CRYSTAL AND MOLECULAR STRUCTURE OF CHLORODIMETHYLTINBIS(p-CHLOROBENZOYLHYDRAZINE) CHLORIDE p-CHLOROBENZOYLHYDRAZINE: A DIORGANOTIN CATION WITH PENTAGONAL BIPYRAMIDAL COORDINATION

Swarnali Basu Baul,[1] Tushar S. Basu Baul[1,2] and Edward R.T. Tiekink*[2]

[1] Chemical Laboratory, Regional Sophisticated Instrumentation Centre,
North-Eastern Hill University, Bijni Complex, Shillong 793 003, India
[2] Department of Chemistry, The University of Adelaide, Adelaide 5005, Australia

Abstract.

The crystal structure of the title compound, $[Me_2Sn(LH)_2Cl]Cl.LH$, shows the tin atom in the cation to exist in a distorted pentagonal bipyramidal geometry. The two *p*-chlorobenzoylhydrazine ligands chelate the tin atom, via the carbonyl-O and amino-N atoms, and the pentagonal plane is completed by a chlorine atom that occupies a position between the two nitrogen donors; the tin-bound methyl substituents occupy axial positions. The structure is stabilised by an extensive hydrogen bonding network which links the ions as well as the uncoordinated *p*-chlorobenzoylhydrazine molecule.

Introduction.

N-Aroyl-hydrazines have received considerable attention owing to their versatility as coordination agents [1 - 3] as well as their antibacterial activity [4 - 6]. In the latter context, the incorporation of various substituents into the acid hydrazides has a marked effect on their biological activity [7]. Further, the activity of some of the biologically active hydrazines has been shown to increase upon coordination to metal ions [8]. It was with this background that the present investigation of the interaction of various acid hydrazines with organotin centres was commenced [9]. The recrystallisation (methanol solution) of an authenticated sample of $[Me_2Sn(LH)Cl_2]$, where LH is $NH_2N(H)C(O)C_6H_4Cl$-*p*, yielded crystals of the title compound, hereafter $[Me_2Sn(LH)_2Cl]Cl.LH$, details of its spectroscopic characterisation and crystal structure analysis are reported herein.

Experimental.

Preparation

$[Me_2Sn(LH)Cl_2]$ A methanolic solution of Me_2SnCl_2 (0.25 g, 20 cm^3) was added slowly to a hot methanol solution of p-chlorobenzoylhydrazine (LH, 0.20 g, 20 cm^3) under stirring for 1 h. The solvent was removed under reduced pressure, the precipitate was washed with a mixture of CH_2Cl_2 and hexane; the cream-coloured product was dried *in* *vacuo*. Yield 85 %, m. pt 179 - 180 °C. Analytical data (Hereaeus elemental analyser), found: C, 27.50; H, 3.40; N, 7.20 %. Calcd for $C_9H_{13}Cl_3N_2OSn$: C, 27.68, H, 3.33; N, 7.17 %. Infrared spectrum (Perkin-Elmer 1720X FT spectrometer; KBr disc): amide I υ(C=O) 1646, amide II (υCN + δNH) 1540, amide III (δNH) 1332 and υ(N-N) 1012 cm^{-1}. ^1H NMR ($CDCl_3$/DMSO-d_6 (1/4) ACF300 NMR spectrometer; TMS internal lock; see Fig. 1 for numbering scheme): δ 7.88 (2H, d, 8.4 Hz, H3, 7), 7.41 (2H, d, 8.4 Hz, H4, 6) and 1.18 ppm (6H, Sn-Me, $^2J(^1H-^{117}Sn)$ 92.1 Hz, $^2J(^1H-^{119}Sn)$ 96.0 Hz). ^{13}C NMR ($CDCl_3$/DMSO-d_6 (1/4); $CDCl_3$ internal lock): δ 166.0 (C=O), 136.9 (C2, 5), 128.3 (C3, 7), 127.9 (C4, 6) and 16.2 ppm (Sn-C). Tin-119*m* Mössbauer parameters [10]: IS 1.45, QS 4.39, Γ_1 0.89, Γ_2 0.84 mm s^{-1}.

