in the Ni (Holian & Marsh, 1970; Coghi, Mangia, Nardelli & Pelizzi, 1969; Ward, Caughlan & Smith, 1971) and Cu (Mathew & Kunchur, 1970) complexes.

The bond distances $N(sp^2)$—$C(sp^2)$ of the ligand molecule are not all equal; the larger ones [1·367 (2)– 1·381 (2) Å] involve the N atoms of the NH groups, the medium ones [1·326 (2)–1·338 (3) Å] involve the N atoms of the NH_2 groups and the shorter ones [1·303 (3)–1·310 (2) Å] include the N atoms coordinating with Mn. All these values indicate that the π-electron cloud is delocalized and spread over the six-membered rings which are stabilized by resonance. All the independent nitrate ions are planar and have normal N—O distances ranging from 1·224 (2) to 1·266 (3) Å.

The packing of the molecule viewed down the c axis is shown in Fig. 2. The molecules are held together by normal van der Waals contacts and by a network of hydrogen bonds. The axial water molecules of the square pyramid are engaged in hydrogen bonding with the nitrate ions and all three nitrate ions are hydrogen bonded to the complex ion.

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Structure of Two Polymorphic Forms of Phenothiazine Tetrachloroantimonate

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Abstract. The crystal structures of two polymorphic forms of phenothiazine tetrachloroantimonate, triclinic (I) and monoclinic (II), were determined. $C_{12}H_9NS.SbCl_4$, $M_r = 462·8$. (I) Triclinic, $P\bar{1}$, $a = 12·149 (10)$, $b = 13·023 (9)$, $c = 11·750 (8)$ Å, $\alpha = 108·57 (5)$, $\beta = 107·78 (6)$, $\gamma = 104·65 (6)°$, $V = 1547 (2)$ Å^3, $Z = 4$, $D_x = 1·99$, $D_m = 1·99$ Mg m^{-3}, λ(Mo $K\alpha) = 0·71073$ Å, $\mu = 2·61$ mm^{-1}, $F(000) = 892$, $T = 293$ K, $R = 0·062$ for 4184 observed reflections. (II) Monoclinic, $P2_1/c$, $a = 12·154 (6)$, $b = 18·116 (3)$, $c = 7·517 (2)$ Å, $\beta = 108·29 (2)°$, $V = 1571·4 (8)$ Å^3, $Z = 4$, $D_x = 1·96$, $D_m = 2·00$ Mg m^{-3}, λ(Mo $K\alpha) = 0·71073$ Å, $\mu = 2·62$ mm^{-1}, $F(000) = 892$, $T = 293$ K, $R = 0·061$ for 1502 observed reflections. All the phenothiazine (PT) molecules exist as cation radicals and are nearly planar. Each Sb atom is coordinated by six Cl atoms to form a distorted octahedral configuration. In form (I), both cation radicals and anions form separate tetramers, while in form (II) they each make up one-dimensional infinite columns.

Introduction. Phenothiazine (PT) is known to have multiple oxidation states : cation radical, cation, and dication (Billon, 1962; Shine & Mach, 1965; Iida, 1971). The neutral PT molecule is known to be folded along the S···N axis with a dihedral angle of 153·3 (monoclinic; Bell, Blount, Briscoe & Freeman, 1968) and 158·5° (orthorhombic; McDowell, 1976). In several structural works on the 'weak' charge-transfer complexes, the PT molecule has been found to have a similar structure to that of the neutral species except for the dihedral angles cited above. Crystals of several kinds of PT complexes were obtained from PT and the strong Lewis acids $SbCl_3$ or $SbCl_5$. In the PT–$SbCl_6$ crystals, PT was found to exist as a cation radical, and to be almost planar with a dihedral angle of 175·8° (Uchida, Ito & Kozawa, 1983). It was concluded that PT is easily changed to a relatively stable cation radical in the complex with strong electron acceptors, and that it adopts a conjugated planar system including the central thiazine ring. This paper concerns the refined

structure of two polymorphic PT–SbCl₄ crystals, the preliminary results of which appeared in our previous communication giving the physical properties (Uchida, Seki, Ito, Nakano, Hoshizaki & Kozawa, 1986).

