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Short Communication

X-ray structure of $HSeO_3SnMe_2Cl$

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Abstract

When H_2SeO_3 is allowed to react with $SnMe_2Cl_2$ in EtOH at a 1:2 ratio colorless crystals of $HSeO_3SnMe_2Cl$ are obtained which have been characterized by X-ray crystallography. The compound crystallizes in triclinic space group P1 with cell parameters a=6.9693(5) Å, b=7.5096(4) Å, c=7.5222(5) Å, α=71.503(4)°, β=75.826(3)°, γ=87.114(4)°, Z=2, V=361.82(4) Å^3. The structure consists of $[Me_3SnO_3SeH]_n$ chains in which Me_3Sn units are bridged by the selenite ligand; chains are further linked into sheets by O—H----Cl bonds. The key role of hydrogen bonds is worth emphasizing.

Keywords: chain polymer; H-bonds; $SnMe_2Cl$ group; tricoordinating SeO_3.

Many research groups are involved in synthesizing and studying new organotin (VI) compounds because of their various applications in many fields (agriculture, medicine, antifouling paints, wood preservatives) (see Davies and Smith, 1982; Jain et al., 2004; Thoonen et al., 2004; Otera et al., 2008). Our group is interested in the coordination behavior of oxy-anion ligands has yet published X ray structure of selenito organotin (IV) compounds – $SeO_3(SnPh_3)_2$, $SeO_3(SnMe_3)_2{\cdot}H_2O$ – reported by Diallo et al. (2007) and Diassé-Sarr et al. (1997). Structures containing $SnPh_2Cl$, $SnEt_2Cl$ and $SnMe_2Cl$ residues have been reported previously (Camacho-Camacho et al., 2005; Zhang et al., 2005; Dostál et al., 2007) but only two are infinite chains, all the others being discrete. As a continuation of this field of research, we report in this paper the X-ray structure of $HSeO_3SnMe_2Cl$.

The environment around the tin (IV) center is a distorted octahedron with methyl groups disposed in a trans manner [C(1)-Sn-C(2) 167.0(4)°], as are O(1) and O(3), though again here the bond angle deviates notably from linearity [174.0(2)°]. The lattice structure consists of a chain with two adjacent rings: a four-atom Sn_2O_2 ring and a larger eight-atom $[SeO_2Sn]_2$ ring, the two rings having a common Sn-O(1) side. The $[HOSeO_2]^-$ anion behaves as a tridentate ligand with one oxygen [O(1)] bridging two tin atoms while another oxygen atom [O(3)] is solely coordinated to one tin and acts in a bridging manner. As expected, the Sn-O(1) bond [2.235(5) Å] is shorter than either bridging Sn-O(1′) [2.546(5) Å] or Sn-O(3) bonds [2.285(6) Å]. The OH group involved in O—H-----Cl hydrogen bonds, ensure the development of the supramolecular structure into a sheet [O(2)…Cl″ 3.150(6), H(2)…Cl″ 2.38 Å, <O(2)-H(2)…Cl″ 153°].

Table 1 Crystal data and structure refinement.

Empirical formula	$C_2H_7ClO_3SeSn$
Formula weight	312.18
Temperature (K)	150(2) K
Wavelength (Å)	0.71073 Å
Crystal system	triclinic
Space group	P 1
a (Å)	6.9693(5) Å
b (Å)	7.5096(4)
c (Å)	7.5222(5)
α (°)	71.503(4)
β (°)	75.826(3)
γ (°)	87.114(4)
Volume (Å^3)	361.82(4)
Z	2
Density (calculated)	2.865 Mg/m^3
Absorption coefficient, mm^{-1}	8.857
θ range (°)	4.10–24.99
Reflections collected	4523
Independent reflections	1244 [R(int) =0.0791]
Data/restraints/parameters	1244/0/72
Extinction coefficient [a]	0.024(4)
Largest difference in peak and hole (eÅ^3)	2.188 and -4.377
R_1 [b] [I>2σ (I)]	0.0586
wR_2 [c] [I>2σ(I)]	0.1429
GOF [d]	1.083
CCDC number	782622

[a] $F_c^* = k F_c[1+0.001F_c^2\lambda^3/\sin(2\theta)]^{-1/4}$.
[b] $R_1 = \Sigma ||F_o|-|F_c|| / \Sigma |F_o|$.
[c] $wR_2 = \{\Sigma[w(F_o^2-F_c^2)^2] / \Sigma[w(F_o^2)^2]\}^{1/2}$.
[d] $GOF = S = \{\Sigma[w(F_o^2-F_c^2)^2]] / (n-p)\}^{1/2}$.

