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Synthesis, spectroscopic characterization, and crystal structures of two chlorodiorganotin(IV) 4-(2-methoxyphenyl)piperazine-1-carbodithioates

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ABSTRACT

Two chlorodiorganotin(IV) complexes of 4-(2-methoxyphenyl)piperazine-1-carbodithioate (MPPDA) have been synthesized by 1:1 mole-ratio reactions of the parent acid (MPPDAH) with Me_2SnCl_2 or Et_2SnCl_2 in dry methanol. The products have been characterized by Raman and multinuclear NMR (^1H, ^{13}C and ^{119}Sn) spectroscopy, elemental analysis, and mass spectrometry. Single-crystal X-ray diffraction studies indicate that both complexes have distorted trigonal bipyramidal geometries around the central Sn atom.

1. Introduction

Interest in the chemistry of organotins remains unabated [1]. The biocidal activity of organotins is well documented [2] and they have a number of industrial applications [3]. For several decades after their introduction, organotins were considered relatively benign actors in the environment since successive loss of organogroups should yield "non-toxic" Sn(II) species. Unfortunately, organotins are remarkably persistent in the environment and the study of their toxicology is a matter of some urgency [4]. It is generally accepted that *in vivo* deactivation of R_3Sn and R_2Sn systems involves enzymes whose active sites contain two sulfur donor atoms [5,6]. Thus considerable attention is being paid to organotin dithiolates, dithiocarboxylates, and dithiocarbamates, the last group being of particular interest [7]. Compounds of the type R_2SnClX, where X is the anion of a piperazinecarbodithioic acid, are a sub-set of these organotin dithiocarbamates, with the first of this type appearing in 1986 [8]. Only in the last decade has more interest been shown [9–12] including two structures from this group [13,14]. In continuation of this work, we now report the synthesis and characterization of two chlorodiorganotin(IV) 4-(2-methoxyphenyl)piperazine-1-carbodithioates, the parent acid being shown in Fig. 1.

2. Experimental

2.1. Materials and methods

Reagents, Me_2SnCl_2, Et_2SnCl_2 and 1-(2-methoxyphenyl)piperazine were obtained from Aldrich and CS_2 from Riedal-de Haën; methanol was dried before use by the literature procedure [15]. Microanalyses were done using a Leco CHNS 932 apparatus. Raman spectra (±1 cm^{-1}) were measured with an InVia Renishaw spectrometer, using argon-ion (514.5 nm) and near-infrared diode (785 nm) lasers. WiRE 2.0 software was used for the data acquisition and spectra manipulations. NMR spectra ($CDCl_3$) were obtained using a Bruker AM-300 MHz spectrometer (^1H and ^{13}C) and a Varian Unity 500-MHz instrument [^{119}Sn; $SnMe_4$(ext) ref]. Electron impact (70 eV) mass spectra were recorded on a Kratos MS25RFA instrument.

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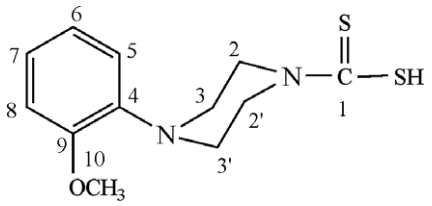


Fig. 1. Numbering scheme for 4-(2-methoxyphenyl)piperazine-1-carbodithioic acid (MPPDAH).

2.2. Synthesis

2.2.1. 4-(2-Methoxyphenyl)piperazine-1-carbodithioic acid (MPPDAH)

Dropwise addition of CS_2 (3.0 mmol) in methanol (50 mL) to 1-(2-methoxyphenyl)piperazine (5.0 g, 3.0 mmol) in methanol (50 mL) followed by stirring for 4 h at room-temperature gave the white product. This was filtered and washed with diethyl ether (Yield: 5.9 g, 85%). M.p. 161–163 °C. *Anal.* Calc. for $C_{12}H_{15}N_2OS_2$: C, 53.70; H, 6.01; N, 10.44; S, 23.89. Found: C, 53.60; H, 6.23; N, 10.56; S, 23.77%. Raman (cm^{-1}): 569 ν(C–S), 1215 ν(C═S), 2447 ν(SH). ^1H NMR (ppm): 3.6 (m, $H_{2,2'}$), 4.6 (m, $H_{3,3'}$), 3.9 (s, H_{10}), 8.5 (s, SH) 7.1–6.9 (m, C_6H_5). ^{13}C NMR (ppm): 209.6 (C-1), 48.4 (C-2,2′), 50.8 (C-3,3′), 55.5 (C-10), 111.4 (C-5), 118.4 (C-8), 121.1 (C-7), 123.8 (C-6), 140.6 (C-4), 152.3 (C-9).

