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Molecular structure of 1,2;3,4,di-μ-nitrobenzoato-OO′-1,3-bis(*o*, *m*-, or *p*-nitrobenzoato)-1,2,4;2,3,4-di-μ_3-oxotetrakis [di-n-butyltin(IV)] compounds

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Abstract

Infrared, Mössbauer and multinuclei (^{1}H, ^{13}C, ^{119}Sn) NMR spectral data for the title compounds and single crystal X-ray data for the *ortho* isomer only reveal that these compounds contain two bridging and two non-bridging (monodentate) nitro-benzoato groups. These data further confirm the existence of two six-coordinate endocyclic and two five-coordinate exocyclic tin atoms in these dimers.

Introduction

A number of organodistannoxanes are known to have ‘ladder’ or ‘staircase’ structures [1,2]. Tetraorgano-diisothiocyanato-, diacetoxy-, or substituted-diacetoxy-distannoxanes are among those which form ladder dimers with central planar four membered Sn_2O_2 rings [3–6]. X-Ray diffraction data for the tri- or di-chloroacetoxystannoxanes have shown the presence of bridging and non-bridging (monodentate) carboxyl groups, corresponding to five and six coordination for the tin atoms in the dimers [3,5,6]. However, recent reports [7–9] have shown tetraorgano- di(amino)- or di(*o*-methoxy)-benzoato)distannoxanes dimers to contain mainly six-coordinate tin atoms.

In triphenyltin benzoates, both electronic and steric effects are known to influence the bonding and the functional substituents on benzoato rings play an important role in determining the structure. For example, triphenyltin(IV) *o*-chlorobenzoate has a *trans* R_3SnO_2 polymeric structure linked through carboxyl

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groups, rather than the monomeric tetrahedral structure observed in other cases [10]. We thus thought it worthwhile to investigate the structure of organodistannoxanes with various substituents on the benzoate groups, and the results obtained for the title compounds are discussed below.

Results and discussion

Azeotropic dehydration reactions between di-n-butyltin(IV) oxide and a nitrobenzoic acid (*o*-, *m*-, or *p*-) in the molar ratio 1 : 1 in benzene gives a white solid in each case. These solids are soluble in organic solvents and have the composition $[\{^nBu_2SnOC(O)\text{-}C_6H_4NO_2\}_2O]_2$.

$$4^nBu_2SnO + 4HOOCC_6H_4NO_2 \rightarrow [\{^nBu_2SnOC(O)C_6H_4NO_2\}_2O]_2 + 2H_2O$$

Spectral studies

The infrared spectra of the compounds in the solid state (KBr) and in solution (CH_2Cl_2) reveal $\nu(CO_2)$ bands at 1640–1630, 1420–1410, and 1370–1360 cm^{-1} assignable to bridging and non-bridging carboxyl groups. $\nu(NO_2)$ bands identified at 1550–1530 and 1350–1340 cm^{-1} remain unchanged after the reaction.

^{119m}Sn Mössbauer spectra of the compounds show chemical isomer shifts (IS) and quadrupole splittings (QS) at liquid nitrogen temperature of 1.36–1.41 and 3.44–3.51 mm sec^{-1}, respectively. The ratio ($\rho = QS/IS \sim 2.5$) reveals the coordination number of the tin atoms to be greater than four in these compounds [11]. Since the R–Sn–R bond angle is available (see below) for the *o*-nitrobenzoato isomer from single crystal data, the QS values can also be calculated [12]; the values obtained for endocyclic and exocyclic tin atoms are 3.36 and 3.20 mm sec^{-1}, respectively. The average value calculated here (3.28 mm sec^{-1}) and the observed value (3.44 mm sec^{-1}) differ by only 0.16 mm sec^{-1}, within the experimental error noted [12]. Consideration of the two values reveals that the exocyclic tin atom is five coordinate and endocyclic tin either five or six-coordinate.

