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Preparation and characterization of triorganotin(IV) derivatives of mercaptocarboxylic acids. Crystal structure of [*O*,*S*-bis(triphenyltin(IV))-2-mercaptobenzoate]

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Abstract

The syntheses and tin-119m Mössbauer spectroscopic data are reported for the tri-n-butyl-, tricyclohexyl- and triphenyltin(IV) derivatives of mercaptoacetic and 2-mercaptobenzoic acids. A four-coordinate tetrahedral geometry at tin is assigned for (cyclo-$C_6H_{11})_3SnSCH_2C(O)OH$ and for the $(C_6H_5)_3SnSCH_2C(O)O^-$ and $(C_6H_5)_3SnSC_6H_4C(O)O^-$ anions, isolated as the dicyclohexylammonium and iso-*S*-benzylthiuronium salts, respectively. The sulfidotin in $R_3SnSCH_2C(O)OSnR_3$ and $R_3SnSC_6H_4C(O)OSnR_3$ is also four-coordinate, but the carboxylatotin is four-coordinate for $R = C_6H_5$ and five-coordinate for $R =$ n-C_4H_9. The Mössbauer quadrupole splitting in the butyltin case indicates a *trans*-C_3SnO_2 trigonal bipyramidal configuration. The coordination assignment for the two tin atoms in bis(triphenyltin)-2-mercaptobenzoate is confirmed by X-ray crystallography. The compound crystallizes in space group $P2_1/c$ with a 14.293(4), b 17.456(2), c 15.624(1) Å, β 107.40(1)°; $Z = 4$. The structure was refined to an R factor of 0.035 for 5837 observed reflections. The carbonyl oxygen of the molecule is positioned roughly halfway between the carboxylatotin (Sn ··· O 2.765(3) Å) and sulfidotin (Sn ··· S 3.016(1) Å), disorting somewhat the tetrahedral geometries at both tin sites.

Introduction

Mercaptocarboxylic acids are useful complexometric agents which form stable complexes with metal ions, and their esters with mono- and di-alkytins comprise an important class of thermal stabilizers for poly(vinyl chloride) [1]. When condensed

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with triorganotin oxides or hydroxides they yield products with 1/1 [2–4] and 2/1 [4–6] tin/ligand stoichiometries, as exemplified by the $[(n\text{-}C_4H_9)_3SnSCH_2C(O)O]^-$ Na^+ and $[(n\text{-}C_4H_9)_3SnSCH_2C(O)OSn(n\text{-}C_4H_9)_3]$ mercaptoacetates [4]. The two tin environments in the bis(trialkylstannyl) mercaptoalkanoates can be distinguished by tin-119m Mössbauer analysis which yields a spectrum consisting of two sets of quadrupole-split doublets. The outer doublet is characteristic of five-coordinate tin (the carboxylatotin), with the magnitude of the quadrupole splitting paralleling those obtained for most triorganotin alkanoates, which, on the basis of crystallographic evidence, are generally five-coordinate, carboxylate-bridged polymers [7]. The inner doublet has been assigned to the four-coordinate sulfidotin [4–6]. Spectroscopic and crystallographic studies on triphenyltin arylcarboxylates [8,9] and arylsulfides [10–12] have shown these compounds to be generally four-coordinate at tin. When the arylsulfido ligand carries a donor atom, higher-than-four coordination is possible, and is seen, for example, in triphenyltin 8-quinolinylsulfide [13], in which the chelating ligand spans the axial–equatorial positions of the *cis*-trigonal bipyramid, and in triphenyltin 4-pyridylsulfide [14], in which the ligand bridges the *trans*-trigonal bipyramid. There is, however, considerable distortion in the five-coordinate geometries of these two molecules. The compound triphenyltin 2-carboxymethylphenylsulfide, which has an organic ester group *ortho* to the sulfido linkage, has been studied by proton NMR spectroscopy but there is little evidence for any intramolecular tin–oxygen interaction in solution [15]. For the related mercaptoaminoacetates, which are tin–sulfur bonded, a zwitterionic formulation, $[R_3SnSC(R')_2CH(NH_3)^+(CO_2)^-]$, has been proposed [16].

