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TRIPHENYLTIN(IV) 8-QUINOLYLOXYACETATE HYDRATE, $[Ph_3SnO_2CCH_2(8\text{-}C_9H_6NO) \cdot H_2O]_n$, AN ORGANOTIN ESTER DERIVATIVE BUILT OF HYDROGEN-BONDED HELICAL CHAINS

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Summary

The X-ray crystal structure of triphenyltin(IV) 8-quinolyloxyacetate hydrate, $[Ph_3SnO_2CCH_2(8\text{-}C_9H_6NO) \cdot H_2O]_n$, has been determined and refined to $R_F = 0.066$. The compound crystallizes in space group $P2_1/c$ with a 13.667(5), b 10.340(5), c 35.92(1) Å, β 93.01(3)° and $Z = 8$. The asymmetric unit consists of two independent $[Ph_3SnO^IC(O^{II})CH_2(8\text{-}C_9H_6NO^{III}) \cdot H_2O^{IV}]$ molecular units (**A** and **B**) which differ in the extent of the intramolecular approach of the carbonyl oxygen O^{II} to tin ($Sn \cdots O^{II}$ 3.53(1) and 3.20(1) Å, respectively). The coordination environments of tin in **A** and **B** are in each case essentially trigonal bipyramidal, with the *ipso*-carbons of the phenyl groups lying in the equatorial plane and the axial positions being occupied by oxygen from a unidentate carboxylate group and a water molecule. The Sn–O bond lengths involving coordinated water are approximately 0.22 Å longer than those involving the carboxylate groups; the axial angles, O^ISnO^{IV}, in **A** and **B** are respectively 175.9(3) and 176.6(3)°. Molecules **A** and **B** are linked sequentially by hydrogen bonds involving the coordinated water molecule and the quinolyloxyacyl O^{III} and N atoms to form one-dimensional helical chains parallel to the a axis. The slope of the variable-temperature ^{119m}Sn Mössbauer plot ($a = d\ln(A_T/A_{80\ K})/dT = -1.99 \times 10^{-2}\ K^{-1}$ for $80 \leq T \leq 110$) is consistent with the model of a weakly associated lattice.

Introduction

Recent X-ray studies on hydrated organotin(IV) derivatives have yielded examples of both inner-sphere and outer-sphere coordination for the water molecules and have drawn attention to the important role played by hydrogen-bonding interactions in determining the molecular packing.

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Coordinated water molecules, for example, have been authenticated among triorganotin compounds in Me_3SnO_2C-2-py · H_2O [1], [n-$Bu_3Sn(H_2O)_2]^+$ [$C_5(CO_2Me)_5]^-$ [2], $Me_3SnO_3SPh \cdot H_2O$ [3], $Me_3SnNO_3 \cdot H_2O$ [4] and $Ph_3SnX \cdot L \cdot H_2O$ (X = Cl: L = 1,10-phenanthroline [5], 3-[2-(1,10-phenanthrolyl)]-5,6-diphenyl-1,2,4-triazine [6]; L = 2,2′: 6′, 2″-terpyridine; X = Cl [7,8], NCS [9]), with tin–water ($Sn-O_w$) bond distances in the range 2.30–2.47 Å. In the case of the above-mentioned triphenyltin complexes, the chelating ligands do not coordinate to tin but serve merely to bind the monomeric [$Ph_3SnX \cdot H_2O$] units through hydrogen bonding with the heterocyclic nitrogens. This bonding choice has been taken as further evidence for the preference for pentacoordination over hexa- or heptacoordination in triorganotin(IV) systems. The pentacoordinated tin environment similarly prevails in the protonated picolinic and quinaldic acid derivatives $Ph_3(NCS)SnO_2C$-2-pyH · H_2O [10] and Ph_3ClSnO_2C-2-quinH · H_2O [11], respectively, both of which constitute examples of outer-sphere or secondary coordination by the water molecules which link two monomeric tin units in such a way that each water moiety hydrogen bonds to the non-coordinated carboxylate oxygen atoms of both picolinic (or quinaldic) acid molecules and the heterocyclic nitrogen atom of one picolinic (or quinaldic) acid. In both of these protonated carboxylic acid derivatives and in Me_3SnO_2C-2-py · H_2O [1], the absence of bridging by the carboxylate moiety (a general feature for triorganotin carboxylates) may be attributed to the hydrogen-bonding interactions provided by the water molecules. This view also finds support in the crystal structure of Ph_3ClSnO_2C-2pyH [12], which reveals a unidentate ester linkage and strong inter- and intramolecular hydrogen-bond involvement by the acidic proton bonded to the heterocyclic nitrogen atom.

In the light of the foregoing, the interpretation of the structure of Ph_3SnOAc [13] as a distorted *meridional*-[R_3SnX_3] octahedron about tin is unique and intriguing. Although another example of six-coordinated triorganotin exists for the case of $Me_3Sn[HB(pz)_3]$, where pz = 1-pyrazolyl [14], it is noteworthy that spectroscopic studies on the 1 : 1 complexes of $Ph_3SnO_2CCCl_3$ [15] and $Ph_3SnO_2CCF_3$ [16] with nitrogen, oxygen and sulfur donor ligands show the carboxylate linkages to be unidentate, and hence the complexes to be essentially five-coordinated. The same is presumably also the case for the adduct $Ph_3SnOAc \cdot N(CH_2CH_3OH)_3$ [17], whose structure has not yet been clarified. Seemingly, coordination of triorganotin esters to external ligands proceeds with attendant disruption of intermolecular carboxylate bridging, a result which is perhaps reconcilable on steric grounds. In as much as the hexacoordinated character of Ph_3SnOAc depends on the additional intramolecular chelation (although weak) by the acyl bridging oxygen (Sn · · · O 3.206 Å [13]), we considered the possibility of introducing an intramolecular bonding option in Ph_3SnOAc by means of a quinolinyl substituent on the acyl β-carbon. This group is widely used in complexometry, and recent studies have strongly suggested a chelating mode for the 8-quinolinyl ligand towards triorganotin acceptor moieties [18,19]. Furthermore, when conjoined to the acetate unit, the quinolinyl group yields a skeletal framework rather akin to a *nido*-crown ether. We thus hoped that the resulting 8-quinolyloxyacetate group might provide ample scope for higher-than-five coordination at the metal center when ligated to triphenyltin. An X-ray crystal structure analysis was therefore conducted on the title compound to examine this possibility, and also to clarify the role of the water molecule in the structure.

