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Six-coordinate tin in a dialkylchlorooxinatotin(IV) complex: skew-trapezoidal bipyramidal bis(2-carbomethoxyethyl)-chloro(quinolin-8-olato)tin(IV)

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Abstract

Bis(2-carbomethoxyethyl)chloro(quinolin-8-olato)tin(IV), $C_{17}H_{20}ClNO_5Sn$, crystallizes in the triclinic space group $P\bar{1}$, with a 7.9001(7), b 10.2636(7), c 12.8287(9) Å; α 108.575(7), β 98.564(6), γ 96.469(7)°; V 960.69 Å^3, $Z = 2$. The coordination geometry around the tin atom approximates to a skew-trapezoidal bipyramid, with the carbonyl oxygen of one of the 2-carbomethoxyethyl groups intramolecularly bonded to the tin (Sn–O 2.847(4) Å).This oxygen atom, the nitrogen and oxygen atoms of the chelating quinolin-8-olato ligand (Sn–N 2.370(4), Sn–O 2.040(4) Å; N–Sn–O 74.4(1)°), and the chlorine atom (Sn–Cl 2.454(2) Å), occupy the four corners of a trapezoid around the metal atom. The two apical tin–carbon bonds (Sn–C 2.122(6), 2.143(7) Å), which are skewed towards the long edge (defined by the carbonyl oxygen and nitrogen atoms) of the trapezoidal plane, subtend an angle of 135.4(3)° at tin.

Introduction

The 2-carboalkoxyethyltin(IV) halides, $[ROC(O)(CH_2)_2]_nSnCl_{4-n}$ ($n = 1$ or 2), are important intermediates in the synthesis of 'estertins', an important class of organotin stabilizers for polyvinylchloride plastics [1]. The intramolecular donor capability of the carboalkoxy function in these chlorides has been unequivocally

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established by X-ray diffraction studies [2,3]. The tin–oxygen distances are in the range 2.321–2.524 Å, which is normal for tin–oxygen coordinate linkages. The presence of the intramolecular bond provides a rationale for the relatively diminished acceptor properties of this class of organotin halides towards most neutral Lewis bases [4,5]. The halogeno group, however, can be metathetically replaced by a uninegative bidentate ligand which is capable of chelating to the tin atom [4,6–8]. On the basis of solid state and solution spectroscopic data, distorted octahedral geometries were proposed for bis(2-carbomethoxyethyl)tin bis(*N*,*N*-dimethyldithiocarbamate) [4] and the corresponding bis(oxinate) [4,6], and a trigonal bipyramidal geometry for the chlorooxinate title compound [4]. In the mono- and bis-chelate compounds, the ester carbonyl band appears around 1700 cm^{-1} in the infrared, effectively ruling out coordinative involvement of the carbonyl oxygen. However, a seven-coordinate environment for the tin resulting from an intramolecular tin–oxygen bond of 2.751 Å arising from a bent carbomethoxyethyl group, has recently been confirmed by a crystal structure study of the bisdithiocarbamate [9]. If this bond is excluded from the coordination polyhedron, the geometry of the dithiocarbamate is changed from the pentagonal bipyramidal to the skew-trapezoidal bipyramid (STB), a geometry often adopted by dialkyltin bischelates, $R_2Sn(A{-}B)_2$ [10–12]. The STB geometry is associated with a small chelate bite angle and a carbon–tin–carbon angle (135–155°) intermediate between the idealized *cis*- and *trans*-$[SnR_2]$ octahedral angles.

Six-fold coordination at tin, and consequently the STB geometry, may be anticipated for the related mono-chelate compounds, $R_2SnX(A{-}B)$, if an additional donor atom is provided either intramolecularly in the R group or intermolecularly, through the bridging X group (typically, a halide). A crystal structure determination of the title compound was therefore undertaken to examine the intramolecular bonding option. We report in this paper the results of this study, and appraise the above-mentioned possibility of bridging by the X group in compounds of a similar formulation whose structures have been crystallographically verified.

