**WATER-COORDINATED TRIORGANOTIN CARBOXYLATES.
CRYSTAL STRUCTURES OF BIS(TRIPHENYLTIN 3-OXA-
PENTAMETHYLENETHIOCARBAMOYL-S-ACETATE) HYDRATE
AND TRICYCLOHEXYLTIN 3-OXAPENTAMETHYLENETHIO-
CARBAMOYL-S-ACETATE HYDRATE**

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Abstract

The crystal structure of bis(triphenyltin 3-oxapentamethylenethiocarbamoyl-S-acetate) hydrate consists of a $(\text{C}_6\text{H}_5)_3\text{SnO}_2\text{CCH}_2\text{SC}(\text{O})\text{N}(\text{CH}_2\text{CH}_2)_2\text{O H}_2\text{O}$ ($\text{Sn-O} = 2.177(4)$, $\text{Sn-O}_{\text{water}} = 2.298(4)$ Å) molecule that uses its carbonyl oxygen atom to bridge to a $(\text{C}_6\text{H}_5)_3\text{SnO}_2\text{CCH}_2\text{SC}(\text{O})\text{N}(\text{CH}_2\text{CH}_2)_2\text{O}$ ($\text{Sn-O} = 2.158(4)$, $\text{Sn-O}_{\text{bridge}} = 2.381(4)$ Å) molecule. The coordinated water forms hydrogen bonds to the carbonyl and carbamoyl ($\text{O}\cdots\text{O} = 2.615(7)$, $2.685(8)$ Å) oxygen atoms of adjacent $(\text{C}_6\text{H}_5)_3\text{SnO}_2\text{CCH}_2\text{SC}(\text{O})\text{N}(\text{CH}_2\text{CH}_2)_2\text{O}$ molecules, the hydrogen bonds giving rise to a layer structure. In tricyclohexyltin 3-oxapentamethylenethiocarbamoyl-S-acetate hydrate, the coordinated water ($\text{Sn-O} = 2.194(2)$, $\text{Sn-O}_{\text{water}} = 2.421(2)$ Å) molecule is hydrogen bonded to the carbonyl and carbamoyl oxygen atoms of only one neighboring molecule, the hydrogen bonds ($\text{O}\cdots\text{O} = 2.683(4)$, $2.785(4)$ Å) linking the molecules into a linear helical chain.

Introduction

Triorganotin carboxylates often crystallize with coordinated water when the carboxylato anions possess either basic donor sites that form strong hydrogen bonds with the water molecule (as with the quinolyl-8-oxyacetato [1] and *N*-phthaloylglycinato [2] anions) or electron-withdrawing substituents that enhance the Lewis acceptor capacity of the triorganotin cation (as with the trifluoroacetato [3] and bis(*N,N*-dimethyldithiocarbamoyl)acetato [4] anions). For the bis(*N,N*-dimethyldithiocarbamoyl)acetato anion in particular, the fulfillment of both conditions for the triphenyltin derivative permits its crystallization with a second molecule of water, which is located in the secondary coordination sphere of the metal atom [4]. Triphenyltin *N,N*-dimethyldithiocarbamoylacetate exhibits no Lewis acidity toward water, however, as the compound exists as a carboxylate-bridged chain [5]. In view of the dipolar nature of the thiocarbamoyl unit that can hydrogen bond with water, the related triorganotin derivatives of 3-oxapentamethylenethiocarbamoyl-S-acetic acid are anticipated to form hydrates. The crystal structures of the condensation products of this carboxylic acid with triphenyltin hydroxide and with tricyclohexyltin hydroxide are described here.

Experimental

3-Oxapentamethylenethiocarbamoyl-S-acetic acid was synthesized from morpholine, carbonyl sulfide and chloroacetic acid [6]. Equimolar quantities of the acid and triphenyltin hydroxide were heated in a small volume of ethanol; slow cooling of the filtered solution gave

crystals of the hydrated dinuclear compound. The use of tricyclohexyltin hydroxide in place of triphenyltin hydroxide afforded the analogous hydrated carboxylate.

