Table 2. *Selected bond distances* (Å) *and angles* (°) *for* [PPh4]4[Pb2WSe4)4]

Pb—W(1)	3·679 (1)	W(1)—Se(4)	2·352 (1)
Pb—W(2)	3·769 (1)	W(2)—Se(5)	2·345 (1)
Pb—W(2)'	3·872 (1)	W(2)—Se(6)	2·325 (1)
Pb—Se(3)	2·904 (1)	W(2)—Se(7)	2·324 (1)
Pb—Se(4)	2·942 (1)	W(2)—Se(8)	2·283 (1)
Pb—Se(5)	3·124 (1)	P(1)—C(1)	1·770 (10)
Pb—Se(5)'	3·185 (1)	P(1)—C(7)	1·795 (10)
Pb—Se(6)	2·984 (1)	P(1)—C(13)	1·794 (10)
Pb—Se(7)'	3·121 (1)	P(1)—C(19)	1·803 (10)
W(1)—Se(1)	2·285 (1)	P(2)—C(25)	1·809 (10)
W(1)—Se(2)	2·298 (1)	P(2)—C(31)	1·785 (11)
W(1)—Se(3)	2·343 (1)	P(2)—C(37)	1·813 (10)
		P(2)—C(43)	1·794 (11)

Se(3)—Pb—Se(4)	79·23 (3)	Se(1)—W(1)—Se(4)	110·69 (5)
Se(3)—Pb—Se(5)	169·32 (4)	Se(2)—W(1)—Se(3)	110·33 (5)
Se(3)—Pb—Se(5)'	96·71 (3)	Se(2)—W(1)—Se(4)	112·41 (5)
Se(3)—Pb—Se(6)	93·40 (3)	Se(3)—W(1)—Se(4)	105·12 (4)
Se(3)—Pb—Se(7)'	98·10 (3)	Se(5)—W(2)—Se(6)	107·33 (4)
Se(4)—Pb—Se(5)	102·97 (3)	Se(5)—W(2)—Se(7)	107·76 (4)
Se(4)—Pb—Se(5)'	170·90 (3)	Se(5)—W(2)—Se(8)	111·51 (5)
Se(4)—Pb—Se(6)	96·13 (4)	Se(6)—W(2)—Se(7)	110·46 (5)
Se(4)—Pb—Se(7)	98·91 (3)	Se(6)—W(2)—Se(8)	109·75 (5)
Se(5)—Pb—Se(5)'	82·54 (3)	Se(7)—W(2)—Se(8)	109·98 (4)
Se(5)—Pb—Se(6)	76·01 (3)	W(1)—Se(3)—Pb	88·36 (4)
Se(5)'—Pb—Se(6)	92·22 (3)	W(1)—Se(4)—Pb	87·29 (4)
Se(5)—Pb—Se(7)	91·92 (3)	W(2)—Se(5)—Pb	85·89 (3)
Se(5)'—Pb—Se(7)	73·46 (3)	W(2)—Se(5)—Pb'	87·51 (4)
Se(6)—Pb—Se(7)	162·53 (4)	W(2)—Se(6)—Pb	89·55 (4)
Se(1)—W(1)—Se(2)	108·65 (5)	W(2)—Se(7)—Pb'	89·43 (4)
Se(1)—W(1)—Se(3)	109·59 (5)	Pb—Se(5)—Pb'	97·46 (3)

structure. Each is tetrahedrally coordinated by four Se atoms. One WSe4 tetrahedron is edge shared to a PbSe6 octahedron while the other contributes the Se vertices that are edge shared between PbSe6 octahedra. The distances (Table 2) from Pb to the doubly bridging Se are 2·904 (1) to 3·121 (1) Å, shorter than those to the triply bridging Se atoms 3·124 (1)–

3·185 (1) Å. The angles around Pb vary from 73·46 (3) to 170·90 (3)°; the distortion in part probably arises from the lone-pair electrons on the Pb^{2+} center. The Se—W(1)—Se angles range from 105·12 (4) to 112·41 (5)°, showing slightly wider variation than the Se—W(2)—Se angles [107·33 (4) to 111·51 (5)°]. The distances from W(2) atoms to the triply bridging Se(5) atoms [2·345 (1) Å] are slightly longer than those to the doubly bridging Se(6) [2·325 (1) Å] or Se(7) [2·324 (1) Å] atoms. The terminal W—Se distances of 2·283 (1)–2·289 (1) Å are comparable with those of 2·281 (1)–2·292 (1) Å in the $W_2Se_6^{2-}$ (Lu, Ansari & Ibers, 1989) and 2·281 (1)–2·288 (1) Å in the $NiWSe_6^{2-}$ anions (Ansari, Chau, Mahler & Ibers, 1989).

