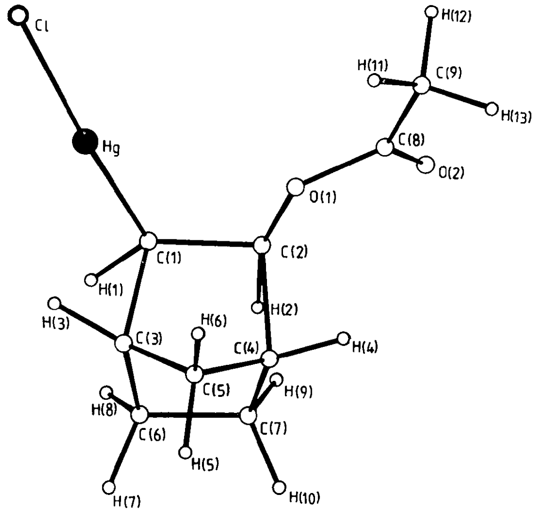

Fig. 1. A view of the $C_9H_{13}ClHgO_2$ molecule, showing the atom numbering scheme used in this analysis.

**Discussion.** An illustration of the molecule drawn using the program *ORTEP* (Johnson, 1970) is given in Fig. 1.

The Hg atom is bonded almost linearly [173 (2)°] to Cl and C(1); the bond lengths 2·32 (2), 2·10 (6) Å are within the range most frequently found for covalent Hg—Cl and Hg—C distances. Similar small deviations from linearity are typically found in two-coordinate Hg compounds. The nearest other contacts to the Hg atoms are Cl atoms of adjacent molecules (3·43 and 3·32 Å); these are in excess of the sum of the van der Waals radii, $r_{\text{Cl}}$ 1·80 Å (Pauling, 1940) and $r_{\text{Hg}}$ 1·50 Å (Grdenić, 1965), and cannot be ascribed to definite interactions. The small torsion angle for Hg—C(1)—C(2)—O(1) of 7 (5)° indicates that addition across the norbornene ethylenic bond is in the *cis* conformation, confirming the prediction of Anderson & Henry (1961) and of Traylor & Baker (1963). The bond distances and angles in the norbornane nucleus agree, within the limits of experimental error, with those in similar compounds.

We thank the Science Research Council for the provision of a Research Studentship to one of us (JH).

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*Acta Cryst.* (1978). B**34**, 3079–3083

# Cobalt Dihydrogen Diphthalate Hexahydrate

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(*Received* 8 *December* 1977; *accepted* 14 *June* 1978)

**Abstract.** $Co(C_8H_5O_4)_2.6H_2O$, monoclinic, $P2_1/c$; $a = 6{\cdot}560$ (1), $b = 30{\cdot}962$ (4), $c = 9{\cdot}918$ (1) Å, $\beta = 91{\cdot}02$ (4)°, $Z = 4$, $D_x = 1{\cdot}618$ g cm$^{-3}$. The structure has been solved by direct methods and refined to $R = 0{\cdot}033$ for 3400 independent observed X-ray reflexions. Co is coordinated to six O atoms of water molecules which form a nearly regular octahedron. These octahedra are linked by hydrogen bonds to the hydrogen

phthalate ions to form layers parallel to (010). The hydrogen phthalate ions do not build intramolecular hydrogen bonds.

**Introduction.** In a few examples of acid salts of phthalic acid (Biagini Cingi, Guastini, Musatti & Nardelli, 1969; Gonschorek & Küppers, 1975; Adiwidjaja & Küppers, 1978) very short intramolecular hydrogen bonds between the two adjacent carboxylic groups of each molecule have been detected. In the present paper a further hydrogen phthalate is investigated to see if such an intramolecular hydrogen bond is formed.

The lattice constants and space group of $Co(C_8H_5O_4)_2.6H_2O$ were first determined by Cingi & Magnano (1959) from Weissenberg photographs.