The following NMR spectral data were obtained for the compounds: ^1H (for each organic moiety), 0.83–0.9 (t, 6H, CH_3), 1.26–1.73 (m, 12H, CH_2), 7.26–8.97 ppm (m, 4H, C_6H_4); ^{13}C, 13.40–13.55 (CH_3), 26.7, 27.2–27.5, 28.01–28.91 ($3CH_2$), 123.47–124.60, 129.61–130.68, 130.57–138.60 (C_6H_4), 148.26–150.17 (CNO_2), 170.52–170.95 ppm (CO_2); ^{119}Sn, −194.4, −197.9 (*ortho*), −204.3, −210 (*meta*) and −205, −210 ppm (*para*). The ^1H and ^{13}C NMR spectral data confirm the presence of the expected organic moieties in the compounds, and ^{119}Sn NMR signals reveal the presence of two non-equivalent tin atoms in each molecule. This feature is well documented for distannoxanes [3–8] in solutions. ^{119}Sn NMR chemical shift values may be used to give tentative indications of the environment around tin atoms. For di-n-butyltin carboxylates, Holeček *et al.* [13] suggested values between +200 and −60 for four-coordinate, −90 and −190 for five-coordinate and −210 and −400 ppm for six coordinate tin atoms in solution. The δ ^{119}Sn NMR values obtained in the present work thus suggest that there are either five- or six-coordinate tin atoms in the distannoxanes.

The structure of one of the title compounds is shown in Fig. 1. As is the case for other known carboxystannoxanes, the compound is a tetranuclear centrosymmetric dimer with a central four-membered ring defined by Sn(2)–O(2)–Sn(2)*–O(2)*.

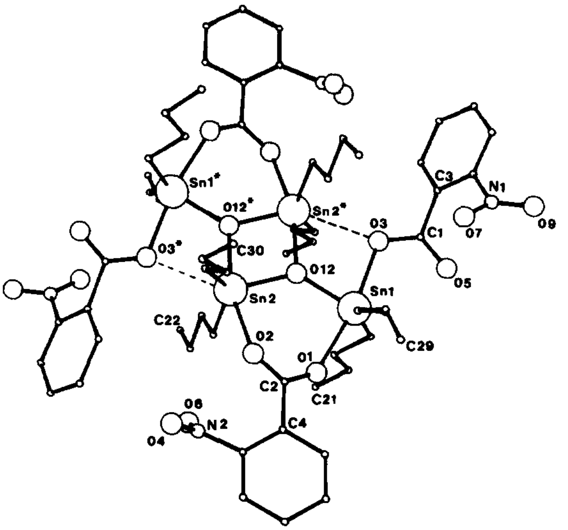

Fig. 1. Perspective diagram showing numbering scheme for 1,2;3,4-di-μ-nitrobenzoato-OO′-1,3-bis(*ortho*-nitrobenzoato)-1,2,4;2,3,4-di-μ_3-oxotetrakis[di-n-butyltin(IV)].

The four tin atoms are linked by two bridging carboxyl groups while the remaining two act as monodentate ligands to the exocyclic tin atoms. Consideration of the axial angles O(1)–Sn(1)–O(3) (168.4(1)°) and O(2)–Sn(2)–O(12)* (163.4(1)°) and the Sn–O distances lead to the conclusion that the two tin atoms are in different environments. Both tin atoms are coordinated by two n-butyl moieties which form C–Sn–C angles of 137.9(3)° and 133.2(2)° respectively. In the case of the exocyclic Sn(1) atom, coordination is completed by O(1), O(3) and O(12) with distances of 2.233(3), 2.209(3) and 2.035(3) Å respectively. For the endocyclic Sn(2) atom, the O(2), O(12) and O(12)* atoms all coordinate at distances of 2.312(3) and 2.053(3) Å, and there is a longer but significant, interaction of 2.798(6) Å to O(3)*. In the case of Sn(1), O(2) is 3.567 Å from the tin atom. However, O(5) is appreciably closer at 3.054(6) Å, but it appears that the interaction is very weak. This is supported by the values of the O–C–O bond angles of the bridging and nonbridging carboxyl groups, which are significantly different (120.7(4)° and 124.1(4)°). Thus it may be concluded that the exocyclic tin atom Sn(1) is best described as 5-coordinate, and the endocyclic Sn(2) as pseudo-6-coordinate.

