not involved in the coordination, in contrast to the barium glucose 6-phosphate heptahydrate structure, where seven atoms of the nine in the coordination sphere are from water molecules. The carboxyl O atom (O1A) not taking part in the coordination has a shorter C—O bond length [1·237 (8) Å] as compared with the average value of 1·254 (7) Å for the other three.

Hydrogen bonding. The single water molecule is prominent in the hydrogen-bond network, accepting two bonds (from equivalent hydroxyl groups of the two half-molecules) and donating a hydrogen bond (bifurcated) to a hydroxyl group and a symmetry-related water molecule. The latter values are weaker with H···O distances of 2·340 and 2·465 Å compared with the average of 1·840 Å for the other four.

Calcium galactarate

The asymmetric unit consists of one half of a Ca^{2+} ion (on a twofold rotation axis), one half of a galactarate ion (related to the other half by a centre of symmetry) and two water molecules. This contrasts with the structure of the Ba salt which has two distinct half-molecules of the galactarate ion, a unique Ba^{2+} ion and one water molecule per asymmetric unit. Bond lengths and angles have normal values for carbohydrates (Foces-Foces, Cano & Garcia-Blanco, 1981).

Ca^{2+} *coordination.* Each Ca^{2+} ion is coordinated to eight O atoms, with the Ca—O distances in the range 2·40 (1)–2·54 (1) with an average = 2·46 Å (Table 3b). The four O atoms listed are symmetry related (twofold rotation axis) to the other four, producing a characteristic square-antiprism arrangement around the Ca^{2+}

ion, with near-normal Ca—O distances for this type of coordination as established by Einspahr & Bugg (1981).

Hydrogen bonding. All hydroxyl H atoms take part in the hydrogen-bonding scheme with the second water molecule (not involved in the calcium coordination) being a receptor for two H bonds and a donor for two.

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Bis(*n*-propylammonium) Tetrachloroplumbate

BY A. MERESSE

Laboratoire de Cristallographie, UA 144, *Université de Bordeaux* I, 33405 *Talence, France*

AND A. DAOUD

Laboratoire de l'Etat Solide, ENIS, BP W, Sfax, Tunisia

Abstract. $[NH_3(C_3H_7)]_2[PbCl_4]$, $M_r = 469$, orthorhombic, *Pnma*, $a = 7.815$ (1), $b = 25.034$ (3), $c = 7.954$ (1) Å, $V = 1556$ Å^3, $Z = 4$, $D_x = 2.0$ g cm^{-3}, D_m not measured, λ (Cu $K\alpha$) = 1.54178 Å, $\mu = 271$ cm^{-1}, $F(000) = 880$, room temperature ($\simeq 295$ K), 1228 non-equivalent diffractometer data up to $(\sin\theta)/\lambda = 0.6$ Å^{-1}, final $R(F) = 0.079$, $wR(F) = 0.091$. This compound crystallizes in a perovskite-type layer structure. The Cl octahedra centred by Pb atoms form infinite two-dimensional layers which are sandwiched between the propylammonium chains. The results show a net deformation of Cl octahedra.

Introduction. The perovskite-type layer compounds, with the general formula $[NH_3(C_nH_{2n+1})]_2[MCl_4]$, where M is a metal, show a two-dimensional structure.

Table 1. *Atomic coordinates and equivalent values of the anisotropic temperature factors*

$$B_{eq} = \tfrac{4}{3}\Sigma_i\Sigma_j B_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$B_{eq}(Å^2)$
Pb(1)	0·3470 (2)	0·25	0·5100 (2)	3·00
Cl(2)	0·3208 (7)	0·3639 (3)	0·4712 (8)	4·13
Cl(3)	0·024 (1)	0·25	0·6726 (9)	4·00
Cl(4)	0·649 (1)	0·25	0·2983 (9)	3·41
C(11)	0·812 (5)	0·121 (1)	0·538 (5)	5·47
C(12)	0·828 (5)	0·061 (2)	0·503 (6)	8·24
C(13)	0·721 (6)	0·029 (2)	0·627 (6)	10·51
N(14)	0·916 (3)	0·152 (1)	0·418 (3)	4·20

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

Pb(1)–Cl(2)	2·879 (7)	N(14)–C(3')	3·30 (2)
Pb(1)–Cl(3)	2·830 (7)	N(14)–Cl(4)	3·36 (2)
Pb(1)–Cl(4)	2·911 (7)	N(14)–Cl(2)	3·22 (2)
Pb(1)–Cl(5)	2·878 (7)	N(14)–Cl(6)	3·50 (2)
Pb(1)–Cl(6)	2·899 (7)	N(14)–Cl(2'')	3·18 (2)
C(11)–C(12)	1·50 (5)	Cl(4)–H(211)	3·49
C(12)–C(13)	1·52 (6)		
C(11)–N(14)	1·51 (4)		

N(14)–C(11)–C(12)	110 (2)	Cl(3)⋯N(14)–C(11)	97 (1)
C(11)–C(12)–C(13)	110 (2)	Cl(4)⋯N(14)–C(11)	103 (1)

The structural arrangement consists of inorganic layers built up from corner-sharing Cl octahedra with M^{2+} in the centres, the cavities between octahedra being occupied by the $[NH_3(C_3H_7)]^+$ groups. In this paper we are interested in the compound with $M =$ Pb and $n = 3$.