2.2.2. $Me_2SnCl(MPPDA)$ (**1**)

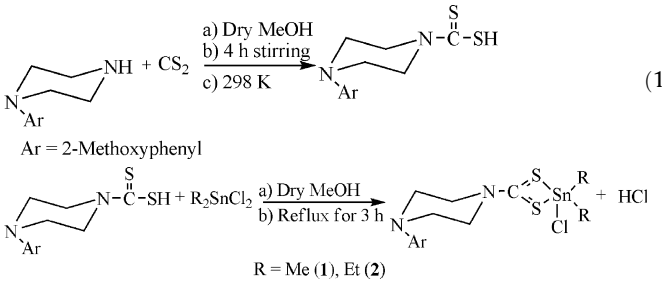
Me_2SnCl_2 (0.40 g, 1.86 mmol) in methanol (30 mL) was added dropwise to MPPDAH (0.50 g, 1.86 mmol) in methanol (30 mL) and the mixture was refluxed for 3 h with stirring. The white product was filtered and recrystallized from chloroform–ethanol to give colourless crystals (Yield: 0.65 g, 66%). M.p. 136–138 °C. *Anal.* Calc. for $C_{14}H_{21}N_2OS_2SnCl$: C, 37.23; H, 4.69; N, 6.20; S, 14.20. Found: C, 36.90; H, 4.67; N, 6.10; S, 13.90%. Raman (cm^{-1}): 286 ν(Sn–Cl), 396 ν(Sn–S), 499 ν(Sn–C), 527 ν(C–S), 1188 ν(C═S). ^1H NMR (ppm): 3.2 (m, $H_{2,2'}$), 4.2 (m, $H_{3,3'}$), 3.9 (s, H_{10}), 7.1–6.9 (m, C_6H_4), 1.4 (s, $SnCH_3$), 78 Hz [$^2J(^{119}Sn, ^1H)$]. ^{13}C NMR (ppm): 196.1 (C-1), 49.8 (C-2,2′), 51.9 (C-3,3′), 55.9 (C-10), 111.4 (C-5), 118.6 (C-8), 121.1 (C-7), 124.2 (C-6), 139.4 (C-4), 152.2 (C-9), 12.8 ($SnCH_3$). ^{119}Sn NMR: δ −192.9 ppm.

2.2.3. $Et_2SnCl(MPPDA)$ (**2**)

Compound **2** was prepared in the same way as **1**, using the same molar amounts, to give colourless crystals (Yield: 0.71 g, 79%). M.p. 184–186 °C. *Anal.* Calc. for $C_{16}H_{25}N_2OS_2SnCl$: C, 40.06; H, 5.25; N, 5.84; S, 13.37. Found: C, 39.81; H, 5.33; N, 5.81; S, 13.15%. Raman (cm^{-1}): 267 ν(Sn–Cl), 398 ν(Sn–S), 468 ν(Sn–C), 563 ν(C–S), 1183 ν(C═S). ^1H NMR (ppm), {$^nJ(^1H, ^1H)$, Hz}, [$^nJ(^{119}Sn, ^1H)$, Hz]: 3.2 (m, $H_{2,2'}$), 4.3 (m, $H_{3,3'}$), 3.9 (s, H_{10}), 7.1–6.9 (m, C_6H_4), 2.1 (q, Sn–CH_2CH_3) {7.8} [71], 1.5 (Sn–CH_2CH_3). ^{13}C NMR (ppm), [$^nJ(^{119}Sn, ^{13}C)$, Hz]: 200.4 (C-1), 50.2 (C-2,2′), 51.3 (C-3,3′), 55.5 (C-10), 111.3 (C-5), 118.5 (C-8), 121.1 (C-7), 123.8 (C-6), 140 (C-4), 152.2 (C-9), 10.9 ($SnCH_3$) [49], 27.9 (Sn–CH_2), [608]. ^{119}Sn NMR: δ −187 ppm.

