X-RAY STRUCTURE OF n-BUTYL-CHLORO-BIS(DIISOBUTYLDITHIOCARBAMATO)TIN(IV)

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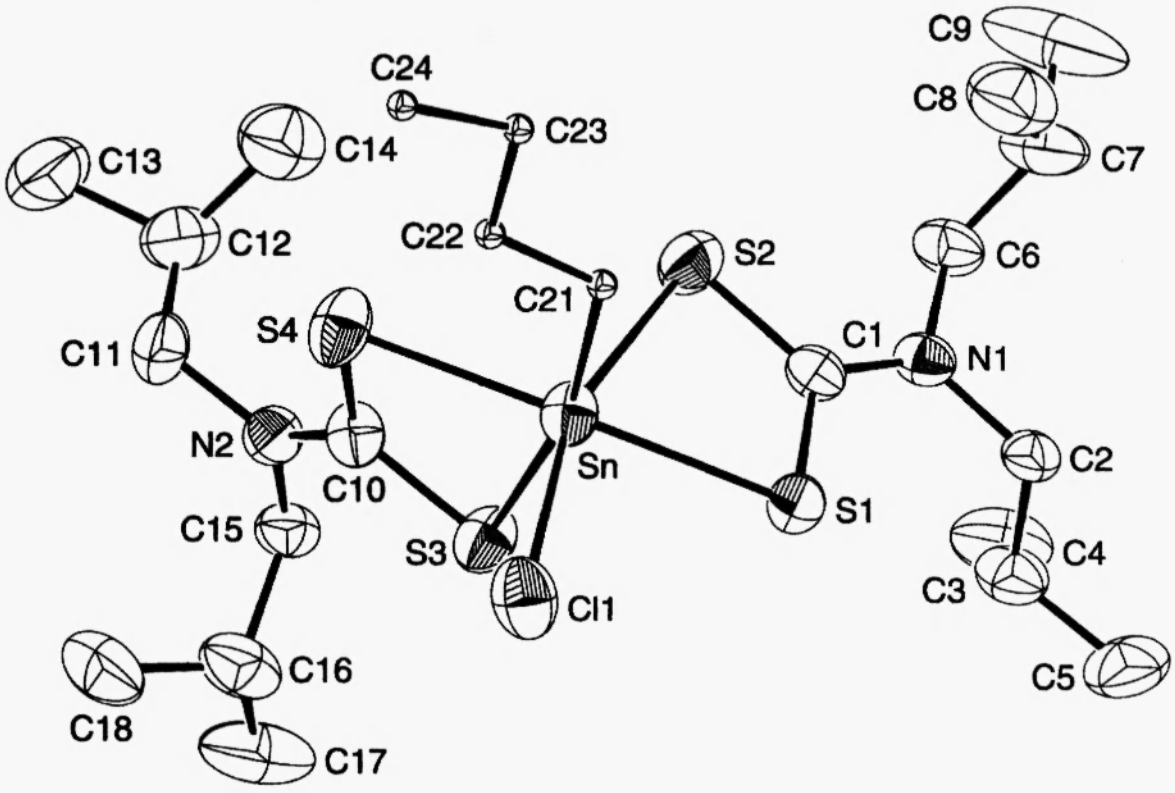

Figure 1. Molecular structure (50% displacement ellipsoids) of [^{n}BuSn($S_2CN^iBu_2$)$_2$Cl]. Selected bond distances and angles: Sn-Cl1 2.469(2), Sn-S1 2.558(1), Sn-S2 2.628(2), Sn-S3 2.520(2), Sn-S4 2.618(2), Sn-C21 2.153(8), S1-C1 1.730(5), S2-C1 1.716(5), S3-C10 1.725(5), S4-C10 1.729(5), C1-N1 1.326(6) and N2-C10 1.322(6) Å; Cl1-Sn-S1 91.24(5), Cl1-Sn-S2 160.56(5), Cl1-Sn-S3 91.12(6), Cl1-Sn-S4 105.08(5), Cl1-Sn-C21 92.2(2), S1-Sn-S2 69.32(4), S1-Sn-S3 87.52(5), S1-Sn-S4 151.92(4), S1-Sn-C2 104.3(2), S2-Sn-S3 87.45(6), S2-Sn-S4 92.62(5), S2-Sn-C21 93.3(2), S3-Sn-S4 69.80(5), S3-Sn-C21 167.7(2), S4-Sn-C21 97.9(2), Sn-S1-C1 87.4(2), Sn-S2-C1 85.5(2), Sn-S3-C10 88.0(2), Sn-S4-C10 84.8(2), S1-C1-S2 117.8(3) and S3-C10-S4 116.7(3)°.

