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Structure of Dimeric Dichlorotetramethyldistannoxane,* $(C_4H_{12}Cl_2OSn_2)_2$

BY R. GRAZIANI
Istituto di Chimica Generale, Università di Padova, Italy

U. CASELLATO
Istituto di Chimica e Tecnologia dei Radioelementi del CNR, Padova, Italy

AND G. PLAZZOGNA
Istituto di Chimica Analitica, Università di Padova, Italy

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Abstract. $M_r = 768{\cdot}6$, monoclinic, $P2_1/c$, $a = 7{\cdot}32$ (1), $b = 18{\cdot}56$ (2), $c = 8{\cdot}73$ (1) Å, $\beta = 110{\cdot}00$ (5)°, $V = 1115$ (1) Å^3, D_m(flotation) $= 2{\cdot}30$, $D_x = 2{\cdot}29$ Mg m^{-3} for $Z = 2$ (the molecule is dimeric and there are four asymmetric units in the cell), Mo $K\alpha$, $\lambda = 0{\cdot}71069$ Å, $\mu = 52{\cdot}4$ cm^{-1}, $F(000) = 712$, room temperature. The structure was solved by standard methods from diffractometer data and refined to a conventional R of 6·1% for 1798 observed reflections. The centrosymmetric dimer consists of a central Sn_2O_2 ring with two additional adjacent Sn_2OCl four-membered rings. The Sn, O, and Cl atoms are approximately coplanar. The Sn atoms are pentacoordinate, approximately trigonal bipyramidal.

Introduction. Several structural studies of dialkyltin derivatives have been prompted in view of the biological activity displayed by some of these compounds and we recently reported the crystal structure of dichlorodimethylbis(3,5-dimethylpyrazole-N^2)tin(IV), $SnMe_2Cl_2(dmp)_2$ (Graziani, Casellato, Ettorre & Plazzogna, 1982).

Experimental. Very stable colourless crystals of the title compound were obtained as a hydrolysis product during the preparation of the above complex. Crystal $0{\cdot}1 \times 0{\cdot}2 \times 0{\cdot}2$ mm, Philips PW 1100 four-circle diffractometer; lattice parameters by least-squares refinement of 25 medium-angle settings. Integrated intensities up to $\theta = 25°$, $h_{\max} = \pm 8$, $k_{\max} = 22$, $l_{\max} = 10$, $\theta/2\theta$ scan mode, graphite-monochromated Mo $K\alpha$ radiation; standard reflections: no variation. 2005 unique reflections measured, 1798 with $I > 3\sigma(I)$ used; intensities corrected for Lorentz and polarization factors, and for absorption (North, Phillips & Mathews, 1968), $0{\cdot}26 < \mu R < 0{\cdot}54$. The diffraction effects are dominated by the heavy-atom contributions and the structure was solved by Patterson and Fourier methods. Full-matrix least-squares anisotropic refinement converged to $R = 6{\cdot}1\%$; $\sum w(k|F_o| - |F_c|)^2$ minimized, $w = 1$; no H atoms introduced; maximum shift-to-error ratio 0·2; maximum and minimum heights in final difference Fourier map 0·7 and –0·7 e Å^{-3}; no correction for secondary extinction. Scattering factors for the Sn atom from Cromer & Waber (1965), with corrections for anomalous dispersion (Cromer, 1965), and those for the other non-hydrogen atoms from Cromer & Mann (1968). Calculations performed using XRAY72 (Stewart, Kruger, Ammon, Dickinson & Hall, 1972).

