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Crystal structure of (dibenzyl sulphoxide-κO)bis (2-chlorobenzyl-κC1)dichloridotin(IV), C_{28}H_{26}Cl_{4}OSSn

Abstract

C_{28}H_{26}Cl_{4}OSSn, monoclinic, P2_1/c (no. 14), a = 10.6342(2) Å, b = 12.9298(3) Å, c = 20.3576(4) Å, β = 101.820(1)°, V = 2739.78(10) Å³, Z = 4, R_{gt}(F) = 0.0197, wR_{ref}(F²) = 0.0520, T = 100(2) K.

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Source of material

The elemental analysis was performed on a Perkin-Elmer EA2400 CHN analyser. The IR spectrum was recorded using a Perkin-Elmer RX1 spectrophotometer in a Nujol mull between KBr plates. The ^1H and ^13C{^1H} NMR spectra were recorded in CDCl₃ solution on a Bruker AVN FT-NMR spectrometer with chemical shifts relative to Me₄Si for ^1H and CDCl₃ for ^13C{^1H}.

Di(2-chlorobenzyl)tin dichloride was prepared from the direct synthesis of tin powder (Sigma-Aldrich) and 2-chlorobenzyl chloride (Sigma-Aldrich) in toluene [6]. The ligand, dibenzyl sulphoxide (Sigma-Aldrich; 0.46 g, 2.0 mmol) and di(2-chlorobenzyl)tin dichloride (0.44 g, 1 mmol) were heated in 95% ethanol (50 mL) for 0.5 h followed by filtration. The filtrate was evaporated until colourless crystals formed. Yield: 0.43 g (64%), M.pt: 404−406 K. Anal. Calcd. for C_{28}H_{26}Cl_{4}OSSn: C, 50.11; H, 3.91. Found: C, 50.00; H, 3.52. IR (cm⁻¹) 988 (s) ν(S=O), 500 (m) ν(Sn−O). ^1H NMR (CDCl₃, p.p.m.): δ 3.22 (s, 4H, 2-ClPh−CH₂), 3.79 (s, 4H, SCH₂), 7.06−7.18 (m, 8H, 2-ClPh—H), 7.21−7.38 (m, 10H, SCH₂Ph—H). ^13C NMR (CDCl₃, p.p.m.): δ 55.7 (s, −SCH₂), 36.2 (s, 2-ClPh−CH₂), 135.1, 133.3, 130.8, 130.3, 129.2, 129.0, 128.9, 128.7, 127.5, 127.1 (phenyl-C).

Table 1: Data collection and handling.

| Crystal: | Slab, colorless |
| Size: | 0.50 × 0.40 × 0.30 mm |
| Wavelength: | Mo Kα radiation (0.71073 Å) |
| µ: | 1.42 mm⁻¹ |
| Diffractometer, scan mode: | Bruker SMART, ϕ and ω-scans |
| θ_{max}, completeness: | 28.4°, >99% |
| N(hkl)_measured, N(hkl)_unique: | 27156, 6871, 0.015 |
| Criterion for I_{obs}, N(hkl)_gt: | I_{obs} > 2σ(I_{obs}), 6626 |
| N(param)_refined: | 316 |
| Programs: | Bruker programs [1], SHELX [2–4], WinGX and ORTEP [5] |

The title complex is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.
Discussion

In general, the reaction of a diorganotin dichloride precursor with monodentately coordinating acyclic dialkylsulphoxide ligands, e.g. dimethylsulphoxide, leads to the formation of 1:2 adducts in which the tin atom is in a six-coordinate, octahedral environment. Two examples relevant to the present study are trans-Sn₂Cl₄·OSMe₂ [7] and trans-(4-FBz)₂SnCl₄·2OSMe₂ [8]; Bz is benzyl. There are only three examples where mononuclear 1:1 diorganotin dichlorido-sulphoxide adducts have been structurally described in the literature, i.e. Me₂SnCl₂·OSBz₂ [9], Ph₂SnCl₂·OS(Ph)Bz [10] and Ph₂SnCl₂·OS(CH₃)₂ [11]. In the present work, as continuation of studies in this area [8], another example of a 1:1 adduct, (2-CIBz)₂SnCl₂·OSBz₂, is described.

The molecular structure (Figure, 50% displacement ellipsoids) features a five-coordinate tin centre defined by two chloride ligands, the oxygen atom of the OSBz₂ ligand and two substituted benzyl-C atoms. The resultant C₆H₆O donor set is highly distorted. A measurement of the distortion in five-coordinate geometries is τ [12] which has a value of 0.0 for an ideal square-pyramidal geometry and 1.0 for a trigonal bipyramid. In the present case, τ computes to 0.60. One contribution to the distortion is found in the presence of an intramolecular Sn⋯Cl interaction of 3.413(6) Å. There is a clear disparity in the Sn—Cl bond lengths with the Sn—Cl₁ bond [2.3728(4) Å], with the Cl₁ atom approximately opposite to the weakly associated C33 atom, being significantly shorter than the Sn—Cl₂ bond [2.4239(4) Å], which is opposite the sulphoxide-O1 atom [2.3764(11) Å]. The structure of
An analysis of the molecular packing points to the importance of phenyl-C–H···Cl interactions in stabilizing the crystal [C3–H13···Cl1: H13···Cl1 = 2.77 Å, C13···Cl1 = 3.4759(17) Å with angle at H13 = 132°; C19–H19···Cl2: H19···Cl2 = 2.80 Å, C19···Cl2 = 3.6532(17) Å with angle at H19 = 150°; C21–H21···Cl3: H21···Cl3 = 2.81 Å, C21···Cl3 = 3.7048(16) Å with angle at H21 = 158°; symmetry operations: i: x, 3/2–y, 1/2+z; ii: 1–x, 1/2+y, 1/2–z; iii: 1–x, 2–y, –z]. The result of these contacts is the formation of a supramolecular layer in the bc-plane. The only other contacts of note are parallel ring-R–H···Cl and literature structures are, to a first approximation, the same so that the main influence of the coordination of OSBz and SnCl 2 is to elongate the Sn–Cl2 bond; both Sn–Cl bonds are equivalent [2.3740(7) Å] in (2-ClBz) 2 SnCl 2 [13].

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References