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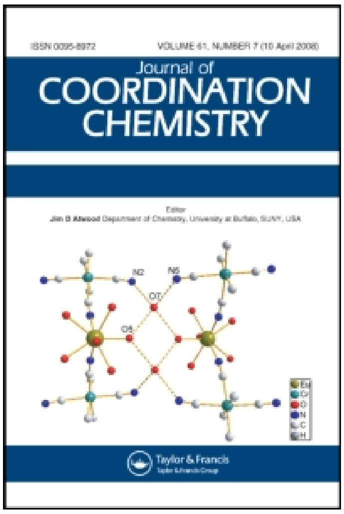

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Synthesis and Crystal Structure of the Tetrabutyl*bis*(3,5-Dichloro-4-Hydroxybenzoato)Distannoxane Dimer, $\{[(C_4H_9)_2SnO_2CC_6H_2(Cl)_2(OH)]_2O\}_2$

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SYNTHESIS AND CRYSTAL STRUCTURE OF THE TETRABUTYL*BIS*(3,5-DICHLORO-4-HYDROXYBENZOATO)DISTANNOXANE DIMER, $\{[(C_4H_9)_2SnO_2CC_6H_2(Cl)_2(OH)]_2O\}_2$

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Reaction of di-*n*-butyltin oxide with 3,5-dichloro-4-hydroxybenzoic acid yields the title compound which crystallizes as the usual dicarboxylatotetraorganodistannoxane dimer. A crystal structure determination of the compound reveals the presence of a centrosymmetric planar Sn_2O_2 unit connected to exocyclic Sn atom *via* two bidentate bridging carboxylate ligands. The remaining two carboxylate ligands are coordinated to the exocyclic Sn atom in the monodentate mode.

Keywords: Dicarboxylatotetraorganodistannoxane; Centrosymmetric; Bidentate; Monodentate; X-ray structure

INTRODUCTION

The chemistry of organotin carboxylates has attracted much attention owing to their industrial [1,2] and agricultural importance [3,4], and more recently their anti-tumour activity [5,6]. These compounds may adopt a variety of structural modes depending on the nature of the organic substituent on the Sn atom and the type of carboxylate ligand [7]. There have been numerous crystallographic studies of dicarboxylato tetraorganodistannoxanes of formula $(R_2SnCO_2CR')_2O\}_2$ [8–12]. In this paper we report the preparation, spectroscopic and X-ray characterization of the tetrabutylbis(3,5-dichloro-4-hydroxybenzoato)distannoxane dimer. The compound was studied in order to reveal the influence of the presence of two chlorine atoms and a hydroxy group residing on the carboxylate ligand.

EXPERIMENTAL

Reagents

Di-*n*-butyltin oxide was obtained from Fluka Chemie AG and 3,5-dichloro-4-hydroxybenzoic acid from the Aldrich Chemical Company.

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Preparation of Tetrabutyl*bis*(3,5-dichloro-4-hydroxybenzoato)distannoxane

Di-*n*-butyltin oxide (1.00 g, 4 mmol), 3,5-dichloro-4-hydroxybenzoic acid (1.66 g, 8 mmol) and 50 cm^3 of toluene were refluxed until a clear solution formed. The solution was filtered while it was still hot. The solvent was removed by evaporation under vacuum until a white solid was obtained. The solid was then recrystallized from toluene/hexane to give colourless, needle-shaped crystals of the distannoxane. It is interesting to note that the oxide does not react with the acid when a molar ratio of 1 : 1 is used. Addition of excess ligand to the reaction mixture initiates the reaction. The sodium salt of 3,5-dichloro-4-hydroxybenzoic acid was prepared by reacting sodium metal with a solution of the acid in methanol.

Characterization

Microanalyses were carried out on a Control Equipment Corporation Model 240XA instrument. IR data were recorded using a Perkin-Elmer FTIR 2000 spectrophotometer in the frequency range 4000–400 cm^{-1} with the sample prepared in KBr discs. ^1H and ^{13}C NMR spectra were obtained with a Bruker AC-P 300 mHz FT-NMR spectrometer in DMSO-d_6.