Table 1. *Atomic coordinates and equivalent isotropic thermal parameters*

$U_{eq} = \frac{1}{3}(U_{11} + U_{22} + U_{33})$.

	x	y	z	U_{eq}(Å^2)
Sn(1)	0·4880 (2)	0·0369 (1)	0·1685 (1)	0·051 (1)
Sn(2)	0·4436 (2)	0·1629 (1)	–0·1574 (2)	0·056 (1)
Cl(1)	0·4182 (13)	0·1796 (3)	0·1523 (8)	0·089 (5)
Cl(2)	0·4678 (12)	0·1110 (4)	–0·4068 (8)	0·092 (5)
O	0·4659 (22)	0·0612 (7)	–0·0727 (16)	0·056 (8)
C(1)	0·7746 (34)	0·0522 (16)	0·3390 (29)	0·082 (17)
C(2)	0·2077 (31)	0·0160 (16)	0·1854 (29)	0·081 (16)
C(3)	0·7210 (35)	0·2138 (15)	–0·0574 (35)	0·082 (17)
C(4)	0·1432 (39)	0·1895 (18)	–0·2527 (38)	0·103 (22)

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* IUPAC name: 1,2;3,4-di-μ-chloro-1,4-dichloro-1,1,2,2,3,3,4,4-octamethyl-1,2,3;2,3,4-di-μ_3-oxo-tetratin(IV).

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Table 2. *Interatomic distances* (Å) *and angles* (°)

(*a*) Bond distances			
Sn(1)—Cl(1)	2·692 (7)	Sn(2)—Cl(1)	2·790 (9)
Sn(1)—O	2·10 (1)	Sn(2)—Cl(2)	2·442 (8)
Sn(1)—O^{i}	2·08 (1)	Sn(2)—O	2·01 (1)
Sn(1)—C(1)	2·13 (2)	Sn(2)—C(3)	2·14 (2)
Sn(1)—C(2)	2·14 (3)	Sn(2)—C(4)	2·13 (3)
(*b*) Contact distances			
Sn(1)···Sn(1^{i})	3·304 (5)	O···O^{i}	2·57 (3)
Sn(1)···Sn(2)	3·611 (4)	O···Cl(1)	3·05 (2)
Sn(1)···Sn(2^{i})	3·748 (5)	O···Cl(2)	3·06 (2)
(*c*) Bond angles			
O—Sn(1)—O^{i}	75·7 (5)	Cl(1)—Sn(2)—Cl(2)	163·1 (2)
O—Sn(1)—Cl(1)	77·8 (4)	O—Sn(2)—Cl(1)	76·8 (4)
O^{i}—Sn(1)—Cl(1)	153·5 (4)	O—Sn(2)—Cl(2)	86·3 (4)
C(1)—Sn(1)—C(2)	135·4 (9)	Cl(1)—Sn(2)—Cl(2)	163·1 (2)
C(1)—Sn(1)—O	112·9 (8)	C(3)—Sn(2)—C(4)	140·2 (12)
C(1)—Sn(1)—O^{i}	98·1 (9)	O—Sn(2)—C(3)	108·6 (9)
C(2)—Sn(1)—O	111·1 (8)	O—Sn(2)—C(4)	107·7 (10)
C(2)—Sn(1)—O^{i}	99·2 (9)	C(3)—Sn(2)—Cl(1)	85·6 (8)
C(1)—Sn(1)—Cl(1)	91·7 (8)	C(4)—Sn(2)—Cl(1)	87·2 (8)
C(2)—Sn(1)—Cl(1)	90·7 (7)	C(3)—Sn(2)—Cl(2)	101·0 (8)
Sn(1)—O—Sn(1^{i})	104·3 (6)	C(4)—Sn(2)—Cl(2)	97·1 (7)
Sn(1)—O—Sn(2)	122·5 (7)	Sn(1)—Cl(1)—Sn(2)	82·4 (2)
Sn(2)—O—Sn(1^{i})	132·6 (7)		

Symmetry code: superscript: none *x*, *y*, *z*; (i) $1-x, \bar{y}, \bar{z}$.

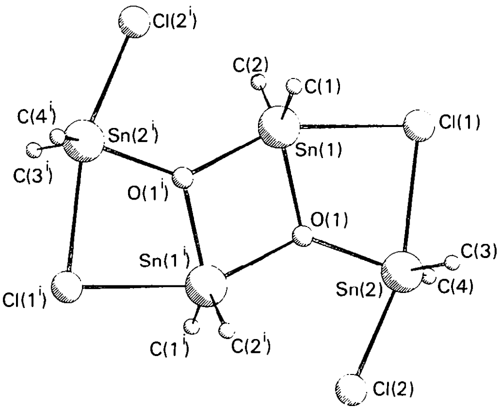

Fig. 1. A perspective view showing the atom numbering of the dimer.