A bis(triorganostannyl) mercaptocarboxylate can be viewed as a triorganotin sulfide carrying a triorganostannylcarboxylato substituent in the sulfido ligand. Although the sulfidotin has been assigned a tetrahedral geometry [4,5], coordination by the carboxyl oxygen, which would raise the coordination number to five, cannot be entirely discounted, because if the carboxyl oxygen is two or three carbon atoms distant from the sulfur end it might be able to coordinate to the sulfidotin to form a five- or six-membered ring. We report in this paper Mössbauer data for some organotin mercaptocarboxylates and the crystal structure of bis(triphenyltin)-2-mercaptobenzoate.

Experimental

Synthesis. The mercaptoacetic and mercaptobenzoic acids, bis(tri-n-butyltin) oxide (TBTO) and triphenyltin hydroxide were purchased from Tokyo Chemical Industries, Japan. Bis(tri-n-butyltin) mercaptoacetate and 2-mercaptobenzoate were obtained as analytically pure liquids by mixing together equimolar amounts of the respective mercaptocarboxylic acids and TBTO in the absence of solvent, and pumping the mixture under vacuum to remove the water formed in the reaction. (When the product was stored for a few months, solid material separated out from the supernatant bis(tri-n-butyltin) mercaptoacetate, and was identified by Mössbauer spectroscopy as di-n-butyltin mercaptoacetate). The bis(triphenyltin) analogues were obtained by reaction of triphenyltin hydroxide and the acids in ethanol or an ethanol/acetone mixture; the pure products separated out when the solvents were allowed to evaporate off slowly. In this way, single crystals of bis(triphenyltin)-2-mercaptobenzoate were obtained. However, diphenyltin-2-mercaptobenzoate (IS

Table 1

Analytical data for organotin(IV) mercaptocarboxylates

Compound	m.p. (°C)	Analysis (Found (calcd.) (%))	
		C	H
$(n\text{-}C_4H_9)_3SnSCH_2C(O)OSn(n\text{-}C_4H_9)_3$	liquid	46.09 (46.61)	8.01 (8.37)
$(C_6H_5)_3SnSCH_2C(O)OSn(C_6H_5)_3$	165–166	57.73 (57.77)	4.05 (4.11)
$(cyclo\text{-}C_6H_{11})_3SnSCH_2C(O)OH$	78–79	52.16 (52.32)	7.91 (7.85)
$[C_6H_5)_3SnSCH_2C(O)O][(cyclo\text{-}C_6H_{11})_2NH_2]$ [a]	177–178	61.77 (61.76)	6.54 (6.59)
$(n\text{-}C_4H_9)_3SnSC_6H_4C(O)OSn(n\text{-}C_4H_9)_3$	liquid	51.18 (51.18)	8.13 (7.93)
$(C_6H_5)_3SnSC_6H_4C(O)OSn(C_6H_5)_3$	138–139	60.31 (60.61)	4.00 (3.99)
$[(C_6H_5)_3SnSC_6H_4C(O)O][C_6H_5CH_2SC(NH_2)_2]$ [b]	131–132	58.97 (59.22)	4.58 (4.49)

[a] N, 2.25 (2.24)%. [b] N, 4.09 (4.19)%.

1.24, QS 3.29 mm s^{-1}) was unexpectedly obtained when triphenyltin hydroxide was treated with mercaptoacetic acid in toluene. Dicyclohexylammonium *S*-triphenylstannylmercaptoacetate separated out when equimolar amounts of mercaptoacetic acid, dicylohexylamine and triphenyltin hydroxide were mixed in ethanol. Conversion of 2-mercaptobenzoic acid to the iso-*S*-benzylthiuronium mercaptobenzoate salt by reaction with iso-*S*-benzylthiuronium chloride in water and subsequent reaction with triphenyltin hydroxide gave a precipitate of iso-*S*-benzylthiouronium *S*-triphenylstannylmercaptobenzoate in quantitative yield. Unfortunately, crystallization of neither salt yielded crystals suitable for X-ray analysis. Analytical data are presented in Table 1.