M.p. 121.1–121.6°C. *Analysis*: Found: C, 40.88%; H, 4.15%. Calculated for $C_{30}H_{42}Cl_4O_7Sn_2$: C, 40.27%; H, 4.70%. The IR spectrum of the free acid shows a sharp band at 3458 cm^{-1} attributed to the pendant OH group, a broad band at 2821–3098 cm^{-1} due to O–H stretching of the carboxylic group and two sharp bands at 768 and 809 cm^{-1} due to C–Cl stretches. The IR spectrum of the complex displays strong bands at 635 and 486 cm^{-1} which can be assigned to Sn–O–Sn and Sn–O stretches, respectively, and two sharp bands at 781 and 801 cm^{-1} due to C–Cl stretching. The carbonyl stretching bands are located at 1590 and 1554 cm^{-1} (asymmetric) and 1389 and 1330 cm^{-1} (symmetric). ^1H NMR (DMSO-d_6) for $[(H_3C^1H_2C^2H_2C^3H_2C^4)Sn)CCH-(Cl)(OH)]2O$: δ (t, 12H, $4C^1H_3$) 0.79; δ (m, 8H, $4C^2H_2$) 1.27; and (s, 16H, $4C^3H_2C^4H_2$) 1.57; δ (s, 4H, $2C_6H_2$) 7.70 ppm. ^{13}C NMR (DMSO-d_6) δ: C^1 13.6; C^2 26.0; C^3 26.9; C^4 29.2; C(O)O 169.0; C_6H_2 123.1, 124.0, 129.6, 154.5 ppm.

X-ray Crystal Structure of Tetrabutylbis(3,5-dichloro-4-hydroxybenzoic)distannoxane

Intensity data for a crystal of size 0.2 × 0.2 × 0.5 mm were measured at 298 K on a Siemens P4 diffractometer using graphite monochramatized MoKα radiation, $\lambda = 0.71073$ Å; the θ–2θ scan technique was employed to measure a total of 27 749 reflections up to $2\theta\text{max} = 55.0°$. Some 10 436 of the 19 096 independent reflections ($R_{\text{int}} = 0.0409$ satisfying the $F > 4.0\sigma(F)$ criterion of observability were used for the solution and refinement. The structure was solved by using direct methods and refined by full-matrix least-squares procedures based on F using the Siemens SHELXTL (PC version) system [13]. Refinement continued to final $R = 0.0668$ and $R_w = 0.0660$. The final difference map had peaks between -0.89 e Å^{-3} and 1.40 e Å^{-3}. Crystal data and refinement parameters are given in Table I. Final atomic coordinates for non-hydrogen atoms in Table II and selected bond lengths and angles in Table III. Lists of additional data including atomic coordinates, anisotropic temperature factors are available from S.-G.T.

TABLE I Crystal data and refinement details for $\{[(C_4H_9)_2SnO_2CC_6H_2(Cl)_2(OH)]_2O\}_2$

Crystal Data	
Formula	$C_{30}H_{42}Cl_4O_7Sn_2$
Crystal system	Triclinic
Space group	$P\bar{1}$
Unit cell dimensions	$a = 13.1331(2)$ Å
	$b = 15.3456(1)$ Å
	$c = 20.4970\ (3)$ Å
	$\alpha = 100.7379\ (9)^\circ$
	$\beta = 97.3973\ (2)^\circ$
	$\gamma = 95.4390\ (10)^\circ$
Volume	3994.74 (9) Å^3
Z	4
Formula weight	893.8
Density(calc.)	1.486 Mg/m^3
Absorption coefficient	1.555 mm^{-1}
$F(000)$	1784
Refinement	
Quantity minimized	$\sum w(F_o - F_c)^2$
Hydrogen atoms	Riding model, fixed isotropic U
Weighting scheme	$w^{-1} = \sigma^2(F) + 0.0000F^2$
Number of parameters refined	615
Final R indices (obs. data)	$R = 0.0668$, $wR2 = 0.0660$
Goodness-of-fit	2.58
Largest and mean Δ/σ	4.503, 0.086
Data-to-parameter ratio	17.0 : 1

RESULTS AND DISCUSSION

The crystal structure of $\{[(C_4H_9)_2SnO_2CC_6H_2(Cl)_2(OH)_2(OH)]_2O\}_2$ reveals the presence of two independent half molecules in the triclinic unit cell, each of which is located about a crystallographic centre of inversion. The molecular structure of one of the independent units and the numbering scheme employed is shown in Fig. 1.

