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Main Group Metal Compounds **AOC**

Crystallographic report

1,4-Bis[bis(trimethylsilyl)methyldichlorostannylmethyldimethylsilyl]benzene, *p*-$\{[(Me_3Si)_2CH]Sn(Cl)_2CH_2SiMe_2\}_2C_6H_4$

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The dinuclear molecule of *p*-$\{[(Me_3Si)_2CH]Sn(Cl)_2CH_2SiMe_2\}_2C_6H_4$ is centrosymmetric and adopts an 'S' conformation that is stabilized by intramolecular C—H···π interactions. The tin atom exists within a distorted tetrahedron defined by a C_2Cl_2 donor set. Copyright © 2002 John Wiley & Sons, Ltd.

KEYWORDS: crystal structure; organotin; C—H···π interactions

COMMENT

The title compound, *p*-$\{[(Me_3Si)_2CH]Sn(Cl)_2CH_2SiMe_2\}_2C_6H_4$ (**I**), is a synthetic precursor for spacer-bridged double ladder molecules.[1] The crystal structure determination shows an 'S' conformation for the centrosymmetric molecule (Fig. 1). This is stabilized by the formation of two intramolecular C—H···π interactions[2] involving the C1-*H* and the central aromatic ring. Thus, the distance between the H and the ring centroid is 2.75 Å and the angle subtended at H is 161°. A C_2Cl_2 donor set is found for the tin atom with the widest angle of 124.29(10)° being subtended by the organic groups.

EXPERIMENTAL AND RESULTS

Compound **I** was prepared from 1,4-bis[bis(trimethylsilyl)methyldiphenylstannylmethyldimethylsilyl]benzene (**II**) as described. Preparation of **II**: to a solution of $LiCH(SiMe_3)_2$[3] in 23 ml Et_2O ($c = 0.4\ \mathrm{mol\ l^{-1}}$) was added at room temperature a solution of 1,4-bis(iododiphenylstannylmethyldimethylsilyl)benzene[1] (4.43 g, 4.35 mmol) in 40 ml Et_2O over 40 min. The reaction mixture was stirred overnight and hydrolysed with saturated NH_4Cl solution. The organic layer was washed twice with water and dried over Na_2SO_4. After filtration, the organic solvent was evaporated *in vacuo* and the resulting yellow oil was kept 1 h at 130 °C and 10^{-3} Torr to remove all volatile by-products. This gave 4.32 g crude product, which was used without further purification. ^{13}C NMR (75.44 MHz, $CDCl_3$): δ CH −2.31, $^1J(^{117/119}Sn-^{13}C) = 246/257$, $^1J(^{29}Si-^{13}C) = 50$; $SiMe_2$ 0.07; CH_2 1.61, $^1J(^{117/119}Sn-^{13}C) = 170/179$, $^1J(^{29}Si-^{13}C) = 40$; $SiMe_3$ 3.62 $^3J(^{117/119}Sn-^{13}C) = 16$, $^1J(^{29}Si-^{13}C) = 51$; C_m 128.10; C_p 128.42; C_{oSi} 132.57; C_o 136.90, $^2J(^{117/119}Sn-^{13}C) = 36$; C_{iSi} 141.73; C_i 141.88, $^1J(^{117/119}Sn-^{13}C) = 457/478$. ^{29}Si NMR (59.6 MHz, $CDCl_3$): $\delta - 2.01$ (s, $^2J(^{117/119}Sn-^{29}Si) = 22$, $SiMe_2$); 1.57 (s, $^2J(^{117/}$