Experimental. Single crystals of (I) and (II) were obtained from the same 1,2-dichloroethane (DCE) solution of PT and $SbCl_3$ in the molar ratio 1:2. Crystals of (II) were also obtained from a DCE solution of PT and $SbCl_5$ in the molar ratio 2:1. Single crystals of both (I) and (II) were black plates and the dimensions of the crystals used were $0.5 \times 0.1 \times 0.03$ mm (I) and $0.15 \times 0.12 \times 0.08$ mm (II). D_m was measured by flotation in an aqueous solution of K_2HgI_4 for (I) and in a bromoform/chloroform solution for (II). Rigaku AFC-5 four-circle diffractometer, $\theta/2\theta$ data collection, scan width $(1 + 0.5\tan\theta)°$ in θ, scan rate $4°$ min^{-1} in θ, $2\theta_{max} = 55$ (I) and $60°$ (II), range of hkl: 0 to 16, -17 to 17, -16 to 16 (I) and 0 to 17, 0 to 25, -10 to 9 (II); standard reflections $\bar{5}23$, $\bar{2}1\bar{6}$, $17\bar{6}$ (I) and $\bar{4}5\bar{1}$, $\bar{4}51$, $\bar{1}53$ (II), monitored every 100 measurements, intensity variations in F 2% (I) and 10% (II); corrected for linear decay only for (II). For (I) 7332 reflections measured, 7094 unique, $R_{int} = 0.02$, 4184 observed with $F_o > 3\sigma(F)$; for (II), 4936 reflections measured, 4595 unique, $R_{int} = 0.021$, 1502 observed with $F_o > 3\sigma(F)$; lattice parameters determined from 24 reflections $(27.3 < 2\theta < 30.3°)$ for (I) and 20 reflections $(17.7 < 2\theta < 22.3°)$ for (II); correction for Lorentz and polarization effects, no correction for absorption for (I) and (II).

Structure solved by heavy-atom method for (I) and by direct methods using MULTAN84 (Main, Germain & Woolfson, 1984) for (II). Block-diagonal least-squares refinements were employed. The quantity minimized was $\sum w(|F_o| - |F_c|)^2$, and $w = 1$ for both (I) and (II). Initial positions of H atoms were calculated. Parameters refined: atomic coordinates for all atoms, anisotropic temperature factors for non-H atoms; isotropic temperature factors for H atoms were fixed at $B_{iso} = 4.0$ Å². (I): $R = 0.061$, $wR = 0.074$, $S = 3.82$, $(\Delta/\sigma)_{max} = 0.15$ for non-H atoms, $\Delta\rho_{min} = -2.8$, $\Delta\rho_{max} = 2.3$ e Å⁻³ near the Sb atom. (II): $R = 0.060$, $wR = 0.069$, $S = 4.54$, $(\Delta/\sigma)_{max} = 0.15$ for non-H atoms, $\Delta\rho_{min} = -2.2$, $\Delta\rho_{max} = 1.0$ e Å⁻³ near the Sb atom.

Scattering factors and anomalous-dispersion factors for Sb, Cl and S were taken from International Tables for X-ray Crystallography (1974). The calculations were carried out using a PANAFACOM U-1200II computer with the Rigaku RASA-5P package and a HITAC M-680H at the Computer Centre of the University of Tokyo with the UNICS (Sakurai, 1967) program system.

Table 1. *Fractional coordinates and equivalent isotropic thermal parameters with e.s.d.'s in parentheses*

