SHAVER, A., J. M. McCALL, P. H. BIRD AND U. SIRIWARDANE   661

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A Dimeric Stannoxane Structure: [Sn2(Cl)(O)(OH)(C6H5)4]2.2C3H7NO

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Abstract. Bis[chloro-1κCl-μ-hydroxo-μ-oxo-(tetraphenyl-1$\kappa^2 C$,2$\kappa^2 C$)ditin]-N,N-dimethylformamide (1/2), [Sn$_2$(Cl)(O)(OH)(C$_6$H$_5$)$_4$]$_2$.2C$_3$H$_7$NO, $M_r =$ 1374·6, orthorhombic, *Pbca*, $a = 17·231$ (4), $b = 17·552$ (2), $c = 18·345$ (3) Å, $V = 5548$ (3) Å^3, $Z = 4$ (dimers), $D_x = 1·646$ Mg m^{-3}, Mo $K\alpha$ radiation, $\lambda = 0·7107$ Å, $\mu = 1·770$ mm^{-1}, $F(000) = 2704$, $T = 293$ (1) K, $R = 0·022$ for 2245 observed reflections. The title compound adopts the familiar tetraorganostannoxane structural motif with a centrosymmetric R_4Sn$_2$O$_2$ moiety connected to two exocyclic R_2SnCl units. The two pairs of Sn atoms are also linked by hydroxy bridges such that each Sn atom exists in a distorted trigonal bipyramidal geometry. For the endocyclic Sn atom, the hydroxy O atom and stannoxane O atom occupy approximate *trans* positions and for the other Sn atom, the axial sites are occupied by the hydroxy O atom and the Cl atom. Connected to the hydroxy H atoms at each end of the 'ladder' structure, *via* hydrogen bonds, are two dimethylformamide molecules of solvation.

Experimental. The title compound is a hydrolysis product (Vollano, Day & Holmes, 1984) of (C$_6$H$_5$)$_2$SnCl$_2$ which was obtained as colourless crystals from a reaction between (C$_6$H$_5$)$_2$SnCl$_2$ (Aldrich) and the sodium salt of pipecolic acid (Sigma) performed in dimethylformamide solution. Enraf–Nonius CAD-4F diffractometer controlled by a PDP8/A computer, graphite-monochromated Mo $K\alpha$ radiation; ω:2θ scan technique. Cell parameters by least squares on 25 reflections ($3 \leq \theta \leq 9°$) (de Boer & Duisenberg, 1984) on a spherical crystal of 0·30 mm diameter, no absorption correction applied. There was significant decomposition of the crystal during the data collection as judged by the fall in the net intensities of two reference reflections ($\overline{2}3\overline{4}$ and $0\overline{4}0$) measured every 7200 s. The data collection was stopped when the average intensity

values were 80% of the initial values. This gave a total of 4084 reflections ($1·5 \leq \theta \leq 22·5°$) measured in the range $-18 \leq h \leq 0$, $-18 \leq k \leq 0$, $-19 \leq l < 1$. 3630 unique reflections (R_{amal} 0·052) and 2245 satisfied $I \geq 2·5\sigma(I)$. Structure solved by Patterson method, full-matrix least-squares refinement on 309 parameters based on F (Sheldrick, 1976). Anisotropic thermal parameters for non-H atoms and H atoms included at their calculated positions except for the hydroxy H atom, H(2), which was located from a difference map but not refined. At convergence, $R =$

