Structural Studies in Main-group Chemistry. Part VII.[1] Crystal Structure of *N*-Benzoyl-*N*-phenyl-*O*-(triphenylstannyl)hydroxylamine

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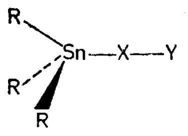
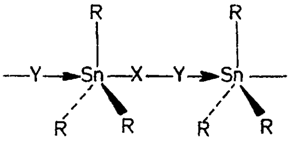
The crystal structure of the title compound has been determined from *X*-ray diffractometer data by Patterson and Fourier methods. Crystals are monoclinic, space group $P2_1/c$ with a = 12·278(3), b = 17·621(2), c = 15·411(2) Å, β = 128·37(2)°, Z = 4. Strong intramolecular carbonyl–tin co-ordination renders the tin atom five-co-ordinate with trigonal-bipyramidal geometry. The equatorial positions are occupied by two phenyl groups [mean Sn–C(Ph) 2·136 Å] and the hydroxylamino-oxygen (Sn–O 2·091 Å). The Sn–C(Ph) axial distance (2·176 Å) is significantly longer than the two equatorial bonds. In the hydroxylamine residue, the C–N bond distance indicates significant multiple-bond character, whilst the carbonyl bond distance is the same as in zwitterionic compounds. Rationalisations of observed spectroscopic data are made in the light of the derived structure. The sign of the electric-field gradient at the tin nucleus is deduced to be negative.

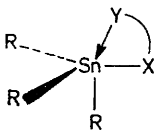
TRIORGANOTIN derivatives of potentially bidentate ligands R_3SnXY may *a priori* assume the three configurations (I)—(III). Essentially tetrahedrally co-ordinated tin is expected for weak or no interaction between the second donor site Y and the metal atom (I), whilst strong inter- or intra-molecular co-ordination would produce the five-co-ordinate configurations (II) and (III). Among the sparse structural data for tin–oxygen-bonded compounds there are examples of types (I) and (II), but there was no crystallographic confirmation of the existence of the intramolecularly co-ordinated type (III). Most triorganotin acetates are considered to possess the same structure as tribenzyltin acetate, in which fairly strong intermolecular carbonyl–tin co-ordination results in chain-like structure of type (II).[2] Tricyclohexyltin acetate on the other hand is monomeric in the crystal, and the intramolecular carbonyl–tin distance is such as to suggest negligible bonding interaction.[3] Previously we reported the preparation of and spectroscopic data for a number of triorganotin derivatives of *N*-benzoylhydroxylamines. The data suggested that these compounds possess monomeric structures of type (III), rather than the acetate structures (I) and (II). Here we report the structure of one of these compounds, *N*-benzoyl-*N*-phenyl-*O*-(triphenylstannyl)hydroxylamine.



(I)



(II)



(III)

EXPERIMENTAL

Crystal Data.—$C_{31}H_{25}NO_2Sn$, M = 561·9, Monoclinic, a = 12·278(3), b = 17·621(2), c = 15·411(2) Å, β = 128·37(2)°, Z = 4, F(000) = 1136. Space group $P2_1/c$ from systematic absences: $h0l$ for l odd, and $0k0$ for k odd. Mo-K_α radiation, λ = 0·71069 Å. μ(Mo-K_α) = 10·41 cm^{-1}.

Rod-like crystals, stable to air and to *X*-rays, were obtained by recrystallisation from benzene, and a crystal of dimensions 0·3 × 0·3 × 0·4 mm was mounted on a glass fibre. The space group was deduced from oscillation and zero- and first-layer Weissenberg photographs. The intensities of 3549 independent non-zero reflections were measured by use of a Hilger and Watts four-circle automatic diffractometer. Reflections with intensities $I < 3\sigma(I)$ were considered non-observed, and were not used further. Accurate unit-cell dimensions were obtained by least-squares refinement of data for *ca.* 20 reflections. Lorentz and polarisation corrections were applied, but none were made for absorption effects.