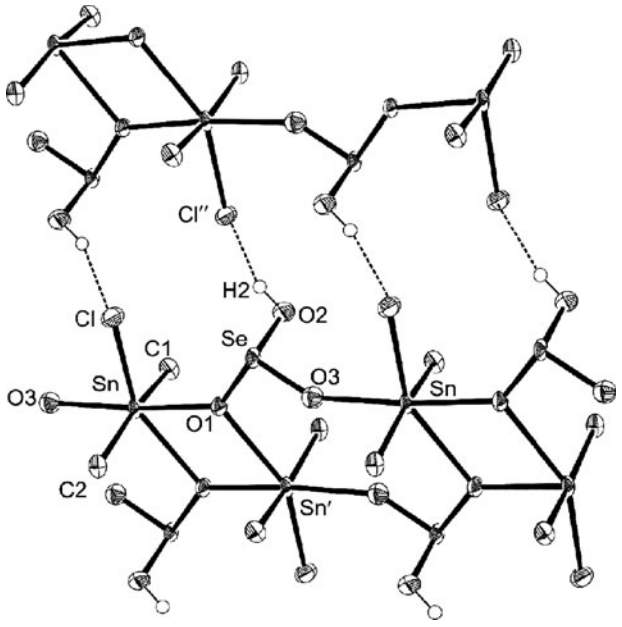

Figure 1 Selected bond lengths and angles Sn-C(1) 2.097(7); Sn-C(2) 2.099(7); Sn-O(1) 2.235(5); Sn-O(1′) 2.546(5); Sn-O(3) 2.285(6); Sn-Cl 2.534(2) Å; C(1)-Sn-C(2) 167.0(4); C(2)-Sn-O(1) 92.2(3); C(2)-Sn-O(3) 87.5(3); O(1)-Sn-O(3) 174.0(2); C(2)-Sn-Cl 95.8(3); O(1)-Sn-Cl 84.50(15); C(2)-Sn-O(1) 86.6(3); O(1)-Sn-O(1) 65.5(2); O(3)#1-Sn-O(1) 120.5(2); Cl-Sn-O(1) 149.96(13). Symmetry transformations used to generate equivalent atoms: ′-x, -y, 2-z; ″-x, 1-y, 1-z.

Experimental section

Synthesis

When 4.130 mmol (0.51584 g) H_2SeO_3 is allowed to react with 7.963 mmol (1.75672 g) $SnMe_2Cl_2$ in EtOH, colorless crystals of $HSeO_3SnMe_2Cl$ are obtained in 60% yield after a slow solvent evaporation.

X-ray crystallography

A crystal of approximate dimensions 0.30×0.20×0.10 mm was used for data collection. Intensity data were collected at 150 K on a Nonius KappaCCD diffractometer equipped with an Oxford cryostream, using graphite monochromated MoKα radiation (λ=0.71070 Å). Data were processed using the Nonius Software [G. M. Sheldrick SHELXS86 (1986); G. M. Sheldrick SHELXL97 (1997)]. A symmetry-related (multi-scan) absorption correction had been applied. Crystal parameters and details on data collection, solution and refinement for the complexes are provided in Table 1. Structure solution, followed by full-matrix least squares refinement was performed using the WINGX-1.80 suite of programs throughout in Department of Chemistry, University of Bath, UK. In the final cycles of least-squares refinement all non-hydrogen atoms except O3 were refined anisotropically. Hydrogen atoms were placed on calculated positions and refined isotropically. A schematic view is shown in Figure 1.

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