Structural Studies in Main-group Chemistry. Part XIII.[1] The Structure of Bis(*N*-benzoyl-*N*-phenylhydroxylaminato)dichlorotin(IV)

By **Philip G. Harrison,* Trevor J. King,** and **John A. Richards,** Department of Chemistry, University of Nottingham, Nottingham NG7 2RD

The structure of the title compound has been determined by single-crystal *X*-ray diffractometry using Patterson and Fourier techniques. Crystals are orthorhombic, space group $P2_12_12_1$, with $Z = 12$ in a unit cell of dimensions $a = 24.336(4)$, $b = 33.192(4)$, and $c = 9.408(2)$ Å. The structure has been solved by constructing a pseudo-cell related to the unit cell such that $a' = a$, $b' = \frac{b}{3}$, and $c' = c$, and re-indexing the reflections such that $h' = h$, $k' = \frac{k}{3}$, and $l' = l$, and refined by block-diagonal least-squares methods to R 0.081 for 1 942 re-indexed reflections. The tin atom is co-ordinated in a distorted octahedral fashion by two chlorine atoms occupying *cis* positions [Sn–Cl 2.361(5) Å, Cl–Sn–Cl 99.0(2)°] and by two *N*-benzoyl-*N*-phenylhydroxylaminato-residues which chelate the tin nearly symmetrically [Sn–O 2.04(2), 2.11(2) Å].

ALTHOUGH there is a relative abundance of structural data for six-co-ordinate dimethyltin(IV) compounds, apparently no data are available for the corresponding dichlorotin(IV) derivatives. The early assumption that the organic groups of the former class always adopt mutually *trans* positions in an octahedral co-ordination sphere has since been shown to be far from the real situation. Observed C–Sn–C bond angles in this type of compound range from 180 to 110.7° depending on the other groups bonded to tin, with chelating ligands with small 'bites' favouring smaller bond angles.[2] It has been reasoned previously [3] that in octahedral complexes of the type $[SnX_2Y_4]$, in which all bonds are to electronegative, potentially π-bonding, atoms, *cis* stereochemistry will be preferred except when steric restrictions are placed on the system,[4,5] since the *cis* ligands would then be able to π-donate into different t_{2g} orbitals thus stabilising the *cis* configuration. In our studies of *N*-hydroxylamides of tin, we have already determined the structure of $[SnMe_2\{ONMe(COMe)\}_2]$, in which both *N*-methyl-*N*-acetyl groups chelate the tin in an unsymmetrical fashion resulting in a C–Sn–C angle of 145.8°.[6] In this paper we report the results of an *X*-ray diffraction study of a dichlorotin(IV) analogue, $[SnCl_2\{ONPh(COPh)\}_2]$, which would be expected to adopt the *cis* configuration.

EXPERIMENTAL

Needle crystals of the compound were obtained by recrystallisation from a methanol–chloroform mixture of the product of the reaction between the lithium salt of *N*-hydroxy-*N*-phenylbenzamide and anhydrous tin(IV) chloride in benzene.[7] A suitable crystal of approximate dimensions 0.3 × 0.3 × 0.5 mm was mounted on to a glass fibre using an epoxy resin, and used for both the initial photography and subsequent intensity-data collection.

---

[1] Part XII, P. G. Harrison and J. A. Richards, *J. Organometallic Chem.*, 1976, **108**, 64.

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

[3] R. J. H. Clark, L. Maresca, and R. J. Puddephatt, *Inorg. Chem.*, 1968, **7**, 1603.

[4] A. J. Corby, T. Hinsperger, L. Mitrichuk, and H. D. Sharma, *Inorg. Chem.*, 1970, **9**, 2573.

[5] P. G. Harrison, B. C. Lane, and J. J. Zuckerman, *Inorg. Chem.*, 1972, **11**, 1537.

[6] P. G. Harrison, T. J. King, and J. A. Richards, *J.C.S. Dalton*, 1975, 826.

[7] J. A. Richards, Ph.D. Thesis, Nottingham University, 1975.

