

(Toniolo, Bonora, Bavoso, Benedetti, Di Blasio, Pavone & Pedone, 1983; Prasad & Balaram, 1984).

Both molecules *A* and *B* show the piperidine ring in a chair (4C_1) conformation and the —CONH— substituent in the axial disposition, as expected from calculations (Toniolo *et al.*, 1989). The urethane and amide groups have the *trans* orientation, but the former is markedly out-of-plane. Interestingly, the tripeptide analogue *Z*-Aib-L-Pro-Aib-OMe is also folded in the crystal state, showing the same type of β -bend (distorted type-I) (Benedetti, Bavoso, Di Blasio, Pavone, Pedone, Toniolo, Bonora & Crisma, 1983).

In the crystal packing intermolecular N—H...O=C hydrogen bonds occur, involving the urethane Aib(1) N—H as donor and the peptide Pip(2) C=O as acceptor. Alternating *A* and *B* molecules form infinite chains along *a*. The N(1) atom of an *A* molecule is hydrogen bonded to the O(4') atom of an enantiomeric *B* molecule ($x, \frac{1}{2} - y, \frac{1}{2} + z$), while the N(1') atom of a *B* molecule is hydrogen bonded to the O(4) atom of an enantiomeric *A* molecule ($x + 1, \frac{1}{2} - y, z - \frac{1}{2}$). The corresponding N...O distances are 2.89 and 2.88 Å.

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Structure of Tetrachlorophosphonium Hexafluoroantimonate

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Abstract. $[\text{PCl}_4][\text{SbF}_6]$, $M_r = 408.526$, tetragonal, $P4/n$, $a = 8.568$ (3), $c = 6.531$ (5) Å, $V = 479.4$ Å³, $Z = 2$, $D_x = 2.830$ Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 4.22$ mm⁻¹, $F(000) = 376$, $T = 169$ K, final $R = 0.059$ for 425 unique observed [$F \geq 4.0\sigma(F)$] diffractometer data. The unit cell contains two $[\text{PCl}_4]^+$

cations $[\text{P—Cl } 1.929$ (2) Å ($\times 4$), $\text{Cl—P—Cl } 109.15$ (8) ($\times 2$), 109.63 (8)^o ($\times 4$)] and two $[\text{SbF}_6]^-$ anions $[\text{Sb—F } 1.864$ (5) ($\times 4$), 1.924 (9) ($\times 1$), 1.912 (8) Å ($\times 1$), $\text{F—Sb—F } 90.0$ or 180.0°]. The anion resides on a fourfold axis and the cation on a fourfold inversion axis.

