

Tetrabutylbis(*N*-phthaloylglycinato)-  
distannoxane dimer<sup>1</sup>Masood Parvez,<sup>a\*</sup> Saeed Anwar,<sup>b</sup> Amin Badshah,<sup>b</sup> Bashir  
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Received 30 June 1999

Accepted 22 October 1999

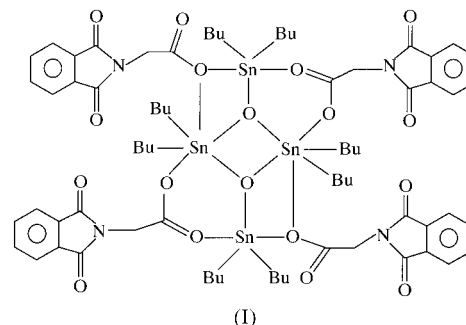
The crystal structure of the title compound, [Sn<sub>4</sub>(C<sub>4</sub>H<sub>9</sub>)<sub>8</sub>(C<sub>10</sub>H<sub>6</sub>NO<sub>4</sub>)<sub>4</sub>O<sub>2</sub>], contains centrosymmetric dimers. It contains a central Sn<sub>2</sub>O<sub>2</sub> core with the O atoms bonded to two dibutylbis(*N*-phthaloylglycinato)tin units. The Sn atoms of the core are six-coordinate in a skew trapezoidal bipyramidal geometry, while the exocyclic Sn atoms are essentially five-coordinate in a distorted trigonal geometry. The Sn—C distances lie in a narrow range of 2.120 (5)–2.138 (4) Å.

## Comment

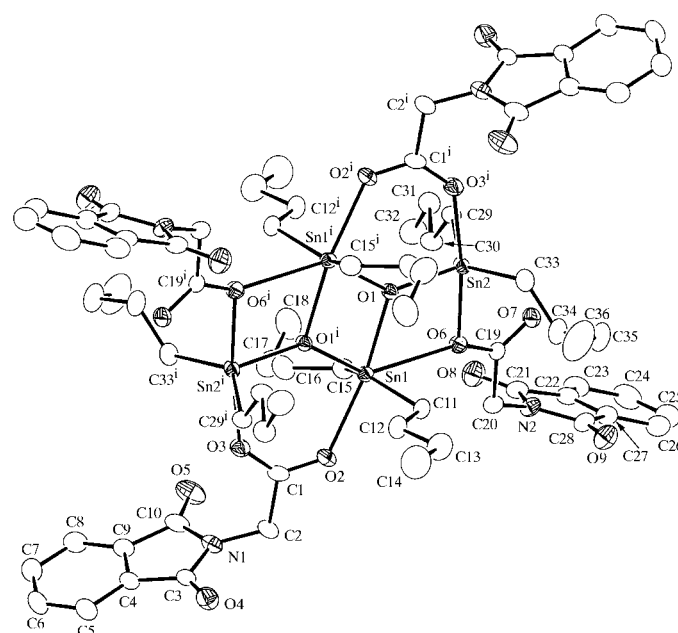
It has been reported that diorganotin complexes containing carboxylic acids, amino acids, purines, pyrimidines and peptides exhibit maximum antitumour activity, low mammalian toxicity and less nephrotoxicity than *cis*-platin (Sandhu *et al.*, 1992; Danish *et al.*, 1996; Gielen *et al.*, 1992). The structural chemistry of organotin complexes of amino acids protected as carboxylates has afforded a great diversity of structural variation in recent years and complexes with a coordination number higher than four are being studied extensively (Tiekink, 1994; Mehring *et al.*, 1998; Parvez *et al.*, 1999). A number of crystal structures of organotin complexes containing the ligand *N*-phthaloylglycinate, a protected amino acid, and showing interesting features have been reported. For example, in triphenyltin *N*-phthaloylglycinate, six molecules were bridged *via* carboxylate groups to form a cyclic hexamer (Ng *et al.*, 1990). In the tricyclohexyltin analogue, the molecules were linked into a helical chain through an amido-O atom (Ng & Kumar Das, 1997). This protected amino acid, when condensed with (*p*-chlorophenyl)diphenyl hydroxide, yielded the corresponding aqua carboxylate wherein the coordinated water molecules had linked adjacent molecules

