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

C(1*) is the centroid of the C(1)—C(5) ring, C(2*) is the centroid of the C(7)—C(11) ring.

Ti(1)—Cl(1)	2.279 (2)	Ti(2)—Cl(3)	2.282 (2)
Ti(1)—Cl(2)	2.295 (2)	Ti(2)—Cl(4)	2.292 (2)
Ti(1)—N(1)	2.121 (4)	Ti(2)—N(1)	2.128 (4)
Ti(1)—N(4)	2.148 (4)	Ti(2)—N(4)	2.112 (4)
Ti(1)—C(1)	2.327 (7)	Ti(2)—C(7)	2.336 (6)
Ti(1)—C(2)	2.321 (8)	Ti(2)—C(8)	2.313 (7)
Ti(1)—C(3)	2.308 (9)	Ti(2)—C(9)	2.308 (8)
Ti(1)—C(4)	2.317 (7)	Ti(2)—C(10)	2.327 (6)
Ti(1)—C(5)	2.367 (7)	Ti(2)—C(11)	2.373 (6)
Ti(1)—C(1*)	2.026	Ti(2)—C(2*)	2.014
N(1)—N(2)	1.225 (7)	N(4)—N(5)	1.222 (7)
N(2)—N(3)	1.136 (7)	N(5)—N(6)	1.123 (7)
Ti(1)···Ti(2)	3.524 (1)	N(1)···N(4)	2.383 (7)
Cl(1)—Ti(1)—Cl(2)	90.96 (7)	Cl(3)—Ti(2)—Cl(4)	90.83 (6)
Cl(1)—Ti(1)—N(1)	133.0 (1)	Cl(4)—Ti(2)—N(1)	134.3 (1)
Cl(1)—Ti(1)—N(4)	84.0 (1)	Cl(4)—Ti(2)—N(4)	84.5 (1)
Cl(2)—Ti(1)—N(4)	136.0 (1)	Cl(3)—Ti(2)—N(4)	134.9 (1)
Cl(2)—Ti(1)—N(1)	85.2 (1)	Cl(3)—Ti(2)—N(1)	84.3 (1)
Cl(1)—Ti(1)—C(1*)	114.4	Cl(3)—Ti(2)—C(2*)	112.1
Cl(2)—Ti(1)—C(1*)	111.2	Cl(4)—Ti(2)—C(2*)	111.2
N(1)—Ti(1)—C(1*)	110.6	N(1)—Ti(2)—C(2*)	112.7
N(4)—Ti(1)—C(1*)	110.6	N(4)—Ti(2)—C(2*)	111.3
Average	111.7	Average	111.8
N(1)—Ti(1)—N(4)	67.9 (2)	N(1)—Ti(2)—N(4)	68.3 (2)
Ti(1)—N(1)—N(2)	122.9 (3)	Ti(2)—N(1)—N(2)	124.9 (3)
Ti(1)—N(4)—N(5)	123.8 (3)	Ti(2)—N(4)—N(5)	124.5 (3)
Ti(1)—N(1)—Ti(2)	112.0 (2)	Ti(1)—N(4)—Ti(2)	111.7 (2)
N(1)—N(2)—N(3)	179.3 (5)	N(4)—N(5)—N(6)	179.6 (5)
N(1)···N(4)—N(5)	179.3 (3)	N(4)···N(1)—N(2)	177.9 (4)
N(4)—Ti(2)—N(1)—Ti(1)	2.0 (2)	Cl(4)—Ti(2)—N(1)—Ti(1)	−55.9 (3)
Cl(1)—Ti(1)—N(1)—Ti(2)	−59.6 (3)	C(1*)—Ti(1)—N(1)—Ti(2)	102.7 (2)
Cl(2)—Ti(1)—N(1)—Ti(2)	−146.5 (2)	C(2*)—Ti(2)—N(1)—Ti(1)	107.0 (2)
Cl(3)—Ti(2)—N(1)—Ti(1)	−141.5 (2)		

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Structure of Triphenyltin Chloride–Triphenylphosphine Oxide (1/1) Complex

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Abstract. Chloro(triphenyl)(triphenylphosphine oxide)tin, $[\mathrm{SnCl}\{(\mathrm{C_6H_5})_3\mathrm{PO}\}(\mathrm{C_6H_5})_3]$, $M_r = 663.76$, monoclinic, $P2_1/c$, $a = 10.671$ (1), $b = 11.777$ (2), $c = 25.494$ (1) Å, $\beta = 98.148$ (5)°, $V = 3171.5$ (7) Å^3, $Z = 4$, $D_x = 1.390\ \mathrm{g\ cm^{-3}}$, $\lambda(\mathrm{Mo}\ K\alpha) = 0.71073$ Å, $\mu = 9.67\ \mathrm{cm^{-1}}$, $F(000) = 1344$, $T = 298$ K, $R = 0.033$ for 3327 $[I \geq 3\sigma(I)]$ reflections. Triphenyltin chloride forms a molecular complex with triphenylphosphine oxide in which Sn—Cl = 2.470 (2) and Sn—O = 2.391 (4) Å. The geometry of Sn is a *trans*-$\mathrm{C_3SnClO}$ trigonal bipyramid.