$$B_{eq} = (4/3)\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
(I)				
Sb(1)	0·0368 (1)	0·2071 (1)	0·0918 (1)	2·77 (3)
Sb(2)	0·3518 (1)	0·1040 (1)	0·1648 (1)	3·24 (3)
Cl(1)	−0·1580 (4)	0·1567 (4)	0·1218 (4)	4·5 (2)
Cl(2)	−0·0286 (4)	0·3213 (4)	−0·0196 (4)	4·3 (2)
Cl(3)	0·0980 (4)	0·0144 (4)	0·1803 (4)	4·2 (2)
Cl(4)	0·1466 (4)	0·3781 (4)	0·3035 (4)	4·9 (2)
Cl(5)	0·4281 (5)	0·2659 (4)	0·3739 (4)	5·1 (2)
Cl(6)	0·4158 (4)	−0·0167 (4)	0·2667 (5)	5·7 (2)
Cl(7)	0·2643 (4)	0·2646 (4)	0·0478 (3)	3·9 (1)
Cl(8)	0·5436 (4)	0·1660 (4)	0·1410 (4)	5·3 (2)
S(A)	0·0608 (4)	0·2117 (4)	0·4733 (4)	4·1 (2)
N(A)	0·0023 (10)	0·1211 (9)	0·6696 (10)	3·0 (4)
C(1A)	−0·0799 (13)	0·1622 (11)	0·6076 (11)	3·0 (5)
C(2A)	−0·1854 (14)	0·1572 (13)	0·6342 (13)	3·9 (5)
C(3A)	−0·2707 (15)	0·1967 (14)	0·5736 (14)	4·2 (6)
C(4A)	−0·2513 (16)	0·2412 (14)	0·4816 (14)	4·5 (6)
C(5A)	−0·1507 (16)	0·2453 (14)	0·4560 (14)	4·2 (6)
C(6A)	−0·0636 (13)	0·2058 (11)	0·5139 (11)	3·0 (5)
C(7A)	0·1445 (13)	0·1530 (11)	0·5640 (12)	3·2 (5)
C(8A)	0·2515 (13)	0·1482 (13)	0·5495 (14)	4·0 (5)
C(9A)	0·3235 (14)	0·1021 (13)	0·6213 (15)	4·1 (6)
C(10A)	0·2859 (14)	0·0645 (13)	0·7092 (14)	3·9 (6)
C(11A)	0·1819 (14)	0·0702 (12)	0·7220 (13)	3·7 (5)
C(12A)	0·1076 (13)	0·1165 (11)	0·6535 (12)	3·0 (4)
S(B)	0·5677 (4)	0·5128 (4)	0·3021 (4)	4·4 (2)
N(B)	0·6522 (10)	0·6250 (9)	0·1339 (10)	3·1 (4)
C(1B)	0·7277 (12)	0·5766 (10)	0·1901 (12)	2·8 (4)
C(2B)	0·8376 (14)	0·5811 (12)	0·1725 (14)	3·6 (5)
C(3B)	0·9139 (14)	0·5340 (13)	0·2280 (14)	4·0 (5)
C(4B)	0·8857 (16)	0·4819 (14)	0·3087 (15)	4·6 (6)
C(5B)	0·7818 (15)	0·4775 (13)	0·3284 (15)	4·1 (6)
C(6B)	0·7006 (13)	0·5243 (11)	0·2739 (12)	3·2 (5)
C(7B)	0·4941 (13)	0·5774 (13)	0·2146 (13)	3·5 (5)
C(8B)	0·3810 (15)	0·5796 (14)	0·2166 (15)	4·3 (6)
C(9B)	0·3188 (15)	0·6314 (15)	0·1497 (16)	5·0 (7)
C(10B)	0·3661 (15)	0·6811 (14)	0·0763 (14)	4·3 (6)
C(11B)	0·4779 (14)	0·6804 (12)	0·0748 (13)	3·7 (5)
C(12B)	0·5432 (12)	0·6265 (11)	0·1396 (12)	3·0 (5)
(II)				
Sb	0·2738 (1)	0·1902 (1)	0·3675 (2)	2·72 (2)
Cl(1)	0·1456 (4)	0·0996 (3)	0·1842 (7)	4·0 (1)
Cl(2)	0·1265 (4)	0·1953 (3)	0·5852 (6)	3·8 (1)
Cl(3)	0·3924 (4)	0·1009 (3)	0·5784 (8)	4·7 (2)
Cl(4)	0·4024 (4)	0·1808 (3)	0·1625 (7)	4·2 (1)
S	0·6339 (4)	0·2084 (3)	−0·0253 (7)	3·8 (1)
N	0·8802 (10)	0·1981 (8)	0·2660 (18)	2·7 (3)
C(1)	0·8186 (14)	0·1345 (9)	0·2038 (23)	2·8 (5)
C(2)	0·8772 (17)	0·0668 (12)	0·2724 (28)	4·4 (6)
C(3)	0·8202 (21)	−0·0008 (12)	0·2219 (30)	4·9 (7)
C(4)	0·7113 (15)	−0·0022 (12)	0·0932 (27)	4·1 (6)
C(5)	0·6511 (18)	0·0630 (12)	0·0211 (28)	4·5 (7)
C(6)	0·7046 (14)	0·1296 (10)	0·0740 (23)	2·9 (5)
C(7)	0·7229 (12)	0·2796 (9)	0·0824 (19)	2·0 (4)
C(8)	0·6816 (13)	0·3509 (11)	0·0401 (25)	3·4 (5)
C(9)	0·7529 (15)	0·4094 (11)	0·1258 (27)	3·8 (6)
C(10)	0·8647 (19)	0·3976 (12)	0·2539 (31)	4·9 (7)
C(11)	0·9030 (18)	0·3282 (10)	0·2942 (28)	4·1 (6)
C(12)	0·8370 (13)	0·2665 (9)	0·2135 (22)	2·3 (4)