References

ANSARI, M. A., CHAU, C.-N., MAHLER, C. H. & IBERS, J. A. (1989). *Inorg. Chem.* **28**, 650–654.
ANSARI, M. A. & IBERS, J. A. (1990). *Coord. Chem. Rev.* **100**, 223–266.
LU, Y.-J., ANSARI, M. A. & IBERS, J. A. (1989). *Inorg. Chem.* **28**, 4049–4050.
MÜLLER, A., DIEMANN, E., JOSTES, R. & BÖGGE, H. (1981). *Angew. Chem. Int. Ed. Engl.* **20**, 934–955.
MÜLLER, A., PAULAT-BÖSCHEN, I., KREBS, B. & DORNFIELD, H. (1976). *Angew. Chem. Int. Ed. Engl.* **15**, 633.
WARDLE, R. W. M., MAHLER, C. H., CHAU, C. N. A. & IBERS, J. A. (1988). *Inorg. Chem.* **27**, 2790–2795.
WATERS, J. M. & IBERS, J. A. (1977). *Inorg. Chem.* **16**, 3273–3277.

Structure of Dichloro[1-(4-methoxyphenyl)iminomethyl-2-naphtholato]diphenyltin(IV)

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Abstract. [SnCl2(C18H15NO2)2(C6H5)2], C48H40Cl2N2-O4Sn, M_r = 898·46, triclinic, $P\bar{1}$, a = 10·291 (1), b = 12·332 (1), c = 8·895 (1) Å, α = 70·62 (1), β = 80·30 (1), γ = 72·62 (1)°, V = 1013 (1) Å³, Z = 1, D_m = 1·451 (flotation), D_x = 1·473 Mg m⁻³, λ(Mo Kα) = 0·71069 Å, μ = 0·728 mm⁻¹, F(000) = 458, T =

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295 K, $R = 0.0415$ for 2928 observed reflections, $I > 2.5\sigma(I)$. The complex possesses octahedral geometry and *trans* configuration of ligands about the Sn atom which is bonded to two Cl atoms, two phenyl groups and two 2-hydroxy-1-naphthalideneanisidine moieties. The H(N)···O distance of 1.874 Å shows the presence of a hydrogen bond. There is also an observed shift of the phenolic proton of the ligand to the imine N atom.

Introduction. The coordinative interactions of organotin(IV) halides with 2-hydroxy-1-napthalideneaniline having different substituents in the aniline fragment have attracted considerable attention in recent years.

From infrared studies of the 1:2 adducts of such interactions, Srivastava & Chauhan (1977) inferred that coordination occurs at the azomethine N atom present in the ligand. A similar mode of coordination is apparently also observed when the ligand interacts with tin(II) halides (Dwivedi, Bhatnagar & Srivastava, 1986).

However, crystallographic studies by Charland, Lee, Gabe, Khoo & Smith (1987) have refuted the above proposal and demonstrated that only the phenolic O atom is coordinated to the Sn atom. These workers further showed that the 3:2 adduct obtained from the interaction of triphenyltin halide with the ligand has a five-coordinate trigonal bipyramidal structure.

Similar work involving tin tetrachloride and mono- and diorganotin halide with salicyclideneaniline (Saraswat, Srivastava & Mehrotra, 1979; Kamwaya & Khoo, 1985) have resulted in the formation of 1:2 adducts having an essentially octahedral geometry about the tin atom.

In this paper we report a full structural investigation of the title adduct.

Experimental. An aqueous mixture of Ph_2SnCl_2 was prepared by dissolving 2.5 mmol (0.8595 g) of Ph_2SnCl_2 in CH_2Cl_2. The solution was then mixed with 5.0 mmol (1.3865 g) of 1-(4-methoxyphenyl-imino)methyl-2-naphthol which was originally dissolved in dichloromethane. The final solution was then filtered and allowed to stand. Red crystals of dichloro-1-(4-methoxyphenyl)iminomethyl-2-naphtholato]diphenyltin(IV) were obtained.