A specimen of the triphenyltin derivative, 0.50 x 0.30 x 0.15 mm, was used for the room-temperature measurements on an Enraf-Nonius CAD4 diffractometer (Mo- $K\alpha$ radiation, 0.71073 Å). Accurate cell dimensions were calculated from the 25 strongest reflections in the $12.0^\circ \leq \theta \leq 12.5^\circ$ shell. The 9398 reflections were measured by ω - 2θ scans [7] up to $2\theta = 50^\circ$ (index ranges: $0 < h \leq 15$, $0 < k < 24$, $-24 \leq l < 23$), and the data were corrected for decay (7.6%) and absorption [8] (transmission factors = 0.756 - 1.000). The raw data were reduced by the *NRCVAX* program [9] for solution by *SHELXS-86* [10]. The $O(CH_2CH_2)_2NC(O)S$ - groups of molecules *a* and *b* are disordered, the disorder in molecule *b* being less severe. The disordered group of molecule *a* when refined as two groups of half site occupancies gave unsatisfactory results, and had to be refined as one group but with $N1-C21 = 1.35 \pm 0.01$ and $C24-C25 = 1.54 \pm 0.01$ Å. Peaks ranging from 1.668 to $-1.122 \text{ e}\text{\AA}^{-3}$ were found in the final difference map, with peaks exceeding $1 \text{ e}\text{\AA}^{-3}$ being found near the $S(1a)$ atom. The disorder also affected the other parts of the *a* and *b* molecules, and all phenyl rings had to be refined as rigid hexagons. H-atoms were generated geometrically, with $U(H) = 1.5U_{eq}(C)$; the hydroxylic H-atoms were also generated. Refinement on F^2 [11] for the 8964 independent reflections ($R_{int} = 0.0329$), 2 restraints and 514 parameters converged to a residual index of 0.0812 for all reflections. The R_1 index for the 6373 $I \geq 2\sigma(I)$ reflections was 0.0570; $S = 1.040$ and $wR_2 = 0.1449$ for $w = [\sigma^2(F_o^2) + (0.0985P)^2]^{-1}$. Atomic coordinates are listed in Table 1. The dinuclear molecule is shown as a *PLUTON* [12] plot in Fig. 1.

Crystal data: $C_{50}H_{52}N_2O_9S_2Sn_2$, $FW = 1126.44$, monoclinic, $P2_1/n$, $a = 12.641(4)$, $b = 20.465(2)$, $c = 20.472(6)$ Å, $\beta = 105.67(1)^\circ$, $V = 5099(2)$ Å³, $D_{calc} = 1.467 \text{ Mg m}^{-3}$, $\mu = 1.116 \text{ mm}^{-1}$, $F(000) = 2280$ for $Z = 4$.

Table 1. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for bis(triphenyltin 3-oxapentamethylenethiocarbamoyl-*S*-acetate) hydrate

Atom	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}
Sn(1 <i>a</i>)	9246(1)	4068(1)	7501(1)	47(1)
S(1 <i>a</i>)	7972(3)	3835(1)	4926(1)	112(1)
O(1 <i>a</i>)	8653(4)	3801(2)	6434(2)	56(1)
O(2 <i>a</i>)	8321(4)	2754(2)	6564(2)	49(1)
O(3 <i>a</i>)	6298(10)	4199(5)	5156(5)	174(5)
O(4 <i>a</i>)	7005(12)	6175(4)	4013(6)	172(5)
O(w1 <i>a</i>)	9781(4)	4376(2)	8622(2)	63(1)
N(1 <i>a</i>)	6967(16)	4920(5)	4511(6)	182(7)
C(1 <i>a</i>)	7744(3)	3790(2)	7695(2)	55(2)
C(2 <i>a</i>)	7669(5)	3181(2)	7979(3)	78(2)
C(3 <i>a</i>)	6683(6)	2981(3)	8094(3)	99(3)
C(4 <i>a</i>)	5773(4)	3391(4)	7925(4)	128(5)
C(5 <i>a</i>)	5849(4)	4001(4)	7641(4)	128(5)
C(6 <i>a</i>)	6834(5)	4200(3)	7526(3)	92(3)

