for close packing of larger molecules which are in general positions.

A description in terms of two-dimensional black–white symmetry is also possible. If those molecules whose carboxylic group C(8), O(3), O(4) [or C(18), O(13), O(14)] points into the positive *b* direction are characterized as being black and those with this carboxylic group pointing into the negative *b* direction are characterized as being white, then, the two-dimensional black–white group p_c, $2_1'ca'$ results.

The diffractometer was supplied by the Deutsche Forschungsgemeinschaft. Calculations were performed at the Rechenzentrum der Universität, Hamburg. Valuable discussions with Professor Dr O. Jarchow, Hamburg, are gratefully acknowledged. The English version of the manuscript was revised by Dr M. Barton. Several numerical calculations were done by Mr P. Drissen.

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Chloro[*N*,*N*-bis(2-dimethylaminoethyl)-2-dimethylammonioethylamine]palladium(II) Chloride Hexafluorophosphate

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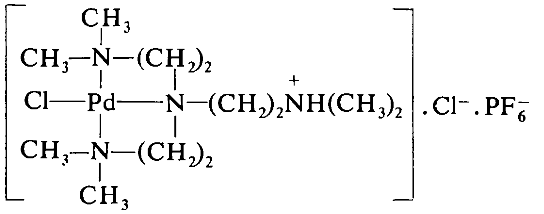
Abstract. $C_{12}H_{31}Cl_2F_6N_4PPd$, $M_r = 553{\cdot}7$, monoclinic, $a = 6{\cdot}711$ (1), $b = 30{\cdot}426$ (5), $c = 13{\cdot}478$ (2) Å, $\beta = 126{\cdot}48$ (1)°, $V = 2212{\cdot}8$ Å^3, $Z = 4$, $D_c = 1{\cdot}66$ g cm^{-3}, $F(000) = 1120$; space group $P2_1/c$ (C_{2h}^5, No. 14) from systematic absences ($h0l$ when $l = 2n + 1$, $0k0$ when $k = 2n + 1$); Mo $K\alpha$ radiation, $\lambda = 0{\cdot}71069$ Å, $\mu = 10{\cdot}9$ cm^{-1}. The structure was solved using Patterson and Fourier techniques with diffractometer data and refined by least-squares methods to a final R of 0·045 for 2111 independent observed reflections. The crystal structure contains chloride and hexafluorophosphate anions and a cation in which a Pd atom is coordinated in a square-planar environment by a Cl atom [Pd—Cl = 2·315 (2) Å] and three N atoms [Pd—N 2·039 (6) (*trans* to Cl), 2·062 (7), 2·079 (8) Å] of the protonated Me_6tren ligand. The chloride ion is hydrogen bonded to the cation (N—H···Cl 3·035 Å).

Introduction. Attempts (Senoff & Kuty, 1978) to prepare $[Pd(Me_6tren)Cl]PF_6$, where Me_6tren = tris(2-dimethylaminoethyl)amine, yielded a complex mixture of microcrystalline materials which could not be separated by crystallization techniques. Examination of the material under a microscope revealed that there were numerous larger crystals interspersed in the microcrystalline mass and a few of these were selected for X-ray examination. We report here the results of our study which establish that the material examined is the title compound (I), $[Pd(Me_6trenH)Cl]ClPF_6$.



$$\left[\begin{array}{l} \quad\ CH_3 \\ \quad\ \ | \\ CH_3-N-(CH_2)_2 \\ \qquad\ | \qquad\ | \qquad\quad\ \ + \\ Cl-Pd-N-(CH_2)_2NH(CH_3)_2 \\ \qquad\ | \qquad\ | \\ CH_3-N-(CH_2)_2 \\ \quad\ \ | \\ \quad\ CH_3 \end{array}\right].Cl^-.PF_6^-$$

(I)

