Related literature. The title compound was obtained as unreacted starting material in an attempt to form $[Mo_5O_{18}(Mo{\equiv}N{-}SiMe_3)]^{2-}$ by reaction with $Me_3SiN{=}PPh_3$. Dahlstrom, Zubieta, Neaves & Dilworth (1982) have reported a different monoclinic polymorph ($P2_1/n$) of the title compound. Several other salts have the same anion: Ph_4As^+ (Clegg, Sheldrick, Garner & Walton, 1982); $C_{22}H_{22}O_2P^+$ and $C_{22}H_{22}P^+$ (Arzoumanian, Baldy, Lai, Odreman, Metzger & Pierrot, 1985); $C_{12}H_{24}O_6.H_3O^+$ (Shoemaker, McAfee, Shoemaker & DeKock, 1986); $C_{20}H_{40}MoN_4S_8{}^+$ (Garner, Howlader, Mabbs, McPhail, Miller & Onan, 1978); $C_{39}H_{33}Mo_2O_4P_2^+$ (Riera, Ruiz, Villafane, Jeannin & Bois, 1988); $C_{12}H_{24}O_6.K^+.H_2O$ (Nagano & Sasaki, 1979); $C_{12}H_{37}N_9P_3^+$ (Allcock, Bissell & Shawl, 1973); and $C_{14}H_{21}ClN_2Rh^+$ (Zhang, Ozawa, Hayashi & Isobe, 1989).

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Structure of Tribenzyl(thiocyanato-*N*)(triphenylarsine oxide-*O*)tin

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Abstract. $[Sn(NCS)(C_{18}H_{15}AsO)(C_7H_7)_3]$, M_r = 772.42, triclinic, $P\bar{1}$, a = 10.099 (2), b = 11.4014 (8), c = 15.682 (3) Å, α = 95.145 (9), β = 92.72 (2), γ = 98.29 (1)°, V = 1776.3 (5) Å^3, Z = 2, D_x = 1.444 g cm^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, μ = 17.30 cm^{-1}, F(000) = 780, T = 300 K, R = 0.030 for 4221 reflections. The Sn atom is five-coordinate in a *trans*-C_3SnNO trigonal bipyramidal environment, being displaced out the equatorial plane by 0.030 (1) Å in the direction of the N occupant.

Experimental. An equimolar amount of triphenylarsine oxide was added to an ethanol solution containing tribenzyltin isothiocyanate, which had been prepared *in situ* from the metathetical reaction between tribenzyltin chloride and potassium thiocyanate. Slow evaporation of the filtered solution furnished crystals of the molecular complex, m.p. 419–420 K. ^{119}Sn NMR in $CDCl_3$ (saturated solution): $\delta = -187.5$ p.p.m. relative to $(CH_4)_4Sn$. ^{13}C NMR in $CDCl_3$: $^1J(^{119}Sn{-}^{13}C)$ = 476.1 Hz. ^{1}H NMR in $CDCl_3$: $^2J(^{119}Sn{-}^1H)$ = 79.9 Hz.

A 0.22 × 0.22 × 0.29 mm crystal was used for the study on an Enraf–Nonius CAD-4 diffractometer. Unit-cell constants were calculated from the 25 strongest reflections in the θ = 13–15° range. Intensity measurements were obtained by using the ω–2θ scan method to $2\theta_{max}$ = 50° (collection range: h = 0 to 12, $k = -13$ to 13, $l = -18$ to 18). 4221 of the 5836 unique reflections (R_{int} = 0.012) obeyed the $I \geq 3\sigma(I)$ criterion. The data were corrected for decay (minimum/maximum correction = 1.0000/1.0281, average correction = 1.0127), which was monitored by three reflections hourly, and for absorption effects by using the ψ-scan data (minimum/maximum correction = 0.9495/0.9996, average correction = 0.9755). The solution by the heavy-atom method presented some difficulties as the Sn–Sn vector in the Patterson map was found to be the third largest, after the Sn–As vectors. Non-H atoms were refined