Experimental. Single crystals were prepared from a saturated solution of $\text{PCl}_4^+ \cdot \text{SbF}_6^-$ in anhydrous HF at 243 K in a KEL-F reactor equipped with a stainless-steel valve. Over a period of 18 d the HF was removed stepwise at this temperature until crystallization began. The solution was then decanted and the crystals dried by pumping off the HF in a high vacuum. A crystal of approximate size $0.18 \times 0.36 \times 0.42$ mm was used. The crystal was sealed in a Lindemann-glass capillary under inert atmosphere. D_m was not determined. Intensity data were collected using $\omega/2\theta$ scans with variable scan speed $1.5\text{--}15.0^\circ \text{ min}^{-1}$ in θ and scan width $1.2^\circ +$ dispersion. A Nicolet *R3m/V* diffractometer with graphite-monochromated $\text{Mo K}\alpha$ radiation and equipped with a liquid- N_2 LT-1 low-temperature device was used for preliminary examinations and data collection. The lattice parameters were determined from a symmetry-constrained least-squares fit of the angular settings for 25 reflections with $2\theta_{\text{max}} = 28.8^\circ$. ω scans of low-order reflections along the three crystal axes showed acceptable mosaicity. Six standard reflections (400, 040, 002, $\bar{4}00$, $\bar{0}40$, $0\bar{0}2$) were recorded every 300 reflections and showed only random deviations during 14.5 h of X-ray exposure. 535 reflections with $2.0 \leq 2\theta \leq 50.0^\circ$ (3378 accessible), $0 \leq h \leq 11$, $0 \leq k \leq 11$, $0 \leq l \leq 8$ were measured. The data were corrected for Lorentz-polarization but not for absorption effects and averaged ($R_{\text{int}} = 0.061$) to 431 unique reflections, 425 of which had $F \geq 4.0\sigma(F)$. The systematic absences ($hk0$) $h+k = 2n+1$ and the diffraction symmetry conform to space group $P4/n$. The structure was solved via a Patterson function and $\Delta\rho$ maps. It was refined (on F) using full-matrix least squares with anisotropic displacement parameters for all atoms. 30 parameters were refined. Weights $w^{-1} = \sigma^2(F) + 0.0037F^2$ led to a featureless analysis of variance in terms of $\sin\theta$ and F_o . The refinement converged to $S = 2.48$, $R = 0.059$, $wR = 0.083$, $(\Delta/\sigma)_{\text{max}} = 0.009$ (no extinction correction). No explanation can be given for the rather high R values. The correctness of the space-group choice was checked by using *MISSYM* (Le Page, 1987). The largest peaks in the final $\Delta\rho$ map were $\pm 1.6(5) \text{ e } \text{\AA}^{-3}$ (near Sb). Atomic scattering factors for neutral atoms and real and imaginary dispersion terms were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). The programs used were *PARST* (Nardelli, 1983), *SHELX76* (Sheldrick, 1976), *SHELXTL-Plus* (Sheldrick, 1987), *PLATON* (Spek, 1982), *SADIAN* (Baur & Wenninger, 1969), and *MISSYM* (Le Page, 1987). The anion and cation and the numbering scheme are shown in Fig. 1 and a stereoscopic view of the unit-cell contents is presented in Fig. 2. Positional parameters and the equivalent values of the anisotropic displacement

parameters are given in Table 1.* Bond lengths and bond angles are given in Table 2.

Related literature. Several crystals containing a PCl_4^+ cation have already been investigated and their crystal structures have been published: $\text{PCl}_4^+ \cdot \text{TeCl}_5^-$ (Collins & Webster, 1972), $\text{PCl}_4^+ \cdot \text{TeCl}_5^-$ (Krebs, Buss & Berger, 1973); $\text{PCl}_4^+ \cdot \text{ICl}_2^-$ (Zelezny & Baenziger, 1952); $\text{PCl}_4^+ \cdot \text{Ti}_2\text{Cl}_9^-$ and $2\text{PCl}_4^+ \cdot \text{Ti}_2\text{Cl}_{10}^-$ (Kistenmacher & Stucky, 1971); $\text{PCl}_4^+ \cdot \text{NbCl}_6^-$ and $\text{PCl}_4^+ \cdot \text{TaCl}_6^-$ (Preiss, 1971c); $\text{PCl}_4^+ \cdot \text{FeCl}_4^-$ (Kistenmacher & Stucky, 1968); $\text{PCl}_4^+ \cdot \text{VCl}_5^-$ (Ziegler, Nuber, Weidenhammer & Hoch, 1977); $\text{PCl}_4^+ \cdot \text{UCl}_6^-$ (Taylor & Waugh, 1983); $3\text{PCl}_4^+ \cdot \text{Sn}_2\text{Cl}_8^{2-} \cdot \text{PCl}_6^-$, $\text{PCl}_4^+ \cdot \text{Sn}_2\text{Cl}_{10}^{2-}$, $\text{PCl}_4^+ \cdot \text{SnCl}_5^-$ (Shamir, Luski, Bino, Cohen & Gibson, 1985); $\text{PCl}_4^+ \cdot \text{AuCl}_4^-$ (Erdbrügger, Jones, Schelbach, Schwarzmann & Sheldrick, 1987);

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55078 (4 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: BX0585]

