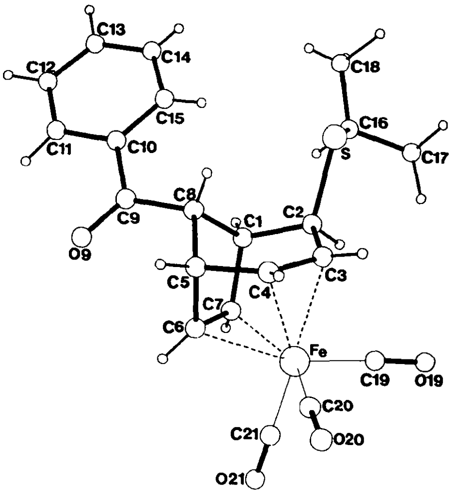

Fig. 1. Perspective view of the molecule, showing the labelling scheme.

bonds, *cf.* 1·81 (1) and 1·80 (1) Å in thiodilactic acid (Martuscelli, Mazzarella & Zagari, 1973) and 1·80 (1) Å in thiodiglycollic acid (Sukla, 1967).

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Bis(triphenylphosphine)iminium Pentadecacarbonyliodopentaosmate

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Abstract. $C_{51}H_{30}INO_{15}Os_5P_2$, monoclinic, $P2_1/c$, a = 18·176 (7), b = 18·540 (7), c = 17·317 (7) Å, β = 106·93 (8)°, U = 5583 Å^3, Z = 4, D_x = 2·423 g cm^{-3}, μ(Mo $K\alpha$) = 115·5 cm^{-1}. The structure was refined to an R of 0·040 for 3500 unique diffractometer data. The anion geometry may be derived from that of $Os_5(CO)_{16}$ by replacing one carbonyl of the equatorial $Os(CO)_4$ group by iodide, and shows a similar pattern of Os—C(O)···Os incipient bridge bonds.

Introduction. Purple crystals of $[(Ph_3P)_2N]^+$-$[IOs_5(CO)_{15}]^-$ were prepared by Eady & Malatesta (1977). 11 586 data were measured on a Nonius CAD-4 diffractometer with graphite-monochromated Mo $K\alpha$ radiation and a crystal 0·06 × 0·13 × 0·20 mm. Lp and numerical absorption corrections were applied, and equivalent reflexions averaged to give 3500 unique reflexions with $F > 6\sigma(F)$ based on counting statistics. Os and I atoms were located by multisolution $\sum_2$ sign expansion, and P, C, N and O from successive difference syntheses. The structure was refined by blocked-cascade least squares (Rivera & Sheldrick, 1978) with anisotropic Os, I and P, complex neutral-atom scattering factors and weights $w = 1/\sigma^2(F)$ to $R' = \sum w^{1/2}\Delta/\sum w^{1/2}|F_o|$ = 0·033 and R = 0·040. H atoms were not included. Final atomic coordinates and thermal parameters are given in Table 1, with bond lengths and angles in Tables 2, 3 and 4.*

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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 33705 (23 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Atom coordinates* (×10⁴) *and isotropic temperature factors* (Å² ×10³)