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Table 1. *Positional parameters and equivalent isotropic thermal 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	0.23721 (3)	0.46798 (3)	0.24947 (2)	3.919 (6)
As	−0.01315 (5)	0.18873 (4)	0.24081 (3)	3.76 (1)
S	0.5600 (2)	0.8499 (1)	0.2730 (1)	8.30 (5)
O	0.0779 (3)	0.3180 (3)	0.2289 (2)	5.15 (8)
N	0.4009 (5)	0.6354 (4)	0.2671 (4)	7.9 (2)
C1	0.3895 (5)	0.3562 (5)	0.2506 (3)	4.9 (1)
C2	0.4049 (4)	0.2930 (4)	0.3288 (3)	4.3 (1)
C3	0.4060 (5)	0.1710 (5)	0.3218 (4)	5.4 (1)
C4	0.4189 (6)	0.1112 (5)	0.3931 (4)	6.5 (1)
C5	0.4321 (6)	0.1699 (6)	0.4723 (4)	7.0 (2)
C6	0.4321 (6)	0.2894 (6)	0.4817 (4)	6.3 (1)
C7	0.4187 (5)	0.3501 (5)	0.4107 (4)	5.2 (1)
C8	0.1517 (5)	0.5344 (5)	0.3636 (4)	5.6 (1)
C9	0.0048 (5)	0.5384 (4)	0.3524 (3)	4.4 (1)
C10	−0.0851 (5)	0.4669 (5)	0.3947 (3)	5.1 (1)
C11	−0.2185 (6)	0.4695 (6)	0.3860 (4)	6.6 (2)
C12	−0.2678 (6)	0.5434 (6)	0.3350 (4)	7.5 (2)
C13	−0.1841 (6)	0.6154 (6)	0.2920 (4)	7.7 (2)
C14	−0.0475 (6)	0.6132 (5)	0.2994 (4)	6.2 (1)
C15	0.1774 (6)	0.5193 (5)	0.1268 (4)	6.0 (1)
C16	0.1972 (5)	0.4328 (4)	0.0536 (3)	4.6 (1)
C17	0.0928 (6)	0.3597 (6)	0.0098 (4)	7.2 (2)
C18	0.112 (1)	0.2797 (7)	−0.0564 (4)	10.7 (3)
C19	0.240 (1)	0.2719 (7)	−0.0804 (5)	10.7 (3)
C20	0.3434 (7)	0.3421 (7)	−0.0392 (4)	9.5 (2)
C21	0.3220 (6)	0.4210 (6)	0.0271 (4)	6.9 (2)
C22	0.4663 (5)	0.7244 (5)	0.2691 (4)	5.2 (1)
C23	−0.0438 (5)	0.1672 (4)	0.3578 (3)	4.2 (1)
C24	0.0637 (5)	0.1852 (5)	0.4174 (3)	5.1 (1)
C25	0.0412 (6)	0.1785 (6)	0.5026 (4)	6.4 (1)
C26	−0.0854 (7)	0.1536 (6)	0.5270 (4)	7.1 (2)
C27	−0.1917 (6)	0.1358 (6)	0.4694 (4)	7.0 (2)
C28	−0.1721 (6)	0.1430 (5)	0.3839 (3)	5.7 (1)
C29	0.0782 (5)	0.0646 (4)	0.1937 (3)	4.2 (1)
C30	0.1254 (6)	0.0730 (5)	0.1146 (4)	6.8 (1)
C31	0.2011 (7)	−0.0098 (7)	0.0824 (4)	8.4 (2)
C32	0.2301 (6)	−0.0975 (5)	0.1280 (5)	7.2 (2)
C33	0.1846 (6)	−0.1066 (5)	0.2068 (4)	6.5 (2)
C34	0.1070 (6)	−0.0264 (5)	0.2396 (3)	5.4 (1)
C35	−0.1803 (5)	0.1855 (4)	0.1779 (3)	3.9 (1)
C36	−0.2659 (5)	0.0806 (4)	0.1577 (3)	4.9 (1)
C37	−0.3874 (5)	0.0817 (5)	0.1138 (4)	5.9 (1)
C38	−0.4212 (5)	0.1842 (6)	0.0895 (4)	6.5 (1)
C39	−0.3369 (6)	0.2877 (5)	0.1088 (4)	6.8 (1)
C40	−0.2153 (5)	0.2900 (5)	0.1542 (4)	5.2 (1)

