THE CRYSTAL STRUCTURE OF BIS[BENZYLTRIPHENYLPHOSPHONIUM]HEXACHLOROTIN(IV) DICHLOROFORM SOLVATE

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Figure 1. Unit cell contents and atomic labeling scheme for $[(\text{PhCH}_2)\text{Ph}_3\text{P}]_2[\text{SnCl}_6] \cdot 2\text{CHCl}_3$ (50% probability ellipsoids). Selected bond distances and angles: Sn-Cl(1) 2.436(1), Sn-Cl(2) 2.424(1), Sn-Cl(3) 2.441(1) Å; Cl(1)-Sn-Cl(2) 90.72(4), Cl(1)-Sn-Cl(2)ⁱ 89.28(4), Cl(1)-Sn-Cl(3) 89.29(4), Cl(1)-Sn-Cl(3)ⁱ 90.71(4), Cl(2)-Sn-Cl(3) 89.28(5), Cl(2)-Sn-Cl(3)ⁱ 90.72(5)°; symmetry operation *i*: -*x*, -*y*, -*z*.

Comment

The compound is a rearrangement product isolated from a solution containing $[(\text{PhCH}_2)\text{Ph}_3\text{P}]_2[\text{Cl}_4\text{Sn}(\text{CH}_2)_6\text{SnCl}_4]$ [1]. The anion is centrosymmetric and is almost a regular octahedron. The most significant intermolecular contact in the lattice occurs between the Cl(2) and the chloroform-H atoms so that $\text{Cl}(2)\cdots\text{H}^{\text{ii}}$ is 2.72 Å, $\text{Cl}(2)\cdots\text{C}(26)^{\text{ii}}$ is 3.565(5) Å and the $\text{Cl}(2)\cdots\text{H}^{\text{ii}} - \text{C}(26)^{\text{ii}}$ angle is 149°; symmetry operation *ii*: 1-x, -y, 1-z.

Experimental

Preparation: Colourless crystals (m.p. 266–267°C) were grown at room temperature from a $\text{CH}_2\text{Cl}_2/\text{CHCl}_3$ (1:5) solution of $[(\text{PhCH}_2)\text{Ph}_3\text{P}]_2[\text{Cl}_4\text{Sn}(\text{CH}_2)_6\text{SnCl}_4]$ prepared from 2 mole equivalents of $[(\text{PhCH}_2)\text{Ph}_3\text{P}]\text{Cl}$ and $\text{Cl}_3\text{Sn}(\text{CH}_2)_6\text{SnCl}_3$. A sample to be used for elemental analysis was kept at 60°C and 10^{-3} Torr for 1 h to remove all volatiles. Anal: Calcd for $\text{C}_{50}\text{H}_{44}\text{Cl}_6\text{P}_2\text{Sn}$, C, 57.86; H, 4.27. Found: C 57.82, H 4.39 %. ^1H NMR (299.98 MHz, CD_3OD) δ 4.94 (d, 2H, $^2J(^{31}\text{P}-^1\text{H}) = 15$ Hz, CH_2); 6.98–7.94 (mm, 20H, Ph). $^{13}\text{C}\{^1\text{H}\}$ NMR (75.44 MHz, CD_3OD) δ 30.54 (d, 1C, $^1J(^{31}\text{P}-^{13}\text{C}) = 48$ Hz); 118.80 (d, 3C, $^1J(^{31}\text{P}-^{13}\text{C}) = 86$ Hz); 128.51 (d, 1C, $^2J(^{31}\text{P}-^{13}\text{C}) = 8$ Hz); 129.57 (d, 1C, $^5J(^{31}\text{P}-^{13}\text{C}) = 4$ Hz); 129.82 (d, 2C, $^nJ(^{31}\text{P}-^{13}\text{C}) = 3$ Hz); 131.16 (d, 6C, $^nJ(^{31}\text{P}-^{13}\text{C}) = 12$ Hz); 132.09 (d, 2C, $^nJ(^{31}\text{P}-^{13}\text{C}) = 5$ Hz); 135.18 (d, 6C, $^nJ(^{31}\text{P}-^{13}\text{C}) = 10$ Hz); 136.18 (d, 3C, $^4J(^{31}\text{P}-^{13}\text{C}) = 3$ Hz). $^{119}\text{Sn}\{^1\text{H}\}$ NMR (111.85 MHz, CD_3OD) δ -652.3.

Crystallography:**Table 1.** Crystal data for $[(\text{PhCH}_2)\text{Ph}_3\text{P}]_2[\text{SnCl}_6]\cdot 2\text{CHCl}_3$

Formula	$\text{C}_{52}\text{H}_{46}\text{Cl}_{12}\text{P}_2\text{Sn}$	Formula weight	1277.0
Crystal system	triclinic	Crystal size, mm	0.07 x 0.10 x 0.18
Space group	$P\bar{6}3$	<i>a</i> , Å	11.757(3)
<i>b</i> , Å	12.027(3)	<i>c</i> , Å	10.307(2)
α , °	92.01(2)	β , °	96.09(2)
γ , °	73.33(2)	<i>V</i> , Å ³	1388.2(6)
<i>Z</i>	1	Diffraction	AFC7R
Temperature, K	173	$\mu(\text{Mo-K}\alpha)$, cm ⁻¹	11.29
Trans. factors	0.928 – 1	D_{calc} , g cm ⁻³	1.527
<i>F</i> (000)	642	<i>A</i>	27.5
Refins meas.	6687	Refins unique, <i>R</i> _{int}	6380, 0.034
Refins with <i>I</i> > 3.0σ(<i>I</i>)	4099	Weighting scheme	1/[σ ² (<i>F</i> _o) + 0.00017 <i>F</i> ²]
<i>R</i> (<i>F</i>), <i>R</i> _w (<i>F</i>)	0.039, 0.044	ρ , e Å ⁻³	0.69
Programs used	teXan [2], DIFABS [3], DIRDIF [4], ORTEP [5]		
Deposition number	CCDC 141374		

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