2.3. X-ray crystallography

For both crystals, X-ray data were collected on a Bruker SMART APEX CCD diffractometer using graphite-monochromated Mo Kα radiation (λ = 0.71073 Å). Data collection used φ- and ω-scans, and a multi-scan absorption correction was applied in both cases. The structure of complex **1** was solved by Patterson synthesis followed by direct methods applied to difference structure factors using the program DIRDIF. The structure of compound **2** was solved by direct methods using the program SIR2004. In both cases, the hydrogen atoms were generated by geometrical considerations; methyl-groups were defined as rigid groups which were allowed to rotate free. Final refinement on F^2 carried out by full-matrix least-squares techniques using SHELXL-97. Some atoms in the solution for **2** showed unrealistic parameters when refined anisotropically due to the weak scattering power of the crystals examined. To improve the parameters, restraints were applied in the refinement of the structure of **2**.



(1)

(2)

3. Results and discussion

3.1. Synthesis

The ligand was obtained from the reaction of 1-(2-methoxyphenyl)piperazine with carbon disulfide in dry methanol (Eq. (1)). The organotin(IV) derivatives, **1** and **2**, were then synthesized by the reaction of MPPDAH with R_2SnCl_2 (R═Me or Et) as shown in Eq. (2). Both products are white crystalline solids, soluble in a range of organic solvents, e.g. benzene, chloroform, as well as THF, acetone, and DMSO. They are less soluble in methanol or ethanol and effectively insoluble in water.

3.2. Raman spectra

The Raman peak corresponding to the NH stretch of 1-(2-methoxyphenyl)piperazine disappears upon reaction with CS_2 and the formation of MPPDAH is also indicated by appearance of features assigned to ν(SH), ν(C–S) and ν(C═S) modes. After the reaction of MPPDAH with R_2SnCl_2, the peak associated with the ν(SH) disappears and the vibrational mode corresponding to tin–sulfur appears at ∼397 cm^{-1}. In addition, Raman spectra of complex **1** and **2** display peaks associated with the chlorodiorganotin moiety, ν(Sn–C) and ν(Sn–Cl).

3.3. NMR spectra

Comparing the ^1H NMR spectra of complexes **1** and **2** with that of MPPDAH showed the disappearance of the signal for the SH proton owing to complexation with the alkyltin moiety. The $^2J(^{119}Sn, ^1H)$ values for complexes **1** and **2** were 78.0 and 71.4 Hz, respectively, in the range normally expected for 5-coordinate tin [16] and consistent with CSnC angles of 128° and 118°. The ^{13}C chemical shifts observed for the two complexes were similar to those for the parent MPPDAH. Only a small shift in the position of C(1) was noted which resulted from deshielding of this particular carbon atom upon deprotonation of the thiol group and coordination through both sulfur atoms. The alkyl groups attached to the tin atoms showed signals in the expected range. Moreover, the $^1J[^{119}Sn, ^{13}C]$ coupling constant observed for complex **2**, 608 Hz, was the same as that reported for analogous 5-coordinate derivatives [17,18]. The ^{119}Sn NMR spectra of **1** and **2** displayed sharp signals at −192.9 and −187 ppm, respectively, which were consistent with those for similar five-coordinate organotin(IV) dithiocarbamates [19,20].

3.4. Mass spectra

Mass-spectral data for both **1** and **2** show rich ion distributions but our interest lies in those ions containing tin. These were easily qualitatively identified by inspection from the characteristic isotope peak patterns for "Sn" and "SnCl" species [21]. Fragmentation patterns are proposed for the tin-containing ions observed in **1** and **2** and these patterns are shown in Fig. 2a and b, respectively. The relative intensities reported for the ions given were qualitatively estimated from the ion current for the ^{120}Sn or $^{120}Sn^{35}Cl$ peak in each species, and must be regarded as approximate.

Both spectra show little (**1**) or no (**2**) molecular ion ($M^{+\cdot}$) present as is generally the case for main-group organometallics, and the first fragmentation appears to be loss or R· (R=CH_3 or C_2H_5) or the organic part of the anion, [$ArN(CH_2)_4NC$·]. For **1** [Fig. 2a] the latter strongly predominates giving the dimethyl species $S_2Sn(CH_3)_2Cl^+$ which can then lose C_2H_6, S_2, or CS to form further (EE^+) ions. The last, due to loss of CS, $H_3SSn(CH_3)Cl^+$, is the most abundant tin-containing ion and can be written as a sulphonium

