

## Synthesis and structures of some diethyl- and diphenyltin bishydroxamates

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### Abstract

Diethyl- and diphenyltin derivatives of *N*-methyl-*N*-*p*-bromobenzoylhydroxylamine,  $R_2Sn(ON(Me)C(O)C_6H_4Br-p)_2$ ,  $R = Et, Ph$ , and *N*-*p*-bromophenyl-*N*-*p*-bromobenzoylhydroxylamine,  $R_2Sn(ON(C_6H_4Br-p)C(O)C_6H_4Br-p)_2$ ,  $R = Et, Ph$ , were made and characterized by IR, Mössbauer and NMR ( $^1H$ ,  $^{13}C$ ,  $^{119}Sn$ ) spectroscopy and, in the case of  $Et_2Sn(ON(Me)C(O)C_6H_4Br-p)_2$ , by an X-ray diffraction study. The geometry at the tin atom is that of a distorted *trans* octahedron with two bidentate, nearly equivalent, *N*-acylhydroxylamine residues and a CSnC angle of 141.1°. On the basis of the Mössbauer data for the diphenyltin hydroxamates, a *cis*-octahedral structure with bidentate ligation of the hydroxylamine residues is suggested.

**Keywords:** Tin; Crystal structure; Hydroxamates; Mössbauer spectroscopy

### 1. Introduction

The synthesis, physicochemical properties and structures of organotin hydroxamates have been the subject of numerous studies [1–9]. As a rule, the hydroxamic ligand is bidentate as a result of carbonyl coordination to the tin atom, which substantially lowers the infrared carbonyl stretching frequencies relative to that for the free hydroxylamines [1–5]. A firmer indication of the structure of an organotin hydroxamate can sometimes be made on the basis of correlation of  $^{119}Sn$  Mössbauer parameters with the relevant crystal structure data [1,2] but only a few X-ray structure determinations have been carried out on organotin *N*-acylhydroxylamine derivatives [6–9].

In a previous paper [10], we described the synthesis, spectroscopic properties and structures of some dimethyl- and di-*n*-butyltin bishydroxamates and the results of X-ray diffraction studies for two of them. We report here the results of a structural study of analogous diethyl- and diphenyltin derivatives of *N*-methyl-*N*-*p*-bromobenzoylhydroxylamine, namely  $R_2Sn(ON(Me)$

$C(O)C_6H_4Br-p)_2$ ,  $R = Et$  (1),  $Ph$  (2), and *N*-*p*-bromophenyl-*N*-*p*-bromobenzoylhydroxylamine,  $R_2Sn(ON(C_6H_4Br-p)C(O)C_6H_4Br-p)_2$ ,  $R = Et$  (3),  $Ph$  (4). The hydroxamates 1–4 have been characterized by IR, Mössbauer and NMR ( $^1H$ ,  $^{13}C$ ,  $^{119}Sn$ ) spectroscopy, and in the case of (1) by an X-ray diffraction study.

### 2. Experimental section

#### 2.1. Synthesis

The oxides  $R_2SnO$  with  $R = Et, Ph$  were prepared by alkaline hydrolysis of  $R_2SnCl_2$ . *N*-Methyl-*N*-*p*-bromobenzoylhydroxylamine and *N*-*p*-bromophenyl-*N*-*p*-bromobenzoylhydroxylamine were obtained by reaction of the appropriate acyl chloride with hydroxylamine hydrochloride in the presence of  $Na_2CO_3$  [11].

The diorganotin bishydroxamates were made by treatment of the  $R_2SnO$  with an appropriate *N*-acylhydroxylamine in the presence of an excess of 2,2-dimethoxypropane in  $CHCl_3$  [10]. The yields were 85–90%. Elemental analysis data are shown in Table 1 together with melting points.

