

(1985), are intermediate between the locations derived from neutron data.

The uranium atom presents a hexagonal-bipyramidal coordination (Table 2). The uranyl ion lies on a threefold axis imposing an uncommon exact linear configuration. It is surrounded by six coplanar oxygen atoms of three acetate ligands. Table 3(b) shows the quasi-planarity of the uranyl equatorial plane and a small shift of the uranium and carbon atoms on both sides of this plane.

The six-coordination polyhedron around sodium, based on the site symmetry C_3 , is formed by two independent triangles of oxygen atoms, O3 and O4. It can be considered as intermediate between an octahedron and a trigonal prism (the rotation angle around the threefold axis between the two triangles is 30°). The six oxygen atoms belong to six different

acetate ligands and each of them is shared between two sodium atoms. Fig. 2 shows the crystal packing.

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Dichloro{1-[(2-methylphenylimino)methyl]-2-naphtholato}diphenyltin(IV): a 1:1 Molecular Adduct Formed by the Interaction of Diphenyltin(IV) Dichloride with *N*-(*o*-Substituted phenyl)-2-hydroxynaphthylmethyleamine

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Abstract. $[\text{Sn}(\text{Cl})_2(\text{C}_6\text{H}_5)_2(\text{C}_{18}\text{H}_{15}\text{NO})]$, $M_r = 605.13$, monoclinic, $P2_1$, $a = 9.586$ (2), $b = 14.269$ (2), $c = 9.765$ (3) Å, $\beta = 97.53$ (1)°, $V = 1324.2$ (6) Å³, $Z = 2$, $D_x = 1.518$ Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 1.085$ mm⁻¹, $F(000) = 608$, $T = 298$ K, $R = 0.0700$ for 2447 observed reflections. The compound crystallizes in the form of an adduct with the organotin and ligand in a 1:1 stoichiometry. The Sn atom in the title compound possesses trigonal bipyramidal geometry, with the phenolic O atom of the Schiff base and one Cl atom occupying the axial positions. The other Cl atom and the two phenyl rings occupy the equatorial positions.

Introduction. Whilst a large number of molecular adducts of organotin halides and 2-hydroxynaphthylmethyleanilines, having different substituents

in the aniline fragment, have been synthesized, crystallographic studies on the compounds have only recently been carried out (Charland, Lee, Gabe, Khoo & Smith, 1987; Khoo, Charland, Gabe & Smith, 1987). These studies, however, involve adducts with *para*-substituted anilines. To date there has been no known report on the corresponding molecular adducts with Schiff bases having *ortho*-substituted anilines. In view of the above, the 1:1 molecular adduct of diphenyltin(IV) dichloride and *N*-(*o*-substituted phenyl)-2-hydroxynaphthylmethyleamine was synthesized and a full X-ray structural investigation carried out.

Experimental. *o*-Toluidine was purified before being used. The other reagents, 2-hydroxy-1-naphthaldehyde and diphenyltin(IV) dichloride, were utilized without further purification.

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The Schiff base was prepared by direct condensation of 2-hydroxy-1-naphthaldehyde and *o*-toluidine in 95% ethanol and was purified by recrystallization from dichloromethane.

The complex was obtained by adding an equimolar amount of the ligand to diphenyltin(IV) dichloride in dichloromethane. The resulting product was filtered and then recrystallized from dichloromethane. Weissenberg and Buerger precession photographs yielded approximate cell dimensions and showed a monoclinic system with space group $P2_1$ from systematic absence $0k0$, $k = 2n + 1$. Data collection on a crystal of dimensions $0.3 \times 0.4 \times 0.2$ mm was obtained with a Syntex $P2_1$ diffractometer using the $2\theta/\theta$ mode in the index ranges ($0 < h < 10$, $0 < k < 15$, $-10 < l < 10$) to the 2θ limit of 53° . A variable scan speed of $5.0\text{--}29.3^\circ \text{ min}^{-1}$ was used. Lattice parameters refined using 25 reflections in the range $29 < \theta < 35^\circ$. Standard reflection 110 checked every 50 reflections: no significant deviation. The data were corrected for Lorentz and polarization effects. 2824 reflections were collected, 2634 unique ($R_{\text{int}} = 0.1700$), of which 2447 observed reflections with $I > 2.5\sigma(I)$ were used for refinement of the structure. The structure was solved using *SHELXS86* (Sheldrick, 1986) with $R(E) = 0.26$ and refined by the full-matrix least-squares procedure based on F through *SHELX76* (Sheldrick, 1976). Coefficients of scattering factors for all the atoms inlaid in *SHELX76*. Structure determination and refinement performed on an IBM4361/4381. Anisotropic thermal factors for all the non-H atoms, isotropic for H atoms. H atoms were fixed (C—H 0.96 Å) and made riding. In order to correct the observed structure factors for absorption effects, *ABSORB* (Ugozzoli, 1987) was used (correction factors min.—max.: 0.9365–1.0655). The final agreement factors were $R = 0.0704$, $wR = 0.0691$. For completeness, the opposite enantiomorph was also refined and convergence was attained at $R = 0.0700$, $wR = 0.0683$. The ratio of wR factors (1.012) is greater than the value of 1.002 required for the Hamilton (1965) R -factor ratio at a probability level of 99.5%. The preferred chirality was chosen as the one having the lower R value. Weights for the observed structure factors calculated according to $w = 1/[\sigma^2(F) + 0.015834F^2]$, $(\Delta/\sigma)_{\text{max}} = 0.098$, $\Delta\rho_{\text{max}} = 1.38$ and $\Delta\rho_{\text{min}} = -1.62 \text{ e } \text{Å}^{-3}$. Geometrical calculations performed with *XANADU* (Roberts & Sheldrick, 1975) and illustrations drawn with *PLUTO* (Motherwell, 1978). Scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV).