	x	*y*	*z*	*U*
Os(1)	2836 (1)	4228 (1)	437 (1)	
Os(2)	1606 (1)	4416 (1)	1116 (1)	
Os(3)	2930 (1)	2731 (1)	152 (1)	
Os(4)	2504 (1)	3178 (1)	1480 (1)	
Os(5)	1496 (1)	3378 (1)	−45 (1)	
I(1)	4155 (1)	4500 (1)	25 (1)	
C(11)	2360 (10)	4200 (13)	−720 (14)	50 (6)
O(11)	2166 (7)	4281 (9)	−1408 (10)	68 (4)
C(12)	3531 (11)	4240 (13)	1499 (14)	57 (6)
O(12)	4008 (8)	4334 (10)	2120 (10)	77 (5)
C(13)	2718 (10)	5251 (13)	477 (13)	48 (6)
O(13)	2682 (7)	5859 (10)	483 (9)	38 (4)
C(21)	2100 (11)	5083 (13)	1969 (14)	49 (6)
O(21)	2408 (8)	5442 (10)	2465 (10)	50 (5)
C(22)	814 (11)	4105 (13)	1532 (14)	61 (7)
O(22)	353 (8)	3896 (8)	1803 (9)	68 (5)
C(23)	1031 (11)	5164 (13)	466 (13)	49 (6)
O(23)	712 (8)	5628 (10)	85 (10)	43 (4)
C(31)	2694 (11)	1772 (16)	103 (15)	71 (8)
O(31)	2529 (8)	1163 (11)	60 (10)	85 (5)
C(32)	3976 (11)	2670 (13)	592 (13)	59 (7)
O(32)	4645 (8)	2593 (9)	847 (9)	74 (5)
C(33)	3089 (11)	2795 (14)	−861 (14)	74 (7)
O(33)	3165 (8)	2861 (10)	−1509 (10)	56 (5)
C(41)	3439 (13)	2691 (14)	1935 (15)	74 (8)
O(41)	4039 (9)	2451 (10)	2303 (11)	96 (6)
C(42)	2496 (10)	3605 (13)	2441 (15)	58 (7)
O(42)	2553 (8)	3809 (9)	3114 (11)	82 (5)
O(43)	1645 (9)	1868 (12)	1749 (11)	65 (5)
C(43)	1949 (12)	2379 (15)	1638 (15)	64 (7)
C(51)	759 (11)	2896 (13)	232 (13)	61 (7)
O(51)	269 (9)	2607 (10)	465 (10)	87 (5)
C(52)	791 (12)	4042 (14)	−723 (15)	63 (7)
O(52)	380 (8)	4448 (10)	−1161 (10)	80 (5)
C(53)	1451 (12)	2747 (16)	−920 (17)	82 (8)
O(53)	1293 (9)	2399 (11)	−1502 (12)	95 (6)
N	7202 (8)	3553 (10)	4987 (10)	87 (5)
P(1)	7991 (3)	3147 (3)	5288 (3)	
C(111)	8625 (10)	3730 (12)	5972 (12)	40 (6)
C(112)	9375 (10)	3490 (13)	6405 (13)	54 (6)
C(113)	9908 (13)	3974 (17)	6894 (16)	85 (9)
C(114)	9697 (13)	4730 (16)	6961 (16)	83 (8)
C(115)	8954 (13)	4960 (15)	6496 (15)	77 (8)
C(116)	8408 (10)	4456 (13)	6035 (12)	54 (6)
C(121)	8452 (9)	2912 (12)	4526 (12)	44 (6)
C(122)	8282 (12)	2279 (16)	4122 (16)	79 (8)
C(123)	8640 (14)	2104 (16)	3505 (17)	93 (9)
C(124)	9042 (11)	2639 (15)	3259 (15)	75 (8)
C(125)	9211 (12)	3298 (16)	3626 (17)	94 (9)
C(126)	8898 (11)	3455 (14)	4308 (14)	68 (7)
C(131)	7914 (10)	2320 (12)	5822 (12)	45 (6)
C(132)	7233 (10)	2235 (13)	6047 (13)	53 (6)
C(133)	7143 (12)	1586 (15)	6427 (15)	70 (7)
C(134)	7732 (13)	1076 (14)	6648 (15)	75 (8)
C(135)	8375 (12)	1212 (14)	6418 (14)	64 (7)
C(136)	8489 (10)	1828 (14)	6005 (13)	55 (6)
P(2)	6416 (3)	3549 (3)	4302 (3)	
C(211)	5674 (9)	3809 (11)	4745 (12)	39 (5)
C(212)	5895 (10)	4066 (13)	5534 (14)	53 (6)
C(213)	5308 (12)	4298 (15)	5925 (15)	82 (8)
C(214)	4598 (11)	4250 (14)	5465 (15)	69 (7)
C(215)	4343 (11)	4005 (13)	4678 (14)	55 (6)
C(216)	4901 (10)	3816 (12)	4266 (13)	55 (6)
C(221)	6419 (10)	4209 (11)	3541 (12)	38 (5)
C(222)	5846 (11)	4216 (14)	2815 (14)	60 (7)
C(223)	5849 (12)	4778 (15)	2261 (15)	68 (7)
C(224)	6385 (12)	5305 (14)	2388 (14)	67 (7)
C(225)	7009 (13)	5250 (15)	3140 (16)	77 (8)
C(226)	7018 (11)	4718 (14)	3718 (14)	64 (7)
C(231)	6129 (10)	2682 (12)	3837 (12)	46 (6)
C(232)	5706 (10)	2208 (14)	4134 (14)	60 (7)
C(233)	5509 (12)	1543 (15)	3753 (15)	70 (7)
C(234)	5750 (12)	1351 (14)	3116 (16)	83 (8)
C(235)	6170 (11)	1811 (15)	2758 (15)	75 (7)
C(236)	6358 (11)	2503 (14)	3112 (14)	67 (7)