Colourless crystals of $[Me_2Sn(LH)_2Cl]Cl.LH$, which were suitable for the X-ray analysis, were obtained from the attempted recrystallisation of $[Me_2Sn(LH)Cl_2]$ from methanol solution; m. pt. 198 - 199 °C. Characteristic bands were observed in the infrared spectrum (Perkin-Elmer 1720X FT spectrometer; KBr disc): amide I υ(C=O) 1644, amide II (υCN + δNH) 1561, amide III (δNH) 1338 and υ(N-N) 1015 cm^{-1}. ^1H NMR ($CDCl_3$/DMSO-d_6; ACP300 NMR spectrometer; TMS internal lock): δ 7.84 (6H, d, 8.4 Hz, H3, 7), 7.39 (6H, d, 8.4 Hz, H4, 6) and 1.17 ppm (6H, Sn-Me, $^2J(^1H-^{117}Sn)$ 103.0 Hz, $^2J(^1H-^{119}Sn)$ 107.1 Hz). ^{13}C NMR ($CDCl_3$/DMSO-d_6 (1/4); $CDCl_3$ internal lock): δ 164.3 (C=O), 135.3 (C1), 18.4 (Sn-C), other C 129.9, 127.7, 127.1, 126.8 and 125.8 ppm.

Crystallography

Intensity data for a colourless crystal (0.06 x 0.22 x 0.42 mm) were measured at room temperature on a Rigaku AFC6R four-circle diffractometer fitted with graphite-monochromatised MoKα radiation, λ = 0.7107 Å. A total of 7646 data were measured (ω:2θ scan technique and θ_{max} was 27.5°), 7171 were unique and of these 2943 satisfied the $I \geq 3.0\sigma(I)$ criterion of observability and were used in the subsequent analysis. The data were corrected for Lorentz and polarization effects [11] and for absorption employing the DIFABS program [12] which resulted in a range of transmission coefficients of 0.982 to 1.

Crystal data for $[Me_2Sn(LH)_2Cl]Cl.LH$: $C_{23}H_{27}Cl_5N_6O_3Sn$, M = 731.5, monoclinic, space group $P2_1/c$, a = 16.898(2), b = 22.007(3), c = 8.338(2) Å, β = 102.85(1)°, V = 3023.0(8) $Å^3$, Z = 4 , D_{calcd} = 1.607 g cm^{-3}, $F(000)$ = 1464, μ = 13.22 cm^{-1}.

The structure was solved by direct methods [13] and refined by a full-matrix least-squares procedure based on F [11]. The non-H atoms were refined with anisotropic thermal parameters and the H atoms were included in the model at their calculated positions (C-H 0.97, N-H 0.95 Å) except for those bound to the N(1c) atom which were located from an analysis of a difference map. At convergence R = 0.042 and R_w = 0.036 (sigma weights, i.e. $1/\sigma^2(F)$ [11]).* The final fractional atomic coordinates are listed

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* where $R = \Sigma||F_o| - |F_c|| / \Sigma|F_o|$ and $R_w = [\Sigma w(|F_o| - |F_c|)^2 / \Sigma w F_o^2]^{1/2}$

in Table 1 and the crystallographic numbering scheme used is shown in Figure 1, which was drawn with ORTEP [14] at 50% probability ellipsoids. Data manipulation was performed with the teXsan package [11] installed on an Iris Indigo workstation.