The core geometry of the molecule consists of a centrosymmetric, planar, four-membered Sn_2O_2 unit with Sn(1)–O(7)–Sn(1A) angle 103.5(3)° and O(7)–Sn(1)–O(7A) angle 76.6(3)°. The Sn_2O_2 unit is connected to a pair of exocyclic Sn atoms *via* the bridging O atoms so that the O atoms are tricoordinate. There are two distinct carboxylate ligands in the structure. The first, defined by the O(1) and O(2) atoms, symmetrically bridges Sn(1) and Sn(2) atoms. The second is essentially monodentate, coordinating to the Sn(2) atom *via* O(3) only. This ligand does not show any weak intramolecular interaction with the Sn(2) atom *via* the O(4) atom. Cl(1), Cl(2) and O(5) do not participate in coordination to the Sn centres, being directed away from the central unit.

The endocyclic Sn atom forms five significant bonds in the structure and exists in distorted trigonal bipyramidal geometry with two atoms occupying *trans* positions. The trigonal plane is defined by the two butyl groups and an O atom and the axial positions by two atoms which form an angle of 167.9(4)°. The exocyclic Sn atom also exists in distorted trigonal bipyramidal geometry with the basal plane defined by an O atom and two C atoms of the butyl groups; the axial sites are occupied by two O atoms which form an angle of 170.9(4)° at the Sn atom.

The IR spectrum of the free acid shows a broad O–H absorption at 2821–3098 cm^{-1} which is absent in the spectrum of the complex, showing the deprotonation and coordination of the carboxylate groups. A sharp peak at 3458 cm^{-1} remains, indicating

TABLE II Atomic coordinates (× 10^4) and equivalent isotropic displacement coefficients ($Å^2 \times 10^3$)