Crystal Data.—$C_{26}H_{20}Cl_2N_2O_4Sn$, *M* 614.05, Orthorhombic, space group $P2_12_12_1$ (from systematic absences $h00$, $h = 2n + 1$; $0k0$, $k = 2n + 1$; and $00l$, $l = 2n + 1$), $a = 24.336(4)$, $b = 33.192(4)$, $c = 9.408(2)$ Å, $U = 7\,599.3$ Å^3, $D_0 = 1.65$ (by flotation), $Z = 12$, $D_c = 1.61$ g cm^{-3}, $F(000) = 3\,672$, $\mu(\text{Mo-}K_\alpha) = 12.62$ cm^{-1}, $\lambda(\text{Mo-}K_\alpha) = 0.710\,69$ Å.

Cell Measurements and Data Collection.—The approximate cell dimensions and crystal class were determined from oscillation and zero- and first-layer Weissenberg photographs obtained on a Leeds equi-inclination Weissenberg camera. The festoons about the *b* reciprocal lattice on the Weissenberg photographs were strong when $k = 3n$, but weak otherwise. Accurate cell parameters were obtained by least-squares refinement using 12 indexed reflections, and the intensities of *ca.* 7 600 reflections and associated background counts were measured using monochromated Mo-K_α radiation on a Hilger and Watts Y290 four-circle diffractometer in the ω—2θ scanning mode. Each reflection was counted for 30 s, and its two associated background counts for 15 s. The reflections were brought to the same relative intensities by reference reflections counted after every 100 reflections, the orientation of the diffractometer being checked after every 200 reflections. The background counts, which were low, were subtracted from each reflection, and reflections with corrected counts less than three times the estimated standard deviation were considered as unobserved and discarded, reducing the number of observed reflections to 5 328. The intensities were corrected for Lorentz and polarisation effects, but no absorption correction was made due to the low μ value. The atomic scattering factors utilised were those of neutral atoms.[8]

Structure Determination and Refinement.—The number of reflections was initially reduced to 2 171 by progressive elimination of the less-intense reflections in order to enable adequate computer storage for calculation. A Patterson synthesis gave a multitude of peaks in the Patterson vector space, which could not initially be interpreted in terms of the three tin atoms within the asymmetric unit as a result of the large number of cross-vectors arising from the tin–tin and tin–chlorine vectors. However, the Patterson space also showed a vector at $0, \frac{1}{3}, 0$ (and also at $0, \frac{2}{3}, 0$), which was essentially as intense as the origin vector. This is perhaps not surprising since by selection of the 2 171 most intense reflections the majority of the reflections correspond to the set hkl where $k = 3n$. Nevertheless, this pseudo-origin vector was maintained in a subsequent Patterson synthesis incorporating the full set of reflections, suggesting that the asymmetric unit of the cell consists of three almost identical units, repeating at $y = \frac{1}{3}$ and $\frac{2}{3}$. The approximate retention of the structure within these units was confirmed in the Patterson space by the appearance of duplicate vectors removed by $b/3$ from the original vector. To facilitate solution of the structure, a pseudo-cell ($a' = a$, $b' = b/3$, $c' = c$) was constructed, which contained four molecules per unit pseudo-cell and retained the symmetry of the original cell. The reflections were re-indexed such that $h' = h$, $k' = k/3$, $l' = l$, and those with non-integer values of k were discarded leaving 1 948 indexed reflections.

