

## Tetrabutylbis[3-(4-methoxyphenyl)-2-phenyl-2-propenoato]distannoxane dimer

Masood Parvez,<sup>a\*</sup>  
Sadiq-ur-Rehman,<sup>b</sup> Khadija  
Shahid,<sup>b</sup> Saqib Ali<sup>b</sup> and  
Muhammad Mazhar<sup>b</sup><sup>a</sup>Department of Chemistry, The University of Calgary, 2500 University Drive NW, Calgary, Alberta, Canada T2N 1N4, and <sup>b</sup>Department of Chemistry, Quaid-i-Azam University, Islamabad 45320, Pakistan

Correspondence e-mail: parvez@ucalgary.ca

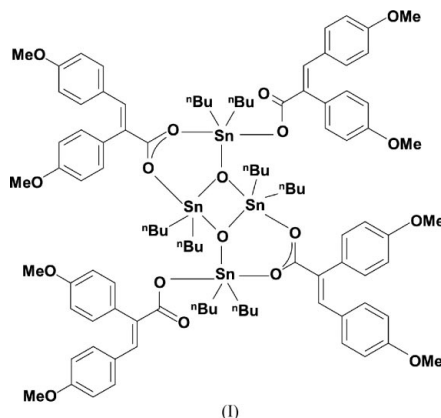
## Key indicators

Single-crystal X-ray study  
T = 173 K  
Mean  $\sigma(\text{C}-\text{C}) = 0.008 \text{ \AA}$   
Disorder in main residue  
R factor = 0.046  
wR factor = 0.097  
Data-to-parameter ratio = 21.7For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

The crystal structure of the title compound, bis[ $\mu_2$ -3-(4-methoxyphenyl)-2-phenyl-2-propenoato]bis[3-(4-methoxyphenyl)-2-phenyl-2-propenoato]di- $\mu_3$ -oxo-tetrakis[dibutyltin(IV)], [Sn<sub>4</sub>(C<sub>4</sub>H<sub>9</sub>)<sub>8</sub>(C<sub>17</sub>H<sub>15</sub>O<sub>2</sub>)<sub>4</sub>O<sub>2</sub>], is composed of centrosymmetric dimers in which dibutylbis[[3-(4-methoxyphenyl)-2-phenyl-2-propenoato]tin] units containing monodentate and bridging bidentate ligands are coordinated to the central Sn<sub>2</sub>O<sub>2</sub> core through its O atoms. Both Sn atoms adopt distorted trigonal bipyramidal geometry, with Sn–C distances lying in a narrow range 2.096 (13)–2.161 (6) Å, while Sn–O distances range between 2.041 (2) and 2.262 (3) Å.