C(7a)	9601(4)	5025(2)	7173(3)	57(2)
C(8a)	9915(5)	5535(3)	7634(2)	82(2)
C(9a)	10202(5)	6137(2)	7417(4)	103(3)
C(10a)	10173(6)	6229(2)	6740(4)	110(4)
C(11a)	9858(6)	5719(3)	6279(3)	100(3)
C(12a)	9572(5)	5117(2)	6496(2)	78(2)
C(13a)	10626(3)	3424(2)	7718(2)	51(2)
C(14a)	10995(4)	3100(3)	8335(2)	66(2)
C(15a)	11929(4)	2711(3)	8460(3)	93(3)
C(16a)	12495(4)	2645(3)	7968(4)	105(4)
C(17a)	12127(4)	2969(3)	7351(3)	96(3)
C(18a)	11192(4)	3358(3)	7226(2)	67(2)
C(19a)	8260(5)	3260(3)	6217(3)	47(2)
C(20a)	7661(8)	3219(3)	5470(4)	75(2)
C(21a)	6891(8)	4352(6)	4879(6)	208(12)
C(22a)	6004(17)	5341(8)	4347(11)	231(10)
C(23a)	6261(18)	5977(7)	4355(11)	212(10)
C(24a)	8004(19)	5837(10)	4284(9)	220(10)
C(25a)	7799(17)	5098(8)	4222(9)	222(11)
Sn(1b)	8031(1)	1633(1)	6255(1)	42(1)
S(1b)	8188(2)	-542(1)	5065(1)	76(1)
O(1b)	7975(4)	644(2)	5883(2)	53(1)
O(2b)	6531(4)	309(2)	6206(3)	68(1)
O(3b)	6820(4)	324(3)	4360(3)	90(2)
O(4b)	8774(5)	-136(4)	2698(3)	102(2)
N(1b)	8162(5)	12(3)	3898(3)	76(2)
C(1b)	9174(3)	1822(2)	5672(2)	44(1)
C(2b)	8920(3)	1620(2)	4999(2)	56(2)
C(3b)	9610(4)	1782(3)	4598(2)	72(2)
C(4b)	10555(4)	2146(3)	4870(3)	85(3)
C(5b)	10809(3)	2348(3)	5543(3)	82(2)
C(6b)	10119(4)	2186(2)	5944(2)	66(2)
C(7b)	8536(4)	1449(2)	7317(2)	50(2)
C(8b)	7814(4)	1119(3)	7607(2)	92(3)
C(9b)	8105(5)	998(3)	8301(2)	118(4)
C(10b)	9118(5)	1206(3)	8705(2)	99(3)
C(11b)	9840(4)	1535(3)	8415(2)	96(3)
C(12b)	9549(4)	1657(3)	7721(2)	78(2)
C(13b)	6341(3)	1861(2)	5849(2)	49(2)
C(14b)	5767(4)	1655(2)	5204(2)	58(2)
C(15b)	4661(4)	1809(3)	4951(2)	85(3)
C(16b)	4129(3)	2170(3)	5342(3)	93(3)
C(17b)	4703(4)	2377(3)	5988(3)	91(3)
C(18b)	5809(4)	2222(2)	6241(2)	65(2)
C(19b)	7226(6)	226(3)	5900(3)	50(2)
C(20b)	7229(8)	-417(4)	5546(4)	79(2)
C(21b)	7635(6)	-15(4)	4379(4)	64(2)
C(22b)	9094(8)	-386(6)	3882(5)	106(4)
C(23b)	9000(12)	-632(7)	3216(6)	153(6)
C(24b)	7799(9)	180(7)	2690(5)	125(5)
C(25b)	7835(11)	471(6)	3343(5)	132(5)