Table 2

^{119m}Sn Mössbauer data [a] for organotin mercaptocarboxylates at 80 K

Compound	IS [b]	QS [c]	T_1; T_2 [d]
$(n\text{-}C_4H_9)_3SnSCH_2C(O)OSn(n\text{-}C_4H_9)_3$ [e]	1.39	1.79	0.94; 1.00
	1.45	3.92	0.94; 0.92
$(C_6H_5)_3SnSCH_2C(O)OSn(C_6H_5)_3$	1.22	1.46	1.06; 0.99
	1.26	2.78	1.05; 1.99
$(cyclo\text{-}C_6H_{11})_3SnSCH_2C(O)OH$	1.49	1.94	1.05; 1.03
$[C_6H_5)_3SnSCH_2C(O)O][(cyclo\text{-}C_6H_{11})_2NH_2]$	1.23	1.25	0.98; 0.95
$(C_6H_5)_3SnSC_6H_4C(O)OSn(C_6H_5)_3$	1.22	1.38	1.06; 0.99
	1.20	2.56	0.92; 1.00
$(n\text{-}C_4H_9)_3SnSC_6H_4C(O)OSn(n\text{-}C_4H_9)_3$	1.43	2.04	1.05; 1.00
	1.46	3.94	1.04; 1.02
$[(C_6H_5)_3SnSC_6H_4C(O)O][C_6H_5CH_2SC(NH_2)_2]$	1.27	1.44	0.90; 0.96

[a] Error ±0.05 mm s^{-1}. [b] Isomer shift relative to $CaSnO_3$ at room temperature. [c] Quadrupole splitting. [d] Full width at half height of the resonant peaks, at larger and lesser velocity than the spectrum centroid respectively. [e] Lit. 1.40, QS 1.75; IS 1.40, QS 3.77 [4].

Table 3

Atomic coordinates ($\times 10^5$ for Sn and S; $\times 10^4$ for other atoms) and thermal parameters [a] (Å^2 $\times 10^4$ for Sn and S; $\times 10^3$ for other atoms)

Atom	x	y	z	U_{eq}
Sn(1)	2435(2)	53498(1)	33387(2)	494(1)
Sn(2)	34592(2)	46308(1)	20424(2)	491(1)
S	20192(8)	41928(6)	8946(8)	671(4)
O(1)	173(2)	5702(2)	2050(2)	59(1)
O(2)	1549(2)	5002(2)	2398(2)	60(1)
C(1)	1480(3)	5092(2)	487(2)	51(1)
C(2)	1518(3)	5323(3)	−353(3)	69(2)
C(3)	1064(3)	5970(3)	−750(3)	80(2)
C(4)	521(3)	6415(3)	−344(3)	71(2)
C(5)	472(3)	6209(2)	494(3)	59(1)
C(6)	963(3)	5551(2)	926(2)	48(1)
C(7)	917(3)	5386(2)	1852(2)	51(1)
C(8)	−116(3)	4168(2)	3221(2)	52(1)
C(9)	491(3)	3620(2)	3030(3)	73(2)
C(10)	162(4)	2862(3)	2866(3)	89(2)
C(11)	−752(4)	2664(3)	2878(3)	89(2)
C(12)	−1351(4)	3196(3)	3082(3)	81(2)
C(13)	−1027(3)	3948(2)	3264(3)	62(2)
C(14)	−990(3)	5997(2)	3418(3)	57(1)
C(15)	−1848(3)	5989(3)	2706(3)	79(2)
C(16)	−2665(4)	6376(4)	2757(4)	102(3)
C(17)	−2637(5)	6778(4)	3505(5)	117(3)
C(18)	−1812(5)	6798(3)	4219(4)	108(3)
C(19)	−979(3)	6407(3)	4175(3)	76(2)
C(20)	1524(3)	5736(2)	4321(2)	56(1)
C(21)	2204(3)	5203(3)	4819(3)	72(2)
C(22)	2999(4)	5451(3)	5470(3)	96(2)
C(23)	3168(4)	6209(3)	5643(3)	95(2)
C(24)	2521(4)	6741(3)	5161(3)	85(2)
C(25)	1691(3)	6506(3)	4499(3)	68(2)
C(26)	4608(3)	4339(2)	1464(2)	50(1)
C(27)	5569(3)	4546(2)	1889(3)	64(2)
C(28)	6306(3)	4356(3)	1522(3)	78(2)
C(29)	6093(4)	3970(3)	727(4)	87(2)
C(30)	5163(4)	3763(3)	312(3)	83(2)
C(31)	4417(3)	3947(2)	667(3)	68(2)
C(32)	3650(3)	5831(2)	2308(2)	50(1)
C(33)	3349(3)	6202(2)	2597(3)	58(1)
C(34)	3582(3)	6963(2)	3151(3)	70(2)
C(35)	4109(3)	7365(2)	2688(3)	70(2)
C(36)	4404(3)	7009(2)	2038(3)	75(2)
C(37)	4176(3)	6236(2)	1844(3)	64(2)
C(38)	3628(3)	3983(2)	3228(2)	50(1)
C(39)	3137(3)	3308(3)	3223(3)	75(2)
C(40)	3301(4)	2865(3)	4003(3)	93(2)
C(41)	3953(4)	3107(3)	4781(3)	93(2)
C(42)	4429(4)	3770(3)	4800(3)	100(2)
C(43)	4277(4)	4212(3)	4025(3)	86(2)