Discussion. Final atomic positional parameters of (I) and (II) are listed in Table 1* and the atomic numbering and conformations of anions are shown in Fig. 1. An asymmetric unit of (I) contains two chemical formula units of the components, whose

* Lists of structure factors, anisotropic thermal parameters, bond angles of the SbCl₄ moieties and least-squares-planes data for the PT moieties have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52639 (26 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

two PT moieties are denoted by A and B, respectively.

The bond lengths and angles of three PT moieties are given in Table 2. There is no significant difference between corresponding bond lengths and angles in (IA) and (IB), and in (II), although their standard deviations are rather large. All three PT molecules are characterized by the following three features. (i) The S—C and N—C bond lengths are shorter than the corresponding values in the neutral molecule, which are 1·77 and 1·41 Å in the monoclinic crystal (M) (Bell, Blount, Briscoe & Freeman, 1968) and 1·76 and 1·40 Å in the orthorhombic crystal (O) (McDowell, 1976), respectively. (ii) The C—S—C angles are larger than those of the neutral PT molecule, which are 99·6 (M) and 100·9° (O). (iii) The dihedral angles between the planes of the benzene moieties are 178·7 (IA), 178·1 (IB) and 176·2° (II); hence the PT moieties are nearly planar while the neutral PT molecules have folded conformations with dihedral angles of 153·3 (M) and 158·5° (O). These characteristics observed for (I) and (II) are very similar to those in PT—SbCl$_6$ (Uchida, Ito & Kozawa, 1983), in which PT was proved spectroscopically to exist as a cation radical. In the previous communication, we proposed that PT(IA) and PT(IB) were in different electronic states according

Table 2. *Bond lengths (Å) and angles (°) of the three phenothiazine molecules* (IA, IB *and* II)