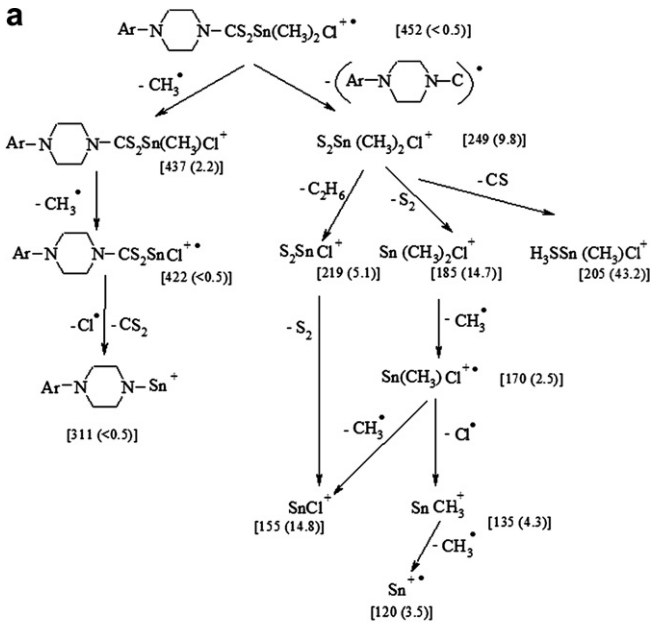


Fig. 2. Proposed fragmentation patterns for (a) $Me_2SnCl(MPPDA)$ (**1**) and (b) $Et_2SnCl(MPPDA)$ (**2**); [mass(% of the total positive current carried by identified tin-containing ions)].

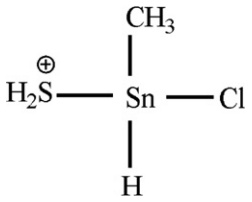


Fig. 3. Proposed structure of $H_3SSn(CH_3)Cl^+$.

species (Fig. 3). This type of ion may well originate from the initial ionization occurring from the CS_2Sn unit rather than $Sn(CH_3)_2Cl$ moiety [22] and be sufficiently stable to survive intact. Ions formed by loss of CH_3· provide approximately <3% of the current carried by tin-containing ions with only small amounts of possible tin-piperazine fragments being observed. The terminal species are $SnCl^+$ and $Sn^{+\cdot}$, the latter perhaps derived from $SnCH_3^+$.

The mass spectrum of **2** [Fig. 2b] significantly differs from that of **1**. Initial loss of ethyl from ($M^{+\cdot}$) is total but then, like **1**, loss of $ArN(CH_2)_4NC$· then greatly predominates. Several "S_2Sn" species are observed including the most abundant ion, S_2SnCl^+ and terminal ($OE^{+\cdot}$) $S_2Sn^{+\cdot}$. The other terminal ion is $SnCl^+$ but no $Sn^{+\cdot}$ was evident. Ions formed by loss of the second ethyl group from ($M-C_2H_5^+$) are only 4.6% of the current carried by tin-containing ions and may also account for some of terminal $S_2Sn^{+\cdot}$.

The steric effects of the larger ethyl groups compared to smaller methyl may be sufficient to account for the ease of their loss from high energy $ArN(CH_2)_4NCS_2Sn(C_2H_5)_2Cl^{+\cdot}$ when compared with the very similar methyl analog. Of more interest is the loss of only part of the anion from ($M^{+\cdot}$) (**1**) or ($M-C_2H_5^+$) (**2**). If as proposed above, ionization occurs from the CS_2Sn region, perhaps from the C=S bond itself, this could then make possible the preferential weakening of the CS_2 structure and thus facilitate loss of the organic fragment [$ArN(CH_2)_4NC$·], leaving the S_2Sn frame intact.

3.5. Crystal structures

Crystal data for both complexes are given in Table 1 with selected geometric parameters listed in Table 2. The ORTEP diagram

Table 1
Crystal data and structure refinement parameter for complexes **1** and **2**

