Crystal structure of bis(*N*-cyclohexyl-*N*-ethyldithiocarbamato)dicyclohexyl tin(IV), [Cy₂Sn(S₂CN(Et)Cy)₂]

V. J. Hall and E. R. T. Tiekink

The University of Adelaide, Department of Chemistry, Australia 5005

Received January 21, 1998, CSD-No. 409205

Source of material: The compound was obtained from the drop-wise addition of [Cy₂SnCl₂] (0.20 g) in hot ethanol (20 cm³) to one molar equivalent of K[S₂CN(Et)Cy] in hot ethanol (20 cm³). After one hour stirring, the solvent was removed in vacuo yielding a white powder. This was recrystallised from a chloroform solution; mp 443 K – 446 K.

The asymmetric mode of coordination of the dithiolate ligands [*d*(Sn–S1, S2, S3, S4) 2.529(1) Å, 2.947(1) Å, 2.539(1) Å and 2.960(1) Å, respectively] and the disposition of the tin-bound cHex groups over the longer Sn–S interactions (C–Sn–C 138.0(2)°) leads to a skew-trapezoidal bipyramidal geometry. This is the common motif for R₂Sn(1,1-dithiolate)₂ compounds (see ref. 1).

C₃₀H₅₄N₂S₄Sn, monoclinic, *P*12₁/*n*1 (No. 14), *a* = 14.129(2) Å, *b* = 16.699(3) Å, *c* = 14.769(2) Å, β = 94.31(1)°, *V* = 3474.8 Å³, *Z* = 4, *R*(*F*) = 0.039, *R*_w(*F*) = 0.035.

Table 1. Parameters used for the X-ray data collection

Crystal:	colorless needle, size 0.06 x 0.08 x 0.45 mm
Wavelength:	Mo K _α radiation (0.7107 Å)
μ:	9.96 cm ⁻¹
Diffractometer:	Rigaku AFC6R
Scan mode:	ω/2θ
<i>T</i> _{measurement} :	293 K
2θ _{max} :	55°
<i>N</i> (<i>hkl</i>) _{unique} :	3794
Criterion for <i>I</i> _o :	<i>I</i> _o > 3 σ(<i>I</i> _o)
<i>N</i> (<i>param</i>) _{refined} :	334
Programs:	SHELXS-86, TEXSAN

Table 2. Final atomic coordinates and displacement parameters (in Å²)

Atom	Site	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{iso}
H(1a)	4e	0.1945	−0.1662	−0.1049	0.059
H(2a)	4e	0.1646	−0.0419	−0.0516	0.079
H(2b)	4e	0.0574	−0.0651	−0.0408	0.079
H(3a)	4e	0.0620	0.0059	−0.1737	0.108
H(3b)	4e	0.1423	−0.0508	−0.2069	0.108
H(4a)	4e	−0.0467	−0.0947	−0.1906	0.114
H(4b)	4e	0.0022	−0.0848	−0.2831	0.114
H(5a)	4e	−0.0093	−0.2203	−0.2424	0.098
H(5b)	4e	0.0977	−0.1944	−0.2492	0.098
H(6a)	4e	0.0081	−0.2137	−0.0862	0.083
H(6b)	4e	0.0897	−0.2693	−0.1190	0.083
H(7a)	4e	0.1626	−0.1626	0.2353	0.057
H(8a)	4e	0.1421	−0.0411	0.1673	0.068
H(8b)	4e	0.2519	−0.0329	0.1538	0.068
H(9a)	4e	0.2192	0.0429	0.2789	0.088
H(9b)	4e	0.1713	−0.0317	0.3235	0.088
H(10a)	4e	0.3667	−0.0184	0.2967	0.097
H(10b)	4e	0.3232	−0.0161	0.3924	0.097
H(11a)	4e	0.3890	−0.1419	0.3652	0.096
H(11b)	4e	0.2789	−0.1488	0.3776	0.096
H(12a)	4e	0.3616	−0.1521	0.2088	0.077
H(12b)	4e	0.3119	−0.2259	0.2532	0.077
H(14a)	4e	−0.2203	−0.3143	0.1617	0.074
H(14b)	4e	−0.1813	−0.2349	0.1191	0.074
H(15a)	4e	−0.1239	−0.1920	0.2639	0.111
H(15b)	4e	−0.1624	−0.2715	0.3066	0.111
H(15c)	4e	−0.2346	−0.2077	0.2620	0.111
H(16a)	4e	−0.0105	−0.4186	0.1670	0.063
H(17a)	4e	−0.1025	−0.4320	0.0360	0.131
H(17b)	4e	−0.1951	−0.4311	0.0901	0.131
H(18a)	4e	−0.0549	−0.5530	0.1026	0.163
H(18b)	4e	−0.1599	−0.5638	0.0587	0.163
H(19a)	4e	−0.1416	−0.6185	0.2050	0.163
H(19b)	4e	−0.2193	−0.5500	0.1983	0.163
H(20a)	4e	−0.0308	−0.5307	0.2707	0.162
H(20b)	4e	−0.1209	−0.5313	0.3285	0.162
H(21a)	4e	−0.1728	−0.4121	0.2632	0.094
H(21b)	4e	−0.0676	−0.4005	0.3061	0.094
H(23a)	4e	0.4840	−0.3578	0.0531	0.079
H(23b)	4e	0.5803	−0.3381	0.0094	0.079
H(24a)	4e	0.4132	−0.3875	−0.0882	0.114
H(24b)	4e	0.5042	−0.4418	−0.0673	0.114
H(24c)	4e	0.5089	−0.3669	−0.1326	0.114
H(25a)	4e	0.5220	−0.1422	−0.0486	0.056
H(26a)	4e	0.6577	−0.2261	0.0631	0.067
H(26b)	4e	0.5918	−0.1569	0.0963	0.067
H(27a)	4e	0.6675	−0.0633	0.0109	0.072
H(27b)	4e	0.7461	−0.1090	0.0731	0.072
H(28a)	4e	0.7908	−0.0963	−0.0758	0.077
H(28b)	4e	0.7829	−0.1875	−0.0478	0.077
H(29a)	4e	0.6431	−0.1028	−0.1549	0.082
H(29b)	4e	0.7099	−0.1694	−0.1921	0.082
H(30a)	4e	0.5583	−0.2233	−0.1695	0.069
H(30b)	4e	0.6377	−0.2673	−0.1065	0.069