Single crystals were grown by evaporation from a stoichiometric aqueous solution. The crystals grow in the form of (010) plates which are terminated by small faces of type (031), (011), (131), and $(13\bar{1})$.

Lattice parameters as given in the *Abstract* were determined from a single crystal by least-squares refinement of 18 diffraction angles measured at 20°C by an automatic diffractometer. Intensities of 3400 unique X-ray reflexions (with $\theta < 27°$) from a specimen of approximately isometric shape with a mean diameter of 0·3 mm, have been gathered by an automatic Hilger & Watts Y290 diffractometer using Mo $K\alpha$ radiation. Averaging reflexions $0kl$ and $0k\bar{l}$ yielded 384 unique reflexions with an internal consistency index

$$[\sum^{384} N \sum^{N} (\bar{F} - F)^2 \sigma^{-2}]/[\sum^{384} (N-1) \sum^{N} F^2 \sigma^{-2}]^{1/2} = 0{\cdot}0084$$

($N$ = number of equivalent reflexions). No correction for absorption has been applied ($\mu = 9{\cdot}1$ cm$^{-1}$).

The positions of the Co ions and of the C and O atoms of the hydrogen phthalate ion were determined by direct methods using *MULTAN* (Germain, Main & Woolfson, 1971). Further calculations were carried out with *SHELX* 76 (Sheldrick, 1976). The positions of the water molecules and the remaining H atoms were detected from difference Fourier syntheses. The structure was refined from 3400 reflexions with $F_o > 3\sigma$ by full-matrix least-squares procedures (assuming anisotropic temperature parameters for the non-hydrogen atoms) to a weighted $R$ value of 3·3% (unweighted $R$ = 3·8%. Squared reciprocal standard deviations were used as weights. Atomic scattering factors including correction for anomalous dispersion were taken from *International Tables for X-ray Crystallography* (1974). Since, in general, $F_o$ values of reflexions with large intensities were not smaller than corresponding $F_c$

Table 1. *Fractional atomic coordinates and* (*for* H *atoms*) *isotropic temperature factors* (× $10^4$)

Standard deviations given in parentheses refer to the last significant digits in the parameter values.