Experimental

Synthesis

The title compound was prepared by treating triphenyltin chloride with the potassium salt of (8-quinolyloxy)acetic acid [20] in stoichiometric proportion in aqueous ethanol at room temperature. Addition of water to the stirred mixture caused precipitation of the product. Single crystals of the compound were obtained by slow evaporation of its solution in a mixed ethanol/chloroform medium. The compound does not melt, but chars over a very wide temperature range. C,H,N analysis: found (calc) (%): C 60.68 (61.08), H 4.35 (4.04); N 2.24 (2.46).

Mössbauer data

The Mössbauer data were obtained on a Cryophysics Microprocessor Mössbauer Spectrometer equipped with a LakeShore Cryotronics, Inc. Temperature Controller. The velocity range was calibrated with $CaSnO_3$ and β-Sn against the $Ca^{119m}SnO_3$ source and the spectra were fitted with a curve-fitting program supplied by the manufacturer. A 50 mg sample (corresponding to approximately 1 mg ^{119}Sn per cm^2 cross-sectional area), finely ground in carbon to eliminate orientation effects, was used in the determinations. Mössbauer data (80 K): isomer shift (*IS*) 1.30, quadru-

(*Continued on p. 38*)

TABLE 1

DATA PROCESSING AND COLLECTION PARAMETERS

Molecular formula	$C_{29}H_{23}NO_3Sn \cdot H_2O$	
Molecular weight	570.21	
Cell constants	a 13.667(5) Å	V 5069(3) Å^3
	b 10.340(5) Å	$F(000) = 2303.7$
	c 35.92(1) Å	$Z = 8$
	β 93.01(3)°	
Density (exptl)	1.497 g cm^{-3} (KI/H_2O)	
Density (calcd)	1.494 g cm^{-3}	
Space group	$P2_1/c$	
Radiation	graphite-monochromatized Mo-K_α ($\lambda = 0.71069$ Å)	
Absorption coefficient	10.43 cm^{-1}	
Crystal size	0.36×0.28×0.18 mm	
Mean μ_r	0.14	
Transmission factors	0.631 to 0.723	
Scan type and speed	$\omega - 2\theta$; 2.02–8.37 deg min^{-1}	
Scan range	1° below K_{α_1} to 1° above K_{α_2}	
Background counting	stationary counts for one-half of scan time at each end of scan	
Collection range	$h, k, \pm l$; $2\theta_{max} = 48°$	
Unique data measured	6754	
Observed data with $\|F_o\| > 3\sigma(\|F_o\|)$, n	5661	
Number of variables, p	379	
$R_F = \Sigma\|\|F_o\| - \|F_c\|\|/\Sigma\|F_o\|$	0.066	
Weighting scheme	$w = [\sigma^2(F_0) + 0.001\|F_0\|^2]^{-1}$	
$R_G = [\Sigma w(\|F_o\| - \|F_c\|)^2/\Sigma w\|F_o\|^2]^{1/2}$	0.096	
$S = [\Sigma w(\|F_o\| - \|F_c\|)^2/(n-p)]^{1/2}$	2.104	
Residual extrema in final difference map	+1.09 to −0.80 e Å^{-3}	

TABLE 2

ATOMIC COORDINATES ($\times 10^5$ for Sn, $\times 10^4$ for other atoms) AND TEMPERATURE FACTORS ($Å^2 \times 10^4$ for Sn, $\times 10^3$ for other atoms)

Atom	x	y	z	U_{eq} [a]
Molecule A				
Sn(1)	61637(5)	20903(7)	40007(2)	494(3)*
O_w(1)	5055(4)	1261(7)	3522(2)	55(2)*
O(1)	7163(5)	2969(7)	4413(2)	64(3)*
O(2)	8565(5)	3318(8)	4125(2)	66(3)*
O(3)	9019(4)	5297(6)	4587(2)	51(2)*
N(1)	10638(5)	6599(8)	4448(2)	53(3)*
C(1)	7979(7)	3567(11)	4359(3)	53(4)*
C(2)	8132(7)	4631(11)	4641(3)	55(4)*
C(3)	9242(6)	6289(10)	4820(3)	45(3)*
C(4)	8724(8)	6673(11)	5103(3)	65(4)*
C(5)	9043(9)	7691(12)	5344(3)	74(5)*
C(6)	9863(8)	8350(12)	5289(3)	70(5)*
C(7)	10432(8)	8004(10)	4977(3)	58(4)*
C(8)	10120(7)	6950(9)	4744(3)	45(3)*
C(9)	11454(8)	7245(12)	4400(4)	77(5)*
C(10)	11829(9)	8231(12)	4629(4)	79(5)*
C(11)	11303(8)	8635(12)	4888(4)	74(5)*
C(12)	7167(4)	3806(7)	3432(2)	69(3)
C(13)	7227(4)	4791(7)	3169(2)	87(4)
C(14)	6429(4)	5607(7)	3094(2)	105(5)
C(15)	5570(4)	5438(7)	3281(2)	80(4)
C(16)	5509(4)	4453(7)	3544(2)	67(3)
C(17)	6308(4)	3637(7)	3620(2)	50(2)
C(18)	6336(4)	−871(7)	3886(2)	72(3)
C(19)	6780(4)	−2086(7)	3911(2)	86(4)
C(20)	7746(4)	−2202(7)	4051(2)	92(4)
C(21)	8270(4)	−1103(7)	4168(2)	95(4)
C(22)	7826(4)	111(7)	4144(2)	78(4)
C(23)	6860(4)	227(7)	4003(2)	47(2)
C(24)	5138(6)	2844(8)	4720(3)	105(5)
C(25)	4453(6)	2749(8)	4993(3)	121(6)
C(26)	3661(6)	1904(8)	4944(3)	110(5)
C(27)	3554(6)	1153(8)	4622(3)	127(6)
C(28)	4239(6)	1248(8)	4349(3)	104(5)
C(29)	5031(6)	2093(8)	4397(3)	57(3)
Molecule B				
Sn(2)	13642(4)	39791(7)	34719(2)	452(2)*
O_w(2)	377(4)	4457(7)	3983(2)	56(2)*
O(4)	2282(4)	3666(7)	3004(2)	55(2)*
O(5)	3428(5)	2628(8)	3347(2)	63(3)*
O(6)	4648(4)	2603(7)	2788(2)	57(3)*
N(2)	6296(5)	1274(9)	2929(2)	56(3)*
C(30)	3146(6)	3172(10)	3061(3)	50(4)*
C(31)	3803(7)	3407(12)	2749(3)	58(4)*
C(32)	5425(6)	2926(11)	2567(3)	51(4)*
C(33)	5408(8)	3829(11)	2291(3)	61(4)*
C(34)	6247(8)	4126(12)	2097(3)	69(5)*
C(35)	7085(8)	3473(11)	2167(3)	61(4)*
C(36)	7125(7)	2497(12)	2434(3)	61(4)*