Table 3. Final atomic coordinates and displacement parameters (in Å²)

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
Sn	4e	0.18523(2)	-0.19842(2)	0.06627(3)	0.0449(2)	0.0460(2)	0.0482(2)	-0.0020(2)	0.0056(1)	0.0003(2)
S(1)	4e	0.09611(9)	-0.32409(8)	0.1035(1)	0.0497(7)	0.0474(9)	0.064(1)	0.0022(7)	0.0153(7)	0.0028(7)
S(2)	4e	-0.0125(1)	-0.17273(9)	0.1102(1)	0.0501(7)	0.0440(9)	0.082(1)	0.0022(7)	0.0101(8)	0.0056(8)
S(3)	4e	0.31661(9)	-0.2984(1)	0.0429(1)	0.0512(7)	0.0503(9)	0.083(1)	0.0011(8)	0.0193(7)	0.0024(9)
S(4)	4e	0.36606(9)	-0.12849(9)	0.0147(1)	0.0498(7)	0.0509(9)	0.073(1)	0.0026(8)	0.0177(7)	0.0016(8)
N(1)	4e	-0.0798(3)	-0.3147(3)	0.1543(3)	0.045(2)	0.048(3)	0.047(3)	-0.002(2)	0.004(2)	0.005(2)
N(2)	4e	0.4822(3)	-0.2510(3)	-0.0103(3)	0.041(2)	0.045(3)	0.056(3)	0.006(2)	0.005(2)	0.003(2)
C(1)	4e	0.1388(3)	-0.1614(3)	-0.0706(4)	0.047(3)	0.054(3)	0.045(3)	-0.002(3)	0.004(3)	0.001(3)
C(2)	4e	0.1115(4)	-0.0739(4)	-0.0762(4)	0.075(4)	0.056(4)	0.066(4)	0.000(3)	-0.002(3)	0.005(3)
C(3)	4e	0.0857(5)	-0.0486(4)	-0.1737(5)	0.125(6)	0.070(5)	0.073(5)	0.020(5)	0.003(5)	0.015(4)
C(4)	4e	0.0121(5)	-0.1008(5)	-0.2199(5)	0.094(5)	0.120(7)	0.068(5)	0.036(5)	-0.012(4)	0.005(5)
C(5)	4e	0.0420(4)	-0.1876(5)	-0.2153(4)	0.078(4)	0.109(6)	0.056(4)	0.001(5)	-0.011(3)	-0.006(4)
C(6)	4e	0.0654(4)	-0.2150(4)	-0.1185(4)	0.079(4)	0.064(5)	0.064(4)	-0.003(4)	-0.003(3)	-0.008(4)
C(7)	4e	0.2157(3)	-0.1437(3)	0.1974(4)	0.045(3)	0.047(3)	0.051(4)	-0.005(3)	0.002(3)	0.001(3)
C(8)	4e	0.2055(4)	-0.0540(3)	0.1928(4)	0.056(3)	0.055(4)	0.058(4)	0.002(3)	-0.002(3)	-0.006(3)
C(9)	4e	0.2212(4)	-0.0149(4)	0.2860(4)	0.080(4)	0.065(4)	0.067(4)	-0.009(4)	0.010(4)	-0.007(4)
C(10)	4e	0.3167(5)	-0.0390(4)	0.3318(4)	0.101(5)	0.084(5)	0.055(4)	-0.021(5)	-0.016(4)	-0.004(4)
C(11)	4e	0.3259(4)	-0.1287(4)	0.3388(4)	0.087(5)	0.079(5)	0.070(5)	-0.022(4)	-0.025(4)	0.010(4)
C(12)	4e	0.3108(4)	-0.1682(3)	0.2457(4)	0.067(4)	0.057(4)	0.066(4)	-0.004(3)	-0.005(3)	0.006(3)
C(13)	4e	-0.0071(3)	-0.2727(3)	0.1256(3)	0.043(3)	0.045(3)	0.043(3)	0.000(3)	0.005(2)	0.000(3)
C(14)	4e	-0.1702(3)	-0.2745(3)	0.1668(4)	0.038(3)	0.060(4)	0.086(5)	0.003(3)	0.002(3)	0.009(3)