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

An irregularly shaped specimen of the tricyclohexyltin derivative, 0.58 x 0.58 x 0.58 mm, was used for the measurements. The unit-cell dimensions were refined from the 25 most intense reflections in the $14.5^\circ < \theta \leq 15.0^\circ$ range. The 8720 reflections which were collected by ω - θ scans up to $2\theta = 60^\circ$ ($-15 \leq h < 0$, $0 < k < 20$, $-24 \leq l \leq 24$) were corrected for absorption (transmission factors = 0.930 - 1.000). The morpholinyl ring and one of the three cyclohexyl rings are disordered over two positions. For the disordered cyclohexyl ring, distance constraints (C-C = 1.54 ± 0.01 Å) were imposed in the C(15), C(16), C(17) and C(15a), C(16a), C(17a) segments. The water H-atoms were located and refined. Refinement on all 8067 independent ($R_{\text{int}} = 0.0217$) data converged to a final R_1 of 0.0470 for all data; $R_1 = 0.0357$ for the 6730 $I \geq 2\sigma(I)$ data; $R_w = 0.0774$ and $S = 1.018$ for 2 restraints and 393 parameters; $w = [\sigma^2(F_o^2) + (0.0462P)^2]^{-1}$. Peaks of 0.700 and -0.291 eÅ^{-3} were found in the final difference map. The absolute structure [13] parameter was $-0.03(2)$.

Crystal data: $\text{C}_{25}\text{H}_{45}\text{NO}_5\text{SSn}$, $FW = 590.37$, orthorhombic, $P2_12_12_1$, $a = 11.2243(6)$, $b = 14.2432(7)$, $c = 17.327(1)$ Å, $V = 2770.0(3)$ Å³, $D_{\text{calc}} = 1.416 \text{ g cm}^{-3}$, $\mu = 1.031 \text{ mm}^{-1}$, $F(000) = 1232$ for $Z = 4$.

Table 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for tricyclohexyltin 3-oxapentamethylenethiocarbamoyl-S-acetate hydrate

Atom	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}
Sn(1)	5935(1)	6230(1)	7189(1)	40(1)
S(1)	5411(1)	3276(1)	5072(1)	59(1)
O(w1)	6461(2)	7432(2)	8117(2)	54(1)
O(1)	5511(2)	5274(2)	6235(1)	56(1)
O(2)	4754(3)	4059(2)	6866(2)	74(1)
O(3)	3388(2)	2354(2)	5282(1)	55(1)
O(4) (0.5)	5326(20)	80(11)	3685(8)	84(4)
O(4a) (0.5)	5238(20)	309(11)	3398(7)	87(4)
N(1)	4666(3)	1696(3)	4454(2)	79(1)
C(1)	6980(3)	5234(2)	7843(2)	48(1)
C(2)	8222(3)	5579(3)	8058(2)	61(1)
C(3)	8925(4)	4805(3)	8476(3)	75(1)
C(4)	8282(4)	4442(3)	9173(3)	70(1)
C(5)	7058(4)	4108(3)	8964(3)	64(1)
C(6)	6335(3)	4864(2)	8546(2)	55(1)
C(7)	4111(3)	6578(3)	7489(2)	52(1)
C(8)	3732(3)	6181(4)	8258(2)	71(1)
C(9)	2491(4)	6503(4)	8495(3)	86(2)
C(10)	1590(4)	6325(4)	7873(3)	88(1)
C(11)	1955(3)	6715(4)	7117(3)	81(1)
C(12)	3193(3)	6391(3)	6879(2)	66(1)
C(13)	6797(3)	7082(3)	6319(2)	51(1)
C(14) (0.5)	5875(18)	7563(11)	5806(8)	61(3)
C(15) (0.5)	6477(19)	8105(12)	5123(8)	79(4)
C(16) (0.5)	7390(28)	8719(15)	5420(11)	130(10)
C(17) (0.5)	8214(19)	8233(25)	5811(21)	213(16)

