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Main Group Metal Compounds **AOC**

Crystallographic report

1,4-Bis[(phenyldichlorostannyl)ethyl]benzene, *p*-$(Cl_2PhSnCH_2CH_2)_2C_6H_4$

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An 'S' conformation, stabilized by intramolecular C—H···π interactions, is found in centrosymmetric *p*-$(Cl_2PhSnCH_2CH_2)_2C_6H_4$. The dinuclear species features distorted tetrahedral tin centres, with the greatest distortion manifested in the C—Sn—C angle of 134.32(16) °. Copyright © 2002 John Wiley & Sons, Ltd.

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

COMMENT

The structure of the title compound was determined in connection with a wider study of rigid spacer-linked tetraorganodistannoxanes.[1] In the centrosymmetric structure of *p*-$(Cl_2PhSnCH_2CH_2)_2C_6H_4$ (**I**); Fig 1, each of the tin centres exists in a distorted tetrahedral geometry defined by a C_2Cl_2 donor set. The greatest deviation from the ideal geometry is found in the C—Sn—C angle of 134.32(16) °. The molecule adopts an 'S' configuration; the reason for this is not immediately apparent. There are no significant intra- or inter-molecular π···π interactions that may be invoked to account for this arrangement. However, there are intramolecular C—H···π interactions[2] so that C6-H is 3.08 Å from the ring centroid of the central phenyl ring with an angle of 108 ° subtended at H. Though these data may not be convincing at first sight, it is noteworthy that the C6-H atom is directed towards the mid-point of the C10—C11^i. Thus, the distance between the H6 atom and the mid-point of C10—C11^i is 2.88 Å with an angle at H of 131 °; symmetry operation *i*: $-x$, $-y$, $1 - z$.

EXPERIMENTAL AND RESULTS

A solution of Ph_3SnH (12.37 g, 35.23 mmol) and AIBN (0.29 g, 1.76 mmol) in benzene (50 ml) was added dropwise to a solution of pure 1,4-divinylbenzene[3] (2.29 g, 17.62 mmol) in benzene (50 ml) at reflux. Stirring was continued at reflux for 2 h after complete addition. After removing the benzene *in vacuo*, the crude product was precipitated from dichloromethane–hexane to give *p*-$(Ph_3SnCH_2CH_2)_2C_6H_4$ as a white powder (14.54 g, 99%), m.p. 172–174 °C. ^1H NMR (299.8 MHz, $CDCl_3$): $\delta = 2.04$ [t, 4H, $^2J(^1H\text{-}^{117/119}Sn) = 54$, α-$CH_2$], 3.19 [t, 4H, $^3J(^1H\text{-}^{117/119}Sn) = 50$, β-$CH_2$], 7.25 (s, 4H, C_6H_4), 7.45–7.90 (m, 30H, Ph); $^{13}C\{^1H\}$ NMR (75.4 MHz, $CDCl_3$): $\delta = 13.10$ [$^1J(^{13}C\text{-}^{117/119}Sn) = 366/383$, α-$CH_2$], 32.06 [$^2J(^{13}C\text{-}^{117/119}Sn) = 18$ β-CH_2] 127.91 (C_6H_4), 128.43 [$^3J(^{13}C\text{-}$

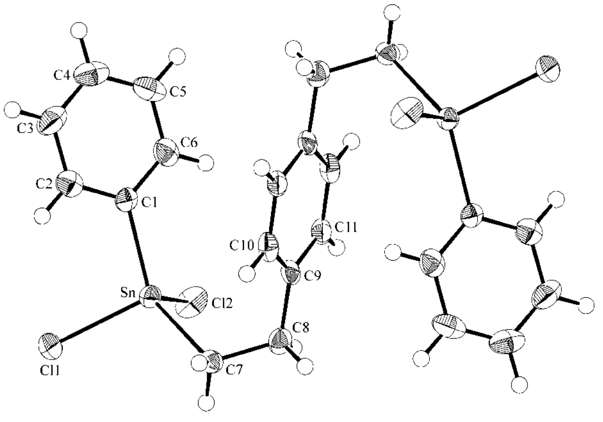

Figure 1. Molecular structure of **I**. Key geometric parameters: Sn—Cl1 2.3546(11), Sn—Cl2 2.3650(14), Sn—C1 2.116(4), Sn—C7 2.133(4) Å, Cl1—Sn—Cl2 102.25(5), Cl1—Sn—C1 103.49(12), Cl1—Sn—C7 107.07(12), Cl2—Sn—C1 104.62(12), Cl2—Sn—C7 101.13(13) and C1—Sn—C7 134.32(16)°.