Determination and Refinement of the Structure.—The positional parameters of the tin atom in the asymmetric unit obtained from a Patterson synthesis were then used to phase the initial structure-factor calculation. A Fourier synthesis then enabled the positions of all but two phenyl-group carbon atoms of the non-hydrogen light atoms to be deduced. Block-diagonal least-squares refinement including all atoms was carried out using programmes written by Ahmed. With anisotropic thermal parameters for all atoms, convergence was reached with R 0·0490. A difference-Fourier synthesis at this stage revealed the positions of all hydrogen atoms. These were then included in the structure-factor calculation to give R 0·0455 after two cycles.

Final atomic co-ordinates for non-hydrogen atoms are in Table 1, anisotropic temperature factor parameters, in Table 2, and co-ordinates and isotropic thermal parameters for hydrogen atoms in Table 3.

The magnetically perturbed spectrum was obtained at the P.C.M.U. (Harwell) using a $BaSn^{119m}O_3$ source and an applied magnetic field of 6 Tesla. Final observed and calculated structure factors are in Supplementary Publication No. SUP 21107 (15 pp., 1 microfiche).†

DISCUSSION

The structure and numbering scheme of the atoms is shown in Figure 1. The geometry at tin is that of a

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† See Notice to Authors No. 7 in *J.C.S. Dalton*, 1973, Index issue.

[1] Part VI, P. G. Harrison, T. J. King, and J. A. Richards, *J.C.S. Dalton*, 1974, 1723.

[2] N. W. Alcock and R. E. Timms, *J. Chem. Soc.* (*A*), 1968, 1873.

[3] N. W. Alcock and R. E. Timms, *J. Chem. Soc.* (*A*), 1968, 1876.

TABLE 1

Fractional atomic co-ordinates, with standard deviations in parentheses

Atom	*x*/*a*	*y*/*b*	*z*/*c*
Sn(1)	0·24118(4)	0·17471(3)	0·20224(3)
N(1)	−0·0323(5)	0·1251(3)	−0·0009(4)
O(1)	0·0608(4)	0·1086(3)	0·1110(3)
O(2)	0·1390(4)	0·1859(3)	0·0169(3)
C(1)	0·0156(6)	0·1613(3)	−0·0446(4)
C(2)	−0·0695(6)	0·1731(4)	−0·1668(4)
C(3)	0·1329(9)	0·3396(4)	0·1175(7)
C(4)	−0·2207(7)	0·1257(5)	−0·3531(5)
C(5)	−0·2054(7)	0·1930(5)	−0·3895(5)
C(6)	−0·1216(8)	0·2512(5)	−0·3169(6)
C(7)	−0·0526(7)	0·2402(4)	−0·2044(5)
C(8)	−0·1734(6)	0·1107(4)	−0·0469(4)
C(9)	−0·2070(7)	0·0397(4)	−0·0289(6)
C(10)	−0·3423(8)	0·0258(5)	−0·0694(6)
C(11)	−0·4423(7)	0·0816(5)	−0·1259(6)
C(12)	−0·4075(7)	0·1507(5)	−0·1421(6)
C(13)	−0·2723(7)	0·1662(4)	−0·1033(5)
C(14)	0·2814(6)	0·1261(3)	0·3495(5)
C(15)	0·1850(7)	0·0856(4)	0·3478(5)
C(16)	0·2136(9)	0·0630(5)	0·4472(6)
C(17)	0·3377(8)	0·0812(4)	0·5486(5)
C(18)	−0·0526(7)	0·2402(4)	−0·2044(5)
C(19)	0·4082(7)	0·1444(5)	0·4527(5)
C(20)	0·4247(6)	0·1537(3)	0·2181(5)
C(21)	0·5295(7)	0·1062(4)	0·3015(6)
C(22)	0·6499(9)	0·0957(5)	0·3154(8)
C(23)	0·6701(8)	0·1304(5)	0·2461(8)
C(24)	0·5664(8)	0·1763(5)	0·1622(7)
C(25)	0·4462(7)	0·1884(4)	0·1486(6)
C(26)	0·1938(6)	0·2902(4)	0·2071(5)
C(27)	0·1239(9)	0·3396(4)	0·1175(7)
C(28)	0·1001(11)	0·4148(5)	0·1260(9)
C(29)	0·1290(9)	0·4405(5)	0·2207(9)
C(30)	0·1900(9)	0·3928(5)	0·3102(7)
C(31)	0·2219(7)	0·3189(5)	0·3033(6)