	(I) Triclinic A	(I) Triclinic B	(II) Monoclinic
S—C(6)	1·71 (2)	1·73 (2)	1·71 (2)
S—C(7)	1·73 (2)	1·71 (2)	1·72 (2)
N—C(1)	1·37 (2)	1·37 (2)	1·37 (2)
N—C(12)	1·36 (2)	1·35 (2)	1·36 (2)
C(1)—C(2)	1·40 (2)	1·40 (2)	1·43 (3)
C(1)—C(6)	1·43 (2)	1·43 (2)	1·43 (3)
C(2)—C(3)	1·38 (3)	1·35 (2)	1·40 (3)
C(3)—C(4)	1·43 (3)	1·40 (3)	1·37 (3)
C(4)—C(5)	1·34 (3)	1·34 (3)	1·41 (3)
C(5)—C(6)	1·38 (2)	1·39 (2)	1·37 (3)
C(7)—C(8)	1·38 (2)	1·39 (3)	1·39 (3)
C(7)—C(12)	1·43 (2)	1·42 (2)	1·45 (2)
C(8)—C(9)	1·40 (2)	1·37 (3)	1·39 (3)
C(9)—C(10)	1·42 (3)	1·40 (3)	1·41 (3)
C(10)—C(11)	1·34 (2)	1·37 (3)	1·34 (3)
C(11)—C(12)	1·40 (2)	1·40 (2)	1·40 (3)
C(6)—S—C(7)	104·8 (8)	104·4 (8)	105·4 (8)
C(1)—N—C(12)	127 (1)	128 (1)	123 (1)
N—C(1)—C(2)	118 (1)	121 (1)	116 (2)
N—C(1)—C(6)	122 (1)	121 (1)	126 (2)
C(2)—C(1)—C(6)	119 (1)	118 (1)	117 (2)
C(1)—C(2)—C(3)	120 (1)	122 (2)	120 (2)
C(2)—C(3)—C(4)	119 (2)	120 (2)	120 (2)
C(3)—C(4)—C(5)	120 (2)	120 (2)	122 (2)
C(4)—C(5)—C(6)	122 (2)	123 (2)	119 (2)
S—C(6)—C(1)	122 (1)	122 (1)	120 (1)
S—C(6)—C(5)	119 (1)	120 (1)	119 (1)
C(1)—C(6)—C(5)	119 (1)	118 (1)	122 (2)
S—C(7)—C(8)	117 (1)	118 (1)	118 (1)
S—C(7)—C(12)	122 (1)	123 (1)	122 (1)
C(8)—C(7)—C(12)	122 (1)	120 (2)	121 (2)
C(7)—C(8)—C(9)	119 (2)	120 (2)	118 (2)
C(8)—C(9)—C(10)	119 (2)	121 (2)	122 (2)
C(9)—C(10)—C(11)	121 (2)	119 (2)	119 (2)
C(10)—C(11)—C(12)	122 (2)	122 (2)	123 (2)
N—C(12)—C(7)	122 (1)	121 (1)	123 (1)
N—C(12)—C(11)	120 (1)	120 (1)	119 (2)
C(7)—C(12)—C(11)	117 (1)	118 (1)	117 (2)

to the bond lengths and angles within the thiazine rings (Uchida, Seki, Ito, Nakano, Hoshizaki & Kozawa, 1986). However, the results of the present refinement show no significant differences between the PT moieties of IA and IB.

The SbCl$_4^-$ ions constitute polyanions; they form segregated tetramers in (I) and infinite chains in (II). Each Sb atom in both (I) and (II) is surrounded by six Cl atoms forming a distorted octahedral configuration with Cl—Sb—Cl angles in the range 79·3–104·1° in (I) and 80·6–102·0° in (II). The Sb—Cl bond lengths in the anions are given in Table 3. The terminal bond lengths are in the range 2·380–2·423 Å in (I) and 2·382–2·403 Å in (II). The bridging Sb—Cl bond lengths about those Cl atoms with rectangular twofold coordination are in the range 2·455–3·499 Å in (I) and 2·522–3·263 Å in (II), and those for the threefold coordinated Cl atoms are in the range 3·093–3·172 Å in (I). These bond lengths are comparable with the values of other SbCl$_4$ moieties reported to date (Porter & Jacobson, 1970; Ensinger, Schwarz & Schmidt, 1982; Lipka, 1980; Lipka & Mootz, 1978; Trinh-Toan & Dahl, 1976).

The crystal structure of (I) is shown in Fig. 2. Both PT and SbCl$_4$ moieties exist as centrosymmetric tetramers. Two independent PT moieties are nearly

Fig. 1. Numbering scheme of (*a*) PT common to IA, IB and II, (*b*) SbCl$_4$ in (I) and (*c*) SbCl$_4$ in (II).