A Patterson synthesis of the re-indexed reflections gave the position of the single tin atom in the asymmetric unit, and this was used to phase a structure-factor calculation. The resulting Fourier synthesis gave the positions of seven light atoms, which were used as a basis of two cycles of isotropic full-matrix least-squares refinement, and reduced the *R* to 0.335. Two successive Fourier syntheses with intermediate least-squares refinement, with the tin and two chlorine atoms varying anisotropically, yielded the positions of the remaining non-hydrogen light atoms. A further two cycles of refinement in the mixed mode followed by six cycles of blocked anisotropic least squares gave convergence at *R* 0.083. Following an analysis of $\Delta(F_o - F_c)^2$ in terms of F_o, the weighting scheme $w = 1/\{1 + [(F_o - 37.0)^2/56.2^2]\}$ was applied, and two cycles of least-squares blocked anisotropic refinement converged at *R* 0.081. At this stage a rejection test $W\Delta F > 9.0$ was applied and six reflections were rejected, and a final cycle of least-squares refinement gave *R* 0.081 for 1 942 reflections. An extension of this solution to the full cell using the full data was not successful, although the peaks of the Patterson synthesis are fully explained in terms of tin and chlorine interatomic vectors. The molecular structure indicated within the pseudo-cell shows considerable disorder in one of the phenyl rings [C(21)–C(26)], suggesting that the true cell may comprise three repeating molecular units with differences in the corresponding phenyl rings probably arising from crystal-packing differences. The final positional atomic parameters and anisotropic thermal parameters of the averaged molecule in the pseudo-cell are listed in Tables 1 and 2 respectively, whilst intramolecular bond distances and angles are given in Table 3. Final observed and calculated structure

TABLE 1

Final fractional atomic co-ordinates of $[SnCl_2\{ONPh(COPh)\}_2]$ in the pseudo-cell, with estimated standard deviations in parentheses

Atom	x/a'	y/b'	z/c'
Sn(1)	0.073 93(5)	0.220 56(11)	0.212 46(18)
Cl(1)	0.167 4(2)	0.247 3(6)	0.274 0(9)
Cl(2)	0.056 0(3)	0.042 7(4)	0.342 1(8)
N(1)	−0.006 5(5)	0.307 5(12)	0.399 7(20)
N(2)	0.111 4(7)	0.192 3(15)	−0.068 3(24)
O(1)	0.048 4(4)	0.334 7(11)	0.371 4(19)
O(2)	−0.010 5(5)	0.240 7(11)	0.174 2(15)
O(3)	0.084 7(7)	0.122 7(11)	0.030 7(19)
O(4)	0.085 0(5)	0.354 1(11)	0.055 2(20)
C(1)	−0.035 5(6)	0.264 9(14)	0.287 3(27)
C(2)	0.109 9(7)	0.301 6(17)	−0.055 7(33)
C(3)	−0.095 1(7)	0.245 8(13)	0.307 2(23)
C(4)	−0.127 5(7)	0.327 1(15)	0.379 3(21)
C(5)	−0.184 8(7)	0.306 1(19)	0.380 3(30)
C(6)	−0.207 3(8)	−0.207 5(21)	0.316 1(30)
C(7)	−0.174 2(9)	0.125 5(20)	0.239 3(32)
C(8)	−0.116 2(8)	0.145 2(15)	0.231 0(23)
C(9)	−0.018 5(6)	0.302 6(15)	0.550 2(22)
C(10)	−0.006 4(8)	0.403 9(24)	0.630 3(32)
C(11)	−0.014 1(10)	0.395 3(25)	0.781 8(39)
C(12)	−0.038 5(7)	0.289 0(26)	0.838 5(26)
C(13)	−0.052 4(8)	0.196 0(22)	0.745 5(22)
C(14)	−0.042 6(7)	0.201 5(17)	0.609 8(24)
C(15)	0.137 7(8)	0.392 7(22)	−0.152 6(28)
C(16)	0.170 6(8)	0.491 5(22)	−0.089 0(29)
C(17)	0.197 0(10)	0.570 7(28)	−0.174 3(26)
C(18)	0.192 0(7)	0.555 6(21)	−0.313 3(38)
C(19)	0.160 9(8)	0.461 5(21)	−0.395 2(32)
C(20)	0.134 9(8)	0.375 0(18)	−0.287 9(34)
C(21)	0.144 4(10)	0.106 7(22)	−0.172 5(33)
C(22)	0.192 8(15)	0.088 3(40)	−0.116 1(38)
C(23)	0.243 9(15)	0.017 1(78)	−0.221 7(95)
C(24)	0.210 8(13)	−0.030 4(39)	−0.255 6(92)
C(25)	0.162 4(22)	−0.018 4(33)	−0.370 1(42)
C(26)	0.121 6(13)	0.072 8(30)	−0.311 0(37)

---

[8] 'International Tables for *X*-Ray Crystallography,' vol. III, Kynoch Press, Birmingham, 1962.