<sup>1</sup> Systematic name: octabutyl-1κ<sup>2</sup>C,2κ<sup>2</sup>C,3κ<sup>2</sup>C,4κ<sup>2</sup>C-bis-μ<sup>3</sup>oxo-1:2:3κ<sup>3</sup>O<sup>-</sup>:1:3:4κ<sup>3</sup>O-tetrakis(μ-phthalimidoacetato)-1:2κ<sup>2</sup>O:2:3κ<sup>2</sup>O:O':3:4κ<sup>2</sup>O;1:4κ<sup>2</sup>O:O'-tetratin(IV).

into sheets through hydrogen bonding (Lo *et al.*, 1997). It has been reported that dicarboxylato tetraorgano distannoxanes are often formed when diorganotin oxides are reacted with carboxylic acids and are the products of incomplete hydrolysis (Ng & Kumar Das, 1995a). In this paper, we report the crystal structure of such a product, the title compound, (I).



The structure of (I) is presented in Fig. 1. It is composed of a centrosymmetric dimer, with Sn1/O1/Sn2/O6 and Sn1<sup>i</sup>/O1<sup>i</sup>/Sn2<sup>i</sup>/O6<sup>i</sup> rings flanking an Sn1/O1/Sn1<sup>i</sup>/O1<sup>i</sup> core ring on opposite sides [symmetry code: (i) 2 - x, -y, 1 - z]. Similar structural units have already been reported (Ng *et al.*, 1991, 1994; Ng, 1998). The endocyclic Sn atoms of the Sn<sub>2</sub>O<sub>2</sub> core are six-coordinate in a C<sub>2</sub>SnO<sub>4</sub> skew-trapezoidal bipyramidal geometry, wherein Sn1 is coordinated to two bridging O atoms, two *N*-phthaloylglycinato anions and two α-C atoms of the *n*-butyl groups. The endocyclic Sn—O distances in the Sn<sub>2</sub>O<sub>2</sub> core, 2.043 (3) and 2.178 (3) Å, are similar to the corresponding distances found in the tetrabutylbis(*N,N*-diethylthiocarbamoylthio)distannoxane dimer (Ng & Kumar



**Figure 1**  
ORTEP II (Johnson, 1976) drawing of (I) with the atomic numbering scheme. Displacement ellipsoids have been plotted at the 30% probability level and H atoms have been omitted for clarity. Atom C18', representing the smaller fraction of the disordered methyl atom C18, has also been omitted [symmetry code: (i) 2 - x, -y, 1 - z].

Das, 1995*a*), bis[1,1,3,3-tetrabutyl-1,3-bis(picolinato *N*-oxide)]-distannoxane hydrate (Ng, 1998) and the isomeric triphenyltin 2-, 3- and 4-pyridinecarboxylate *N*-oxides (Ng & Kumar Das, 1995*b*). The exocyclic distances Sn1—O2 and Sn1—O6 comprise a strong and a relatively weak Sn···O interaction [2.304 (3) and 2.746 (3) Å, respectively] and are similar to those observed in the above-mentioned complexes. The exocyclic Sn2 atom is essentially five-coordinate in a distorted trigonal geometry, the C—Sn—C angle being 135.34 (19)°.

The Sn—C distances, which are almost identical, lie within the narrow range of 2.120 (5)–2.138 (4) Å and are in agreement with the values reported for related structures. The molecular dimensions in the ligand, *N*-phthaloylglycinate, are normal and agree with the corresponding molecular dimensions reported in the structure of 2-phthalimidoethanoic acid monohydrate (Feeder & Jones, 1994).

## Experimental

The title compound was prepared by treating equimolar quantities of phthaloylglycine and dibutyltin oxide in toluene/ethanol (3:1) under azeotropic reflux conditions for 4 h. The solid thus obtained was recrystallized from ethanol.

### Crystal data

[Sn<sub>2</sub>(C<sub>4</sub>H<sub>9</sub>)<sub>8</sub>(C<sub>10</sub>H<sub>6</sub>NO<sub>4</sub>)<sub>4</sub>O<sub>2</sub>]  
*M<sub>r</sub>* = 1780.28  
 Triclinic, *P* $\bar{1}$   
*a* = 13.344 (5) Å  
*b* = 14.393 (4) Å  
*c* = 11.683 (2) Å  
 $\alpha$  = 93.51 (3)°  
 $\beta$  = 110.00 (2)°  
 $\gamma$  = 64.19 (2)°  
*V* = 1888.0 (10) Å<sup>3</sup>

