Crystal structure of 2,6-dithiopurinato-bis[chlorodiphenyltin(IV)] ethanol solvate, $[(C_6H_5)_2SnCl]_2(C_5H_2N_4S_2) \cdot C_2H_5OH$

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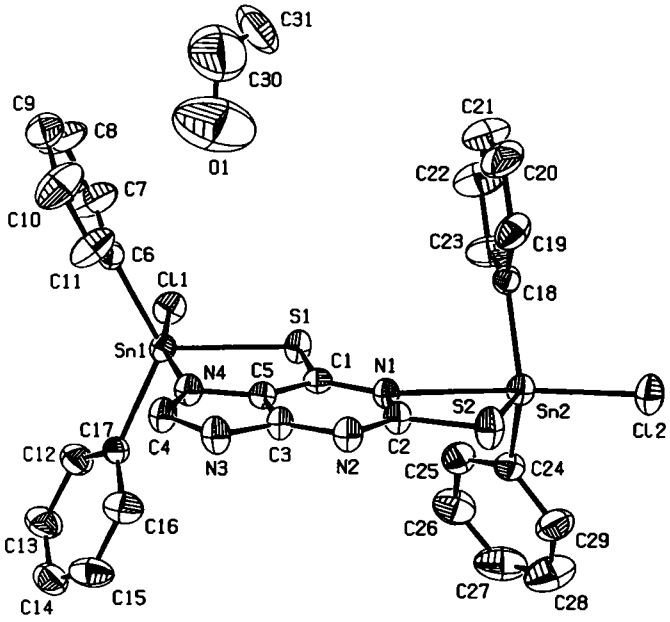

Abstract

$C_{31}H_{28}Cl_2N_4OS_2Sn_2$, triclinic, $P\bar{1}$ (no. 2), $a = 9.526(2)$ Å, $b = 11.078(2)$ Å, $c = 17.245(3)$ Å, $\alpha = 99.475(3)°$, $\beta = 102.564(3)°$, $\gamma = 102.145(3)°$, $V = 1693.8$ Å^3, $Z = 2$, $R_{gt}(F) = 0.039$, $wR_{ref}(F^2) = 0.125$, $T = 298$ K.

Source of material

The reaction was carried out under nitrogen atmosphere using of standard Schlenk technique. 2,6-dithiopurine (0.184 g, 1 mmol) and sodium ethoxide (0.136 g, 2 mmol) were added to ethanol, stirred for 10 min, then Ph_2SnCl_2 (0.344 g, 1 mmol) added to the mixture being kept reacting for 12 h at 40 °C. After cooling down to room temperature, product was filtered. Solvent of the filtrate was removed under vacuum and the solid product was obtained. Solid was recrystallized from ethanol and colorless crystals were formed (m.p. > 200 °C).

Elemental analysis: found – C, 44.19 %; H, 3.34 %; N, 6.72 %; calc. for $C_{31}H_{27}Cl_2N_4OS_2Sn_2$ – C, 44.06 %; H, 3.34 %; N, 6.63 %. ^{1}H NMR and IR data are available in the CIF file.

Discussion

The coordination chemistry of tin is extensive and highly interesting with various coordination numbers and environments [1,2]. To understand the nature of versatile bonding modes of organotin purinates and to study their biological activities [3-5], we prepared the complex $(Ph_2SnCl)_2(C_5H_2N_4S_2) \cdot EtOH$ with a dinuclear structure. The ligand is tetradentate and chelates to tin atoms with two deprotonated heterocyclic thioamide groups (N–*R*–S)-. The environments of tin atoms are distorted trigonal bipyramidal. Two dinuclear complexes are linked with each other through two intermolecular hydrogen bonds, N3–H3···N2′ and N3′–H3′···N2, with d(N3–H3···N2′) = 2.842 Å and ∠N3–H3···N2′ = 163.8°.

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.31 × 0.38 × 0.42 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	17.86 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{max}$:	50.04°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	8969, 5914
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 4168
$N(param)_{refined}$:	379
Programs:	SHELXS-97 [6], SHELXL-97 [7]