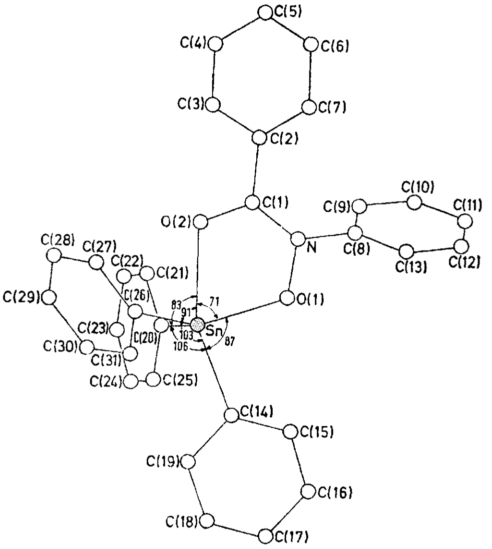


FIGURE 1 Structure and atomic numbering scheme showing bond angles (°) at the tin atom

trigonal bipyramid distorted by the chelation of the benzoylhydroxylamine residue. The equatorial positions are occupied by two phenyl groups and the hydroxylamino-oxygen. Intramolecular co-ordination of the carbonyl group oxygen atom to the tin *via* an axial site produces a five-membered $\overline{\mathrm{Sn{\cdot}O{\cdot}N{\cdot}C{:}O}}$ heterocyclic ring. The other axial site is occupied by the third phenyl group. The equatorial bond angles range from 111 to 125·5° (total 353·5°), the tin atom lying below the plane of the three bonded atoms. There appears to be

TABLE 2

Anisotropic thermal parameters ($\times 10^4$),* with standard deviations in parentheses

Atom	B_{11}	B_{22}	B_{33}	B_{23}	B_{13}	B_{12}
Sn(1)†	1011(4)	404(1)	605(2)	11(4)	826(5)	−143(6)
N(1)	105(6)	46(2)	48(3)	12(4)	67(7)	−6(6)
O(1)	122(5)	51(2)	55(3)	13(4)	71(6)	−27(5)
O(2)	122(5)	66(2)	60(3)	−5(5)	89(7)	−37(6)
C(1)	95(6)	34(3)	50(3)	−16(5)	56(8)	−2(6)
C(2)	95(6)	57(3)	57(4)	4(6)	90(8)	17(8)
C(3)	123(8)	57(3)	63(4)	−24(6)	104(10)	−6(8)
C(4)	134(9)	67(4)	61(5)	−6(7)	91(11)	9(9)
C(5)	131(9)	91(5)	57(4)	10(7)	96(11)	50(10)
C(6)	167(11)	78(5)	86(6)	40(8)	175(14)	32(11)
C(7)	157(9)	38(3)	71(4)	17(6)	129(11)	32(8)
C(8)	117(7)	41(3)	52(4)	(5)	89(9)	−3(7)
C(9)	141(9)	41(3)	89(5)	18(6)	113(12)	5(8)
C(10)	146(10)	54(4)	108(6)	14(8)	126(13)	−36(10)
C(11)	129(9)	73(4)	86(6)	7(8)	113(12)	−20(10)
C(12)	130(9)	65(4)	100(6)	27(8)	137(13)	36(9)
C(13)	157(9)	38(3)	71(4)	17(6)	129(10)	32(8)
C(14)	121(7)	37(2)	58(4)	22(5)	101(9)	17(7)
C(15)	135(8)	45(3)	67(5)	−3(6)	98(11)	−35(8)
C(16)	203(12)	68(4)	89(6)	6(8)	166(15)	−8(11)
C(17)	177(10)	53(3)	70(5)	3(7)	142(12)	−15(9)
C(18)	133(9)	64(4)	55(4)	7(7)	70(11)	23(9)
C(19)	120(8)	67(4)	64(4)	−0(6)	85(10)	−19(9)
C(20)	114(7)	33(3)	84(5)	−8(5)	119(10)	−9(6)
C(21)	130(8)	47(3)	105(6)	20(7)	149(12)	9(8)
C(22)	162(11)	57(4)	141(9)	44(9)	162(17)	32(11)
C(23)	144(10)	59(4)	166(9)	4(10)	208(17)	15(10)
C(24)	160(10)	57(3)	153(8)	34(10)	227(16)	18(11)
C(25)	143(8)	43(3)	91(5)	9(6)	144(12)	−3(8)
C(26)	100(7)	42(3)	86(5)	19(6)	128(10)	2(7)
C(27)	255(14)	48(4)	127(8)	41(8)	261(19)	62(11)
C(28)	283(17)	58(4)	194(11)	81(11)	344(25)	101(13)
C(29)	216(14)	52(4)	209(11)	−6(10)	327(23)	26(11)
C(20)	199(12)	52(4)	147(8)	−56(9)	231(18)	−29(11)
C(31)	152(9)	52(3)	88(5)	−14(7)	136(12)	−16(9)