[a] The equivalent isotropic temperature factor U_{eq} is defined as 1/3 of the trace of the orthogonalized U matrix.

Table 4

Selected bond lengths (Å) and bond angles (°), with estimated standard deviations in parentheses

Bond lengths			
Sn(1)-O(1)	2.079(3)	Sn(1)-C(8)	2.121(4)
Sn(2)-C(14)	2.129(4)	Sn(1)-C(20)	2.116(3)
Sn(2)-S	2.414(1)	Sn(2)-C(26)	2.159(4)
Sn(2)-C(32)	2.136(3)	Sn(2)-C(38)	2.122(4)
S-C(1)	1.781(4)	O(1)-C(7)	1.315(5)
O(2)-C(7)	1.238(4)		
Bond angles			
O(1)-Sn(1)-C(8)	105.3(1)	O(1)-Sn(1)-C(14)	95.6(1)
O(1)-Sn(1)-C(20)	112.9(1)	C(8)-Sn(1)-C(14)	109.7(2)
C(8)-Sn(1)-C(20)	120.5(1)	C(14)-Sn(1)-C(20)	109.9(1)
S-Sn(2)-C(26)	101.6(1)	S-Sn(2)-C(32)	119.0(1)
S-Sn(2)-C(38)	109.2(1)	C(26)-Sn(2)-C(32)	104.0(1)
C(26)-Sn(2)-C(38)	109.8(1)	C(32)-Sn(2)-C(38)	112.3(1)
Sn(1)-O(1)-C(7)	107.2(2)	Sn(2)-S-C(1)	99.7(1)
O(1)-C(7)-O(2)	122.6(4)	O(1)-C(7)-O(2)	122.6(4)
O(2)-C(7)-C(6)	122.5(4)		

Mössbauer spectra. The Mössbauer spectra were recorded at 80 K on a constant acceleration Cryophysics Microprocessor Mössbauer spectrometer. The velocity range was calibrated with β-tin and calcium stannate against the Ca ^{119m}SnO$_3$ source and the spectra were fitted with a Lorentzian curve-fitting programme supplied by the manufacturer. The Mössbauer data are listed in Table 2.