Preliminary space-group and unit-cell information were obtained by photographic measurements. Least-squares analysis of the diffractometer coordinates of 12 reflections with θ(Mo $K\alpha$) between 10 and 15° led to precise values for the lattice parameters. Intensity data for the 3067 reflections with $\theta < 23°$ were collected on a computer-controlled Hilger & Watts Y290 four-circle diffractometer using graphite-monochromated Mo $K\alpha$ radiation and the θ/ω step-scan technique. For each reflection, 60 steps of 0·01°(θ) and 1 s duration were taken through the diffracting position and 15 s background counts were measured at each end of the scan. Standard deviations in intensity were calculated using $\sigma^2(I) = S + 4B + (0{\cdot}03S)^2$, where S is the scan count, B is the sum of the two backgrounds, $I = S - 2B$ and the term $(0{\cdot}03S)^2$ is used to down-weight the most intense reflections. 2111 independent reflections had $I \geq 3\sigma(I)$ and were retained as the observed data. Lorentz and polarization factors were applied but absorption corrections were not, on the grounds that the small value of μ and the regular needle shape of the crystal would combine to minimize uneven counting losses due to absorption.

The structure was solved [using the programs of Sheldrick (1976)] by a combination of Patterson and Fourier methods and refined by full-matrix least-squares techniques minimizing $\sum w(|F_o| - |F_c|)^2$ where $w = [\sigma(F)]^{-2}$ and $\sigma(F)$ is derived from counting statistics. All H atoms were located from a difference map, but rather than refine them independently we chose to constrain them to idealized positions (with C—H 1·08 Å) and to describe their thermal motion with a single overall isotropic temperature factor U which subsequently refined to the value of 0·069 (5) Å^2. The scattering factors of Stewart, Davidson & Simpson (1965) were used for the H atoms while the non-hydrogen atoms were refined using the scattering factors of Cromer & Mann (1968). The PF_6^- group is disordered over two sites for which refinement gave occupancy factors of 0·61 and 0·39. It was treated as a regular octahedron with a single variable P—F bond length and was allowed to vibrate anisotropically with individual anisotropic temperature factors, as were the Pd and Cl atoms.

Final values of R and R'* are 0·045 and 0·047 respectively. A final difference synthesis confirmed the completeness of the refinement, there being no chemically significant maxima. Atomic coordinates and their standard deviations are given in Table 1, while Table 2 contains bond lengths, valency angles and their standard deviations calculated using the full covariance matrix of the last refinement cycle.†

Table 1. *Final coordinates* (*fractional*, $\times 10^5$ *for* Pd, $\times 10^3$ *for* H, $\times 10^4$ *for others*)