Experimental

The title compound was prepared as previously reported [4], and single crystals suitable for an X-ray diffraction study were obtained by slow evaporation of a chloroform solution of the compound.

Intensity data

Intensity data were collected on a 0.15 × 0.30 × 0.30 mm, clear yellow crystal, using graphite-monochromatized Mo-K_α radiation. A total of 2883 reflections were measured on a Nonius CAD4 diffractometer controlled by the NRCCAD software [13] using the $\omega/2\theta$ scan mode up to $2\theta_{max}$ 45°. The three intensity standards monitored during the data collection did not show any significant decay. After symmetry-averaging, the unique set consisted of 2525 reflections ($-8 \le h \le 8$; $0 \le k \le 11$; $-13 \le l \le 13$) of which 2200 were considered observed reflections with $I_{net} \ge 3.0\sigma(I_{net})$. The intensity data were corrected in the standard fashion for Lorentz and measured direct-beam polarization ratio [14], but not for absorption because of the low μ value. The least-squares refinement of the setting angles of 35

Table 1

Atomic coordinates and isotropic temperature factors

Atom	x	y	z	B_{iso} Å^2
Sn	0.73764(5)	0.17029(4)	0.19448(3)	3.92(2)
Cl	0.44635(19)	0.20603(16)	0.12463(14)	5.56(8)
O(1)	0.6127(4)	0.0021(4)	0.2212(3)	4.8(2)
O(2)	0.8385(6)	0.4122(4)	0.1403(4)	6.3(3)
O(3)	0.7453(7)	0.4446(5)	−0.0191(4)	7.7(3)
O(4)	0.7384(7)	0.5593(5)	0.5522(4)	7.7(3)
O(5)	0.6984(6)	0.3847(4)	0.6166(4)	5.9(2)
N(1)	0.9601(6)	0.0650(4)	0.2636(4)	4.3(2)
C(2)	1.1332(7)	0.0948(6)	0.2804(5)	5.1(3)
C(3)	1.2376(8)	0.0234(7)	0.3294(6)	5.7(4)
C(4)	1.1701(8)	−0.0761(7)	0.3645(6)	5.5(4)
C(5)	0.9013(9)	−0.2161(7)	0.3832(6)	5.6(4)
C(6)	0.7242(9)	−0.2414(8)	0.3623(6)	5.8(4)
C(7)	0.6270(9)	−0.1691(6)	0.3094(6)	5.2(4)
C(8)	0.7022(7)	−0.0694(5)	0.2742(5)	4.1(3)
C(9)	0.8892(7)	−0.0388(5)	0.2968(4)	3.9(3)
C(10)	0.9872(7)	−0.1134(6)	0.3500(5)	4.4(3)
C(11)	0.8339(11)	0.1293(8)	0.0434(6)	5.4(4)
C(12)	0.7686(12)	0.2148(8)	−0.0263(6)	5.7(4)
C(13)	0.7880(8)	0.3659(6)	0.0414(5)	5.0(3)
C(14)	0.7540(30)	0.5900(15)	0.0416(14)	10.1(10)
C(15)	0.7910(10)	0.3370(8)	0.3525(6)	5.6(3)
C(16)	0.6727(11)	0.3150(8)	0.4261(6)	6.9(4)
C(17)	0.7068(7)	0.4343(7)	0.5366(5)	5.1(3)
C(18)	0.7307(16)	0.4804(9)	0.7277(6)	7.5(5)

reflections with 40° ≤ 2θ ≤ 44° (λ(Mo-K_{α_1}) 0.70932 Å) was performed to give the accurate cell parameters.