Table 1. *Fractional atomic coordinates and B_{eq} values (Å^2)*

$$B_{eq} = 8\pi^2(U_{11} + U_{22} + U_{33})/3.$$

	x	y	z	B_{eq}
Sn(1)	0·04713 (2)	−0·06808 (2)	0·34159 (2)	3·97
Sn(2)	−0·06489 (2)	−0·07016 (2)	0·49161 (2)	3·48
O(1)	0·0253 (2)	−0·0180 (2)	0·4387 (2)	3·69
O(2)	0·0487 (2)	−0·1279 (2)	0·3901 (2)	4·48
Cl	0·1528 (1)	0·0272 (1)	0·3277 (1)	5·22
C(111)	−0·0200 (3)	−0·0267 (3)	0·2523 (3)	4·29
C(112)	−0·0948 (3)	−0·0498 (4)	0·2411 (4)	5·80
C(113)	−0·1373 (4)	−0·0218 (5)	0·1826 (4)	7·11
C(114)	−0·1082 (4)	0·0299 (5)	0·1369 (4)	6·82
C(115)	−0·0337 (5)	0·0540 (5)	0·1478 (4)	9·28
C(116)	0·0093 (4)	0·0273 (5)	0·2056 (3)	7·73
C(121)	0·1246 (3)	−0·1621 (4)	0·3502 (3)	5·15
C(122)	0·1094 (4)	−0·2198 (4)	0·3983 (4)	6·64
C(123)	0·1656 (6)	−0·2764 (5)	0·4123 (5)	9·41
C(124)	0·2349 (6)	−0·2737 (6)	0·3753 (7)	10·02
C(125)	0·2499 (5)	−0·2175 (6)	0·3285 (6)	10·68
C(126)	0·1960 (4)	−0·1610 (4)	0·3148 (4)	7·18
C(211)	−0·1781 (3)	−0·0290 (3)	0·4675 (3)	4·14
C(212)	−0·2262 (3)	−0·0701 (4)	0·4230 (4)	6·24
C(213)	−0·3016 (4)	−0·0450 (4)	0·4093 (4)	7·94
C(214)	−0·3286 (4)	0·0186 (5)	0·4415 (4)	7·64
C(215)	−0·2814 (4)	0·0618 (4)	0·4850 (4)	7·29
C(216)	−0·2057 (4)	0·0375 (4)	0·4989 (3)	5·52
C(221)	−0·0387 (3)	−0·1654 (3)	0·5573 (3)	4·41
C(222)	−0·0067 (4)	−0·1577 (4)	0·6259 (4)	6·01
C(223)	0·0080 (5)	−0·2209 (5)	0·6690 (4)	8·08
C(224)	−0·0076 (5)	−0·2926 (5)	0·6449 (6)	9·37
C(225)	−0·0391 (5)	−0·3014 (5)	0·5760 (6)	9·88
C(226)	−0·0564 (4)	−0·2385 (4)	0·5333 (4)	6·58
OF(1)	−0·1347 (3)	−0·2534 (3)	0·3559 (3)	9·98
NF(1)	−0·2464 (3)	−0·3173 (3)	0·3398 (3)	6·14
CF(1)	−0·1735 (4)	−0·3021 (4)	0·3260 (4)	6·59
CF(2)	−0·2892 (4)	−0·3764 (4)	0·3005 (4)	6·94
CF(3)	−0·2901 (5)	−0·2718 (4)	0·3921 (5)	9·85

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Table 2. *Selected interatomic distances (Å) and bond angles (°)*

Sn(1)—O(1)	2·021 (3)		Sn(1)—O(2)	2·149 (3)
Sn(1)—Cl	2·485 (1)		Sn(1)—C(111)	2·132 (5)
Sn(1)—C(121)	2·129 (6)		Sn(2)—O(1)	2·048 (3)
Sn(2)—O(2)	2·138 (3)		Sn(2)—O(1')	2·121 (3)
Sn(2)—C(211)	2·127 (5)		Sn(2)—C(221)	2·110 (5)
O(2)···OF(1)	2·729 (7)		H(2)···OF(1)	1·80 (1)

