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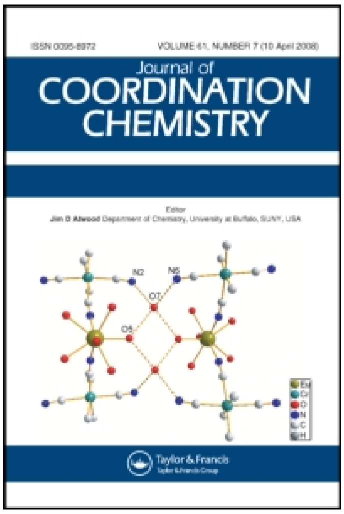

Journal of Coordination Chemistry
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713455674

PREPARATION AND SPECTROSCOPIC STUDY OF 1:2 ADDUCTS OF DIMETHYLTIN(IV)DICHLORIDE WITH AROMATIC *N*-OXIDES (IQNO, 4-C1QNO, 4-MeQNO, 6-MeOQNO AND 4-PhPyNO). CRYSTAL AND MOLECULAR STRUCTURE OF *TRANS*-DICHLORODIMETHYL *BIS*(4-PHENYLPYRIDINE-TV-OXIDE)TIN(IV)

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To cite this Article Papadaki, Hariklia , Christofides, Aristides , Jeffery, John C. and Bakas, Thomas(1999) 'PREPARATION AND SPECTROSCOPIC STUDY OF 1:2 ADDUCTS OF DIMETHYLTIN(IV)DICHLORIDE WITH AROMATIC *N*-OXIDES (IQNO, 4-C1QNO, 4-MeQNO, 6-MeOQNO AND 4-PhPyNO). CRYSTAL AND MOLECULAR STRUCTURE OF *TRANS*-DICHLORODIMETHYL *BIS*(4-PHENYLPYRIDINE-TV-OXIDE)TIN(IV)', Journal of Coordination Chemistry, 47: 4, 559 — 572

To link to this Article: DOI: 10.1080/00958979908024355

URL: http://dx.doi.org/10.1080/00958979908024355

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J. Coord. Chem., 1999, Vol. 47, pp. 559–572
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PREPARATION AND SPECTROSCOPIC STUDY OF 1:2 ADDUCTS OF DIMETHYLTIN(IV)DICHLORIDE WITH AROMATIC *N*-OXIDES (IQNO, 4-ClQNO, 4-MeQNO, 6-MeOQNO AND 4-PhPyNO). CRYSTAL AND MOLECULAR STRUCTURE OF *TRANS*-DICHLORODIMETHYL*BIS*(4-PHENYLPYRIDINE-*N*-OXIDE)TIN(IV)

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(Received 10 June 1998)

Five adducts of general formula $Me_2SnCl_2L_2$ [L = IQNO(isoquinoline-*N*-oxide), 4-ClQNO, 4-MeQNO, 6-MeOQNO (6-methoxyquinoline-*N*-oxide) and 4-PhPyNO (4-Phenylpyridine-*N*-oxide)] have been prepared by the addition of L to chloroform solutions of Me_2SnCl_2. The adducts have been characterized by analysis and spectroscopic (IR, ^1H, ^{13}C and Mössbauer) data. Structural predictions were derived from ^{119}Sn NMR parameters such as $^1J(^{119}Sn-^{13}C)$ and $^2J(^{119}Sn-^1H)$. The single crystal diffraction study of the adduct $Me_2SnCl_2(4\text{-}PhPyNO)_2$ shows the metal to be six-coordinate in an all *trans*-octahedral configuration; Sn–O and Sn–Cl distances are 2.227(2) and 2.5774(9) Å, respectively, and the $CH_3-Sn-CH_3$ bond angle is 180.0°.