Preliminary Weissenberg and Buerger precession photographs yielded approximate cell dimensions and showed triclinic symmetry. Data collection on crystal $0.5 \times 0.5 \times 0.2$ mm, Syntex $P2_1$ diffractometer, $2\theta/\theta$ scan with data index $h -12 \rightarrow 12$, $k -10 \rightarrow 10$, $l 0 \rightarrow 9$; to a 2θ limit of 52°. Variable scan speed of 5.0–$29.3°$ min^{-1}. One standard reflection (132) checked every 15 reflections: no significant deviation. Data corrected for Lorentz and polari-

zation effects. 3626 reflections were collected, 3389 unique ($R_{int} = 0.0435$), of which 2928 observed reflections with $I > 2.5\sigma(I)$ were used for structure refinement.

Structure solved using *SHELXS*86 (Sheldrick, 1986) and refined using *SHELX*76 (Sheldrick, 1976) with coefficients of scattering factors included in *SHELX*76. Space group $P\bar{1}$ confirmed from successful solution and refinement of structure. Structure determination and refinement performed on IBM 4361/4381. Refinement by full-matrix least squares based on F with idealized riding H atoms (C—H 0.96 Å); anisotropic refinement for non-H atoms, isotropic refinement for H atoms. In order to correct the observed structure factors for absorption effects, program *ABSORB* (Ugozzoli, 1987) was used (max. and min. correction factors were 1.3790 and 0.6193 respectively). Final agreement factors were $R = 0.0415$, $wR = 0.0441$. Weight for every observed structure factor calculated according to $w = 12.033/[\sigma^2(F) + 0.000058|F|^2]$. $(\Delta/\sigma)_{max} = 1.298$, $\Delta\rho_{max} = 0.794$, $\Delta\rho_{min} = -0.699$ e Å^{-3}; no extinction correction. Geometrical calculations performed with *XANADU* (Roberts & Sheldrick, 1979) and illustration drawn with *ORTEP* (Johnson, 1965).

Discussion. The final atomic coordinates and equivalent isotropic thermal parameters for the non-H atoms are given in Table 1.* Bond distances and angles are listed in Table 2. Fig. 1 shows a perspective view of a molecule.

The complex possesses octahedral geometry and a *trans* configuration of ligands about the Sn atom which is bonded to two Cl atoms, two phenyl groups and two 2-hydroxy-1-naphthalideneanisidine moieties. The coordination bond angles involving the equivalent *trans* ligands are 180° while the other angles vary from 84.2 to 95.2°.

The Sn—C (Ph) distance of 2.147 (4) Å is within the range of Sn—C (Ph) distances (2.105–2.160 Å) reported in the literature (Harrison, King & Richards, 1974) while the Sn—O bond length of 2.228 (3) Å is close to that found in the Me_2SnCl_2-salicylideneaniline adduct (2.262 Å; Kamwaya & Khoo, 1985).

One salient feature of the structure is the observed shift of the phenolic proton of the ligand to the imine N atom. As such, the Schiff-base ligand is related to tin in the form of a zwitterion. Such proton transfer has been similarly observed elsewhere (Gabe, Lee,

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* Lists of structure factors, anisotropic thermal parameters, bond distances and angles involving H atoms and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53931 (16 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

