(1B) -0.01, C(2B) 0.05, C(21B) -0.10, C(11B) 0.35]				
Angles (deg.) between planes: (1)-(2) 10.7, (1)-(3) 15.5, (2)-(3) 25.4				

25.4°. The distortions are such that the molecule has C_2 rather than m symmetry, the rotation axis being coincident with the Ta-Cl(3) bond (see Figure 1).

The Cl(3) \cdots Prⁱ(2) contacts* are increased by the twist in the rings, and in addition by (a) the large Ta-N(2)-C(21) angles [138.0(12) and 136.3(15) †] and (b) the bending of the N(2)-C(21) bonds out of the

* Prⁱ(n) consists of atoms C($n1$), C($n2$), C($n3$), and H($n1$).

† Where two dimensions are given, the first pertains to ligand A and the second to ligand B.

planes of the respective rings by -0.20 and -0.11 Å. The Cl(3) \cdots C(21) distances are still short (3.18 and 3.24 Å), but not excessively so. The isopropyl groups are twisted around the N(2)-C(21) bonds such that the hydrogen atoms H(21) are directed towards Cl(3). Calculated positions for H(21), give Cl(3) \cdots H(21) to be 2.47 and 2.57 Å. However, with this orientation of the isopropyl groups, the remaining two carbon atoms [C(22) and C(23)] are pointed away from the chlorine atom and indeed the C(1)-N(2)-C(21)-C(2 n) ($n = 2$ or 3) dihedral angles are close to 60° (see Table 5).

TABLE 5
Dihedral angles (°)

	(A)	(B)
N(1)-Ta-N(1)-C(11)	-41.2	-47.4
Cl(3)-Ta-N(2)-C(21)	-25.3	-25.3
C(2)-C(1)-N(2)-C(21)	10.5	8.9
C(2)-C(1)-N(1)-C(11)	26.3	12.4
C(1)-N(1)-C(11)-C(12)	48.6	55.5
C(1)-N(1)-C(11)-C(13)	-78.3	-70.3
Ta-N(1)-C(11)-H(11)	21.8	16.1
C(1)-N(2)-C(21)-C(23)	-77.5	-77.1
C(1)-N(2)-C(21)-C(22)	52.3	55.7
Ta-N(2)-C(21)-H(21)	11.2	4.8

The Ta-N(2) bonds [2.193 (16) and 2.180(20) Å] are significantly longer than the Ta-N(1) bonds [2.098(14) and 2.058(15) Å] and can be compared to 2.231(15) and 2.185(13) in $\text{NbCl}_3[\text{MeNC}(\text{S})\text{Me}]_2$.² These values suggest that the increase to *ca.* 2.18 Å is due to the requirement of increasing the Cl \cdots C distances in the two structures. That the Ta-N(1) bonds in (I) are stronger suggests that the Prⁱ(1A) \cdots Prⁱ(1B) contacts are of lesser import in fixing the molecular configuration; this is in fact so, since they are rotated well away from each other.

In a planar pentagonal girdle the C(11A) \cdots C(11B) distance would be similar to the N(1A) \cdots N(1B) distance (2.74 Å). As it is, however, the twist in the planar rings together with the large deviation (in opposite directions) of the C(11) atoms from their respective planes (-0.53 and 0.34 Å) increase the C(11A) \cdots C(11B) distance to a most reasonable 3.33 Å. The dihedral angles N(1A)-Ta-N(1B)-C(11B) and N(1B)-Ta-N(1A)-C(11A) are -41.2 and -47.4° respectively. The two Prⁱ(1) groups are twisted such that the hydrogen atoms H(11) are in sterically crowded positions. Thus the C(1)-N(1)-C(11)-C(1 n) ($n = 2$ or 3) dihedral angles are within 21° of the staggered value of 60°. Distances <3 Å between these two Prⁱ(1) groups are: H(11A) \cdots H(11B) 2.50, H(11B) \cdots C(13A) 2.73, H(11A) \cdots C(13B) 2.72, H(11A) \cdots C(11B) 2.76, and H(11B) \cdots C(11A) 2.76 Å.

The two Ta-Cl(1) and Ta-Cl(2) axial bonds are shorter than the Ta-Cl(3) equatorial bonds by *ca.* 0.05 Å [2.386(6) and 2.370(6) *vs.* 2.436(6) Å], a normal occurrence in a pentagonal bipyramid.^{2,7,8} This is presumably a steric effect with the chlorine atoms in the less crowded

⁷ R. B. VonDreele, J. J. Stezowski, and R. C. Fay, *J. Amer. Chem. Soc.*, 1971, **93**, 2887.

⁸ M. G. B. Drew and R. Mandyczewsky, *Chem. Comm.*, 1970, 292.

sites of the PB forming the stronger bonds. This disparity in Ta-Cl bond lengths lends credence to the view (see earlier) that the Ta-N(2) bond lengths are lengthened to decrease steric strain between the methyl groups and the equatorial chlorine atom. The two Ta-Cl_{ax} bonds are not significantly distorted from axial positions, and all Cl-Ta-Cl angles are close to 90 or 180°.

The two chelate angles are 62.4(6) and 60.1(7)°.

are similar (135–138°), having values which increase intramolecular contacts involving the Prⁱ groups.

We can conclude from the structure of (I) that the tendency towards a PB with the chlorine atoms in T formation, for complexes MCl₃L₂ (L = bidentate) is very strong. The distortions from ideal symmetry found in (I) are a very logical way of minimising the non-bonded repulsions.