	x	y	z		x	y	z
Pd	5932 (13)	17150 (2)	42 (6)	H(N4)	84	5	−131
Cl(1)	−1237 (5)	2173 (1)	622 (3)	H(1*A*)	−134	160	−286
Cl(2)	−2600 (5)	4681 (1)	4804 (2)	H(1*B*)	36	206	−279
P	1827 (5)	1122 (1)	5276 (2)	H(2*A*)	391	181	−79
F(1)	−154 (9)	1182 (2)	3858 (4)	H(2*B*)	291	138	−187
F(2)	3800 (10)	1066 (2)	6690 (4)	H(3*A*)	304	66	−147
F(3)	3098 (21)	767 (4)	5007 (7)	H(3*B*)	25	80	−289
F(4)	3472 (20)	1477 (4)	5259 (8)	H(4*A*)	20	71	−70
F(5)	570 (21)	1485 (4)	5527 (7)	H(4*B*)	−157	106	−198
F(6)	183 (22)	770 (4)	5277 (8)	H(5*A*)	556	91	235
F(3′)	3897 (16)	1096 (11)	5102 (9)	H(5*B*)	261	72	110
F(4′)	2122 (48)	1625 (2)	5422 (10)	H(6*A*)	507	85	37
F(5′)	−249 (16)	1141 (11)	5456 (9)	H(6*B*)	580	138	97
F(6′)	1514 (49)	617 (2)	5129 (10)	H(7*A*)	−383	235	−188
N(1)	2656 (13)	1376 (2)	1651 (7)	H(7*B*)	−428	186	−269
N(2)	2043 (12)	1295 (2)	−593 (6)	H(7*C*)	−373	236	−316
N(3)	−662 (14)	2080 (3)	−1571 (8)	H(8*A*)	269	244	−53
N(4)	149 (13)	192 (2)	−2196 (7)	H(8*B*)	20	269	−63
C(1)	107 (19)	1830 (4)	−2256 (10)	H(8*C*)	29	270	−191
C(2)	2435 (17)	1580 (3)	−1374 (9)	H(9*A*)	52	−40	−289
C(3)	1044 (16)	656 (3)	−2002 (9)	H(9*B*)	313	−6	−216
C(4)	252 (16)	926 (3)	−1321 (8)	H(9*C*)	45	8	−364
C(5)	3857 (18)	1000 (3)	1471 (9)	H(10*A*)	−321	34	−248
C(6)	4406 (17)	1128 (3)	573 (9)	H(10*B*)	−311	−19	−300
C(7)	−3292 (21)	2168 (4)	−2373 (11)	H(10*C*)	−339	28	−384
C(8)	724 (20)	2506 (4)	−1128 (11)	H(11*A*)	37	146	223
C(9)	1129 (19)	−67 (3)	−2755 (10)	H(11*B*)	248	102	295
C(10)	−2572 (18)	153 (3)	−2926 (10)	H(11*C*)	−16	97	141
C(11)	1244 (19)	1196 (4)	2093 (10)	H(12*A*)	371	195	272
C(12)	4586 (18)	1685 (4)	2594 (10)	H(12*B*)	564	181	228
				H(12*C*)	581	151	346

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* $R' = [\sum w\Delta^2/\sum wF_o^2]^{1/2}$.

† Lists of structure factors and anisotropic thermal parameters, and an analysis of variance have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33652 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

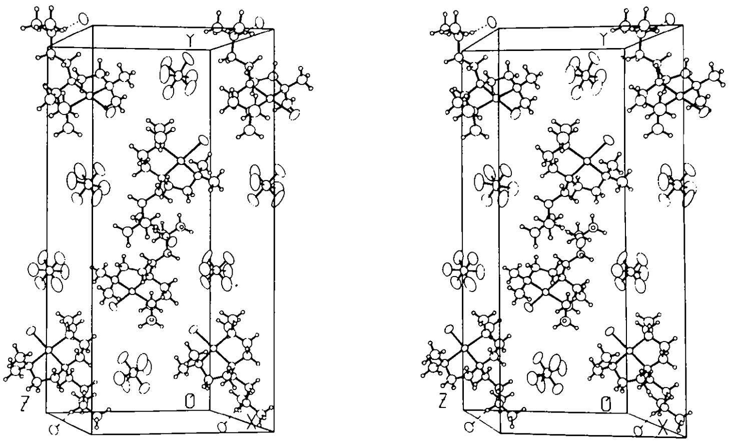

Fig. 1. A stereoscopic view of the crystal packing projected onto the *bc* plane. The N—H···Cl hydrogen bonds are indicated by dashed lines.

Table 2. *Interatomic distances* (Å) *and angles* (°)

Values in parentheses are the standard deviations in the last decimal place and are calculated using the covariance matrix of the last refinement cycle.