## Comment

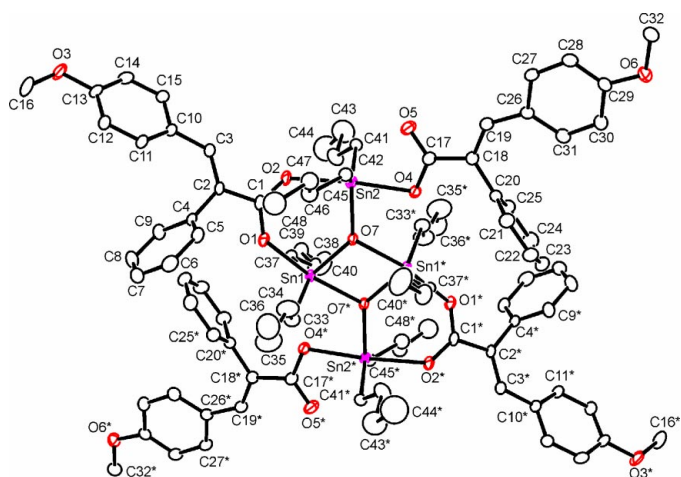
Organotin compounds have important applications in several areas. By far the major use of these compounds is in the stabilization of PVC to prevent thermal degradation during processing and long-term photodegradation (Ahmad *et al.*, 2000). Dialkyltin derivatives are the additives of choice for this purpose, *e.g.* dioctyltin compounds are used in PVC for food packaging, drink bottles and portable water piping (Davies & Smith, 1982). Dibutyltin dilaurate is widely employed as a catalyst for the production of polyurethane foams or for the room-temperature curing of silicone rubber (Al-Allaf *et al.*, 1999). Organotin carboxylates have been extensively used as antitumor agents (Davies & Smith, 1982). In continuation of our studies on the structural aspects of organotin carboxylates (Parvez, Ali, Mazhar, Bhatti & Khokhar *et al.*, 1999; Parvez, Ali, Bhatti *et al.*, 1999; Parvez, Ali, Mazhar, Bhatti & Choudhary, 1999; Parvez *et al.*, 2000, 2002; Sadiq-ur-Rehman, Shouldice *et al.*, 2004; Sadiq-ur-Rehman, Abdelrahman *et al.*, 2004), we have synthesized a new compound, the tetrabutylbis[3-(4-methoxyphenyl)-2-phenyl-2-propenoato]distannoxane dimer, (I), the structure of which is reported here.



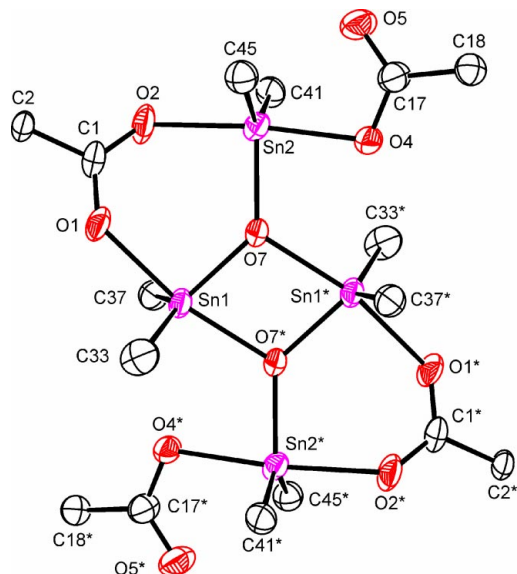
Received 10 September 2004

Accepted 15 September 2004

Online 25 September 2004



**Figure 1**  
ORTEPII (Johnson, 1976) drawing of (I), with displacement ellipsoids drawn at the 25% probability level; the minor components of the disordered C atoms of butyl groups have been omitted, as have the H atoms.



**Figure 2**  
ORTEPII (Johnson, 1976) drawing showing the central core of (I), with displacement ellipsoids plotted at the 50% probability level.

The structure of (I) is composed of a centrosymmetric dimer. The endocyclic Sn—O distances in the central core, Sn1/O7/Sn1<sup>i</sup>/O7<sup>i</sup> [symmetry code: (i) 1 - x, 1 - y, 2 - z], of 2.041 (2) and 2.182 (2) Å and the exocyclic distance Sn1—O1 of 2.249 (3) Å are similar to those observed in the tetrabutylbis(*N,N*-diethylthiocarbamoylthio)distannoxane dimer (Ng & Kumar Das, 1995*a*), bis[1,1,3,3-tetrabutyl-1,3-bis-(picolinato *N*-oxide)]distannoxane hydrate (Ng, 1998), the isomeric triphenyltin-2-(3-,4-)pyridinecarboxylate *N*-oxides (Ng & Kumar Das, 1995*b*), the tetrabutylbis(*N*-phthaloylglycinato)distannoxane dimer (Parvez *et al.*, 2000) and the tetrabutylbis(*N*-phthaloylphenylalaninato)distannoxane dimer (Hans *et al.*, 2002). However, the Sn1...O4<sup>i</sup> interaction of 3.012 (3) Å in (I) is significantly longer than the corresponding interaction observed in both the tetrabutylbis(*N*-

phthaloylphenylalaninato)distannoxane dimer (Hans *et al.*, 2002) [2.725 (3) Å] and the tetrabutylbis(*N*-phthaloylglycinato)distannoxane dimer [2.746 (3) Å; Parvez *et al.*, 2000].