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Table 1  
Elemental analyses and melting points of the diorganotin bishydroxamates

No.	Compound	M.p. (°C)	Analysis (%)					
			C		H		N	
			Calc.	Found	Calc.	Found	Calc.	Found
1	Et <sub>2</sub> Sn(ON(Me)C(O)C <sub>6</sub> H <sub>4</sub> Br-p) <sub>2</sub>	165–166	37.8	38.1	3.8	3.9	4.4	4.2
2	Ph <sub>2</sub> Sn(ON(Me)C(O)C <sub>6</sub> H <sub>4</sub> Br-p) <sub>2</sub>	> 150 <sup>a</sup>	46.0	46.0	3.3	3.3	3.8	3.8
3	Et <sub>2</sub> Sn(ON(C <sub>6</sub> H <sub>4</sub> Br-p)C(O)C <sub>6</sub> H <sub>4</sub> Br-p) <sub>2</sub>	193–194	39.3	39.3	2.8	2.7	3.1	3.1
4	Ph <sub>2</sub> Sn(ON(C <sub>6</sub> H <sub>4</sub> Br-p)C(O)C <sub>6</sub> H <sub>4</sub> Br-p) <sub>2</sub>	> 150 <sup>a</sup>	45.0	45.1	2.6	2.6	2.8	3.0

<sup>a</sup> Decomposition.

## 2.2. Crystal structure determination

Single crystals of Et<sub>2</sub>Sn(ON(Me)C(O)C<sub>6</sub>H<sub>4</sub>Br-p)<sub>2</sub> (**1**) were obtained by recrystallization from CHCl<sub>3</sub> after addition of light petroleum.

X-ray diffraction data for a colourless crystal (0.3 mm × 0.3 mm × 0.2 mm) were collected at room temperature on a four-circle diffractometer. Crystal data and experimental details are given in Table 2. The

Table 2  
Experimental data for the crystal structure determination of **1**

Crystal data	
Formula	C <sub>20</sub> H <sub>30</sub> Br <sub>2</sub> N <sub>2</sub> O <sub>4</sub> Sn
Formula weight	640.97
Absorption	$\mu$ (cm <sup>-1</sup> ) = 45.0, empirical correction ( $\Psi$ -scans)
Space group	<i>P</i> 2 <sub>1</sub> / <i>n</i> , <i>Z</i> = 4
Lattice constants	<i>a</i> = 11.121(3) Å
(Mo K $\alpha$ )	<i>b</i> = 17.322(3) Å
	<i>c</i> = 12.663(3) Å
	$\beta$ = 105.04(3)°
Temperature	293 K
Density	$d_c$ = 1.807 g cm <sup>-3</sup>
Data collection	
Diffractometer	Four-circle, CAD4 (Enraf–Nonius)
Radiation	Mo K $\alpha$ , graphite monochromator
Scan type	$\omega$ -Scans
Scan width	(0.85 + 0.6 tan $\theta$ )° and 25% on the left-hand and right-hand sides of a reflection for background determination
Measuring range	$\theta$ = 2°–31°, + <i>h</i> , + <i>k</i> , ± <i>l</i>
Reflections	7461 total, 7135 unique
Computing	
Programs	SHELXS-86[14], SHELXL-93[15]
Solution	Patterson and difference Fourier methods
Atomic scattering factors for neutral atoms	$\Delta f'$ , $\Delta f''$ from [16]
Refinement	Full matrix least squares on $F^2$ , 335 parameters
Extinction correction	$\epsilon$ = 0.0008(2), after [15]
Residuals	$wR_2$ = 0.147 for all reflections $R$ = 0.053 for $F_o > 4\sigma(F)$ (for 2580 reflections)
$\Delta\rho_{\max,\min}$	0.91, -0.95 e Å <sup>-3</sup>

structure was solved by Patterson and difference Fourier methods. All hydrogen atoms were located and refined with common isotropic temperature factors for those in the methyl groups and separately for those in the methylene fragments. For all heavier atoms anisotropic temperature factors were used. Full matrix least-squares refinement for the  $F^2$  data led to final  $\omega R_2$  = 0.147 for all reflections, corresponding to a conventional  $R$  of 0.053 for the  $F_o$  data with  $F_o > 4\sigma(F)$ . Fractional atomic coordinates for **1** are given in Table 3.

