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The Crystal and Molecular Structure of an Unusual Triorganotin Complex: 1-[(4′-Methylphenylimino)-methyl]-2-naphtholchlorotriphenyltin(IV)(3:2)

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Abstract

The crystal and molecular structure of the triphenyltin complex of 1-[4′-methylphenylimino)-methyl]-2-naphthol at 295 and 120 K is reported. The compound crystallises with a ligand:triphenyltin chloride ratio of 3:2.

At 295 K, the crystals are monoclinic, space group *C*2/*c*, $a = 10.525(3)$, $b = 23.274(4)$, $c = 30.150(9)$ Å, $\beta = 96.150(23)^\circ$, $V = 7343.13$ Å^3, $Z = 8$, $D_{calc} = 1.407$ Mg m^{-3}, $\mu = 0.82$ mm^{-1}, final $R = 4.7\%$ for 4014 unique observed reflections.

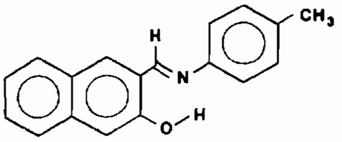
At 120 K, the crystals are monoclinic, space group *C*2/*c*, $a = 10.399(2)$, $b = 23.145(5)$, $c = 30.139(4)$ Å, $\beta = 95.837(12)^\circ$, $V = 7216.40$ Å^3, $Z = 8$, $D_{calc} = 1.431$ Mg m^{-3}, $\mu = 0.82$ mm^{-1}, final $R = 4.3\%$ for 7212 unique observed reflections.

The complex has a five-coordinate trigonal bipyramidal structure, with the phenyl groups taking up the equatorial positions around the tin atom. The ligand, which exists in the form of a zwitterion in the complex, binds to the tin via the phenolic oxygen atom. Free ligand has cocrystallised with the complex in the ratio of one free ligand molecule to every two of the organotin complex units. The free ligand molecules pack in parallel strings in the crystal, between the organotin complex moieties, which are arranged as pairs of centrosymmetrically-related dimers.

Introduction

Complexes of triphenyltin chloride and isothiocyanate almost invariably crystallise with 1:1 stoichiometries, and five-coordinate trigonal bipyramidal structures [1–5].

We now report the crystal and molecular structure of the triphenyltin complex of 1-[(4′-methylphenylimino)-methyl]-2-naphthol, which has been found to crystallise with a ligand:triphenyltin chloride ratio of 3:2.



1-[(4′-methylphenylimino)-methyl]-2-naphthol(L)

Experimental

The reagents triphenyltin chloride, 2-hydroxynaphthaldehyde and 4-methylaniline were obtained commercially, and used without further purification.

The Schiff base was prepared by the condensation of 2-hydroxy-naphthaldehyde with 4-methylaniline in refluxing benzene, the water formed being removed azeotropically. The ligand was purified by recrystallisation from 95% ethanol.

The complex was prepared by refluxing the calculated amount (for 1:1 complex formation) of ligand and triphenyltin chloride in 95% ethanol for 30 min. The complex crystallised when the solution was cooled in the freezer overnight. It was filtered off and recrystallised from ethanol (melting point 128–130 °C).

The conductivity of the complex in nitrobenzene was measured at room temperature using a Phillips PW 9501 conductivity bridge with a cell constant of 0.71 cm^{-1}. Molar conductance value obtained = 153 ohm^{-1} cm^{-1} mol^{-1}.

Tin was determined gravimetrically at SnO_2. Microanalyses were performed at the Chemistry Department, National University of Singapore, Singapore.

Anal. for $Ph_3SnCl \cdot 1.5L[C_{45}H_{37.5}N_{1.5}O_{1.5}SnCl]$: Calc.: C, 69.52; H, 4.86; N, 2.70; Sn, 15.27. Found: C, 70.07; H, 4.96; N, 2.94; Sn, 15.40%.

