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### The Crystal Structure of Tetraphenyldichloro( $\mu$ -hydroxo)-Distannate-quinoline

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## THE CRYSTAL STRUCTURE OF TETRAPHENYLDICHLORO( $\mu$ -HYDROXO)- DISTANNATE · QUINOLINE

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Hydrolysis of diphenyltin dichloride in the presence of quinoline and diethyl ether proceeds via formation of a monomeric diorganodistannoxane compound. The single crystal X-ray diffraction study of  $[\text{Sn}(\text{C}_6\text{H}_5)_2\text{Cl}(\mu\text{-OH})]_2 \cdot \text{quin}$  is reported together with IR and H-NMR data. The dimer consists of five-coordinate tin(IV) units with di- $\mu$ -hydroxo bridging. The coordination geometry of the tin unit is a distorted trigonal bipyramid with two phenyl groups and one  $\mu$ -hydroxo in equatorial positions and the other  $\mu$ -hydroxo and chlorine atoms in axial positions.

**Keywords:** Tin; Quinoline; Stannoxane; Crystal structure; Hydrolysis

### INTRODUCTION

The chemistry of tin has been an active area for a considerable time, both in the fields of coordination and organometallic chemistry. Organotin complexes may interact with biological systems in many different ways, as, for instance, bactericides, fungicides, and agricultural biocides, where the triorganotin derivatives are most useful. The advantage of these compounds is their selectivity, posing little danger to mammalian species and the fact that their degradation products are completely nontoxic [1]. In recent years, several investigations have been carried out to test their antitumor activity, and it has been observed that several organotin complexes are effective antineoplastic agents. The mechanism of their activity may involve the transportation of the complexed organotin compound into the tumor cells followed by reaction of uncomplexed organotin and its hydrolysis product at the active sites. This suggestion is supported by the fact that the hydrolysis product is active [2]. Hydrolysis of diorganotin and monoorganotin compounds,  $\text{R}_2\text{SnCl}_2$  and  $\text{RSnCl}_3$ , results in formation of four-membered cyclic distannoxane rings [3]. One distannoxane ring is present in the structure of dimethyltin nitrate hydroxide  $[(\text{CH}_3)_2\text{Sn}(\text{NO}_3)\text{OH}]_2$ , a dimer through bridging

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hydroxyl groups with distorted trigonal-bipyramidal geometry [4], and in the dimeric structure of six-coordinated ethyldichlorotin oxide monohydrate  $[(C_2H_5)_2SnCl_2OH(H_2O)]_2$  [5].

As an extension of the research on tin compounds with heterocyclic derivatives of biological and pharmaceutical importance, we report here the synthesis and solid state X-ray characterization of  $[Sn(C_6H_5)_2Cl(\mu-OH)]_2 \cdot$  quinoline.

## EXPERIMENTAL

Quinoline ( $C_9H_7N$ ) and diphenyltin dichloride ( $Ph_2SnCl_2$ ) were used as received. Diethyl ether was refluxed with calcium hydride to remove water followed by distillation.

### Synthesis

Diphenyltin dichloride (0.25 mmol) was dissolved in cold diethyl ether (25 mL), stirred and quinoline (3 mmol) was slowly added to the above solution. The reaction mixture was stirred at  $10^\circ C$  during about 2 h and then, after standing for 24 h at  $6^\circ C$ , the solution was evaporated to 10 mL and then was refrigerated. After three days the white crystals that formed were washed with the same solvent. M.p.  $123\text{--}125^\circ C$ . Anal. Found: C, 55.69; H, 3.92; N, 3.20. Anal. Calc.: C, 55.27; H, 3.74; N, 3.47,  $[Sn(C_6H_5)_2Cl(\mu-OH)]_2 \cdot$  (quin). Yield: 30%.