	x/a	y/b	z/c	$U_{(eq)}$
Sn(1)	10336 (1)	894 (1)	– 5310 (1)	43 (1)
Sn(2)	7884 (1)	807 (1)	– 4535 (1)	48 (1)
Sn(3)	4890 (1)	– 4064 (1)	522 (1)	51 (1)
Sn(4)	4539 (1)	– 4044 (1)	– 1322 (1)	61 (1)
C1(1)	8034 (3)	– 3834 (3)	– 3850 (3)	99 (2)
C1(2)	4445 (3)	– 2855 (3)	– 3129 (3)	87 (2)
C1(3)	9848 (4)	5269 (3)	– 6116 (4)	144 (4)
C1(4)	6171 (4)	4770 (3)	– 5281 (3)	124 (3)
C1(5)	4186 (6)	561 (3)	1588 (3)	165 (4)
C1(6)	2886 (5)	285 (3)	– 1023 (3)	142 (3)
C1(7)	5879 (4)	– 8616 (2)	– 2514 (2)	82 (2)
C1(8)	4883 (3)	– 7365 (3)	– 4769 (2)	79 (2)
O(1)	9585 (8)	2165 (6)	– 5340 (7)	90 (6)
O(2)	8118 (8)	2014 (6)	– 4994 (6)	88 (5)
O(3)	7811 (6)	– 496 (5)	– 4189 (5)	50 (3)
O(4)	6410 (7)	– 103 (5)	– 3802 (5)	63 (4)
O(5)	6059 (7)	– 4092 (5)	– 3397 (5)	72 (5)
O(6)	7818 (11)	5710 (9)	– 5824 (10)	182 (11)
O(7)	9190 (6)	388 (5)	– 4849 (4)	48 (3)
O(8)	4915 (7)	– 5354 (5)	– 1787 (4)	57 (4)
O(9)	4630 (7)	– 4776 (5)	– 2692 (4)	60 (4)
O(10)	4722 (9)	– 2650 (5)	348 (5)	79 (5)
O(11)	4121 (10)	– 2777 (6)	– 710 (6)	102 (6)
O(12)	5562 (7)	– 8659 (5)	– 3994 (4)	64 (4)
O(13)	3295 (11)	1156 (6)	380 (6)	125 (7)
O(14)	4847 (6)	– 4542 (5)	– 483 (4)	51 (3)
C(1)	8786 (11)	2434 (8)	– 5234 (8)	57 (6)
C(2)	8506 (10)	3298 (8)	– 5401 (7)	49 (5)
C(3)	7565 (10)	3571 (8)	– 5280 (7)	60 (6)
C(4)	7315 (12)	4369 (10)	– 5425 (10)	83 (8)
C(5)	8046 (15)	4907 (10)	– 5659 (11)	106 (10)
C(6)	8987 (12)	4604 (10)	– 5777 (10)	90 (9)
C(7)	9183 (11)	3808 (9)	– 5644 (9)	73 (7)
C(8)	7008 (9)	– 661 (7)	– 3933 (7)	44 (5)
C(9)	6774 (9)	– 1557 (7)	– 3763 (6)	40 (4)
C(10)	7451 (10)	– 2230 (8)	– 3857 (8)	52 (5)
C(11)	7175 (10)	– 3056 (8)	– 3741 (7)	55 (6)
C(12)	6275 (10)	– 3275 (8)	– 3497 (7)	52 (5)
C(13)	5628 (10)	– 2610 (8)	– 3395 (7)	54 (6)
C(14)	5863 (9)	– 1785 (8)	– 3521 (7)	49 (5)
C(15)	11497 (10)	1718 (9)	– 4598 (7)	63 (4)
C(16)	11168 (12)	2120 (10)	– 3948 (8)	86 (5)
C(17)	12116 (15)	2561 (14)	– 3428 (11)	124 (7)
C(18)	11818 (26)	3050 (21)	– 2900 (18)	344 (25)
C(19)	8276 (11)	1580 (9)	– 3548 (7)	67 (4)
C(20)	8198 (13)	2615 (10)	– 3485 (9)	92 (5)
C(21)	8396 (19)	3167 (16)	– 2758 (13)	166 (10)
C(22)	8186 (19)	4157 (16)	– 2796 (14)	192 (12)
C(23)	9813 (14)	563 (12)	– 6385 (9)	107 (6)
C(24)	10041 (28)	914 (21)	– 6982 (11)	306 (20)
C(25)	9917 (25)	444 (19)	– 7692 (14)	245 (16)
C(26)	10331 (49)	428 (43)	– 8332 (22)	2073 (173)
C(27)	6601 (11)	366 (10)	– 5344 (8)	77 (4)
C(28)	6717 (21)	344 (18)	– 6055 (9)	194 (12)
C(29)	6708 (30)	189 (24)	– 6792 (9)	343 (25)
C(30)	6768 (45)	799 (33)	– 7272 (26)	2045 (168)
C(31)	4839 (10)	– 5410 (8)	– 2437 (7)	50 (5)
C(32)	5020 (9)	– 6288 (7)	– 2825 (6)	42 (5)
C(33)	4888 (10)	– 6391 (7)	– 3522 (6)	49 (5)
C(34)	5072 (10)	– 7208 (8)	– 3900 (6)	48 (5)

TABLE II (Continued)