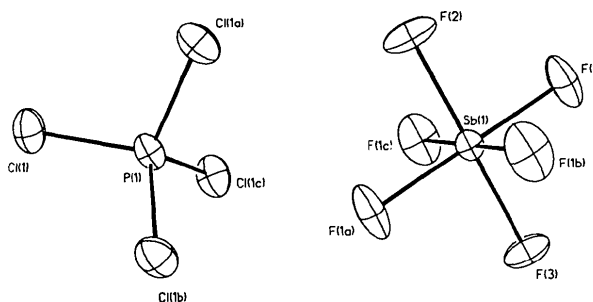


Fig. 1. General view (Sheldrick, 1987) of the cation and the anion, with the atom numbering [symmetry code at Cl: (a) $0.5 - x, 1.5 - y, z$; (b) $-0.5 + y, 1 - x, 1 - z$; (c) $1 - y, 0.5 + x, 1 - z$; and at F: (a) $1.5 - x, 1.5 - y, z$; (b) $0.5 - y, x, z$; (c) $1.5 - x, z$]. Anisotropic ellipsoids represent 50% probability boundaries.

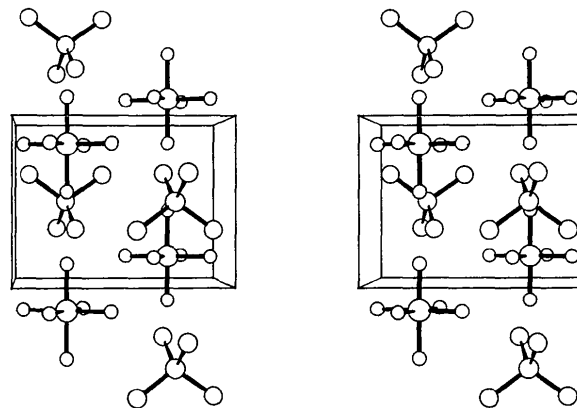


Fig. 2. Stereoscopic view (*SCHAKAL*; Keller, 1986) of the unit cell.

Table 1. Atomic coordinates and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^4$)
$$U_{eq} = (1/3) \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	U_{eq}
Sb(1)	0.75	0.75	0.8461 (1)	206
Cl(1)	0.0719 (2)	0.7059 (2)	0.3288 (3)	361
P(1)	0.25	0.75	0.5	241
F(1)	0.0523 (6)	0.6698 (7)	0.8461 (8)	485
F(2)	0.75	0.75	0.552 (1)	566
F(3)	0.75	0.75	1.139 (1)	458

Table 2. Bond distances (\AA) and bond angles ($^\circ$)

Anion		Cation	
Sb(1)—F(1)	1.864 (5) × 4	P(1)—Cl(1)	1.929 (2) × 4
Sb(1)—F(2)	1.924 (9) × 1	Cl—P—Cl	109.15 (8) × 2
Sb(1)—F(3)	1.912 (8) × 1	Cl—P—Cl	109.63 (8) × 4
F—Sb—F	90.0 or 180.0		

PCl₄⁺·(Sb,Nb,Ta)Cl₆⁻·AsCl₃ (Preiss & Reich, 1971); PCl₄⁺·ICl₄⁻ (Shamir, Schneider, Bino & Cohen, 1986); PCl₄⁺·PCl₆⁻ (Clark, Powell & Wells, 1942). The investigated crystal of the title compound is isomorphous with the crystals of the compounds AsCl₄⁺·AsF₆⁻ (Preiss, 1971a; Minkwitz, Nowicki & Borrmann, 1991) and PCl₄⁺·PCl₆⁻ (Preiss, 1971b).

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Structure of Aluminium Disodium Trihydrogenbis(diphosphate)

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Abstract. Na₂AlH₃(P₂O₇)₂, $M_r = 423.9$, triclinic, $P\bar{1}$, $a = 8.311$ (4), $b = 7.363$ (4), $c = 4.902$ (3) \AA , $\alpha =$

99.67 (2), $\beta = 81.77$ (2), $\gamma = 114.48$ (1) $^\circ$, $V = 268.2$ (4) \AA^3 , $Z = 1$, $D_x = 2.624$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.7107$ \AA , $\mu = 9.31$ cm⁻¹, $F(000) = 210$, $T = 293$ K, $R = 0.032$ for 761 independent observed reflections. The Al octahedron shares two opposite edges with

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