	$Me_2SnCl(MPPDA)$ (**1**)	$Et_2SnCl(MPPDA)$ (**2**)
Empirical formula	$C_{14}H_{21}N_2OS_2SnCl$	$C_{16}H_{25}N_2OS_2SnCl$
Formula mass	451.63	479.68
Crystal system	orthorhombic	monoclinic
Space group	*Pbcn* (no. 60)	*C*2/*c* (no. 15)
a (Å)	20.190(6)	58.88(6)
b (Å)	6.906(2)	9.387(10)
c (Å)	25.676(8)	26.08(3)
β (°)		114.026(16)
V (Å^3)	3580.1(19)	13166(25)
Z (*Z*′)	8 (1)	24 (3)
Crystal habit/size (mm)	needle/ 0.43 × 0.09 × 0.07	platelet/ 0.42 × 0.31 × 0.23
T (K)	100(1)	293(1)
μ(Mo Kα) (cm^{-1})	14.81	18.07
Total reflections	24431	33353
Independent reflections		
All	3621	9430
For $F_o > 4.0\sigma(F_o)$	2855	3964
$R(F) = \sum(\|\|F_o\| - \|F_c\|\|)/\sum\|F_o\|$ For $F_o > 4.0\sigma(F_o)$	0.0332	0.1122
$wR(F^2) = [\sum[w(F_o^2 - F_c^2)^2]/\sum[w(F_o^2)^2]]^{1/2}$ all reflections	0.0692	0.3306
Goodness-of-fit	0.996	0.972
θ range for data collection (°)	3.12–26.37	2.22–23.53
Data/restraints/parameters	3621/0/193	9430/515/631

Table 2
Selected bond lengths (Å) and angles (°) for complexes **1** and **2**

$Me_2SnCl(MPPDA)$ (**1**)		$Et_2SnCl(MPPDA)$ (**2**)			
			$x = 1$	$x = 2$	$x = 3$
Sn–Cl	2.438(11)	Sn–Clx	2.520(6)	2.505(7)	2.536(9)
Sn–S1	2.760(12)	Snx–Sx1	2.500(5)	2.779(8)	2.451(6)
Sn–S2	2.464(11)	Snx–Sx2	2.747(6)	2.471(5)	2.710(8)
Sn–C13	2.104(3)	Snx–Cx13	2.181(16)	2.13(2)	2.19(3)
Sn–C14	2.107(4)	Snx–Cx15	2.156(15)	2.20(3)	2.01(4)
S1–C1	1.704(3)	Sx1–Cx1	1.722(19)	1.785(17)	1.84(2)
S2–C1	1.750(3)	Sx2–Cx1	1.719(15)	1.82(2)	1.722(18)
Cl–Sn–S1	154.23(3)	Clx–Snx–Sx1	85.89(15)	153.74(15)	87.7(2)
Cl–Sn–S2	85.81(3)	ClxSnx–Sx2	154.53(12)	85.06(18)	157.7(2)
Cl–Sn–C13	101.81(9)	Clx–Snx–Cx13	94.2(5)	98.2(8)	94.6(9)
Cl–Sn–C14	100.09(10)	Clx–Snx–Cx15	95.3(6)	96.6(10)	97.6(12)
S1–Sn–S2	68.43(3)	Sx1–Snx–Sx2	68.65(14)	68.70(17)	70.07(18)
S1–Sn–C13	89.47(9)	Sx1–Snx–Cx13	114.9(5)	92.8(8)	116.2(8)
S1–Sn–C14	92.35(10)	Sx1–Snx–Cx15	114.5(5)	89.8(10)	128.6(10)
S2–Sn–C13	116.56(9)	Sx2–Snx–Cx13	95.9(5)	112.5(6)	94.9(8)
S2–Sn–C14	115.99(10)	Sx2–Snx–Cx15	95.9(7)	105.1(8)	96.8(12)
C13–Sn–C14	123.93(13)	Cx13–Snx–Cx15	130.2(7)	140.5(10)	114.2(13)
Sn–S1–C1	82.84(11)	Snx–Sx1–Clx1	90.0(5)	85.1(7)	91.4(6)
Sn–S2–C1	91.43(10)	Snx–Sx2–Clx1	82.2(6)	94.0(6)	85.7(7)
S1–C1–S2	117.19(18)	Sx1–Cx1–Sx2	119.0(1)	111.1(10)	112.9(10)

