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Crystallographic report

Bis[tri(*o*-chlorobenzyl)tin(IV)] terephthalate

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The centrosymmetric structure of $(o\text{-}ClC_6H_4)_3SnO_2CC_6H_4CO_2Sn(C_6H_4Cl\text{-}o)_3$ features an unsymmetrically chelating carboxylate group, so that a distorted trigonal bipyramidal *cis*-C_3O_2 coordination geometry for tin results. Copyright © 2004 John Wiley & Sons, Ltd.

KEYWORDS: crystal structure; organotin; terephthalate

COMMENT

The centrosymmetric dinuclear structure of $(o\text{-Cl-}C_6H_4)_3SnS_2O_2CC_6H_4CO_2Sn(C_6H_4\text{-Cl-}o)_3$, Fig. 1, features a chelating terephthalate ligand which forms unsymmetric Sn–O bonds so that a five-coordinate *cis*-C_3O_2 geometry results in which the axial positions are the O1 and C12 atoms. The structure is similar, for example, to those reported for $[Ph_3Sn(CH_3OH)O_2CC_6H_5CO_2(CH_3OH)SnPh_3]\cdot 2CH_3OH$,[1] but it is different from complexes $Ph_3SnO_2C(CH_2)_3CO_2SnPh_3$,[2] $(n\text{-}C_4H_9)_3SnO_2C(CH_2)_2CO_2Sn(C_4H_9-n)_3$[3] and $Cy_2MeSnO_2CFcCO_2SnMeCy_2$[4] (Fc = ferrocene), underscoring the rich diversity in organotin carboxylates.[5,6]

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EXPERIMENTAL

Anhydrous disodium terephthalate (2.0 mmol) was added to a methanol solution (30 ml) of tri(*o*-chlorobenzyl)tin chloride (4.0 mmol) and stirred for 10 h at 60 °C. The precipitated sodium chloride was removed by filtration and the filtrate was concentrated to about 5 ml under reduced pressure. Hexane (5 ml) was added to this solution, and a precipitate was formed immediately. The product was recrystallized from methanol to give a colorless crystal. M.p. 384–386 K. IR (KBr): $\nu(CO_2)$1536, 1355, ν(Sn–C) 558 and ν(Sn–O) 469 cm^{-1}. Intensity data were collected at 298 K

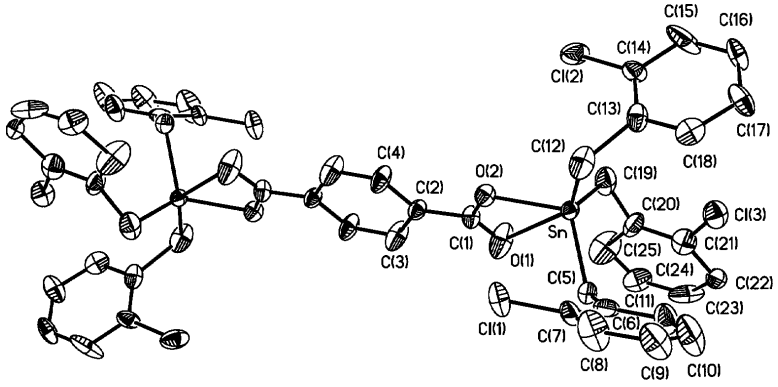

Figure 1. Molecular structure of $(o\text{-}ClC_6H_4)_3SnS_2O_2CC_6H_4CO_2Sn(C_6H_4Cl\text{-}o)_3$. Key geometric parameters: Sn–O1 2.724(11), Sn–O2 2.100(8), Sn–C5 2.053(8), Sn–C12 2.099(12), Sn–C19 2.236(16) Å; O1-Sn–O2 49.9(3), O1–Sn–C5 85.6(4), O1–Sn–C12 147.1(9), O1–Sn–C19 74.0(2), O2–Sn–C5 112.4(3), O2–Sn–C12 97.4(5), O2–Sn–C19 104.0(5), C5–Sn–C12 113.8(7), C5–Sn–C19 108.7(6), C12–Sn–C19 119.6(9)°.

on a Bruker Smart 1000 CCD for a colorless block $0.15 \times 0.30 \times 0.40$ mm^3. $C_{50}H_{40}Cl_6O_4Sn_2$, $M = 1154.90$, triclinic, $P\bar{1}$, $a = 10.372(5)$, $b = 10.921(5)$, $c = 11.141(5)$ Å, $\alpha = 103.495(8)$, $\beta = 92.133(7)$, $\gamma = 8.938(8)°$, $V = 1208.6(9)$ Å^3, $Z = 1$, 3568 unique data ($\theta = 25.0°$), $R = 0.067$ (1472 data with $I>2\sigma(I)$), $wR = 0.159$ (all dataxs); $\rho_{max} = 0.46$ e$^-$ Å^{-3}. Programs used: SHELXL and ORTEP. CCDC deposition number: 223 929.

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