	x/a	y/b	z/c	$U_{(eq)}$
C(35)	5379 (9)	−7906 (7)	−3589 (7)	50 (5)
C(36)	5481 (10)	−7778 (8)	−2892 (6)	49 (5)
C(37)	5298 (10)	−6986 (8)	−2530 (6)	50 (5)
C(38)	4327 (12)	−2363 (9)	−129 (8)	65 (6)
C(39)	4051 (12)	−1417 (8)	11 (8)	62 (6)
C(40)	3651 (12)	−1041 (9)	−499 (7)	70 (6)
C(41)	3397 (13)	−202 (9)	−374 (8)	79 (7)
C(42)	3557 (14)	307 (9)	277 (9)	83 (8)
C(43)	3979 (14)	−60 (9)	782 (8)	84 (7)
C(44)	4247 (12)	−923 (9)	649 (8)	76 (7)
C(45)	6406 (11)	−3495 (10)	963 (8)	79 (5)
C(46)	7090 (12)	−3077 (11)	557 (8)	102 (6)
C(47)	8172 (12)	−2652 (16)	765 (13)	180 (11)
C(48)	8406 (51)	−2113 (38)	262 (27)	2971 (170)
C(49)	2921 (12)	−4345 (11)	−1721 (9)	98 (6)
C(50)	2620 (22)	−3567 (16)	−1998 (16)	262 (17)
C(51)	1527 (19)	−3540 (21)	−2274 (25)	502 (40)
C(52)	1463 (23)	−2714 (21)	−2544 (18)	447 (36)
C(53)	3269 (12)	−4152 (12)	684 (9)	92 (5)
C(54)	2286 (20)	−3822 (26)	461 (24)	480 (39)
C(55)	1839 (57)	−3105 (34)	167 (39)	2727 (170)
C(56)	1593 (53)	−2193 (27)	129 (37)	4000 (170)
C(57)	5860 (11)	−3190 (10)	−1445 (9)	86 (5)
C(58)	6770 (13)	−3558 (12)	−1626 (10)	109 (6)
C(59)	7648 (15)	−2869 (14)	−1663 (12)	133 (8)
C(60)	8474 (21)	−3263 (18)	−1820 (16)	250 (16)

Equivalent isotropic U defined is as one third of the trace of the orthogonalized U_{ij} tensor.

TABLE III Selected Bond Lengths (Å) and Angles (°)

Sn(1)–O(1)	2.275 (10)
Sn(1)–O(7)	2.044 (9)
Sn(1)–C(15)	2.105 (12)
Sn(1)–C(23)	2.169 (18)
Sn(2)–O(2)	2.243 (12)
Sn(2)–O(3)	2.241 (8)
Sn(2)–O(7)	2.028 (8)
Sn(2)–C(19)	2.118 (13)
Sn(2)–C(27)	2.166 (14)
Sn(3)–O(10)	2.290 (9)
Sn(3)–O(14)	2.045 (8)
Sn(3)–C(45)	2.112 (13)
Sn(3)–C(53)	2.192 (16)
Sn(4)–O(8)	2.189 (8)
Sn(4)–O(11)	2.264 (11)
Sn(4)–O(14)	2.020 (9)
Sn(4)–C(49)	2.149 (15)
Sn(4)–C(57)	2.143 (16)
O(1)–Sn(1)–O(7)	91.5 (4)
O(1)–Sn(1)–C(15)	85.3 (5)
O(7)–Sn(1)–C(15)	110.7 (5)
O(1)–Sn(1)–C(23)	85.2 (6)
O(7)–Sn(1)–C(23)	107.7 (6)
C(15)–Sn(1)–C(23)	140.6 (7)
O(1)–Sn(1)–O(7A)	167.9 (4)
O(7)–Sn(1)–O(7A)	76.5 (3)
C(15)–Sn(1)–O(7A)	97.2 (4)

(Continued)

TABLE III (Continued)

C(23)–Sn(1)–O(7A)	99.9 (5)
O(2)–Sn(2)–O(3)	170.9 (4)
O(2)–Sn(2)–O(7)	89.9 (4)
O(3)–Sn(2)–O(7)	81.6 (3)
O(2)–Sn(2)–C(19)	92.1 (5)
O(3)–Sn(2)–O(19)	93.7 (5)
O(7)–Sn(2)–C(19)	108.5 (5)
O(10)–Sn(3)–O(14)	91.5 (3)
O(10)–Sn(3)–O(14A)	168.1 (4)
O(14)–Sn(3)–O(14A)	77.3 (4)
C(45)–Sn(3)–O(14A)	96.5 (5)
C(53)–Sn(3)–O(14A)	99.7 (5)
O(8)–Sn(4)–O(11)	171.7 (4)
O(8)–Sn(4)–O(14)	81.4 (3)
O(11)–Sn(4)–O(14)	90.7 (4)
Sn(1)–O(1)–C(1)	137.6 (10)
Sn(2)–O(2)–C(1)	140.8 (10)
Sn(2)–O(3)–C(8)	113.0 (7)
Sn(1)–O(7)–Sn(2)	135.8 (4)
Sn(1)–O(7)–Sn(1A)	103.5 (3)
Sn(2)–O(7)–Sn(1A)	120.7 (4)
Sn(4)–O(8)–C(31)	109.1 (7)
Sn(3)–O(14)–Sn(4)	135.0 (4)
Sn(3)–O(14)–Sn(3A)	102.7 (4)
Sn(4)–O(14)–Sn(3A)	122.1 (4)