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

Atom	Site	x	y	z	U_{iso}
H(3)	2*i*	0.5042	1.1173	0.0590	0.068
H(1)	2*i*	0.2049	0.1971	0.1369	0.473
H(4)	2*i*	0.5662	1.2604	0.1895	0.067
H(7)	2*i*	0.6618	1.1656	0.5314	0.119
H(8)	2*i*	0.4439	1.1663	0.5692	0.146
H(9)	2*i*	0.2361	1.1808	0.4798	0.115
H(10)	2*i*	0.2472	1.1953	0.3509	0.144
H(11)	2*i*	0.4610	1.1904	0.3106	0.111
H(12)	2*i*	1.0447	1.3859	0.4813	0.078
H(13)	2*i*	1.2180	1.5554	0.4700	0.096
H(14)	2*i*	1.2220	1.5923	0.3424	0.096
H(15)	2*i*	1.0611	1.4529	0.2280	0.098
H(16)	2*i*	0.8856	1.2867	0.2425	0.084
H(19)	2*i*	0.4767	0.4949	0.1465	0.090
H(20)	2*i*	0.3437	0.4397	0.2377	0.134
H(21)	2*i*	0.4585	0.4734	0.3724	0.126
H(22)	2*i*	0.7028	0.5640	0.4187	0.131
H(23)	2*i*	0.8429	0.6208	0.3320	0.099
H(25)	2*i*	1.0300	0.8140	0.2883	0.075
H(26)	2*i*	1.2842	0.9015	0.3321	0.100
H(27)	2*i*	1.4355	0.8462	0.2522	0.138
H(28)	2*i*	1.3285	0.7139	0.1249	0.151
H(29)	2*i*	1.0766	0.6234	0.0831	0.097
H(30A)	2*i*	0.0279	0.0884	0.1899	0.304
H(30B)	2*i*	0.0203	–0.0228	0.1186	0.304
H(31A)	2*i*	–0.0226	0.2045	0.0699	0.249
H(31B)	2*i*	–0.1574	0.1249	0.0934	0.249
H(31C)	2*i*	–0.1013	0.0656	0.0207	0.249