* In the form: $\exp[-(B_{11}h^2 + B_{22}k^2 + B_{33}l^2 + B_{23}kl + B_{13}hl + B_{12}hk)]$. † Values for Sn $\times 10^5$.

TABLE 3

Positions and thermal parameters for hydrogen atoms

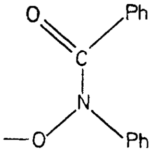
Atom *	*x*/*a*	*y*/*b*	*z*/*c*	*B*/Å^2
H(3)	−0·1655	0·0625	−0·2100	5·1
H(4)	−0·2861	0·0804	−0·4127	5·7
H(5)	−0·2602	0·2001	−0·4780	5·7
H(6)	−0·1119	0·3050	−0·3480	6·1
H(7)	0·0156	0·2846	−0·1446	5·2
H(9)	−0·1268	−0·0043	0·0155	5·5
H(10)	−0·3697	−0·0297	−0·0545	6·6
H(11)	−0·5497	0·0704	−0·1584	6·2
H(12)	−0·4862	0·1947	−0·1862	6·0
H(13)	−0·2459	0·2220	−0·1173	4·9
H(15)	0·0857	0·0711	0·2693	5·1
H(16)	0·1364	0·0298	0·4444	6·4
H(17)	0·3397	0·0790	−0·0145	5·4
H(18)	0·5324	0·1333	0·6228	5·6
H(19)	0·4862	0·1781	0·4565	5·4
H(21)	0·5143	0·0769	0·3563	5·6
H(22)	0·7324	0·0587	0·3817	7·4
H(23)	0·7667	0·1222	0·2577	7·1
H(24)	0·5799	0·2032	0·1059	6·6
H(25)	0·3665	0·2256	0·0819	5·3
H(27)	0·1116	0·3200	0·0408	7·1
H(28)	0·0493	0·4525	0·05400	8·5
H(29)	0·1036	0·4990	0·2264	7·6
H(30)	0·2129	0·4142	0·3868	7·0
H(31)	0·2704	0·2812	0·3755	5·7

* Numbered according to the carbon atom to which they are attached.

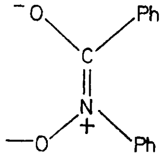
no reason for the large difference between the two O–Sn–C(eq) bond angles (111, 125·5°) except perhaps crystal packing forces. The angles between axial and equatorial sites range from 71·3 to 105·7°, and that between the two axial positions 157·2°.

The tin–carbon bond distances all fall within the range measured for other phenyltin compounds,[5] but the phenyl group in the axial position is at a significantly greater distance [2·176(9) Å] from the tin than are the two equatorial groups [mean 2·136(8) Å]. Both the covalent and co-ordinate tin–oxygen bond distances are among the shortest of each type yet recorded.[5] The co-ordinate distance [2·308(4) Å] is slightly less than that in the complex (Ph$_3$P:CH·COMe)Me$_3$SnCl (2·332 Å)[6] but much lower than the intermolecular Sn · · · O:C distance in tribenzyltin acetate (2·65 Å)[2] and the intramolecular (presumed to be a non-bonding interaction) Sn · · · O:C distance in tricyclohexyltin acetate (2·95 Å).[3] The observed bond angles at tin and tin–ligand distances are thus in accord with the expectations of simple *sp*^{3}*d* hybridisation for tin.