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Crystal Data

(295 K) Monoclinic, space group *C2/c*, $a = 10.525(3)$, $b = 23.274(4)$, $c = 30.150(9)$ Å, $\beta = 96.150(23)°$, $V = 7343.13$ Å^3, $Z = 8$, $D_{calc} = 1.407$ Mg m^{-3}, $\mu = 0.82$ mm^{-1}.

(120 K) Monoclinic, space group *C2/c*, $a = 10.399(2)$, $b = 23.145(5)$, $c = 30.139(4)$ Å, $\beta = 95.837(12)°$, $V = 7216.40$ Å^3, $Z = 8$, $D_{calc} = 1.431$ Mg m^{-3}, $\mu = 0.82$ mm^{-1}.

Intensity Data

First, intensity data were collected at 295 K from a 0.1 × 0.1 × 0.2 mm dimension crystal using graphite monochromatised Mo Kα radiation. A total of 7218 reflections were measured on a Nonius CAD-4 diffractometer using the omega scan mode ($2\theta \leqslant 45°$). Of the reflections measured, 6450 were unique, of which 4014 were considered significant, with $I_{net} > 2.5\sigma(I_{net})$. They were used to solve and refine the crystal structure. Then it was decided, for reasons mentioned in the following section, to collect intensity data again from the same crystal, but at a low temperature (120 K). A total of 15 838 reflections were measured on a Picker four-circle diffractometer equipped with a low-temperature device, using the $\theta/2\theta$ scan mode with profile analysis ($2\theta \leqslant 60°$). After eliminating the systematically absent reflections, the data set consisted of 10627 unique reflections of which 7212 were retained as significantly above background, with $I_{net} > 2.5\sigma(I_{net})$, for refinement of the structure at 120 K. The intensities were corrected for direct beam polarisation [6], but absorption correction was not applied ($\mu = 0.82$ mm^{-1}).

The cell dimensions at 295 K were obtained from 45 reflections with 2-theta angle in the range 40.00–45.00 degrees. Accurate cell parameters at 120 K were calculated from the setting angles of 45 high-angle reflections ($45° \leqslant 2\theta \leqslant 50°$). [$\lambda$(Mo K$\alpha$) = 0.70930 Å].

Structure Solution and Refinement

The structure at room temperature was solved using direct methods. Atoms of the tin complex were located first and refined. A difference map then revealed many peaks with low peak height. These peaks formed a molecular fragment which looked different from the free Schiff base expected to have co-crystallised with the tin complex, based on elemental analysis. As peaks were close to a two-fold axis and a centre of symmetry, an infinite string arrangement as shown in Fig. 4 (see also Fig. 6, supplementary material) was produced. Figures 4 and 6 show uncoordinated ligands disordered over two locations. Least-squares refinement of the peak occupancies indicated that they were present in a 1:1 ratio. The naphthalene rings are shifted about centres of symmetry in such a way that three of the atomic positions of each ring, C1′, C6′ and C8′ are shared between the two images. The unit cell contains eight tin complex molecules and eight halves of the free ligand. The Schiff base/tin ratio is 3:2 instead of the 2:1 ratio first proposed from elemental analysis.

Two refinements were carried out in the acentric space group *Cc*, with the two possible orientations of the free ligand, using an ordered asymmetric unit which includes two tin complex molecules and one full non-disordered free ligand. In both attempts, the refinements yielded unsatisfactory results and they gave severely distorted structures showing irregular C–C bond lengths and bent aromatic rings. Further refinement was performed in the centric space group *C2/c*. All non-H atoms were refined anisotropically. The H atoms except those of the free ligand were then located on a difference map and their positional parameters were refined together with isotropic thermal parameters, with full-matrix least-squares. The final residuals at 295 K, are $R_f = 0.047$ and $R_w = 0.027$, for 4014 significant reflections. The same procedure was applied again, using low temperature data in the hope to solve the disorder problem. The refinements produced similar results to those obtained at room temperature, again forcing the choice of the space group *C2/c*. The final residuals at 120 K, are $R_f = 0.043$ and $R_w = 0.034$, for 7212 significant reflections. Weights based on counting statistics were used throughout. All calculations were performed with the NRCVAX system of programs [7]. Literature values [8] were used for the scattering factors.