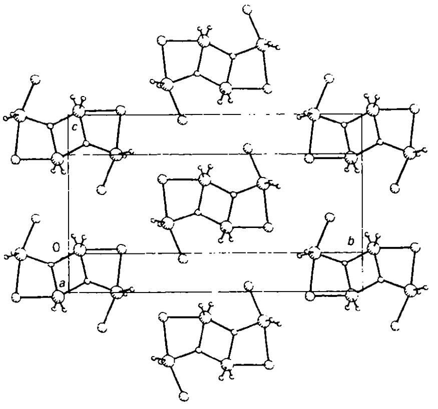

Fig. 2. The molecular packing viewed down **a**.

Discussion. Final coordinates and temperature factors are given in Table 1.* The molecular structure of $\{[(CH_3)_2SnCl]_2O\}_2$ is shown in Figs. 1 and 2. Bond and contact distances and angles are reported in Table 2. The molecule, which is dimeric with crystallographically imposed $\bar{1}$ symmetry, contains a central four-membered Sn_2O_2 ring and two additional adjacent four-membered Sn_2OCl rings. The Sn, O, and Cl atoms of the dimer are roughly coplanar (within 0·08 Å) although the Sn(1)—O—Sn(2)—Cl(1) ring shows a small but significant puckering. The Sn(1)—O distances of 2·10 (1) and 2·08 (1) Å for the equatorial and for the axial bond are chemically equivalent, whereas the Sn(2)—O distance of 2·01 (1) Å is only slightly shorter than the sum of the single-bond covalent radii (2·05 Å). These values can be compared with 2·03 (2), 2·12 (2), and 2·05 (2) Å, respectively, found in $\{[(n\text{-}C_4H_9)SnO_2C_2Cl_3]_2O\}_2$ (Graziani, Bombieri, Forsellini, Furlan, Peruzzo & Tagliavini, 1977).

A good agreement is also observed for the internal angles of the Sn_2O_2 ring: 75·7 (5) and 104·3 (6)° in this work; 75 (1) and 105 (1)° in the above-mentioned compound. The Sn(2)—Cl(2) bond length of 2·442 (8) Å is not significantly different from that found in $Me_3SnCl.py$: 2·42 (4) Å (Beattie, McQuillan & Hulme, 1962) and is in the range of values observed for Sn—Cl in trigonal-bipyramidal organotin complexes; whereas the two Sn—Cl bonds with the bridging Cl are significantly longer. The Sn—C bond lengths agree with those found for other organotin complexes (Zubieta & Zuckerman, 1978).

Both Sn atoms have a severely distorted trigonal-bipyramidal coordination geometry: Sn(1) has Cl(1) and O(1^{i}) as apices and O(1), C(1) and C(2) as base atoms; Sn(2) has Cl(1) and Cl(2) as apices and O(1), C(3) and C(4) as base atoms. However, all the C—Sn—Cl(1) angles with the apical bridging Cl are very close to orthogonality being in the range 86 to 92°, so that the major distortions from the idealized coordination geometry seem to be due to bonding constraints introduced by the triple bridging of the O atoms. Apart from the difference in the chemical stoichiometry, $\{[(CH_3)_2SnCl]_2O\}_2$ is structurally similar to $\{[(CH_3)_2SnNCS]_2O\}_2$ (Chow, 1971). In this latter compound distortions of the coordination geometry are more pronounced and a distorted octahedral hexacoordination for Sn(2) is proposed on the basis of intermolecular interactions.