The bond distances within the hydroxylamine residue reveal some contribution of the canonical resonance form (V) to the overall electronic distribution. Structural parameters for the unsubstituted hydroxylamine are unavailable, but those of the similar *N*-acetylhydroxylamine hemihydrate, HO·NH·COMe,$\frac{1}{2}$H$_2$O,[7] are compared with Ph$_3$Sn(ONPh·COPh) in Table 8. In both compounds, the heavy-atom framework is essentially planar; the sums of the angles at the carbonyl carbon and nitrogen atoms being 360°. The N–O bond distance in Ph$_3$SnONPh·COPh is slightly shorter (1·384 Å) than in HONH·COMe,$\frac{1}{2}$H$_2$O, but this difference is most probably due to the lack of substitution at nitrogen and hydrogen bonding present in the latter compound. The respective relative shortening and lengthening of the C(:O)–N and C=O bonds is more significant. The latter is increased to 1·265 from 1·245 Å in HONH·COMe,$\frac{1}{2}$H$_2$O and 1·23 Å generally found in esters, aldehydes, and ketones.[8] This value compares well with the distances in zwitterionic compounds such as DL-serine (1·26 Å)[8] and (Ph$_3$P:CH·COMe)Me$_3$SnCl (1·268 Å).[6] The endocyclic C–N bond distance (1·304 Å) is much lower than the corresponding exocyclic distance (1·430 Å) which is of normal single N–C(Ph) bond length, indicating a greater double-bond character for the endocyclic bond than in HONH·COMe,$\frac{1}{2}$H$_2$O (1·333 Å)[7] or HCO·NH$_2$ (1·322 Å).[8] Corresponding C=N double-bond distances in hydroximate derivatives fall in the range 1·26—1·29 Å.[9,10]



(IV)



(V)

The determined structure rationalises observed spectroscopic data for Ph$_3$Sn(ONPh·COPh).[4] The monomeric character observed for both solution and vapour is preserved in the crystal [no intermolecular Sn · · · O interaction is present (Table 5)], and the strong intramolecular carbonyl–tin co-ordination is reflected by the very low i.r. carbonyl stretching frequency (1540 cm^{-1}), some 120 cm^{-1} lower than in Me$_3$Si(ONPh·COPh) where such co-ordination is absent. The presence of the $\overline{\text{Sn·O·NPh·C:O}}$ heterocyclic ring also rationalises the formation in the mass spectra of this and closely related compounds of fragments resulting from ring-contraction processes which take place by loss of [O] and [PhN] from the heterocycle.

It is possible to obtain semiquantitative estimates of the tin-119*m* Mössbauer quadrupole splitting, Δ, using point-charge calculations together with empirically derived partial quadrupole splitting (PQS) for the ligands attached to tin.[11,12] Thus, for the five-co-ordinate configurations (II) and (III), in which the tin atom is bonded to three phenyl groups and two oxygen atoms, assuming idealized geometry and equivalence of

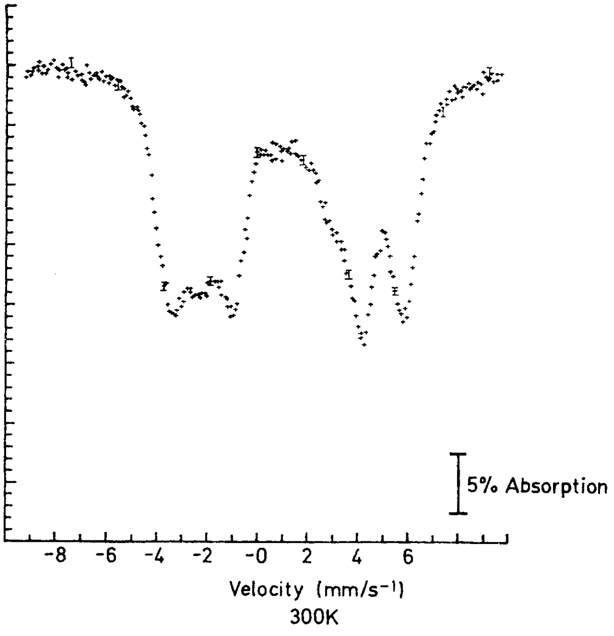


FIGURE 2 Magnetically perturbed Mössbauer spectrum

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[4] P. G. Harrison, *Inorg. Chem.*, 1973, **12**, 1545.