Results and Discussion

The chloride and isothiocyanate of triphenyltin readily form 1:1 complexes with a variety of ligands. It appears that all the complexes so far reported are five-coordinate, having trigonal bipyramidal structures, even with potentially bi- and tridentate ligands. For example, with the bidentate chelating agents phenanthroline [2] and bipyridine [2] and the tridentate terpyridine [3, 4], the triphenyltin chloride and isothiocyanate complexes display a most unusual structure in which the heterocyclic ligands are only indirectly bound to the tin atom (by a coordinated water molecule), giving a trigonal bipyramidal arrangement around the tin. On the other hand, in the corresponding complexes with the potential bidentates picolinic acid [9] and quinaldic acid [10], the ligands are forced into a unidentate mode (with the carboxylate oxygen as donor atom) by the transfer of the carboxylic acid proton to the heterocyclic nitrogen atom, which effectively blocks coordination at this site, and leads to the observed five-coordination around the tin. In addition, we have recently reported [11] the characterisation of a series of triphenyltin complexes with a range of potentially

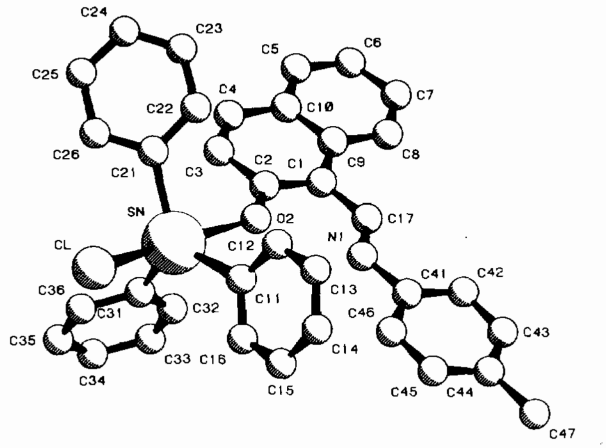

Fig. 1. Molecular configuration and atomic numbering scheme.

bidentate Schiff bases. These complexes have also been found to posssess trigonal bipyramidal structures. Consequently, the isolation of another such complex, displaying a completely different stoichiometry, led us to suspect that this time, the coordination around the tin atom might be different. In a previous report [12] of some 2:1 (ligand:triphenyltin pseudohalide) complexes, it has been suggested that the tin atom displays an octahedral geometry. But, in the present instance, the results of the X-ray study show that this complex again involves five-coordinate tin, and that its structure is virtually the same as those of the 1:1 complexes, except that some free ligand has cocrystallised with the complex, in the ratio of one free ligand molecule to every two of the organotin complex units.

Since the overall structures at 295 and 120 K were the same, discussion is restricted to the geometrical features of the room temperature structure.

The structure of the organotin complex moiety and the atomic numbering scheme are shown in Fig. 1, while a stereoscopic view is given in Fig. 2. Table I lists the bond distances and angles, while the final atomic positional parameters and the equivalent isotropic temperature factors are given in Table II.

The organotin moieties have trigonal bipyramidal structures, with the three phenyl groups occupying the equatorial positions around the tin atom, while a phenolic oxygen from the ligand, and the chlorine atom, take up the axial positions. The C–Sn–C angles between the three phenyl rings are 118.4°, 121.0° and 119.5° while the O–Sn–Cl angle is 175.7°. showing only very slight deviation from trigonal bipyramidal geometry around the tin atom. The average Sn–C(Ph) distance of 2.13 Å is within the range of Sn–C(Ph) distances (2.105–2.16 Å) reported in the literature [13]. The C–C bond lengths in the phenyl groups are all normal.