### Crystal Structure Determination

A single crystal of dimensions  $0.20\text{ mm} \times 0.20\text{ mm} \times 0.30\text{ mm}$  was mounted on a thin glass fiber using a clear epoxy adhesive. X-ray data were collected on a Siemens SMART CCD diffractometer equipped with a normal focus, 2.4 kW sealed tube X-ray source (Mo  $K\alpha$  radiation,  $\lambda = 0.71073\text{ \AA}$ ) operating at 45 kV and 40 mA. The data collection covered the full sphere of reciprocal space by a combination of five sets of exposures; each set had a different  $\varphi$  angle ( $0, 88, 180, 270^\circ$ ) for the crystal and each exposure of 12 s covered in  $0.3^\circ$  in  $\omega$  to give a total of 2252 frames. The crystal-to-detector distance was 5.029 cm and the detector swing angle was  $-30^\circ$ . Coverage of the unique set was over 99% complete. Crystal decay was monitored by repeating fifty frames from the initial set at the end of the data collection. Analysis of the duplicate reflections indicated that crystal decay was negligible. Unit cell parameters were determined by a least-squares fit of 5570 reflections in the range  $2.46^\circ < 2\theta < 56.58^\circ$ . Absorption and other corrections were made using SADABS [6].

Preliminary positions for the Sn atoms were obtained by interpretation of the Patterson map obtained with SHELXS-97 [7]. The rest of the nonhydrogen atoms were located in successive difference Fourier maps with SHELXL-97 [8]. The refinement of atomic coordinates and anisotropic thermal parameters for all nonhydrogen atoms was carried out by full matrix least squares with SHELXL-97. Hydrogen atoms in the phenyl and quinoline moieties were identified in the difference Fourier maps but were included in calculated positions using a "riding" model with aromatic C–H distances maintained at  $0.93\text{ \AA}$ . The possibility of having a protonated quinoline was examined. The analysis revealed short contacts between the nitrogen atoms of the

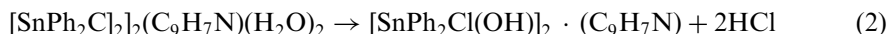
TABLE I Summary of crystal for  $[\text{Sn}(\text{C}_6\text{H}_5)_2\text{Cl}(\mu\text{-OH})]_2 \cdot (\text{C}_9\text{H}_7\text{N})$ 

Formula	$\text{C}_{24}\text{H}_{22}\text{Cl}_2\text{O}_2\text{Sn}_2, (\text{C}_9\text{H}_7\text{N})$
<i>M</i>	909.05
Crystal size (mm <sup>3</sup> )	0.20 × 0.20 × 0.30
Crystal system	Triclinic
Space group	<i>P</i> -1 <i>N</i> <sup>o</sup> 2
<i>a</i> , <i>b</i> , <i>c</i> (Å)	10.1475(6), 12.2679(7), 16.9664(10)
$\alpha$ , $\beta$ , $\gamma$ (°)	79.4400(10), 78.8610(10), 70.3970(10)
<i>U</i> (Å <sup>3</sup> )	1936.1(2)
<i>Z</i>	4
<i>D</i> (Mg m <sup>-3</sup> )	1.559
<i>F</i> (000)	904
$\mu$ (Mo <i>K</i> $\alpha$ ) (/mm)	1.5
Temperature (K)	293
Number of reflections collected	20,236
<i>R</i> <sub>int</sub> / <i>R</i> <sub><math>\sigma</math></sub>	0.0579 (8670 refxs.) / 0.0798
<i>R</i> ( <i>F</i> )/ <i>wR</i> ( <i>F</i> <sup>2</sup> )/ <i>S</i>	0.0358/0.0696/0.91
Weighting scheme	$w = 1/[\sigma^2(F_0^2) + (0.0223 P)^2 + 0.1289P]$ where $P = (F_0^2 + 2F_c^2)/3$

quinoline molecules and the oxygen atoms in the dimeric units, and residual electron density at distances less than 1.00 Å from these oxygens. This indicated the presence of bridging hydroxyl groups which form hydrogen bonds with the quinoline nitrogen atoms in the dimers. The protons of the  $\mu$ -hydroxo bridges were included in the refinement in positions obtained from the difference map. The isotropic thermal parameters for all hydrogen atoms were restricted to be equal to 1.2 times the  $U_{\text{eq}}$  of the parent carbon or oxygen atom. A summary of crystal data, data collection, and structure refinement results is presented in Table I. Fractional coordinates and isotropic thermal parameters for all nonhydrogen atoms are reported in Table II while selected bond distances and angles and hydrogen bond geometry obtained with the program PLATON [9] are summarized in Tables III and IV, respectively. Hydrogen atomic coordinates, thermal parameters, and complete lists of bond lengths and angles have been deposited with the Cambridge Crystallographic Data Center, CCDC N<sup>o</sup> 168401.