Keywords: Crystal structure; quinoline-*N*-oxide; 4-phenylpyridine-*N*-oxide

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INTRODUCTION

The preparation of several dichlorodimethyltin(IV) complexes of pyridine-*N*-oxide and its derivatives has been reported.[1-6] Several years ago the *X*-ray structure analysis of $Me_2SnCl_2(PyNO)_2$ confirmed the expected *trans*-octahedral configuration.[7] Moreover, it was shown that the compound acquires a crystallographic centre of symmetry and is essentially monomeric in the solid state. However, the interaction of dimethyltin dichloride with 2,6-dimethylpyridin-*N*-oxide afforded the 1 : 1 adduct; its structure determination revealed dimer formation through bridging chlorine atoms.[8] A search of the literature showed that similar work with quinoline-*N*-oxide and its derivatives is very limited and restricted to $Me_2SnCl_2(QNO)_2$. The compound was prepared by Ng *et al.*[9] and the Lewis-acid tin complex again preferentially formed 1 : 2 adducts with monodentate oxygen donor ligands. The crystal structure of the above adduct showed a stereochemically crowded all-*trans*-octahedral compound, the coordination polyhedron being similar to that found in its pyridine-*N*-oxide counterpart.

Our motives in undertaking this work have been the current interest in organotin compounds, the possible use of such adducts in a variety of antifouling preparations,[10] and our interest in examining the spectroscopic behaviour, especially NMR parameters, of compounds bearing metals with spin-active nuclei.

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EXPERIMENTAL

IR spectra were recorded on a Perkin Elmer FT 16500 spectrometer and samples prepared as KBr disks. C, H and N analyses were carried out in our microanalytical laboratory with a Perkin Elmer 240 instrument. ^1H and ^{13}C NMR spectra were measured by the staff of the NMR Service of the School of Chemistry, University of Bristol, U.K. Molecular weight determinations were measured on a Perkin Elmer molecular weight apparatus in chloroform solutions. Conductivities were measured in 10^{-3} M chloroform solutions with a WTW LF 530 conductivity bridge.

Me_2SnCl_2 and 4-PhPyNO were purchased from Alfa Products Uentron and Aldrich, respectively. IQNO[11] and 4-ClQNO[12] were prepared by published procedures. 4-MeQNO and 6-MeOQNO were prepared by applying Ochiai's method[12] employed for QNO; the latter was recrystallized from acetone. All operations were performed in air at room temperature.

Preparation of the Complexes $Me_2SnCl_2L_2$ (1–5)

(1) To a solution of Me_2SnL_2 (0.109 g, 0.5 mmol) in $CHCl_3$ (10 cm^3), quinoline- or pyridine-*N*-oxide L was added with constant stirring at room temperature, the molar ratio being 1 : 2. The solution was stirred for 1 h and left to crystallize slowly . The mother liquid was discarded and the precipitated crystals were dried in air.

In the case of complex **4** a white precipitate was formed just after the addition of the ligand. It was left to stir overnight; the residue was collected by filtration and recrystallized from CH_2Cl_2. Complexes **1** and **5** were also recrystallized from CH_2Cl_2.

(2) In an alternative preparation the reaction mixture was refluxed for half an hour, then left to cool. Work up was as above.

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^{119}Sn Mössbauer Data

^{119}Sn Mössbauer spectra of powder samples were obtained at 85 K in an exchange gas cryostat, using a constant acceleration spectrometer and a 10 mCi calcium stannate source kept at room temperature. Spectrometer calibration was effected using a metallic iron foil. Isomer shifts are reported relative to $CaSnO_3$, assuming that they are the same as those of the $BaSnO_3$.

X-ray Structure Determination of 5

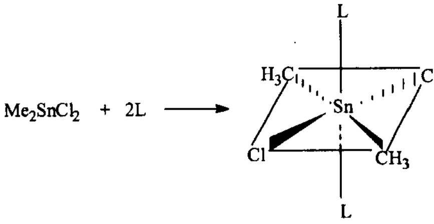
Crystals were grown from CH_2Cl_2. The crystal selected was sealed in a glass capillary tube under nitrogen gas. Data were collected using a Siemens R 3m/U four-circle diffractometer (293 K, Mo-Kα X-radiation, graphite monochromator, $\lambda = 0.71073$ Å). The data were corrected for Lorentz and polarization effects, and for absorption effects using an empirical method based on azimuthal scan data (SHELXTL PLUS program system (s320), Nicolet Instrument Corporation, 1987; SHELXTL PLUS TM program system Siemens Analytical X-ray Instruments, 1989).