TABLE 2 (continued)

Atom	x	y	z	U_{eq} [a]
Molecule B				
C(37)	6292(7)	2192(10)	2655(3)	49(3)*
C(38)	7127(7)	595(11)	3001(3)	57(4)*
C(39)	7982(7)	832(13)	2814(3)	72(5)*
C(40)	7973(7)	1732(13)	2538(3)	69(5)*
C(41)	2885(5)	4715(6)	4115(2)	70(3)
C(42)	3599(5)	5480(6)	4298(2)	91(4)
C(43)	3925(5)	6606(6)	4130(2)	85(4)
C(44)	3536(5)	6967(6)	3779(2)	73(3)
C(45)	2822(5)	6203(6)	3595(2)	63(3)
C(46)	2496(5)	5077(6)	3763(2)	49(2)
C(47)	45(4)	1640(7)	3522(2)	73(3)
C(48)	−223(4)	359(7)	3584(2)	96(4)
C(49)	492(4)	−558(7)	3683(2)	104(5)
C(50)	1475(4)	−195(7)	3720(2)	93(4)
C(51)	1743(4)	1085(7)	3658(2)	72(3)
C(52)	1028(4)	2003(7)	3559(2)	52(2)
C(53)	106(5)	4582(6)	2760(2)	61(3)
C(54)	−529(5)	5293(6)	2522(2)	69(3)
C(55)	−931(5)	6447(6)	2645(2)	72(3)
C(56)	−698(5)	6889(6)	3006(2)	90(4)
C(57)	−63(5)	6178(6)	3243(2)	75(3)
C(58)	339(5)	5024(6)	3120(2)	45(2)

[a] Starred atoms mean equivalent isotropic U_{eq} defined as one third of the trace of the orthogonalized U_{ij} tensor.

TABLE 3

BOND LENGTHS (Å) [a]

Molecule A

Sn(1)–O_w(1)	2.391(6)	Sn(1)–O(1)	2.162(7)	Sn(1)–C(17)	2.120(7)	Sn(1)–C(23)	2.148(7)
Sn(1)–C(29)	2.159(9)	O(1)–C(1)	1.298(12)	O(2)–C(1)	1.221(13)	O(3)–C(2)	1.416(11)
O(3)–C(3)	1.349(11)	N(1)–C(8)	1.355(13)	N(1)–C(9)	1.319(13)	C(1)–C(2)	1.503(15)
C(3)–C(4)	1.327(15)	C(3)–C(8)	1.420(13)	C(4)–C(5)	1.419(17)	C(5)–C(6)	1.336(17)
C(6)–C(7)	1.444(17)	C(7)–C(8)	1.426(14)	C(7)–C(11)	1.408(16)	C(9)–C(10)	1.391(17)
C(10)–C(11)	1.276(18)						

Molecule B

Sn(2)–O_w(2)	2.388(7)	Sn(2)–O(4)	2.174(6)	Sn(2)–C(46)	2.146(7)	Sn(2)–C(52)	2.121(7)
Sn(2)–C(58)	2.130(6)	O(4)–C(30)	1.294(11)	O(5)–C(30)	1.219(12)	O(6)–C(31)	1.424(12)
O(6)–C(32)	1.398(11)	N(2)–C(37)	1.368(13)	N(2)–C(38)	1.348(13)	C(30)–C(31)	1.492(14)
C(32)–C(33)	1.361(15)	C(32)–C(37)	1.429(13)	C(33)–C(34)	1.407(16)	C(34)–C(35)	1.342(16)
C(35)–C(36)	1.392(16)	C(36)–C(37)	1.456(14)	C(36)–C(40)	1.436(15)	C(38)–C(39)	1.400(14)
C(39)–C(40)	1.358(18)						

Weak interactions

Sn(1)···O(2)	3.53(1)	O_w(1)···O(5)	2.68(1)	O_w(1)···O(6)	3.01(1)	O_w(1)···N(2)	2.79(1)
Sn(2)···O(5)	3.20(1)	O_w(2)′···O(2)	2.81(1)	O_w(2)′···O(3)	3.05(1)	O_w(2)′···N(1)	2.79(1)

[a] Symmetry transformation: ′ = 1 + x, y, z.