The Sn–Cl distance of 2.48 Å is similar to those found in the picolinic acid [9] (2.52 Å) and quinaldic acid [10] (2.53 Å) complexes of triphenyltin chloride, and within the range of Sn–Cl distances in other chloro complexes [13] (2.32–2.58 Å).

An unusual feature of the structure is that the phenolic proton of the ligand has moved to the imine nitrogen. The Schiff base is thus coordinated in the form of a zwitterion, as observed for the 1:1 triphenyltinisothiocyanate complex of this ligand [11]. Similar proton transfers from oxygen to nitrogen have been reported recently for some organotin complexes of picolinic [9] and quinaldic [10] acids, which also coordinate to tin(IV) atoms in the form of zwitterions. For these two acids, a five-membered ring is formed by the intramolecular hydrogen bonding, whereas in the present instance a six-membered ring must be involved. However, the protonation of the nitrogen effectively blocks that atom from any coordinative interaction with the tin in all these examples.

These organotin complex units pack together in the crystal in pairs of centrosymmetrically-related dimers, with the Schiff bases lying flat, one above the other, and head-to-tail relative to each other in a similar manner to that reported for the 1:1 isothiocyanato complex [11]. The head-to-tail packing of some of these dimers and their arrangement within the unit cell is illustrated by the stereoview in Fig. 3. The free ligand molecules are arranged in parallel strings running between the organotin moieties, as shown in Fig. 4. These ligand strings, which show some disorder, occur at two different locations

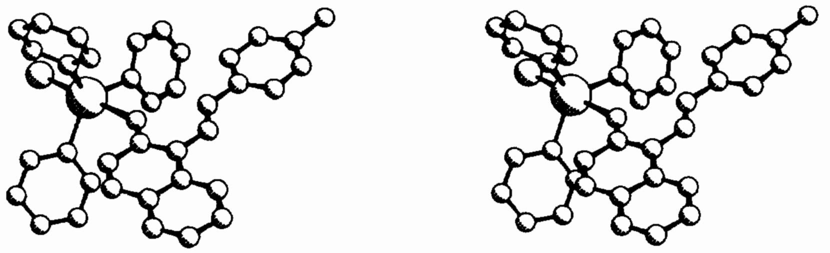

Fig. 2. Stereoview of the complex.

TABLE I. Bond Distances (Å) and Angles (°)