## RESULTS AND DISCUSSION

Diphenyltin dichloride reacts with commercial quinoline (1:12 molar ratio) and a hydrolysis pathway yields the distannate ring.

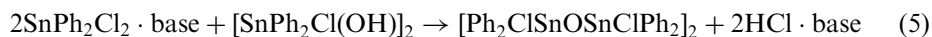
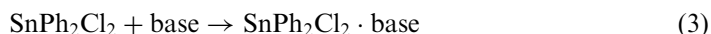


Holmes and coworkers [10] studied the base-catalyzed (base = quinuclidine, 1,4-diazobicyclo[2,2,2]octane or acridine) hydrolysis of  $\text{SnPh}_2\text{Cl}_2$  and mechanistic sequence was proposed, leading to the formation of the hydroxydistannoxane (Eqs. (3)–(5)). The hydroxyhalide species  $[\text{R}_2\text{SnX}(\text{OH})]_2$  was postulated as a precursor to the formation of the dihalodistannoxane  $\text{R}_2\text{XSnOSnR}_2\text{X}$  although no evidence was found for

TABLE II Final coordinates and equivalent isotropic displacement parameters of the nonhydrogen atoms for  $[\text{Sn}_2(\text{C}_6\text{H}_5)_4\text{Cl}_2(\text{OH})_2] \cdot (\text{C}_9\text{H}_7\text{N})$ 

Atom	x	y	z	$U_{\text{eq}} [\text{\AA}^2]$
<b>Dimer 1</b>				
Sn1	0.66141(2)	0.01533(2)	0.50201(1)	0.0348(1)
Cl1	0.89421(9)	-0.13562(9)	0.48805(7)	0.0532(4)
O1	0.5663(2)	-0.1010(2)	0.49165(6)	0.0398(9)
C10	0.7049(3)	0.1215(3)	0.3991(2)	0.0362(11)
C11	0.6201(4)	0.1537(3)	0.3320(2)	0.0449(14)
C12	0.6467(5)	0.2283(4)	0.2629(3)	0.0599(17)
C13	0.7559(5)	0.2730(4)	0.2557(3)	0.0700(19)
C14	0.8415(5)	0.2408(4)	0.3149(3)	0.0720(2)
C15	0.8168(4)	0.1654(4)	0.3831(3)	0.0533(16)
C20	0.6658(4)	0.0446(3)	0.6215(2)	0.0394(12)
C21	0.5438(4)	0.0766(3)	0.6767(3)	0.0509(17)
C22	0.5520(5)	0.0900(4)	0.7551(3)	0.0698(19)
C23	0.6782(6)	0.0753(4)	0.7788(3)	0.080(2)
C24	0.7993(6)	0.0448(4)	0.7251(3)	0.073(2)
C25	0.7943(4)	0.0288(3)	0.6464(3)	0.0536(17)
<b>Dimer 2</b>				
Sn2	0.42584(3)	0.38848(2)	0.01307(2)	0.0423(1)
Cl2	0.54733(9)	0.18404(9)	-0.00301(8)	0.0778(5)
O2	0.3885(2)	0.5777(2)	0.01464(9)	0.0461(9)
C40	0.2978(4)	0.4231(4)	-0.0785(2)	0.0491(16)
C41	0.1623(4)	0.5000(4)	-0.0683(3)	0.0627(19)
C42	0.0716(5)	0.5190(5)	-0.1233(3)	0.082(2)
C43	0.1131(7)	0.4604(6)	-0.1883(4)	0.064(3)
C44	0.2478(7)	0.3848(5)	-0.2014(3)	0.093(3)
C45	0.3389(5)	0.3654(4)	-0.1457(3)	0.0734(19)
C50	0.3449(4)	0.3652(3)	0.1377(2)	0.0437(17)
C51	0.3077(4)	0.2659(4)	0.1702(3)	0.0588(14)
C52	0.2528(5)	0.2498(4)	0.2496(3)	0.074(2)
C53	0.2354(5)	0.3310(5)	0.2989(3)	0.0741(19)
C54	0.2716(4)	0.4307(4)	0.2684(3)	0.0652(19)
C55	0.3274(4)	0.4476(4)	0.1875(2)	0.0523(17)

the presence of this transient species, considered to be responsible for the lability of this class of substances. The presence of water impurities must be considered.



