THE CRYSTAL STRUCTURE OF TRICYCLOHEXYLTIN N-n-BUTYL DITHIOCARBAMATE

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Figure 1. Molecular structure of $[\text{Cy}_3\text{SnS}_2\text{CNHBU}]$. Selected bond distances and angles: Sn(1)-C(11) 2.147(8), Sn(1)-C(21) 2.157(9), Sn(1)-C(31) 2.105(11), Sn(1)-S(1) 2.472(3), C(1)-S(1) 1.750(9), C(1)-S(2) 1.656(9), C(1)-N(1) 1.342(9); C(11)-Sn(1)-C(21) 107.5(4), C(11)-Sn(1)-C(31) 120.0(4), C(21)-Sn(1)-C(31) 104.9(4), S(1)-Sn(1)-C(11) 110.2(3), S(1)-Sn(1)-C(21) 96.1(3), S(1)-Sn(1)-C(31) 114.8(3), S(1)-C(1)-S(2) 122.7(5), C(1)-S(1)-Sn(1) 99.8(3), N(1)-C(1)-S(1) 114.9(7), N(1)-C(1)-S(2) 122.4(7). H-atoms omitted for clarity.

Comment

Dithiocarbamate (dtc) complexes have been examined extensively in organotin complexes of the type $\text{R}_n\text{Sn}(\text{dtc})_{4-n}$ ($n=1-4$) using various methods such as ^{119}Sn NMR, Infrared and Mössbauer spectroscopies as well as crystallographic techniques [1]. The bidentate behavior of the dithiocarbamate moiety in $\text{R}_2\text{Sn}(\text{IV})$ and $\text{RSn}(\text{IV})$ N, N'-disubstituted dithiocarbamates is well established [1]. The coordination of dithiocarbamate ligands has been reported in the literature as either of bidentate or anisobidentate. Anisobidentate coordination involves the second sulfur atom being partially weakly bonded to the tin atom. Bidentate bonding is exhibited in the crystal structure of $\text{Ph}_2\text{Sn}(\text{S}_2\text{CNEt}_2)_2$, whereas $\text{Ph}_3\text{SnS}_2\text{CNEt}_2$, $\text{Ph}_3\text{SnS}_2\text{CN}(\text{CH}_2)_4$ and other organotin dithiocarbamates display anisobidentate coordination [1-4]. In the crystal structure of tricyclohexyltin N-n-butyl dithiocarbamate, $\text{Cy}_3\text{SnS}_2\text{CNHBU}$, the overall chelating mode of the dithiocarbamate is anisobidentate, as evidenced by the presence of two different Sn-S distances: Sn(1)-S(1), 2.472(3) and Sn(1)-S(2), 3.239(3) Å.

Experimental

Preparation: The title compound was prepared by the slow addition, with stirring, of the sodium salt of N, N'-n-butyl dithiocarbamate (0.35g, 2 mmol) to a solution of tricyclohexyltin chloride (0.77g, 2 mmol) in 50 mL of acetone. The temperature was kept below -20°C using an acetone/liquid nitrogen bath. The resulting solution was then allowed to evaporate at -10°C under vacuum and a white powder was obtained. Colorless prism crystals were then obtained by the slow evaporation of a dilute solution in ether. The melting point for the crystals was $99-101^{\circ}\text{C}$. Elemental analysis (Schwarzkopf Microanalytical Laboratory, USA) calculated (found) for $\text{C}_{23}\text{H}_{43}\text{NS}_2\text{Sn}$: C 53.96 (53.49), H 8.69 (8.39), Sn 22.98 (22.98) %.

Crystallography: Some positional disorder is present in one cyclohexane ring (C31-C36). In light of this, these carbon atoms were refined isotropically.

Table 1. Crystal data for $[\text{C}_{23}\text{H}_{43}\text{NS}_2\text{Sn}]$

Formula	$\text{C}_{23}\text{H}_{43}\text{NS}_2\text{Sn}$	Formula weight	516.39
Crystal system	Monoclinic	Crystal size, mm	$0.046 \times 0.070 \times 0.232$
Space Group	$\text{P}2_1/\text{c}$	a, Å	12.245(5)
b, Å	17.968(5)	c, Å	11.916(5)
β , °	93.796(5)	V, Å ³	2616.0(17)
Z	4	Diffractometer	Bruker, SMART CCD
Temperature, K	293(2)	μ (Mo-K α), cm ⁻¹	11.45
D_{calcd} , g cm ⁻³	1.311	F(000)	1080
θ_{max} , °	25.60	Reflns unique	4190
Reflns with $I \geq 2\sigma(I)$	1855	No. parameters	232
Weighting Scheme	$1/[\sigma^2(\text{Fo}^2) + (0.0440\text{P})^2]$; $\text{P} = (\text{Fo}^2 + 2\text{Fc}^2)/3$	R(F), $R_w(\text{F})$	0.053, 0.113
ρ , e Å ⁻³	0.55	Goodness-of-fit	0.98
Programs used	SIR97 [5], SHELXTL [6], WINGX [7], ORTEP [8]		
Deposition number	CCDC 168717		

Acknowledgements

Financial support from the National Institutes of Health Minority Biomedical Research Support Program (MBRS/SCORE, GM08005) is gratefully acknowledged.

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