The structure was solved by conventional heavy-atoms methods. Successive difference Fourier syntheses were used to locate all non-hydrogen atoms; hydrogen atoms were included in calculated positions. The Sn atom lies on an inversion centre and the complex has crystallographically imposed $\bar{1}$ symmetry.

All initial calculations were performed with a DEC micro-Vax II computer with the SHELXTL PLUS system of programs. The final least-squares refinements on F^2 were carried out on a Silicon Graphics Indigo R 4000 computer using SHELX93.

RESULTS AND DISCUSSION

Treatment of chloroform solutions of Me_2SnCl_2 with L (L = IQNO, 4-ClQNO, 4-MeQNO, 6-MeOQNO, 4-PhPyNO) in 1 : 2 mol ratio, results in the formation of the adducts **1**–**5**.



	L
1	IQNO
2	4-ClQNO
3	4-MeQNO
4	6-MeOQNO
5	4-PhPyNO

The elemental analyses (carbon, hydrogen and nitrogen) of all compounds fit well with the proposed formulae. Their analyses, colours and other physical data are given in Table I. The chloroform solutions of all the new adducts do not show appreciable electrical conductivity.

IR Spectra

The coordination of *N*-oxides to the tin is achieved *via* the oxygen atom of the N–O group. As a result, a negative shift of *ca* 21 cm^{-1} for ν(N–O) of the complexes is observed (Table II), as compared to those of the free ligands, which is attributed to the reduction of the double bond character in the N–O group.[2,6,13]

The single (Sn–Me) frequency present in some IR spectra is indicative of a *trans*-methyl configuration. This asymmetric stretching frequency for complexes IQNO, 4-ClQNO and 6-MeOQNO, occurs at *ca* 570 cm^{-1}, being

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TABLE I Analytical and other physical data for complexes **1–5**

Complex		*Colour*	*m.p.* (°C)[a]	*Yield* (%)	*Elemental analysis*[b]			*Molecular weight*[b]
					%C	*%H*	*%N*	
$Me_2SnCl_2(IQNO)_2$	**1**	White	124–127	82	47.08 (47.12)	3.95 (4.01)	5.52 (5.58)	509.9 (235)
$Me_2SnCl_2(4\text{-}ClQNO)_2$	**2**	Flesh	160–162	70	41.49 (41.62)	3.13 (3.38)	4.83 (4.77)	578.9 (224)
$Me_2SnCl_2(4\text{-}MeQNO)_2$	**3**	White	173–177	75	49.11 (48.98)	4.50 (4.49)	5.20 (5.10)	535.1 (233)
$Me_2SnCl_2(6\text{-}MeOQNO)_2$	**4**	Off white	185–188	65	46.35 (46.38)	4.24 (4.57)	4.91 (4.80)	570.1 (227)
$Me_2SnCl_2(4\text{-}PhPyNO)_2$	**5**	Off white	165–169	73	51.28 (51.33)	4.30 (4.35)	4.90 (4.90)	562.1 (196)

[a] Melting points were determined in open tubes. [b] Found values are given in parentheses.

TABLE II IR data (cm^{-1}) for the [$Me_2SnCl_2L_2$] adducts

Complex–ligand	*Ligand* $\nu(N-O)$	*Complex* $\nu(N-O)$	$\Delta\nu(N-O)$	$\nu(Sn-C)$	$\nu(Sn-O)$
IQNO	1180				
$Me_2SnCl_2(IQNO)_2$		1156	−24	568	330
4-ClQNO	1304				
$Me_2SnCl_2(4\text{-}ClQNO)_2$		1300	−4	572	
4-MeQNO	1209				
$Me_2SnCl_2(4\text{-}MeQNO)_2$		1173	−36	537	325
6-MeOQNO	1212–1183[a]				
$Me_2SnCl_2(6\text{-}MeOQNO)_2$		1216–1170[a]	+4, −13	572	348
4-PhPyNO	1239				
$Me_2SnCl_2(4\text{-}PhPyNO)_2$		1213	−26	512	

[a] The assignment is not unequivocal as there are two peaks in the region which are shifted upon complexation [J.H. Nelson and R.O. Ragsdale, *Inorg. Chim. Acta*, **2**, 230 (1968)].