Structure solution and refinements

The structure was solved by direct-methods and standard difference map techniques. The tin atom was found from an *E*-map derived by MULTAN [15], and its

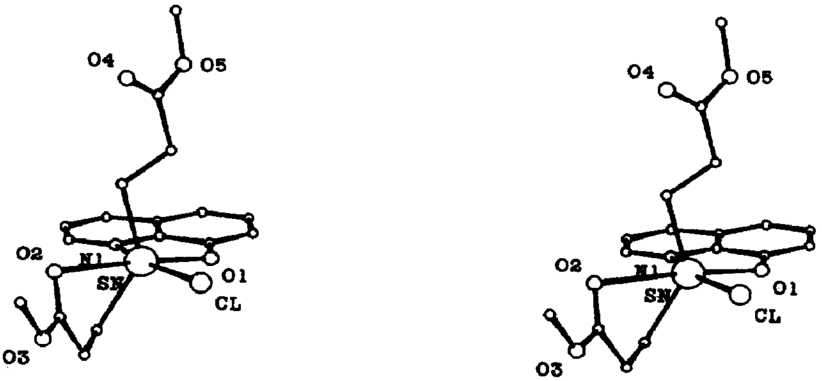

Fig. 1. Stereoview of the molecular structure of bis(2-carbomethoxyethyl)chloro(quinolin-8-olato)tin, with the atom numbering scheme.

position was cross-checked with a Patterson map. The atomic coordinates for the tin were then refined by full-matrix least-squares. All the remaining non-hydrogen atoms were located on the next heavy atom-phased difference map. All non-hydrogen atoms were refined anisotropically. The hydrogen atoms located on the subsequent difference map and their fractional coordinates were refined together with isotropic thermal parameters. Full matrix least-squares refinement with weighting scheme $w^{-1} = \sigma^2(F_o) + 0.00025F_o^2$, converged to the following residuals: R_f 0.032

Table 2

Bond distances (Å) and angles (°)

Bond distances			
Sn–Cl	2.454(2)	C(2)–C(3)	1.374(9)
Sn–O(1)	2.040(3)	C(3)–C(4)	1.331(11)
Sn–O(2)	2.847(4)	C(4)–C(10)	1.421(9)
Sn–N(1)	2.370(4)	C(5)–C(6)	1.363(10)
Sn–C(11)	2.122(6)	C(5)–C(10)	1.402(9)
Sn–C(15)	2.143(7)	C(6)–C(7)	1.384(10)
O(1)–C(8)	1.339(7)	C(7)–C(8)	1.356(8)
O(2)–C(13)	1.190(8)	C(8)–C(9)	1.440(7)
O(3)–C(13)	1.323(7)	C(9)–C(10)	1.401(8)
O(3)–C(14)	1.436(15)	C(11)–C(12)	1.516(11)
O(4)–C(17)	1.223(9)	C(12)–C(13)	1.494(10)
O(5)–C(18)	1.416(9)	C(15)–C(16)	1.472(11)
N(1)–C(2)	1.336(7)	C(16)–C(17)	1.512(11)
N(1)–C(9)	1.362(7)		
Bond angles			
Cl–Sn–O(1)	85.7(1)	N(1)–C(2)–C(3)	121.5(5)
Cl–Sn–O(2)	82.0(1)	C(2)–C(3)–C(4)	121.2(6)
Cl–Sn–N(1)	160.0(1)	C(3)–C(4)–C(10)	120.7(6)
Cl–Sn–C(11)	98.4(2)	C(4)–C(10)–C(5)	125.9(6)
Cl–Sn–C(15)	98.0(2)	C(4)–C(10)–C(9)	114.9(5)
O(1)–Sn–O(2)	167.7(1)	C(10)–C(9)–N(1)	123.8(5)
O(1)–Sn–N(1)	74.4(1)	C(9)–N(1)–C(2)	118.0(5)
O(1)–Sn–C(11)	116.5(2)	N(1)–C(9)–C(8)	115.9(5)
O(1)–Sn–C(15)	105.9(2)	C(9)–C(8)–O(1)	118.7(5)
O(2)–Sn–N(1)	117.9(2)	C(9)–C(8)–C(7)	117.5(5)
O(2)–Sn–C(11)	65.7(3)	O(1)–C(8)–C(7)	123.8(5)
O(2)–Sn–C(15)	75.9(3)	C(8)–C(7)–C(6)	122.0(6)
N(1)–Sn–C(11)	88.8(2)	C(8)–C(9)–C(10)	120.3(5)
N(1)–Sn–C(15)	89.7(3)	C(7)–C(6)–C(5)	121.5(6)
C(11)–Sn–C(15)	135.4(3)	C(6)–C(5)–C(10)	119.4(6)
Sn–O(1)–C(8)	120.2(3)	C(5)–C(10)–C(9)	119.3(5)
Sn–O(2)–C(13)	99.3(4)	C(11)–C(12)–C(13)	112.5(6)
Sn–N(1)–C(2)	132.1(4)	C(12)–(13)–O(2)	123.6(6)
Sn–N(1)–C(9)	109.8(3)	C(12)–C(13)–O(3)	113.6(6)
Sn–C(11)–C(12)	112.8(5)	O(2)–C(13)–O(3)	122.8(6)
Sn–C(15)–C(16)	111.9(5)	C(13)–O(3)–C(14)	116.4(8)
		C(15)–C(16)–C(17)	112.1(6)
		C(16)–C(17)–O(4)	127.7(7)
		C(16)–C(18)–O(5)	109.2(6)
		O(4)–C(17)–O(5)	123.2(5)
		C(17)–O(5)–C(18)	117.9(5)