| | $x$ | $y$ | $z$ | $U$ (Å$^2$) |
|---|---|---|---|---|
| Co(1) | 0 (0) | 5000 (0) | 0 (0) | |
| Co(2) | 5000 (0) | 0 (0) | 0 (0) | |
| C(1) | 7305 (5) | 3371 (1) | 1336 (3) | |
| C(2) | 8292 (5) | 2977 (1) | 1291 (3) | |
| C(3) | 8217 (5) | 2724 (1) | 140 (4) | |
| C(4) | 7201 (6) | 2873 (1) | −984 (4) | |
| C(5) | 6259 (5) | 3272 (1) | −977 (3) | |
| C(6) | 6258 (4) | 3525 (1) | 183 (3) | |
| C(7) | 7389 (5) | 3639 (1) | 2580 (3) | |
| C(8) | 4932 (5) | 3916 (1) | 163 (3) | |
| C(11) | 7713 (4) | 6674 (1) | 4893 (3) | |
| C(12) | 6709 (5) | 7065 (1) | 4795 (3) | |
| C(13) | 6789 (6) | 7307 (1) | 3631 (4) | |
| C(14) | 7859 (6) | 7153 (1) | 2553 (4) | |
| C(15) | 8791 (5) | 6756 (1) | 2616 (3) | |
| C(16) | 8775 (4) | 6510 (1) | 3792 (3) | |
| C(17) | 7670 (4) | 6415 (1) | 6163 (3) | |
| C(18) | 9932 (5) | 6093 (1) | 3782 (3) | |
| O(1) | 7647 (4) | 3412 (1) | 3695 (2) | |
| O(2) | 7277 (4) | 4031 (1) | 2560 (2) | |
| O(3) | 3341 (3) | 3896 (1) | 846 (2) | |
| O(4) | 5351 (3) | 4229 (1) | −579 (2) | |
| O(11) | 7083 (3) | 6636 (1) | 7220 (2) | |
| O(12) | 8153 (4) | 6040 (1) | 6205 (2) | |
| O(13) | 11715 (3) | 6095 (1) | 4256 (2) | |
| O(14) | 9114 (3) | 5778 (1) | 3208 (2) | |
| O*W*(1) | 1518 (4) | 5318 (1) | 1601 (2) | |
| O*W*(2) | 293 (4) | 4440 (1) | 1017 (3) | |
| O*W*(3) | 7181 (4) | 5084 (1) | 996 (3) | |
| O*W*(4) | 4835 (5) | 573 (1) | 1035 (3) | |
| O*W*(5) | 2097 (5) | 9807 (1) | 774 (3) | |
| O*W*(6) | 6366 (6) | 9743 (1) | 1689 (3) | |
| H(2) | 987 (48) | 7886 (10) | 2963 (29) | 351 (94) |
| H(3) | 1061 (55) | 7551 (11) | −134 (31) | 550 (113) |
| H(4) | 2848 (52) | 7311 (12) | 1782 (33) | 481 (103) |
| H(5) | 4518 (47) | 6623 (10) | 1763 (28) | 370 (90) |
| H(12) | 5945 (46) | 7839 (10) | 555 (28) | 305 (88) |
| H(13) | 3791 (52) | 2578 (11) | 1436 (31) | 495 (107) |
| H(14) | 1863 (54) | 2334 (12) | 3182 (33) | 570 (114) |
| H(15) | 408 (47) | 1626 (10) | 3102 (28) | 365 (91) |
| HO(1) | 7934 (109) | 3564 (21) | 4303 (60) | 1783 (294) |
| HO(11) | 3407 (73) | 3536 (15) | 2131 (41) | 1041 (172) |
| H*W*(11) | 836 (57) | 5460 (12) | 2089 (33) | 469 (124) |
| H*W*(12) | 2360 (65) | 5467 (13) | 1331 (38) | 626 (156) |
| H*W*(21) | −592 (54) | 4298 (12) | 1430 (32) | 453 (114) |
| H*W*(22) | 1138 (57) | 4273 (12) | 928 (35) | 446 (129) |
| H*W*(31) | 6594 (62) | 5321 (14) | 983 (39) | 613 (145) |
| H*W*(32) | 6892 (122) | 4916 (22) | 1152 (70) | 2062 (342) |
| H*W*(41) | 5685 (79) | 736 (17) | 986 (49) | 466 (210) |
| H*W*(42) | 3846 (74) | 659 (16) | 1287 (46) | 375 (198) |
| H*W*(51) | 2404 (78) | 9660 (14) | 937 (48) | 1299 (194) |
| H*W*(61) | 6806 (71) | 9880 (15) | 1995 (43) | 610 (178) |
| H*W*(62) | 7314 (86) | 9577 (18) | 1441 (54) | 1546 (260) |

values, extinction was not considered. Final positional parameters are given in Table 1.*

**Discussion.** In Fig. 1 a stereoscopic view of the unit cell is given. The Co ions are represented by black ellipsoids. For all H atoms a uniform radius of 0·1 Å was assumed.

The structure may be roughly described as being composed of different layers parallel to the (010) plane. Nets of Co ions which are surrounded by six water molecules are situated at $y = 0$ and $y = 0{\cdot}5$. In between these nets there are double layers of hydrogen phthalate ions. Their carboxylic groups are turned to the Co/water nets and linked by hydrogen bonds. The interconnexion of the two hydrogen phthalate layers at approximately $y = 0{\cdot}25$ and $y = 0{\cdot}75$ is achieved merely by van der Waals forces.

The two independent Co ions are in special positions and occupy inversion centers. They are coordinated to six O atoms forming nearly regular octahedra which are outlined in Fig. 1 by thin lines. Bond distances and angles in the octahedra are given in Table 2.