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almost insensitive to the nature of L. Moreover, ν(Sn–C) does not correlate well with $J^2(^{119}Sn-^1H)$ and $J^1(^{119}Sn-^{13}C)$ as the *s*-character of the Sn–C bond increases or decreases. Similar behaviour has been noticed by Kumar and Kitching[4] for related compounds. Complexes of 4-MeQNO and 4-PhPyNO have a band at 524 cm^{-1}, which may be the (Sn–C) symmetric stretching frequency. Only in the spectra of adducts of IQNO, 4-MeQNO and 6-MeOQNO could we locate Sn–O stretching bands around 345 cm^{-1}.[12] We cannot be sure about bands assignable to ν(Sn–Cl) because of overlap problems.

NMR Spectra

^1H and ^{13}C NMR data of the new adducts are listed in Table III. In all ^1H and in some (**1**, **2**, **5**) ^{13}C NMR spectra, the peaks assignable to respective methyl nuclei exhibit two sets of symmetrically disposed ^{119}Sn and ^{117}Sn satellites. Due to solubility reasons we have been unable to see the same pattern in ^{13}C NMR spectra of **3** and **4**.

In the ^1H NMR spectra the methyl protons resonate at *ca* 1.16 ppm, their chemical shift being almost the same in comparison with that of the corresponding peak of the parent Lewis-acid (1.22 ppm) in $CDCl_3$ solution. In the ^{13}C NMR spectra the methyl carbons resonate at *ca* 12.02 ppm and are shifted downfield as compared to the analogous peak of Me_2SnCl_2 (6.58 ppm) in the same solvent. This downfield shift is expected because the methyl carbons are in the tin coordination sphere and are subjected to ligand-bonding variations, whereas the protons attached to carbons are not.[14]

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TABLE III ^1H and ^{13}C NMR data of $Me_2SnCl_2L_2$

Adduct	^1H			^{13}C		
	$\delta(CH_3)$	$^2J(^{119}Sn-^1H)$	$^2J(^{117}Sn-^1H)$	$\delta(CH_3)$	$^1J(^{119}Sn-^{13}C)$	$^1J(^{117}Sn-^{13}C)$
(1) $Me_2SnCl_2(IQNO)_2$	1.10[a]	91.5	87.6	12.5	764.5	727.9
(2) $Me_2SnCl_2(4\text{-}ClQNO)_2$	1.19[b]	85.2	81.5	12.3	724.8	691.2
(3) $Me_2SnCl_2(4\text{-}MeQNO)_2$[c]	1.16[b]	84.0	80.3	10.9		
(4) $Me_2SnCl_2(6\text{-}MeOQNO)_2$[c]	1.17[b]	82.7	79.1			
(5) $Me_2SnCl_2(4\text{-}PhPyNO)_2$	1.16[b]	88.3	84.5	12.4	764.5	732.5

[a] In CD_2Cl_2 on a Jeol 400 spectrometer. [b] In $CDCl_3$, adduct (**2**) and (**5**) on a Jeol 270 and 400, respectively, (**3**) and (**4**) on a Jeol Λ-300 spectrometer. [c] The low solubility prevents the evaluation of all ^{13}C NMR data.