Table 1. Fractional atomic coordinates for $[Me_2Sn(LH)_2Cl]Cl.LH$

Atom	x	y	z
Sn	0.11062(3)	0.07251(2)	0.30480(6)
Cl(1)	-0.0400(1)	0.0481(1)	0.2881(2)
Cl(2)	0.0104(1)	-0.1671(1)	0.1899(2)
Cl(5a)	0.4411(1)	0.3842(1)	0.5674(4)
Cl(5b)	0.5911(1)	-0.1094(1)	0.3884(4)
Cl(5c)	0.3441(2)	-0.0308(1)	-0.1470(3)
O(1a)	0.2026(3)	0.1512(2)	0.2844(6)
O(1b)	0.2406(3)	0.0300(2)	0.3490(6)
O(1c)	0.2175(3)	-0.3114(2)	-0.0420(7)
N(1a)	0.0460(3)	0.1706(2)	0.2241(7)
N(1b)	0.1159(3)	-0.0382(2)	0.3784(7)
N(1c)	0.0855(3)	-0.3160(2)	0.0938(7)
N(2a)	0.1024(4)	0.2188(2)	0.2668(7)
N(2b)	0.1950(3)	-0.0627(2)	0.3935(7)
N(2c)	0.1284(4)	-0.2630(2)	0.0717(7)
C(1a)	0.1814(5)	0.2042(3)	0.2998(8)
C(1b)	0.2542(4)	-0.0247(3)	0.3726(8)
C(1c)	0.1914(4)	-0.2635(3)	0.0014(9)
C(1)	0.1340(5)	0.0951(3)	0.5553(9)
C(2)	0.1033(4)	0.0477(3)	0.0575(8)
C(2a)	0.2423(4)	0.2533(3)	0.3572(8)
C(2b)	0.3364(4)	-0.0497(3)	0.3784(9)
C(2c)	0.2286(4)	-0.2042(3)	-0.0245(9)
C(3a)	0.3223(5)	0.2368(3)	0.3720(11)
C(3b)	0.3643(5)	-0.1037(4)	0.4491(13)
C(3c)	0.2995(5)	-0.2058(3)	-0.0795(11)
C(4a)	0.3837(5)	0.2780(4)	0.4338(12)
C(4b)	0.4426(6)	-0.1225(4)	0.4491(14)
C(4c)	0.3368(5)	-0.1526(4)	-0.1142(13)
C(5a)	0.3632(5)	0.3338(3)	0.4807(10)
C(5b)	0.4926(5)	-0.0867(4)	0.3797(11)
C(5c)	0.3002(5)	-0.0979(4)	-0.0962(10)
C(6a)	0.2851(5)	0.3513(3)	0.4649(10)
C(6b)	0.4650(5)	-0.0334(4)	0.3096(12)
C(6c)	0.2301(5)	-0.0951(3)	-0.0422(10)
C(7a)	0.2238(4)	0.3103(3)	0.4014(10)
C(7b)	0.3865(5)	-0.0142(4)	0.3086(11)
C(7c)	0.1960(4)	-0.1485(3)	-0.0042(10)

Results and Discussion.

The [$Me_2Sn(LH)Cl_2$] compound has been prepared from the reaction between Me_2SnCl_2 and LH in methanol solution. The ^1H and ^{13}C NMR spectra showed the expected resonances and multiplicities (see Experimental). In the infrared spectrum evidence for the participation in coordination of the carbonyl and amino groups is indicated by shifts in certain absorptions compared with the free ligand consistent with previous work [1, 15 - 17]. In [$Me_2Sn(LH)Cl_2$] the amide I - III bands have been shifted to lower frequency, compared to the free ligand, suggesting coordination of the carbonyl group [18 - 19] and the band due to υ(N-N) at 994 cm^{-1} in the free ligand has shifted to higher frequency (i.e. 1012 cm^{-1}) in the compound indicating coordination of the -NH_2 group [3, 15]. The Mössbauer data show the characteristic doublet with Γ_1 0.89 and Γ_2 0.84 mm s^{-1}, indicating the presence of a single tin site and the QS value of 4.39 mm s^{-1} shows a *trans*-Me_2Sn arrangement with a calculated C-Sn-C angle of 180°. Thus, in summary, the spectroscopic data indicate an octahedrally coordinated tin atom with *trans*-Me groups and *cis*-Cl. The attempted recrystallisation of [$Me_2Sn(LH)Cl_2$] from methanol solution yielded a small quantity of crystals which have been formulated as [$Me_2Sn(LH)_2Cl$]Cl.LH on the basis of spectroscopic and crystallographic analyses.

The ^1H NMR spectrum of [$Me_2Sn(LH)_2Cl$]Cl.LH is consistent with the formulation of the compound, i.e. three ligands and two tin-bound Me groups. The resonances did not show multiplicity that could be interpreted in terms of the presence of both free LH and coordinated L which could indicate rapid exchange between the bound and unbound ligands in solution; no evidence was found for δ N-H. A similar situation pertains in the ^{13}C NMR spectrum for which only single resonances were observed for the C=O, C(1) and Me carbons. In the infrared spectrum shifts in the amide I - III and υ(N-N) bands indicate coordination of the carbonyl and -NH_2 groups (see above). A full characterisation of the structure was afforded by a crystal structure analysis.