C(15)	4e	-0.1730(4)	-0.2326(4)	0.2581(5)	0.076(4)	0.078(5)	0.129(6)	0.004(4)	0.049(4)	-0.022(5)
C(16)	4e	-0.0766(4)	-0.4030(3)	0.1680(4)	0.058(3)	0.046(4)	0.056(4)	-0.008(3)	0.023(3)	-0.002(3)
C(17)	4e	-0.1287(6)	-0.4463(4)	0.0926(5)	0.197(8)	0.065(5)	0.065(5)	-0.026(6)	0.007(5)	-0.024(4)
C(18)	4e	-0.1205(7)	-0.5371(5)	0.1062(6)	0.21(1)	0.072(6)	0.135(8)	-0.048(7)	0.043(7)	-0.039(6)
C(19)	4e	-0.1522(7)	-0.5615(4)	0.1964(8)	0.163(8)	0.050(5)	0.21(1)	-0.033(5)	0.108(8)	-0.023(6)
C(20)	4e	-0.0974(7)	-0.5164(5)	0.2708(6)	0.187(9)	0.079(6)	0.151(9)	0.025(7)	0.091(7)	0.051(6)
C(21)	4e	-0.1071(5)	-0.4273(4)	0.2588(4)	0.112(5)	0.063(4)	0.061(4)	-0.011(4)	0.019(4)	0.011(4)
C(22)	4e	0.3972(3)	-0.2265(3)	0.0126(3)	0.043(3)	0.052(4)	0.046(3)	-0.001(3)	0.009(2)	0.001(3)
C(23)	4e	0.5116(4)	-0.3362(4)	0.0001(5)	0.062(4)	0.059(4)	0.077(5)	-0.003(3)	0.016(3)	-0.005(4)
C(24)	4e	0.4819(5)	-0.3875(4)	-0.0788(5)	0.100(5)	0.070(5)	0.118(6)	0.006(4)	0.033(5)	-0.001(5)
C(25)	4e	0.5547(3)	-0.1922(3)	-0.0343(3)	0.043(2)	0.053(3)	0.043(3)	0.005(3)	0.007(2)	0.000(3)
C(26)	4e	0.6255(4)	-0.1765(3)	0.0458(3)	0.056(3)	0.067(4)	0.043(3)	-0.003(3)	0.003(3)	-0.001(3)
C(27)	4e	0.6990(4)	-0.1143(4)	0.0223(4)	0.058(3)	0.063(4)	0.058(4)	-0.004(3)	-0.003(3)	-0.001(3)
C(28)	4e	0.7475(4)	-0.1385(4)	-0.0607(4)	0.055(3)	0.076(4)	0.064(4)	-0.003(3)	0.012(3)	0.008(4)
C(29)	4e	0.6766(4)	-0.1524(4)	-0.1403(4)	0.067(4)	0.090(5)	0.049(4)	0.000(4)	0.017(3)	0.005(3)
C(30)	4e	0.6049(3)	-0.2170(3)	-0.1183(3)	0.054(3)	0.075(4)	0.042(3)	-0.001(3)	0.006(3)	-0.005(3)

Acknowledgments. V. J. H. was the holder of a Commonwealth of Australia Postgraduate Research Award. The Australian Research Council is thanked for support.

References

1. Tiekink, E. R. T.: Structural chemistry of tin 1,1-dithiolate complexes. *Main Group Met. Chem.* **15** (1992) 161-186.
2. Sheldrick, G. M.: SHELXS-86. Program for the automatic solution of crystal structure. University of Göttingen, Germany 1986.
3. TEXSAN: Single crystal structure analysis software. Version 1.7.1. Molecular Structure Corporation. The Woodlands, TX, USA 1995.