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Crystallographic report

Dichloro(β-methoxycarbonylethyl)tin(IV) isopropylxanthate

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The Sn atom in $CH_3OCOCH_2CH_2SnCl_2[S_2COCH(CH_3)_2]$ adopts a distorted octahedral geometry via the bidentate xanthate ligand and intramolecular carbonyl coordination. Copyright © 2004 John Wiley & Sons, Ltd.

KEYWORDS: crystal structure; organotin; estertin; isopropylxanthate

COMMENT

The structural chemistry of organotin (IV) xanthates continues to be the focus of much research because of their varied structures and biological activities.[1–4] The title compound exists as a discrete molecule which contains a five-membered chelate ring formed via carbonyl coordination to Sn and a four-membered chelate ring from the bidentate xanthate ligand that forms unsymmetric Sn–S bonds [Δ(Sn–S) = 0.2732(1) Å]. The Sn atom is in a distorted octahedral geometry (Fig. 1). The small bite angles [C1-Sn1-O1 74.83(15) and S2-Sn1-S1 68.76(5)°] of the ligands are in part responsible for the distortion of the octahedral geometry.

EXPERIMENTAL

A solution of $KS_2COCH(CH_3)_2$ (1.05 g, 6 mmol) dissolved in ethanol (40 ml) was added dropwise to a solution of β-methoxycarbonylethyltin trichloride (1.87 g, 6 mmol) in the same solvent (40 ml) at room temperature. The reaction mixture was stirred for 1 h and filtered. The filtrate, after distilling off the excess solvent, yielded a crystalline solid, which was recrystallized from trichloromethane/*n*-hexane (1 : 1, v/v). Yield 83.3%, m.p. 96.8–98.0 °C. IR (KBr) ν: 1664 (C=O), 1278, 1226 (C–O), 1024 cm^{-1} (C–S). ^1H NMR (500 MHz, $CDCl_3$) δ: 5.08 (1H, sept, $J = 6.2$ Hz, OCH), 4.00 (3H, s, CH_3O), 2.98 [2H, t, $J = 7.4$ Hz, $J(^{119/117}Sn\text{-}^1H) = 220.2/215.1$ Hz, $COCH_2$], 2.08 [2H, t, $J = 7.4$ Hz, $J(^{119/117}Sn\text{-}^1H) = 112.6/107.8$ Hz, CH_2Sn], 1.48 ppm (6H, d, $J = 6.2$ Hz, $2CH_3$). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 223.11 (C=S), 181.62 [C=O, $J(^{119}Sn\text{-}^{13}C) = 123.4$ Hz], 87.72 (OCH), 55.27 (CH_3O), 33.44 [$SnCH_2$, $J(^{119/117}Sn\text{-}^{13}C) = 961.8/919.2$ Hz], 28.60 [CH_2CO, $J(^{119}Sn\text{-}^{13}C) = 77.9$ Hz], 21.58, 21.49 ppm [$CH(CH_3)_2$]. Analysis calculated for $C_8H_{14}Cl_2O_3S_2Sn$: C 23.33, H 3.42; found: C 23.39, H 3.27%. Intensity data were collected at 293(2) K on a Bruker P4 four-circle diffractometer using a colorless crystal 0.26 × 0.32 × 0.36 mm^3. $C_8H_{14}Cl_2O_3S_2Sn$, $M = 411.90$, monoclinic, $P2_1/n$, $a = 12.094(5)$, $b = 10.523(5)$, $c = 13.266(5)$ Å, $\beta = 113.920(5)$, $V = 1543.3(11)$ Å^3, $Z = 4$, 2719 unique data (θ max 25.0°), $R = 0.043$ (all data), $\omega R = 0.082$ (all data). Programs used: SHELXTL, XSCANS, ORTEP. CCDC deposition no. 214282.

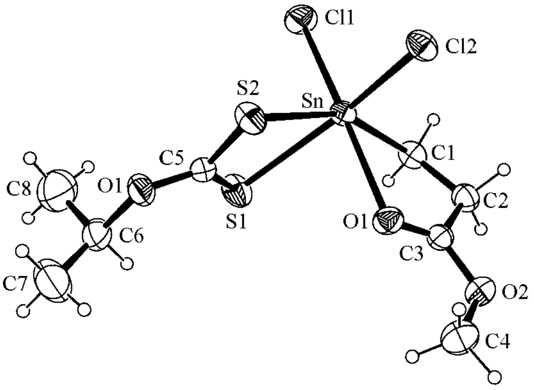

Figure 1. Molecular structure of $CH_3OCOCH_2CH_2SnCl_2[S_2COCH(CH_3)_2]$. Selected geometric parameters: Sn-Cl1 2.3827(16), Sn-Cl2 2.4115(16), Sn-S1 2.7325(17), Sn-S2 2.4593(16), Sn-O1 2.458(3), Sn-C1 2.134(5), S1-C5 1.669(5), S2-C5 1.715(5), O1-C3 1.217(6), O2-C3 1.313(6) Å; Cl1-Sn1-O1 176.61(9), Cl2-Sn-S1 158.99(5), S2-Sn-C1 154.60(13)°.

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