The molecular structure of the cation in [$Me_2Sn(LH)_2Cl$]Cl.LH is shown in Figure 1 and selected interatomic parameters for the structure are listed in Table 2. The structure is comprised of [$Me_2Sn(LH)_2Cl$]$^+$ cations, Cl^- anions and neutral LH molecules in the ratio 1:1:1. The lattice is stabilised via hydrogen bonding contacts involving all species. In the cation, the tin atom is chelated by two coplanar LH ligands, each of which coordinate via the carbonyl-O and amino-N atoms thereby forming five-membered rings. The arrangement of the two bidentate ligands is such that the oxygen atoms occupy adjacent positions (O-Sn-O 72.2(2)°) with the nitrogen atoms opposite to each other (N-Sn-N 156.1(2)°). The coordination environment about the tin atom is completed by a chloride atom and two carbon atoms of the tin-bound methyl substituents. The overall geometry is based on a pentagonal bipyramid with the two methyl groups defining the axial positions; C(1)-Sn-C(2) 172.7(3)°. The deviations of the Cl(1), O(1a), O(1b), N(1a) and N(2a) atoms from the least-squares plane through them are -0.000(2), 0.068(5), -0.075(5), -0.057(5) and 0.037(5) Å, respectively with the tin atom being effectively coplanar. There are no major distortions from the ideal geometry with the angles around the basal plane deviating by no more than ±6°. The mode of coordination of the two LH ligands is essentially equivalent, there being few significant differences in comparable geometric parameters. The major difference between the coordinated ligands is found in the Sn-O(1)-C(1) angles of 118.3(4) and 123.3(4)°, respectively for ligands *a* and *b*. The five-membered chelate rings that form upon coordination are each essentially planar as seen in the O(1)/C(1)/N(2)/N(1) torsion angles of 4(1) and 3(1)°, respectively; the comparable angle in the non-coordinating ligand is -3(1)°. The chelate angle is 66.0(2)° in each case. There are some notable differences in the derived interatomic parameters between the coordinated and non-coordinated LH ligands with evidence for elongation of both the C(1)-O(1) and N(1)-N(2) bonds upon coordination. Further, the C(1)-N(2)-N(1) angle is significantly wider in the free ligand, i.e. 122.6(6)° *cf.* 117.1(5) and 117.7(5)°, reflecting additional strain owing to the formation of the five-membered chelate rings. As expected there are significant intermolecular contacts in the lattice.

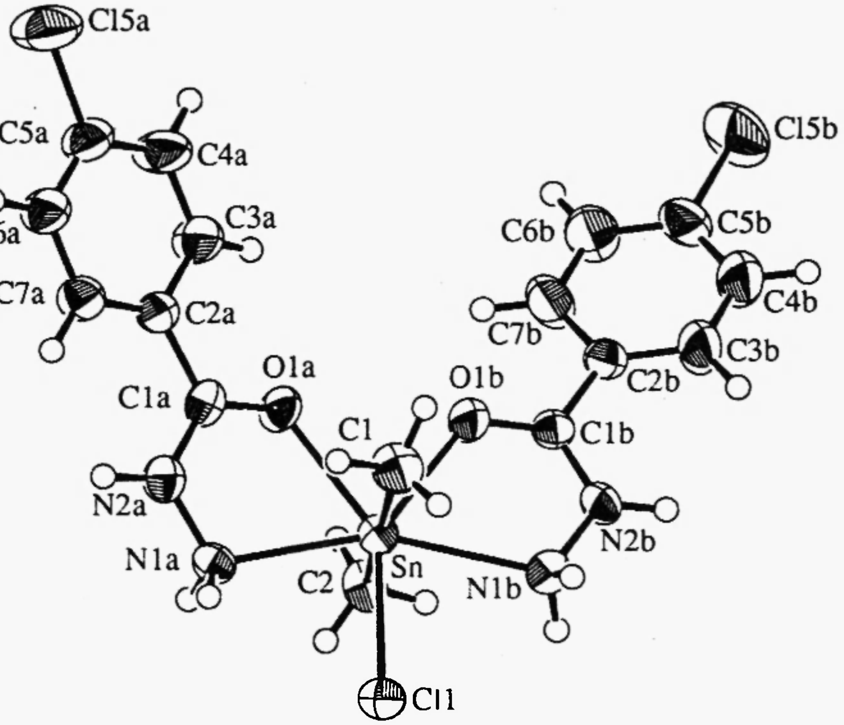

Figure 1. Molecular structure and crystallographic numbering scheme for the cation in $[Me_2Sn(LH)_2Cl]Cl.LH$

