THE CRYSTAL STRUCTURE OF 1,4-BIS(AQUOTRICHLORO-STANNYL)BUTANE: $[(\text{H}_2\text{O})\text{Cl}_3\text{Sn}(\text{CH}_2)_4\text{SnCl}_3(\text{OH}_2)]$

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Figure 1. Molecular structure of $[(\text{H}_2\text{O})\text{Cl}_3\text{Sn}(\text{CH}_2)_4\text{SnCl}_3(\text{OH}_2)]$ (50% probability ellipsoids). Selected bond distances and angles: Sn-Cl(1) 2.438(2), Sn-Cl(2) 2.330(1), Sn-Cl(3) 2.319(1), Sn-C(1) 2.127(2), Sn-O(1) 2.336(3), C(1)-C(2) 1.513(3), C(2)-C(2)ⁱ 1.526(7) Å; Cl(1)-Sn-Cl(2) 92.32(5), Cl(1)-Sn-Cl(3) 93.89(4), Cl(1)-Sn-O(1) 172.70(7), Cl(1)-Sn-C(1) 103.46(9), Cl(2)-Sn-Cl(3) 109.17(4), Cl(2)-Sn-O(1) 80.97(8), Cl(2)-Sn-C(1) 121.2(1), Cl(3)-Sn-O(1) 85.67(8), Cl(3)-Sn-C(1) 125.3(1), O(1)-Sn-C(1) 82.6(1), Sn-C(1)-C(2) 115.7(2), C(1)-C(2)-C(2)ⁱ 110.1(3)°. Symmetry operation *i*: -*x*, -*y*, -*z*.

Comment

The dimeric structure of $[(\text{H}_2\text{O})\text{Cl}_3\text{Sn}(\text{CH}_2)_4\text{SnCl}_3(\text{OH}_2)]$ is centrosymmetric and features two tin atoms linked by a butylene bridge. Each tin atom is coordinated by three chlorides, a water molecule and a carbon atom. The coordination geometry is distorted trigonal bipyramidal with the Sn atom lying 0.2702(1) Å out of the CCl_2 plane in the direction of the Cl(1) atom. The structure reported here is similar to that found in $[\text{CH}_3\text{OCH}_2\text{CH}_2\text{OCH}_2\text{CH}_2\text{CH}_2\text{SnCl}_3(\text{OH}_2)]$ [1] except that in the latter there is a close intramolecular Sn...O contact of 2.442(4) Å leading to a distorted octahedral geometry. The closest intermolecular contact in the lattice of 2.36 Å occurs between the Cl(1) and OH(5)ⁱⁱ atoms so that Cl(1)...O(1)ⁱⁱ is 3.288(2) Å; symmetry operation *ii*: 0.5 + *x*, 0.5 - *y*, -0.5 + *z*.

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Experimental

Experimental details on the preparation and properties of 1,4-bis(trichlorostannyl)butane have been described in another manuscript [2]. Briefly, the compound was prepared via reaction of Ph₃Sn(CH₂)₄SnPh₃ [3] with conc. hydrochloric acid. Colourless crystals (m.p. 103 – 105 °C) were obtained from a toluene solution of the compound. ¹H NMR δ 2.02 - 2.16 (m, ³J(^{117/119}Sn-¹H) = 164 Hz, 1H, CH₂); 2.26 - 2.44 (m, ²J(^{117/119}Sn-¹H) = 86/90 Hz, 1H, CH₂); ¹³C{¹H} NMR δ 27.15 (s, ²J(^{117/119}Sn-¹³C) = 53 Hz, ³J(^{117/119}Sn-¹³C) = 133 Hz, 2C, CH₂); δ 30.41 (s, ¹J(^{117/119}Sn-¹³C) = 648/680 Hz, 2C, CH₂); ¹¹⁹Sn{¹H} δ 6.3 (s).

Crystallography: The O-H atoms were located from a difference map.

Table 1. Crystal data for [(H₂O)Cl₃Sn(CH₂)₄SnCl₃(OH₂)]

Formula	C ₄ H ₁₂ Cl ₆ O ₂ Sn ₂	Formula weight	542.2
Crystal system	monoclinic	Crystal size, mm	0.19 x 0.24 x 0.37
Space group	<i>P</i> 2 ₁ / <i>n</i>	<i>a</i> , Å	6.296(4)
<i>b</i> , Å	11.218(3)	<i>c</i> , Å	10.791(3)
<i>β</i> , °	96.94(3)	<i>V</i> , Å ³	756.6(5)
<i>Z</i>	2 (dimers)	Diffractometer	Rigaku AFC7R
Temperature, K	173	μ(Mo-Kα), cm ⁻¹	43.35
Trans. factors	0.914 – 1	<i>D</i> _{calcd} , g cm ⁻³	2.380
<i>F</i> (000)	508	<i>θ</i> _{max} , °	27.5
Reflns meas.	1975	Reflns unique, <i>R</i> _{int}	1819, 0.027
Reflns with <i>I</i> ≥ 3.0σ(<i>I</i>)	1569	Weighting scheme	1/[σ ² (<i>F</i> _o) + 0.00001 <i>F</i> _o ²]
<i>R</i> (<i>F</i>), <i>R</i> _w (<i>F</i>)	0.019, 0.023	ρ, e Å ⁻³	0.46
Programs used	teXan [4], DIFABS [5], DIRDIF [6], ORTEP [7]		
Deposition number	CCDC 140356		

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References

- [1] J. Susperregui, M. Bayle, J.M. Léger, G. Deléris, M. Biesemans, R. Willem, M. Kemmer and M. Gielen, *J. Organomet. Chem.* **545** (1997) 559.
- [2] B. Zobel, D. Dakternieks and E.R.T. Tiekink, *Organometallics* submitted for publication.
- [3] Y. Azuma and M. Newcomb, *Organometallics* **3** (1984) 9.
- [4] *teXsan, Single Crystal Structure Analysis Software*, Version 1.04 (1997), Molecular Structure Corporation, The Woodlands, TX, USA.
- [5] N. Walker and D. Stuart, *Acta Crystallogr.*, **A39** (1983) 158.
- [6] P.T. Beurskens, G. Admiraal, G. Beurskens, W.P. Bosman, S.R.O. Garcia-Granda, R.O. Gould, J.M.M. Smits and C. Smykalla, The DIRDIF program system, Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands, 1992.
- [7] C.K. Johnson, ORTEP. Report ORNL-5138 (1976), Oak Ridge National Laboratory, TN, USA.

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