Pd—Cl(1)	2·315 (2)	Pd—N(2)	2·039 (6)
Pd—N(1)	2·062 (7)	Pd—N(3)	2·079 (8)
N(1)—C(5)	1·50 (1)	N(3)—C(8)	1·50 (1)
N(1)—C(11)	1·49 (1)	N(4)—C(3)	1·50 (1)
N(1)—C(12)	1·49 (1)	N(4)—C(9)	1·49 (1)
N(2)—C(2)	1·50 (1)	N(4)—C(10)	1·48 (1)
N(2)—C(4)	1·51 (1)		
N(2)—C(6)	1·51 (1)	C(1)—C(2)	1·49 (1)
N(3)—C(1)	1·50 (1)	C(3)—C(4)	1·54 (1)
N(3)—C(7)	1·44 (1)	C(5)—C(6)	1·51 (1)
Cl(1)—Pd—N(1)	94·2 (2)	Pd—N(3)—C(1)	106·2 (6)
Cl(1)—Pd—N(2)	177·2 (2)	Pd—N(3)—C(7)	115·9 (6)
Cl(1)—Pd—N(3)	93·8 (2)	Pd—N(3)—C(8)	106·1.(6)
N(1)—Pd—N(2)	86·5 (3)	C(1)—N(3)—C(7)	110·3 (8)
N(1)—Pd—N(3)	166·2 (3)	C(1)—N(3)—C(8)	108·8 (8)
N(2)—Pd—N(3)	86·0 (3)	C(7)—N(3)—C(8)	109·3 (8)
Pd—N(1)—C(5)	106·8 (5)	C(3)—N(4)—C(9)	109·6 (7)
Pd—N(1)—C(11)	115·8 (6)	C(3)—N(4)—C(10)	113·7 (7)
Pd—N(1)—C(12)	106·8 (6)	C(9)—N(4)—C(10)	111·3 (7)
C(5)—N(1)—C(11)	108·3 (7)		
C(5)—N(1)—C(12)	109·9 (7)	N(3)—C(1)—C(2)	110·4 (8)
C(11)—N(1)—C(12)	109·2 (7)	N(2)—C(2)—C(1)	108·4 (7)
		N(4)—C(3)—C(4)	111·2 (7)
Pd—N(2)—C(2)	103·9 (5)	N(2)—C(4)—C(3)	111·1 (6)
Pd—N(2)—C(4)	110·0 (4)	N(1)—C(5)—C(6)	110·0 (7)
Pd—N(2)—C(6)	104·6 (5)	N(2)—C(6)—C(5)	108·4 (7)
C(2)—N(2)—C(4)	112·2 (6)		
C(2)—N(2)—C(6)	114·2 (6)		
C(4)—N(2)—C(6)	111·3 (6)		

Discussion. The crystal structure (Fig. 1) consists of chloride and hexafluorophosphate anions in association with a cation (Fig. 2) in which a Pd atom is coordinated in a slightly distorted square-planar fashion by a Cl and by the central N and two of the dimethylamino N atoms of the tris(2-dimethylaminoethyl)amine ligand.

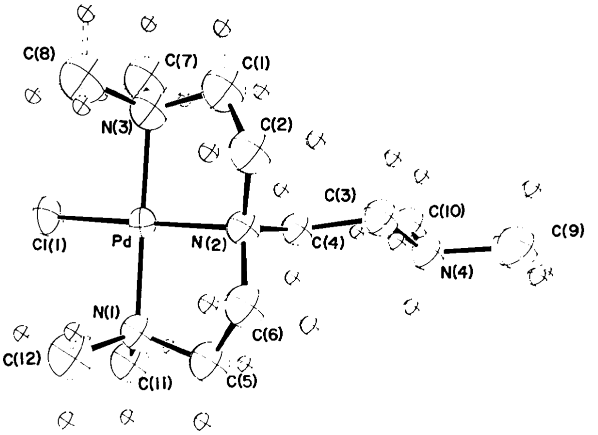

Fig. 2. The [Pd(Me_6trenH$^+$)Cl] cation, showing the numbering scheme used.