TABLE 2

Final anisotropic thermal parameters of $[SnCl_2\{ONPh(COPh)\}_2]$ for the pseudo-cell,* with estimated standard deviations in parentheses

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Sn(1)	7.44(6)	5.09(5)	14.68(12)	0.76(7)	6.02(8)	2.37(8)
Cl(1)	6.4(3)	11.5(5)	22.7(8)	2.8(3)	4.5(4)	6.8(6)
Cl(2)	16.0(6)	5.1(3)	13.0(6)	0.6(3)	8.2(5)	1.5(3)
N(1)	5.0(1)	4.0(1)	13.0(1)	−0.5(6)	−3.5(9)	−0.2(9)
N(2)	11.0(1)	7.0(1)	16.0(2)	3.0(1)	9.8(13)	5.8(12)
O(1)	4.0(1)	5.0(1)	20.0(2)	−1.1(5)	2.8(8)	−0.1(9)
O(2)	8.0(1)	0.7(1)	10.0(1)	−0.5(7)	4.6(8)	−1.2(8)
O(3)	14.0(1)	5.0(1)	15.0(2)	1.3(8)	9.6(12)	3.9(9)
O(4)	6.0(1)	6.0(1)	17.0(2)	1.9(6)	5.4(10)	4.1(9)
C(1)	5.0(1)	4.0(1)	12.0(2)	0.1(7)	−1.8(11)	1.5(12)
C(2)	5.0(1)	0.7(1)	20.0(3)	1.9(9)	7.5(15)	5.1(16)
C(3)	6.0(1)	4.0(1)	11.0(1)	0.4(7)	−3.6(10)	0.4(10)
C(4)	6.0(1)	5.0(1)	9.0(1)	−0.1(8)	0.3(10)	−0.5(10)
C(5)	5.0(1)	7.0(1)	16.0(2)	0.1(9)	1.0(13)	−1.2(15)
C(6)	8.0(1)	11.0(2)	18.0(2)	0.2(12)	−4.9(15)	−10.5(19)
C(7)	0.9(1)	10.0(1)	16.0(3)	3.9(12)	0.5(17)	−3.6(18)
C(8)	9.0(1)	6.0(1)	9.0(2)	1.7(9)	−1.1(12)	−2.1(12)
C(9)	5.0(1)	5.0(1)	10.0(1)	−0.1(7)	−0.6(9)	−0.5(11)
C(10)	6.0(1)	10.0(2)	16.0(2)	1.7(12)	−0.6(14)	−4.6(19)
C(11)	8.0(1)	11.0(2)	16.0(3)	−0.4(14)	−2.2(18)	−2.4(23)
C(12)	6.0(1)	15.0(2)	11.0(2)	−2.3(14)	0.1(11)	−0.2(20)
C(13)	10.0(1)	12.0(2)	6.0(1)	−1.0(13)	2.2(10)	1.7(13)
C(14)	8.0(1)	5.0(1)	9.0(2)	−1.8(9)	2.7(11)	0.2(11)
C(15)	5.0(1)	10.0(2)	10.0(2)	2.0(1)	6.0(1)	5.0(1)
C(16)	5.0(1)	10.0(2)	15.0(2)	0.3(12)	2.0(1)	6.0(2)
C(17)	11.0(2)	16.0(3)	6.0(2)	−1.0(2)	−2.0(1)	−7.0(2)
C(18)	5.0(1)	9.0(2)	21.0(3)	0.4(10)	−2.0(2)	−6.0(2)
C(19)	7.0(1)	10.0(2)	18.0(3)	3.0(1)	3.7(1)	4.0(2)
C(20)	6.0(1)	8.0(1)	18.0(3)	3.0(1)	9.0(2)	6.0(2)
C(21)	12.0(2)	12.0(2)	10.0(3)	5.0(1)	11.0(2)	14.0(2)
C(22)	22.0(3)	31.0(5)	17.0(3)	20.0(4)	14.0(3)	18.0(3)
C(23)	24.0(6)	53.0(11)	66.0(12)	25.0(7)	26.3(8)	54.0(10)
C(24)	8.0(2)	14.0(3)	45.0(12)	6.0(2)	−15.0(4)	−16.0(5)
C(25)	30.0(5)	13.0(3)	15.0(3)	−2.0(3)	12.0(4)	−2.0(3)
C(26)	15.0(2)	15.0(2)	15.0(3)	2.0(2)	−0.5(22)	−1.0(3)

