Crystal structure of *n*-butyl-chlorido-bis[benzyl(isopropyl)carbamodithioato-$\kappa^2 S:S'$]-tin(IV), $C_{26}H_{37}ClN_2S_4Sn$

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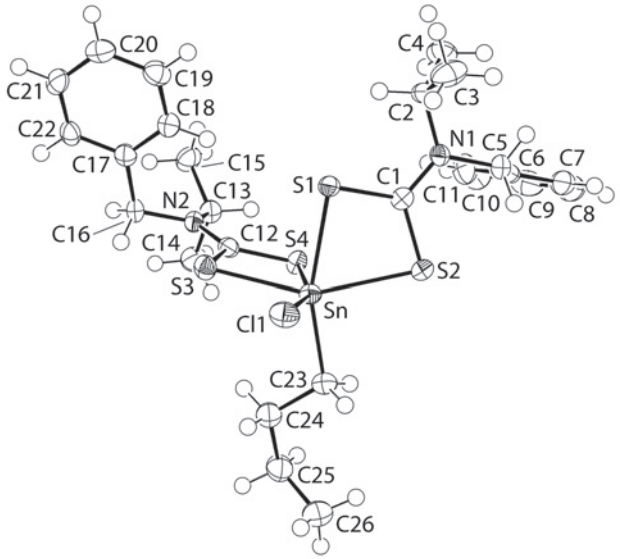

Abstract

$C_{26}H_{37}ClN_2S_4Sn$, monoclinic, *C*2/*c* (no. 15), a = 29.2206(11) Å, b = 10.6749(4) Å, c = 19.6877(7) Å, β = 104.411(4)°, V = 5947.9 Å^3, Z = 8, $R_{gt}(F)$ = 0.0235, $wR_{ref}(F^2)$ = 0.0545, T = 100 K.

Table 1. Data collection and handling.

Crystal:	colourless prisms, size 0.10×0.15×0.20 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	12.48 cm^{-1}
Diffractometer, scan mode:	Agilent Technologies SuperNova Dual diffractometer with Atlas detector, ω
$2\theta_{max}$:	55°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	22095, 6823
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 6062
$N(param)_{refined}$:	312
Programs:	CrysAlis PRO, SHELX, ORTEP-3 [13–15]

Source of material

Carbon disulfide (0.015 mol) was added drop-wise to an ethanol solution (20 ml) of *N*-benzyl-*N*-isopropylamine (0.015 mol). The solution was kept at 277 K for 2 h. *n*-Butyltin trichloride (0.005 mol) dissolved in ethanol (20 ml) was added to give a white precipitate. This was collected and recrystallized from a methanol/dichloromethane (1/2) mixture. Yield: 76%. **m. p.**: 349 K. **^{1}H NMR** ($CDCl_3$, ppm) δ: 2.05 t (Sn–CH_2); 1.76 m, 1.43 m (Bu–CH_2); 0.94 t (Bu–CH_3); 1.21 d (^{i}Pr–CH_3); 5.02 m (^{i}Pr–CH); 5.00 m (Ben–CH_2); 7.32–7.26 m (Ben–C_6H_5). **^{13}C NMR** ($CDCl_3$, ppm) δ: 46.93 (Sn–CH_2); 28.49, 25.33 (Bu–CH_2); 13.84 (Bu–CH_3); 20.49 (^{i}Pr–CH_3); 52.85 (^{i}Pr–CH); 58.45 (Ben–CH_2); 135.39, 128.71, 127.50, 126.55 (Ben–C_6H_5); 200.69 (NCS_2). **IR** (KBr, cm^{-1}): 1480 ν(C–N), 985 ν(C–S_2), 340 ν(Sn–S). UV–Vis ($CHCl_3$, nm): 256 (π-π* transition from NCS_2).

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Experimental details

The C-bound H atoms were geometrically placed (C–H = 0.95-1.00 Å) and refined as riding with U_{iso}(H) = 1.2-1.5U_{eq}(C).

Discussion

Organotin dithiocarbamates continue to attract attention owing to their utility as precursors for chemical vapour deposition (CVD) as well as their various biological activities [1]. These compounds also present fascinating structural diversity in their molecular structures [1]. As revealed in a very recent study [2], structures of the general formula RSn(S_2CN$R'R''$)$_2$Cl, R, R', R'' = alkyl and/or aryl, are exceptional in that they adopt a common structural motif based on $CClS_4$ donor sets that define distorted octahedral geometries. The structure of the title compound was determined as a continuation of these studies. The Sn atom exists within a distorted octahedral geometry defined by a *n*-butyl-C [2.1501(19) Å], Cl [2.4812(5) Å] and four S atoms derived from two almost symmetrically coordinating dithiocarbamate ligands [Sn–S1–S4 = 2.5309(5), 2.6299(5), 2.5301(5) and 2.6387(5) Å]; the longer Sn–S4 bond length has the S4 atom almost *trans* to the Cl atom [158.958(16)°]. The present structure conforms to literature precedents [2–12]. The most prominent intermolecular contacts in the crystal packing are C–H···S [C2–H2···S1^i = 2.85 Å, C2···S1^i = 3.6198(19) Å with an angle at H2 = 134° for i: $-x$, y, ½$-z$] and π···π [Cg(C6–C11)···Cg(C6–C11)^{ii} = 3.7876(12) Å for ii: ½$-x$, ½$-y$, 1$-z$] interactions which link molecules into supramolecular chains aligned along [101].