In our case, the base reaction with  $\text{SnPh}_2\text{Cl}_2$  stabilizes the hydroxo transient species in the formation of the covalent compound. The structure consists of two crystallographically independent  $[\text{Sn}_2(\text{C}_6\text{H}_5)_4\text{Cl}_2(\text{OH})_2]$  dimers and two quinoline molecules. Figure 1 shows the geometry of each dimer as well as the atom labeling scheme.

The coordination geometry of the tin atoms can be described as distorted trigonal bipyramidal, with the phenyl groups and the shorter Sn–O(H) bond in the equatorial plane. The longest Sn–O(H) bond (see Table III) and the Cl atoms occupy the apical positions. However, the bond lengths around Sn1 are different than those

TABLE III Selected bond distances (Å) and bond angles (°) for  $[\text{Sn}_2(\text{C}_6\text{H}_5)_4\text{Cl}_2(\text{OH})_2]$ 

Sn1–Cl1	2.4648(11)	Sn2–Cl2	2.4353(12)
Sn1–O1	2.0200(2)	Sn2–O2	2.227(2)
Sn1–C10	2.120(3)	Sn2–C40	2.110(4)
Sn1–C20	2.134(3)	Sn2–C50	2.119(3)
Sn1–O1 <sup>a</sup>	2.184(2)	Sn2–O2 <sup>b</sup>	2.013(2)
O1–H1	0.87(2)	O2–H2	0.91(3)
C11–Sn1–O1	90.48(7)	C11–Sn1–O1 <sup>a</sup>	160.95(7)
C11–Sn1–C10	95.12(12)	Sn1–O1–H1	122(2)
C11–Sn1–C20	96.15(11)	C12–Sn2–C50	97.92(10)
O1–Sn1–C10	115.18(12)	O1–Sn1–C20	117.20(13)
O1–Sn1–O1 <sup>a</sup>	70.49(9)	O1 <sup>a</sup> –Sn1–C10	92.47(11)
C10–Sn1–C20	126.12(14)	O1 <sup>a</sup> –Sn1–C20	93.40(13)
C40–Sn2–C50	122.01(16)	Sn1 <sup>a</sup> –O1–H1	128(2)
Sn2–O2–H2	133.2(19)	O2–Sn2–C40	92.20(14)
Sn2 <sup>b</sup> –O2–H2	117.2(19)	Cl2–Sn2–O2 <sup>b</sup>	90.23(8)
C12–Sn2–O2	160.69(7)	C12–Sn2–C40	96.51(13)
O2–Sn2–O2b	70.49(9)	O2–Sn2–C50	91.94(12)
Sn1–O1–Sn1 <sup>a</sup>	109.51(10)	Sn2–O2–Sn2 <sup>b</sup>	109.51(10)

<sup>a</sup>1 – x, – y, 1 – z; <sup>b</sup>1 – x, 1 – y, – z.

TABLE IV Hydrogen bond geometry for  $[\text{Sn}_2(\text{C}_6\text{H}_5)_4\text{Cl}_2(\text{OH})_2] \cdot (\text{C}_9\text{H}_7\text{N})$ 

	<i>Don–H</i> (Å)	<i>H ... Acc</i> (Å)	<i>Don ... Acc</i> (Å)	<i>Don–H ... Acc</i> (°)
O1 ... H1 ... N1 <sup>a</sup>	0.87(2)	1.89(2)	2.756(4)	171(3)
O2 ... H2 ... N2 <sup>b</sup>	0.91(3)	1.91(3)	2.787(5)	163(3)
C55–H55 ... O2	0.9298	2.5478	3.127(4)	120.76

<sup>a</sup>1 – x, – y, 1 – z; <sup>b</sup>– x, 1 – y, – z.

