Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Bis[2,3-bis(4-chlorophenyl)propenoato-κ^2O:O']-di-*n*-butyltin(IV)

Sadiq-ur-Rehman,[a] Hanh Vien Ly,[b] Saqib Ali,[a] Amin Badshah[a] and Masood Parvez[b]*

[a]Department of Chemistry, Quaid-i-Azam University, Islamabad 45320, Pakistan, and [b]Department of Chemistry, The University of Calgary, 2500 University Drive NW, Calgary, Alberta, Canada T2N 1N4

Correspondence e-mail: parvez@ucalgary.ca

Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.007 Å
Disorder in main residue
R factor = 0.047
wR factor = 0.138
Data-to-parameter ratio = 19.4

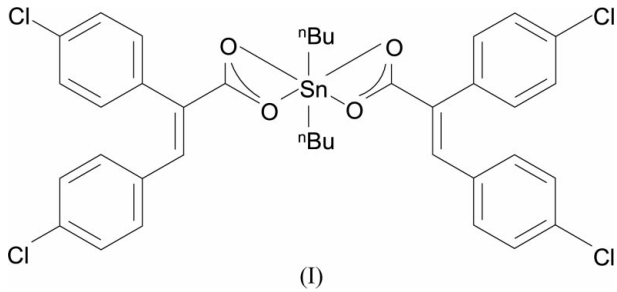
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 7 July 2004
Accepted 13 July 2004
Online 24 July 2004

The crystal structure of the title compound, $[Sn(C_4H_9)_2(C_{15}H_9Cl_2O_2)_2]$, has a highly distorted octahedral geometry that may be best described as a skew-trapezoid planar geometry with two additional axial ligands. The carboxylate ligands are asymmetrically coordinated to the Sn atoms, with short Sn—O covalent bonds [mean 2.098 (6) Å] and long dative bonds [mean 2.602 (12) Å]. The mean Sn—C distance for the *n*-butyl groups is 2.117 (13) Å, with a C—Sn—C angle of 139.18 (18)°.

Comment

The synthesis and structural chemistry of organotin compounds are still a fertile area of research because of their extensive biological applications. Early discoveries meant that these organotin compounds were utilized as antitumor and anticancer agents (Crowe, 1989; Gielen *et al.*, 1994, 2000; de Vos *et al.*, 1998). More recently, the structural features of the organotin carboxylates of general formula R_2SnL_2 (where R = alkyl or aryl, and L = carboxylate ligand) have attracted much attention, and these organotin derivatives are known to be excellent anticancer agents (Gielen, 1996, 2002; Parvez *et al.*, 1997). Continuing our interest in studying the structural chemistry of organotin carboxylates (Parvez, Ali, Mazhar, Bhatti & Khokhar, 1999; Parvez, Ali, Bhatti *et al.*, 1999; Parvez, Ali, Mazhar, Bhatti & Choudhary, 1999; Parvez *et al.*, 2000, 2002; Sadiq-ur-Rehman *et al.*, 2004), in this paper we report the crystal structure of the title compound, (I).



(I)

The structure of (I) is composed of discrete monomeric molecules (Fig. 1), in whic six-coordinated Sn atoms are bonded to two *n*-butyl groups and two 2,3-bis(4-chlorophenyl)propenoate ligands. The geometry around the Sn atom is highly distorted octahedral and can best be described as a skew-trapezoid planar geometry with two additional axial ligands. The Sn atom lies 0.190 (2) Å from the plane formed by the asymmetrically bonded O atoms of the carboxylate ligands, while the two *n*-butyl groups lie above and below this plane. The mean Sn—C distance of 2.117 (13) Å and a C—