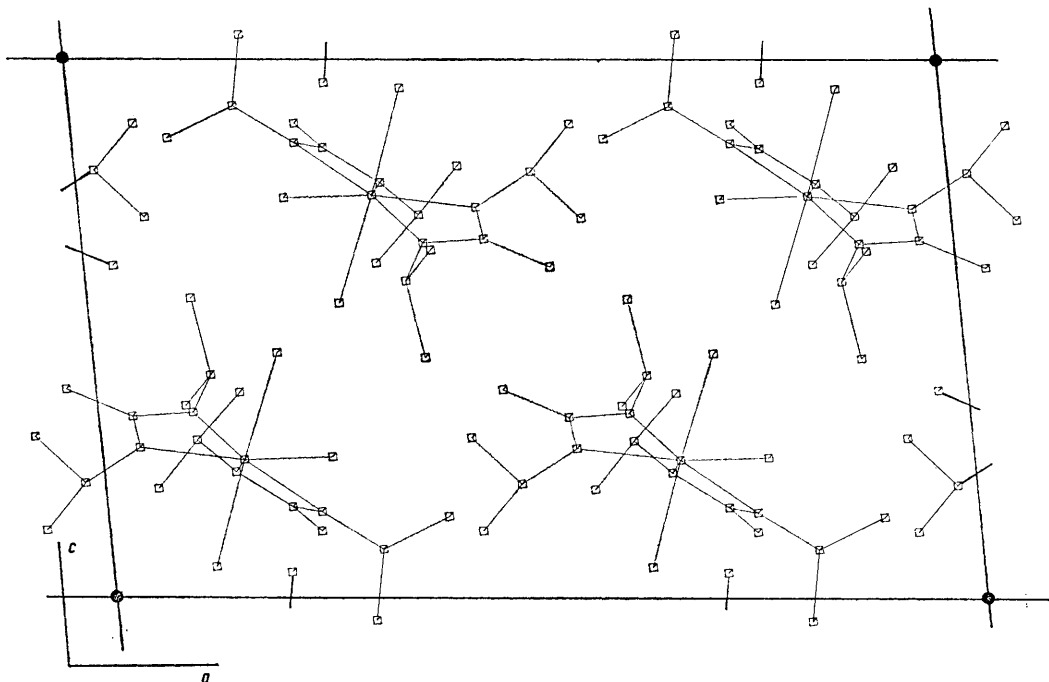


FIGURE 2 The unit cell of (I) in the *b* projection

This short bite leaves space for the other angles in the girdle to be greater than the ideal 72°. The Cl(3)-Ta-N(2) angles are 78.2(4) and 81.1(4)° and the non-chelating N(1A)-Ta-N(1B) angle is 82.4(6)°. The sum of these five angles is 364.2°.*

The N(1)-C(1) and N(2)-C(2) bond lengths are the same within experimental error and the mean value (1.35 Å) suggests a bond order between 1 and 2. The N-C(Me)-N moiety is isoelectronic with the allyl group but in (I) the bonding occurs between the metal and the terminal nitrogen atoms with no significant interaction with the C(Me) group. The angles subtended at the nitrogens N(1) and N(2) vary, presumably owing to the differing relative positions of the Prⁱ groups, but all four Ta-N(*n*)-C(*n*1) (*n* = 1 or 2) angles

* Compared to 360.8° for the less distorted PB in ref. 2. The requirement that the sum of the five angles in the girdle be close to 360° can be used to predict the viability of the PB for a molecule of particular stoichiometry. For example, TaCl₃[O(CH₂)₂·OME]₂ is a six-co-ordinate monomer with one ligand bi- and the other uni-dentate.⁹ This could have been predicted, since in a PB with two bidentate ligands and one chlorine atom in the girdle, the angles would be *ca.* O···O 70, 70 (chelating), 73 (non-chelating) and Cl···O 80, 80° with a sum well in excess of 360°.

The shortest intermolecular contacts in the unit cell are given in Table 6. There are no values appreciably

TABLE 6

Intermolecular contacts < 3.85 Å (not involving hydrogen atoms)

C(12B)···C(2A ^I)	3.62	Cl(1)···C(2 ^{IV})	3.67
C(13A)···Cl(3 ^I)	3.83	Cl(1)···C(12A ^{IV})	3.84
Cl(3)···C(12B ^{II})	3.75	C(12A)···Cl(2 ^V)	3.83
C(22A)···C(12B ^{III})	3.84	C(12A)···C(1B ^V)	3.81
Cl(2)···C(2B ^{III})	3.60		
C(22B)···C(2B ^{III})	3.63		

Roman numeral superscripts refer to atoms in the following equivalent positions, relative to the reference molecule at *x, y, z*:

I	- <i>x</i> , - <i>y</i> , 1 - <i>z</i>	IV	$\frac{1}{2} + x, \frac{1}{2} - y, z$
II	$\frac{1}{2} - x, \frac{1}{2} + y, 1 - z$	V	- <i>x</i> , - <i>y</i> , - <i>z</i>
III	$\frac{1}{2} - x, \frac{1}{2} + y, -z$		

less than the sum of van der Waals radii. Figure 2 is a packing diagram in the *b* projection.

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⁹ M. G. B. Drew and J. D. Wilkins, *Inorg. Nuclear Chem. Letters*, in the press.