Experimental. The synthesis of the title compound is realized through the following procedure: first, by reaction of HCl on PbO_2, we obtain the lead chloride $PbCl_4$; then, a reaction between propylammonium chloride and $PbCl_4$, in HCl solution, in the stoichiometric ratio 2:1, gives $[NH_3(C_3H_7)]_2[PbCl_4]$ directly. The product crystallizes as single platelets. The composition of the final compound is confirmed by elementary analysis.

A spherical crystal 0·3 mm in diameter was used for the experiment. Weissenberg photographs showed the space group to be either $Pna2_1$ or $Pnma$; the latter was confirmed during the structure refinement. Lattice parameters were obtained from least-squares adjustment of the setting angles of 25 reflections with $9 < \theta < 35°$. Data were collected in the four quadrants of reciprocal space up to $(\sin\theta)/\lambda = 0·63$ Å^{-1}, with Enraf–Nonius CAD-4 diffractometer, graphite-monochromated Cu $K\alpha$ radiation, ψ scan; range of hkl: h –9→9, k –10→10, l 0→31. Empirical absorption correction was applied to 5240 intensities measured, transmission factors ranged from 0·60 to 0·99. Three monitored reflections ($\overline{1}1\overline{1}$, 531, $\overline{2}2\overline{1}$) observed after every 60 reflections showed no change in intensities. Symmetry-equivalent reflections were averaged, $R_{int} = 0·039$ for all reflections and 0·062 for the observed reflections only, resulting in 1756 reflections of which 1228 were observed with $F > 3\sigma(F)$.

The structure was determined by heavy-atom and Fourier techniques. Atomic scattering factors were from *International Tables for X-ray Crystallography* (1974), except for H (Stewart, Davidson & Simpson, 1965). Anomalous-dispersion factors were applied to Pb. The H atoms were located in theoretical positions and not refined. No correction was made for secondary extinction. The structure was refined, with $w = 1$ if $F_o < p$ and $w = p^2/F_o^2$ if $F_o > p$ where $p = (F_o{}^2{}_{max}/10)^{1/2}$, to $R(F) = 0·079$, $wR(F) = 0·091$ (poor quality of crystal and hence of intensity data) and $S = 8·49$. Max. and min. electron densities in final difference map 0·47 and $-0·38$ e Å^{-3}, max. shift/e.s.d. in final cycle 0·070.

The calculations were carried out with local programs *CRISAFFI* and *CRISUTIL* on the Mini 6/92 Bull computer. The positional parameters of the atoms are reported in Table 1, bond lengths and angles in Table 2.*
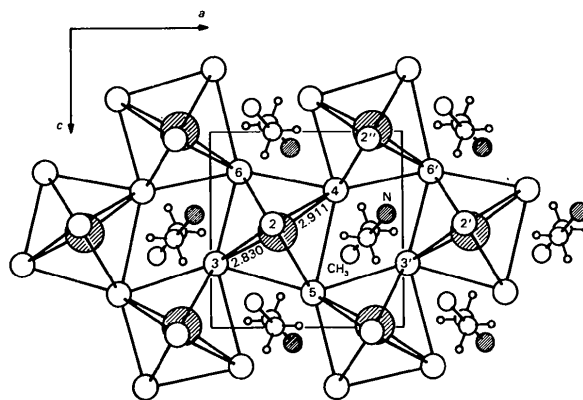
Fig. 1. Projection of the structure along **b**.
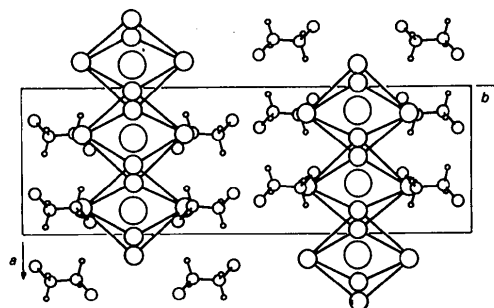
Fig. 2. Projection of the structure along **c**.