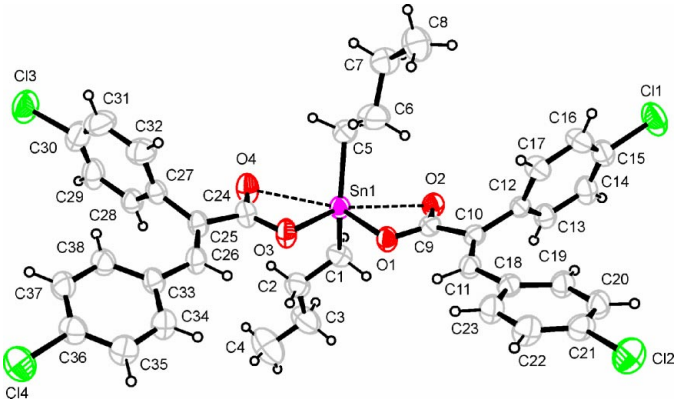


Figure 1
*ORTEP*II (Johnson, 1976) drawing of (I), with displacement ellipsoids plotted at the 30% probability level; the minor components of the disordered atoms C7′, C8′ and Cl3′ have been omitted.

Sn—C angle of 139.18 (18)° agree with the corresponding distances and angles reported previously for other complexes (Gibson *et al.*, 1997; Parvez *et al.*, 1997; Hans *et al.*, 2002; Sadiq-ur-Rehman *et al.*, 2004). The carboxylate ligands are asymmetrically coordinated to the Sn atoms, with Sn—O covalent bonds [mean 2.098 (6) Å] that are significantly shorter than the dative bonds [mean 2.602 (12) Å]. These bond distances are also very similar to the corresponding distances reported in related organotin compounds (Hans *et al.*, 2002; Stocco *et al.*, 1996; Gielen *et al.*, 1998; Gibson *et al.*, 1997; Parvez *et al.*, 2000; Ramirez *et al.*, 2002; Parvez *et al.*, 1997; Sadiq-ur-Rehman *et al.*, 2004).

The molecular dimensions in the ligands are comparable to those reported previously (Sadiq-ur-Rehman *et al.*, 2004). The O2═C9 and O4═C24 bond distances, 1.240 (4) and 1.234 (5) Å, respectively, indicate that these are double bonds, while the O1—C9 and O3—C24 distances, 1.294 (5) and 1.295 (5) Å, respectively, represent single bonds. The orientations of the benzene rings in the two ligands differ significantly. The C12–C17 and C18–C23 benzene rings are inclined at 69.38 (16)° with respect to each other, while the corresponding angle in the second ligand (between the C27–C32 and C33–C38 rings) is 82.88 (14)°. The propenoate moieties, O1/O2/C9–C11 and O3/O4/C24–C26, are individually planar, with maximum deviations of 0.094 (3) and 0.095 (3) Å for atoms C10 and C25, respectively. The C12–C17 and C18–C23 benzene rings are inclined at 69.38 (16) and 8.1 (3)°, respectively, from the mean plane of the propenoate moiety, O1/O2/C9–C11. In the second ligand, the corresponding angles between the planes of the benzene rings, C27–C32 and C33–C38, and the propenoate moiety, O3/O4/C24–C26, are 76.78 (14) and 14.8 (2)°, respectively.

Experimental

Di-*n*-butyltin oxide (0.85 g, 3.42 mmol) and 2,3-bis(4-chlorophenyl)-propenoic acid (2.0 g, 6.83 mmol) were suspended in dry toluene (100 ml) in a two-necked round-bottomed flask (250 ml) equipped with a Dean–Stark funnel and water condenser. The mixture was refluxed for 8–10 h, and water that formed during the condensation