Table 3. *The* Sb—Cl *bond lengths* (Å) *in* (I) *and* (II)

(I)

Sb(1)—Cl(1)	2·455 (5)	Sb(2)—Cl(3)	3·093 (5)
Sb(1)—Cl(2)	2·400 (5)	Sb(2)—Cl(5)	2·380 (6)
Sb(1)—Cl(3)	3·172 (5)	Sb(2)—Cl(6)	2·399 (6)
Sb(1)—Cl(4)	2·423 (5)	Sb(2)—Cl(7)	3·075 (5)
Sb(1)—Cl(7)	2·922 (5)	Sb(2)—Cl(8)	2·398 (6)
Sb(1)—Cl(3')	3·125 (5)	Sb(2)—Cl(1')	3·499 (5)

(II)

Sb—Cl(1)	2·382 (5)	Sb—Cl(4)	2·522 (6)
Sb—Cl(2)	2·780 (5)	Sb—Cl(2'')	3·104 (5)
Sb—Cl(3)	2·403 (6)	Sb—Cl(4''')	3·263 (6)

Symmetry codes: (i) $-x$, $-y$, $-z$; (ii) x, $\frac{1}{2} - y$, $\frac{1}{2} + z$; (iii) x, $\frac{1}{2} - y$, $-\frac{1}{2} + z$.

planar as mentioned above and overlap in the order *BAAB*. The mean interplanar distances in the region of overlap are 3·34 Å between *A* and *A'* ($-x$, $-y$, $1 - z$) (N side overlap) and 3·30 Å between *A* and *B* (S side overlap). These values are comparable to that of graphite (3·35 Å) and indicate the existence of a rather strong interaction within a tetramer. Although the inter-tetramer interaction is considered to be very small because of a minute amount of molecular overlap between *B* and *B''* ($1 - x, 1 - y, -z$), the short contact of C(2*B*) and C(10*B''*) [3·36 (3) Å] suggests that there is still a weak interaction between tetramers.

In the tetramer of SbCl$_4^-$, the four Sb atoms are individually coordinated by six Cl atoms, and form edge-sharing octahedra. The tetramer of SbCl$_4^-$ has been reported in Et$_4$N$^+$.SbCl$_4^-$ (Ensinger, Schwarz & Schmidt, 1982) and [Fe(η^5-C$_5$H$_5$)(CO)$_2$Cl]$_4$(SbCl$_3$)$_4$ (Trinth-Toan & Dahl, 1976), which also have a cubane-like structure. A similar cubane-like structure was also found in TeCl$_4$ (Buss & Krebs, 1971). The SbCl$_4^-$ tetramer in (I), however, has a different structure. The octahedron of Sb(1) shares three edges with neighbouring octahedra and that of Sb(2) shares two edges (see Fig. 1). There are two kinds of Cl coordination, *i.e.* twofold and threefold coordinated Cl as mentioned above. However, each octahedron in the tetramers of the other structures reported to date shares its three edges with the neighbouring three octahedra; so all the bridge Cl atoms are threefold coordinated. Although a non-cubane-like condensed tetramer structure of SbBr$_4$ has recently been reported in tetrakis(2-amino-1,3,4-thiadiazolium)–Sb$_4$Br$_{16}$ (Antolini, Benedetti, Fabretti & Giusti, 1988) as another type of tetramer, it is also different from the structure of (I).

The crystal structure of (II) is shown in Fig. 3. PT and SbCl$_4$ moieties form infinite stacks and chains, respectively, elongated along the *c* axis. The PT moieties are nearly planar and overlap in a face-to-face manner. The mean interplanar distance in the region of overlap is 3·47 Å with almost constant spacing. This spacing, which is comparable to the van der Waals distance for neutral aromatic com-

pounds, is intermediate in value between those of the intra- and inter-dimeric cation radical pair in chlorpromazine–CuCl$_4$ (3·38 and 3·57 Å respectively) (Obata, Kawazura & Miyamae, 1984).

An octahedron of SbCl$_4^-$ in (II) shares two *cis* edges with two other neighbouring octahedra, and a skew infinite chain is formed in a zigzag manner. Such an infinite chain of SbCl$_4^-$ moieties is also reported in pyridinium–SbCl$_4$ (Porter & Jacobson, 1970) and some other metal tetrahalides (Wells, 1984), but is different from those in anilinium–SbCl$_4$ (Lipka, 1980) and diphenylammonium–SbCl$_4$ (Lipka & Mootz, 1978), and these three structures are different from each other.