Of most interest and a valuable source of information are the values of the coupling constants between the methyl protons and/or carbons and the spin-active tin isotopes. The $^2J(^{119}Sn-^1H)$ and $^1J(^{119}Sn-^{13}C)$ values of the new adducts are *ca* 86.3 and 751.3 Hz, respectively, being substantially increased as compared to those of $(CH_3)_2SnCl_2$ (68.7 and 478 Hz) in chloroform solution. However, the values obtained for the new adducts are lower than the expected ones, suggesting complex dissociation.[15] In fact, molecular weight determinations of **1, 2, 3, 4, 5** revealed significant dissociation, the values being less than the half of the calculated ones (Table I). The $^2J(^{117}Sn-^1H)$ and $^1J(^{117}Sn-^{13}C)$ values are in line with the above observations and their calculated values from the respective ^{119}Sn coupling constants, using the gyromagnetic ratio of the two isotopes ($\gamma^{119}Sn/\gamma^{117}Sn = 1.046$) are in satisfactory agreement with those evaluated from the NMR spectra.[16]

The $\theta(C-Sn-C)$ value for **5** can be calculated by applying the Lockhart[17] and Howard[14] equations. The values obtained are significantly lower than that found in the determination of the crystal structure (180°); the best result is obtained from equation (V) (Table IV). This suggests that the adduct in question is no hexacoordinated in solution, as it is in the solid state. As a result the magnitudes of $^2J(^{119}Sn-^1H)$ and $^1J(^{117}Sn-^{13}C)$ are somewhat lower than hexacoordinated tin(IV),[18] thereby the angles derived are also lower. Moreover, the values of θ obtained from equations (II) and (III) are greater than 130° and less than 180°, the upper limits for penta- and hexacoordinated dimethyltin(IV) species, respectively.[17] However, we cannot be sure of latter argument, since the limits have been established by Lockhart *et al.*[17] and the same authors admit several errors may occur in developing the two empirical relationships (Equations (II) and (III)).

The $^2J(^{119}Sn-^1H)$ value appears to respond to the nature of the 4-substituent of 4-PhPyNO in the case of **5** (Table II); the value is lower than that

TABLE IV ^1H and ^{13}C NMR data[a] for adduct **5** and angles derived by Lockhart and Howart equations

Adduct	$^2J(^{119}Sn-^1H)$	$^1J(^{119}Sn-^{13}C)$	$\theta(C-Sn-C)$ (deg)				
			Eq(I)	Eq(II)	Eq(III)	Eq(IV)	Eq(V)[b]
$Me_2SnCl_2(4\text{-}PhPyNO)_2$	88.3	764.5	143.8	142.2	133.7	154.9	150.8

[a] J in Hz; [b] Eq(I): $\theta(C-Sn-C) = [^1J(^{119}Sn-^{13}C) + 875/11.4$; Eq(II): $\theta(C-Sn-C) = 0.0161\ [^2J(^{119}Sn-^1H)]^2 - 1.32[^2J(^{119}Sn-^1H)] + 133.4$; Eq(III): $\theta(C-Sn-C) = 0.0105\ [^2J(^{119}Sn-^1H)]^2 - 0.799[^2J(^{119}Sn-^1H)] + 122.4$; Eq(IV): $\theta(C-Sn-C) = 2.28[^2J(^{119}Sn-^1H)] - 46.4$; Eq(V): $\theta(C-Sn-C) = 0.178[^1J(^{119}Sn-^{13}C)] + 14.74$.

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obtained for its methyl counterpart (99.5 Hz).[3] It seems to follow the trend established elsewhere, in that electron withdrawing substituents reduce the value of the coupling constant. However, data for **2** and **3** are not consistent with the above view.

^{119}Sn Mössbauer Data

All adducts have been investigated by Mössbauer spectroscopy. The isomer shifts (IS), quadrupole splitting (QS), average line widths at half maximum $\Gamma/2^a$ and calculated C–Sn–C bond angles are given Table V.

IS values are typical for octahedral quadrivalent tin compounds. Complexes **1** and **4** show the higher (1.46 mm s^{-1}) and lower (1.34 mm s^{-1}) IS values, respectively, whereas the average value of the five compounds (**1**–**4**, **6**) is 1.40 (0.02) mm s^{-1}. The decrease in the *s*-electron density at the tin nucleus in the series $Me_2SnCl_2L_2$ (L = IQNO, QNO, 4-ClQNO, 4-MeQNO, 6-MeOQNO), albeit small, could not be explained satisfactorily. Increase or decrease in donation by some of the ligands, as justified by resonance forms,[19] does not exert an analogous effect on IS values.[20,21] Perhaps steric influences play more decisive roles on this effect.