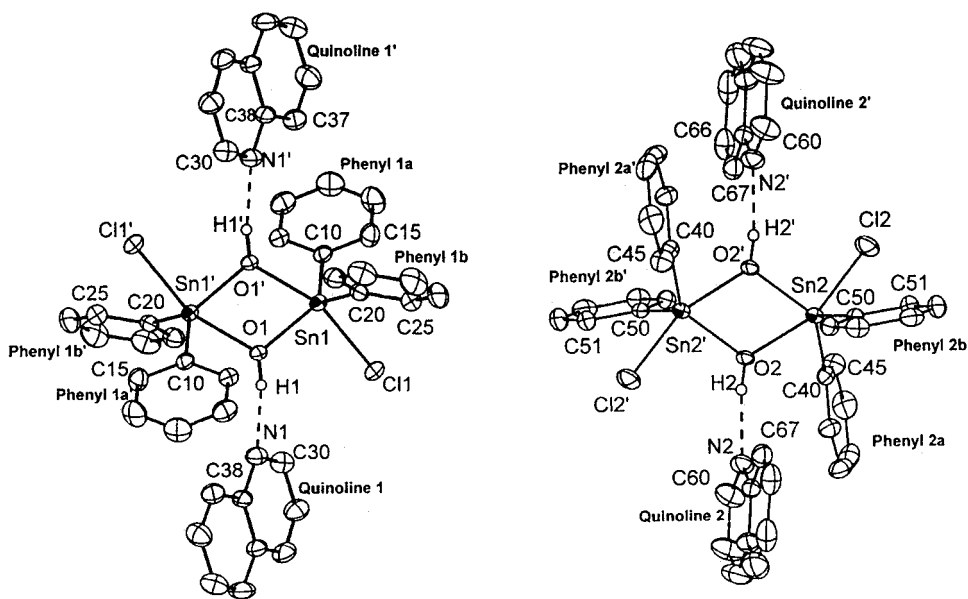


FIGURE 1 The atom-labeling scheme of the dimer  $[\text{Sn}_2(\text{C}_6\text{H}_5)_4\text{Cl}_2(\mu\text{-OH})_2] \cdot (\text{C}_9\text{H}_7\text{N})$ . All hydrogen atoms have been omitted.

around Sn2. The Sn1 to Cl1 bond is longer than the corresponding bond for Sn2 (Sn1–Cl1 2.4648(11) vs Sn2–Cl2 2.4353(12) Å) but for the apical oxygens, the Sn1–O1<sup>a</sup> bond is shorter than the Sn2–O2 bond (Sn1–O1<sup>a</sup> 2.184(2) vs Sn2–O2 2.227(2) Å). For the atoms in the basal plane, the distances to Sn1 are longer than the distances to Sn2. In the dimers, the trigonal pyramids that can be described around each Sn atom share an edge defined by O1–O1<sup>a</sup> in the case of Sn1 and by O2–O2<sup>b</sup> for Sn2. Figure 2 shows a diagram of the coordination sphere of the Sn atoms with the unique bonds labeled.

The centers of the four-membered [Sn(OH)<sub>2</sub>]<sub>2</sub> rings in each dimer occupy centers of inversion with coordinates 1/2, 0, 1/2 for dimer **1** and 1/2, 1/2, 0 for dimer **2**. The distribution of the dimers in the unit cell gives rise to a “honeycomb” type of arrangement

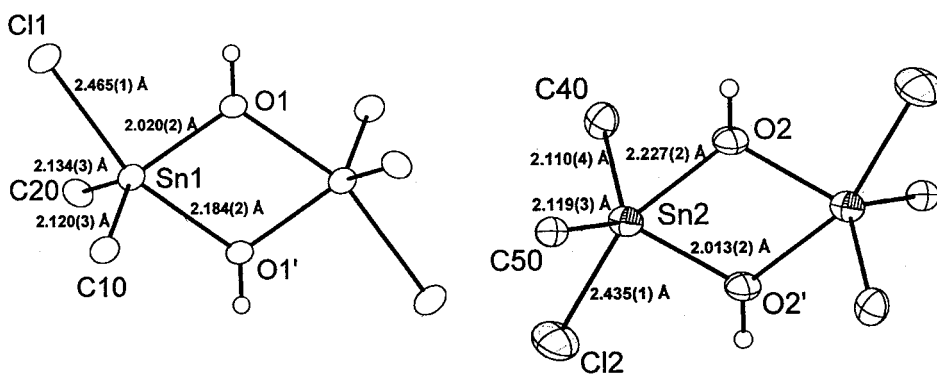


FIGURE 2 Diagram of the coordination sphere of the Sn atoms with the unique bonds labeled.

