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Crystallographic report

Dimeric tetraphenyl-1-hydroxo-3-trifluoromethanesulfonatodistannoxane, $[Ph_2(HO)SnOSn(O_3SCF_3)Ph_2]_2$

Jens Beckmann*, Dainis Dakternieks, Andrew Duthie and Cassandra Mitchell

Centre for Chiral and Molecular Technologies, Deakin University, Geelong 3217, Australia

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The molecular structure of the title compound, obtained by an adventitious phenyl group cleavage of $Ph_3SnOSnPh_3$ with triflic acid, reveals discrete centrosymmetric units of $[Ph_2(HO)SnOSn(O_3SCF_3)Ph_2]_2$ that are loosely associated via hydrogen bonding. Copyright © 2004 John Wiley & Sons, Ltd.

KEYWORDS: crystal structure; tetraorganodistannoxane; tin

COMMENT

Several crystals of $[Ph_2(HO)SnOSn(O_3SCF_3)Ph_2]_2$ were isolated from the reaction of an equimolar ratio of $Ph_3SnOSnPh_3$ and triflic acid, with the intention of

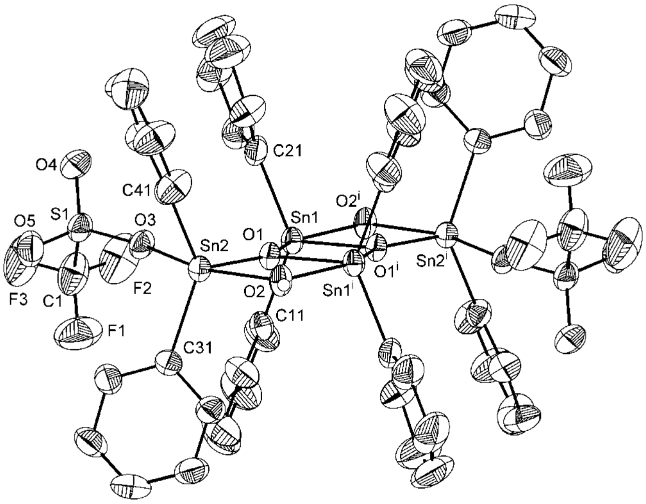

Figure 1. Molecular structure of $[Ph_2(HO)SnOSn(O_3SCF_3)Ph_2]_2$. Hydrogen atoms attached to carbon atoms are omitted for clarity. Selected geometric parameters: Sn1–O1 2.117(3), Sn1–O1^{i} 2.064(3), Sn1–O2^{i} 2.177(4), Sn1–C11 2.109(5), Sn1–C21 2.091(6), Sn2–O1 2.017(3), Sn2–O2 2.093(4), Sn2–O3 2.306(3), Sn2–C31 2.121(5), Sn2–C41 2.112(5) Å; O1–Sn1–O1^{i} 73.97(13), O1–Sn1–O2^{i} 146.98(13), O1–Sn1–C11 99.61(18), O1–Sn1–C21 98.7(2), O1^{i}–Sn1–O2^{i} 73.20(13), O1^{i}–Sn1–C11 113.21(17), O1^{i}–Sn1–C21 115.99(20), O2^{i}–Sn1–C11 96.38(18), O2^{i}–Sn1–C21 92.41(19), C11–Sn1–C21 130.5(2), O1–Sn2–O2 75.95(14), O1–Sn2–O3 80.40(14), O1–Sn2–C31 111.00(17), O1–Sn2–C41 117.62(18), O2–Sn2–O3 156.17(14), O2–Sn2–C31 98.57(19), O2–Sn2–C41 95.33(19), O3–Sn2–C31 92.49(18), O3–Sn2–C41 93.04(19), C31–Sn2–C41 131.3(2)°. Hydrogen bonding contact: O2···O4^{ii} 2.806(6) Å. Symmetry operations: i, $2-x$, $-y$, $2-z$; ii, $1.5-x$, $-0.5+y$, $1.5-z$.

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*Correspondence to: Jens Beckmann, Centre for Chiral and Molecular Technologies, Deakin University, Geelong 3217, Australia.
E-mail: beckmann@deakin.edu.au
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producing $[Ph_3SnOHSnPh_3](O_3SCF_3)$. The formation of $[Ph_2(HO)SnOSn(O_3SCF_3)Ph_2]_2$ (Fig. 1) may be rationalized by an adventitious phenyl group cleavage similar to that reported recently for the reaction of $Ph_3SnOSnPh_3$ with 2,4,6-tris(trifluoromethyl)benzoic acid.[1] The title compound is an analogue of $[R_2(HO)SnOSn(O_3SCF_3)R_2]_2$ (R = Et, *n*-Bu, *n*-Oct),[2] and an example of the broader class of dimeric tetraorganodistannoxanes.[3]

EXPERIMENTAL

Triflic acid (0.204 g, 1.36 mmol) was added to Ph_3SnOH (1.00 g, 2.72 mmol) suspended in MeCN (50 ml) and stirred at room temperature for 1 h. The solvent was removed from the clear solution *in vacuo*, yielding a clear oil. Crystals (m.p. 270–272 °C) suitable for X-ray crystallography were isolated from a dichloromethane–petroleum spirit (60–80 °C) solution of the compound. Intensity data were collected at 293 K on a Bruker SMART Apex CCD diffractometer for a block $0.08 \times 0.10 \times 0.25$ mm^3. $C_{50}H_{42}F_6O_{10}S_2Sn_4$, $M = 1455.72$, monoclinic, $P2_1/n$, $a = 11.4025(7)$, $b = 16.3884(10)$, $c = 15.3933(9)$ Å, $\beta = 110.285(1)°$, $V = 2698.1(3)$, $Z = 2$, 6127 unique data ($\theta_{max} = 27.5°$), $R = 0.046$ (4259 $[I > 2\sigma(I)]$ reflections), $wR = 0.109$ (all data), $\rho_{max} = 1.18$ e^{-1} Å^{-3} (near tin). Programs used: SAINT, SADABS, SHELX-97, ORTEP. CCDC deposition number: 218 184.

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