IR spectra were recorded for KBr discs with a Bruker IR spectrometer. The <sup>1</sup>H NMR, {<sup>1</sup>H} <sup>13</sup>C and <sup>119</sup>Sn

Table 3  
Atomic fractional coordinates and equivalent isotropic temperature factors (10<sup>-20</sup> m<sup>2</sup> = Å<sup>2</sup>) for **1**

Atom	<i>x</i>	<i>y</i>	<i>z</i>	$U_{eq}$ <sup>a</sup>
Sn(1)	0.04699(5)	0.21241(3)	0.14983(4)	0.0456(2)
Br(1)	0.03226(12)	0.49263(6)	0.75563(8)	0.0969(4)
Br(2)	0.17529(10)	0.04817(7)	-0.49905(7)	0.0903(4)
O(1)	0.0888(5)	0.2959(3)	0.3038(4)	0.0629(15)
C(1)	0.0145(7)	0.2838(4)	0.3603(5)	0.045(2)
N(1)	-0.0676(6)	0.2277(3)	0.3373(5)	0.050(2)
O(10)	-0.0738(5)	0.1820(3)	0.2462(4)	0.0564(14)
C(11)	0.0179(6)	0.3355(4)	0.4572(5)	0.039(2)
C(12)	-0.0876(7)	0.3752(5)	0.4632(6)	0.051(2)
C(13)	-0.0812(8)	0.4236(5)	0.5535(7)	0.057(2)
C(14)	0.0279(8)	0.4294(4)	0.6322(6)	0.051(2)
C(15)	0.1341(8)	0.3928(5)	0.6242(6)	0.055(2)
C(16)	0.1288(7)	0.3443(4)	0.5369(7)	0.048(2)
O(2)	0.1189(5)	0.1849(3)	-0.0104(4)	0.0613(15)
C(2)	0.0475(7)	0.1378(4)	-0.0723(6)	0.047(2)
N(2)	-0.0465(6)	0.1076(3)	-0.0419(5)	0.0472(15)
O(20)	-0.0617(5)	0.1229(3)	0.0606(4)	0.0568(14)
C(21)	0.0714(6)	0.1160(4)	-0.1782(5)	0.039(2)
C(22)	0.0806(8)	0.0382(4)	-0.2068(6)	0.050(2)
C(23)	0.1103(8)	0.0181(5)	-0.3001(6)	0.056(2)
C(24)	0.1333(7)	0.0747(5)	-0.3676(6)	0.052(2)
C(25)	0.1299(8)	0.1523(5)	-0.3410(6)	0.061(2)
C(26)	0.0979(7)	0.1718(4)	-0.2462(6)	0.051(2)
C(3)	0.2183(9)	0.1618(6)	0.2315(7)	0.065(3)
C(4)	0.3316(9)	0.2115(6)	0.2385(9)	0.074(3)
C(5)	-0.0268(11)	0.3135(6)	0.0631(9)	0.079(3)
C(6)	0.0666(18)	0.3706(8)	0.0486(17)	0.123(7)
C(10)	-0.1459(9)	0.1981(6)	0.4039(8)	0.062(2)
C(20)	-0.1498(8)	0.0635(6)	-0.1082(8)	0.060(2)

<sup>a</sup>  $U_{eq}$  is calculated as 1/3 of the trace of the orthogonalized  $U_{ij}$  tensor.