* U_{ij} are of the form $\exp[-2\pi^2 10^{-2}(h^2U_{11}a^{*2} + k^2U_{22}b^{*2} + l^2U_{33}c^{*2} + hkU_{12}a^*b^* + hlU_{13}a^*c^* + klU_{23}b^*c^*)]$.

factors are listed in Supplementary Publication No. SUP 21758 (10 pp.).*

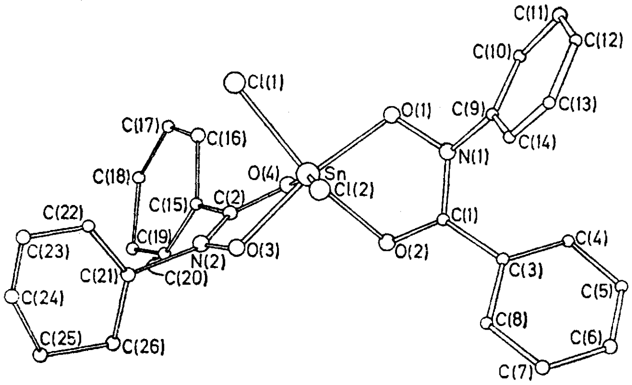

FIGURE 1 Structure of $[SnCl_2\{ONPh(COPh)\}_2]$ showing atomic numbering

RESULTS AND DISCUSSION

The title compound possesses a distorted octahedral structure, with both hydroxylamine residues chelating tin and the two chlorine atoms occupying *cis* positions (Figure 1). The bond angles at tin range from 76.3(5) to 99.0(2)°, and the Cl–Sn–Cl angle (99.0°) compares with those observed for $[SnPh_2Cl_2]\cdot$bipy [103.5(1)°] (bipy = 2,2′-bipyridyl),[9] $[SnMe_2Cl_2(dmso)_2]$ [95.9(4)°] (dmso = dimethyl sulphoxide),[10] and $[SnCl_4(NCMe)_2]$ [93.9(3)—102.6(2)°] [11] (Table 4). The tin–chlorine bond distances [mean 2.361(6) Å] are much shorter than those in octa-

TABLE 3

Intramolecular bond distances (Å) and angles (°) in $[SnCl_2\{ONPh(COPh)\}_2]$ with standard deviations in parentheses

(*a*) Bond distances

Sn(1)–Cl(1)	2.366(5)	Sn(1)–Cl(2)	2.356(6)
Sn(1)–O(1)	2.054(15)	Sn(1)–O(3)	2.041(16)
Sn(1)–O(2)	2.099(12)	Sn(1)–O(4)	2.180(15)
O(1)–N(1)	1.40(2)	O(3)–N(2)	1.37(2)
N(1)–C(1)	1.36(3)	N(2)–C(2)	1.31(2)
N(1)–C(9)	1.45(3)	N(2)–C(21)	1.58(3)
C(1)–O(2)	1.25(3)	C(2)–O(4)	1.30(3)
C(1)–C(3)	1.48(2)	C(2)–C(15)	1.45(3)
C(3)–C(4)	1.37(2)	C(15)–C(16)	1.48(3)
C(4)–C(5)	1.41(2)	C(16)–C(17)	1.35(4)
C(5)–C(6)	1.36(3)	C(17)–C(18)	1.32(4)
C(6)–C(7)	1.41(3)	C(18)–C(19)	1.50(4)
C(7)–C(8)	1.43(3)	C(19)–C(20)	1.53(4)
C(8)–C(3)	1.42(2)	C(20)–C(15)	1.29(4)
C(9)–C(10)	1.38(3)	C(21)–C(22) *	1.31(4)
C(10)–C(11)	1.44(5)	C(22)–C(23) *	1.78(8)
C(11)–C(12)	1.42(4)	C(23)–C(24) *	1.01(8)
C(12)–C(13)	1.39(3)	C(24)–C(25) *	1.60(8)
C(13)–C(14)	1.30(3)	C(25)–C(26) *	1.52(5)
C(14)–C(9)	1.38(2)	C(26)–C(21) *	1.46(4)