C(18) (0.5)	7741(17)	7787(9)	6550(11)	90(6)
C(14a) (0.5)	6050(18)	7853(12)	6077(11)	110(7)
C(15a) (0.5)	6453(20)	8365(18)	5418(11)	123(8)
C(16a) (0.5)	7709(18)	8751(13)	5582(14)	173(20)
C(17a) (0.5)	8604(13)	8023(12)	5883(8)	85(4)
C(18a) (0.5)	8025(14)	7387(8)	6546(11)	72(3)
C(19)	4956(3)	4505(2)	6273(2)	48(1)
C(20)	4480(3)	4157(2)	5504(2)	51(1)
C(21)	4346(3)	2364(2)	4947(2)	48(1)
C(22) (0.5)	5600(10)	1746(9)	3866(7)	63(3)
C(23) (0.5)	5946(18)	1115(13)	3491(10)	148(9)
C(24) (0.5)	4126(16)	404(13)	3757(9)	148(9)
C(25) (0.5)	3774(12)	1004(9)	4129(6)	60(3)
C(22a) (0.5)	5948(12)	1558(13)	4201(10)	141(9)
C(23a) (0.5)	6169(10)	795(8)	3897(8)	75(3)
C(24a) (0.5)	4396(13)	89(7)	4207(7)	74(3)
C(25a) (0.5)	3974(15)	807(10)	4456(7)	97(6)

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

Results and discussion

Triphenyltin hydroxide condenses with 3-oxapentamethylenethiocarbamoyl-*S*-acetic acid in ethanol to yield the ester as a hemihydrate; formally, the compound is the 1/1 complex of $(C_6H_5)_3SnO_2CCH_2SC(O)N(CH_2CH_2)_2O \cdot H_2O$ (molecule *a*) with $(C_6H_5)_3SnO_2CCH_2SC(O)N(CH_2CH_2)_2O$ (molecule *b*). Tricyclohexyltin hydroxide reacts with the acid to give monohydrate. These hydrated products contrast with the anhydrous carboxylates derived from the condensation with 3-oxapentamethylenedithiocarbamoylacetic acid [14].

In bis(triphenyltin 3-oxapentamethylenethiocarbamoyl-*S*-acetate) hydrate, molecule *a* uses its carbonyl oxygen atom to bridge to molecule *b* ($Sn(1a)-O(1a) = 2.177(4)$, $Sn(1a)-O(w1a) = 2.298(4)$; $Sn(1b)-O(1b) = 2.158(4)$, $Sn(1b)-O(2a) = 2.381(4)$ Å). The compound constitutes the first example of a triorganotin carboxylate having half a water ligand. One hydrogen bond connects the dinuclear chain to the carbonyl oxygen ($O \cdots O = 2.615(7)$ Å) atom of molecule *b* of an adjacent chain; a second hydrogen bond links it to the carbamoyl oxygen ($O \cdots O = 2.685(8)$ Å) atom of another neighboring chain (symmetry transformation: $0.5 + x, 0.5 - y, 0.5 + z$). The hydrogen bonds link the dinuclear chains into a tightly-held layer structure. The hydrogen bonding distances are comparable with that ($O \cdots O = 2.641(6)$ Å) found in *N,N*-dimethyldithiocarbamoyl-*S*-acetic acid [15]. The tin-water distance is shorter than those ($2.388(7)$, $2.391(6)$ Å) found in triphenyltin quinolyl-8-oxyacetate hydrate [1] and that ($2.388(3)$ Å) found in triphenyltin bis (*N,N*-dimethyldithiocarbamoyl)acetate dihydrate [3]. On the other hand, the carboxylate bridging distance is similar to those [16] observed for five-coordinate carboxylate-bridged triorganotin systems.