*Z* = 1  
*D<sub>x</sub>* = 1.566 Mg m<sup>-3</sup>  
 Mo K $\alpha$  radiation  
 Cell parameters from 25 reflections  
 $\theta$  = 9.0–15.0°  
 $\mu$  = 1.38 mm<sup>-1</sup>  
*T* = 170 (2) K  
 Prism, colourless  
 0.48 × 0.26 × 0.21 mm

### Data collection

Rigaku AFC-6S diffractometer  
 $\omega$ -2 $\theta$  scans  
 Absorption correction:  $\psi$  scans  
 (North *et al.*, 1968)  
*T<sub>min</sub>* = 0.70, *T<sub>max</sub>* = 0.75  
 9112 measured reflections  
 8728 independent reflections  
 5278 reflections with *I* > 2 $\sigma$ (*I*)

*R<sub>int</sub>* = 0.027  
 $\theta_{\text{max}}$  = 27.56°  
*h* = 0 → 17  
*k* = -16 → 18  
*l* = -15 → 14  
 3 standard reflections  
 every 200 reflections  
 intensity decay: 1.1%

### Refinement

Refinement on *F*<sup>2</sup>  
*R*[*F*<sup>2</sup> > 2 $\sigma$ (*F*<sup>2</sup>)] = 0.032  
*wR*(*F*<sup>2</sup>) = 0.082  
*S* = 1.02  
 8728 reflections  
 447 parameters

H atoms constrained  
 $w = 1/[\sigma^2(F_o^2) + (0.0115P)^2 + 2.325P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} = 0.002$   
 $\Delta\rho_{\text{max}} = 0.93 \text{ e } \text{Å}^{-3}$   
 $\Delta\rho_{\text{min}} = -0.74 \text{ e } \text{Å}^{-3}$

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1988); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *TEXSAN* (Molecular Structure Corporation, 1994); program(s) used to solve structure: *SAPI91* (Fan, 1991); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *TEXSAN*; software used to prepare material for publication: *SHELXL97*.

**Table 1**

Selected geometric parameters (Å, °).

|                          |             |                         |             |
|--------------------------|-------------|-------------------------|-------------|
| Sn1—C11                  | 2.125 (5)   | Sn2—C33                 | 2.133 (5)   |
| Sn1—C15                  | 2.120 (5)   | Sn2—C29                 | 2.138 (4)   |
| Sn1—O1 <sup>i</sup>      | 2.043 (3)   | Sn2—O1                  | 2.037 (3)   |
| Sn1—O1                   | 2.178 (3)   | Sn2—O3 <sup>i</sup>     | 2.320 (3)   |
| Sn1—O2                   | 2.304 (3)   | Sn2—O6                  | 2.157 (3)   |
| O1 <sup>i</sup> —Sn1—C15 | 108.09 (15) | O1—Sn2—C33              | 111.46 (16) |
| O1 <sup>i</sup> —Sn1—C11 | 107.38 (15) | O1—Sn2—C29              | 111.28 (15) |
| C15—Sn1—C11              | 143.71 (18) | C33—Sn2—C29             | 135.34 (19) |
| O1 <sup>i</sup> —Sn1—O1  | 76.73 (12)  | O1—Sn2—O6               | 79.67 (11)  |
| C15—Sn1—O1               | 99.41 (15)  | C33—Sn2—O6              | 97.95 (16)  |
| C11—Sn1—O1               | 95.88 (15)  | C29—Sn2—O6              | 101.78 (15) |
| O1 <sup>i</sup> —Sn1—O2  | 91.03 (11)  | O1—Sn2—O3 <sup>i</sup>  | 90.29 (11)  |
| C15—Sn1—O2               | 87.44 (16)  | C33—Sn2—O3 <sup>i</sup> | 82.44 (16)  |
| C11—Sn1—O2               | 84.49 (16)  | C29—Sn2—O3 <sup>i</sup> | 84.98 (15)  |
| O1—Sn1—O2                | 167.32 (11) | O6—Sn2—O3 <sup>i</sup>  | 169.40 (11) |

Symmetry code: (i) 2 - *x*, -*y*, 1 - *z*.

MP thanks the Natural Sciences and Engineering Research Council, Canada, for providing the diffractometer through an equipment grant to the University of Calgary.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: BK1497). Services for accessing these data are described at the back of the journal.

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