Sn–Cl	2.4819(16)	C(35)–C(36)	1.375(9)	C(4)–C(10)–C(9)	118.3(4)
Sn–O(2)	2.391(3)	C(41)–C(42)	1.355(8)	C(5)–C(10)–C(9)	120.2(5)
Sn–C(11)	2.143(5)	C(41)–C(46)	1.347(8)	Sn–C(11)–C(12)	120.9(4)
Sn–C(21)	2.129(5)	C(42)–C(43)	1.377(7)	Sn–C(11)–C(16)	118.0(4)
Sn–C(31)	2.110(6)	C(43)–C(44)	1.333(8)	C(12)–C(11)–C(16)	120.7(5)
O(2)–C(2)	1.288(6)	C(44)–C(45)	1.352(9)	C(11)–C(12)–C(13)	118.4(6)
N(2)–C(17)	1.295(6)	C(44)–C(47)	1.535(7)	C(12)–C(13)–C(14)	120.5(6)
N(2)–C(41)	1.426(6)	C(45)–C(46)	1.389(8)	C(13)–C(14)–C(15)	120.6(5)
C(1)–C(2)	1.423(7)			C(14)–C(15)–C(16)	120.2(6)
C(1)–C(9)	1.450(7)	Cl–Sn–O(2)	175.67(9)	C(11)–C(16)–C(15)	119.5(6)
C(1)–C(17)	1.398(7)	Cl–Sn–C(11)	93.07(15)	N(2)–C(17)–C(1)	125.9(5)
C(2)–C(3)	1.439(7)	Cl–Sn–C(21)	92.64(14)	Sn–C(21)–C(22)	122.6(4)
C(3)–C(4)	1.328(7)	Cl–Sn–C(31)	95.18(14)	Sn–C(21)–C(26)	119.6(4)
C(4)–C(10)	1.417(7)	O(2)–Sn–C(11)	82.91(16)	C(22)–C(21)–C(26)	117.6(5)
C(5)–C(6)	1.356(8)	O(2)–Sn–C(21)	88.08(16)	C(21)–C(22)–C(23)	121.7(5)
C(5)–C(10)	1.396(7)	O(2)–Sn–C(31)	88.21(16)	C(22)–C(23)–C(24)	120.5(6)
C(6)–C(7)	1.379(8)	C(11)–Sn–C(21)	120.99(21)	C(23)–C(24)–C(25)	118.8(5)
C(7)–C(8)	1.353(7)	C(11)–Sn–C(31)	119.46(21)	C(24)–C(25)–C(26)	121.2(5)
C(8)–C(9)	1.390(7)	C(21)–Sn–C(31)	118.37(20)	C(21)–C(26)–C(25)	120.2(5)
C(9)–C(10)	1.402(7)	Sn–O(2)–C(2)	136.3(3)	Sn–C(31)–C(32)	122.5(4)
C(11)–C(12)	1.361(8)	C(17)–N(2)–C(41)	127.0(4)	Sn–C(31)–C(36)	121.3(4)
C(11)–C(16)	1.364(8)	C(2)–C(1)–C(9)	120.8(4)	C(32)–C(31)–C(36)	116.2(5)
C(12)–C(13)	1.393(8)	C(2)–C(1)–C(17)	119.3(4)	C(31)–C(32)–C(33)	123.1(5)
C(13)–C(14)	1.357(10)	C(9)–C(1)–C(17)	119.9(4)	C(32)–C(33)–C(34)	120.3(6)
C(14)–C(15)	1.332(11)	O(2)–C(2)–C(1)	120.8(4)	C(33)–C(34)–C(35)	118.5(6)
C(15)–C(16)	1.386(8)	O(2)–C(2)–C(3)	122.6(4)	C(34)–C(35)–C(36)	120.6(5)
C(21)–C(22)	1.387(8)	C(1)–C(2)–C(3)	116.7(4)	C(31)–C(36)–C(35)	121.4(6)
C(21)–C(26)	1.377(7)	C(2)–C(3)–C(4)	121.7(5)	N(2)–C(41)–C(42)	122.6(5)
C(22)–C(23)	1.385(8)	C(3)–C(4)–C(10)	123.2(4)	N(2)–C(41)–C(46)	116.2(5)
C(23)–C(24)	1.367(9)	C(6)–C(5)–C(10)	120.7(5)	C(42)–C(41)–C(46)	121.3(5)
C(24)–C(25)	1.366(9)	C(5)–C(6)–C(7)	119.5(5)	C(41)–C(42)–C(43)	117.2(5)
C(25)–C(26)	1.407(8)	C(6)–C(7)–C(8)	120.4(5)	C(42)–C(43)–C(44)	124.6(5)
C(31)–C(32)	1.336(8)	C(7)–C(8)–C(9)	122.3(5)	C(43)–C(44)–C(45)	116.2(5)
C(31)–C(36)	1.386(8)	C(1)–C(9)–C(8)	124.1(4)	C(43)–C(44)–C(47)	122.8(6)
C(32)–C(33)	1.379(9)	C(1)–C(9)–C(10)	119.1(4)	C(45)–C(44)–C(47)	121.0(6)
C(33)–C(34)	1.348(9)	C(8)–C(9)–C(10)	116.9(4)	C(44)–C(45)–C(46)	122.3(6)
C(34)–C(35)	1.359(9)	C(4)–C(10)–C(5)	121.5(4)	C(41)–C(46)–C(45)	118.5(5)