$$[SnCl_2(C_{18}H_{15}NO_2)_2(C_6H_5)_2]$$

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

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

	x	y	z	B_{eq}
Sn(1)	5000	5000	5000	4·24
Cl(1)	6586 (1)	4450 (1)	7206 (1)	5·60
O(1)	6888 (3)	5108 (3)	3374 (4)	5·27
C(1)	4451 (3)	6827 (4)	4976 (5)	4·76
C(2)	4496 (5)	7104 (5)	6349 (6)	5·69
C(3)	4104 (5)	8274 (5)	6349 (7)	6·81
C(4)	3676 (5)	9179 (5)	5014 (8)	7·02
C(5)	3629 (6)	8919 (5)	3663 (8)	7·19
C(6)	4004 (5)	7749 (4)	3632 (6)	5·74
C(7)	7375 (4)	5744 (4)	2066 (5)	4·54
C(8)	8793 (4)	5675 (3)	1817 (5)	3·96
C(9)	9327 (4)	6452 (4)	392 (5)	4·48
C(10)	8430 (5)	7240 (4)	−778 (5)	5·06
C(11)	7049 (5)	7204 (5)	−521 (6)	6·11
C(12)	6535 (4)	6511 (4)	813 (6)	5·97
C(13)	10692 (5)	6495 (4)	97 (5)	5·46
C(14)	11145 (6)	7246 (5)	−1248 (6)	6·68
C(15)	10262 (7)	8036 (5)	−2382 (6)	6·95
C(16)	8918 (6)	8011 (5)	−2154 (6)	6·40
C(17)	9705 (4)	4808 (4)	2911 (5)	4·18
N(1)	9372 (3)	4087 (3)	4237 (4)	4·36
C(18)	10267 (4)	3141 (3)	5305 (5)	4·19
C(19)	11666 (4)	3031 (4)	5170 (5)	4·70
C(20)	12478 (4)	2090 (4)	6239 (6)	5·24
C(21)	11926 (5)	1288 (4)	7425 (5)	5·43
C(22)	10548 (5)	1383 (5)	7543 (6)	6·31
C(23)	9728 (4)	2318 (4)	6481 (6)	5·79
O(2)	12814 (5)	389 (4)	8405 (5)	8·27
C(24)	12539 (14)	−303 (10)	9559 (11)	18·56

Table 2. *Bond distances* $(\mathring{A})$ *and angles* $(°)$ *with e.s.d.'s in parentheses for non-H atoms*

Cl(1)—Sn(1)	2·552 (1)	C(11)—C(10)	1·413 (7)
C(1)—Sn(1)	2·228 (3)	C(16)—C(10)	1·400 (7)
C(1)—Sn(1)	2·147 (4)	C(12)—C(11)	1·343 (7)
C(7)—O(1)	1·286 (5)	C(14)—C(13)	1·357 (6)
C(2)—C(1)	1·384 (6)	C(15)—C(14)	1·386 (8)
C(6)—C(1)	1·380 (6)	C(16)—C(15)	1·372 (8)
C(3)—C(2)	1·378 (7)	N(1)—C(17)	1·284 (5)
C(4)—C(3)	1·361 (7)	C(18)—N(1)	1·425 (5)
C(5)—C(4)	1·354 (8)	C(19)—C(18)	1·392 (5)
C(6)—C(5)	1·387 (7)	C(23)—C(18)	1·367 (6)
C(8)—C(7)	1·420 (5)	C(20)—C(19)	1·380 (6)
C(12)—C(7)	1·421 (6)	C(21)—C(20)	1·362 (6)
C(9)—C(8)	1·449 (6)	C(22)—C(21)	1·376 (6)
C(17)—C(8)	1·411 (5)	O(2)—C(21)	1·359 (5)
C(10)—C(9)	1·413 (6)	C(23)—C(22)	1·378 (6)
C(13)—C(9)	1·399 (6)	C(24)—O(2)	1·152 (12)