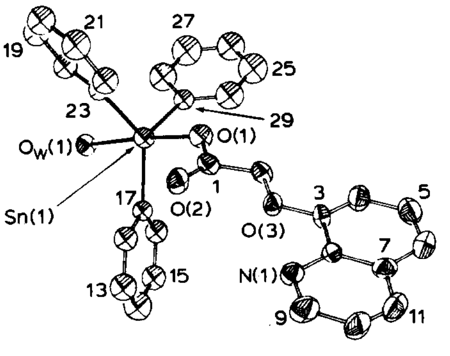

Fig. 1. Molecular geometry and atom numbering of molecule **A**.

pole splitting (*QS*) 3.57, Γ_1 1.11, Γ_2 1.10 mm s^{-1}; variable-temperature data ($80 \le T \le 110$ K): a -1.99×10^{-2} K^{-1} (7 points, correlation coefficient −0.999).

Infrared data

The IR spectrum was recorded in Nujol on a Perkin-Elmer 881 Infrared Spectrophotometer. $\nu(CO_2)_{asym}$ 1658s, 1629s, 1600m, 1575m; $\nu(CO_2)_{sym}$ 1429s, 1410s; $\nu(H_2O)$ 3400–3100b cm^{-1}.

X-Ray crystallography

Diffraction measurements were made on a Nicolet R3m four-circle diffractometer (graphite-monochromatized Mo-K_α radiation, λ 0.71069 Å), and determination of the crystal class, orientation matrix and accurate unit-cell parameters were performed by established procedures [21]. Intensities were recorded at 22°C, and data collection and processing parameters are summarized in Table 1. Application of absorption corrections was based on a pseudo-ellipsoidal fit to azimuthal scans of selected strong reflections over a range of 28 values [22,23]. The intensities were processed by the learned-profile procedure [24]. Structure solution by means of Patterson and Fourier methods revealed two independent molecules, designated **A** and **B**, corresponding to the molecular formula (Figs. 1 and 2, respectively). The phenyl rings, including their associated H atoms, were treated as rigid groups

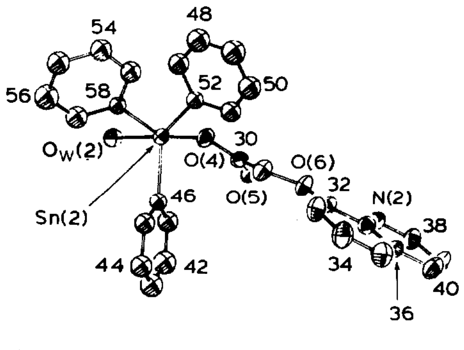

Fig. 2. Molecular geometry and atom numbering of molecule **B**.

TABLE 4

BOND ANGLES (°) [a]

Molecule A					
O_w(1)-Sn(1)-O(1)	175.9(3)	O_w(1)-Sn(1)-C(17)	83.3(2)	O(1)-Sn(1)-C(17)	92.8(3)
O_w(1)-Sn(1)-C(23)	86.9(2)	O(1)-Sn(1)-C(23)	96.3(3)	C(17)-Sn(1)-C(23)	128.5(3)
O_w(1)-Sn(1)-C(29)	91.5(3)	O(1)-Sn(1)-C(29)	89.8(3)	C(17)-Sn(1)-C(29)	121.3(3)
C(23)-Sn(1)-C(29)	109.3(3)	Sn(1)-O(1)-C(1)	128.0(7)	C(2)-O(3)-C(3)	116.8(7)
C(8)-N(1)-C(9)	116.6(9)	O(1)-C(1)-O(2)	127.0(10)	O(1)-C(1)-C(2)	109.8(8)
O(2)-C(1)-C(2)	123.2(9)	O(3)-C(2)-C(1)	110.7(8)	O(3)-C(3)-C(4)	126.1(9)
O(3)-C(3)-C(8)	114.2(8)	C(4)-C(3)-C(8)	119.7(9)	C(3)-C(4)-C(5)	121.4(11)
O(3)-C(3)-C(8)	114.2(8)	C(4)-C(3)-C(8)	119.7(9)	C(3)-C(4)-C(5)	122.0(10)
C(4)-C(5)-C(6)	121.4(11)	C(5)-C(6)-C(7)	118.7(11)	C(6)-C(7)-C(8)	119.2(9)
C(6)-C(7)-C(11)	123.8(10)	C(8)-C(7)-C(11)	116.9(10)	N(1)-C(8)-C(3)	120.3(8)
N(1)-C(8)-C(7)	120.7(9)	C(3)-C(8)-C(7)	118.9(9)	N(1)-C(9)-C(10)	125.6(12)
C(9)-C(10)-C(11)	117.8(11)	C(7)-C(11)-C(10)	121.9(11)		
Molecule B					
O_w(1)-Sn(2)-O(4)	176.6(3)	O_w(2)-Sn(2)-C(46)	86.4(2)	O(4)-Sn(2)-C(46)	91.5(3)
O_w(2)-Sn(2)-C(52)	87.2(3)	O(4)-Sn(2)-C(52)	96.2(3)	C(46)-Sn(2)-C(52)	126.4(3)
O_w(2)-Sn(2)-C(58)	88.3(2)	O(4)-Sn(2)-C(58)	90.3(2)	C(46)-Sn(2)-C(58)	117.1(2)
C(52)-Sn(2)-C(58)	115.8(2)	Sn(2)-O(4)-C(30)	119.7(6)	C(31)-O(6)-C(32)	116.1(8)
C(37)-N(2)-C(38)	118.1(8)	O(4)-C(30)-O(5)	124.2(9)	O(4)-C(30)-C(31)	113.4(8)
O(5)-C(30)-C(31)	122.3(8)	O(6)-C(31)-C(30)	110.4(8)	O(6)-C(32)-C(33)	126.3(9)
O(6)-C(32)-C(37)	113.2(9)	C(33)-C(32)-C(37)	120.5(9)	C(32)-C(33)-C(34)	121.9(10)
C(33)-C(34)-C(35)	120.4(11)	C(34)-C(35)-C(36)	119.8(10)	C(35)-C(36)-C(37)	121.9(9)
C(35)-C(36)-C(40)	125.5(10)	C(37)-C(36)-C(40)	112.5(10)	N(2)-C(37)-C(32)	120.0(9)
N(2)-C(37)-C(36)	124.6(9)	C(32)-C(37)-C(36)	115.4(9)	N(2)-C(38)-C(39)	122.2(10)
C(38)-C(39)-C(40)	119.6(10)	C(36)-C(40)-C(39)	122.9(10)		
Geometry about water oxygen atoms					
Sn(1)-O_w-O(5)	117.7(8)	Sn(1)-O_w(1)-N(2)	99.2(7)	O(5)-O_w-N(2)	110.5(10)
Sn(2)′-O_w(2)′-O(2)	126.2(8)	Sn(2)′-O_w(2)′-N(1)	124.5(7)	O(2)-O_w(2)′-N(1)	108.1(10)