Experimental. Triphenyltin chloride and triphenylphosphine were dissolved in equimolar amounts in chloroform; slow evaporation of the solvent furnished clear crystals of the complex. A crystal measuring approximately 0.14 × 0.22 × 0.25 mm was used in the structure analysis. Measurements were performed on an Enraf–Nonius diffractometer. Accurate unit-cell parameters were obtained from the 25 most intense reflections in the $13 \leq 2\theta \leq 15°$ thin shell. Intensity data were gathered to a Bragg angle of 50° ($0 \leq h \leq 12$, $0 \leq k \leq 14$, $-30 \leq l \leq 30$) by

Table 1. *Positional parameters and equivalent isotropic displacement parameters* (Å^2)

$$B_{eq} = (4/3)[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)].$$

	x	y	z	B_{eq}
Sn	0.19699 (3)	0.00038 (4)	0.15623 (1)	3.406 (6)
Cl	0.1401 (2)	−0.1616 (1)	0.20973 (6)	5.33 (4)
P	0.2593 (1)	0.2812 (1)	0.09021 (5)	3.00 (3)
O	0.2547 (3)	0.1594 (3)	0.1064 (1)	3.71 (8)
C(1)	0.3031 (5)	−0.0942 (5)	0.1077 (2)	3.7 (1)
C(2)	0.2690 (6)	−0.2040 (5)	0.0932 (2)	5.0 (1)
C(3)	0.3373 (7)	−0.2664 (6)	0.0611 (3)	7.2 (2)
C(4)	0.4392 (7)	−0.2219 (6)	0.0429 (3)	7.2 (2)
C(5)	0.4757 (7)	−0.1149 (7)	0.0569 (3)	7.1 (2)
C(6)	0.4093 (5)	−0.0520 (6)	0.0886 (3)	5.1 (1)
C(7)	0.2888 (5)	0.0945 (5)	0.2206 (2)	4.0 (1)
C(8)	0.4148 (7)	0.0783 (7)	0.2399 (3)	6.8 (2)
C(9)	0.4772 (9)	0.1459 (8)	0.2794 (3)	10.2 (3)
C(10)	0.414 (1)	0.2276 (8)	0.3014 (3)	11.6 (3)
C(11)	0.291 (1)	0.2465 (7)	0.2839 (3)	9.1 (3)
C(12)	0.2285 (7)	0.1803 (5)	0.2441 (3)	6.0 (2)
C(13)	0.0067 (5)	0.0393 (4)	0.1250 (2)	3.7 (1)
C(14)	−0.0277 (6)	0.0650 (5)	0.0717 (2)	4.5 (1)
C(15)	−0.1490 (6)	0.0973 (6)	0.0523 (3)	6.0 (2)
C(16)	−0.2381 (7)	0.1059 (7)	0.0852 (3)	7.3 (2)
C(17)	−0.2075 (6)	0.0795 (7)	0.1372 (3)	7.0 (2)
C(18)	−0.0868 (6)	0.0459 (6)	0.1568 (2)	5.1 (1)
C(19)	0.1697 (5)	0.3718 (4)	0.1272 (2)	3.2 (1)
C(20)	0.2145 (6)	0.4720 (5)	0.1497 (2)	5.1 (1)
C(21)	0.1433 (7)	0.5340 (6)	0.1812 (3)	6.3 (2)
C(22)	0.0290 (6)	0.4956 (6)	0.1901 (2)	6.3 (1)
C(23)	−0.0162 (6)	0.3959 (6)	0.1682 (3)	5.7 (2)
C(24)	0.0516 (5)	0.3350 (5)	0.1366 (2)	4.7 (1)
C(25)	0.1999 (4)	0.2970 (4)	0.0212 (2)	3.1 (1)
C(26)	0.2135 (5)	0.2076 (5)	−0.0124 (2)	4.1 (1)
C(27)	0.1712 (6)	0.2170 (6)	−0.0657 (2)	5.3 (2)
C(28)	0.1149 (6)	0.3130 (6)	−0.0861 (2)	5.4 (2)
C(29)	0.1001 (6)	0.4029 (6)	−0.0533 (2)	5.9 (2)
C(30)	0.1435 (6)	0.3962 (5)	0.0002 (2)	4.6 (1)
C(31)	0.4167 (5)	0.3376 (4)	0.1014 (2)	3.3 (1)
C(32)	0.4589 (5)	0.4188 (6)	0.0688 (2)	5.0 (1)
C(33)	0.5748 (6)	0.4720 (6)	0.0834 (3)	6.6 (2)
C(34)	0.6477 (6)	0.4421 (7)	0.1289 (3)	7.0 (2)
C(35)	0.6088 (6)	0.3608 (6)	0.1613 (3)	6.2 (2)
C(36)	0.4933 (5)	0.3083 (6)	0.1474 (3)	4.9 (1)