TABLE II. Positional Parameters and Isotropic Thermal Parameters

	x	y	z	B_{iso}[a]		x	y	z	B_{iso}[a]
Sn	0.52824(5)	0.721203(18)	0.612387(14)	4.205(21)	C17	0.6480(5)	0.51577(21)	0.57547(16)	3.8(3)
Cl	0.46270(17)	0.82367(6)	0.61150(6)	6.57(11)	C21	0.3950(5)	0.69830(22)	0.65777(16)	4.2(3)
O2	0.5814(4)	0.62161(13)	0.60804(11)	4.44(22)	C22	0.3238(6)	0.64806(25)	0.65355(18)	5.5(4)
N1	0.9761(9)	0.4726(4)	0.1221(3)	5.3(6)	C23	0.2438(6)	0.6322(3)	0.68501(20)	6.4(4)
N2	0.6329(4)	0.55458(17)	0.54457(13)	4.3(3)	C24	0.2348(6)	0.6653(3)	0.72193(19)	7.1(4)
C1	0.6363(5)	0.52434(21)	0.62075(15)	3.4(3)	C25	0.3066(6)	0.7142(3)	0.72733(16)	6.4(4)
C2	0.6069(5)	0.58019(22)	0.63591(16)	3.9(3)	C26	0.3853(5)	0.73172(24)	0.69493(17)	5.2(3)
C3	0.6075(6)	0.58748(22)	0.68333(16)	4.5(3)	C31	0.7195(6)	0.73693(20)	0.63810(17)	4.0(3)
C4	0.6286(5)	0.54370(24)	0.71138(16)	4.6(3)	C32	0.8127(6)	0.69868(24)	0.63499(20)	6.2(4)
C5	0.6665(6)	0.44150(24)	0.72776(17)	5.5(4)	C33	0.9368(6)	0.7073(3)	0.65349(22)	8.4(5)
C6	0.6895(6)	0.38746(24)	0.71384(18)	6.4(4)	C34	0.9694(6)	0.7562(3)	0.67596(20)	7.3(4)
C7	0.6946(6)	0.37704(23)	0.66904(18)	5.4(4)	C35	0.8779(6)	0.7968(3)	0.67898(19)	7.2(4)
C8	0.6798(6)	0.42059(22)	0.63921(17)	4.6(3)	C36	0.7545(6)	0.7875(3)	0.66048(18)	5.9(4)
C9	0.6559(5)	0.47672(22)	0.65176(15)	3.6(3)	C41	0.6465(5)	0.54724(24)	0.49836(16)	4.3(3)
C10	0.6490(5)	0.48669(22)	0.69729(16)	3.9(3)	C42	0.6699(6)	0.49544(24)	0.48042(16)	5.1(4)
C11	0.4813(6)	0.71177(23)	0.54182(16)	4.9(3)	C43	0.6835(6)	0.49401(25)	0.43551(17)	5.8(4)
C12	0.3736(6)	0.6835(3)	0.52499(18)	6.5(4)	C44	0.6724(6)	0.5396(3)	0.40863(17)	5.6(4)
C13	0.3399(7)	0.6846(3)	0.47903(20)	8.7(5)	C45	0.6486(7)	0.5903(3)	0.42794(19)	8.3(5)
C14	0.4144(7)	0.7124(3)	0.45185(19)	9.6(5)	C46	0.6342(7)	0.5950(3)	0.47306(18)	7.6(5)
C15	0.5205(8)	0.7393(3)	0.46864(19)	9.8(6)	C47	0.6847(6)	0.5357(3)	0.35845(18)	7.7(5)
C16	0.5555(7)	0.7397(3)	0.51426(19)	7.6(4)					

(continued)