Both independent Sn atoms in (I) are in a five-coordinate C<sub>2</sub>SnO<sub>3</sub> distorted trigonal-bipyramidal geometry. The angles O1—Sn1—O7<sup>i</sup> and O2—Sn2—O4 of 164.87 (10) and 173.93 (10)°, respectively, are approximately linear. Atom Sn1 lies 0.091 (2) Å out of the equatorial plane defined by atoms O7/C33/C37, towards O7<sup>i</sup>, while Sn2 lies essentially in the equatorial plane defined by atoms O7/C41/C45 [maximum deviation = 0.008 (3) Å]. The distortion of the trigonal-bipyramidal geometry is reflected in the trigonal angles around Sn1 and Sn2 lying in wide ranges, *viz.* 108.72 (14)–136.00 (18) and 109.89 (14)–136.3 (2)°, respectively. Similarly, the remaining angles around Sn1 and Sn2, in the ranges 76.01 (10)–100.78 (14) and 82.66 (9)–96.32 (13)° [ignoring the minor components of the disordered α-C atoms of the butyl groups], respectively, also deviate significantly from the ideal value of 90°.

The Sn—C distances involving the ordered C atoms are almost identical, with a mean value of 2.127 (3) Å, while those involving disordered C atoms are 2.096 (13) and 2.161 (6) Å for the major and minor components of C45. These values are in agreement for the corresponding distances reported in similar structures cited above. It is interesting to note that one of the ligands is coordinated to both Sn atoms, with C—O distances [O1—C1 = 1.277 (5) Å and O2—C1 = 1.252 (5) Å] lying between single- and double-bond length, representing a delocalized system. In the other ligand coordinated to Sn2 *via* O4, the distances O4—C17 [1.292 (5) Å] and O5—C17 [1.241 (5) Å] indicate a single and a double bond, respectively. The remaining dimensions in the ligands are in agreement with the corresponding dimensions reported for the derivatives of 2,3-diphenylpropenoic acid (Sadiq-ur-Rehman, Shouldice *et al.*, 2004; Sadiq-ur-Rehman, Abdelrahman *et al.*, 2004; Sadiq-ur-Rehman, Ali *et al.*, 2004).

## Experimental

A mixture of (*E*)-3-(4-methoxyphenyl)-2-phenyl-2-propenoic acid (2.398 g, 9.441 mmol) and di-*n*-butyltin oxide (2.350 g, 9.551 mmol) was refluxed in dry toluene (80 ml) for 8–10 h using a Dean–Stark apparatus. The reaction mixture was cooled to room temperature and the solvent was removed on a rotary evaporator. The resulting solid mass was crystallized from a mixture of chloroform and *n*-hexane (4:1), resulting in prismatic crystals of (I) (yield 75%; m.p. 376–378 K).

### Crystal data

[Sn<sub>4</sub>(C<sub>4</sub>H<sub>9</sub>)<sub>8</sub>(C<sub>17</sub>H<sub>15</sub>O<sub>2</sub>)<sub>4</sub>O<sub>2</sub>]  
*M<sub>r</sub>* = 1976.72  
 Triclinic, P $\bar{1}$   
*a* = 12.971 (2) Å  
*b* = 13.429 (3) Å  
*c* = 15.088 (3) Å  
 $\alpha$  = 92.490 (11)°  
 $\beta$  = 96.820 (11)°  
 $\gamma$  = 118.531 (8)°