Discussion. The structure consists of infinite two-dimensional $PbCl_6$ octahedra layers with organic $NH_3(C_3H_7)$ groups situated between the layers (Figs. 1 and 2); the ammonium ends are linked to the octahedra by NH···Cl hydrogen bonds. The results show no disorder of the organic part unlike the structure of the similar compound $[NH_3(C_3H_7)]_2[CdCl_4]$ [C_3Cd (Chapuis, 1978)]. Another difference is observed in the perovskite layer: in the C_3Cd compound, the octahedra have nearly tetragonal symmetry but this is not true in the C_3Pb derivative. For the lead compound, the deformation of octahedra can be seen through the following facts: in the equatorial plane – which is a mirror plane – the bond distances between the atoms

C—C vary from 3·86 to 4·32 Å and the bond distances between an axial Cl atom to each equatorial Cl atom vary from 3·81 to 4·32 Å. An angle of nearly 45° is observed between the c axis and the mean plane of the organic part, which is parallel to the **b** direction perpendicular to the perovskite layer.

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Hydrido{tris[2-(diphenylphosphino)ethyl]phosphino-*P,P',P'',P'''*}platin(II) Tetraphenylborat

VON ALFRED GIEREN, PETER BRÜGGELLER, KLAUS HOFER, THOMAS HÜBNER UND CATALINA RUIZ-PÉREZ

Institut für Anorganische und Analytische Chemie, Universität Innsbruck, A-6020 Innsbruck, Österreich

Abstract. $[Pt(H)\{(C_6H_5)_2PC_2H_4\}_3P][(C_6H_5)_4B]$, $M_r = 1186·06$, triclinic, $P\bar{1}$, $a = 11·677$ (2), $b = 13·661$ (3), $c = 18·286$ (3) Å, $\alpha = 94·65$ (2), $\beta = 106·98$ (1), $\gamma = 96·36$ (2)°, $V = 2752·8$ Å^3, $Z = 2$, $D_m = 1·41$, $D_x = 1·431$ Mg m^{-3}, $\lambda(Mo\ K\alpha) = 0·71069$ Å, $\mu = 2·73$ mm^{-1}, $F(000) = 1204$, $T = 294$ K, final $R = 0·021$ for 9934 observed reflections. The X-ray structure analysis confirms the trigonal-bipyramidal coordination geometry of the title compound. The Pt atom is shifted out of the equatorial plane towards the hydride ligand by 0·179 Å. The apical Pt—P bond (2·27 Å) is significantly shorter than the equatorial ones (2·33 Å). The equatorial Pt—P bonds differ slightly in length ($\Delta_{max} = 0·03$ Å) in accordance with a slight distortion of the threefold symmetry of the cation.

Einleitung. King, Kapoor, Saran & Kapoor (1971) beschrieben die Synthese von (2) und haben eine quadratisch-planare Koordination des Platins(II) vorgeschlagen. Kürzlich wurde durch Umsetzung von (2) mit NaBH$_4$ und Na(BPh$_4$) das analoge (1) erhalten und diesem jedoch NMR-spektroskopisch eine trigonal-bipyramidale Struktur zugeschrieben (Brüggeller, 1987). Insbesonders da auch Komplexe des Co^I und Ni^{II} mit Tris[2-(diphenylphosphino)ethyl]phosphin (PP$_3$) bzw. vergleichbaren Liganden ebenfalls trigonal-bipyramidale Strukturen besitzen (Morassi, Bertini &

Sacconi, 1973; Mani & Sacconi, 1983; Hohman, Kountz & Meek, 1986), erschien auch eine Kristallstrukturanalyse von (1) erforderlich.

X = H, Y = (BPh$_4$): (1)
X = Cl, Y = Cl: (2)
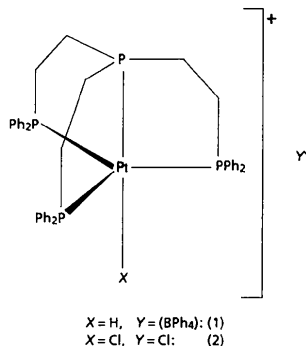
Experimentelles. (1) (Brüggeller, 1987) wurde aus Methylenchlorid umkristallisiert und ein Kristall mit den Dimensionen 0,14 × 0,32 × 0,64 mm wurde auf einen Glasfaden montiert. D_m mittels Schwebemethode, Enraf–Nonius CAD-4-Diffraktometer, graphit-monochromatisierte Mo $K\alpha$-Strahlung, Gitterkonstanten aus den Orientierungsparametern von 25 Reflexen im Bereich $4,5 \le \theta \le 16,8°$. Datensammlung mit $\omega/2\theta$-Abtastung, Referenzreflexe ($\bar{1}17$, $06\bar{1}$, 311)