(*b*) Bond angles

Cl(1)–Sn(1)–Cl(2)	99.0(2)	O(1)–Sn(1)–O(2)	76.3(5)
Cl(1)–Sn(1)–O(1)	92.1(4)	O(1)–Sn(1)–O(3)	167.2(6)
Cl(1)–Sn(1)–O(3)	166.0(4)	O(1)–Sn(1)–O(4)	96.8(6)
Cl(1)–Sn(1)–O(3)	98.5(5)	O(2)–Sn(1)–O(3)	92.2(6)
Cl(1)–Sn(1)–O(4)	87.8(4)	O(2)–Sn(1)–O(4)	86.0(5)
Cl(2)–Sn(1)–O(1)	94.6(4)	O(3)–Sn(1)–O(4)	76.5(5)
Cl(2)–Sn(1)–O(2)	89.8(4)		
Cl(2)–Sn(1)–O(3)	90.8(4)		
Cl(2)–Sn(1)–O(4)	166.5(5)		
Sn(1)–O(1)–N(1)	107(1)	Sn(1)–O(3)–N(2)	109(1)
Sn(1)–O(2)–C(1)	111(1)	Sn(1)–O(4)–C(2)	111(1)
O(1)–N(1)–C(9)	113(1)	O(3)–N(2)–C(21)	109(2)
O(1)–N(1)–C(1)	115(2)	O(3)–N(2)–C(2)	119(2)
C(9)–N(1)–C(1)	130(1)	C(21)–N(2)–C(2)	132(2)
N(1)–C(1)–C(3)	118(2)	N(2)–C(2)–C(15)	124(2)
N(1)–C(1)–O(2)	119(1)	N(2)–C(2)–O(4)	117(2)
C(3)–C(1)–O(2)	124(2)	C(15)–C(2)–O(4)	119(2)
C(1)–C(3)–C(4)	122(1)	C(2)–C(15)–C(16)	117(2)
C(1)–C(3)–C(8)	114(2)	C(2)–C(15)–C(20)	120(2)
C(4)–C(3)–C(8)	124(2)	C(16)–C(15)–C(20)	123(2)
C(3)–C(4)–C(5)	117(2)	C(15)–C(16)–C(17)	120(2)
C(4)–C(5)–C(6)	122(2)	C(16)–C(17)–C(18)	117(3)
C(5)–C(6)–C(7)	120(2)	C(17)–C(18)–C(19)	130(3)
C(6)–C(7)–C(8)	120(2)	C(18)–C(19)–C(20)	108(2)
C(7)–C(8)–C(3)	117(2)	C(19)–C(20)–C(15)	122(2)
N(1)–C(9)–C(10)	117(2)	N(2)–C(21)–C(22)	107(3)
N(1)–C(9)–C(14)	121(2)	N(2)–C(21)–C(26)	121(2)
C(10)–C(9)–C(14)	122(2)	C(22)–C(21)–C(26) *	132(3)
C(9)–C(10)–C(11)	117(2)	C(21)–C(22)–C(23) *	118(4)
C(10)–C(11)–C(12)	119(3)	C(22)–C(23)–C(24) *	81(6)
C(11)–C(12)–C(13)	118(2)	C(23)–C(24)–C(25) *	139(7)
C(12)–C(13)–C(14)	123(2)	C(24)–C(25)–C(26) *	107(3)
C(13)–C(14)–C(9)	121(2)	C(25)–C(26)–C(21) *	104(3)

* Region of disorder.

---

* For details see Notice to Authors No. 7, *J.C.S. Dalton*, 1975, Index issue.

[9] P. G. Harrison, T. J. King, and J. A. Richards, *J.C.S. Dalton*, 1975, 1723.

[10] N. W. Isaacs and C. H. L. Kennard, *J. Chem. Soc.* (*A*), 1970, 1257.