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(2)	8*f*	0.0289	0.1809	0.3357	0.022
H(3A)	8*f*	0.0232	–0.0354	0.3237	0.048
H(3B)	8*f*	–0.0103	0.0109	0.3718	0.048
H(3C)	8*f*	0.0395	–0.0532	0.4069	0.048
H(4A)	8*f*	0.0590	0.1434	0.4833	0.043
H(4B)	8*f*	0.0101	0.2066	0.4430	0.043
H(4C)	8*f*	0.0590	0.2760	0.4454	0.043
H(5A)	8*f*	0.1505	–0.0008	0.4121	0.024
H(5B)	8*f*	0.1129	0.0096	0.4585	0.024
H(7)	8*f*	0.1970	0.0204	0.5451	0.033
H(8)	8*f*	0.2468	0.1588	0.6204	0.041
H(9)	8*f*	0.2402	0.3736	0.6006	0.040
H(10)	8*f*	0.1851	0.4495	0.5030	0.041
H(11)	8*f*	0.1366	0.3121	0.4251	0.034
H(13)	8*f*	0.1274	0.6641	0.2721	0.023
H(14A)	8*f*	0.1746	0.7237	0.1977	0.040
H(14B)	8*f*	0.1654	0.8429	0.2418	0.040

Table 2. continued.

Atom	Site	*x*	*y*	*z*	U_{iso}
H(14C)	8*f*	0.1368	0.8275	0.1616	0.040
H(15A)	8*f*	0.0620	0.8512	0.2054	0.039
H(15B)	8*f*	0.0893	0.8474	0.2866	0.039
H(15C)	8*f*	0.0494	0.7455	0.2555	0.039
H(16A)	8*f*	0.0612	0.6220	0.0693	0.023
H(16B)	8*f*	0.0574	0.7521	0.1076	0.023
H(18)	8*f*	0.0176	0.4926	0.1883	0.026
H(19)	8*f*	–0.0610	0.4451	0.1832	0.029
H(20)	8*f*	–0.1220	0.5550	0.1075	0.028
H(21)	8*f*	–0.1038	0.7102	0.0352	0.028

Table 2. continued.

Atom	Site	*x*	*y*	*z*	U_{iso}
H(22)	8*f*	–0.0253	0.7560	0.0385	0.024
H(23A)	8*f*	0.2282	0.2495	0.2055	0.026
H(23B)	8*f*	0.2119	0.1342	0.1532	0.026
H(24A)	8*f*	0.1927	0.3835	0.1031	0.032
H(24B)	8*f*	0.1940	0.2606	0.0571	0.032
H(25A)	8*f*	0.2614	0.3847	0.0612	0.035
H(25B)	8*f*	0.2756	0.3788	0.1450	0.035
H(26A)	8*f*	0.2918	0.1642	0.1411	0.054
H(26B)	8*f*	0.3224	0.2408	0.0980	0.054
H(26C)	8*f*	0.2760	0.1669	0.0572	0.054