[5] B. Y. K. Ho and J. J. Zuckerman, *J. Organometallic Chem.*, 1973, **49**, 1.

[6] J. Buckle, P. G. Harrison, T. J. King, and J. A. Richards, *J.C.S. Chem. Comm.*, 1972, 1104.

[7] B. H. Bracher and R. W. H. Small, *Acta Cryst.*, 1970, **B26**, 1705.

[8] 'Molecular Structures and Dimensions,' vol. A1, eds. O. Kennard, D. G. Watson, F. H. Allen, N. W. Isaacs, W. D. S. Motherwell, R. C. Petersen, and W. G. Town, N. V. A. Oosthoek's Vitgevers Mij, Utrecht, 1972.

[9] I. K. Larsen, *Acta Chem. Scand.*, 1971, **25**, 2409.

[10] M. G. Waite and G. A. Sim, *J. Chem. Soc.* (*B*), 1971, 752, 1102.

[11] R. V. Parish and R. H. Platt, *Inorg. Chim. Acta*, 1970, **4**, 65.

[12] R. C. Poller and J. N. R. Ruddick, *J. Organometallic Chem.*, 1972, **39**, 121.

phenyl groups and oxygen atoms, expressions (1) and (2) may be derived for Δ_{cis} and Δ_{trans} in terms of [Ph] and [O], the respective PQS values for phenyl groups and oxygen atoms. Poller and Ruddick[12] have derived

$$\Delta_{cis} = \{3[\mathrm{Ph}]^2 - 6[\mathrm{Ph}][\mathrm{O}] + 4[\mathrm{O}]^2\}^{\frac{1}{2}} \qquad (1)$$

$$\Delta_{trans} = -3[\mathrm{Ph}] + 4[\mathrm{O}] \qquad (2)$$

values of $-0{\cdot}19$ for [Ph], 0·72 for [O_2CMe], and 0·85 for [O_2CCF_3]. Using these values, the calculated quadrupole splittings for the *trans*-derivatives, Ph_3SnO_2CMe (3·45 mm s^{-1}) and $Ph_3SnO_2CCF_3$ (3·97 mm s^{-1}), are in reasonable accord with observed data (3·34 and 4·00 mm s^{-1}, respectively). In the present case, it might be expected that [O] would fall somewhere between these extreme values. On this basis, therefore, the quadrupole splitting for $Ph_3Sn(ONPh{\cdot}COPh)$ would lie in the range 1·73—1·99 mm s^{-1}, compared with the experimentally observed value of 1·94 mm s^{-1}.[4] From the magnetically perturbed ($H = 6$ T) Mössbauer spectrum (Figure 2) obtained at 4·2 K, the sign of the electric-field gradient at tin in $Ph_3Sn(ONPh{\cdot}COPh)$ is easily deduced to be negative.