| C(1)—Sn(1)—Cl(1)	84·2 (1)	C(16)—C(10)—C(9)	120·3 (4)
C(1)—Sn(1)—Cl(1)	90·0 (1)	C(16)—C(10)—C(11)	121·8 (4)
C(1)—Sn(1)—O(1)	95·2 (1)	C(12)—C(11)—C(10)	123·4 (4)
C(7)—O(1)—Sn(1)	143·5 (2)	C(11)—C(12)—C(7)	121·1 (4)
C(2)—C(1)—Sn(1)	120·0 (3)	C(14)—C(13)—C(9)	122·1 (5)
C(6)—C(1)—Sn(1)	121·7 (3)	C(15)—C(14)—C(13)	121·3 (5)
C(6)—C(1)—C(2)	118·3 (4)	C(16)—C(15)—C(14)	118·5 (5)
C(3)—C(2)—C(1)	120·0 (5)	C(15)—C(16)—C(10)	121·0 (5)
C(4)—C(3)—C(2)	121·4 (5)	N(1)—C(17)—C(8)	125·9 (3)
C(5)—C(4)—C(3)	119·1 (5)	C(18)—N(1)—C(17)	127·3 (3)
C(6)—C(5)—C(4)	120·8 (5)	C(19)—C(18)—N(1)	121·8 (4)
C(5)—C(6)—C(1)	120·4 (5)	C(23)—C(18)—N(1)	118·5 (3)
C(8)—C(7)—O(1)	120·3 (4)	C(23)—C(18)—C(19)	119·7 (4)
C(12)—C(7)—O(1)	121·9 (4)	C(20)—C(19)—C(18)	119·0 (4)
C(12)—C(7)—C(8)	117·8 (4)	C(21)—C(20)—C(19)	120·9 (4)
C(9)—C(8)—C(7)	120·3 (3)	C(22)—C(21)—C(20)	120·2 (4)
C(17)—C(8)—C(7)	120·4 (4)	O(2)—C(21)—C(20)	116·3 (4)
C(17)—C(8)—C(9)	119·3 (3)	O(2)—C(21)—C(22)	123·4 (5)
C(10)—C(9)—C(8)	119·2 (4)	C(23)—C(22)—C(21)	119·3 (4)
C(13)—C(9)—C(8)	124·1 (4)	C(22)—C(23)—C(18)	120·9 (4)
C(13)—C(9)—C(10)	116·7 (4)	C(24)—O(2)—C(21)	126·7 (8)
C(11)—C(10)—C(9)	117·8 (4)		
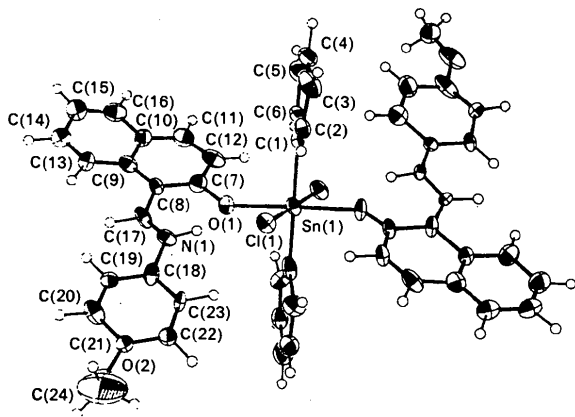
Fig. 1. A perspective view of the molecule.

Khoo & Smith, 1985, 1986; Khoo, Charland, Gabe & Smith, 1987).

A six-membered ring is formed as a result of the intramolecular hydrogen bonding. The H(N)···O distance of 1·874 Å shows the presence of a hydrogen bond. Protonation of the nitrogen effectively prevents that atom from any coordinative interaction with the tin atom in all cases where such a phenomenon is observed.

References

CHARLAND, J. P., LEE, F. L., GABE, J., KHOO, L. E. & SMITH, F. E. (1987). *Inorg. Chim. Acta*, **130**, 55–60.

DWIVEDI, B. K., BHATNAGAR, K. & SRIVASTAVA, A. K. (1986). *Synth. React. Inorg. Met. Org. Chem.* **16**(6), 841–855.

GABE, E. J., LEE, F. L., KHOO, L. E. & SMITH, F. E. (1985). *Inorg. Chim. Acta*, **105**, 103–106.

GABE, E. J., LEE, F. L., KHOO, L. E. & SMITH, F. E. (1986). *Inorg. Chim. Acta*, **112**, 41–46.

HARRISON, P. G., KING, T. J. & RICHARDS, J. A. (1974). *J. Chem. Soc. Dalton Trans.* pp. 1723–1729.

JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.

KAMWAYA, M. E. & KHOO, L. E. (1985). *Acta Cryst.* C41, 1027–1029.

KHOO, L. E., CHARLAND, J. P., GABE, E. J. & SMITH, F. E. (1987). *Inorg. Chim. Acta*, **128**, 139–145.

ROBERTS, P. & SHELDRICK, G. M. (1979). *XANADU*. Program for crystallographic calculations. Univ. of Cambridge, England.

SARASWAT, B. S., SRIVASTAVA, G. & MEHROTRA, R. C. (1979). *J. Organomet. Chem.* **164**, 153–158.

SHELDRICK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

SHELDRICK, G. M. (1986). *SHELXS86*. Program for the solution of crystal structures. Univ. of Göttingen, Germany.

SRIVASTAVA, T. N. & CHAUHAN, A. K. S. (1977). *Synth. React. Inorg. Met. Org. Chem.* **7**(4), 373–385.

UGOZZOLI, G. (1987). *Comput. Chem.* **2**, 109–120.