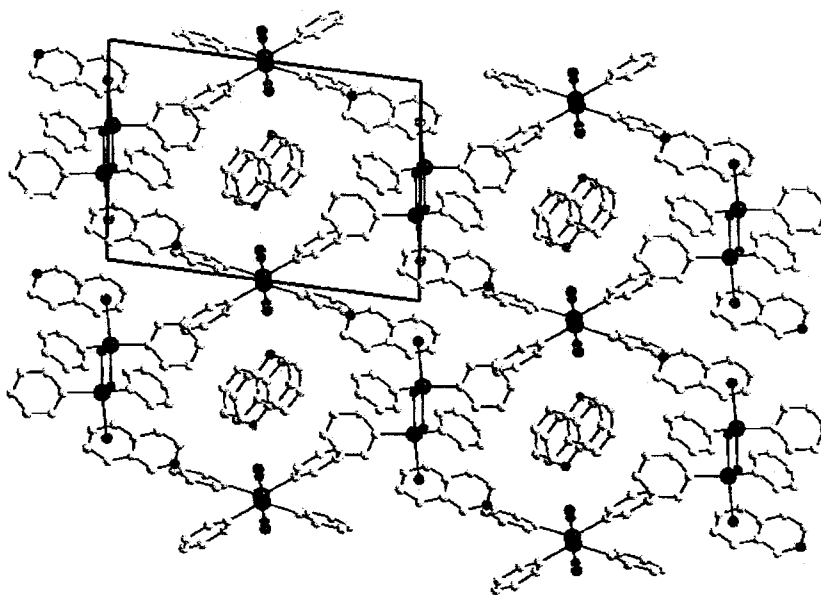


FIGURE 3 View of the unit cell of [Sn<sub>2</sub>(C<sub>6</sub>H<sub>5</sub>)<sub>4</sub>Cl<sub>2</sub>(μ-OH)<sub>2</sub>] · (C<sub>9</sub>H<sub>7</sub>N) down the *a* axis.

with open channels which run parallel to the *a* and *b* axes, as shown in Fig. 3. The two independent quinoline molecules are located within these channels. Quinoline molecule **1** (which contains N1, and C30 to C38) stack along the *a* axis and form angles of 57.88(18)° with quinoline molecules **2** (N2, C60 to C68) which stack along the *b* axis. The two arrays of quinoline molecules produce a lattice with rhombohedral holes occupied by the [Sn<sub>2</sub>(C<sub>6</sub>H<sub>5</sub>)<sub>4</sub>Cl<sub>2</sub>(OH)<sub>2</sub>] dimers. Therefore, the structure can be described either as a network formed by [Sn<sub>2</sub>(C<sub>6</sub>H<sub>5</sub>)<sub>4</sub>Cl<sub>2</sub>(OH)<sub>2</sub>] dimers with quinoline molecules occupying the voids, or as an array of quinoline molecules with [Sn<sub>2</sub>(C<sub>6</sub>H<sub>5</sub>)<sub>4</sub>Cl<sub>2</sub>(OH)<sub>2</sub>] dimers in the voids.

As mentioned before, the nitrogen atoms of quinoline molecules participate in hydrogen bonds of medium strength with the hydroxo groups of each dimer, based on distance and angle criteria and taking into account the electronegativity of the acceptor and donor atoms [11] (see Table IV). Given the centrosymmetric nature of the units, two quinoline molecules related by a center of symmetry are in hydrogen-bonding contact with one [Sn<sub>2</sub>(C<sub>6</sub>H<sub>5</sub>)<sub>4</sub>Cl<sub>2</sub>(OH)<sub>2</sub>] dimer. As a result, dimer **1** and quinoline **1** form isolated chains of finite contacts N1...H1-O1-Sn1-O1<sup>a</sup>-H1<sup>a</sup>...N1<sup>a</sup> along the *b* axis [O1...N1 distance 2.756(4) Å and O1-H1...N1 angle 171(3)°]. Similarly, dimer **2** and quinoline **2** form a chain of contacts N2...H2-O2-Sn2-O2<sup>b</sup>-H2<sup>b</sup>...N2<sup>b</sup> along the *a* axis [O2...N2 distance 2.787(5) Å and O2-H2...N2 angle 163(3)°].