Table 4  
Selected bond distances and angles in structure 1

Atoms	<i>d</i> (Å)	Atoms	$\omega$ (°)	Atoms	$\omega$ (°)
Sn–O(1)	2.375(5)	O(1)Sn(1)O(2)	140.7(2)	Sn(1)O(1)C(1)	110.6(4)
Sn–O(2)	2.413(5)	O(1)Sn(1)O(10)	72.2(2)	Sn(1)O(2)C(2)	111.0(4)
Sn–O(10)	2.102(5)	O(1)Sn(1)O(20)	148.4(2)	Sn(1)O(10)N(1)	116.1(4)
Sn–O(20)	2.106(5)	O(1)Sn(1)C(3)	83.8(3)	Sn(1)O(20)N(2)	117.0(4)
Sn–C(3)	2.109(10)	O(1)Sn(1)C(5)	83.6(3)	O(1)C(1)N(1)	121.7(6)
Sn–C(5)	2.117(10)	O(2)Sn(1)O(10)	147.1(2)	O(1)C(1)C(11)	119.2(6)
O(1)–C(1)	1.242(8)	O(2)Sn(1)O(20)	70.9(2)	O(2)C(2)N(2)	119.8(6)
O(2)–C(2)	1.259(8)	O(2)Sn(1)C(3)	82.9(3)	O(2)C(2)C(21)	119.7(6)
O(10)–N(1)	1.386(7)	O(2)Sn(1)C(5)	84.0(3)	O(10)N(1)C(1)	119.2(6)
O(20)–N(2)	1.377(7)	O(10)Sn(1)O(20)	76.3(2)	O(10)N(1)C(10)	112.0(6)
N(1)–C(1)	1.314(8)	O(10)Sn(1)C(3)	104.1(3)	O(20)N(2)C(2)	120.0(6)
N(1)–C(10)	1.453(10)	O(10)Sn(1)C(5)	106.8(3)	O(20)N(2)C(20)	112.0(6)
N(2)–C(2)	1.313(8)	O(20)Sn(1)C(3)	106.1(3)	N(1)C(1)C(11)	119.2(6)
N(2)–C(20)	1.452(9)	O(20)Sn(1)C(5)	103.9(4)	N(2)C(2)C(21)	120.4(6)
C(1)–C(11)	1.511(9)	C(3)Sn(1)C(5)	141.1(4)	C(1)N(1)C(10)	128.2(6)
C(2)–C(21)	1.483(9)			C(2)N(2)C(20)	127.6(6)

NMR spectra were obtained with a Varian VXR-400 spectrometer operated at 400, 100 and 149.2 MHz, respectively.

The  $^{119}\text{Sn}$  Mössbauer spectra were obtained at about 80 K with a conventional constant-acceleration spectrometer operated in the time mode using a  $\gamma$ -ray source of 5 mCi  $^{119}\text{Sn}$  in  $\text{BaSnO}_3$  at 80 K. The velocity of the transducer was calibrated by the conventional method involving the Mössbauer spectrum of metallic iron; the peak positions and line widths were determined in the usual way, by fitting the experimental data to Lorentzian lines.

### 3. Results and discussion

The structure and atomic numbering scheme for **1** are depicted in Fig. 1 and selected bond distances and angles are reported in Table 4. The geometry around the tin atom is similar to that in  $^n\text{Bu}_2\text{Sn}\{\text{ON}(\text{Ph})\text{C}(\text{O})\text{P}\}_2$  and  $^n\text{Bu}_2\text{Sn}\{\text{ON}(\text{Me})\text{C}(\text{O})\text{C}_6\text{H}_4\text{Br-}p\}_2$  [10] and some other diorganotin bishydroxamates [6,7]. In the structure the two *N*-acylhydroxylamine residues are almost equivalent and function as bidentate ligands, forming one short covalent and one longer coordinate oxygen–tin bond (Table 4). The two heterocyclic rings are almost

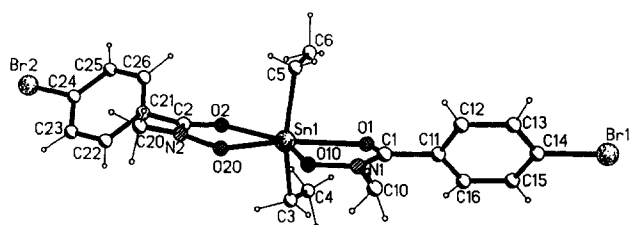


Fig. 1. Structure and atomic numbering scheme for  $\text{Et}_2\text{Sn}\{\text{ON}(\text{Me})\text{C}(\text{O})\text{C}_6\text{H}_4\text{Br-}p\}_2$ .

planar, the deviation of the tin atom from these planes being 0.01 Å. The phenyl rings are rotated by 49.3° and 55.8° relative to the planes of the *N*-acylhydroxylamine residues. The CSnC linkage is not linear, having an angle of 141.1°: this geometry at the tin atom is usually described as a distorted *trans*-octahedron or a bicapped tetrahedron [10].