The Pd—Cl bond length [2·315 (2) Å] is virtually identical to those found in *cis*-dichloro(*meso*-2,3-diaminobutane)palladium(II) (Ito, Marumo & Saito, 1971) and *cis*-dichloroethylenediaminepalladium(II) (Iball, MacDougall & Scrimgeour, 1975) [2·317 (1) and 2·309 (3) Å respectively]. The Pd—N bond lengths are of two types and are in line with trends reported in other similar complexes (Alyea, Dias, Ferguson, McAlees, McCrindle and Roberts, 1977); those *trans* to N atoms average 2·071 (9) Å while that *trans* to the Cl is considerably shorter [2·039 (6) Å]. The N—Pd—N angles are less than 90° [mean 86·3 (3)°] whereas the Cl—Pd—N angles are slightly greater than 90° [mean 94·0 (2)°]. Pd, Cl(1), N(1), N(2) and N(3) are almost coplanar, with a r.m.s. deviation of 0·12 Å from the best least-squares plane through them. The fourth N atom of the ligand is protonated and forms an N—H···Cl hydrogen bond with the chloride ion [H···Cl 1·99, N···Cl 3·035 Å, ∠N—H···Cl 163·0°]. The F atoms of the PF_6^- moiety are disordered over two sites; this was allowed for in the refinement and the value of the P—F bond length obtained is 1·549 (4) Å, in accord with accepted values. Chemical studies on the other crystalline material mentioned in the *Introduction* are in progress (Senoff, 1978).

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μ_4-Benzylidyne-μ_3-benzylidyne-hexadecacarbonylhexaosmium

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Abstract. $Os_6(CO)_{16}[C(C_6H_5)]_2$, monoclinic, $P2_1/c$, a = 22·813 (10), b = 10·603 (5), c = 29·003 (10) Å, β = 91·55 (3)°, U = 7012·8 Å^3, Z = 8, D_c = 3·35 g cm^{-3}, μ(Mo $K\alpha$) = 217·24 cm^{-1}. The structure was refined to an R of 0·062 for 5811 unique diffractometer data. The two independent molecules are structurally equivalent; the six Os atoms, in each cluster, define a monocapped square-based pyramid, with one benzylidyne ligand capping the square face, and the other an adjacent triangular face. The carbonyl ligands are all terminal.

Introduction. $Os_6(CO)_{16}[C(C_6H_5)]_2$ was isolated as one of the products of the photolysis of $Os_6(CO)_{18}$ with phenylacetylene (Fernandez, Johnson & Lewis, 1977). Spectroscopic data showed the compound to be an organometallic cluster based on the Os_6 unit, but neither the geometry of the metal cluster nor the nature of the organic species could be established. A single-crystal X-ray analysis was therefore undertaken.

Crystals of $Os_6(CO)_{16}[C(C_6H_5)]_2$ were obtained as dark-red tablets from an ethyl acetate/hexane mixture. 8193 intensities were measured (for $3·0 < 2\theta \leq 60·0°$) on a Syntex $P2_1$ four-circle diffractometer, using graphite-monochromated Mo $K\alpha$ radiation, a θ–2θ scan procedure, and a crystal with dimensions 0·315 × 0·175 × 0·105 mm. Lp corrections and semi-empirical absorption corrections (based on a pseudo-ellipsoid model and 460 azimuthal scan data from 44 independent reflections which gave transmission coefficients ranging from 0·665 to 0·206 for the full data set) were applied. The data were averaged to give 5811 unique observed reflections [$I > 1·5\sigma(I)$]. Cell dimensions were derived from the angular measurements of 15 strong reflections ($20·0 < 2\theta < 30·0°$).

The Os atoms were located by multisolution $\sum_2$ sign expansion, and the remaining non-hydrogen atoms by difference syntheses. The structure was refined by blocked-full-matrix least squares with anisotropic Os and isotropic light atoms. In view of the relatively large uncertainties in the light-atom positions, the X-ray scattering being dominated by the Os contributions, the Os—C and C—O bond lengths in each molecule were constrained to be equal; these distances were refined [molecule 1: Os—C 1·89 (1), C—O 1·16 (1) Å; molecule 2: Os—C 1·86 (1), C—O 1·18 (1) Å]. The phenyl rings were refined as rigid groups with idealized geometry (C—C 1·395 Å, C—C—C 120·0°). Complex neutral-atom scattering factors (*International Tables for X-ray Crystallography*, 1974), and a weighting scheme of the form $w = 1·384/[\sigma^2(F) + 0·001|F_o|^2]$ were employed. The refinement converged to R = 0·062 and $R' = \sum w^{1/2}\Delta/\sum w^{1/2}|F_o| = 0·059$. The final