The electrical resistivity of a single crystal of (II) shows little anisotropy ($\rho_{300K,\parallel c} = 2 \times 10^3$ Ω m, $\rho_{300K,\parallel b} = 1 \times 10^5$ Ω m) (Uchida, Seki, Ito, Nakano, Hoshizaki & Kozawa, 1986). The $\rho_{\parallel c}$ value is not as small as expected for a one-dimensional structure,
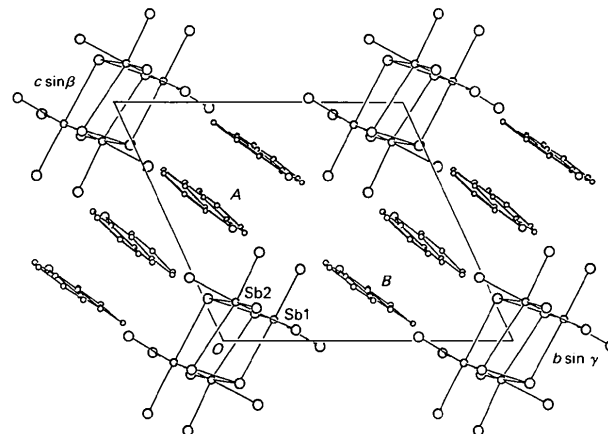
Fig. 2. Projection of the crystal structure of (I) along **a**.
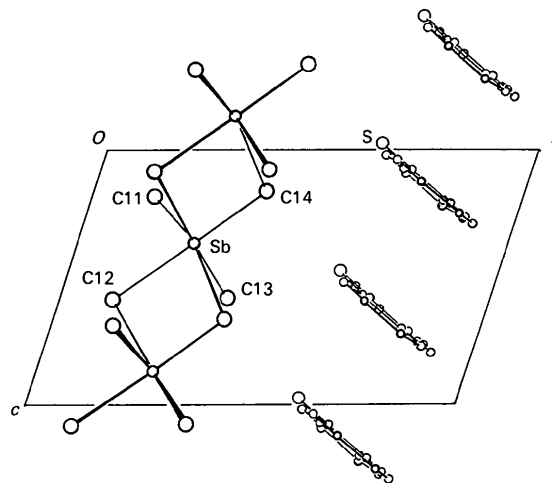
Fig. 3. Projection of the crystal structure of (II) along **b**.

because all the PT moieties exist as cation radicals possessing unit charge, and the mean interplanar distances are somewhat large compared with that of graphite (3·35 Å). The polarized absorption spectrum parallel to the c axis of a single crystal of (II) shows the characteristic peaks of the PT cation radical, and it also shows that the PT moieties have some mutual interaction, since there is a broad band around 1000 nm which one may assign to the charge-transfer band between PT cation radicals (Uchida, Seki, Ito, Nakano, Hoshizaki & Kozawa, 1986).

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Ten-Vertex Metallaborane Chemistry. Structure of 3-(OC2H5)-4,9-(PPh3)2-7,7-Cl(Ph2P-ortho-C6H4)-isonido-7-IrB9H5-10-.CH2Cl2

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Abstract. 7-Chloro-7,10-μ-(ortho-diphenylphosphinophenyl-P,C)-3-ethoxy-4,9-bis(triphenylphosphino)-7-irida-isonido-decaborane(5) dichloromethane solvate, $C_{56}H_{54}B_9ClIrOP_3.CH_2Cl_2$, $M_r = 1245·87$, monoclinic, $P2_1/n$, $a = 19·543$ (3), $b = 13·199$ (3), $c = 23·258$ (3) Å, $\beta = 107·93$ (1)°, $V = 5708$ Å³, $Z = 4$, $D_x = 1·45$ Mg m⁻³, $\lambda(Mo\ K\alpha) = 0·71069$ Å, $\mu = 2·480$ mm⁻¹, $F(000) = 2496$, room temperature, $R = 0·0349$, 5954 unique reflections with $F > 4·0\sigma(F)$.

The IrB9 cluster structure is of the rare isonido ten-vertex geometry that is formally derived from an eleven-vertex closo geometry by removal of a four-connected vertex.

Introduction. As part of our investigation of the chemical and thermolytic reactions of unsubstituted and phosphine-substituted nido-iridadecaboranes, we have determined the crystal and molecular structure of the title compound which was isolated in about 0·1% yield as the only identified metallaborane prod-

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