It is well known that the QS value is a good criterion for distinguishing between *cis* or *trans*-R_2 isomers in dialkyl tin complexes.[22] The QS values of the investigated complexes are consistent with a *trans*-Me_2 configuration. Moreover, the QS values were used to calculate the C–Sn–C bond angles by employing the Parish relationship[20] connecting the two parameters. As it is also known that the above relationship was derived due to the fact that a change in the bond angle between the organic groups affects markedly the QS.

TABLE V ^{119}Sn Mössbauer data

Compound	IS^a (mm s^{-1})	QS^a (mm s^{-1})	$\Gamma/2^a$ (mm s^{-1})	C–Sn–C^b (deg)
(**1**) $Me_2SnCl_2(IQNO)_2$	1.46	3.90	0.41	158
(**2**) $Me_2SnCl_2(4\text{-}ClQNO)_2$	1.40	3.89	0.41	158
(**3**) $Me_2SnCl_2(4\text{-}MeQNO)_2$	1.36	4.03	0.42	166
(**4**) $Me_2SnCl_2(6\text{-}MeOQNO)_2$	1.34	3.93	0.40	160
(**5**) $Me_2SnCl_2(4\text{-}PhPyNO)_2$	1.32	3.84	0.41	155
(**6**) $Me_2SnCl_2(QNO)_2^c$	1.42	4.00	0.43	164

[a] ±0.02 mm s^{-1}. [b] Calculated by using the literature partial quadrupole splitting: $[CH_3] = -1.03$ mm s^{-1} [c] Prepared by others.[9]

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X-ray Structure Determination of 5

The molecular structure of **5** is shown in Figure 1 together with the atom numbering scheme. It was found that **5** in the crystalline state is precisely *trans*-octahedral; the tin atom lies astride an inversion centre and the molecule has crystallographically imposed $\bar{1}$ symmetry.

The two rings of the 4-PhPyNO ligands are essentially colinear, as expected,[23] and are twisted with respect to each other about the C–C bond, the dihedral angle C(12)–N(11)–C(21)–C(22) being −35° The angle at oxygen is 117.7(2)°, similar to that found in the octahedral complexes $Me_2SnCl_2(PyNO)_2$[7] [117(1)°] and $Me_2SnCl_2(QNO)_2$[9] [123.2(1)°]. It has been pointed out by others[7] that the Sn–O–N angles for such complexes are of interest, because they might mean sp^2 hybridization of the oxygen orbitals, providing that the character in O–N bond is essentially single and steric factors favour a large angle. That was said for a related compound having a ligand (PyNO) less sterically demanding than 4-PhPyNO.[7] Consequently, in the case of **5** one should be more reserved considering sp^2 hybridization of the oxygen orbitals in terms of the observed angles.

There are no particularly close non-bonded contacts which would influence the lengths of Sn–Me (2.11(3) Å) and Sn–Cl(2.5774(9) Å) bonds, which are near to those found for $Me_2SnCl_2(PyNO)_2$[7] [2.225(25), 2.584(10) Å] and $Me_2SnCl_2(QNO)_2$[9] [2.122(3), 2.5902(7) Å].

The tin–oxygen distance here is 2.227(2) Å and lies between the corresponding lengths of $Me_2SnCl_2(QNO)_2$ [2.220(2) Å] and $Me_2SnCl_2(PyNO)_2$ [2.251(16) Å], which may mean that the coordination ability of 4-PhPyNO is also somewhere between QNO and PyNO. However, the same bond length is longer [2.289(2) Å] in the dimeric adduct of Me_2SnCl_2 with 2,6-dimethylpyridine N-oxide[24] and even longer [2.459(5) Å] in $(p\text{-tolyl})_3SnBrQNO$[25], probably due to steric reasons.

