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Crystallographic report

Dicyclohexyltin *N*-[(*E*)-(5-chloro-2-hydroxyphenyl) methylene]-(L)-isoleucinate

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The tin atom in the title compound is in a distorted trigonal bipyramidal geometry and forms a five- and six-membered chelate rings with the tridentate ligand. Copyright © 2004 John Wiley & Sons, Ltd.

KEYWORDS: crystal structure; diorganotin; amino acid; Schiff base

COMMENT

The structural chemistry of organotin complexes with Schiff bases derived from α-amino acids continues to receive attention owing to their biological properties, especially their anti-tumour activities.[1–5] The coordination geometry about the tin atom in the title compound is that of a distorted trigonal bipyramid with two cyclohexyl groups (C14 and C20) and the imino N1 atom in equatorial positions and a unidentate carboxylate group oxygen O1 and a phenoxide O3 atom in axial sites (Fig. 1). The bond angle of O1–Sn–O3 is 153.84(12)° and the sum of trigonal angles is 359.6°. Distortions from the ideal geometry may be rationalized, in part, by the restricted bite angles of the tridentate ligand. Neither of the five- or six-membered rings formed upon chelation are planar, as seen in the following torsion angles: Sn–O1–C1–C2 −4.2(3)°, Sn–N1–C2–C1 19.9(2)°, Sn–O3–C18–C8 −32.2(2)°, and Sn–N1–C7–C8 19.6(3)°. There are no intermolecular tin–oxygen interactions. The above features are comparable to those of reported compounds such as $Ph_2Sn(OC_{10}H_6CH{=}NCH_2COO)$,[3] $Ph_2Sn(OC_6H_4C(CH_3){=}NCH_2COO)$,[6] $Bu_2Sn(OC_6H_4CH{=}NCH(i\text{-}Pr)COO)$,[7] and $Ph_2Sn(OC_6H_4CH{=}NCH_2COO)$.[8]

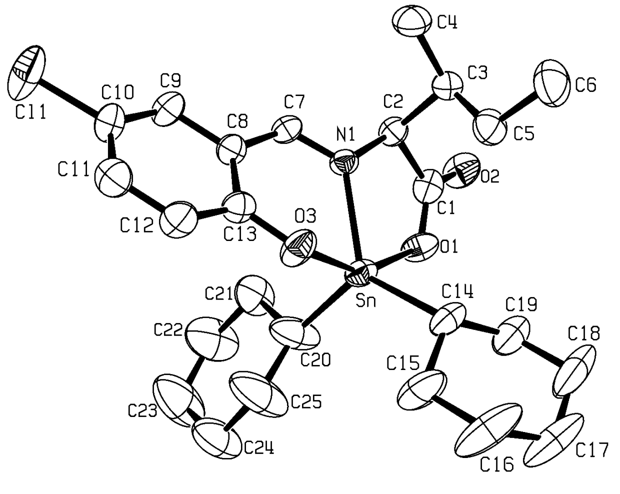

Figure 1. Molecular structure of the title compound. Hydrogen atoms have been removed for clarity. Selected geometric parameters: Sn–O1 2.158(3), Sn–O3 2.108(3), Sn–N1 2.184(3), Sn–C14 2.128(5), Sn–C20 2.074(5), O1–C1 1.284(6), Do–C1 1.231(6), O3–C13 1.314(5) Å; O1–Sn–O3 153.84(12), O1–Sn–N1 73.87(12), O1–Sn–C14 96.11(18), O1–Sn–C20 92.8(2), O3–Sn–N1 80.71(12), O3–Sn–C14 93.74(16), O3–Sn–C20 102.6(3), N1–Sn–C14 125.86(17), N1–Sn–C20 112.42(17), C14–Sn–C20 121.3(2), Sn–O1–C1 120.3(3), Sn–O3–C13 126.1(3)°.

EXPERIMENTAL

Into a dry benzene solution (30 ml), Et_3N (2 mmol), potassium *N*-[(*E*)-(5-chloro-2-hydroxyphenyl)methylene]-(L)-isoleucinate (1.5 mmol) and dicyclohexyltin dichloride (1.5 mmol) were dropped within an hour. The mixture was continued to stir for 3 h at room temperature and then filtered. The yellow filtrate was concentrated in a rotary evaporator to 10 ml and hexane (10 ml) was added. The yellow product obtained was recrystallized from dichloromethane–hexane (1 : 1, v/v). Yield 62%, m.p. 199–200 °C,

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$[\alpha]_D^{25} - 234.2°$. IR (cm^{-1}): ν_{as}(COO) 1668, ν (C=N) 1617, ν_s (COO) 1389. ^1H NMR δ (ppm): 0.97 (t, $J = 7.4$ Hz, 3H, CH_3), 1.06 (d, $J = 6.8$ Hz, 3H, CH_3), 1.23–2.35 (m, 25 H, 2Cy + $CHCH_2$), 3.90 (d, $J = 5.1$ Hz, $J(^{119/117}Sn-^1H) = 35.1/25.5$ Hz, 1H, CHN), 6.78 (d, $J = 9.1$ Hz, 1H, H-3 in Ar), 7.15 (d, $J = 2.6$ Hz, 1H, H-6 in Ar), 7.34 (dd, $J = 2.6$, 9.1 Hz, 1H, H-4 in Ar), 8.19 (s, $J(^{119}Sn-^1H) = 40.6$ Hz, 1H, N=CH). ^{13}C NMR δ (ppm): 173.07 (C=O), 171.23 (CH=N), 168.99, 137.66, 133.70, 124.56, 121.34, 118.00 (Ar), 74.01 (=NCH), 42.42 (CH), 41.71, 39.94 (C-4 in Cy; the cyclohexyl groups are magnetically distinct), 30.52, 30.48, 30.44, 30.33 (C-3 in Cy), 28.93, 28.85, 28.70 (C-2 in Cy, i.e. only three resonances were apparent), 26.75, 26.58 (C-1 in Cy), 25.69 (CH_2), 15.43 (CH_3), 11.98 (CH_3). Intensity data were collected at 293 K on a Rigaku RAXIS-RAPID diffractometer using a yellow block 0.13 × 0.32 × 0.48 mm^3. $C_{25}H_{36}ClNO_3Sn$, $M = 552.69$, orthorhombic, $P2_12_12_1$, $a = 10.2090(4)$, $b = 10.7429(3)$, $c = 23.1681(9)$ Å, $V = 2540.94(16)$ $Å^3$, $Z = 4$, 4550 unique data (θ_{max}25.2°), $R = 0.036$ (all data), $wR = 0.081$ (all data). Flack parameter: −0.03(2). Programs used: SHELXTL, WINGX, ORTEP. CCDC deposition number: 226857.

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