TABLE 4

Bond lengths (Å) and angles (°), with standard deviations in parentheses

(*a*) Distances	
Sn(1)–O(1)	2·091(5)
Sn(1)–O(2)	2·308(4)
Sn(1)–C(20)	2·142(9)
Sn(1)–C(26)	2·131(7)
Sn(1)–C(14)	2·176(9)
C(14)–C(15)	1·368(12)
C(15)–C(16)	1·400(11)
C(16)–C(17)	1·382(11)
C(17)–C(18)	1·351(14)
C(18)–C(19)	1·388(10)
C(19)–C(14)	1·408(9)
C(20)–C(21)	1·399(10)
C(21)–C(22)	1·369(16)
C(22)–C(23)	1·380(15)
C(23)–C(24)	1·379(13)
C(24)–C(25)	1·372(15)
C(25)–C(20)	1·394(11)
C(26)–C(27)	1·393(10)
C(27)–C(28)	1·414(13)
C(28)–C(29)	1·349(16)
C(29)–C(30)	1·373(13)
C(30)–C(31)	1·384(12)
C(31)–C(26)	1·390(10)
O(1)–N(1)	1·384(6)
N(1)–C(1)	1·304(9)
C(1)–O(2)	1·265(9)
C(1)–C(2)	1·495(9)
C(2)–C(3)	1·388(10)
C(3)–C(4)	1·398(9)
C(4)–C(5)	1·375(12)
C(5)–C(6)	1·391(12)
C(6)–C(7)	1·392(10)
C(7)–C(2)	1·389(11)
N(1)–C(8)	1·430(10)
C(8)–C(9)	1·399(10)
C(9)–C(10)	1·386(14)
C(10)–C(11)	1·380(13)
C(11)–C(12)	1·364(13)
C(12)–C(13)	1·398(14)
C(13)–C(8)	1·369(10)
(*b*) Angles	
O(1)–Sn(1)–O(2)	71·3(2)
O(2)–Sn(1)–C(20)	83·1(2)
O(2)–Sn(1)–C(26)	90·7(2)
O(2)–Sn(1)–C(14)	157·2(2)
O(1)–Sn(1)–C(20)	125·5(2)
O(1)–Sn(1)–C(26)	110·9(2)
O(1)–Sn(1)–C(14)	86·7(2)
C(14)–Sn(1)–C(20)	105·7(3)
C(14)–Sn(1)–C(26	103·2(3)
C(20)–Sn(1)–C(26)	116·9(3)
Sn(1)–O(1)–N(1)	115·6(4)
Sn(1)–O(2)–C(1)	112·1(3)
O(1)–N(1)–C(1)	117·3(6)
O(1)–N(1)–C(8)	113·0(5)
C(1)–N(1)–C(8)	128·8(6)
N(1)–C(1)–C(2)	122·1(6)
N(1)–C(1)–O(2)	119·7(6)
O(2)–C(1)–C(2)	118·1(6)
C(1)–C(2)–C(3)	120·7(6)
C(1)–C(2)–C(7)	118·3(6)
C(3)–C(2)–C(7)	120·6(6)
C(2)–C(3)–C(4)	119·1(7)
C(3)–C(4)–C(5)	119·6(8)
C(5)–C(6)–C(7)	118·3(8)
C(6)–C(7)–C(2)	120·4(7)
N(1)–C(8)–C(9)	118·4(6)
N(1)–C(8)–C(13)	120·9(6)
C(9)–C(8)–C(13)	120·6(7)
C(8)–C(9)–C(10)	119·1(7)
C(9)–C(10)–C(11)	120·4(8)
C(10)–C(11)–C(12)	119·6(8)
C(11)–C(12)–C(13)	121·2(8)
C(12)–C(13)–C(8)	118·8(7)
Sn(1)–C(14)–C(15)	123·5(5)
Sn(1)–C(14)–C(19)	117·7(5)
C(15)–C(14)–C(19)	118·5(7)
C(14)–C(15)–C(16)	120·1(7)
C(15)–C(16)–C(17)	121·6(8)
C(16)–C(17)–C(18)	117·8(8)
C(17)–C(18)–C(19)	122·4(8)
C(18)–C(19)–C(14)	119·6(7)
Sn(1)–C(20)–C(21)	120·7(5)
Sn(1)–C(20)–C(25)	121·6(5)
C(21)–C(20)–C(25)	117·6(7)
C(20)–C(21)–C(22)	120·8(8)
C(21)–C(22)–C(23)	121·0(9)
C(22)–C(23)–C(24)	118·7(9)
C(23)–C(24)–C(25)	120·9(9)
C(24)–C(25)–C(20)	120·9(8)
Sn(1)–C(26)–C(27)	123·5(6)
Sn(1)–C(26)–C(31)	119·5(6)
C(27)–C(26)–C(31)	117·0(7)
C(26)–C(27)–C(28)	120·1(9)
C(27)–C(28)–C(29)	121·1(11)
C(28)–C(29)–C(30)	119·6(11)
C(29)–C(30)–C(31)	120·2(9)
C(30)–C(31)–C(26)	121·9(8)