A search of the Cambridge Structural Database (CSD, Version 5.20 October 2000) [12] indicated one closely related structure (Refcode: TISVUM) among the oxo-chloro-diphenyl tin derivatives, namely bis(2-ammoniopyrimidine)bis((μ<sub>2</sub>-oxo)-chloro-diphenyl-tin(IV)) [13], based on units with only two tin atoms. Other structures are based on tetra-tin units, for example bis(μ<sub>3</sub>-oxo)-bis(μ<sub>2</sub>-hydroxo)-dichloro-octaphenyl-tetra-tin acetone solvate (CIJJEK) and bis(μ<sub>3</sub>-oxo)-bis(μ<sub>2</sub>-chloro)-dichloro-octaphenyl-tetra-tin (CIJJIO) [10], bis(chloro-(μ<sub>2</sub>-hydroxo)-(μ<sub>2</sub>-oxo)-tetraphenyl-di-tin) dimethylformamide solvate (JIGJAK) [14], bis(μ<sub>3</sub>-oxo)-bis(μ<sub>2</sub>-hydroxo)-dichloro-octaphenyl-tetra-tin(IV) (LAZVUD and LAZVUD01) [15,16]. However, when the source of coordinating oxygen atoms is provided by nitrate, dimethylsulfoxide, or in general bulkier ligands, the structures tend to be monomeric. Examples of these structures are tetrakis(triphenylarsine)-silver chloro-diphenyl-bis-(nitrato-*O,O'*)-tin [17] and *cis*-dichloro-*cis*-bis(dimethylsulfoxide)-*trans*-diphenyltin [18].

The IR spectrum shows bands at 3580 and 380 cm<sup>-1</sup> attributable to ν(O-H) and ν(Sn-Cl) respectively, two bands at 450 and 440 cm<sup>-1</sup> assignable to ν<sub>as</sub>(Sn-O) and two bands at 280 and 260 cm<sup>-1</sup> assignable to ν<sub>as</sub>(Sn-C), indicating nonlinear Sn-O<sub>2</sub> and Sn-C<sub>2</sub> moieties [19]. The H-NMR spectrum shows, besides the aromatic proton resonances, a triplet at 2.55 ppm due to the tin-OH coupling indicating the presence of the [Sn(OH)]<sub>2</sub> ring. These arrangements are consistent with the X-ray diffraction study.

Summarized in Table V is a comparison of bond parameters of related tin(IV) compounds that have the distannoxane ring system. The range of Sn-O bond lengths is narrow, 2.01–2.23 Å, coinciding with the single bond covalent radius value of 2.13 Å [19,20], reflecting the strong coordination within the dimer, and consistent with similar bond lengths in other distannoxanes. The range of Sn-Cl bonds is also surprisingly narrow, 2.42–2.48 Å. The range of O-Sn-O and Sn-O-Sn bond angles, 69.6–74.7° and 100.5–110.4° respectively, are close to one another for all the distannoxanes. The Sn-C bonds to both tins are consistent with the values observed in other phenyltin derivatives [21].



TABLE V Bond lengths and bond angles for dimeric tin(IV) derivatives with distannoxane ring

Compound	Bond length		Bond angle		Reference
	Sn–O	Sn–Cl	O–Sn–O	Sn–O–Sn	
[SnEt(OH)(OH <sub>2</sub> )Cl <sub>2</sub> ] <sub>2</sub>	2.110	2.423	70.9	109.1	[4]
[SnBu(OH)(OH <sub>2</sub> )Cl <sub>2</sub> ] <sub>2</sub>	2.047	2.484	69.6	110.4	[22]
	2.169	2.419			
(C <sub>3</sub> H <sub>5</sub> N <sub>2</sub> ) <sub>2</sub> [SnMe <sub>2</sub> (NO <sub>3</sub> ) <sub>2</sub> (OH) <sub>2</sub> ]	2.129	–	74.7	100.5	[23]
	2.162	–			
[SnPh <sub>2</sub> Cl(μ–OH)] <sub>2</sub> Dimer <b>1</b>	2.020	2.4648	70.5	109.5	This work
	2.184				
[SnPh <sub>2</sub> Cl[(μ–OH)] <sub>2</sub> Dimer <b>2</b>	2.227	2.4353	70.5	109.5	This work
	2.013				

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