As in other organotin hydroxamates [6–10], the bond lengths in the *N*-acylhydroxylamine groups are consistent with a significant contribution of the zwitterionic canonical form to the electronic distribution, the C=O distance being longer and the endocyclic C–N distance shorter than normal double bond C=O and single bond C–N distance, respectively [6].

The  $^{119}\text{Sn}$  Mössbauer parameters (isomeric shift  $\Delta IS$  and quadrupole splitting  $\Delta QS$ ) of the synthesized hydroxamates are given in Table 5. The experimental  $QS$  values for compounds **1** and **3** are similar, and close to that found for a number of other diorganotin bishydroxamates with distorted *trans*-octahedral structures [1,2,10]. They are in good agreement with a value of  $\Delta QS = 3.09 \text{ mm s}^{-1}$  calculated in terms of the point-charge model formalism using published values for the partial quadrupole splittings of the alkyl and ligand groups and real angle values determined from the X-ray study of  $^n\text{Bu}_2\text{S}\{\text{ON}(\text{Me})\text{C}(\text{O})\text{C}_6\text{H}_4\text{Br-}p\}_2$  [10]. Thus,

Table 5

Mössbauer parameters and infrared carbonyl stretching frequencies for diorganotin bishydroxamates

Compound	$IS^a$ ( $\text{mm s}^{-1}$ )	$QS^b$ ( $\text{mm s}^{-1}$ )	$\nu(\text{CO})$ ( $\text{cm}^{-1}$ )
<b>1</b>	1.28	3.39	1590
<b>2</b>	0.73	1.66	1587
<b>3</b>	1.27	3.30	1543
<b>4</b>	0.78	1.87	1528

<sup>a</sup>  $\pm 0.004 \text{ mm s}^{-1}$ .

<sup>b</sup>  $\pm 0.004 \text{ mm s}^{-1}$ .

Table 6

<sup>1</sup>H, <sup>13</sup>C and <sup>119</sup>Sn NMR spectroscopic data for diorganotin bishydroxamates <sup>a</sup>

$$R_2Sn(ON(R'')^1C(O)R')_2, R = ^2CH_2^3CH_3,$$

R	Et	Et	Ph	Ph
R'	C <sub>6</sub> H <sub>4</sub> Br- <i>p</i>	C <sub>6</sub> H <sub>4</sub> Br- <i>p</i>	C <sub>6</sub> H <sub>4</sub> Br- <i>p</i>	C <sub>6</sub> H <sub>4</sub> Br- <i>p</i>
R''	Me	C <sub>6</sub> H <sub>4</sub> Br- <i>p</i>	Me	C <sub>6</sub> H <sub>4</sub> Br- <i>p</i>
δ <sup>2</sup> CH <sub>2</sub>	1.43q	1.58q	–	–
δ <sup>3</sup> CH <sub>3</sub> or δ <sup>3</sup> CH	1.26t	1.40t	7.74m	7.74m
δ <sup>4</sup> CH and δ <sup>5</sup> CH	–	–	7.35m	7.32m
δR'	7.41m	7.25m (7.29m)	7.39m	7.13m (7.19m)
δR''	3.42s	7.29m (7.25m)	3.49s	7.19m (7.13m)
δ <sup>1</sup> C	162.9	163.3	162.1	163.3
δ <sup>2</sup> C	19.4	19.1	147.9	147.2
<sup>1</sup> J or <i>ipso</i> J( <sup>13</sup> C– <sup>119</sup> Sn)	765	730	925	919
δ <sup>3</sup> C	9.6	9.6	135.5	135.7
<sup>2</sup> J or <i>ortho</i> J( <sup>13</sup> C– <sup>117/119</sup> Sn)	–	–	54	54
δ <sup>4</sup> C	–	–	128.0	128.1
<i>meta</i> J( <sup>13</sup> C– <sup>117/119</sup> Sn)	–	–	78	80
δ <sup>5</sup> C	–	–	128.5	128.8
<i>para</i> J( <sup>13</sup> C– <sup>117/119</sup> Sn)	–	–	16	16
δR'	131.9; 131.7; 129.3; 124.8	140.4; 132.1; 131.5; 131.0; 130.2; 127.8;	131.8; 131.1; 129.5; 125.1	140.0; 132.2; 131.5; 130.4; 129.7; 127.6;
δR''	41.0	125.2; 121.7	40.9	125.5; 122.3
δ <sup>119</sup> Sn	–240	–227	–361	–354