Distances and angles within the two hydrogen phthalate ions, which are given in Fig. 2, scatter in the range of the usual values. Apparently, the hydrogen phthalate ions do not give rise to any intramolecular hydrogen bonds or nearly planar configurations. The planes of the carboxylic groups form the following angles with the mean planes through the respective benzene rings I and II: ring I with the plane through C(7), O(1), O(2): 28·7°; ring I with C(8), O(3), O(4): 69·6°; ring II with C(17), O(11), O(12): 15·7°; ring II with C(18), O(13), O(14): 98·8°.

The least-squares planes through atoms C(1) to C(8) and C(11) to C(18) are (as related to a Cartesian ångström coordinate system with $x \parallel a^*$, $y \parallel b$ and $z \parallel c$), respectively $0{\cdot}8191x + 0{\cdot}4673y - 0{\cdot}3327z = 8{\cdot}3477$, $0{\cdot}8297x + 0{\cdot}4595y + 0{\cdot}3170z = 7{\cdot}5596$. The maximum deviations from the least-squares planes are shown by C(8) (−0·087 Å) and C(15) (0·025 Å).

The intracrystalline connexion between the hydrogen phthalate ions and the cobalt–water octahedra is achieved by several hydrogen bonds which are listed in

Table 2. *Bond distances* (Å) *and angles* (°) *in the cobalt–water octahedra*

| | | | |
|---|---|---|---|
| Co(1)–O*W*(1) | 2·105 (2) | Co(2)–O*W*(4) | 2·054 (3) |
| Co(1)–O*W*(2) | 2·014 (2) | Co(2)–O*W*(5) | 2·150 (3) |
| Co(1)–O*W*(3) | 2·128 (2) | Co(2)–O*W*(6) | 2·047 (3) |
| O*W*(1)–Co(1)–O*W*(2) | 90·8 (1) | O*W*(4)–Co(2)–O*W*(5) | 90·4 (1) |
| O*W*(1)–Co(1)–O*W*(3) | 90·3 (1) | O*W*(4)–Co(2)–O*W*(6) | 92·7 (1) |
| O*W*(2)–Co(1)–O*W*(3) | 92·9 (1) | O*W*(5)–Co(2)–O*W*(6) | 91·3 (1) |
| O*W*(1)–O*W*(2) | 2·892 (3) | O*W*(4)–O*W*(5) | 2·983 (3) |
| O*W*(1)–O*W*(3) | 2·986 (3) | O*W*(4)–O*W*(6) | 2·830 (3) |
| O*W*(2)–O*W*(3) | 2·855 (3) | O*W*(5)–O*W*(6) | 2·935 (3) |

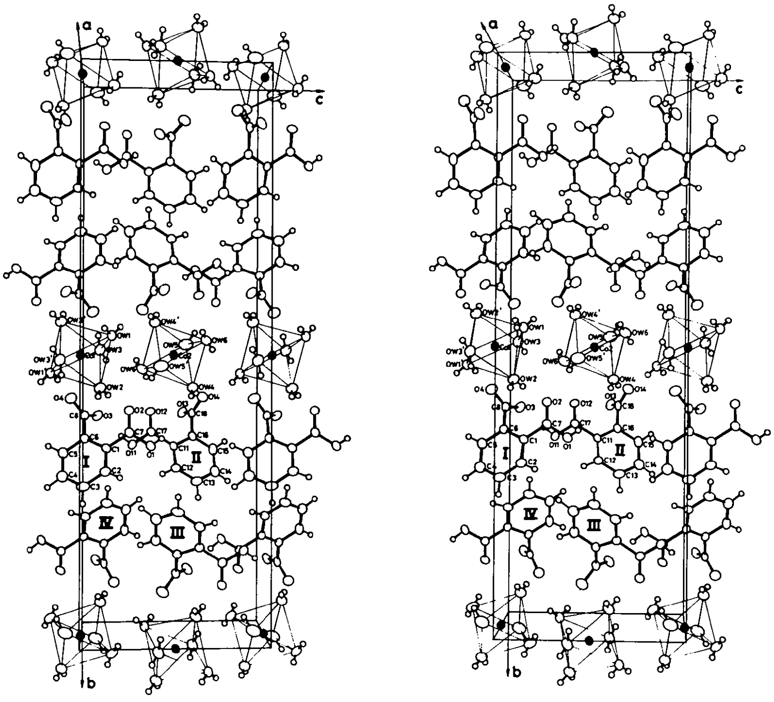

Fig. 1. Stereoscopic view of the unit cell as seen down the *a* axis.