anisotropically; H atoms were located and refined with $B = 5$ Å^2. Full-matrix least-squares refinement based on F for the 514 variables converged to $R = 0.038$, $wR = 0.035$ $\{w = [\sigma(F)^2 + (0.02F)^2 + 1]^{-1}\}$; $S = 0.483$; $(\Delta/\sigma)_{max} = 0.42$ for all atoms; $(\Delta\rho)_{min/max} = -0.17/0.37$ (4) e Å^{-3}. Scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV, Tables 2.3.1 and 2.2B). Calculations were performed using the *MolEN* structure determination package (Delft Instruments, 1990) on a DEC MicroVAX minicomputer. Atomic coordinates are listed in Table 1* and selected bond distances and angles in Table 2; the structure is depicted in Fig. 1.

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

Sn—O	2.162 (3)	As—O	1.652 (3)
Sn—N	2.325 (5)	As—C23	1.907 (5)
Sn—C1	2.135 (6)	As—C29	1.912 (5)
Sn—C8	2.148 (6)	As—C35	1.908 (5)
Sn—C15	2.145 (6)		
O—Sn—N	176.7 (2)	O—As—C23	112.8 (2)
O—Sn—C1	92.8 (2)	O—As—C29	108.3 (2)
O—Sn—C8	91.2 (2)	O—As—C35	106.6 (2)
O—Sn—C15	88.2 (2)	C23—As—C29	109.2 (2)
N—Sn—C1	90.0 (2)	C23—As—C35	109.7 (2)
N—Sn—C8	88.7 (2)	C29—As—C35	110.2 (2)
N—Sn—C15	89.0 (3)		
C1—Sn—C8	123.2 (2)		
C1—Sn—C15	116.3 (2)		
C8—Sn—C15	120.4 (3)		

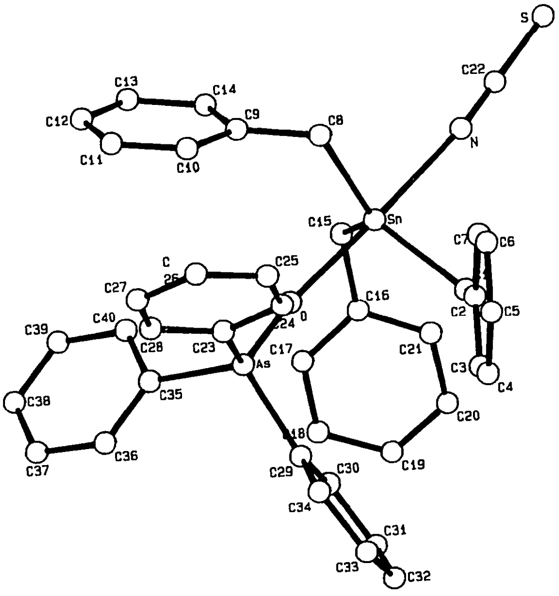

Fig. 1. Plot of the title compound with H atoms omitted.

Related literature. The triphenylarsine oxide ligand is involved in bent coordination to Sn [Sn—O—As = 160.1 (2)°]. The Sn—O—As angle and the Sn—O bond distance are similar to the corresponding values [145.9 (3)°; 2.219 (5), 2.205 (5) Å] found in the $[(p\text{-ClC}_6H_4)(C_6H_5)_2Sn.2(C_6H_5)_3AsO]^+$ cation (Ng, Chen, Kumar Das & Butcher, 1989). The Sn—N bond distance is slightly longer than that [2.15 (6) Å] in the five-coordinate *trans*-C_3SnNS trigonal bipyramidal trimethyltin isothiocyanate (Forder & Sheldrick, 1970), and that [2.22 (5) Å] in triphenyltin isothiocyanate (Domingos & Sheldrick, 1974). The dihedral angles between the $Sn/C_{methylene}/C_{ipso\text{-}phenyl}$ plane and the phenyl rings of the benzyl groups are 40.7 (4), 51.5 (3) and 77.9 (4)°, compared to the dihedral angle of 85° in carboxylate-bridged tribenzyltin acetate, for which nearly maximal σ–π hyperconjugation between the Sn—C_α bond and the π system of the benzyl group has been suggested (Holecek, Lycka, Handlir & Nadvornik, 1988). The

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* Lists of structure factors, anisotropic thermal parameters, calculated H-atom positional parameters, and complete bond lengths and bond angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55677 (51 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: OH1003]

five-coordinate geometry of the complex is retained in chloroform solution, as shown by the magnitudes of the one- and two-bond NMR coupling constants (Holecek, Lycka, Handlir & Nadvornik, 1988).