TABLE II. *(continued)*

	x	y	z	B_{iso}[a]		x	y	z	B_{iso}[a]
C1′	0.9892(6)	0.4910(3)	0.04045(19)	6.4(4)	H12	0.317(5)	0.6591(22)	0.5468(16)	10.5(18)
C2′	1.0037(14)	0.5688(5)	−0.0381(5)	10.4(11)	H13	0.258(5)	0.6606(24)	0.4682(16)	12.8(21)
C4′	0.9762(13)	0.5580(6)	0.0423(4)	8.1(9)	H14	0.379(5)	0.7061(22)	0.4234(14)	10.3(17)
C5′	0.9893(13)	0.4674(8)	0.0746(4)	10.1(11)	H15	0.570(5)	0.7595(21)	0.4518(15)	10.4(18)
C6′	1.0017(6)	0.60445(23)	−0.06841(19)	10.5(5)	H16	0.631(4)	0.7616(18)	0.5248(13)	7.3(15)
C7′	0.9798(15)	0.6164(6)	−0.0250(4)	9.0(10)	H17	0.682(4)	0.4842(16)	0.5674(12)	4.6(12)
C8′	0.9790(7)	0.5881(3)	0.01020(21)	7.8(5)	H22	0.317(5)	0.6267(20)	0.6253(15)	9.2(17)
C9′	0.9901(14)	0.5252(5)	0.0067(5)	5.4(8)	H23	0.148(6)	0.593(3)	0.6783(20)	9.9(27)
C17′	0.9822(10)	0.5078(5)	0.0909(3)	5.3(7)	H24	0.153(5)	0.6460(24)	0.7462(21)	8.0(23)
C41′	0.9805(10)	0.4923(5)	0.1680(3)	4.5(7)	H25	0.322(4)	0.7424(18)	0.7526(17)	9.1(16)
C42′	0.9912(12)	0.5481(5)	0.1783(3)	5.2(7)	H26	0.435(4)	0.7651(16)	0.7000(11)	4.6(12)
C43′	1.0023(13)	0.5633(6)	0.2250(4)	7.2(9)	H32	0.804(4)	0.6599(19)	0.6193(14)	7.5(15)
C44′	0.997(8)	0.5213(5)	0.255(3)	7.7(17)	H33	0.990(5)	0.6822(21)	0.6413(15)	10.8(18)
C45′	0.977(3)	0.4628(5)	0.2415(8)	5.6(12)	H34	1.048(4)	0.7647(17)	0.6847(12)	5.4(13)
C46′	0.9676(10)	0.4473(5)	0.1974(4)	5.6(7)	H35	0.898(4)	0.8280(16)	0.6928(12)	4.4(12)
C47′	0.9962(11)	0.5385(7)	0.3039(3)	7.9(9)	H36	0.683(4)	0.8162(18)	0.6599(13)	6.3(14)
H2	0.602(4)	0.5864(16)	0.5512(12)	4.3(11)	H42	0.690(4)	0.4576(19)	0.5000(14)	6.3(14)
H3	0.590(3)	0.6227(15)	0.6904(11)	3.0(10)	H43	0.699(4)	0.4620(18)	0.4250(12)	5.8(13)
H4	0.620(3)	0.5506(15)	0.7401(12)	3.4(10)	H45	0.623(4)	0.6172(17)	0.4123(12)	5.4(13)
H5	0.662(4)	0.4536(17)	0.7602(14)	6.2(13)	H46	0.629(4)	0.6288(18)	0.4865(13)	6.0(13)
H6	0.692(4)	0.3585(18)	0.7344(13)	6.5(14)	H471	0.614(4)	0.5484(20)	0.3402(14)	8.1(15)
H7	0.737(5)	0.3441(21)	0.6634(15)	10.0(17)	H472	0.807(7)	0.556(3)	0.3538(23)	12.1(33)
H8	0.689(4)	0.4098(18)	0.6096(13)	6.2(13)	H473	0.743(6)	0.506(3)	0.3574(20)	9.1(28)

[a] B_{iso} is the mean of the principal axes of the thermal ellipsoid. e.s.d.s in parentheses refer to the last digit printed.

in the unit cell: (i) one string parallel to the bc plane at $y = ½$ (along the *b* direction); (ii) a second string, also parallel to the *bc* plane, intersecting the *ac* plane at $x = ½$.