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33707 (23 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 3. The first two (short) hydrogen bonds connect the hydrogen phthalate ions with each other. The following nine hydrogen bonds provide linkage between the hydrogen phthalate ions with water molecules. The final hydrogen bond connects two water molecules. All H atoms with the exception of H*W*(32) and H*W*(52) are involved in hydrogen bonds. Consequently, the localization of H*W*(32) and H*W*(52) was very difficult in the course of the structure determination. H*W*(32) shows the largest temperature factor of all the H atoms. The position of H*W*(52) could not be determined by refinement. H*W*(52) is, therefore, missing in Table 1 and in Fig. 1.

Special attention should be drawn to the packing of the hydrogen phthalate molecules within the double layers mentioned previously. Careful inspection of the stereoscopic drawing (Fig. 1) shows that among the molecules of a particular double layer a pseudo symmetry exists which is not due to the space group. The two symmetrically independent molecules I and II (Fig. 1) are found to be intercorrelated by a pseudo glide plane parallel to (001) with a translation parallel to the viewing direction [100]. Neglecting the fact that $\beta$ deviates by 1° from 90°, the position of the pseudo glide plane can be determined by averaging the $z$ values of corresponding atoms of the two molecules. Thus, a mean value of $z = 0{\cdot}3214\ (47)$ results for the double layer in the lower part of the unit cell in Fig. 1. In the upper part of the unit cell, the pseudo glide plane lies at $z = 0{\cdot}1786$ (and 0·6786), according to the symmetry of the space group. The water molecules around the Co atoms do not obey the pseudo symmetry. Obviously, these pseudo glide planes, together with the true glide plane $c$ of the space group, generate pseudo screw axes $2_1$ parallel to [100] at $y = 0{\cdot}25$ and $z = 0{\cdot}0714$ or 0·5714, in the lower part of the unit cell. By means of this pseudo-$2_1$-operation, for example, molecule I is approximately transformed into molecule IV, or II into III (Fig. 1).

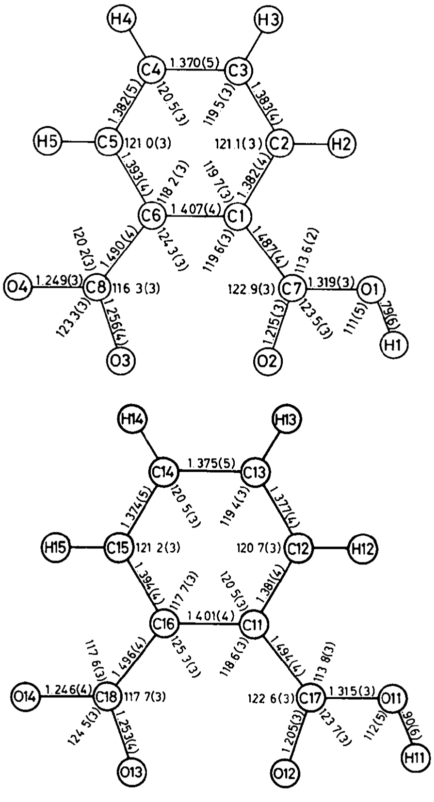

Fig. 2. Bond lengths (Å) and angles (°) in the hydrogen phthalate ions.