Table 2. *Bond lengths* (Å) *in the* $[IOs_5(CO)_{15}]^-$ *anion*

Os(1)–Os(2)	2·833 (5)	Os(4)–C(42)	1·848 (28)
Os(1)–Os(3)	2·832 (5)	Os(4)–C(43)	1·858 (28)
Os(1)–Os(4)	2·835 (5)	Os(5)–C(51)	1·787 (25)
Os(1)–Os(5)	2·815 (4)	Os(5)–C(52)	1·913 (24)
Os(1)–I(1)	2·740 (5)	Os(5)–C(53)	1·898 (32)
Os(2)–Os(4)	2·780 (5)	C(11)–O(11)	1·150 (29)
Os(2)–Os(5)	2·748 (5)	C(12)–O(12)	1·182 (25)
Os(3)–Os(4)	2·758 (5)	C(13)–O(13)	1·131 (32)
Os(3)–Os(5)	2·799 (4)	C(21)–O(21)	1·102 (28)
Os(4)–Os(5)	2·766 (4)	C(22)–O(22)	1·141 (30)
Os(1)–C(11)	1·939 (24)	C(23)–O(23)	1·136 (27)
Os(1)–C(12)	1·904 (22)	C(31)–O(31)	1·165 (35)
Os(1)–C(13)	1·912 (27)	C(32)–O(32)	1·174 (24)
Os(2)–C(21)	1·935 (23)	C(33)–O(33)	1·176 (33)
Os(2)–C(22)	1·879 (26)	C(41)–O(41)	1·179 (28)
Os(2)–C(23)	1·896 (23)	C(42)–O(42)	1·200 (33)
Os(3)–C(31)	1·825 (30)	C(43)–O(43)	1·141 (34)
Os(3)–C(32)	1·834 (20)	C(51)–O(51)	1·205 (31)
Os(3)–C(33)	1·862 (28)	C(52)–O(52)	1·170 (28)
Os(4)–C(41)	1·882 (25)	C(53)–O(53)	1·160 (36)