Table 2. *Selected bond distances* (Å) *and angles* (°)

Sn—Cl	2.470 (2)	Sn—O	2.391 (4)
Sn—C(1)	2.109 (6)	Sn—C7	2.103 (6)
Sn—C(13)	2.124 (6)	P—O	1.496 (4)
P—C(19)	1.789 (5)	P—C25	1.793 (5)
P—C(31)	1.791 (5)		
Cl—Sn—O	178.6 (1)	Cl—Sn—C(1)	96.3 (2)
Cl—Sn—C(7)	95.7 (2)	Cl—Sn—C(13)	94.4 (2)
O—Sn—C(1)	84.4 (2)	O—Sn—C(7)	82.8 (2)
O—Sn—C(13)	86.4 (2)	C(1)—Sn—C(7)	120.4 (2)
C(1)—Sn—C(13)	117.3 (2)	C(7)—Sn—C(13)	119.6 (2)
O—P—C(19)	112.7 (2)	O—P—C(25)	110.5 (2)
O—P—C(31)	112.3 (2)	C(19)—P—C(25)	108.3 (2)
C(19)—P—C(31)	104.7 (2)	C(25)—P—C(31)	108.1 (2)
Sn—O—P	156.8 (2)		

using the $\theta/2\theta$ scan technique. 5880 data were measured, of which 5244 were unique and 3327 satisfying the $I \geq 3\sigma(I)$ criterion were used in the refinement. Three reflections ($0,0,\overline{16}$, $1,1,\overline{17}$, 606) monitored hourly showed negligible decrease in intensity. The structure was solved by using *MULTAN* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982). An empirical (Fourier series) absorption correction (Walker & Stuart, 1983) was applied after the non-H atoms had been refined isotropically. The non-H atoms were anisotropically refined; 361 variables were refined. H atoms were placed at calculated positions (C—H = 0.95 Å, $B = 5$ Å^2) and were allowed to ride on their parent C atoms. The refinement, based on F, converged at $R = 0.033$, $wR = 0.038$ $\{w = [\sigma(F)^2 + (0.02F)^2 + 1]^{-1}\}$; $S = 0.447$; $\Delta/\sigma = 0.01$. The maximum and minimum $\Delta\rho$ were 0.34 (5) and −0.17 (5) e Å^{-3}. All computations were performed on a MicroVAX minicomputer with the *MolEN* (Fair, 1990) structure determination package. Scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV, Tables 2.2B and 2.3.1). The atomic coordinates are listed in Table 1;* selected bond distances and angles are listed in Table 2. The atom-labeling scheme is shown in Fig. 1.

Related literature. The Sn—Cl bond in triphenyltin chloride, which is 2.32 (2) Å (Bokii, Zakharova & Struchkov, 1970), has been lengthened to 2.470 (2) Å in the triphenylphosphine oxide complex. The coordination around Sn in the complex is similar to that in the 1,2-(diphenylphosphinyl)ethylene adducts (Pelizzi & Pelizzi, 1980*a*,*b*). The Sn—O—P angle in