*V* = 2277.5 (8) Å<sup>3</sup>  
*Z* = 1  
*D<sub>x</sub>* = 1.441 Mg m<sup>-3</sup>  
 Mo K $\alpha$  radiation  
 Cell parameters from 43035 reflections  
 $\theta$  = 2.7–28.3°  
 $\mu$  = 1.15 mm<sup>-1</sup>  
*T* = 173 (2) K

Prism, colorless	0.10 × 0.08 × 0.08 mm
<i>Data collection</i>	
Nonius KappaCCD diffractometer	7022 reflections with $I > 2\sigma(I)$
$\omega$ and $\varphi$ scans	$R_{\text{int}} = 0.084$
Absorption correction: multi-scan ( <i>SORTAV</i> ; Blessing, 1997)	$\theta_{\text{max}} = 28.3^\circ$
$T_{\text{min}} = 0.894$ , $T_{\text{max}} = 0.914$	$h = -17 \rightarrow 17$
43035 measured reflections	$k = -17 \rightarrow 17$
11049 independent reflections	$l = -19 \rightarrow 19$
<i>Refinement</i>	
Refinement on $F^2$	$w = 1/[\sigma^2(F_o^2) + (0.031P)^2 + 2.28P]$
$R[F^2 > 2\sigma(F^2)] = 0.046$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.097$	$(\Delta/\sigma)_{\text{max}} = 0.002$
$S = 1.02$	$\Delta\rho_{\text{max}} = 0.72 \text{ e } \text{\AA}^{-3}$
11049 reflections	$\Delta\rho_{\text{min}} = -0.92 \text{ e } \text{\AA}^{-3}$
510 parameters	
H-atom parameters constrained	

**Table 1**  
Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ ).

Sn1—O7	2.041 (2)	Sn2—O2	2.262 (3)
Sn1—C33	2.123 (4)	O1—C1	1.277 (5)
Sn1—C37	2.127 (4)	O2—C1	1.252 (5)
Sn1—O7 <sup>i</sup>	2.182 (2)	O3—C13	1.365 (5)
Sn1—O1	2.249 (3)	O3—C16	1.431 (6)
Sn2—C45'	2.096 (13)	O4—C17	1.292 (5)
Sn2—O7	2.043 (2)	O5—C17	1.241 (5)
Sn2—C41	2.130 (4)	O6—C29	1.369 (5)
Sn2—C45	2.161 (6)	O6—C32	1.427 (5)
Sn2—O4	2.192 (2)		
O7—Sn1—C33	114.70 (16)	C45—Sn2—O4	88.76 (18)
O7—Sn1—C37	108.72 (14)	O7—Sn2—O2	91.50 (10)
C33—Sn1—C37	136.00 (18)	C41—Sn2—O2	84.11 (14)
O7—Sn1—O7 <sup>i</sup>	76.01 (10)	C45—Sn2—O2	95.12 (19)
C33—Sn1—O7 <sup>i</sup>	96.21 (14)	O4—Sn2—O2	173.93 (10)
C37—Sn1—O7 <sup>i</sup>	100.78 (13)	C1—O1—Sn1	132.3 (2)
O7—Sn1—O1	90.42 (10)	C1—O2—Sn2	128.1 (3)
C33—Sn1—O1	83.25 (14)	C13—O3—C16	116.6 (4)
C37—Sn1—O1	89.80 (14)	C17—O4—Sn2	105.4 (2)
O7 <sup>i</sup> —Sn1—O1	164.87 (10)	C17—O5—Sn2	80.5 (2)
O7—Sn2—C41	109.89 (13)	C29—O6—C32	117.0 (3)
O7—Sn2—C45	113.78 (18)	Sn1—O7—Sn2	132.27 (12)
C41—Sn2—C45	136.3 (2)	Sn1—O7—Sn1 <sup>i</sup>	103.99 (10)
O7—Sn2—O4	82.66 (9)	Sn2—O7—Sn1 <sup>i</sup>	122.48 (11)
C41—Sn2—O4	96.32 (13)		