Table 3. *Bond angles* (°) *in the* $[IOs_5(CO)_{15}]^-$ *anion*

Os(2)–Os(1)–Os(3)	106·8 (1)	Os(1)–Os(4)–Os(2)	60·6 (1)
Os(2)–Os(1)–Os(4)	58·8 (1)	Os(1)–Os(4)–Os(3)	60·8 (1)
Os(3)–Os(1)–Os(4)	58·3 (1)	Os(2)–Os(4)–Os(3)	110·4 (1)
Os(2)–Os(1)–Os(5)	58·2 (1)	Os(1)–Os(4)–Os(5)	60·3 (1)
Os(3)–Os(1)–Os(5)	59·4 (1)	Os(2)–Os(4)–Os(5)	59·4 (1)
Os(4)–Os(1)–Os(5)	58·6 (1)	Os(3)–Os(4)–Os(5)	60·9 (1)
Os(2)–Os(1)–I(1)	160·2 (1)	Os(1)–Os(4)–C(41)	105·2 (9)
Os(3)–Os(1)–I(1)	91·9 (2)	Os(2)–Os(4)–C(41)	152·8 (8)
Os(4)–Os(1)–I(1)	131·0 (1)	Os(3)–Os(4)–C(41)	76·5 (9)
Os(5)–Os(1)–I(1)	140·6 (1)	Os(5)–Os(4)–C(41)	136·9 (8)
Os(2)–Os(1)–C(11)	105·3 (7)	C(12)–Os(4)–C(41)	79·0 (10)
Os(3)–Os(1)–C(11)	79·9 (8)	Os(1)–Os(4)–C(42)	109·8 (9)
Os(4)–Os(1)–C(11)	121·7 (8)	Os(2)–Os(4)–C(42)	72·1 (8)
Os(5)–Os(1)–C(11)	65·9 (7)	Os(3)–Os(4)–C(42)	163·3 (6)
I(1)–Os(1)–C(11)	84·0 (7)	Os(5)–Os(4)–C(42)	128·7 (7)
Os(2)–Os(1)–C(12)	88·8 (8)	C(41)–Os(4)–C(42)	94·1 (11)
Os(3)–Os(1)–C(12)	97·1 (8)	Os(1)–Os(4)–C(43)	150·1 (7)
Os(4)–Os(1)–C(12)	66·2 (8)	Os(2)–Os(4)–C(43)	112·5 (8)
Os(5)–Os(1)–C(12)	124·5 (8)	Os(3)–Os(4)–C(43)	100·4 (9)
I(1)–Os(1)–C(12)	82·2 (8)	Os(5)–Os(4)–C(43)	90·6 (8)
C(11)–Os(1)–C(12)	165·8 (9)	C(41)–Os(4)–C(43)	91·1 (11)
Os(2)–Os(1)–C(13)	75·7 (8)	C(42)–Os(4)–C(43)	93·5 (12)
Os(3)–Os(1)–C(13)	172·3 (7)	Os(1)–Os(5)–Os(2)	61·2 (1)
Os(4)–Os(1)–C(13)	127·7 (8)	Os(1)–Os(5)–Os(3)	60·6 (1)
Os(5)–Os(1)–C(13)	118·0 (6)	Os(2)–Os(5)–Os(3)	110·1 (1)
I(1)–Os(1)–C(13)	86·7 (8)	Os(1)–Os(5)–Os(4)	61·1 (1)

Table 3 (*cont.*)