[a] Symmetry transformation: ′ = 1 + x, y, z.

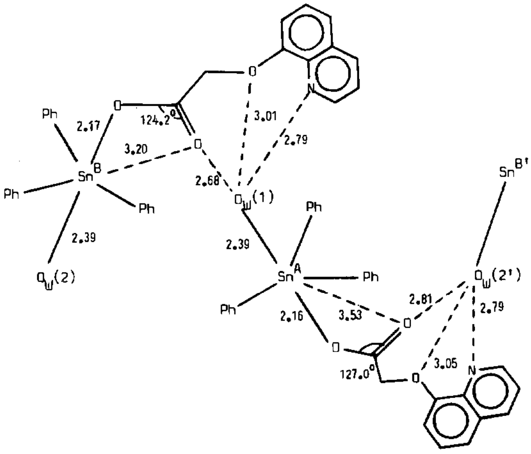

Fig. 3. Environments about the coordinated water molecules in a polymeric chain extending along the a axis. The tin atoms Sn^A and Sn^B correspond to Sn(1) and Sn(2), respectively, in Tables 2–5. Symmetry transformation: ′1 + x,y,z. Standard deviations of all interatomic distances approximately 0.01 Å.

TABLE 5

LEAST-SQUARES PLANES AND SELECTED DIHEDRAL ANGLES

Least-squares planes

Plane No.	Atoms fitted	Equation of plane [a] l	m	n	d	rms Δ ($\times 10^3$ Å)
Molecule A						
1	Sn(1), C(17), C(23), C(29)	0.6811	0.3816	0.6249	15.173	49
2	C(12)–C(17)	0.3459	0.6245	0.7002	14.114	–
3	C(18)–C(23)	0.3214	0.1139	−0.9401	−10.020	–
4	C(24)–C(29)	0.5416	−0.7272	0.4218	8.655	–
5	O(1), O(2), C(1), C(2)	0.4797	−0.6345	0.6060	12.034	4
6	O(3), N(1), C(3)–C(11)	0.5192	−0.6380	0.5686	11.867	26
Molecule B						
7	Sn(2), C(46), C(52), C(58)	0.5793	−0.2094	−0.7877	−9.606	44
8	C(41)–C(46)	0.7140	−0.5232	−0.4652	−6.159	–
9	C(47)–C(52)	−0.1022	0.2067	0.9731	12.652	–
10	C(53)–C(58)	0.7775	0.5055	−0.3741	−1.198	–
11	O(4), O(5), C(30), C(31)	0.3058	0.8670	0.3935	8.414	11
12	O(6), N(2), C(32)–C(40)	0.2962	0.6849	0.6656	10.147	15

Dihedral angles between planes [b]

Planes	Angle (°)	Planes	Angle (°)
1–2	24	7–8	27
1–3	109	7–9	150
1–4	69	7–10	50
1–5	62	7–11	108
5–6	3	11–12	19

[a] Equation of plane is in the form $lX_o + mY_o + nZ_o = d$, where X_o, Y_o, Z_o are orthogonal coordinates in Å referred to axes x_o, y_o, z_o respectively, with x_o parallel to a^*, y_o to b and z_o to c. [b] Standard deviation approximately 1°.

(regular hexagons of bonds 1.395 Å long) [25] with individual isotropic temperature factors for the C atoms. The remaining non-hydrogen atoms in the asymmetric unit were subjected to anisotropic refinement. The H atoms of the methylene and quinolyl groups were generated geometrically (d(C–H) fixed at 0.96 Å), allowed to ride on their respective parent C atoms, and included in structure factor calculations with assigned isotropic thermal parameters. The H atoms of the water molecule could not be located in the final difference map. All computations were performed on a Data General Nova 3/12 minicomputer with the SHELXTL system [26]. Analytic expressions of neutral-atom scattering factors incorporating the real and imaginary components of anomalous dispersion were employed [27]. Blocked-cascade least-squares refinement [28] converged to the R indices and other parameters listed in Table 1. The final atomic parameters are listed in Table 2. Bond distances and angles are listed in Tables 3 and 4, respectively, and least-squares planes data in Table 5. The geometry about the coordinated water molecules is illustrated in Fig. 3, and a stereoview of the molecular packing is given in Fig. 4. Tables of hydrogen