In order to obtain a measure of how well this pseudo symmetry is fulfilled the coordinates $x$, $y$, and $z$ of molecule II were transformed according to a glide plane operation of type '$a$' at $z = 0{\cdot}3214$ (again assuming $\beta = 90°$). The values obtained differ only slightly from those of the corresponding atoms in molecule I and the magnitude of the deviations can be characterized by the following standard deviations $\sigma$: $\sigma(x) = 0{\cdot}1496$, $\sigma(y) = 0{\cdot}0688$, $\sigma(z) = 0{\cdot}0930$ Å.

The C atoms obey the pseudo symmetry to a better degree. If the C atoms only are considered the respective standard deviations are: $\sigma(x) = 0{\cdot}0156$, $\sigma(y) = 0{\cdot}0237$, $\sigma(z) = 0{\cdot}0468$ Å. These low values show that the pseudo symmetry within the layers, although not being required by the space group, is fulfilled to an astonishingly high degree.

The pseudo symmetry causes a pseudo extinction: $hk0$ reflexions with $h = 2n + 1$ are found to have low intensities. The $F_o$ values averaged over $k$ are as follows: $\langle F_{1k0}\rangle = 3{\cdot}6$; $\langle F_{2k0}\rangle = 32{\cdot}4$; $\langle F_{3k0}\rangle = 8{\cdot}2$; $\langle F_{4k0}\rangle = 28{\cdot}5$; $\langle F_{5k0}\rangle = 4{\cdot}9$; $\langle F_{6k0}\rangle = 17{\cdot}3$; $\langle F_{7k0}\rangle = 3{\cdot}8$; $\langle F_{8k0}\rangle = 11{\cdot}4$. It should be mentioned that this pseudo extinction is correctly obeyed also by the Co atoms which lie in special positions and which contribute about 10% to the total scattering power.

The pseudo symmetry within the double layers can be described in terms of one of the 80 symmetry groups for layers of three-dimensional figures which were first derived by Weber (1929) (see also Kitaigorodskii, 1955). The present symmetry corresponds to the group $p2_1(c)a$. In this notation, according to the usual convention, the symmetry element referring to the direction of missing periodicity is enclosed in round brackets. Kitaigorodskii (1955) points out that the space group $P2_1ca$ is one of the groups very well suited

Table 3. *Hydrogen bonds and* O–O *distances* (Å)

| | | | |
|---|---|---|---|
| O(1)–HO(1)···O(13) | 2·571 | O(4)···H*W*(31)–O*W*(3) | 2·721 |
| O(11)–HO(11)···O(3) | 2·550 | O(12)···H*W*(62)–O*W*(6) | 2·747 |
| O(2)···H*W*(21)–O*W*(2) | 2·820 | O(13)···H*W*(41)–O*W*(4) | 2·794 |
| O(2)···H*W*(51)–O*W*(5) | 2·904 | O(14)···H*W*(11)–O*W*(1) | 2·674 |
| O(3)···H*W*(22)–O*W*(2) | 2·626 | O(14)···H*W*(42)–O*W*(4) | 2·787 |
| O(4)···H*W*(12)–O*W*(1) | 2·697 | O*W*(6)–H*W*(61)···O*W*(1) | 2·803 |

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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*Acta Cryst.* (1978). B**34**, 3083–3086

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

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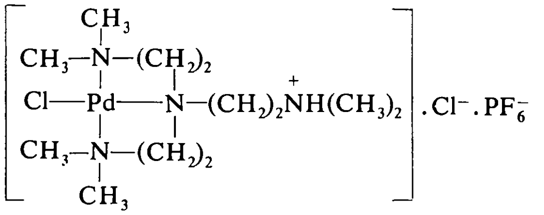
(*Received* 7 *March* 1978; *accepted* 26 *May* 1978)

**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 $P2_1/c$ ($C^5_{2h}$, 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$_6$tren 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$_6$tren = 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), $[Pd(Me_6trenH)Cl]ClPF_6$.



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

(I)

Preliminary space-group and unit-cell information were obtained by photographic measurements. Least-squares analysis of the diffractometer coordinates of 12 reflections with $\theta$(Mo $K\alpha$) between 10 and 15° led to precise values for the lattice parameters. Intensity data