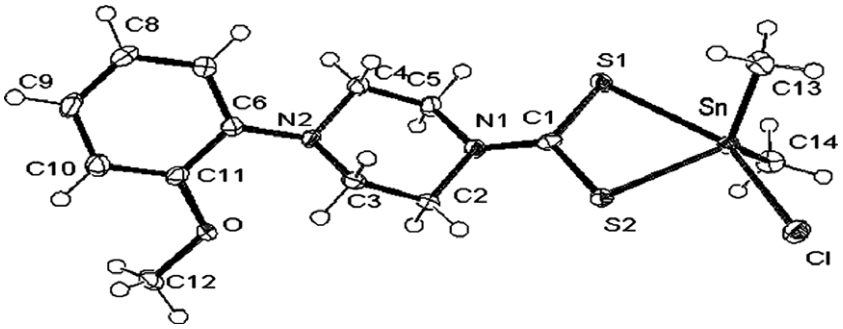


Fig. 4. Numbering scheme (ORTEP) for $Me_2SnCl(MPPDA)$ (**1**).

for the molecule of **1** together with the atom numbering scheme used here is shown in Fig. 4. The configuration about the tin atom is five-coördinate, with a distorted trigonal–bipyramidal geometry with atoms S2, C13 and C14 occupying the equatorial positions. The sum of the equatorial angles [356.5(3)°] at the tin atom involving the C13 C14, and S2 atoms deviates only by 3.5° from the ideal angle of 360°. The Cl atom occupies one of the apical positions of the trigonal bipyramid with the ClSnS2 angle of 85.81(3)°. As a result of MPPDA being a chelating ligand with a small bite forming a four-membered ring, the S1–Sn–S2 angle is not 90°, but only 68.43(3)°. Therefore the S1 atom cannot occupy exactly the corresponding *trans* axial position to Cl and the Cl–Sn–S1 angle is 154.23(3)°. Thus the thiocarboxylate group is asymmetrically coordinated as shown by Sn–S1(ax), 2.760(12), and Sn–S2(eq), 2.464(11) Å, bond lengths. The S–C bond lengths [S1–C1 = 1.704(3) Å and S2–C1 = 1.750(3) Å] also show the asymmetric nature of the dithiocarboxylate group.

The asymmetric unit of **2** contains three different molecules; The ORTEP diagram for one of these, molecule (1), together with an example of the atom numbering scheme used here is shown in Fig. 5. As with complex **1**, the configuration about the tin atom is five-coördinate, with a distorted trigonal–bipyramidal geometry at the central tin atom. The sums of the equatorial angles involving the two α-carbons of the ethyl groups and a S atom, (1): 360(2)°, (2): 358(2)°, (3): 359(3)°, show little deviation from the ideal angle of 360°. For each molecule, the Cl atom occupies one of the apical positions of the trigonal bipyramid but the quasi-axial S atom again cannot occupy exactly the position *trans* to Cl. Thus the thiocarboxylate groups are asymmetrically coordinated as in the structure of **1**. While there appear to be differences in detail for the geometries around tin for the three different molecules in the asymmetric unit of **2**, the less than optimal data set and the large number of constraints used in the structure solution would indicate that more detailed discussion would be premature at this point.

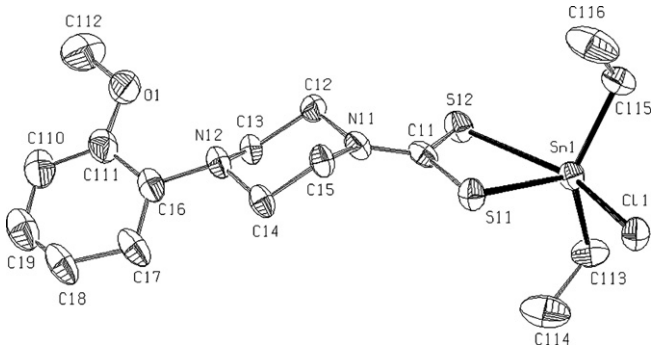


Fig. 5. Numbering scheme (ORTEP) for one of three independent molecules of $Et_2SnCl(MPPDA)$ (**2**).

4. Conclusions

Both complexes have five-coördinate tin as evidenced by the multinuclear solution NMR (^{1}H, ^{13}C and ^{119}Sn) spectra while X-ray single-crystal diffraction data show they have quasi-trigonal bipyramidal geometry in the solid state. The dithioate ligand is asymmetrically coördinated to tin in each case. The EI–MS fragmentation patterns show surprising differences for two ostensibly very similar species. However this parallels the differing biological properties of analogues methyl- and ethyltin systems. In the near future, we plan to undertake a preliminary investigation of the pesticidial activity of these two new organotin(IV) complexes.

Acknowledgements

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Appendix A. Supplementary material

CCDC 664573 and 664574 contain the supplementary crystallographic data for compounds **1** and **2**. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif. Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.ica.2008.05.006.

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