[11] M. Webster and H. E. Blayden, *J. Chem. Soc.* (*A*), 1969, 2443.

TABLE 4

Comparison of bond parameters in $[SnCl_2\{ONPh(COPh)\}_2]$ and related compounds

Compound		Distance/Å			Angle/°	
		r(Sn–Cl)	r(Sn–O)	r(Sn · · · O)	Cl–Sn–Cl	O–Sn–O
$[SnCl_2\{ONPh(COPh)\}_2]$	(i)	2.366(5)	2.05(2)	2.10(1)	99.0(2)	76.3(5)
	(ii)	2.356(6)	2.04(2)	2.11(2)		76.5(6)
$[SnMe_2\{ONMe(COMe)\}_2]$ [a]	(i)		2.107(4)	2.374(5)		73.2(1)
	(ii)		2.126(4)	2.384(4)		71.1(1)
$[SnPh_3\{ONPh(COPh)\}]$ [b]			2.091(5)	2.308(4)		71.3(2)
$[SnMe_2Cl_2(dmso)_2]$ [c]		2.48(1)		2.32(3)	95.9(4)	
		2.53(1)		2.38(3)		
$[SnPh_2Cl_2(bipy)]$ [d]		2.152(8)			103.5(1)	
$[SnCl_4(NCMe)_2]$ [e]		2.339(8)—2.356(8)			93.9(3)—102.6(3)	
$[SnMe_2(pd)_2]$ [f]			2.18(1)	2.20(1)		86(1)

[a] Ref. 6. [b] Ref. 12. [d] Ref. 10. [d] Ref. 9. [e] Ref. 11. [f] pd= Pentane-2,4-dionato; G. A. Miller and E. O. Schlemper, *Inorg. Chem.*, 1973, **12**, 677.

TABLE 5

Comparison of bond distances (Å)

Bond	$[SnMe_2\{ONMe(COMe)\}_2]$ [a]	$[SnCl_2\{ONPh(COPh)\}_2]$ [b]	$[SnPh_3\{ONPh(COPh)\}]$ [c]	$MeCONHOH\cdot\frac{1}{2}H_2O$ [d]
N–O	1.381(6), 1.372(6)	1.40(2), 1.37(3)	1.384(6)	1.400(5)
C(O)–N	1.303(6), 1.314(8)	1.36(3), 1.32(3)	1.304(9)	1.333(6)
C=O	1.264(7), 1.250(6)	1.26(3), 1.30(3)	1.265(9)	1.245(6)
C–R	1.498(8), 1.502(9) [e]	1.48(2), 1.45(3) [f]	1.495(7) [f]	1.505(6) [e]
N–C(R)	1.450(9), 1.436(8) [e]	1.45(3), 1.58(3) [f]	1.430(10) [f]	

[a] Ref. 6. [b] This work. [c] Ref. 12. [d] B. H. Bracher and R. W. H. Small, *Acta Cryst.*, 1970, **B26**, 1705. [e] R = Me. [f] R = Ph.

hedral complexes of diorganotin(IV) dichlorides (2.48—2.58 Å) [2,9] but are similar to those in $[SnCl_4(NCMe)_2]$ (mean 2.35 Å) [11] in which the tin is also only bonded to electronegative ligands. The high effective nuclear charge developed on the tin atom as a result of the electronegative groups also results in a shortening of the tin–oxygen bonds compared to $[SnMe_2\{ONMe(COMe)\}_2]$ [6] and $[SnPh_3\{ONPh(COPh)\}]$.[12] Whereas in the two latter compounds the *N*-acylhydroxylaminato-groups chelate the tin in an unsymmetrical fashion with distinct covalent and co-ordinate interactions, the tin–oxygen distances in $[SnCl_2\{ONPh(COPh)\}_2]$ are nearly equal [2.04(2) and 2.05(2), and 2.11(2) and 2.10(1) Å] and are among the shortest such distances observed. In consequence, the angles subtended at tin by the ligands in $[SnCl_2\{ONPh(COPh)\}_2]$ are significantly larger (76.3, 76.6°) than in $[SnMe_2\{ONMe(COMe)\}_2]$ [71.1(1), 73.2(1)°] [6] and in $[SnPh_3\{ONPh(COPh)\}]$ (71.3°).[12]