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$^{117/119}$Sn) = 48, Ph$_m$], 128.81 [$^4J(^{13}C–^{117/119}Sn) = 11$, Ph$_p$], 137.01 [$^2J(^{13}C–^{117/119}Sn) = 36$, Ph$_o$], 138.67 [$^1J(^{13}C–^{117/119}Sn) = 468/489$, Ph$_i$], 142.42 [$^3J(^{13}C–^{117/119}Sn) = 60$, C_6H_4]; ^{119}Sn NMR (111.9 MHz, $CDCl_3$): $\delta = -100.2$. Anal. Found: C, 66.15; H, 4.72. Calc. for $C_{46}H_{42}Sn_2$: C, 66.39; H, 5.09%.

Conc. HCl (2 ml) was added to *p*-$(Ph_3SnCH_2CH_2)_2C_6H_4$ (0.20 g, 0.24 mmol) and stirred at 60 °C overnight. The crude product was extracted with dichloromethane (5 ml), dried over Na_2SO_4, filtered, and the solvent removed *in vacuo*. Crystallization from chloroform gave **I** as colourless crystals (0.13 g, 81%), m.p. 143–145 °C. ^{1}H NMR (299.8 MHz, $CDCl_3$): $\delta = 2.30$ [t, 4H, $^2J(^1H–^{117/119}Sn) = 61/63$, α-CH_2], 3.13 [t, 4H, $^3J(^1H–^{117/119}Sn) = 124/130$, β-CH_2], 7.15 (s, 4H, C_6H_4), 7.25–7.50 (m, 10H, Ph); $^{13}C\{^1H\}$ NMR (75.4 MHz, $CDCl_3$): $\delta = 28.09$ [$^1J(^{13}C–^{117/119}Sn) = 485/507$, α-CH_2], 30.27 [$^2J(^{13}C–^{117/119}Sn) = 31$, β-CH_2], 128.72 (C_6H_4), 129.31 [$^3J(^{13}C–^{117/119}Sn) = 81$, Ph$_m$], 131.35 [$^4J(^{13}C–^{117/119}Sn) = 17$, Ph$_p$], 134.46 [$^2J(^{13}C–^{117/119}Sn) = 64$, Ph$_o$], 139.09 (Ph$_i$), 140.94 [$^3J(^{13}C–^{117/119}Sn) = 71$, C_6H_4]; ^{119}Sn NMR (111.9 MHz, $CDCl_3$): $\delta = 38.8$. Anal. Found: C, 39.55; H, 3.30. Calc. for $C_{22}H_{22}Cl_4Sn_2$: C, 39.70; H, 3.33%.

Intensity data for **I** were collected at 173 K on a Rigaku AFC7R diffractometer for a colourless crystal $0.08 \times 0.16 \times 0.36$ mm^3. $C_{22}H_{22}Cl_4Sn_2$, $M = 665.6$, orthorhombic, *Pbca*, $a = 20.739(4)$, $b = 16.054(9)$, $c = 7.140(1)$ Å, $V = 2377(1)$ Å^3, $Z = 4$, 2730 unique data ($\theta_{\max}$ 27.5 °), 1579 data with $I \geq 2\sigma(I)$, R(obs.) = 0.026, wR(all data) = 0.062, $\rho_{\max} = 0.48$ e$^-$ Å^{-3}. Programs used: teXsan, DIRDIF, DIFABS, SHELXL, PLATON, and ORTEP. CCDC deposition number: 185038.

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REFERENCES

1. Dakternieks D, Duthie A, Zobel B, Jurkschat K, Schürmann M and Tiekink ERT. *Organometallics* 2002; **21**: 647.
2. Jennings WB, Farrell BM and Malone JF. *Acc. Chem. Res.* 2001; **34**: 885.
3. Wiley RH, Jin JI and Kamath Y. *J. Polym. Sci., Polym. Chem. Ed.* 1968; **6**: 1065.