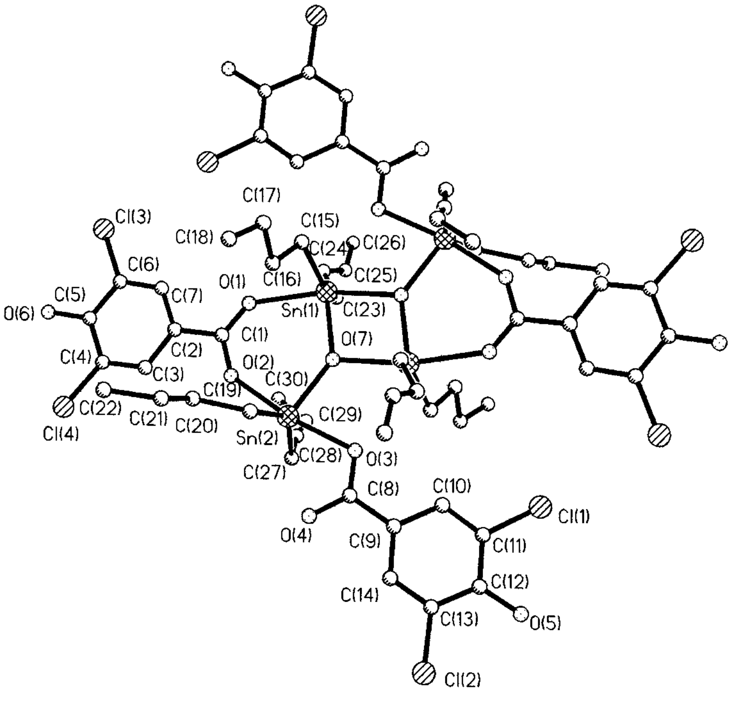

FIGURE 1 Crystal Structure of $\{[(C_4H_9)_2SnO_2CC_6H_2(Cl)_2(OH)]_2O\}_2$.

that the pendant OH group does not participate in coordination. The presence of strong bands due to C–Cl at 768 and 809 cm^{-1} in the free acid and at 781 and 801 cm^{-1} in the complex imply that the two Cl atoms residing on the ring do not coordinate. The two values for $\nu(\mathrm{COO})_{\mathrm{asym}}$ and $\nu(\mathrm{COO})_{\mathrm{sym}}$ so obtained show the presence of two different chelating carboxylate groups, one monodentate and the other bidentate. For the bidentate carboxylate, the difference between $\nu(\mathrm{COO})_{\mathrm{asym}}$ and $\nu(\mathrm{COO})_{\mathrm{sym}}$ ($\Delta\nu$) is 201 cm^{-1}, comparable to that of the corresponding sodium salt ($\Delta\nu = 199$ cm^{-1}) [14]. $\Delta\nu$ for the monodentate carboxylate is 224 cm^{-1} which is higher than that of the salt [15].

^{1}H NMR spectral data for the free acid shows the COOH resonance at δ 13.00 ppm, which is absent in the spectrum of the complex, indicating the deprotonation and coordination of the carboxylate group. The signals observable at δ 0.79, 1.27 and 1.57 are ascribable to protons of the butyl group. Resonance of the aromatic protons appears as a singlet at δ 7.70 ppm and this does not change on complexation. The carbonyl carbon shifts downfield from 159.0 to 169.0 ppm upon coordination of the carboxylate group to the Sn atom.

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