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38603 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Pseudo-Polymeric Chain Structure of Dibromo(1,2-cyclohexanedione dioxime)copper(II),[*] $CuBr_2(C_6H_{10}N_2O_2)$

By Michel Mégnamisi-Bélombé

Department of Chemistry, University of Ife, Ile-Ife, Nigeria

and Helmut Endres

Anorganisch-Chemisches Institut der Universität, Im Neuenheimer Feld 270, *D*-6900 *Heidelberg* 1, *Federal Republic of Germany*

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Abstract. $M_r = 365·51$, triclinic, $P\bar{1}$, $a = 7·702\ (1)$, $b = 7·942\ (2)$, $c = 10·500\ (3)$ Å, $\alpha = 70·05\ (2)$, $\beta = 68·98\ (2)$, $\gamma = 63·37\ (2)$°, $V = 523·3$ Å^3, $Z = 2$, $D_x = 2·32$ g cm^{-3}, Mo $K\alpha$ radiation, $\lambda = 0·71069$ Å, $\mu = 9·63$ mm^{-1}, $F(000) = 350$, $T = 295 \pm 2$ K, $R_w(R) = 0·039\ (0·045)$ for 1659 observed independent reflections. Pseudo-polymeric columns occur where the Cu^{II} sites form infinite zig-zag chains along **b** with alternating Cu–Cu separations of 4·054 (4) and 4·453 (4) Å. The chains consist of dimers with bridging oxime O atoms and Cu–O distances of 3·240 (10) Å. Within a chain these dimers are linked by weak interdimer Cu–O contacts of 3·912 (10) Å.

Introduction. We recently reported the structure of di-μ-chloro-bis[chloro(1,2-cyclohexanedione dioxime)-copper(II)][†] (Mégnamisi-Bélombé & Endres, 1983) which matches the dimeric structure type commonly found in adducts of Cu^{II} halides (CuX_2) with α, β-dione dioximes (α,β-dodoH). However, two examples of adducts of this kind were recently found to crystallize in a polymeric chain structure, notably the adducts of $CuCl_2$ with oxamide dioxime (Endres, Genç & Nöthe, 1983), and with diphenylethanedione dioxime (Mégnamisi-Bélombé, Singh, Bolster & Hatfield, 1983). Magnetic data analysis has revealed that the latter system represents the first ferromagnetic Heisenberg spin $-\frac{1}{2}$ linear chain based on an α,β-dodoH ligand. In the following we describe the preparation and structure of the adduct of $CuBr_2$ with 1,2-cyclohexanedione dioxime (nioxime, niox) which crystallizes in a pseudo-polymeric chain structure brought about by bridges of O atoms.

Experimental. 510 mg (2·3 mmol) of finely powdered $CuBr_2$ crystals were suspended at room temperature in a mixture of dry diethyl ether (50 ml) and reagent grade (98%) acetonitrile (5 ml). 350 mg (2·4 mmol) of fine-powder niox (EGA-Chemicals, 97%) was added in portions. The mixture turned green, and a dark-green precipitate formed instantaneously. Stirring was continued for 1 h, solid was separated by filtration, washed with dry Et_2O and dried at room temperature. Yield: 718 mg (86%) of dark-green microcrystals decomposing violently at 416–417 K. Material is readily soluble in H_2O (red-brown) and in acetone (green), but virtually insoluble in Et_2O. In many respects, it behaves like the $CuCl_2$(niox) adduct (Mégnamisi-Bélombé & Endres, 1983). Single crystals grown by dissolving the solid in a *ca* 313 K mixture of dry acetone and Et_2O (50:50 ml), and slowly adding dry Et_2O to the filtered solution over a period of 2 d. Rhombohedral crystal, 0·1 × 0·1 × 0·2 mm, lattice parameters from the setting angles of 25 reflections centered on a diffractometer (Syntex $R3$, monochromatic Mo $K\alpha$ radiation), θ–2θ scans, background–peak–background, $2\theta \leq 60°$, 1659 observed independent reflections with $I > 2·5\sigma(I)$, index range h −9–+10, k −9–+11, l 0–+14, 1393 reflections

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[*] *catena*-Dibromo-μ-[(1,2-cyclohexanedione dioxime)-N,N',μ-O,μ-O']copper(II).

[†] Henceforth $CuCl_2$(niox).

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