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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(1)	2*i*	0.78550(4)	1.16758(4)	0.38201(2)	0.0436(3)	0.0432(3)	0.0379(2)	0.0135(2)	0.0094(2)	0.0063(2)
Sn(2)	2*i*	0.79859(5)	0.61859(4)	0.14659(3)	0.0543(3)	0.0494(3)	0.0397(3)	0.0178(2)	0.0065(2)	0.0076(2)
Cl(1)	2*i*	0.9290(2)	1.1513(2)	0.51265(9)	0.060(1)	0.068(1)	0.0385(8)	0.0200(8)	0.0055(7)	0.0111(7)
Cl(2)	2*i*	0.7928(2)	0.4149(2)	0.0695(1)	0.098(2)	0.060(1)	0.069(1)	0.031(1)	0.016(1)	–0.0040(9)
N(1)	2*i*	0.7278(5)	0.8277(4)	0.1721(3)	0.050(3)	0.041(3)	0.036(3)	0.018(2)	0.003(2)	0.008(2)
N(2)	2*i*	0.5902(6)	0.8964(5)	0.0591(3)	0.062(3)	0.055(3)	0.034(3)	0.023(3)	–0.003(2)	0.009(2)
N(3)	2*i*	0.5498(6)	1.1035(5)	0.1042(3)	0.073(4)	0.055(3)	0.042(3)	0.032(3)	–0.005(3)	0.015(3)
N(4)	2*i*	0.6521(6)	1.1335(5)	0.2374(3)	0.053(3)	0.049(3)	0.040(3)	0.021(3)	0.003(2)	0.011(2)
O(1)	2*i*	0.193(2)	0.122(2)	0.139(1)	0.15(1)	0.48(3)	0.25(2)	0.04(1)	–0.01(1)	0.04(2)
S(1)	2*i*	0.8139(2)	0.9530(2)	0.32915(9)	0.063(1)	0.0491(9)	0.0312(8)	0.0266(8)	0.0031(7)	0.0074(6)
S(2)	2*i*	0.6534(2)	0.6734(2)	0.0291(1)	0.098(2)	0.062(1)	0.0355(9)	0.036(1)	–0.0067(9)	–0.0018(8)
C(1)	2*i*	0.7313(6)	0.9307(5)	0.2269(3)	0.040(3)	0.046(3)	0.033(3)	0.011(3)	0.007(2)	0.013(3)
C(2)	2*i*	0.6566(7)	0.8151(6)	0.0931(4)	0.059(4)	0.049(4)	0.037(3)	0.020(3)	0.002(3)	0.006(3)
C(3)	2*i*	0.6003(7)	1.0000(6)	0.1151(4)	0.051(4)	0.046(4)	0.040(3)	0.019(3)	–0.001(3)	0.004(3)
C(4)	2*i*	0.5863(8)	1.1815(6)	0.1800(4)	0.069(4)	0.054(4)	0.044(4)	0.028(3)	0.005(3)	0.007(3)
C(5)	2*i*	0.6630(6)	1.0191(5)	0.1973(3)	0.038(3)	0.042(3)	0.034(3)	0.009(3)	0.004(2)	0.006(3)
C(6)	2*i*	0.5852(7)	1.1736(6)	0.4159(4)	0.044(4)	0.039(3)	0.060(4)	0.011(3)	0.017(3)	0.004(3)
C(7)	2*i*	0.578(1)	1.170(1)	0.4932(6)	0.096(7)	0.158(9)	0.090(6)	0.073(7)	0.055(6)	0.062(6)
C(8)	2*i*	0.447(1)	1.171(1)	0.5162(7)	0.116(9)	0.18(1)	0.125(9)	0.075(9)	0.087(8)	0.077(9)
C(9)	2*i*	0.323(1)	1.1801(9)	0.4638(8)	0.061(6)	0.093(7)	0.14(1)	0.022(5)	0.050(6)	0.018(7)
C(10)	2*i*	0.330(1)	1.188(1)	0.3879(7)	0.065(6)	0.18(1)	0.108(9)	0.052(7)	0.020(6)	0.003(8)
C(11)	2*i*	0.4595(9)	1.185(1)	0.3637(5)	0.068(5)	0.149(9)	0.070(5)	0.049(6)	0.025(4)	0.013(5)
C(12)	2*i*	1.0468(8)	1.3999(6)	0.4298(5)	0.072(5)	0.054(4)	0.064(5)	0.003(4)	0.020(4)	0.010(4)
C(13)	2*i*	1.1508(9)	1.5016(7)	0.4236(6)	0.072(5)	0.066(5)	0.089(6)	–0.011(4)	0.026(5)	0.009(4)
C(14)	2*i*	1.1539(9)	1.5225(8)	0.3477(6)	0.067(5)	0.067(5)	0.128(8)	0.027(4)	0.044(6)	0.046(5)
C(15)	2*i*	1.0568(9)	1.4408(9)	0.2796(6)	0.076(6)	0.104(7)	0.080(6)	0.020(5)	0.028(5)	0.055(5)
C(16)	2*i*	0.9532(8)	1.3409(8)	0.2886(5)	0.058(4)	0.084(5)	0.070(5)	0.016(4)	0.012(4)	0.033(4)
C(17)	2*i*	0.9473(7)	1.3192(6)	0.3639(4)	0.048(4)	0.041(3)	0.056(4)	0.019(3)	0.020(3)	0.018(3)
C(18)	2*i*	0.6761(7)	0.5652(6)	0.2287(4)	0.044(4)	0.044(3)	0.053(4)	0.012(3)	0.013(3)	0.006(3)
C(19)	2*i*	0.5248(8)	0.5098(8)	0.2015(5)	0.054(5)	0.087(6)	0.072(5)	0.016(4)	0.015(4)	–0.008(4)
C(20)	2*i*	0.446(1)	0.477(1)	0.2563(8)	0.066(6)	0.116(8)	0.15(1)	0.008(5)	0.058(7)	–0.005(8)
C(21)	2*i*	0.513(1)	0.497(1)	0.3364(7)	0.095(8)	0.14(1)	0.098(8)	0.026(7)	0.057(7)	0.031(7)
C(22)	2*i*	0.657(1)	0.550(1)	0.3634(6)	0.089(7)	0.17(1)	0.086(7)	0.044(7)	0.037(6)	0.054(7)
C(23)	2*i*	0.7412(9)	0.5843(9)	0.3115(5)	0.054(5)	0.128(8)	0.068(5)	0.018(5)	0.016(4)	0.041(5)
C(24)	2*i*	1.0284(7)	0.7083(6)	0.1826(4)	0.057(4)	0.053(4)	0.054(4)	0.015(3)	0.020(3)	0.017(3)
C(25)	2*i*	1.0906(8)	0.7920(7)	0.2557(4)	0.060(5)	0.066(5)	0.061(4)	0.016(4)	0.016(4)	0.013(4)
C(26)	2*i*	1.2432(9)	0.8448(8)	0.2820(5)	0.066(5)	0.089(6)	0.079(6)	–0.001(5)	0.005(5)	0.019(5)
C(27)	2*i*	1.333(1)	0.814(1)	0.2342(8)	0.071(6)	0.14(1)	0.13(1)	–0.008(6)	0.034(7)	0.039(8)
C(28)	2*i*	1.269(1)	0.733(1)	0.1589(8)	0.090(8)	0.17(1)	0.14(1)	0.029(8)	0.061(8)	0.035(9)
C(29)	2*i*	1.118(1)	0.6792(8)	0.1335(5)	0.083(6)	0.092(6)	0.072(5)	0.018(5)	0.039(5)	0.011(5)
C(30)	2*i*	0.042(2)	0.069(2)	0.136(1)	0.29(3)	0.24(2)	0.32(3)	0.09(2)	0.19(3)	0.11(2)
C(31)	2*i*	–0.071(2)	0.121(1)	0.074(1)	0.16(1)	0.19(1)	0.22(2)	0.11(1)	0.11(1)	0.05(1)

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