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Structure of (η^4-1,5-Cyclooctadiene)(1-ferrocenyl-1,3-butanedionato-κ^2O,O')rhodium(I)

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Abstract. [Rh($C_{14}H_{13}FeO_2$)(C_8H_{12})], $M_r = 480.2$, triclinic, $P\bar{1}$, $a = 11.658$ (1), $b = 13.001$ (1), $c = 15.251$ (2) Å, $\alpha = 70.621$ (8), $\beta = 68.538$ (8), $\gamma = 66.049$ (7)°, $V = 1919.8$ (3) Å^3, $Z = 4$, $D_m = 1.63$, $D_x = 1.66$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 16.15$ cm^{-1}, $T = 295$ K, $F(000) = 976$, final $R = 0.0419$ for 8290 observed reflections. Two crystallographically independent molecules A and B are present in the unit cell. Both molecules have an approximately square-planar configuration about the Rh atom, but only the cyclopentadienyl (Cp) rings of B approach an eclipsed conformation. The O—Rh—O plane does not pass through the middle of the C=C bonds of the boat-like 1,5-cyclooctadiene (cod) ligand.

Experimental. The title compound was prepared by stirring 0.24 g (0.9 mmol) of solid 1,3-dioxo-1-butylferrocene (FcA) (Hauser & Lindsay, 1957) with a solution of 0.22 g (0.45 mmol) of [$Rh_2Cl_2(cod)_2$] (Chatt & Venanzi, 1957) in a minimum amount of DMF for 5 min at room temperature. Excess water was added to the homogeneous reaction mixture and the precipitate was filtered, dissolved in ether then washed with water. Drying ($MgSO_4$) of the ethereal solution and removal of the solvent, followed by recrystallization from an acetone–water mixture, gave 0.26 g (61%) of well formed red crystals of [Rh(FcA)(cod)] suitable for X-ray analysis. The density was determined by flotation in sodium iodide solution. Data were collected on an Enraf–Nonius CAD-4F diffractometer with graphite-monochromated Mo $K\alpha$ radiation, using a crystal of size 0.19 × 0.11 × 0.08 mm. The $\omega/2\theta$-scan technique was used with variable scan width $\Delta\omega = (0.45 + 0.34\tan\theta)°$, a max. scan speed of 5.49° min^{-1} in ω and a max. scan time of 60 s per reflection. The unit-cell parameters were determined from a least-squares refinement of 25 reflections with $18 < \theta < 20°$. Data were corrected for Lorentz and polarization effects, as well as for absorption (North, Phillips & Mathews, 1968) with min. and max. correction factors of 0.970 and 0.999, respectively. Three control reflections were measured every 3600 s of X-ray exposure time and varied from the initial value by less than 2%. Intensity data were corrected for this decay. The intensities of 11 384 reflections in the index range $0 < h < 16$, $-18 < k < 18$, $-21 < l < 21$ with $0.07 < \sin\theta/\lambda < 0.7$ Å^{-1}, were measured. Of 10 106 unique reflections ($R_{\rm int} = 0.0204$), 8291 were considered observed with $I > 3\sigma(I)$. The structure was solved by the heavy-atom method using *SHELX*86 (Sheldrick, 1990) and was subjected to least-squares refinement on F with 476 positional and anisotropic thermal parameters for all the non-H atoms using *SHELX*76 (Sheldrick, 1976). The H-atom positions were calculated by assuming C—H = 1.08 Å, and refined with an overall temperature factor. Neutral-atom scattering factors (Cromer & Mann, 1968) and anomalous-dispersion corrections

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