C(11)–Os(1)–C(13)	92·5 (10)	Os(2)–Os(5)–Os(4)	60·6 (1)
C(12)–Os(1)–C(13)	90·2 (10)	Os(3)–Os(5)–Os(4)	59·4 (1)
Os(1)–Os(2)–Os(4)	60·7 (1)	Os(1)–Os(5)–C(51)	148·3 (7)
Os(1)–Os(2)–Os(5)	60·6 (1)	Os(2)–Os(5)–C(51)	93·1 (9)
Os(4)–Os(2)–Os(5)	60·0 (1)	Os(3)–Os(5)–C(51)	119·8 (8)
Os(1)–Os(2)–C(21)	98·5 (8)	Os(4)–Os(5)–C(51)	91·0 (7)
Os(4)–Os(2)–C(21)	104·0 (7)	Os(1)–Os(5)–C(52)	100·9 (8)
Os(5)–Os(2)–C(21)	157·6 (6)	Os(2)–Os(5)–C(52)	83·8 (9)
Os(1)–Os(2)–C(22)	155·0 (7)	Os(3)–Os(5)–C(52)	141·6 (8)
Os(4)–Os(2)–C(22)	97·2 (8)	Os(4)–Os(5)–C(52)	144·2 (7)
Os(5)–Os(2)–C(22)	99·5 (8)	C(51)–Os(5)–C(52)	93·8 (11)
C(21)–Os(2)–C(22)	98·1 (11)	Os(1)–Os(5)–C(53)	114·5 (9)
Os(1)–Os(2)–C(23)	102·1 (8)	Os(2)–Os(5)–C(53)	173·3 (9)
Os(4)–Os(2)–C(23)	157·5 (7)	Os(3)–Os(5)–C(53)	69·8 (7)
Os(5)–Os(2)–C(23)	99·7 (8)	Os(4)–Os(5)–C(53)	122·8 (8)
C(21)–Os(2)–C(23)	92·3 (10)	C(51)–Os(5)–C(53)	92·5 (12)
C(22)–Os(2)–C(23)	95·8 (10)	C(52)–Os(5)–C(53)	92·4 (12)
Os(1)–Os(3)–Os(4)	60·9 (1)	Os(1)–C(11)–O(11)	167·8 (19)
Os(1)–Os(3)–Os(5)	60·0 (1)	Os(1)–C(12)–O(12)	170·2 (21)
Os(4)–Os(3)–Os(5)	59·7 (1)	Os(1)–C(13)–O(13)	176·2 (18)
Os(1)–Os(3)–C(31)	159·5 (8)	Os(2)–C(21)–O(21)	177·0 (19)
Os(4)–Os(3)–C(31)	102·2 (9)	Os(2)–C(22)–O(22)	177·3 (16)
Os(5)–Os(3)–C(31)	102·3 (8)	Os(2)–C(23)–O(23)	177·3 (19)
Os(1)–Os(3)–C(32)	95·5 (8)	Os(3)–C(31)–O(31)	178·6 (11)
Os(4)–Os(3)–C(32)	100·4 (9)	Os(3)–C(32)–O(32)	175·9 (18)
Os(5)–Os(3)–C(32)	153·3 (7)	Os(3)–C(33)–O(33)	176·9 (20)
C(31)–Os(3)–C(32)	99·3 (11)	Os(4)–C(41)–O(41)	171·1 (23)
Os(1)–Os(3)–C(33)	97·8 (9)	Os(4)–C(42)–O(42)	171·3 (18)
Os(4)–Os(3)–C(33)	157·5 (8)	Os(4)–C(43)–O(43)	176·2 (20)
Os(5)–Os(3)–C(33)	104·6 (7)	Os(5)–C(51)–O(51)	175·2 (21)
C(31)–Os(3)–C(33)	96·7 (12)	Os(5)–C(52)–O(52)	177·1 (22)
C(32)–Os(3)–C(33)	88·2 (11)	Os(5)–C(53)–O(53)	168·3 (20)

Table 4. *Selected bond lengths* (Å) *and angles* (°) *in the* $(Ph_3P)_2N^+$ *cation*

N–P(1)	1·570 (18)	N–P(2)	1·568 (16)
P(1)–C(111)	1·762 (21)	P(2)–C(211)	1·801 (23)
P(1)–C(121)	1·810 (25)	P(2)–C(221)	1·800 (24)
P(1)–C(131)	1·817 (25)	P(2)–C(231)	1·805 (24)
P(1)–N–P(2)	142·3 (13)		
N–P(1)–C(111)	106·5 (11)	N–P(2)–C(211)	107·8 (10)
N–P(1)–C(121)	116·5 (10)	N–P(2)–C(221)	110·7 (10)
N–P(1)–C(131)	112·0 (10)	N–P(2)–C(231)	115·1 (10)
C(111)–P(1)–C(121)	106·7 (11)	C(211)–P(2)–C(221)	107·0 (10)
C(111)–P(1)–C(131)	107·9 (10)	C(211)–P(2)–C(231)	105·8 (10)
C(121)–P(1)–C(131)	106·8 (11)	C(221)–P(2)–C(231)	110·0 (11)
P(1)–C(111)–C(112)	120·1 (18)	P(2)–C(211)–C(212)	118·1 (14)
P(1)–C(111)–C(116)	119·5 (13)	P(2)–C(211)–C(216)	119·6 (17)
P(1)–C(121)–C(122)	120·0 (19)	P(2)–C(221)–C(222)	120·7 (17)
P(1)–C(121)–C(126)	116·5 (18)	P(2)–C(221)–C(226)	117·7 (15)
P(1)–C(131)–C(132)	116·0 (16)	P(2)–C(231)–C(232)	121·8 (19)
P(1)–C(131)–C(136)	121·3 (18)	P(2)–C(231)–C(236)	117·7 (18)