<sup>a</sup> Abbreviations: s = singlet; t = triplet; q = quadruplet; m = complex pattern. *p*-Substituted aryls all provide a <sup>1</sup>H NMR AA'XX' pattern, described here as multiplets.

it is logical on the basis of the Mössbauer data to ascribe a distorted octahedral structure, with ethyl groups *trans* to each other, also to the hydroxamate **3**.

The diphenyltin derivatives **2** and **4** show substantially lower Δ*QS* values, viz. 1.66 and 1.87 mm s<sup>–1</sup>). Usually such low Δ*QS* values can be associated with a tetrahedral structure for such compounds, but for **2** and **4** the infrared data make this unlikely, since they are similar to those obtained for the *trans*-octahedral chelate diethyl- and dibutyltin analogues [10]. Thus we suggest for both **2** and **4** the *cis*-octahedral structure shown in Fig. 2, as suggested for some other diphenyltin bishydroxamates on the basis of their Mössbauer parameters (Δ*QS* = 1.61–1.95 mm s<sup>–1</sup> [1,2]). In the IR spectra of the solids discussed here, the carbonyl stretching vibration appears at lower frequencies (Table 5) than those in the spectra of the free hydroxylamines, HON(Me)C(O)C<sub>6</sub>H<sub>4</sub>Br-*p* (1610 cm<sup>–1</sup>) and HON(C<sub>6</sub>H<sub>4</sub>Br-*p*)C(O)-

C<sub>6</sub>H<sub>4</sub>Br-*p* (1602 cm<sup>–1</sup>), confirming again the bidentate mode of bonding of the hydroxylamine ligands in metal-organic hydroxamates [1–5,10].

The <sup>1</sup>H, {<sup>1</sup>H} <sup>13</sup>C and <sup>119</sup>Sn NMR spectroscopic parameters for the compounds are listed in Table 6. The <sup>1</sup>H NMR spectra exhibit the expected proton signals with correct integrals. On the basis of the established correlation of the spin–spin coupling constant <sup>1</sup>J(<sup>13</sup>C–<sup>119</sup>Sn) with the C–Sn–C angle [12], we suggest that upon dissolution of the diethyltin bishydroxamates in CDCl<sub>3</sub> chelated distorted *trans*-octahedral structure is retained. The C–Sn–C angles, estimated on the basis of Lockhart's equation, are 144° and 141°, respectively.

The δ<sup>119</sup>Sn data (Table 6) for **1–4** are typical for six-coordinate diorganotin compounds [13], and confirm the chelated structures of the bishydroxamates in CDCl<sub>3</sub> solution.

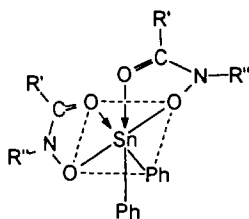


Fig. 2. *cis*-Octahedral structure of the diphenyltin derivatives **2** and **4**.

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