A comparison between the structure of the title compounds and those of the (amino- or *o*-methoxybenzoato)distannoxanes published earlier [7–9] reveal that endocyclic tin atoms in both types of compounds are six-coordinate while the exocyclic tins are five-coordinate in the nitrobenzoato and 6-coordinate in the (amino- or (*o*-methoxy-)benzoato)stannoxanes. In the infrared spectra also, the lower $\nu(CO_2)$ absorption range for the aminobenzoato derivatives [1605–1600 cm^{-1}] compared with that for the nitrobenzoato derivatives [1640–1630 cm^{-1}] may reflect a difference between the coordination numbers of the tin atoms in these compounds. The existence of such 6-coordinate and 5-coordinate tins have also been shown by X-ray and IR data [5,6] in tetra n-butyl bis-(trichloroacetoxy-or-(dichloroacetoxy)-distannoxanes. A close scrutiny of the available structural data for diorganostannoxanes [4–8] reveal that the choice between five- and six-coordinated exocyclic tins in this type of compound may depend upon the electronic effects of the substituents in the carboxyl ligands. This is supported by similar observations on triphenyltin benzoates [10]. Interestingly the electronegative ligands influence the bonding characteristics of non-bridging but not those of the bridging carboxyl groups.

Experimental

Synthesis

A mixture of di-n-butyltin (IV) oxide (2.5 g, 10 mmole) and *o*-, *m*-, or *p*-nitrobenzoic acid (1.67 g, 10 mmol) in benzene (50 cm^3) was heated under reflux for 4 h in a Dean-Stark azeotropic distillation apparatus with continuous removal of water liberated. The solvent was evaporated and the residue extracted with light petroleum ether (b.p. 40–60°). The crude product was recrystallised from 1/1 acetonitrile-diethyl ether.

(i) *o*-Nitrobenzoato compound (90% yield), m.p. 130°C (Found: C 44.3; H, 5.5; N, 3.2; Sn, 29.8); (ii) *m*-nitrobenzoato compound (85% yield), m.p. 150°C (Found: C, 44.1; H, 5.5; N, 3.0; Sn, 28.6%); (iii) *p*-nitrobenzoato compound (yield 88%), m.p. 181°C (Found: C, 44.0; H, 5.4; N, 3.1; Sn, 28.8%. $C_{60}H_{88}Sn_4O_{18}N_4$ calc.: C, 44.2; H, 5.4; N, 3.4; Sn, 29.2%). M.p.'s are corrected to within ±1°C.

Spectroscopy

FT-IR spectra were recorded in KBr pellets and CH_2Cl_2 solvent on a Perkin Elmer Model 1850 spectrometer. ^{119m}Sn Mössbauer spectra were recorded at 78 K using a constant acceleration drive coupled with a multichannel analyser. The source was $Ba^{119m}SnO_3$ at room temperature, and isomer shifts are reported with respect to this source. Observed spectra were fitted by least squares using a Lorentzian line shape. ^{1}H, ^{13}C, ^{119}Sn NMR spectra were recorded with $CDCl_3$ solutions on a Brüker (AM 500) 500 MHz instrument at the National Facility Centre at Tata Institute of Fundamental Research, Bombay. The chemical shifts δ ^{13}C were determined by reference to a suitable signal of the solvent δ ($CDCl_3$ = 77.00 ppm). The values of δ ^{1}H and δ ^{119}Sn are relative to Me_4Si and Me_4Sn respectively. Elemental analyses were obtained with a Carlo Erba Elemental Analyser Model 1106.

Crystal structure determination

The structure was solved by the heavy atom method, the correct choice of space group being determined by the N(z) test. A Fourier map determined the positions of all the remaining non-hydrogen atoms. Full matrix anisotropic refinement of all non hydrogen atoms with the hydrogen atoms fixed at calculated positions converged at $R = 0.043$, $R_W = 0.065$, with a weighting scheme of $w = 1/[\sigma(F^2) + (0.05F)^2 + 5.0]$ with the esd of an observation of unit weight = 1.121.