Fig. 1. Geometries of the tin atoms in bis(triphenyltin 3-oxapentamethylenethiocarbamoyl-S-acetate) hydrate. Selected bond distances and angles: Sn(1a)-C(1a) = 2.121(4), Sn(1a)-C(7a) = 2.155(4), Sn(1a)-C(13a) = 2.136(3), Sn(1a)-O(1a) = 2.177(4), Sn(1a)-O(w1a) = 2.298(4), Sn(1b)-C(1b) = 2.143(3), Sn(1b)-C(7b) = 2.129(3), Sn(1b)-C(13b) = 2.124(3), Sn(1b)-O(1b) = 2.158(4), Sn(1b)-O(2a) = 2.381(4) Å; C(1a)-Sn(1a)-C(7a) = 125.0(2), C(1a)-Sn(1a)-C(13a) = 121.1(2), C(1a)-Sn(1a)-O(1a) = 92.2(2), C(1a)-Sn(1a)-O(w1a) = 85.6(2), C(7a)-Sn(1a)-C(13a) = 113.5(2), C(7a)-Sn(1a)-O(1a) = 87.8(2), C(7a)-Sn(1a)-O(w1a) = 91.8(2), C(13a)-Sn(1a)-O(1a) = 95.4(2), C(13a)-Sn(1a)-O(w1a) = 87.6(2), O(1a)-Sn(1a)-O(w1a) = 176.9(2), C(1b)-Sn(1b)-C(7b) = 122.7(2), C(1b)-Sn(1b)-C(13b) = 120.1(2), C(1b)-Sn(1b)-O(1b) = 86.3(2), C(1b)-Sn(1b)-O(2a) = 84.1(2), C(7b)-Sn(1b)-C(13b) = 115.5(2), C(7b)-Sn(1b)-O(1b) = 99.5(2), C(7b)-Sn(1b)-O(2a) = 85.3(2), C(13b)-Sn(1b)-O(1b) = 97.7(2), C(13b)-Sn(1b)-O(2a) = 87.6(2), O(1b)-Sn(1b)-O(2a) = 170.3(2)°.

Tricyclohexyltin 3-oxapentamethylenethiocarbamoyl-S-acetate crystallizes as a monohydrate (Sn(1)-O(1) = 2.194(2), Sn(1)-O(w1) = 2.421(2) Å) whose coordinated water interacts with the carbonyl and carbamoyl oxygen atoms of only one adjacent molecule. The tin atom shows trigonal bipyramidal coordination ($\Sigma\text{C-Sn-C} = 359.2(3)^\circ$), and is an unusual example of a five-coordinate tricyclohexyltin carboxylate. Most tricyclohexyltin carboxylates adopt arrangements with the tin atom in a four-coordinate environment [17] as the three sterically bulky cyclohexyl rings prevent the formation of carboxyl-bridged [16] chains. For example, tricyclohexyltin acetate has a tetrahedral configuration, although its carbon-tin-carbon angles are considerably widened [18] from the idealized tetrahedral angle of 109.5° . On the other hand, the benzylsulfonylacetate is five-coordinate ($\Sigma\text{C-Sn-C} = 352.0(9)^\circ$) [18], but the geometry is severely distorted owing to the long bridging distance. In the 3-oxapentamethylenethiocarbamoyl-S-acetate, the hydrogen bonding interaction with the carbonyl donor ($\text{O}\cdots\text{O} = 2.683(4)$ Å) is much stronger than that with the carbamoyl donor ($\text{O}\cdots\text{O}$

= 2.785(4) Å) despite the dipolar form of the =C=N(O⁻)S- group that places a negative charge on the oxygen end. The hydrogen bonds, which are somewhat weaker than those found in the triphenyltin derivative, give rise to a linear helical chain structure.

Fig. 2. Geometry of the tin atom in tricyclohexyltin 3-oxapentamethylenethiocarbamoyl-S-acetate. Selected bond distances and angles: Sn(1)-C(1) = 2.162(3), Sn(1)-C(7) = 2.170(4), Sn(1)-C(13) = 2.163(3), Sn(1)-O(1) = 2.194(2), Sn(1)-O(w1) = 2.421(2) Å; C(1)-Sn(1)-C(7) = 122.4(1), C(1)-Sn(1)-C(13) = 119.4(1), C(1)-Sn(1)-O(1) = 96.1(1), C(1)-Sn(1)-O(w1) = 89.1(1), C(7)-Sn(1)-C(13) = 117.4(1), C(7)-Sn(1)-O(1) = 96.8(1), C(7)-Sn(1)-O(w1) = 84.8(1), C(13)-Sn(1)-O(1) = 85.4(1), C(13)-Sn(1)-O(w1) = 87.5(1), O(1)-Sn(1)-O(w1) = 172.7(1)°.

Acknowledgments

This work was supported by the University of Malaya (PJP 66/95).

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**Received: October 27, 1995 - Accepted: November 13, 1995 -
Received in revised camera-ready format: December 13, 1995**