O(1)—Sn(1)—O(2)	72·8 (1)		O(1)—Sn(1)—Cl	86·2 (1)
O(1)—Sn(1)—C(111)	115·3 (1)		O(1)—Sn(1)—C(121)	112·9 (2)
O(2)—Sn(1)—Cl	159·0 (1)		O(2)—Sn(1)—C(111)	93·9 (2)
O(2)—Sn(1)—C(121)	94·2 (2)		Cl—Sn(1)—C(111)	95·1 (2)
Cl—Sn(1)—C(121)	94·0 (2)		C(111)—Sn(1)—C(121)	131·4 (2)
O(1)—Sn(2)—O(2)	72·5 (1)		O(1)—Sn(2)—O(1')	73·4 (3)
O(1)—Sn(2)—C(211)	116·5 (2)		O(1)—Sn(2)—C(221)	117·5 (2)
O(1')—Sn(2)—O(2)	146·0 (1)		O(1')—Sn(2)—C(211)	99·9 (3)
O(1')—Sn(2)—C(221)	99·5 (3)		O(2)—Sn(2)—C(211)	95·8 (2)
O(2)—Sn(2)—C(221)	95·4 (2)		C(211)—Sn(2)—C(221)	125·8 (2)
Sn(1)—O(1)—Sn(2)	111·4 (1)		Sn(1)—O(1)—Sn(2')	142·1 (1)
Sn(2)—O(1)—Sn(2')	106·6 (1)		Sn(1)—O(2)—Sn(2)	103·3 (1)
O(2)—H(2)···OF(1)	159 (1)			

Primed atoms are related by centre of inversion.

$0·022$, $wR = 0·025$, $w = [\sigma^2(F) + 0·001|F|^2]^{-1}$, $S = 1·16$, $(\Delta/\sigma)_{max} \leq 0·002$, $\Delta\rho_{max} = 0·36$, $\Delta\rho_{min} = -0·40\,e\,\text{Å}^{-3}$; no extinction correction applied. Scattering factors for all atoms were as incorporated in *SHELX*76 (Sheldrick, 1976). All calculations were performed on a Sun4/280 computer system. Atomic parameters are given in Table 1, selected interatomic parameters in Table 2* and the numbering scheme used is shown in Fig. 1, which was drawn with *ORTEP* (Johnson, 1971) at 15% probability levels.

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and all interatomic parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53533 (12 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
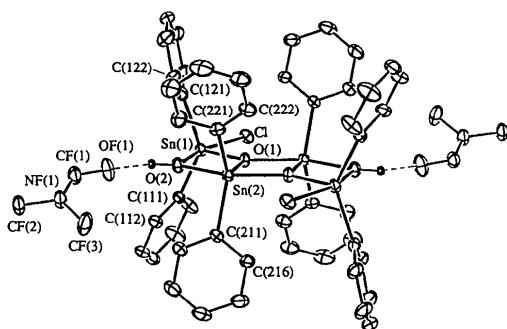
Fig. 1. Molecular structure and crystallographic numbering scheme employed for [Sn$_2$(Cl)(O)(OH)(C$_6$H$_5$)$_4$]$_2$.2C$_3$H$_7$NO (Johnson, 1971).

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Structure of 1,4,5,8,9,10-Hexabromo-11,11-dimethoxytricyclo[6.2.1.0²,⁷]undeca-4,9-diene-3,6-dione

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Abstract. C$_{13}$H$_8$Br$_6$O$_4$, $M_r = 707·7$, orthorhombic, $Pna2_1$, $a = 12·711$ (5), $b = 16·921$ (4), $c = 8·455$ (3) Å, $V = 1818·5$ (11) Å^3, $Z = 4$, $D_x = 2·584$ g cm^{-3}, $\lambda(\text{Mo }K\alpha) = 0·71073$ Å, $\mu = 131·4$ cm^{-1}, $F(000) = 1312$, $T = 100$ (4) K, final $R = 0·043$ for 883 reflections with $I > 3\sigma(I)$. The title compound is an *endo* addition product of 2,3-dibromobenzoquinone and 1,2,3,4-tetrabromo-5,5-dimethoxycyclopentadiene. The two remaining double bonds are parallel and separated by