All calculations were performed on a DEC PDP 11/73 computer using SDP crystallographic programmes.

Bond lengths and bond angles are given in Table 1, and atomic coordinates with anisotropic temperature factors in Table 2. Crystal data for $C_{30}H_{44}N_2O_9Sn_2$; $M = 814.06$, space group $P\bar{1}$, $a = 12.097(9)$, $b = 12.384(8)$, $c = 13.150(9)$ Å, $\alpha =$

Table 1

Bond lengths (Å) and angles (deg.) in $[^{n}Bu_2SnOC(O)C_6H_4NO_2\text{-}o]_2O$

Sn(1)–Sn(2)	3.760(0)	O(6)–N(2)	1.210(5)	C(12)–C(14)	1.396(8)
Sn(1)–Sn(2)*	3.644(0)	O(7)–N(1)	1.198(8)	C(15)–C(17)	1.523(7)
Sn(2)–Sn(2)*	3.313(0)	O(9)–N(1)	1.231(7)	C(16)–C(18)	1.490(6)
Sn(1)–O(1)	2.233(3)	N(1)–C(13)	1.472(6)	C(17)–C(19)	1.517(8)
Sn(1)–O(3)	2.209(3)	N(2)–C(14)	1.464(8)	C(18)–C(20)	1.517(7)
Sn(1)–O(12)	2.053(3)	C(1)–C(3)	1.521(6)	C(19)–C(21)	1.473(12)
Sn(1)–C(15)	2.121(5)	C(2)–C(4)	1.502(6)	C(20)–C(22)	1.505(8)
Sn(1)–C(23)	2.110(4)	C(3)–C(5)	1.367(6)	C(23)–C(25)	1.404(12)
Sn(2)–O(2)	2.312(3)	C(3)–C(13)	1.381(6)	C(24)–C(26)	1.333(9)
Sn(2)–O(12)	2.053(3)	C(4)–C(6)	1.403(7)	C(25)–C(27)	1.500(12)
Sn(2)–O(12)*	2.149(2)	C(4)–C(14)	1.376(8)	C(26)–C(28)	1.472(12)
Sn(2)–C(16)	2.127(4)	C(5)–C(7)	1.410(8)	C(27)–C(29)	1.00(2)
Sn(2)–C(24)	2.111(6)	C(6)–C(8)	1.374(8)	C(28)–C(30)	1.33(2)
O(1)–C(2)	1.247(6)	C(7)–C(9)	1.366(8)		
O(2)–C(2)	1.241(6)	C(8)–C(10)	1.365(11)		
O(3)–C(1)	1.274(6)	C(9)–C(11)	1.366(7)		
O(4)–N(2)	1.221(8)	C(10)–C(12)	1.363(11)		
O(5)–C(1)	1.224(5)	C(11)–C(13)	1.362(7)		
Sn(2)*–Sn(1)–Sn(2)	53.14(1)	C(16)–Sn(2)–C(24)	137.9(3)	C(7)–C(9)–C(11)	121.8(5)
Sn(1)*–Sn(2)–Sn(1)	126.86(1)	O(7)–N(1)–O(9)	124.7(4)	C(8)–C(10)–C(12)	119.8(6)
