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Crystallographic report

Bis[tris(2-methyl-2-phenylpropyl)tin] piperazinyldithiocarbamate

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The centrosymmetric structure of bis[tris(2-methyl-2-phenylpropyl)tin]piperazinyldithiocarbamate contains four-coordinated tin and monodentate dithiocarbamate ligands.

KEYWORDS: crystal structure; organotin; piperazinyldithiocarbamate

COMMENT

The structural chemistry of triorganotin dithiocarbamates continues to be the focus of much research,[1–4] as these compounds are noted for their biological activity.[5,6] The centrosymmetric title compound adopts a distorted tetrahedral geometry at tin, with each half of the piperazinyldithiocarbamate ligand functioning in a monodentate manner (Fig. 1). The monodentate mode of coordination is confirmed by the disparity in the Sn–S bond distances, i.e. 2.4588(9) Å for Sn–S1 and 3.329(3) Å for Sn–S2, as found in related species.[1–4,7] The coordination geometry is not influenced to a great extent by the close approach of the S2 atom, as all the bond angles at tin are close to the tetrahedral value except for the S1–Sn–C30 angle of 98.20(9)°.

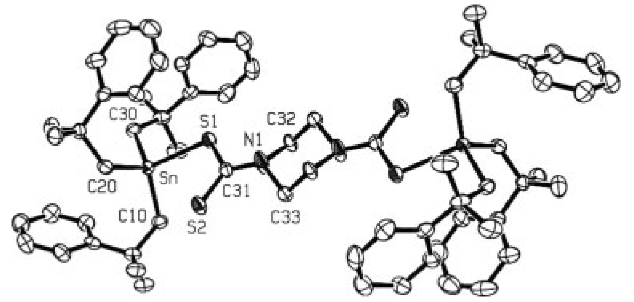

Figure 1. Molecular structure of $(PhC(CH_3)_2CH_2)_3SnS_2CN(CH_2)_2NCS_2Sn(CH_2C(CH_3)_2Ph)_3$; hydrogen atoms have been removed for clarity. Selected geometric parameters: Sn–S1 2.4588(9), Sn–C10 2.170(3), Sn–C20 2.169(3), Sn–C30 2.182(3), S1–C31 1.761(3), S2–C31 1.663(3) Å; S1–Sn–C10 105.90(10), S1–Sn–C20 114.45(10), S1–Sn–C30 98.20(9), C10–Sn–C20 115.75(13), C10–Sn–C30 109.21(14), C20–Sn–C30 111.79(13)°.

EXPERIMENTAL

Anhydrous sodium piperazinyldithiocarbamate (0.28 g, 1 mmol) was added to a trichloromethane solution (30 ml) of tris(2-methyl-2-phenylpropyl)tin chloride (1.11 g, 2 mmol). The reaction mixture was stirred for 5 h at room temperature and filtered. After evaporating under vacuum, a white crystalline material was obtained that was recrystallized from a dichloromethane/*n*-hexane mixture (1:1, v/v). The product (yield 78%, m.p. 173.1–173.6 °C) was then dissolved in dichloromethane/*n*-hexane (1:1, v/v) and, upon slow evaporation, crystals were obtained. ^{1}H NMR ($CDCl_3$, 500 MHz) δ: 1.24 (s, 36H, $12CH_3$), 1.48 (s, $J(^{119}Sn-^1H) = 49.5$ Hz, 12H, $6CH_2Sn$), 4.20 (s, 8H, $4CH_2N$), 7.17–7.29 (m, 30H, $6C_6H_5$). ^{13}C NMR ($CDCl_3$, 125 MHz) δ: 33.16 ($J(^{119}Sn-^{13}C) = 41.8$ Hz, CH_3), 38.12 ($J(^{119/117}Sn-^{13}C) = 328.8/314.5$ Hz, CH_2Sn), 38.58 ($J(^{119}Sn-^{13}C) =$ 19.5 Hz, CCH_2), 50.02 (CH_2N), 125.79, 125.95, 128.41, 150.92 (C_6H_5), 199.76 (CS_2). Intensity data were collected at 293 K on a Bruker SMART CCD diffractometer using a colorless crystal 0.26 × 0.35 × 0.36 mm^3. $C_{66}H_{86}N_2S_4Sn_2$, $M = 1272.99$, triclinic, $P\bar{1}$, $a = 9.3075(17)$, $b = 10.1099(18)$, $c = 19.054(3)$ Å, $\alpha = 91.587(3)$, $\beta = 101.232(3)$, $\gamma = 115.995(2)$, $V = 1567.7(5)$ Å^3, $Z = 1$, 4471 unique data (θ_{max}23.3), $R = 0.033$ (all data), $\omega R = 0.084$ (all data), $\rho_{max} = 0.52$ e$^-$ Å^{-3}. The atoms C32 and C33 in the piperazine ring were found to be disordered over two positions; from refinement, these had 50% occupancy each. The molecules were refined isotropically and hydrogen atoms were not included. Programs used: SHELXTL, WINGX, ORTEP. CCDC deposition number: 224 363.

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