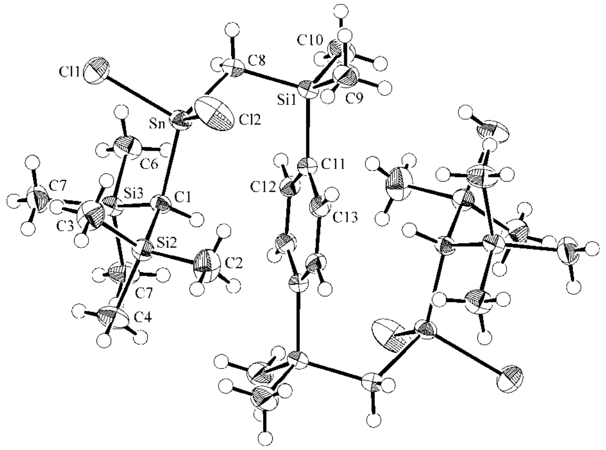

Figure 1. Molecular structure of **I**. Key geometric parameters: Sn—Cl1 2.3471(9), Sn—Cl2 2.3482(9), Sn1—C1 2.130(2), Sn—C8 2.119(3) Å, Cl1—Sn—Cl2 100.40(4), Cl1—Sn—C1 108.01(7), Cl1—Sn—C8 105.09(8), Cl2—Sn—C1 111.68(7), Cl2—Sn—C8 104.68(8), C1—Sn—C8 124.29(10)°.

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^{119}Sn–^{29}Si) = 31, $^{1}J(^{13}C–^{29}Si) = 51$, SiMe$_3$) ^{119}Sn NMR (111.85 MHz, CDCl$_3$): $\delta - 55.2$ $^{1}J(^{13}C–^{119}Sn) = 479$, $^{1}J(^{13}C–^{119}Sn) = 256$.

Preparation of **I**: to a magnetically stirred solution (0 °C) of **II** (4.32 g, 3.99 mmol) in 50 ml acetone was added dropwise a solution of HgCl$_2$ (5.50 g, 20.26 mmol) in 50 ml acetone over 30 min. The reaction mixture was stirred for 7 days and the acetone removed *in vacuo*. Hexane (100 ml) was added and the suspension was refluxed for 10 min. The remaining PhHgCl was removed by hot filtration and the organic solvent evaporated *in vacuo* to give 3.74 g yellow oil. This was recrystallized from CHCl$_3$ (15 ml) to give 1.96 g (51%) of **I**, m.p. 133–136 °C. ^{1}H NMR (299.98 MHz, CDCl$_3$): $\delta - 0.11$ (s, 1H, $^{2}J(^{117/119}Sn–^{1}H) = 101/106$, CH); 0.16 (s, 18H, SiMe$_3$); 0.51 (s, 6H, SiMe$_2$); 1.00 (s, 2H, $^{2}J(^{117/119}Sn–^{1}H) = 84/89$, CH$_2$); 7.59 (s, 2H, C$_6H_4$). ^{13}C NMR (75.44 MHz, CDCl$_3$): δ SiMe$_2$ $- 0.67$ $^{3}J(^{117/119}Sn–^{13}C) = 20$, $^{1}J(^{29}Si–^{13}C) = 55$; SiMe$_3$ 2.77 $^{3}J(^{117/119}Sn–^{13}C) = 27$, $^{1}J(^{29}Si–^{13}C) = 52$; CH 14.81, $^{1}J(^{117/119}Sn–^{13}C) = 314/329$, $^{1}J(^{29}Si–^{13}C) = 46$; CH$_2$ 18.24, $^{1}J(^{117/119}Sn–^{13}C) = 204/213$, $^{1}J(^{29}Si–^{13}C) = 35$; C$_o$ 133.21; C$_i$ 140.66. ^{119}Sn NMR (111.85 MHz, CDCl$_3$): δ 123.7 ppm.

Intensity data for **I** were collected at 173 K on a Rigaku AFC7R diffractometer for a colourless crystal $0.23 \times 0.23 \times 0.29$ mm^3. $C_{26}H_{58}Cl_4Si_6Sn_2$, $M = 918.5$, monoclinic, $P2_1/c$, $a = 11.380(1)$, $b = 11.209(2)$, $c = 17.133(1)$ Å, $\beta = 100.968(8)°$, $V = 2145.6(4)$ Å^3, $Z = 2$, 4927 unique data (θ_{max} 27.5°), 3786 data with $I \geq 2\sigma(I)$, R(obs.) = 0.026, wR(all data) = 0.072, $\rho_{max} = 0.38$ e$^-$ Å^{-3}. Programs used: teXsan, DIRDIF, SHELXL, PLATON, and ORTEP. CCDC deposition number: 186483.

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