The distance separating the dimers and the strings of free ligand averages about 3.5 Å, and there are stacking interactions between them which probably explains the observed disorder of the free ligand.

This most unusual structure, whose solution turend out to be quite challenging, provides yet another example of the reluctance of triorganotin chloride and isothiocyanate complexes to adopt a six-coordinate configuration.

Supplementary Material

The following items of additional data have been deposited as supplementary material, and are available from the Editor-in-Chief:

(a) For both 120 K and 295 K structures – anisotropic thermal parameters of all atoms, fractional coordinates of H atoms and their final geometries, mean-plane calculations and the observed and calculated structure factors.

(b) Additionally for the 120 K structure – fractional coordinates for non-hydrogen atoms, thermal parameters for non-hydrogen atoms, and fractional

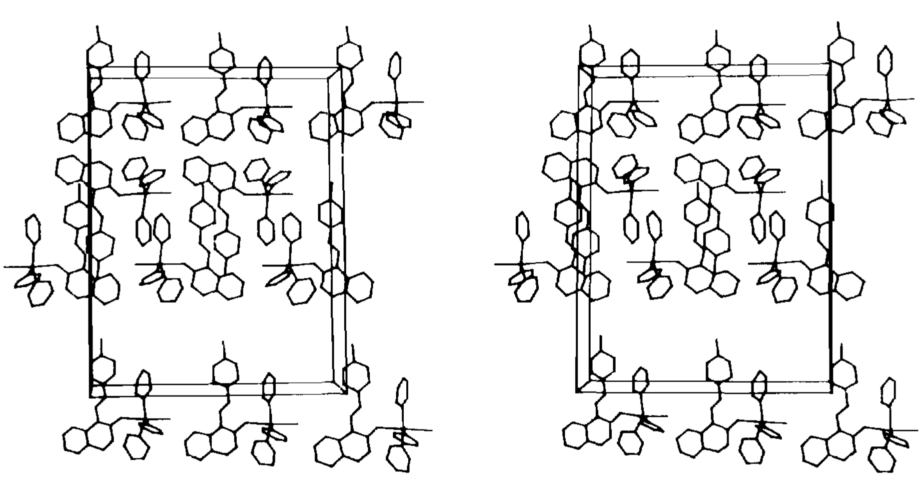

Fig. 3. Stereoview of the packing arrangement of organotin units within the unit cell.

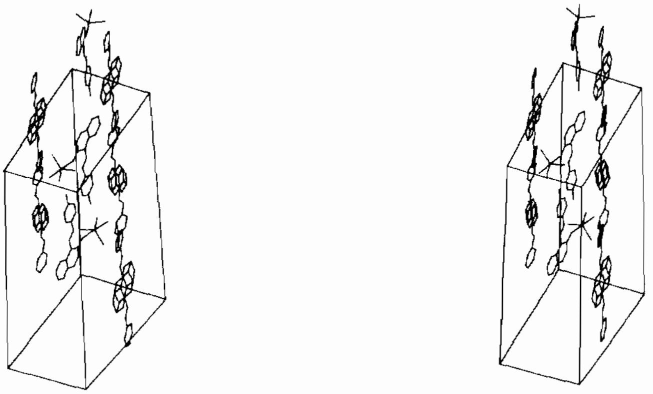

Fig. 4. Stereoview of the packing of the free ligand strings.

and thermal parameters of H atoms along with geometries.

(c) Another Figure, illustrating the disorder in the cocrystallised free ligand.

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