Sn(1)–Sn(2)–Sn(2)*	61.64(1)	O(7)–N(1)–C(13)	118.3(5)	C(9)–C(11)–C(13)	118.2(4)
Sn(1)*–Sn(2)–Sn(2)*	65.22(1)	O(9)–N(1)–C(13)	116.8(5)	C(10)–C(12)–C(14)	119.2(7)
O(1)–Sn(1)–O(3)	168.4(1)	O(4)–N(2)–O(6)	124.1(6)	N(1)–C(13)–C(3)	119.7(4)
O(1)–Sn(1)–O(12)	89.9(1)	O(4)–N(2)–C(14)	117.0(4)	N(1)–C(13)–C(11)	117.1(4)
O(1)–Sn(1)–C(15)	86.4(2)	O(6)–N(2)–C(14)	118.9(5)	C(3)–C(13)–C(11)	122.9(4)
O(1)–Sn(1)–C(23)	91.3(2)	O(3)–C(1)–O(5)	124.1(4)	N(2)–C(14)–C(4)	116.7(6)
O(3)–Sn(1)–O(12)	78.7(1)	O(3)–C(1)–C(3)	116.1(4)	N(2)–C(14)–C(12)	116.7(6)
O(3)–Sn(1)–C(15)	96.2(2)	O(5)–C(1)–C(3)	119.8(5)	C(4)–C(14)–C(12)	122.0(6)
O(3)–Sn(1)–C(23)	95.1(2)	O(1)–C(2)–O(2)	120.7(4)	C(15)–C(17)–C(19)	113.1(6)
O(12)–Sn(1)–C(15)	114.2(1)	O(1)–C(2)–C(4)	116.6(4)	C(16)–C(18)–C(20)	113.6(4)
O(12)–Sn(1)–C(23)	112.5(2)	O(2)–C(2)–C(4)	118.7(5)	C(17)–C(19)–C(21)	114.2(7)
C(15)–Sn(1)–C(23)	133.2(2)	C(1)–C(3)–C(5)	120.2(4)	C(18)–C(20)–C(22)	113.9(5)
O(2)–Sn(2)–O(12)	88.8(1)	C(1)–C(3)–C(13)	121.9(4)	C(23)–C(25)–C(27)	113.6(9)
O(2)–Sn(2)–O(12)	163.4(1)	C(5)–C(3)–C(13)	117.8(4)	C(24)–C(26)–C(28)	123.0(9)
O(2)–Sn(2)–C(16)	85.5(1)	C(2)–C(4)–C(6)	118.6(5)	C(25)–C(27)–C(29)	136.0(2)
O(2)–Sn(2)–C(24)	82.7(2)	C(2)–C(4)–C(14)	123.9(4)	C(26)–C(28)–C(30)	114.0(1)
O(12)–Sn(2)–O(12)*	75.9(1)	C(6)–C(4)–C(14)	117.3(4)	Sn(1)–O(12)–Sn(2)	133.7(1)
O(12)–Sn(2)–C(16)	107.4(2)	C(3)–C(5)–C(7)	120.7(4)	Sn(1)–O(12)–Sn(2)*	121.1(2)
O(12)–Sn(2)–C(24)	112.6(2)	C(4)–C(6)–C(8)	120.0(6)	Sn(2)–O(12)–Sn(2)*	104.1(1)
O(12)–Sn(2)–C(16)	105.1(1)	C(5)–C(7)–C(9)	118.5(5)		
O(12)–Sn(2)–C(24)	96.9(2)	C(6)–C(8)–C(10)	121.5(6)		