A [001] projection of the unit cell contents for $[Me_2Sn(LH)_2Cl]Cl.LH$ is shown in Figure 2 with the association between the various components occurring along the crystallographic *b*-axis. In the lattice, layers of associated molecules are separated from adjacent layers by regions occupied by the *p*-chlorophenyl groups. There is no evidence of alignment of the aromatic rings which are separated in any case by approximately *c*/2 Å, thereby precluding significant π-interactions between them. Significant hydrogen bonding contacts are listed in Table 3. The tin-bound Cl(1) atom forms two weak

Table 2. Selected bond distances (Å) and angles (°) for $[Me_2Sn(LH)_2Cl]Cl.LH$

Sn-Cl(1)	2.575(2)	Sn-O(1a)	2.358(4)
Sn-O(1b)	2.340(5)	Sn-N(1a)	2.445(5)
Sn-N(1b)	2.510(5)	Sn-C(1)	2.098(7)
Sn-C(2)	2.110(7)	Cl(5a)-C(5a)	1.751(8)
Cl(5b)-C(5b)	1.725(8)	Cl(5c)-C(5c)	1.746(8)
O(1a)-C(1a)	1.235(7)	O(1b)-C(1b)	1.233(7)
O(1c)-C(1c)	1.227(7)	N(1a)-N(2a)	1.418(7)
N(1b)-N(2b)	1.420(7)	N(1c)-N(2c)	1.406(7)
N(2a)-C(1a)	1.340(8)	N(2b)-C(1b)	1.345(8)
N(2c)-C(1c)	1.324(8)	C(1a)-C(2a)	1.496(9)
C(1b)-C(2b)	1.486(9)	C(1c)-C(2c)	1.486(9)
Cl(1)-Sn-O(1a)	144.0(1)	Cl(1)-Sn-O(1b)	143.8(1)
Cl(1)-Sn-N(1a)	78.1(1)	Cl(1)-Sn-N(1b)	78.0(1)
Cl(1)-Sn-C(1)	93.9(2)	Cl(1)-Sn-C(2)	92.9(2)
O(1a)-Sn-O(1b)	72.2(2)	O(1a)-Sn-N(1a)	66.0(2)
O(1a)-Sn-N(1b)	138.0(2)	O(1a)-Sn-C(1)	85.2(2)
O(1a)-Sn-C(2)	90.9(2)	O(1b)-Sn-N(1a)	137.9(2)
O(1b)-Sn-N(1b)	66.0(2)	O(1b)-Sn-C(1)	88.6(3)
O(1b)-Sn-C(2)	84.2(2)	N(1a)-Sn-N(1b)	156.1(2)
N(1a)-Sn-C(1)	92.2(2)	N(1a)-Sn-C(2)	91.9(2)
N(1b)-Sn-C(1)	89.9(2)	N(1b)-Sn-C(2)	88.8(2)
C(1)-Sn-C(2)	172.7(3)	Sn-O(1a)-C(1a)	118.3(4)
Sn-O(1b)-C(1b)	123.3(4)	Sn-N(1a)-N(2a)	110.9(4)
Sn-N(1b)-N(2b)	111.8(4)	N(1a)-N(2a)-C(1a)	117.1(5)
N(1b)-N(2b)-C(1b)	117.7(5)	N(1c)-N(2c)-C(1c)	122.6(6)
O(1a)-C(1a)-N(2a)	120.3(6)	O(1a)-C(1a)-C(2a)	121.4(7)
N(2a)-C(1a)-C(2a)	118.3(6)	O(1b)-C(1b)-N(2b)	120.8(6)
O(1b)-C(1b)-C(2b)	120.4(6)	N(2b)-C(1b)-C(2b)	118.8(6)
O(1c)-C(1c)-N(2c)	121.0(7)	O(1c)-C(1c)-C(2c)	121.3(7)
N(2c)-C(1c)-C(2c)	117.7(6)	C(1a)-C(2a)-C(3a)	115.4(6)
C(1b)-C(2b)-C(3b)	124.6(7)	C(1c)-C(2c)-C(3c)	117.0(6)
C(1a)-C(2a)-C(7a)	124.7(7)	C(1b)-C(2b)-C(7b)	115.8(7)
C(1c)-C(2c)-C(7c)	125.0(6)		