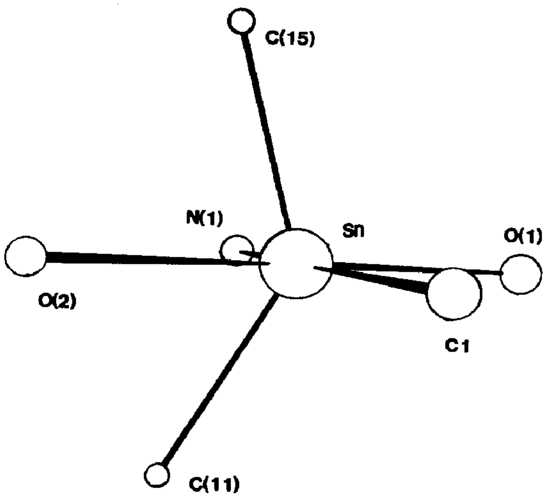

Fig. 2. View of bis(2-carbomethoxyethyl)chloro(quinolin-8-olato)tin showing the trapezoidal plane.

and R_w 0.039, for the significant reflections and R_f 0.038 and R_w 0.041 for all reflections, with a goodness-of-fit ratio of 1.63. The $(\Delta/\sigma)_{max}$ in the last cycle was 0.8 (an H atom). The final difference map was featureless with a background of less than 0.44 eÅ^{-3}. All calculations were performed with the NRCVAX system of programs [16]. The atomic scattering factors were from standard sources [17]. The final atomic coordinates of the non-hydrogen atoms are given in Table 1. The structure of the complex with numbering scheme is depicted in Fig. 1 and Table 2 lists bond lengths and angles involving non-hydrogen atoms. Lists of anisotropic temperature factors, hydrogen atom coordinates, least-squares planes and observed, unobserved and calculated structure factor amplitudes are available from the authors on request.

Crystal data. 2-Carbomethoxyethyl-2-carbomethoxyethylchloro-quinolin-8-olato-tin(IV), $[CH_3OC(O)(CH_2)_2]_2SnCl(C_9H_6NO)$: Triclinic, space group $P\bar{1}$, a 7.9001(7), b 10.2636(7), c 12.8287(9) Å; α 108.575(7), β 98.564(6), γ 96.469(7)°; V 960.69 Å^3, $Z = 2$, D_x 1.633 g cm^{-3}, μ 1.5 mm^{-1}.