Discussion. Diphenyltin(IV) dichloride has been known to form 1:2 molecular adducts with salicylideneanilines or 2-hydroxynaphthylmethylene-

Table 1. Atomic coordinates ($\times 10^4$) and B_{eq} (Å^2) values for non-H atoms with e.s.d.'s in parentheses

$$B_{\text{eq}} = 8\pi^2(U_{11} + U_{22} + U_{33})/3.$$

	x	y	z	B_{eq}
Sn1	-1034 (1)	0	-8561 (1)	3.80
Cl1	-454 (4)	-1662 (2)	-8100 (4)	6.39
Cl2	-2365 (3)	31 (3)	-6709 (3)	5.47
C22	-3630 (6)	64 (7)	-10772 (7)	4.78
C23	-4405 (6)	-131 (7)	-12052 (7)	5.73
C24	-3820 (6)	-680 (7)	-13012 (7)	6.18
C25	-2461 (6)	-1035 (7)	-12693 (7)	6.47
C26	-1686 (6)	-841 (7)	-11413 (7)	4.37
C21	-2271 (6)	-292 (7)	-10453 (7)	3.91
C32	1449 (8)	1329 (5)	-8813 (9)	5.18
C33	2852 (8)	1614 (5)	-8689 (9)	7.72
C34	3901 (8)	1051 (5)	-7987 (9)	6.62
C35	3548 (8)	204 (5)	-7409 (9)	8.86
C36	2145 (8)	-81 (5)	-7533 (9)	5.54
C31	1096 (8)	481 (5)	-8236 (9)	4.28
O1	-1754 (8)	1487 (5)	-8913 (7)	4.28
C1	-1631 (12)	2261 (8)	-8190 (11)	4.31
C2	-935 (13)	2249 (8)	-6797 (11)	4.75
C3	-784 (13)	3031 (9)	-6073 (10)	4.79
C4	-1218 (12)	3944 (8)	-6609 (11)	4.36
C5	-972 (14)	4767 (10)	-5845 (12)	5.47
C6	-1291 (14)	5595 (9)	-6395 (15)	5.36
C7	-1888 (13)	5666 (10)	-7779 (15)	5.30
C8	-2174 (10)	4881 (8)	-8556 (10)	4.04
C9	-1871 (10)	3988 (8)	-7968 (10)	3.65
C10	-2111 (9)	3114 (7)	-8779 (9)	3.47
C11	-2907 (10)	3135 (7)	-10095 (9)	3.63
N1	-3092 (9)	2420 (6)	-10908 (7)	2.95
C12	-3973 (10)	2387 (7)	-12207 (9)	3.90
C13	-5309 (12)	2788 (8)	-12311 (13)	4.79
C14	-6200 (15)	2708 (13)	-13521 (14)	6.47
C15	-5711 (18)	2292 (12)	-14652 (17)	6.96
C16	-4426 (14)	1913 (9)	-14504 (11)	5.26
C17	-3507 (11)	1960 (7)	-13312 (9)	3.98
C18	-2070 (13)	1555 (10)	-13215 (13)	5.26

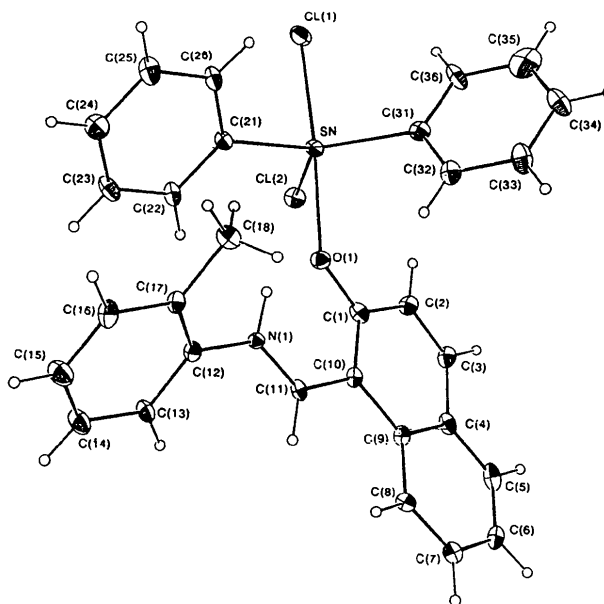


Fig. 1. A perspective view of the molecule.

anilines having *para*-substituents in the aniline fragment (Srivastava & Chauhan, 1977; Saraswat, Srivastava & Mehrotra, 1979; Kamwaya & Khoo, 1985).

