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Crystallographic report

1,3-Bis[(trimethylsilylmethyl)dichlorostannyl]propane, $[(Me_3SiCH_2)Cl_2Sn]_2(CH_2)_3$

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The dinuclear molecule of $[(Me_3SiCH_2)Cl_2Sn]_2(CH_2)_3$ adopts an extended conformation and features distorted tetrahedral tin centres, with the greatest distortion manifested in the C−Sn−C angles of approximately 128°. The distortions are ascribed to the influence of intermolecular Sn···Cl interactions. Copyright © 2002 John Wiley & Sons, Ltd.

KEYWORDS: 1,3-bis[(trimethylsilylmethyl)dichlorostannyl]propane; crystal structure; organotin; Sn···Cl interactions

COMMENT

The title compound (**I**) is a synthetic precursor for propylene-bridged double ladder molecules.[1] A C_2Cl_2 donor set is found for both independent tin centres in the crystal structure. The greatest deviation from the ideal geometry is found in the C−Sn−C angle of 128.0(5)° for Sn1 and 129.1(6)° for Sn2. In the lattice, molecules associate via weak Sn···Cl interactions that result in the formation of $(Sn\cdots Cl)_2$ rectangles. In this way, Sn1 is 3.319(5) Å from Cl3 (symmetry operation: $0.5 + x$, $0.5 - y$, $1 - z$) and Sn2 is 3.510(5) Å from Cl2 (symmetry operation: $-1/2 + x$, $1/2 - y$, $1 - z$).

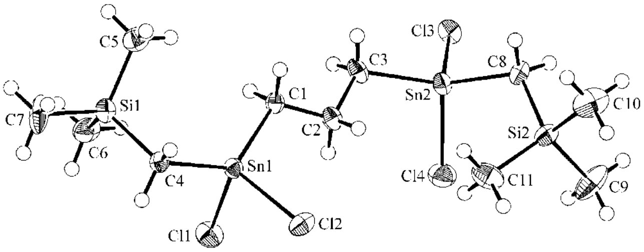

Figure 1. Molecular structure of **I**. Key geometric parameters: Sn1—Cl1 2.373(5), Sn1—Cl2 2.357(4), Sn1—C1 2.127(14), Sn1—C4 2.102(13), Sn2—Cl3 2.370(4), Sn2—Cl4 2.359(5), Sn2—C3 2.119(15), Sn2—C8 2.110(15) Å, Cl1—Sn1—Cl2 97.37(16), Cl1—Sn1—C1 102.9(4), Cl1—Sn1—C4 104.1(4), Cl2—Sn1—C1 109.4(4), Cl2—Sn1—C4 110.1(4), C1—Sn1—C4 128.0(5), Cl3—Sn2—Cl4 97.6(2), Cl3—Sn1—C3 105.5(5), Cl3—Sn1—C8 107.0(4), Cl4—Sn1—C3 105.1(5), Cl4—Sn1—C8 108.1(4), C3—Sn2—C8 129.1(6)°.

EXPERIMENTAL

Synthesis

To a solution of $[(Me_3SiCH_2)Ph_2Sn]_2(CH_2)_3$ (27.1 g, 35.6 mmol; prepared according to a literature procedure[1]) in chloroform (100 ml) was added excess concentrated HCl (200 ml, 32%). The reaction mixture was stirred and maintained at 70 °C for 24 h. The organic layer was separated and the aqueous layer extracted with chloroform (3 × 100 ml). The combined organic extracts were dried ($CaCl_2$) and the solvent was removed *in vacuo*. The residue was crystallized from chloroform/hexane (1/2.5) to afford the title compound as colourless crystals (16.5 g, 78 %); m.p. 93–94 °C (lit. 88–90 °C[1]). ^1H NMR ($CDCl_3$, δ ppm): 0.18 (s, 18H, Me_3Si), 0.89 (s, 4H, $SiCH_2$), 1.83 [t, 4H, $SnCH_2$, $^2J(^1H\text{-}^{117/119}Sn) = 88/92$ Hz], 2.32 (quint, 2H, CH_2). ^{13}C NMR ($CDCl_3$, δ ppm): 1.24 [$SiMe_3$, $^3J(^{13}C\text{-}^{117/119}Sn) = 26$ Hz, $^1J(^{13}C\text{-}^{29}Si) = 52$ Hz], 12.31 [$SiCH_2$, $^1J(^{13}C\text{-}^{117/119}Sn) = 281/294$ Hz], 20.90 [$^2J(^{13}C\text{-}^{117/119}Sn) = 33$ Hz], 29.73 [$^1J(^{13}C\text{-}^{117/119}Sn) = 430/450$ Hz, $^3J(^{13}C\text{-}^{117/119}Sn) = 98/103$ Hz]. ^{119}Sn NMR ($CDCl_3$, δ ppm): 132.0 ppm. Anal. Found: C, 22.0; H, 4.6. Calc. for $C_{11}H_{28}Cl_2Si_2Sn_2$: C, 22.2; H, 4.7%.

Crystallography

Intensity data for **I** were collected at 173 K on a Rigaku AFC7R diffractometer for a colourless crystal $0.07 \times 0.19 \times 0.36$ mm^3, $C_{11}H_{28}Cl_4Si_2Sn_2$, $M = 595.7$, orthorhombic, $P2_12_12_1$, $a = 12.195(12)$, $b = 25.689(14)$, $c = 7.230(7)$ Å, $V = 2265(3)$ Å^3, $Z = 4$; 2973 unique data ($\theta_{max} = 27.5°$); 2504 data with $I \geq 2\sigma(I)$; (obs.) $R = 0.048$; (all data) $wR = 0.185$; Flack parameter: 0.10(10); $\rho_{max} = 1.30$ e Å^{-3}. Programs used: teXsan, DIRDIF, DIFABS, SHELXL, and ORTEP. CCDC deposition number: 185038.

Acknowledgements

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REFERENCES

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