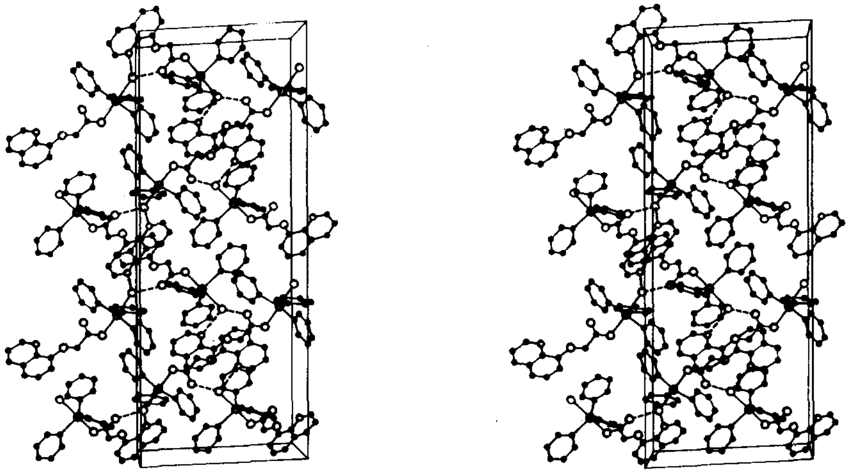

Fig. 4. Stereoview of the molecular packing. The origin of the unit cell lies at the upper left corner, with *b* pointing towards the reader, *a* from left to right, and *c* downwards. Hydrogen bonds are represented by broken lines.

coordinates, anisotropic thermal parameters, and structure factors are available from the authors on request.

Results and discussion

The title compound contains two crystallographically-independent [$Ph_3SnO_2CCH_2(C_9H_6NO) \cdot H_2O$] molecular units, **A** (Fig. 1) and **B** (Fig. 2), having the same basic trigonal bipyramidal geometry at tin, each with a unidentate carboxylate group and a coordinated water molecule at the apical positions, and phenyl groups located in the trigonal girdle. The sum of the angles subtended at tin by the *ipso*-carbons of the phenyl rings is 359.1(9) for **A** and 359.3(7)° for **B**; the corresponding axial (O_{acyl}–Sn–O_w) angles are 175.9(3) and 176.6(3)°. The Sn–O_w bonds (2.391(6)^A, 2.388(7)^B Å) are significantly longer than the Sn–O_{acyl} bonds (2.162(7)^A, 2.174(6)^B Å), but are comparable to those in other organotin inner-sphere coordination hydrates (Table 6). There is no evidence for intermolecular carboxylate bridging in either **A** or **B**. Although such bridging is general for organotin carboxylates [29–31], the absence of this feature in the title compound may be the result of the steric bulk of the quinolinoyl-substituted acetate moiety. Another possible reason, as outlined in the Introduction, is the relative preference of the acyl unit to engage in hydrogen-bonding interactions whenever possible, and this is, indeed, observed in the title compound (vide infra).

The tin–acyl bonds (Sn–O^I) in the title compound (2.162(7)^A, 2.174(6)^B Å) are in good agreement with published data (Table 7), and lie between the extremes of 2.043 Å in $Ph_3SnO_2CC_6H_4$-2-NH_2 [32] and 2.350 Å in Ph_3ClSnO_2C-2-quinH · H_2O [11]. However, a comparison of the second intramolecular acyl–oxygen to tin distances (Sn ··· O^{II}) in **A** and **B** reveals that a closer location of O^{II} to the metal center exists in **B** (3.201(9) vs. 3.525(9) Å in **A**). The values are well within the sum of the Van der Waals radii (3.70 Å), and the value for **B** is close to that observed for the pseudo-octahedral Ph_3SnOAc molecule (3.206 Å) [13]. However, the interpretation of the $Sn \cdots O^{II}$ distance as a weak bonding interaction in Ph_3SnOAc is supported by two additional pieces of evidence, viz., the small difference between the lengths of the two C–O bonds of the carboxylate group compared to that in other organotin carboxylates (Table 7), and the distortion of the equatorial bond angle in the direction of O^{II} approach to tin (135.2 vs. 111.2 and 113.0° for the other angles). In molecule **B**, on the other hand, the double-bond character remains localized in the $C–O^{II}$ bond, as reflected in the dissimilar C–O bond lengths, and the observed distortion of the equatorial C(46)–Sn–C(52) angle relative to the other equatorial angles is less dramatic (126.4(3) vs. 117.1(2) and 115.8(2)°). On the basis of the otherwise near-identical molecular dimensions of **A** and **B** at the metal centers, and the ^{119m}Sn Mössbauer spectral homogeneity of the compound, an essentially five-coordinated environment at tin is thus envisaged for both cases. This is further corroborated by the infrared data of the compound in the carboxyl region (ν(C–O): 1658, 1629 (*asym*); 1429, 1410 (*sym*) cm^{-1}), where the difference between the asymmetric and symmetric stretching frequencies exceeds 200 cm^{-1}, effectively discounting any significant chelating or bridging interactions [33,34]. We consider the apparent difference in the $Sn \cdots O^{II}$ bond lengths in **A** and **B** to be an artefact of the packing arrangement engendered by hydrogen bonding by the water molecules, which accentuate the dihedral angle differences (Table 5) between the Ph_3Sn trigonal girdle and the acyl $O^ICO^{II}C$ planes in the two cases (62° for **A** vs. 108° for **B**).