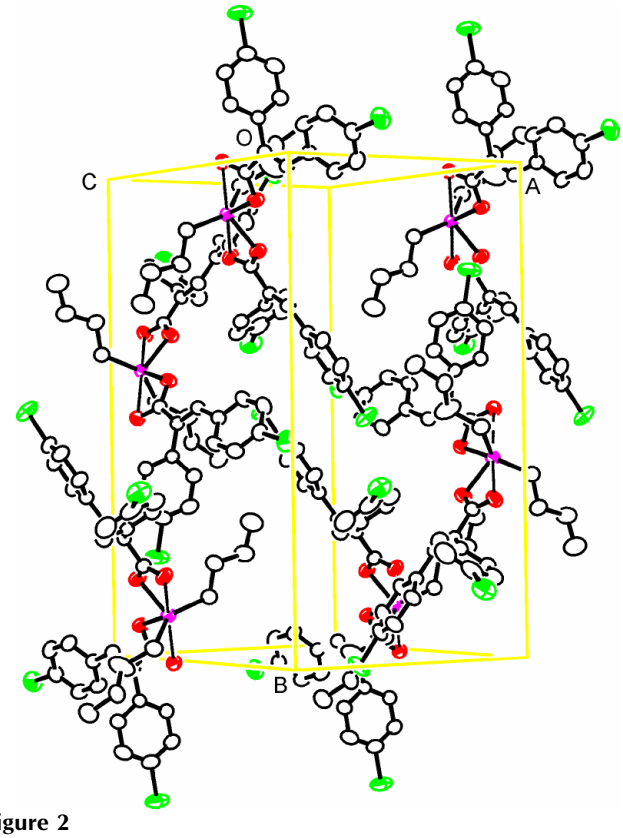


Figure 2
*ORTEP*II (Johnson, 1976) drawing of the unit cell contents. H atoms have been omitted.

reaction was periodically removed *via* a Dean–Stark separator. The mixture was cooled to room temperature and solvent was removed on a rotary evaporator (yield 80%, m.p.379–381 K). The solid was recrystallized from chloroform to which a few drops of *n*-hexane were added to obtain crystals suitable for X-ray analysis.

Crystal data

$[Sn(C_4H_9)_2(C_{15}H_9Cl_2O_2)_2]$
M_r = 817.16
Monoclinic, $P2_1/c$
a = 11.877 (2) Å
b = 21.552 (3) Å
c = 14.817 (2) Å
β = 91.696 (7)°
V = 3791.1 (10) Å^3
Z = 4
D_x = 1.432 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 16 543 reflections
θ = 3.2–27.5°
μ = 0.99 mm^{-1}
T = 295 (2) K
Block, colorless
0.12 × 0.11 × 0.10 mm

Data collection

Nonius KappaCCD diffractometer
ω and φ scans
Absorption correction: multi-scan (*SORTAV*; Blessing, 1997)
T_{min} = 0.890, T_{max} = 0.907
16 543 measured reflections
8637 independent reflections
5271 reflections with $I > 2\sigma(I)$
R_{int} = 0.035
θ_{max} = 27.5°
$h = -15 \rightarrow 15$
$k = -25 \rightarrow 27$
$l = -19 \rightarrow 19$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.138
S = 1.02
8637 reflections
445 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.065P)^2 + 1.86P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.020
$\Delta\rho_{max}$ = 0.96 e Å^{-3}
$\Delta\rho_{min}$ = −0.61 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0013 (3)

Table 1
Selected geometric parameters (Å, °).

Sn1—O3	2.092 (3)	Cl3—C30	1.819 (10)
Sn1—O1	2.104 (3)	Cl3′—C30	1.710 (11)
Sn1—C1	2.104 (5)	Cl4—C36	1.739 (5)
Sn1—C5	2.129 (4)	O1—C9	1.294 (5)
Sn1—O2	2.590 (3)	O2—C9	1.240 (4)
Sn1—O4	2.614 (3)	O3—C24	1.295 (5)
Cl1—C15	1.750 (5)	O4—C24	1.234 (5)
Cl2—C21	1.740 (4)		
O3—Sn1—O1	81.07 (11)	O3—Sn1—O4	54.29 (10)
O3—Sn1—C1	105.52 (15)	O1—Sn1—O4	134.59 (10)
O1—Sn1—C1	105.29 (16)	C1—Sn1—O4	95.29 (15)
O3—Sn1—C5	106.19 (17)	C5—Sn1—O4	83.04 (15)
O1—Sn1—C5	104.44 (14)	O2—Sn1—O4	166.53 (9)
C1—Sn1—C5	139.18 (18)	C9—O1—Sn1	103.2 (2)
O3—Sn1—O2	135.49 (9)	C9—O2—Sn1	82.0 (2)
O1—Sn1—O2	54.51 (9)	C24—O3—Sn1	104.0 (2)
C1—Sn1—O2	90.35 (15)	C24—O4—Sn1	81.2 (2)
C5—Sn1—O2	84.70 (14)		

Two C atoms of an *n*-butyl group were disordered over sites C7 and C8, with 0.784 (12) site-occupancy factors and minor components at C7′ and C8′. Equal anisotropic displacement parameters were used for these C atoms. A Cl atom was also disordered over two sites, Cl3 and Cl3′, with unequal site-occupancy factors of 0.57 (4) and 0.43 (4), respectively. H atoms were located in difference Fourier syntheses and were included in the refinement at idealized positions, with C—H distances of 0.93–0.97 Å and U_{iso}(H) values of 1.5 (methyl H atoms) and 1.2 (other H atoms) times U_{eq} of the atoms to which they were bonded. The final difference map was free of any chemically significant features, with the highest electron density located in the vicinity of the disordered *n*-butyl group.