X-ray crystallography. Bis(triphenyltin)-2-mercaptobenzoate, $C_{43}H_{34}O_2SSn_2$: space group $P2_1/c$, a 14.293(4), b 17.456(2), c 15.624(1) Å, β 107.40(1)°; V 3720(1) Å^3, $Z = 4$; D_m 1.517 g cm^{-3} (flotation in $ZnBr_2/H_2O$), D_c 1.522 g cm^{-3}. The structure was solved from 5837 observed out of 7411 unique Mo-K_α (λ 0.71069 Å) reflections, and refined to an R factor of 0.035 ($R_w = 0.043$). Details of the experimental and computational methods were as described previously [17]. The

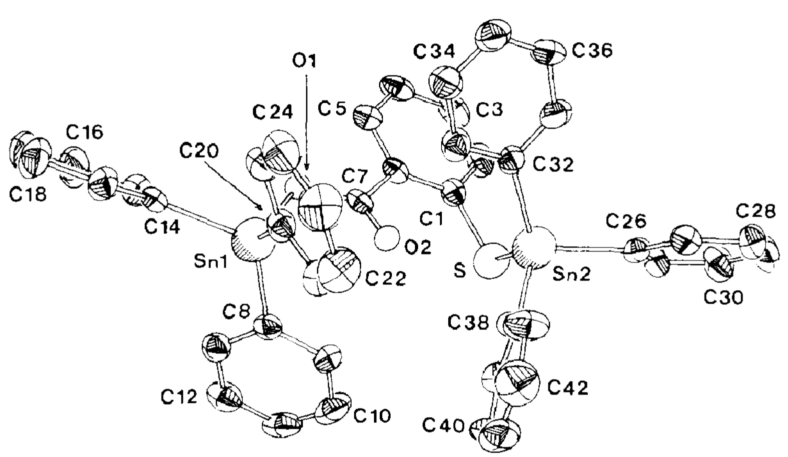

Fig. 1. Perspective view of the molecular structure of bis(triphenyltin)-2-mercaptobenzoate with atom labelling. The thermal ellipsoids are drawn at the 30% probability level.

atomic coordinates and thermal parameters are listed in Table 3, and selected bond lengths and bond angles are presented in Table 4. The molecular structure of the compound is illustrated in Fig. 1.

Results and discussion

The Mössbauer isomer shift (IS) and quadrupole splitting (QS) values for the two $[(C_6H_5)_3SnSCH_2C(O)O]^-$ and $[(C_6H_5)_3SnSC_6H_4C(O)O]^-$ anions are 1.23 (IS), 1.25 (QS) and 1.27 (IS), 1.44 (QS) mm s^{-1}, respectively (Table 2). A chelating mode for the ligand in either anion would render the tin five-coordinate, but rigorous support for this was not provided by the Mössbauer data. Accumulated Mössbauer studies on $R_3Sn(IV)$ compounds indicate that for trigonal bipyramidal coordination, the QS for the commonly encountered *trans* geometry (equatorial R's) lies in the 3.0–3.9 mm s^{-1} range, whereas for the isomeric *cis* (one R apical) and *meridional* (two R's apical) structures, the QS values are in the range 1.7–2.3 and 3.5–4.1 mm s^{-1}, respectively [18,19]. For tetrahedral triorganotin compounds, the QS varies from 1.0 to 2.4 mm s^{-1} [19]. Although the overlap in the QS ranges for four-coordinate tetrahedral and five-coordinate *cis*-trigonal bipyramidal triorganotin compounds precludes a secure assignment of the coordination number at tin, it is noteworthy that examples of the *cis*-C_3SnXY structures are rare compared with those of the ubiquitous *trans*-C_3SnXY. For the crystallographically authenticated triorganotin *N*-phenyl-*N*-benzoylhydroxamates, the QS is 1.94 mm s^{-1} for the triphenyltin and 2.36 mm s^{-1} for the trimethyltin derivative [20,21], compared to 1.65 mm s^{-1} predicted for the *cis*-geometry from point-charge model calculations [22]. By contrast, the QS values for the two triphenylstannylthioester anions are seen to be lower than the observed and predicted values for the above-mentioned hydroxamate derivatives, and are hence considered to have tetrahedral tin atoms. The assignment means that the tin is sulfur-bonded, and that the charge on anion resides not on tin but on the carboxyl group. Given the propensity of tin to achieve high coordination numbers whenever possible, this seemingly unfavorable electrostatic situation can be understood if the amino group of the cation and the carboxylato group are engaged in hydrogen-bonding *. With a stronger Lewis acid acceptor such as the dimethylchlorostannate cation, the 2-mercaptobenzoate ligand, however, functions as a chelate [25].

