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Crystallographic report

1,3,5,7-Tetraisopropyl-2,4,6,8,9,10-hexathia-1,3,5,7-tetrastanna-adamantane

C. Wagner, C. Raschke and K. Merzweiler*

Institut für Anorganische Chemie, Martin-Luther-Universität Halle-Wittenberg, Kurt-Mothes-Str. 2, D-06120 Halle, Germany

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The crystal structure of $^{i}Pr_4Sn_4S_6$ consists of isolated molecules that contain an adamantane-like Sn_4S_6 core. The tin atoms are coordinated nearly tetrahedrally, with Sn–S distances ranging from 2.397(1) to 2.411(1) Å.

KEYWORDS: crystal structure; tin; sulfur

COMMENT

$^{i}Pr_4Sn_4S_6$ exhibits an adamantane-like Sn_4S_6 core analogous to six organotin sesquisulfides $R_4Sn_4S_6$ (R = Me,[1] 1,3,5-$Me_3C_6H_2$,[2] 4-Me-C_6H_4,[2] C_6F_5,[2] $(Me_3Si)_3C$,[3] and $\{Cp(CO)_2Fe\}$[4]) published so far. The $^{i}Pr_4Sn_4S_6$ molecule (Fig. 1) possesses crystallographic mirror symmetry, with the mirror plane passing through the atoms Sn(1), Sn(2), S(1), S(3), C(1) and C(3). The Sn–S distances (2.397(1)–2.411(1) Å) agree with the values for the Sn_4S_6 cages cited above. The coordination geometry of the tin atoms is essentially tetrahedral, with S–Sn–S angles ranging from 110.5(1) to 112.8(1)° and C–Sn–S angles between 104.2(2) and 109.2(1)°.

EXPERIMENTAL

$^{i}Pr_4Sn_4S_6$ was obtained in 95% yield from the reaction of $^{i}PrSnCl_3$ with sodium sulfide in acetone/water (1 : 1). Recrystallization from chloroform–toluene gave colourless rodlike crystals, m.p. 220 °C (dec.), ^{119}Sn NMR ($CDCl_3$, δ ppm): 154.8, $J(^{117}Sn–^{119}Sn)$: 161.7 Hz. Intensity data were collected at 220 K on a STOE IPDS diffractometer for a crystal $0.12 \times 0.12 \times 0.52$ mm^3, $C_{12}H_{28}S_6Sn_4$, $M = 839.46$, orthorhombic, *Pnma*, $a = 11.032(1)$, $b = 15.504(2)$, $c = 14.645(3)$Å, $V = 2504.9(6)$ Å^3, $Z = 4$; 2534 unique data ($\theta_{max} = 26.1°$), $R_1 = 0.028$ (all data), $wR_2 = 0.050$ (all data), $\rho_{max} = 1.48$ e Å^{-3} (near tin). Programs used: SHELXL, DIAMOND. CCDC deposition number: 226 390.

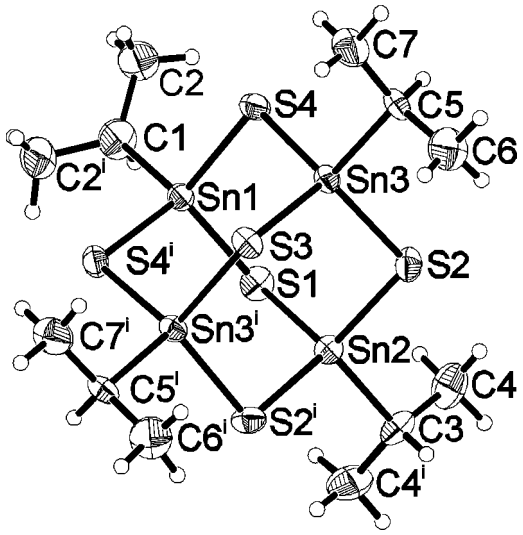

Figure 1. Molecular structure of $^{i}Pr_4Sn_4S_6$. Key geometric parameters: Sn1–S1 2.397(1), Sn1–S4 2.407(1), Sn2–S1 2.402(1), Sn2–S2 2.400(1), Sn3–S2 2.401(1), Sn3–S3 2.411(1), Sn3–S4 2.405(1), Sn1–C1 2.169(5), Sn2–C3 2.150(5), Sn3–C5 2.158(3) Å; S1–Sn1–S4 111.4(1), S4–Sn1–S4^{i} 111.5(1), S2–Sn2–S2^{i} 112.8(1), S1–Sn2–S2 110.5(1), S2–Sn3–S4 111.1(1), S2–Sn3–S3 111.9(1), S3–Sn3–S4 110.8(1), Sn1–S1–Sn2 105.9(1), Sn2–S2–Sn3 110.8(1), Sn3–S3–Sn3^{i} 105.9(1), Sn1–S4–Sn3 105.3(1)°. Symmetry operation i: x, $\frac{1}{2} - y$, z.

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*Correspondence to: K. Merzweiler, Institut für Anorganische Chemie, Martin-Luther-Universität Halle-Wittenberg, Kurt-Mothes-Str. 2, D-06120 Halle, Germany.
E-mail: merzweiler@chemie.uni-halle.de