Consideration of the geometrical environments of the coordinated water molecules (Fig. 3 and Table 3) shows that each forms donor hydrogen bonds with the O^{II} and N atoms of a neighboring molecular unit, generating a helical chain extending in the direction of the a axis (Fig. 4). Each water molecule is three-coordinated, with pyramidal and virtually planar bonding configurations about $O_w(1)$ and $O_w(2)$, respectively (Table 4). The relatively short $O_w \cdots O^{III}$ contacts (3.01 and 3.05 Å) are not considered hydrogen bonds, and in this regard they differ from the water–ether oxygen interactions of 2.92–3.02 Å found in a low-temperature diffraction study of $\{[Me_2SnCl_2 \cdot H_2O]_2 \cdot 18\text{-crown-}6\}_n$ [35]. The intermolecular $O_w \cdots O$ hydrogen bonds in the title compound are comparable to those found in $Me_3SnO_2C\text{-}2\text{-py} \cdot H_2O$ ($2.71_{av.}$ Å) [1], $Me_3SnO_3SPh \cdot H_2O$ (2.54 Å) [3] and $Me_3SnNO_3 \cdot H_2O$ (2.72 Å) [4]. The $O_w \cdots N$ hydrogen bonds also appear to be at the lower end of the scale of similar interactions in other triorganotin hydrate compounds (Table 7). As indicated in Fig. 3, the shorter $Sn \cdots O^{II}$ contact in **B** (3.201(9) Å) is consistent with the concomitant smaller $Sn–O^I–C–O^{II}$ torsion angle (−15(1)°) relative to the respective dimensions (3.525(9) Å and −35(1)°) for molecule **A**.

The crystal lattice is made up by packing of parallel helical chains (Fig. 4). Our attempt to disrupt this by recrystallizing the title compound from DMSO led to regeneration of the starting material. A rigid polymeric structure is, however, considered unlikely, and this view is supported by the variable-temperature Mössbauer data recorded in the temperature range $80 \leq T \leq 110$ K.

In the 'thin absorber' approximation, the recoil-free fraction $f(T)$ of the ^{119}Sn absorber is given by

$$df/dT = d\exp\left(-6E_R/k\theta_D{}^2\right)/dT$$

The experimentally accessible area under the Mössbauer resonance curve, A_T, is a

(*Continued on p. 46*)

TABLE 6

STRUCTURAL DATA FOR HYDROGEN-BONDED ORGANOTIN HYDRATE COMPOUNDS

Organotin hydrate compounds	$d[\mathrm{Sn{-}O_w}]$ (Å)	$d[\mathrm{O_w \cdots N}]$ (Å)	$d[\mathrm{O_w \cdots O}]$ (Å)	$d[\mathrm{O_w \cdots X}]$	Ref.
$EtCl_2SnOH \cdot H_2O$	2.347		2.863, 3.041	3.307 (X = Cl)	[a]
$Me_2Sn[ONHC(O)Me]_2 \cdot H_2O$	[b]	2.78, 2.85	2.75, 2.89		[c]
$\{[Me_2SnCl_2 \cdot H_2O]_2 \cdot 18\text{-crown-}6\}_n$	2.313, 2.360		2.881–3.023		35
$[Me_2Sn(NCS)_2 \cdot 2H_2O \cdot 18\text{-crown-}6]_n$	2.278		2.80, 3.03		[d]
$\{[Me_2SnCl_4]^{2-} \cdot 2[H_3NC_6H_4\text{-}2\text{-}C(O)NH_2]^+ \cdot 2H_2O\}_n$	[b]	2.721			[e]
$Me_2SnCl_2 \cdot 2H_2O \cdot 4(\text{purine})$	2.135	2.722, 2762, 2.786, 2.805			[f]
$Me_3SnO_2C\text{-}2\text{-}py \cdot H_2O$	2.44	2.88	2.79		1
	2.43	2.80	2.74		
	2.41	2.74	2.66		
	2.44	2.72	2.65		
$Me_3SnNO_3 \cdot H_2O$	2.47		2.72, 2.77, 2.92		4
$Me_3SnO_3SPh \cdot H_2O$	2.30		2.54		3
$[n\text{-}Bu_3Sn(H_2O)_2]^+ [C_5(CO_2Me)_5]^-$	2.295, 2.326				2
$Ph_3SnCl \cdot terpy \cdot H_2O$	2.317	2.766, 2.807, 2.949			7,8
$Ph_3(NCS)Sn \cdot terpy \cdot H_2O$	2.252	2.756, 2.798			9
$Ph_3(NCS)SnO_2C\text{-}2\text{-}pyH \cdot H_2O$	[b]	2.667	2.801, 3.246		10
$Ph_3SnCl \cdot phen \cdot H_2O$	2.42	2.96–3.31			5
$Ph_3SnCl \cdot 3\text{-}[2\text{-}(1\text{-}10\text{-phenanthrolyl})]\text{-}5,6\text{-diphenyl-}1,2,4\text{-triazine} \cdot H_2O$	2.359	2.83, 2.85, 3.04			6
$Ph_3ClSnO_2C\text{-}2\text{-}quinH \cdot H_2O$	[b]	Not given	Not given		11
$[Ph_3SnO_2CCH_2(8\text{-}C_9H_6NO) \cdot H_2O]_n$	2.388(6)	2.79(1)	2.68(1), 3.01(1)		This work
	2.391(6)	2.79(1)	2.81(10, 3.05(1)		