Symmetry code: $\star = -x, -y, -z$.

72.55(4)°, $\beta = 83.26(4)$, $\gamma = 67.64(3)°$, $V = 1738.3(2)$ Å^3, $Z = 2$, $F(000) = 820.0$, $D_c = 1.65$ g cm^{-3}, μ(Mo-K_α) = 15.0 cm^{-1}, $\lambda = 0.71069$ Å and $T = 298$ K.

Data collection and processing

Intensity data were collected using graphite monochromated Mo-K_α radiation ($\lambda = 0.71069$ Å) on an Enraf Nonius CAD$_4$ four circle diffractometer in $\omega/2\theta$ scan mode. The scan width = $0.7 + 0.35 \tan\theta$ with a scan speed 3.33 min^{-1} to a

Table 2

Positional parameters and their estimated standard deviations

Atom	x	y	z	B (Å^2)
Sn(1)	0.77729(2)	0.49512(2)	0.47135(2)	3.975(6)
Sn(2)	0.51949(2)	0.42744(2)	0.40818(2)	3.636(6)
O(1)	0.8025(3)	0.4414(3)	0.3202(2)	5.12(7)
O(2)	0.6982(3)	0.3251(3)	0.3356(2)	4.85(7)
O(3)	0.7145(3)	0.5521(3)	0.6184(2)	4.88(7)
O(4)	0.6951(5)	0.1574(4)	0.1784(5)	11.1(2)
O(5)	0.8932(3)	0.5567(3)	0.6295(3)	6.08(8)
O(6)	0.6387(4)	0.3517(4)	0.1199(3)	8.1(1)
O(7)	0.9494(4)	0.3328(3)	0.8114(3)	7.7(1)
O(9)	1.0487(4)	0.4056(4)	0.8800(3)	7.9(1)
O(12)	0.6124(2)	0.4834(2)	0.4896(2)	3.85(6)
N(1)	0.9559(4)	0.4080(4)	0.8480(3)	5.9(1)
N(2)	0.7144(4)	0.2519(4)	0.1527(3)	6.7(1)
C(1)	0.7954(4)	0.5585(4)	0.6677(3)	4.48(9)
C(2)	0.7822(4)	0.3594(3)	0.2984(3)	4.06(9)
C(3)	0.7642(4)	0.5709(4)	0.7801(3)	4.71(9)
C(4)	0.8673(4)	0.2983(4)	0.2223(3)	4.5(1)
C(5)	0.6573(4)	0.6536(5)	0.8003(4)	5.7(1)
C(6)	0.9860(4)	0.2917(4)	0.2183(4)	5.5(1)
C(7)	0.6314(5)	0.6730(5)	0.9021(4)	6.9(1)
C(8)	1.0673(5)	0.2320(6)	0.1527(5)	6.8(2)
C(9)	0.7154(5)	0.6069(5)	0.9803(4)	7.0(1)
C(10)	1.0354(6)	0.1790(5)	0.0897(5)	7.5(2)
C(11)	0.8221(5)	0.5223(5)	0.9624(4)	6.1(1)
C(12)	0.9214(6)	0.1815(5)	0.0935(5)	7.6(2)
C(13)	0.8446(4)	0.5063(4)	0.8629(3)	5.1(1)
C(14)	0.8380(4)	0.2414(4)	0.1601(4)	5.4(1)
C(15)	0.7799(4)	0.6656(4)	0.3751(4)	5.2(1)
C(16)	0.4833(4)	0.5520(4)	0.2534(3)	4.5(1)
C(17)	0.6998(5)	0.7258(4)	0.2772(4)	5.7(1)
C(18)	0.3773(5)	0.6670(4)	0.2408(4)	5.3(1)
C(19)	0.7015(6)	0.8504(5)	0.2165(6)	8.1(2)
C(20)	0.3582(6)	0.7483(5)	0.1272(4)	6.7(2)
C(21)	0.6250(9)	0.9103(7)	0.1213(7)	11.4(3)
C(22)	0.2523(7)	0.8657(6)	0.1141(6)	8.5(2)
C(23)	0.9123(5)	0.3304(5)	0.5473(4)	6.3(1)
C(24)	0.5246(5)	0.2494(5)	0.4848(6)	7.3(2)
C(25)	0.9026(8)	0.2184(7)	0.5566(7)	11.3(3)
C(26)	0.6188(8)	0.1528(7)	0.5299(9)	13.1(3)
C(27)	1.003(1)	0.1110(8)	0.6182(9)	15.9(4)
C(28)	0.6151(8)	0.0299(7)	0.5735(9)	10.9(3)
C(29)	1.082(2)	0.054(1)	0.600(2)	27.4(8)
C(30)	0.691(1)	−0.041(1)	0.653(1)	23.6(6)

maximum of 90 secs. for weak reflections ($h \pm k \pm l$, $1 < \theta < 26°$) yielding, after averaging and rejection of systematic absences, 6121 observed reflections of which 5309 had $I > 3\sigma(I)$. During the data collection a standard reflection indicated a decay of about 5% in the crystal and a correction was made for this.

A list of structure factors is available from D.C.P.

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