Comment

The tin atom exists in a distorted octahedral geometry defined by a $C_1Cl_1S_4$ donor set so that the C and Cl atoms occupy *cis*-positions. Each of the dithiocarbamato ligands chelates the tin atom forming almost symmetric Sn-S bonds and this is reflected in the near equivalence of the associated S-C bonds. The overall coordination geometry matches closely those found in related RSn($S_2CNR'_2$)$_2$Cl systems [1], *i.e.* R = Ph, R' = Et [2], R' = $^cC_4H_4$ [3]; R = Vin, R' = Et [4], and R = $CH_2CH_2C(=O)OMe$, R' = Me [5].

Experimental

Preparation: The compound was prepared using a literature procedure [6]. Colourless crystals were obtained from the slow evaporation of an ether solution of the compound; m.p. 101-102 °C. ^{1}H NMR: δ 1.95-2.05 m, 1.66-1.77 m, 1.34-1.47 m, 0.89-0.91 m (Cα-Cδ, n-butyl), 3.60 d (CH_2, 3.9 Hz), 2.30-2.44 m (CH), 0.88-0.95 m (CH_3). ^{13}C NMR: δ 46.9, 28.3, 25.2, 13.7 (Cα-Cδ, n-butyl), 199.8 (Cq), 63.5 (CH_2), 27.0 (CH), 20.1 (CH_3). ^{119}Sn NMR: δ -577.7 ppm. IR (KBr disk) ν(C-N) 1494, ν(C-S) 977 cm^{-1}.

Crystallography: The tin-bound n-butyl group was found to be disordered so that two positions were found for the C(22)-C(24) atoms. All atoms of this group were refined isotropically, the disordered components were assigned 50% occupancy factors (from refinement) and hydrogen atoms were not included for this group. An empirical absorption correction was applied [7].

Table 1. Crystallographic data for [^{n}BuSn($S_2CN^iBu_2$)$_2$Cl]

Formula	$C_{22}H_{45}ClN_2S_4Sn$	Formula weight	620.0
Crystal system	orthorhombic	Space group	*Pbca*
a, Å	18.67(1)	*b*, Å	30.123(8)
c, Å	10.830(5)	V, Å^3	6091(4)
Z	8	Crystal size, mm	0.19 x 0.19 x 0.40
Diffractometer	Rigaku AFC7R	Temperature, K	173
μ(Mo-Kα), cm^{-1}	12.13	D_{calcd}, g cm^{-3}	1.352
F(000)	2576	θ_{max}, °	27.5
No. reflns meas., unique	7712, 6980	No. reflns with $I \geq 2\sigma(I)$	3770
R, *wR* (F^2, obs. data)	0.043, 0.113	*R*, *wR* (F^2, all data)	0.118, 0.140
Weighting scheme	$w = 1/[\sigma^2(F_o^2) + (0.0623P)^2 + 5.2587P]$ where $P = (F_o^2 + 2F_c^2)/3$		
No. parameters	264	GoF	1.01
ρ, e Å^{-3}	0.81		
Programs used	DIFABS [7], teXsan [8], DIRDIF [9], SHELXL97 [10], ORTEP [11]		
Deposition no.	CCDC 162239		

Acknowledgements

The Australian Research Council is thanked for support.

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Received: April 20, 2001 - Accepted: April 27, 2001 - Accepted in publishable format: May 2, 2001