Data collection: *COLLECT* (Hooft, 1998); cell refinement: *HKL DENZO* (Otwinowski & Minor, 1997); data reduction: *HKL SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SAPI*91 (Fan, 1991); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *SHELXL*97.

References

Blessing, R. H. (1997). *J. Appl. Cryst.* **30**, 421–426.
Crowe, A. J. (1989). *Metal-Based Antitumour Drugs*, Vol. 1, edited by M. Giele, pp. 103–149. London: Freund.
Fan, H.-F. (1991). *SAPI*91. Rigaku Corporation, Tokyo, Japan.
Gibson, D. H., Mehta, J. M., Mashuta, M. S. & Richardson, J. F. (1997). *Organometallics*, **16**, 4828–4832.
Gielen, M. (1996). *Coord. Chem. Rev.* **151**, 41–51.
Gielen, M. (2002). *Appl. Organomet. Chem.* **16**, 481–494.
Gielen, M., Biesemans, M., de Vos, D. & Willem, R. (2000). *J. Inorg. Biochem.* **79**, 139–145.
Gielen, M., Boualam, M., Mahieu, B. & Tiekink, E. R. T. (1994). *Appl. Organomet. Chem.* **8**, 19–23.
Gielen, M., Dalil, H., Ghys, L., Boduszek, B., Tiekink, E. R. T., Martins, J. C., Biesemans, M. & Willem, R. (1998). *Organometallics*, **17**, 4259–4262.
Hans, K., Parvez, M., Ahmad, F., Ali, S., Mazhar, M. & Munir, A. (2002). *Acta Cryst.* E**58**, m441–m443.
Hooft, R. (1998). *COLLECT*. Nonius BV, Delft, The Netherlands.
Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.
Parvez, M., Ali, S., Ahmad, S., Bhatti, M. H. & Mazhar, M. (2002). *Acta Cryst.* C**58**, m334–m335.
Parvez, M., Ali, S., Bhatti, M. H., Khokhar, M. N. Mazhar, M. & Qureshi, S. I. (1999). *Acta Cryst.* C**55**, 1427–1429.
Parvez, M., Ali, S., Masood, T. M., Mazhar, M. & Danish, M. (1997). *Acta Cryst.* C**53**, 1211–1213.
Parvez, M., Ali, S., Mazhar, M., Bhatti, M. H. & Choudhary, M. A. (1999). *Acta Cryst.* C**55**, 1429–1431.
Parvez, M., Ali, S., Mazhar, M., Bhatti, M. H. & Khokhar, M. N. (1999). *Acta Cryst.* C**55**, 1280–1282.
Parvez, M., Bhatti, M. H., Ali, S., Mazhar, M. & Qureshi, S. I. (2000). *Acta Cryst.* C**56**, 327–328.
Ramirez, A. R., Parvez, M., Ahmad, V. U., Hussain, J. & Hidayat, H. (2002). *Acta Cryst.* E**58**, m278–m280.
Sadiq-ur-Rehman, Shouldice, S. R., Ali, S., Badshah, A. & Parvez, M. (2004). *Acta Cryst.* E**60**, m670–m672.
Sheldrick, G. M. (1997). *SHELXL*97. University of Göttingen, Germany.
Stocco, G., Gul, G., Girasolo, M. A., Bruno, G., Nicol, F. & Scopelliti, R. (1996). *Acta Cryst.* C**52**, 829–832.
Vos, D. de, Willem, R., Gielen, M., van Wingerden, K. E. & Nooter, K. (1998). *Met. Based Drugs*, **5**, 179–188.