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Crystallographic report

Bis(triphenyltin) oxalate

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The centrosymmetric structure of bis(triphenyltin) oxalate contains essentially four-coordinated tin and monodentate carboxylate moieties despite a Mössbauer quadrupole splitting of 2.8 mm s^{-1} and a ^{119}Sn NMR chemical shift of −503 ppm. Copyright © 2003 John Wiley & Sons, Ltd.

KEYWORDS: crystal structure; tin; oxalate

COMMENT

The structures of organotin carboxylates continue to be the focus of much research[1,2] while triphenyltin compounds are noted for their biological activity.[3–6] The structure of compounds such as bis(triphenyltin) oxalate is thus of interest. The centrosymmetric title compound adopts an essentially tetrahedral geometry at tin with each half of the carboxylate ligand acting in a monodentate manner (Fig. 1). Both C–O [1.283(2) Å] and C=O [1.223(3) Å] fragments are distinguishable, and the Sn–O bond [2.111(1) Å] is typical of related species.[1,2] Although the Sn(1)–O(2) separation is only 2.592(1) Å the impact on the bond angles at tin is minor. However, the <C(1)–Sn(1)–C(13) opens slightly due to the proximity of O(2) [117.29(8)°] and <C(7)–Sn(1)–O(1) is unusually acute [86.78(7)°]. All other bond angles at tin are close to the tetrahedral value. The Mössbauer quadrupole splitting (2.80 mm s^{-1}) for the title compound is unusual for a tetrahedral species[7] and probably arises from the noted distortions. The ^{119}Sn chemical shift (−503 ppm) is also unusual for a four-coordinated tin.

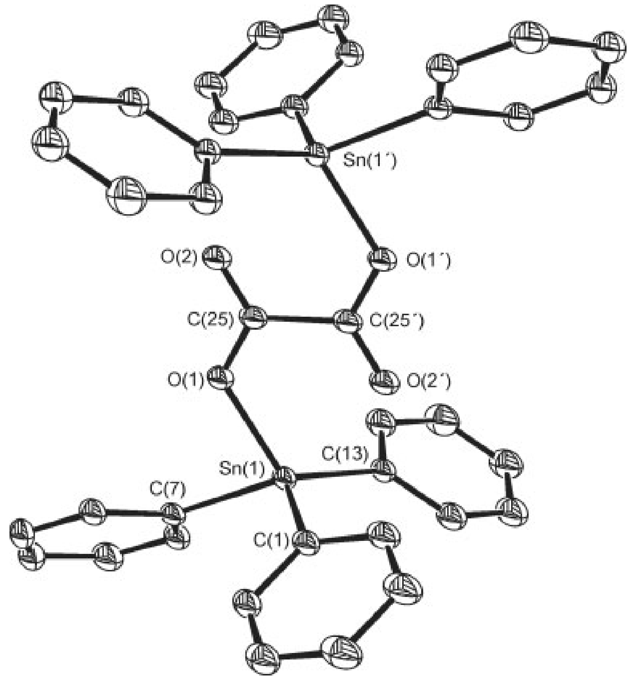

Figure 1. The structure of $(Ph_3Sn)_2(C_2O_4)$; thermal ellipsoids are at the 40% probability level. Selected geometric data: Sn(1)–O(1) 2.111(1), Sn(1)–C(1) 2.123(2), Sn(1)–C(7) 2.141(2), Sn(1)–C(13) 2.116(2), O(1)–C(25) 1.283(2), O(2)–C(25) 1.223(3), C(25)–C(25′) 1.538(4) Å; C(1)–Sn(1)–C(7) 109.72(8), C(1)–Sn(1)–C(13) 117.29(8), C(7)–Sn(1)–C(13) 110.79(8), C(1)–Sn(1)–O(1) 115.70(7), C(7)–Sn(1)–O(1) 86.78(7), C(13)–Sn(1)–O(1) 112.38(7)°. Symmetry operation for primed atoms: 1/3 −*x*, 2/3 −*y*, −*z* −1/3.

EXPERIMENTAL

$(Ph_3Sn)_2(C_2O_4)$ was prepared from the 1:1 reaction of $[H_3NCH_2CH_2NH_3]^{2+}$ $[C_2O_4]^{2-}$ in water and Ph_3SnCl in CH_3CN. Yield 72%, m.p. 250 °C. ^{119}Sn NMR ($CDCl_3$): −503 ppm. Mössbauer (mm s^{-1}): IS = 1.26, QS = 2.80. Intensity data were collected at

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150(2) K on a Nonius Kappa CCD diffractometer using a colourless block $0.30 \times 0.35 \times 0.35$ mm^3. $C_{38}H_{30}O_4Sn_2$, $M = 788.00$, trigonal, $R\bar{3}$, $a = 30.6690(4)$, $c = 11.7270(2)$ Å, $U = 9552.5(2)$ Å^3, $Z = 12$, 5917 independent reflections, $\theta_{\max}$ 30.1°, $R = 0.037$ (all data), $wR = 0.065$ (all data), $\rho_{\max,\ \min} = 0.95, -1.12$ e Å^{-3}. The asymmetric unit comprises two independent molecular halves. The first fragment, based on Sn(1), is located such that the half oxalate anion therein straddles an inversion centre, which serves to generate the remainder on the dimer. The second fragment consists of one phenyl ring attached to Sn(2) and an oxalate fragment containing O(1A), O(2A) and C(25A). The location of Sn(2) on a crystallographic -3 axis serves to generate the Ph_3Sn moiety. Initially, it seemed as though the metal-bound oxygen of the oxalate (O1A) was also located on the -3 axis, but the associated elongated thermal parameter did not appear to support this model. Early refinements of the model were also complicated by the proximity of the oxalate half to an inversion centre, which, as in the case of the Sn(1)-based motif, generates the dimer. Hence, the oxalate is disordered in this second fragment. Convergence was optimized by refinement of O(1A), O(2A) and C(25A) at 1/3 occupancy, with O(1A) located close to, but not on, the three fold axis. Metrical data for the ordered fragment based on Sn(1) are given in Fig. 1. Software used: SHELX-86, SHELX-97, ORTEX. CCDC deposition number 212936.

REFERENCES

1. Tiekink ERT. *Appl. Organometal. Chem.* 1991; **5**: 1.
2. Tiekink ERT. *Trends Organometal. Chem.* 1994; **1**: 71.
3. Gielen M. *Chem. Rev.* 1996; **151**: 41.
4. Kamruddin SK, Chattopadhyaya TK, Roy A, Tiekink ERT. *Appl. Organometal. Chem.* 1996; **10**: 513.
5. Gielen M, Bouhdid A, Tiekink ERT, de Vos D, Willem R. *Met. Based Drugs* 1996; **3**: 75.
6. Gielen M, Kaysar F, Zhidkova OB, Kampel VT, Bregadze VI, de Vos D, Biesemans M, Mahieu B, Willem R. *Met. Based Drugs* 1995; **2**: 37.
7. Davies AG, Smith PJ. In *Comprehensive Organometallic Chemistry*, vol. 2, Wilkinson G, Stone FGA, Abel EW (eds). Pergamon Press: Oxford, 1982; 525.