Discussion. The anion (Fig. 1) consists of a trigonal bipyramidal cluster of five $Os(CO)_3$ groups, with a terminal I atom attached to one of the equatorial Os atoms. The Os–I distance of 2·740 (5) Å is close to the value of 2·749 (5) Å in the Os–I–Os bridge of $H_3Os_4(CO)_{12}I$ (Johnson, Lewis, Raithby, Sheldrick, Wong & McPartlin, 1978), although the latter Os–I bond might have been expected to be weaker and longer. This suggests that some of the negative charge in the anion is localized on the terminal I, weakening the Os–I bond. The $[IOs_5(CO)_{15}]^-$ structure may be derived from that of $Os_5(CO)_{16}$ (Reichert & Sheldrick, 1977) by replacing a carbonyl by I on the Os atom with highest formal oxidation state or 'electron connectivity' (Eady *et al.*, 1978). Iodide substitution lowers the electron connectivity, so the observed structure is consistent with the hypothesis that the most stable product is the one with the smallest imbalance of electron connectivities. The relatively long Os–Os bonds involving Os(1) in $Os_5(CO)_{16}$ are reduced by about 0·05 Å in the iodide; Eady *et al.* (1978) observed that Os–Os bonds are normally longer when the difference in electron connectivities is greater.

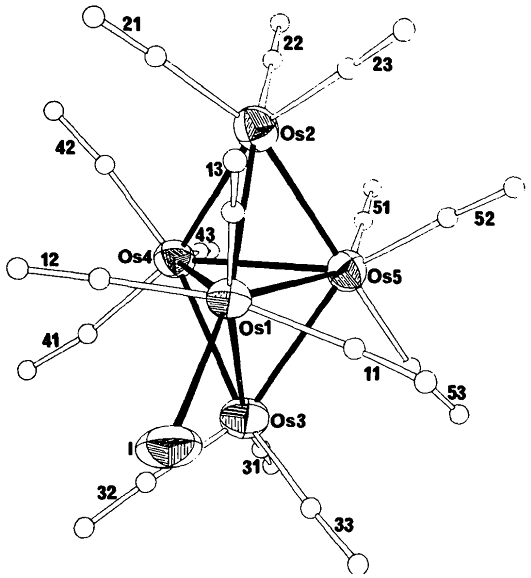

Fig. 1. The $[IOs_5(CO)_{15}]^-$ anion, showing the labelling scheme and 50% probability thermal ellipsoids for the osmium atoms.

There are four short Os···C interactions, all involving relatively bent carbonyls: Os(5)···C(11) 2·69 Å, Os(1)–C(11)–O(11) 167·8 (19)°; Os(4)···C(12) 2·71 Å, Os(1)–C(12)–O(12) 170·2 (21)°; Os(3)···C(53) 2·79 Å, Os(5)–C(53)–O(53) 168·3 (20)°; Os(2)···C(42) 2·83 Å, Os(4)–C(42)–O(42) 171·3 (18)°. In each case these weak incipient bridge bonds tend to redistribute the bonding electrons so as to equalize the electron connectivities [Os(1) + 1, Os(2) and Os(3) −1, Os(4) and Os(5) 0] as discussed by Eady *et al.* (1978). The corresponding incipient bridge bonds are found in $Os_5(CO)_{16}$. The cation dimensions are normal (Table 4), with staggered PPh_3 groups and P–N–P 142·3 (13)°.