Discussion

The title compound, bis(2-carbomethoxyethyl)tinchlorooxinate, is a discrete molecule (Figs. 1, 2) with no close intermolecular contacts. If consideration is given first to a five-coordinate trigonal bipyramidal geometry, we note that the oxygen and two carbon atoms comprise the trigonal plane about which the chlorine and nitrogen atoms are apically positioned. The geometry is, however, distorted, as is evident from the widened carbon–tin–carbon angle of 135.4(3)°, and the bent axial chlorine–tin–nitrogen skeleton (Cl–Sn–N 160.0(1)°). The distortion is also seen in the sum of the angles subtended in the equatorial plane (357.8(7)°); the tin atom is displaced out of this plane by 0.178(5) Å towards the chlorine. The distortion appears to be caused by one of the 2-carbomethoxyethyl groups, which bends back to engage the tin in a weak coordination through the carbonyl oxygen atom (Sn–O 2.847(4) Å). This contact raises the coordination number at the metal to six, a result which is readily rationalized in terms of the known propensity of diorganotin compounds to achieve higher coordination numbers whenever possible. The 2-carboalkoxyl group is a poor Lewis base towards tin, and there are few structural examples of the interaction of this group with tin. Nevertheless, the tin–oxygen distance found in the title compound is not significantly longer than the intramolecular bonds in the seven-coordinate bis(2-carbomethoxyethyl)tin bis(*N*,*N*-dimethyldithiocarbamate (2.751 Å [9]) and in the two six-coordinate bis(1,2-diethoxycarbonylethyl)tin dibromide isomers (2.44–2.49 Å [18]).

The tin atom in the $R_2SnX(A-B)$ compound dimethyltin chlorocysteinate [19] cannot increase its coordination number beyond five because there is steric hindrance to participation of the carbonyl oxygen in either intra- or inter-molecular bonding. Furthermore, the tin–chlorine distance of 4.268 Å precludes a halogen bridging interaction. In the dimethyltin chloro(2-mercaptobenzoate) anion [20], the carbonyl oxygen can only bond to the neighbouring tin because it is a constituent atom of the chelate ligand. As detailed in Table 3, the tin-oxygen distance in the anion is 3.676 Å, which is close to the sum of the Van der Waals radii for tin and oxygen (3.70 Å [21]). We have previously noted large tin–oxygen distances in polymeric triorganotin carboxylates [22]. In the dimethyltin iodo(2-phenoxymercaptate) anion [20] and in dimethyltin hydroxynitrate [23], six-coordination at tin is achieved by dimerization: the intermolecular tin–oxygen bond distances are 2.646 and 2.182 Å, respectively.

There are no suitable basic atoms in the chelating ligand in dimethyltin chlorobis(pyrazolyl)borate and dimethyltin chlorodimethyldithiocarbamate, but there are weak tin–chlorine contacts (3.923 Å [24] and 3.906 Å [25], respectively) within the sum of the Van der Waals radii for tin and chlorine (4.10 Å [22]). In a previous communication [26], we noted that 1/1 adducts of dimethyltin dichloride with 'pointed' oxygen-donor ligands, stated in the literature to be five-coordinate molecules, are likely to six-coordinate dimers as a result of chlorine bridging. The tin–chlorine bridges in the diphenylcyclopropenone [27], 2,6-dimethylpyridine *N*-oxide [28], tetramethylurea [29] and (methylphenylamino)phenylsulfine [30] adducts, for example, range from 3.31 to 3.66 Å. The Sn–Cl bridges in the dimethyltin chlorobis(pyrazolyl)borate and – chlorodimethyldithiocarbamate compounds are not significantly longer than the 3.54 Å bridge noted in six-coordinate dimethyltin dichloride [31]. The structures of these two compounds are therefore re-interpreted as six-coordinate dimers. There is an intramolecular tin–oxygen distance of 2.582 Å in the monomeric 1/1 adduct of di-n-butyltin dichloride with the bidentate ligand bis(diphenylphosphoryl)methane [32]. The chlorine and oxygen atoms in this structure make up a flat plane which is almost perpendicular to the plane carrying the C_2Sn skeleton (dihedral angle 88.5°); the adduct should also be regarded as being six-coordinate. On the other hand, the dimethyltin fluoro(2-phenoxymercaptate) anion [20] appears to be a truly monomeric five-coordinate dialkyltinhalochelate, whose intermolecular contacts exceed 4 Å. Intermolecular contact distances also exceed 4 Å in the mixed alkylaryltinhalochelate, n-butylphenyltin chlorodiethyldithiocarbamate [33] and the diaryltinhalochelate, diphenyltin chloro-i-propylxanthate [34].