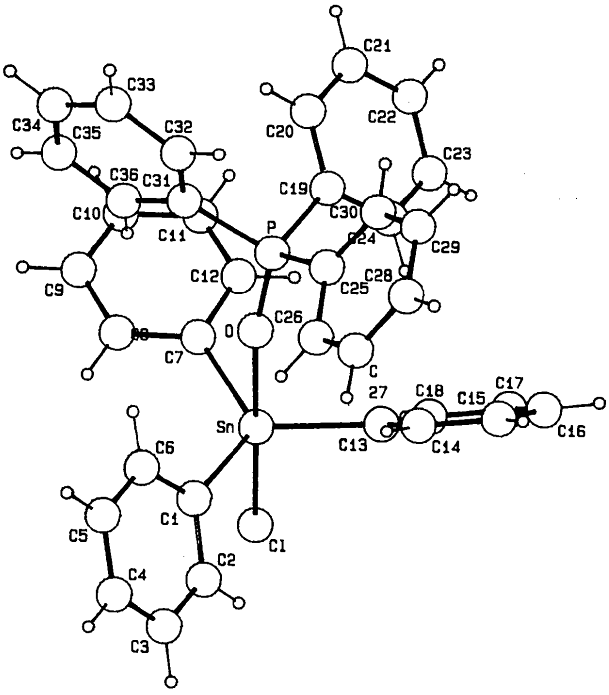

Fig 1. The labeling scheme for triphenyltin chloride–triphenylphosphine oxide.

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* Lists of structure factors, anisotropic thermal parameters, bond distances and angles, and H-atom positional parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55102 (41 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AS0567]

triphenyltin halide–phosphine oxide complexes is large, typically about 156° (Rheingold, Ng & Zuckerman, 1984).

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Carbonatobis(1,10-phenanthroline)cobalt(III) Perchlorate

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Abstract. $[\mathrm{Co(CO_3)(C_{12}H_8N_2)_2}](\mathrm{ClO_4})$, $M_r = 578.81$, monoclinic, $P2_1/n$, $a = 11.732\,(5)$, $b = 12.404\,(4)$, $c = 16.194\,(5)$ Å, $\beta = 108.80\,(3)°$, $V = 2231\,(1)$ Å^3, $Z = 4$, $D_x = 1.723$ Mg m^{-3}, $\lambda(\mathrm{Mo}\ K\alpha) = 0.71069$ Å, $\mu = 0.944$ mm^{-1}, $F(000) = 1176$, $T = 293$ K, $R = 0.063$, 2547 unique reflexions $[I \geq 2\sigma(I)]$. The cation has typical non-crystallographic twofold symmetry with the Co environment approximating a *cis* octahedron. There is a tendency for Co—N bonds *trans* to O atoms to be slightly longer than the *cis* bonds [1.936 (5), 1.951 (5) Å *cf* 1.922 (5), 1.931 (5) Å]; however, both coordinated carbonate O atoms are equidistant from Co [1.889 (4), 1.886 (4) Å].

Experimental. The compound was obtained during the synthesis of Co complexes of 2(1*H*)-pyridone by use of the electrochemical method proposed by Tuck (Habeeb, Tuck & Walters, 1978). The electrochemical oxidation of a Co anode in an acetonitrile solution (70 ml) containing 1,10-phenanthroline (0.25 g), 2(1*H*)-pyridone (0.20 g) and tetramethylammonium perchlorate (*ca* 10 mg) led to the formation of a brown solid. Crystals of the title compound, suitable for X-ray studies, were obtained by slow evaporation of the mother liquor at room temperature.

A crystal of dimensions 0.3 × 0.3 × 0.1 mm was used for data collection on a Rigaku AFC-6*S* diffractometer, with graphite-monochromated Mo $K\alpha$ radiation. Unit-cell dimensions were determined from the setting angles of 25 accurately centered reflexions $(12.4 \leq 2\theta \leq 28.3°)$. Reflexions were measured using the ω–2θ scan mode with ω-scan width $(1.26 + 0.30\tan\theta)°$ and scan speed of 8° min^{-1}, with up to two additional scans of weak reflexions $[I < 10\sigma(I)]$; $0 \leq h \leq 13$, $0 \leq k \leq 14$, $-18 \leq l \leq 18$, $0 \leq \theta \leq 25°$. 4354 reflexions were measured, of which 4140 were unique ($R_{\mathrm{int}} = 0.072$) and 2547 observed $[I \geq 2\sigma(I)]$. Intensity standards (025, 033, 132) measured every 150 reflexions showed no decline. Lp and absorption (maximum/minimum transmission 0.86/1.0) corrections were applied. *MITHRIL* (Gilmore, 1984) was used to solve the phase problem. All non-H atoms were found in Fourier maps, H atoms from ΔF synthesis. Full-matrix least-squares refinement was based on F using *TEXSAN* (Molecular Structure Corporation, 1985) crystallographic software. Final $R = 0.063$ $\{wR = 0.062$, $w = 1/[\sigma^2(F_o) + (0.03F_o)^2]\}$, $S = 2.01$. Anisotropic thermal parameters were refined for heavier atoms, isotropic for H atoms. Maximum fluctuation in the final ΔF map was in the range -0.41 to 0.71 e Å^{-3}. Maxi-

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