Table 3. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Sn	8*f*	0.13533(2)	0.23028(2)	0.18163(2)	0.01567(7)	0.01613(7)	0.01571(7)	0.00053(5)	0.00537(5)	–0.00156(5)
Cl(1)	8*f*	0.09865(2)	0.07403(4)	0.09111(2)	0.0259(2)	0.0226(2)	0.0208(2)	–0.0018(2)	0.0077(2)	–0.0074(2)
S(1)	8*f*	0.06772(2)	0.20322(4)	0.23951(2)	0.0139(2)	0.0234(2)	0.0138(2)	0.0009(2)	0.0029(2)	0.0003(2)
S(2)	8*f*	0.16405(2)	0.11341(4)	0.30235(2)	0.0149(2)	0.0205(2)	0.0202(2)	0.0031(2)	0.0049(2)	0.0018(2)
S(3)	8*f*	0.08769(2)	0.40038(5)	0.10643(2)	0.0230(2)	0.0208(2)	0.0147(2)	0.0016(2)	0.0024(2)	–0.0021(2)
S(4)	8*f*	0.15181(2)	0.44846(4)	0.24609(2)	0.0181(2)	0.0166(2)	0.0178(2)	0.0023(2)	0.0011(2)	–0.0005(2)
N(1)	8*f*	0.09574(5)	0.1138(1)	0.37015(7)	0.0152(7)	0.0185(8)	0.0131(7)	0.0005(6)	0.0020(6)	0.0010(6)
N(2)	8*f*	0.09094(5)	0.6173(1)	0.17352(8)	0.0170(7)	0.0165(8)	0.0155(7)	0.0020(6)	0.0034(6)	0.0016(6)
C(1)	8*f*	0.10783(6)	0.1400(2)	0.31147(9)	0.0151(8)	0.0134(9)	0.0160(8)	–0.0013(7)	0.0019(7)	–0.0038(7)
C(2)	8*f*	0.04607(6)	0.1283(2)	0.37601(9)	0.0155(9)	0.023(1)	0.0165(9)	0.0012(7)	0.0055(7)	–0.0009(8)
C(3)	8*f*	0.02256(7)	0.0015(2)	0.3690(1)	0.021(1)	0.025(1)	0.052(1)	–0.0027(8)	0.013(1)	–0.004(1)
C(4)	8*f*	0.04330(7)	0.1944(2)	0.4428(1)	0.029(1)	0.039(1)	0.021(1)	0.0036(9)	0.0108(8)	–0.0057(9)
C(5)	8*f*	0.13024(6)	0.0583(2)	0.43012(9)	0.0194(9)	0.022(1)	0.0169(9)	0.0030(8)	0.0028(7)	0.0045(8)
C(6)	8*f*	0.16152(6)	0.1528(2)	0.47715(9)	0.0153(9)	0.030(1)	0.0146(9)	–0.0013(8)	0.0045(7)	0.0003(8)
C(7)	8*f*	0.19461(7)	0.1079(2)	0.5360(1)	0.021(1)	0.040(1)	0.020(1)	0.0046(9)	0.0038(8)	0.0041(9)
C(8)	8*f*	0.22394(7)	0.1903(3)	0.5810(1)	0.020(1)	0.061(2)	0.019(1)	0.002(1)	–0.0012(8)	0.001(1)
C(9)	8*f*	0.22038(7)	0.3173(2)	0.5692(1)	0.023(1)	0.055(2)	0.022(1)	–0.011(1)	0.0031(8)	–0.010(1)
C(10)	8*f*	0.18776(8)	0.3619(2)	0.5114(1)	0.034(1)	0.034(1)	0.031(1)	–0.009(1)	0.0029(9)	–0.005(1)
C(11)	8*f*	0.15868(7)	0.2801(2)	0.4653(1)	0.027(1)	0.032(1)	0.021(1)	–0.0027(9)	–0.0022(8)	0.0006(9)
C(12)	8*f*	0.10862(6)	0.5025(2)	0.17574(9)	0.0162(9)	0.0183(9)	0.0149(8)	–0.0007(7)	0.0050(7)	0.0003(7)
C(13)	8*f*	0.11208(7)	0.7111(2)	0.2284(1)	0.0218(9)	0.0167(9)	0.0183(9)	0.0006(7)	0.0041(7)	–0.0013(7)
C(14)	8*f*	0.15066(7)	0.7826(2)	0.2053(1)	0.025(1)	0.023(1)	0.033(1)	–0.0038(8)	0.0093(9)	0.0004(9)
C(15)	8*f*	0.07489(7)	0.7964(2)	0.2455(1)	0.027(1)	0.023(1)	0.029(1)	0.0072(8)	0.0070(9)	–0.0014(9)
C(16)	8*f*	0.05467(6)	0.6602(2)	0.11177(9)	0.0202(9)	0.021(1)	0.0156(9)	0.0035(8)	0.0027(7)	0.0061(8)
C(17)	8*f*	0.00433(6)	0.6287(2)	0.11362(9)	0.0219(9)	0.0169(9)	0.0150(9)	0.0023(7)	0.0052(7)	–0.0028(7)
C(18)	8*f*	–0.00697(7)	0.5368(2)	0.1567(1)	0.023(1)	0.022(1)	0.022(1)	0.0035(8)	0.0061(8)	0.0039(8)
C(19)	8*f*	–0.05377(7)	0.5090(2)	0.1539(1)	0.027(1)	0.021(1)	0.027(1)	–0.0016(8)	0.0109(8)	0.0008(8)
C(20)	8*f*	–0.08997(7)	0.5736(2)	0.1090(1)	0.0197(9)	0.029(1)	0.022(1)	–0.0027(8)	0.0060(8)	–0.0104(9)
C(21)	8*f*	–0.07910(7)	0.6654(2)	0.06625(9)	0.022(1)	0.032(1)	0.0142(9)	0.0055(8)	–0.0004(7)	–0.0040(8)
C(22)	8*f*	–0.03234(7)	0.6928(2)	0.06832(9)	0.026(1)	0.020(1)	0.0130(9)	0.0033(8)	0.0042(7)	–0.0004(8)
C(23)	8*f*	0.20464(7)	0.2222(2)	0.1627(1)	0.021(1)	0.023(1)	0.022(1)	0.0031(8)	0.0090(8)	0.0000(8)
C(24)	8*f*	0.20942(7)	0.3033(2)	0.1015(1)	0.021(1)	0.032(1)	0.027(1)	0.0016(9)	0.0076(8)	0.0056(9)
C(25)	8*f*	0.26091(7)	0.3309(2)	0.1020(1)	0.022(1)	0.035(1)	0.030(1)	–0.0023(9)	0.0077(8)	0.007(1)
C(26)	8*f*	0.29036(8)	0.2156(2)	0.0993(1)	0.024(1)	0.050(2)	0.037(1)	0.006(1)	0.013(1)	0.007(1)

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