*intra*molecular contacts as well as two *inter*molecular contacts. The chloride anion, Cl(2), also forms four weak contacts ranging from 2.34 Å to 2.43 Å. The most significant hydrogen bonding contact in the lattice occurs between the carbonyl group of the non-coordinating ligand and the only N-H atom that is not associated with a chloride atom, i.e. the O(1c) and H(23b)-N(2b) atoms.

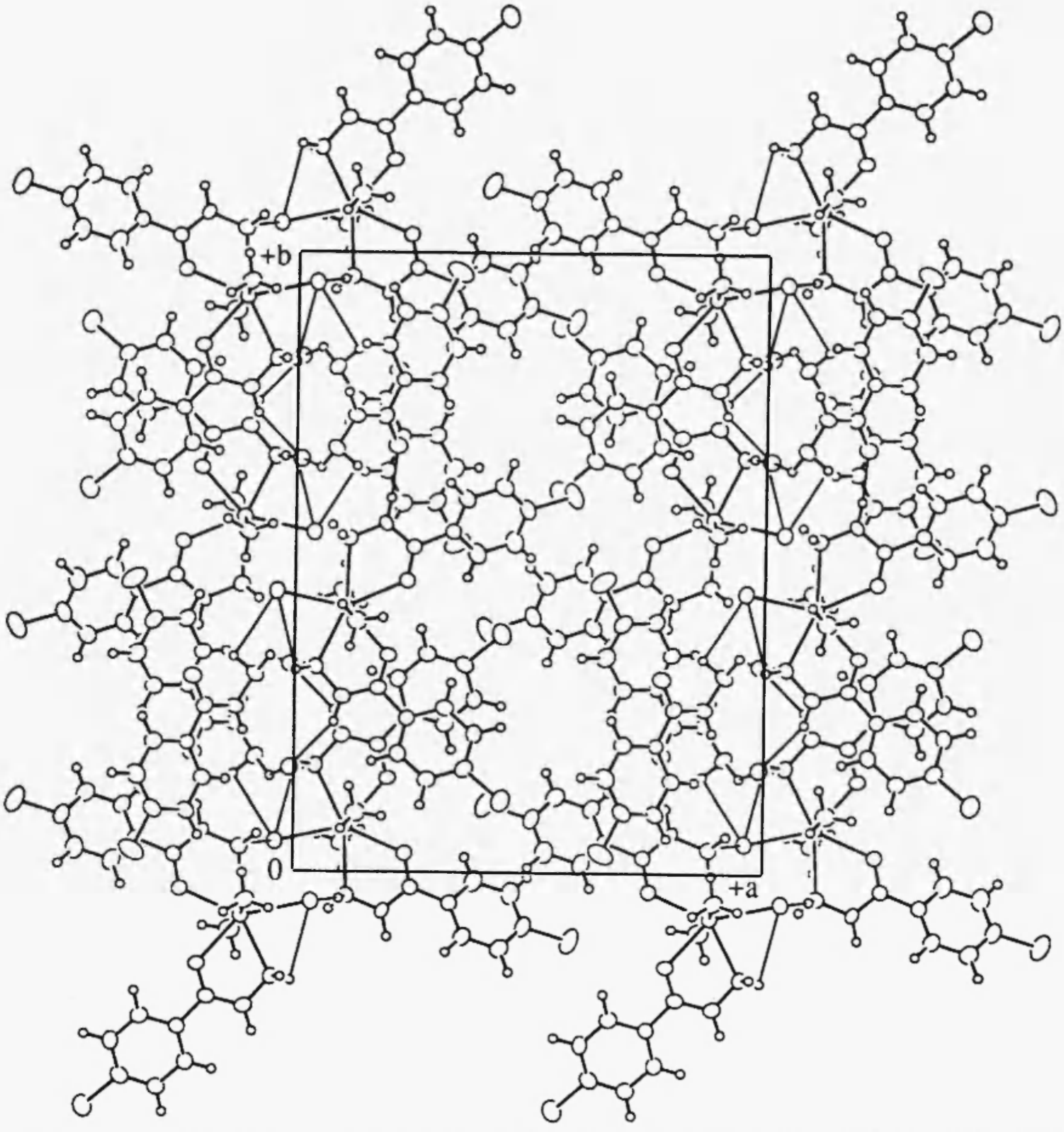

Figure 2. A [001] projection of the unit cell contents for $[Me_2Sn(LH)_2Cl]Cl.LH$

The pentagonal bipyramidal geometry found in the present study is not without precedence in the literature; all structures feature a *trans*-C_2Sn arrangement. Neutral species containing $N_2O_3C_2$ [20, 21], $N_3Cl_2C_2$ [22] and O_5C_2 [23] donor sets are known for example. Anionic species have been characterized with an O_4ClC_2 donor set as in the $[Ph_2Sn(NO_3)_2Cl]^-$ anion [24] and O_5C_2 donor sets as in the $[Me_2Sn(O_2CMe)_3]^-$ [25] and $[Ph_2Sn(NO_3)_3]^-$ [26] anions as well as in the $\{[nBu_2Sn(O_2C_2O_2)(OH_2)]_2(O_2C_2O_2)\}^{2-}$

dianion [27]. Three cationic species have been characterised crystallographically, namely in $[Ph_2Sn(NO_3)(DMSO)_3]^+$ [28], with a O_5C_2 donor set, and in the two dicationic species $[Me_2SnL]^{2+}$ with an N_5C_2 donor set [29] (L is 6,6"-bis(α-methylhydrazino)-4'-phenyl-2,2':6',2"-terpyridine) and $[Et_2SnL']^{2+}$ with an $N_3O_2C_2$ donor set [30], where H_2L' is bis(2,6-diacetylpyridine-bis(isonicotinoylhydrazone)).