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

Atom C44 of one butyl group and atoms C45–C48 of another butyl group are disordered over two sites each with unequal site-occupancy factors [occupancies of 0.784 (18):0.216 (18) and 0.682 (7):0.318 (7), respectively]. Disordered C atoms were assigned

isotropic displacement parameters. H atoms were included in the refinement at geometrically idealized positions, with C–H = 0.95, 0.98 and 0.99  $\text{\AA}$ , and  $U_{\text{iso}}(\text{H}) = 1.5$  (methyl) and 1.2 (aromatic and  $\text{CH}_2$  type) times  $U_{\text{eq}}$  of the atoms to which they are bonded. The final difference map was free of any chemically significant features.

Data collection: *COLLECT* (Hooft, 1998); cell refinement: *HKL DENZO* (Otwinowski & Minor, 1997); data reduction: *SCALE-PAK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SAP91* (Fan, 1991); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

## References

- Ahmad, Z., Rehman, H. U., Ali, S. & Sarwar, M. I. (2000). *Int. J. Pol. Mater.* **46**, 547–559.
- Al-Allaf, T. A. K., Khuzaic, F. R., Rashan, L. J. & Halaseh, W. F. (1999). *Boll. Chim. Farm.* **138**, 267–271.
- Blessing, R. H. (1997). *J. Appl. Cryst.* **30**, 421–426.
- Davies, A. G. & Smith, P. J. (1982). *Comprehensive Organometallic Chemistry, The Synthesis, Reactions, and Structures of Organometallic Compounds*, Vol. 2, edited by G. Wilkinson, F. G. Stone and E. W. Abel, pp. 519–527. New York: Pergamon.
- Fan, H.-F. (1991). *SAP91*. Rigaku Corporation, Tokyo, Japan.
- Hans, K., Parvez, M., Ahmad, F., Ali, S., Mazhar, M. & Munir, A. (2002). *Acta Cryst.* **E58**, m441–m443.
- Hooft, R. (1998). *COLLECT*. Nonius BV, Delft, The Netherlands.
- Johnson, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
- Ng, S. W. (1998). *Acta Cryst.* **C54**, 752–755.
- Ng, S. W. & Kumar Das, V. G. (1995a). *Acta Cryst.* **C51**, 1774–1776.
- Ng, S. W. & Kumar Das, V. G. (1995b). *Main Group Met. Chem.* **18**, 315–328.
- Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr and R. M. Sweet, pp. 307–326. New York: Academic Press.
- Parvez, M., Ali, S., Ahmad, S., Bhatti, M. H. & Mazhar, M. (2002). *Acta Cryst.* **C58**, m334–m335.
- Parvez, M., Ali, S., Bhatti, M. H., Khokhar, M. N., Mazhar, M. & Qureshi, S. I. (1999). *Acta Cryst.* **C55**, 1427–1429.
- Parvez, M., Ali, S., Mazhar, M., Bhatti, M. H. & Choudhary, M. A. (1999). *Acta Cryst.* **C55**, 1429–1431.
- Parvez, M., Ali, S., Mazhar, M., Bhatti, M. H. & Khokhar, M. N. (1999). *Acta Cryst.* **C55**, 1280–1282.
- Parvez, M., Bhatti, M. H., Ali, S., Mazhar, M. & Qureshi, S. I. (2000). *Acta Cryst.* **C56**, 327–328.
- Sadiq-ur-Rehman, Abdelrahman, D., Ali, S., Badshah, A. & Parvez, M. (2004). *Acta Cryst.* **E60**, m1076–m1078.
- Sadiq-ur-Rehman, Ali, S., Mazhar, M. & Parvez, M. (2004). *Acta Cryst.* **E60**, m1394–m1396.
- Sadiq-ur-Rehman, Shouldice, S. R., Ali, S., Badshah, A. & Parvez, M. (2004). *Acta Cryst.* **E60**, m670–m672.
- Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.