TABLE 5

Closest intermolecular Sn · · · O contacts (Å)

Sn(1) · · · O(1)	6·30, 9·89, 9·68, 8·74, 8·76, 8·80
Sn(1) · · · O(2)	7·35, 6·22, 9·20

TABLE 6

Equations of least-squares planes, and, in square brackets, deviations (Å) of atoms from these planes

Plane (1): Sn(1), O(1), O(2), N(1), C(1)

$$0{\cdot}5634x - 0{\cdot}8243y - 0{\cdot}0297z + 1{\cdot}9344 = 0$$

[Sn(1) −0·0953(5), O(1) 0·139(5), O(2) 0·102(6), N(1) −0·101(6), C(1) −0·044(7)]

Plane (2): C(8)—(12)

$$0{\cdot}3885x - 0{\cdot}3284y - 0{\cdot}8609z + 0{\cdot}8060 = 0$$

[C(8) 0·001(6), C(9) −0·004(7), C(10) 0·003(8), C(11) 0·001(8), C(12) −0·004(8), C(13) 0·003(7)]

Plane (3): C(2)—(4)

$$-0{\cdot}9163x + 0{\cdot}3860y - 0{\cdot}1070z - 0{\cdot}7068 = 0$$

[C(2) 0·006(9), C(3) 0·001(9), C(4) −0·007(10), C(5) 0·006(10), C(6) 0·002(11), C(7) −0·008(9)]

Dihedral angles (°): (1)–(2) 58·9
(1)–(3) 164·9

TABLE 7

Comparison of the tin–ligand bond distances (Å) in $Ph_3Sn(ONPh{\cdot}COPh)$ with those in related compounds

Molecule [a]	*r*(Sn–C)	*r*(Sn–O)	Confign. at Sn
(1) [b]	Eq. 2·136(8), ax. 1·176(9)	Cov. 2·091(5), Co-ord. 2·308(4)	Trig. bipy.
(2) [d]	2·122(22)		Tet.
(3) [e]	2·087(14), 2·134(14), 2·170(15)	2·010(9)	Tet.
(4) [f]	2·15(2)	2·25(2)	Trig. bipy.
(5) [g]	2·15(3), 2·18(3), 2·18(3)	Cov. 2·14(2) Co-ord. 2·65(2)	Trig. bipy.
(6) [h]	2·19(3) [c]	Cov. 2·12(3)	Dist. tet.
(7) [i]	2·133(9)	2·332(6)	Trig. bipy.

[a] (1) $Ph_3Sn(ONPh{\cdot}COPh)$; (2) Ph_3SnCl; (3) $Ph_3SnOC_5Ph_4$-$[Mn(CO)_3]$; (4) $(MeCO_2)_2Sn_2Ph_4$; (5) $(PhCH_2)_3SnO_2CMe$; (6) $C_6H_{11}SnO_2CMe$; (7) $(Ph_3PCH{\cdot}COMe)Me_3SnCl$. [b] This work. [c] Mean value. [d] N. G. Bokii, G. N. Zakharova, and Yu. T. Struchkov, *J. Strukt. Chem.*, 1970, **11**, 828. [e] R. F. Bryan and H. P. Weber, *J. Chem. Soc.* (*A*), 1967, 843. [f] G. Bandoli, D. A. Clemente, and C. Panattoni, *Chem. Comm.*, 1971, 311. [g] Ref. 2. [h] Ref. 3. [i] Ref. 6.

TABLE 8

Comparison of bond distances (Å) in (*A*) $HO{\cdot}NH{\cdot}COMe,\frac{1}{2}H_2O$ and (*B*) $Ph_3Sn(ONPh{\cdot}COPh)$

Bond	(*A*)	(*B*)
N–O	1·400(5)	1·384(6)
C(:O)–N	1·333(6)	1·304(9)
C=O	1·245(6)	1·265(9)
C–R	1·505(6) *	1·495(7) †
C–NR		1·430(10)

* R = Me. † R = Ph.

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