Interactions between the chlorine atoms and the hydrogens of the phenyl rings, seen in the packing diagram viewed down the *a* axis (Figure 2), are

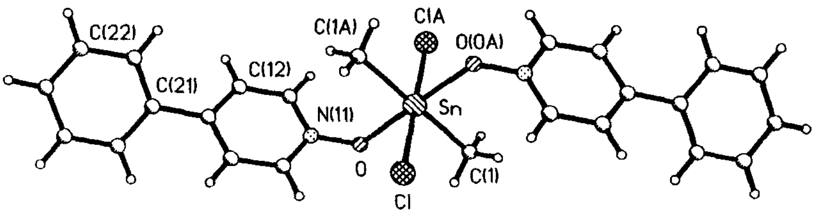

FIGURE 1 Molecular structure of **5** with the atom labelling scheme.

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weak, so we observe descrete molecules in solid state. Details of the crystal data and intensity collection are summarized in Table VI; selected bond lengths and angles for **5** are shown in Table VII, and final atomic constants for the non-hydrogen atoms are listed in Table VIII. Tables of final atomic

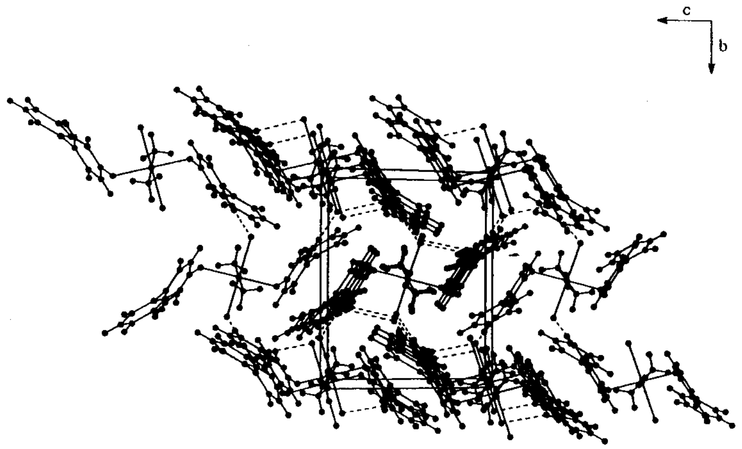

FIGURE 2 Unit-cell view of the structure of $Me_2SnCl_2(4\text{-}PhPyNO)_2$ (**5**) viewed down the crystallographic *a* axis.

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TABLE VI Crystal data and structure refinement details for **5**

Empirical formula	C12 H12 Cl N O Sn0.50
Formula weight	281.02
Temperature	293(2) K
Wavelength	0.71073 Å
Crystal system	monoclinic
Space group	$P2_{(1)}/c$
Unit cell dimensions	$a = 10.350\ (2)$ Å
	$b = 12.423\ (3)$ Å, beta = 110.07(2)°
	$c = 10.202(3)$ Å
Volume	1232.1(5) $Å^3$
Z	4
Density (calculated)	1.515 mg/m^3
Absorption coefficient	1.276 mm^{-1}
$F(000)$	564
Crystal size	0.25 × 0.30 × 0.40 mm
Theta range for data collection	2.66°–25.00°
Index ranges	$0 \leq h \leq 12, 0 \leq k \leq 14, -12 \leq l \leq 11$
Reflections collected	2290
Independent reflections	2164 [$R(int) = 0.0132$]
Data/restraints/parameters	2164/0/143
Goodness-of-fit on F^2	1.033
Final R indices [$I > 2\text{sigma}(I)$]	$R_1 = 0.025$, $wR_2 = 0.063$
R indices (all data)	$R_1 = 0.030$, $wR_2 = 0.066$