As in the case of the reported triphenyltin analogues [3], tricyclohexyltin mercaptoacetic acid contains a sulfidotin and a free C(O)OH group (carboxyl absorptions in the infrared: 3500b, 1700s cm^{-1}), and shows a QS of 1.94 mm s^{-1}. The sulfidotins in bis(triphenyltin)mercaptoacetate (QS 1.46 mm s^{-1}) and 2-mercaptobenzoate (QS 1.38 mm s^{-1}) are, by analogy, also assigned the tetrahedral geometry. The carboxylatotins in the last two compounds show moderately large QS values (QS 2.78 mm s^{-1} for the mercaptoacetate and 2.04 mm s^{-1} for the mercaptobenzoate). We are, however, inclined to consider these tin sites as also

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* There are two examples in the literature of metal complexes of 2-mercaptobenzoic acid whose carboxylate group is free; copper 2-mercaptobenzoate hexamethyltetraazocyclotetradecane hydrate [23] and mercury bis(2-mercaptobenzoic acid) dioxane [24] are extensively hydrogen-bonded in the crystal lattice.

tetrahedral, although on the basis of the empirical QS/IS ratio [26] rule which states that ratios > 2.1 are associated with higher coordination and < 1.8 with four coordination, five-coordinate tin environments might be implied. The sulfidotins in the corresponding bis(tri-n-butyltin) compounds are assigned the tetrahedral geometry, but the carboxylatotins show significantly larger QS (3.92, 3.94 mm s^{-1}) values, which clearly argue for five-coordination and *trans*-C_3SnO_2 trigonal bipyramidal geometries. In a test of our Mössbauer interpretations of the bis(triphenyltin) mercaptoesters we undertook the determination of the crystal structure of bis(triphenyltin)-2-mercaptobenzoate.

Molecules of the title compound (Fig. 1) are well separated in the unit cell. The two tin atoms in the molecule are essentially four-coordinate, and the carboxyl oxygen atom appears to point into one face of each of the two tetrahedra. Since it is a 1,2-disubstituted benzene, the compound is severely crowded, and to alleviate crowding by the bulky tin-bearing substituents, the carboxyl carbon and the sulfur atoms twist in opposite directions out of the plane of the benzene ring, the carbon deviating from it by 0.09(1) Å and the sulfur by 0.18(1) Å. The steric effect of *ortho* substitution is also seen in the 23.3(8)° dihedral angle between this aromatic ring and the plane of the carboxyl group. In the carboxylatotin part of the molecule, the tin–oxygen–carbon bond angle of 107.2(2)° is close to the tetrahedral value of 109.5°; the carboxyl oxygen makes a contact of 2.765(3) Å, but this distance is not regarded as arising from a formal bond. A contact of a similar length has been encountered, for example, in four-coordinate triphenyltin benzoate [27]. The oxygen atom opens up the carbon–tin–carbon angles to 109.7(2), 109.9(1) and 120.5(1)°, in accordance with Bent's rule [28], which predicts that the more electronegative oxygen will tend to concentrate *p*-character along the tin–oxygen bond, thereby imparting more *s*-character to the tin–carbon bonds [8]. In the sulfidotin fragment, the tin–sulfur distance (2.414(1) Å) and angle at sulfur (99.7(1)°) are in good agreement with values found for other triphenyltin arylsulfides [10–12]. The essentially tetrahedral geometries at tin thus substantiate the Mössbauer assignment.

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