Table 2. Selected bond distances (Å) and angles (°) with e.s.d.'s in parentheses for non-H atoms

C11—Sn1	2.464 (3)	C8—C7	1.360 (17)
C12—Sn1	2.346 (2)	C9—C8	1.413 (15)
C21—Sn1	2.102 (6)	C10—C9	1.479 (13)
C31—Sn1	2.138 (7)	C11—C10	1.407 (13)
O1—Sn1	2.244 (7)	N1—C11	1.291 (12)
C1—O1	1.307 (13)	C12—N1	1.429 (10)
C2—C1	1.434 (15)	C13—C12	1.394 (15)
C10—C1	1.398 (15)	C17—C12	1.363 (14)
C3—C2	1.319 (17)	C14—C13	1.369 (16)
C4—C3	1.444 (16)	C15—C14	1.388 (23)
C5—C4	1.395 (17)	C16—C15	1.336 (20)
C9—C4	1.392 (15)	C17—C16	1.367 (14)
C6—C5	1.317 (20)	C18—C17	1.486 (17)
C7—C6	1.401 (20)		
C12—Sn1—C11	90.6 (1)	C6—C5—C4	121.5 (10)
C21—Sn1—C11	93.3 (3)	C7—C6—C5	120.2 (11)
C21—Sn1—C12	112.3 (2)	C8—C7—C6	120.4 (13)
C31—Sn1—C11	95.4 (2)	C9—C8—C7	120.0 (10)
C31—Sn1—C12	119.0 (2)	C8—C9—C4	118.1 (9)
C31—Sn1—C21	127.7 (3)	C10—C9—C4	119.4 (9)
O1—Sn1—C11	174.7 (2)	C10—C9—C8	122.3 (9)
O1—Sn1—C12	85.0 (2)	C9—C10—C1	119.6 (8)
O1—Sn1—C21	85.8 (3)	C11—C10—C1	120.3 (9)
O1—Sn1—C31	89.3 (3)	C11—C10—C9	119.9 (9)
C22—C21—Sn1	121.8 (1)	N1—C11—C10	124.0 (9)
C26—C21—Sn1	118.2 (1)	C12—N1—C11	126.1 (8)
C32—C31—Sn1	119.8 (2)	C13—C12—N1	118.8 (9)
C36—C31—Sn1	120.1 (2)	C17—C12—N1	120.0 (9)
C1—O1—Sn1	135.4 (6)	C17—C12—C13	121.2 (9)
C2—C1—O1	120.0 (10)	C14—C13—C12	119.5 (11)
C10—C1—O1	120.8 (9)	C15—C14—C13	119.0 (9)
C10—C1—C2	119.2 (10)	C16—C15—C14	119.3 (13)
C3—C2—C1	120.2 (10)	C17—C16—C15	123.7 (13)
C4—C3—C2	124.2 (9)	C16—C17—C12	117.1 (10)
C5—C4—C3	123.0 (10)	C18—C17—C12	121.3 (9)
C9—C4—C3	117.3 (10)	C18—C17—C16	121.6 (10)
C9—C4—C5	119.7 (11)		

In the present compound, where the substituent is in the *ortho* position, a 1:1 adduct is formed.

The molecular structure of the compound and the numbering scheme are shown in Fig. 1. Final atomic coordinates are shown in Table 1* while bond distances and angles are given in Table 2.

The title compound has a trigonal bipyramidal geometry about the Sn atom, with one Cl atom lying in the equatorial plane while the phenolic O atom of the ligand and the remaining Cl atom occupy the axial positions. The C(21)—Sn—C(31) angle

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and bond lengths and angles involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53980 (15 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

[127.7 (3)°] is observed to be larger than the average C(Ph)—Sn—Cl(2) angle [115.6 (2)°]. This may be attributed to the stronger repulsion between the two phenyl rings compared to that incurred by Cl(2) and C(Ph).

The Sn—Cl(2) bond length [2.346 (2) Å] is shorter than Sn—Cl(1) [2.464 (3) Å]. This feature can be explained by looking at the average C(Ph)—Sn—Cl(2) angle [115.6 (2)°] which is larger than the average C(Ph)—Sn—Cl(1) angle [94.4 (3)°]. Hence the Cl(1) atom which is situated closer to the phenyl rings is subject to stronger repulsive forces.

The Schiff base is coordinated to the Sn atom in the form of a zwitterion similar to that reported for similar Schiff bases (Bullock, Lad, Povey & Tajmir-Riahi, 1979; Kamwaya & Khoo, 1985). The O(1)—H(N1) distance (1.829 Å) is characteristic of an intramolecular hydrogen bond. The Sn—O bond distance of 2.249 (7) Å is in accord with that reported for 1-[(4-methylimino)methyl]-2-naphthol(in)(IV) (Khoo *et al.*, 1987).

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