Table 3. Hydrogen-bonding contacts (A-H···B; Å, °) in $[Me_2Sn(LH)_2Cl]Cl.LH$

A	H	B	A-H	A···B	H···B	A-H...B	symmetry operation
N(1a)	H(22a)	Cl(1)	0.95	3.164(6)	2.92	96	i
N(1b)	H(22b)	Cl(1)	0.95	3.199(6)	3.11	87	i
N(1c)	H(21c)	Cl(1)	1.04	3.292(6)	2.91	103	ii
N(1b)	H(21b)	Cl(1)	0.95	3.317(6)	2.38	171	iii
N(1c)	H(22c)	Cl(2)	1.23	3.345(6)	2.34	137	iv
N(2a)	H(23a)	Cl(2)	0.95	3.221(6)	2.35	152	v
N(2c)	H(23c)	Cl(2)	0.95	3.205(6)	2.35	149	i
N(1a)	H(21a)	Cl(2)	0.95	3.371(6)	2.43	172	vi
N(2b)	H(23b)	O(1c)	0.95	2.832(7)	1.88	175	vii
N(1a)	H(22a)	N(1c)	0.95	2.972(7)	2.02	176	v

i *x*, *y*, *z*; ii -*x*, 0.5+*y*, 0.5-*z*; iii -*x*, -*y*, 1-*z*; iv *x*, -0.5-*y*, 0.5+*z*; v -*x*, -0.5+*y*, 0.5-*z*; vi -*x*, -*y*, 1-*z*; vii *x*, -0.5-*y*, -0.5+*z*

Acknowledgments.

The Department of Science and Technology (India) for the award of a BOYSCAST Fellowship, and North-Eastern Hill University are thanked for enabling TSBB to work in Adelaide. The support of the crystallographic facility by the Australian Research Council is gratefully acknowledged.

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Received: November 29, 1995 - Accepted: December 12, 1995 - Accepted in revised camera-ready format: January 23, 1996