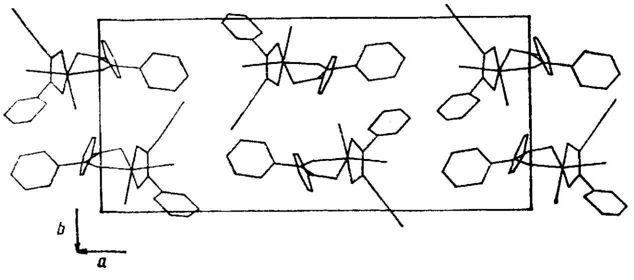

FIGURE 2 Projection of the unit cell on to the *ab* plane

Despite the shorter Sn–O bond distances in $[SnCl_2\{ONPh(COPh)\}_2]$, the similarity of the bond distances within the *N*-acylhydroxylaminato-residues of the three derivatives of tin (Table 5) show that effects resulting from electronic redistribution about the tin do not penetrate beyond the first co-ordination sphere. The ONCO skeleton of each ligand is essentially planar (Table 6),

TABLE 6

Weighted least-squares planes

(*a*) Equations with deviations * (Å) of atoms in square brackets

Plane (1): C(3)—C(8)

$$0.1118x - 0.5480y + 0.8290z = 0.6269$$

[C(3) 0.0200, C(4) 0.0012, C(5) −0.0201, C(6) 0.01653, C(7) 0.0047, C(8) −0.0223, C(1) −0.0887]

Plane (2): C(9)—C(14)

$$0.9119x - 0.3876y + 0.1348z = -1.0373$$

[C(9) 0.0278, C(10) −0.0381, C(11) 0.0210, C(12) 0.0068, C(13) −0.0197, C(14) 0.0021, N(1) 0.0811]

Plane (3): C(15)—C(20)

$$0.8144x - 0.5802y + 0.0055z = 0.2224$$

[C(15) −0.0215, C(16) −0.0005, C(17) 0.0101, C(18) −0.0009, C(19) −0.0171, C(20) 0.0298, C(2) −0.0417]

Plane (4): C(21)—(C26)

$$0.3423x + 0.7823y - 0.5137z = 3.0438$$

[C(21) −0.0476, C(22) −0.0660, C(23) 0.2662, C(24) −0.2636, C(25) −0.0213, C(26) 0.1323, N(2) −0.0947]

Plane (5): O(1), O(3), O(4), Cl(2), Sn(1)

$$0.9654x + 0.0090y + 0.2606z = 2.1524$$

[O(1) −0.0726, O(3) −0.0761, O(4) 0.0150, Cl(2) 0.0064, Sn(1) 0.1274]

Plane (6): O(1), N(1), C(1), O(2)

$$-0.2346x + 0.9195y - 0.3155z = 2.0107$$

[O(1) 0.0162, N(1) −0.0317, C(1) 0.0341, O(2) −0.0186, Sn(1) −0.8196]

Plane (7): O(3), N(2), C(2), O(4)

$$0.8459x - 0.0452y + 0.5315z = 1.8416$$

[O(3) −0.0069, N(2) 0.0135, C(2) −0.0138, O(4) 0.0071, Sn(1) 0.6323]

(*b*) Angles (°) between planes

(1)—(6) 37.7, (2)—(6) 52.2, (3)—(7) 44.1, (4)—(7) 89.4, (6)—(7) 65.9

* According to V. Schomaker, J. Waser, R. E. Marsh, and G. Bergman, *Acta Cryst.*, 1959, **12**, 600.

---

[12] P. G. Harrison and T. J. King, *J.C.S. Dalton*, 1974, 2298.

but the tin atom lies substantially out of both planes. The two phenyl groups of each hydroxylamine residue are significantly twisted with respect to the ONC=O skeletons.

As may be seen from Figure 2, the crystal is composed of isolated molecules, although crystal packing is most likely responsible for the differences in the three pseudocells.

One of us (J. A. R.) thanks the S.R.C. for support in the form of an Instant Award.

[5/2323 *Received, 28th November, 1975*]