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TABLE 7

STRUCTURAL DATA FOR ORGANOTIN CARBOXYLATES

Organotin carboxylate	d[Sn–O^{I}] (Å)	d[O^{I}–C] (Å)	d[C–O^{II}] (Å)	d[Sn ··· O^{II}] (Å)	Sn–O^{I}–C (°)	O^{I}–C–O^{II} (°)	Geometry at Sn	Ref.
Me_2PhSnO_2CMe	2.202	1.252	1.256		121.4	122.5	tbp	*a*
Me_3SnO_2CMe	2.205	1.269	1.240		122.5	122.6	tbp	*b*
$Me_3SnO_2CCF_3$	2.177	1.28	1.21		121	129	tbp	*b*
$[Me_3SnO_2CCH_2NH_2]_2$	2.21	1.35	1.23	3.23	117.5	126.1	tbp	*c*
$Me_3SnO_2CCH_2CO_2SnMe_3$	2.17	1.28	1.28		121		tbp	*d*
	2.19	1.27	1.22		121		tbp	
Me_3SnO_2C-2-py$\cdot H_2O$	2.21	1.29	1.18		118.6	126.7	tbp	1
	2.18	1.29	1.25		125.2	120.6	tbp	
	2.15	1.28	1.25		118.3	125.8	tbp	
	2.20	1.30	1.23		118.3	128.5	tbp	
Me_2ClSnO_2CMe	2.165	1.260	1.262	2.782	108.4	121.3	tbp	*e*
$[Me_2Sn(O_2CCH_2Cl)]_2$	2.241	1.27	1.25		123	126	tbp	*f*
$[Me_2Sn(O_2CCF_3)]_2$	2.319	1.218	1.251		121.4	129.9	tbp	*g*
$\{[Me_2Sn(O_2CCF_3)]_2O\}_2$	2.367	1.212	1.213		133.3	130.1	tbp	*h*
	2.253	1.263	1.190	3.164	115.8	127.7	tbp	
$[(CH_2{=}CH)_4Sn_2(O_2CCF_3)]_2O$	2.25			3.12			*trans*-O$_h$	*i*
	2.31							
$[CH_2{=}CH]_2Sn(O_2CCF_3)_2\cdot$bipy	2.178	1.319	1.172	3.471		126.3	*cis*-O$_h$	*j*
	2.250	1.215	1.172	3.008		127.5		
$\{[n\text{-}Bu_2Sn(O_2CCCl_3)]_2O\}_2$	2.22	1.26	1.20		112	129	tbp	*k*
$(CH_2{=}CH)_3Sn(O_2CCCl_3)$	2.17	1.25	1.21		122.8	128.2	tbp	*l*
$(CH_2{=}CH)_3SnO_2C(C_5H_4FeC_5H_5)$	2.12	1.27	1.21		123	124	tbp	*m*
Bz_3SnO_2CMe	2.14	1.31	1.21	3.25			tbp	*n*
Cyh_3SnO_2CMe	2.12	1.39	1.25	2.95			T_d	*o*

$Cyh_3SnO_2CCF_3$	2.08	1.28	1.20	3.11	122	122	T_d/tbp	[p]
Cyh_3Sn-indole-3-acetate	2.086	1.302	1.213	2.929	112.8	123.3	T_d	[q]
Ph_3SnO_2CMe	2.185	1.263	1.251	3.206	121.0	122.9	*mer*-O_h	13
$Ph_3SnO_2CC_6H_4$-2-(N_2-C_6H_3-2-OH-5-Me)	2.070	1.296	1.224	2.463	105.5	120.4	*cis*-tbp	[r]
$Ph_3SnO_2CC_6H_4$-2-NH_2	2.043	1.310	1.237	2.823	112.3	119.8	T_d/tbp	[s]
$Ph_3SnO_2CC_6H_4$-2-NMe_2	2.115	1.272	1.22	2.564	102.2	120.2	T_d/tbp	[s]
$Ph_3SnO_2CC_6H_4$-4-$NH_2 \cdot 0.5C_6H_6$	2.072	1.306	1.236	2.629	105.7	118.9	T_d/tbp	[s]
$Ph_3SnO_2CC_6H_4$-2-OH	2.083	1.301	1.232	3.071	118.8	122.4	T_d/tbp	32
$Ph_3SnO_2CC_6H_4$-2-OMe	2.054	1.321	1.214	2.781	110.1	120.3	T_d/tbp	32
$Ph_3SnO_2CC_6H_4$-4-SMe	2.060	1.310	1.232	2.783	109.7	120.7	T_d/tbp	32
Ph_3ClSnO_2C-2-pyH	2.347	1.256	1.232		135.5	127.4	tbp	12
Ph_3ClSnO_2C-2-quinH$\cdot H_2O$	2.350	1.267	1.222		116.5	129.9	tbp	11
$Ph_3(NCS)SnO_2C$-2-pyH$\cdot H_2O$	2.221	1.229	1.237		120.8	130.2	tbp	10
$[Ph_2Sn(O_2CMe)]_2$	2.25	1.31	1.20		128.1	125.6	tbp	[t]
$[Ph_3SnO_2CCH_2(8$-$C_4H_6NO)\cdot$	2.162(7)	1.298(12)	1.221(13)	3.53(1)	128.0(7)	127.0(10)	tbp	This
$H_2O]_n$	2.174(6)	1.294(11)	1.219(12)	3.20(1)	119.7(6)	124.2(9)	tbp	work

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dimensionless value obtained by dividing the area under the curve by the baseline (both parameters measured in channel × count units). The temperature-dependent, time-independent parameter, a, is the slope of the equation

$$\mathrm{d}[\ln(A_T/A_{80\,\mathrm{K}})] = a\,\mathrm{d}T + \text{constant}$$

The plot is linear and the value of a, which reflects the mean-square amplitude vibration of the tin atom in the crystal lattice, is a measure of how tightly-bound the tin atom is in the crystal lattice and hence, the degree of molecular association. Normalization to 80 K facilitates the calculations.

A survey of a/structure systematics for methyl, phenyl and cyclohexyltin derivatives reveals that in general, rigid polymeric lattices gives rise to $-a$ values of $< 1.1 \times 10^{-2}$, while polymers possessing more flexible tertiary structures that permit greater vibrational freedom for the tin give rise to a values approaching those of non-associated lattices, i.e. in the range $(1.3\text{–}2.8) \times 10^{-2}$ K^{-1} [36,37]. The a value of -1.99×10^{-2} K^{-1} for the title compound suggests a monomeric or weakly polymeric structure. Direct comparison of this result can be made with the value for Ph_3SnOAc (-1.91×10^{-2} K^{-1}), which incorrectly predicts a monomeric formulation, but this has been accounted for in terms of the fact that the axis of propagation of the Ph_3SnOAc polymer lies at about a 60° angle to the general alignment of the OSnO unit, with the result that the effective bridging mass is concentrated away from the line joining any two adjacent tin atoms [13].

Acknowledgements

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