Returning to the title compound, we note that the carbonyl oxygen atom involved in intramolecular coordination, together with the chlorine, and the nitrogen and oxygen donor atoms of the oxinato ligand, make an approximate trapezoid. The tin–carbon bonds are skewed towards the long edge of this quadrilateral. The tin atom is displaced by 0.024(2) Å from the trapezoidal plane (sum of internal angles is

Table 3

Structural data for $R_2SnX(A-B)$ [a] compounds

$R_2SnX(A-B)$	R–Sn–R (°)	Sn–Y [b] (Å)	A–Sn–B (°)	B–Sn–X (°)	X–Sn–Y (°)	Y–Sn–A (°)	Dihedral [c] angle (°)	Ref.
$(CH_3)_2SnCl[OC(O)CH_3]$ [d]	140.9	2.392(Y=O)	50.9	87.0	83.2	138.9	90.6	[e]
$(CH_3)_2SnCl[OC(O)C_6H_4S\text{-}2]^-$	130.3	3.676 (Y=O)	84.9	87.6	86.4	101.3	89.8	20
$(CH_3)_2SnI[OC_6H_4S\text{-}2]^-$	140.1	2.646 (Y=O)	82.1	83.5	67.1	127.7	89.4	20
$(CH_3)_2Sn(OH)(NO_3)$ [f]	139.9	2.182 (Y=O) [g]	48.1	77.9	71.5	162.5	91.2	23
$(CH_3)_2SnCl[(C_3H_3N_2)_2BH_2]$	133.7	3.923 (Y=Cl)	82.1	84.5	72.6	120.3	89.5	24
$(CH_3)_2SnCl[SC(S)N(CH_3)_2]$ [h]	127.9	3.906 (Y=Cl)	68.2	86.4	74.0	131.4	90.6	25
$(CH_3)_2SnCl[SCH_2CH(NH_2)C(O)OC_2H_5]$	119.5	4.268 (Y=Cl)	80.1	86.8	81.6	111.5	88.4	19
$(CH_3)_2SnF[OC_6H_4S\text{-}2]^-$	117.6		81.6		85.1			20
$[CH_3OC(O)(CH_2)_2]_2SnCl[C_9H_6NO]$	135.4(3)	2.847(4) (Y=O)	74.4(1)	85.7(1)	82.0(1)	117.9(1)	91.1(2)	This work

[a] A–B represents a uninegative bidentate ligand. [b] Intermolecular 'bond' distances; Y identifies the bridging atom (of the X group or A–B ligand). In $[CH_3OC(O)(CH_2)_2]_2SnCl[C_9H_6NO]$, Y represents the intramolecular quinolin-8-olato oxygen atom. [c] Angle between C–Sn–C and A–B–X–Y planes. [d] Error in published coordinates: C(3) should read *z* 0.2530. The acetato group is assumed to be chelating in dimethyltin chloroacetate. [e] D.W. Allen, I.W. Nowell, J.S. Brooks and R.W. Clarkson, J. Organomet. Chem., 219 (1981) 29. [f] The nitrato group is assumed to be chelating in dimethyltin hydroxynitrate. [g] O refers to the hydroxyl oxygen atom. [h] Error in published coordinates: C(5) should read *z* 0.081.

(360.0(5)°), which makes an 8.1(1)° angle with the flat quinolin-8-olato ligand. The plane containing the tin–carbon bonds is almost perpendicular to the trapezoidal plane (dihedral angle 91.1(2)°). The overall geometry of the molecule, shown in Fig. 2, approximates to a distorted STB. The geometry is also involved in several of the compounds listed in Table 3.

Acknowledgements

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