TABLE VII Selected bond lengths [Å] and angles [deg] for **5**

Sn–C(1) #1	2.119(3)
Sn–C(1)	2.119(3)
Sn–O #1	2.227(2)
Sn–O	2.227(2)
Sn–Cl	2.5774(9)
Sn–Cl #1	2.5774(9)
O–N(11)	1.347(3)
N(11)–C(16)	1.336(4)
N(11)–C(12)	1.339(4)
C(12)–C(13)	1.372(4)
C(13)–C(14)	1.389(4)
C(14)–C(15)	1.392(4)
C(14)–C(21)	1.482(4)
C(15)–C(16)	1.365(4)
C(21)–C(26)	1.387(4)
C(21)–C(22)	1.389(4)
C(22)–C(23)	1.389(5)
C(23)–C(24)	1.362(6)
C(24)–C(25)	1.378(5)
C(25)–C(26)	1.376(4)
C(1) #1–Sn–C(1)	180.0
C(1) #1–Sn–O #1	84.69(10)
C(1)–Sn–O #1	95.32(10)
C(1) #1–Sn–O	95.31(10)
C(1)–Sn–O	84.69(10)
O #1–Sn–O	180.0
C(1) #1–Sn–Cl	90.23(9)
C(1)–Sn–Cl	89.77(9)
O #1–Sn–Cl	89.50(7)
O–Sn–Cl	90.50(7)
C(1) #1–Sn–Cl #1	89.77(9)
C(1)–Sn–Cl #1	90.23(9)
O #1–Sn–Cl #1	90.50(7)
O–Sn–Cl #1	89.50(7)
Cl–Sn–Cl #1	180.0
N(11)–O–Sn	117.7(2)
C(16)–N(11)–C(12)	121.1(2)
C(16)–N(11)–O	119.3(2)
C(12)–N(11)–O	119.6(2)
N(11)–C(12)–C(13)	120.1(3)
C(12)–C(13)–C(14)	120.9(3)
C(13)–C(14)–C(15)	116.5(2)
C(13)–C(14)–C(21)	123.1(2)
C(15)–C(14)–C(21)	120.3(2)
C(16)–C(15)–C(14)	121.1(3)
N(11)–C(16)–C(15)	120.2(3)
C(26)–C(21)–C(22)	118.7(3)
C(26)–C(21)–C(14)	120.6(2)
C(22)–C(21)–C(14)	120.7(3)
C(23)–C(22)–C(21)	119.9(3)
C(24)–C(23)–C(22)	120.9(3)
C(23)–C(24)–C(25)	119.6(3)
C(26)–C(25)–C(24)	120.4(3)
C(25)–C(26)–C(21)	120.6(3)

Symmetry transformations used to generate equivalent atoms: #1 $-x+1, -y, -z$.

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TABLE VIII Atomic coordinates ($\times 10^4$ C) and equivalent isotropic displacement parameters for **5**. *U(eq)* is defined as one third of the trace of the orthogonalized U_{ij} tensor

	x/a	*y/b*	*z/c*	*U(eq)*
Sn	5000	0	0	37(1)
Cl	6059(1)	1872(1)	776(1)	64(1)
C(1)	6999(3)	−647(3)	436(3)	54(1)
O	5304(2)	−431(2)	2205(2)	55(1)
N(11)	4358(3)	−84(2)	2744(2)	45(1)
C(12)	3210(3)	−659(2)	2534(3)	53(1)
C(13)	2224(3)	−300(3)	3048(3)	52(1)
C(14)	2407(3)	648(2)	3811(3)	41(1)
C(15)	3638(3)	1195(2)	4033(3)	47(1)
C(16)	4587(3)	826(2)	3486(3)	49(1)
C(21)	1353(3)	1096(2)	4340(3)	43(1)
C(22)	−38(3)	999(3)	3567(3)	60(1)
C(23)	−1007(3)	1493(4)	4027(4)	73(1)
C(24)	−616(4)	2055(3)	5248(4)	69(1)
C(25)	760(4)	2132(3)	6038(4)	64(1)
C(26)	1737(3)	1658(2)	5590(3)	52(1)

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coordinates equivalent isotropic displacement parameters, hydrogen coordinates and isotropic displacement parameters, anisotropic thermal parameters are observed and calculated structure factors are available from the authors upon request.

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