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Trichlorobis(triphenylphosphine oxide)antimony(III)

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Abstract. $SbCl_3[(C_6H_5)_3PO]_2$, monoclinic, $C2/c$ (No. 15); $a = 17{\cdot}296$ (2), $b = 12{\cdot}212$ (2), $c = 17{\cdot}140$ (3) Å, $\beta = 104{\cdot}84$ (2)°; $D_o = 1{\cdot}47$ (1), $D_c = 1{\cdot}488$ g cm^{-3}, $Z = 4$, $R = 0{\cdot}027$ for 2713 reflections [$I > 3\sigma(I)$]. Coordination around Sb is a nearly regular square pyramid with the O-coordinated triphenylphosphine oxide ligands in the *cis* position. There is disorder of the apical Cl atoms [refined population parameters are 0·503 (4) for the apical and 0·999 (5) for the Cl atom in the base].

Introduction. A number of interesting configurations in the complexes of Sb^{III} or Bi^{III} halides with organic oxobases, $(C_6H_5)_3PO$ and $(C_6H_5)_3AsO$, have been determined by crystal structure analysis (Lazarini, Golič & Pelizzi, 1975, 1976; Lazarini & Milićev, 1976) or suggested by vibrational spectroscopy (Milićev & Hadži, 1977). The crystal structure determination of $SbCl_3[(C_6H_5)_3PO]_2$ is reported here. The complex $SbBr_3[(C_6H_5)_3PO]_2$ is isomorphous, as shown by powder diffraction data and vibrational spectra (Milićev & Hadži, 1977).

Crystals of $SbCl_3[(C_6H_5)_3PO]_2$ were obtained by the method of Milićev & Hadži (1977). The unit-cell parameters were determined by single crystal diffractometry. A unique set of 5032 reflections was measured in the range $1{\cdot}5 < \theta < 30°$ on an Enraf–Nonius CAD-4 diffractometer with ω–2θ scan and Mo $K\alpha_1$ radiation [graphite monochromator, $\lambda = 0{\cdot}7107$ Å, $t = 20\,(1)$°C]. A crystal ground to a sphere ($r = 0{\cdot}2$ mm) was used for intensity measurements. 2713 reflections having $I > 3\sigma(I)$ were taken as observed. The intensities were corrected for Lorentz, polarization and absorption ($\mu = 11{\cdot}5$ cm^{-1}) effects.

The structure was solved from a Patterson summation and successive Fourier syntheses. Because of the stoichiometry an Sb as well as a Cl atom should be expected in a special position. Just one peak on the twofold axis belonging to the Sb was observed and an additional peak of half the intensity expected for the Cl atom was found in the general position. Consequently the Cl atom with a population parameter of 0·5 was placed in this position. Population parameters of both Cl atoms, positional parameters and anisotropic temperature factors of all non-hydrogen atoms were included in the refinement of the structure. The H-atom parameters were calculated and included in the structure factor calculation (with an isotropic temperature factor $U = 0{\cdot}07$ Å^2) but not refined. The function minimized in the least-squares procedure was $\sum w(|F_o| - |F_c|)^2$. The weighting scheme applied was as follows:

$$|F_o| < 35\colon w_F = (|F_o|/35);\qquad \sin\theta < 0{\cdot}22\colon w_S = (\sin\theta/0{\cdot}22)$$

$$35 \le |F_o| < 64\colon w_F = 1{\cdot}0;\qquad 0{\cdot}22 \le \sin\theta < 0{\cdot}40\colon w_S = 1{\cdot}0$$

$$|F_o| \ge 64\colon w_F = (64/|F_o|);\qquad \sin\theta \ge 0{\cdot}40\colon w_S = (0{\cdot}40/\sin\theta)$$

$$w = w_F\, w_S.$$

Final R values are $R = \sum||F_o| - |F_c||/\sum|F_o| = 0{\cdot}027$ and $R_w = [\sum w(F_o - F_c)^2/\sum wF_o^2]^{1/2} = 0{\cdot}031$ for

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