

## Compounds of germanium and tin

# XIV <sup>☆</sup>. Rearrangement of bis(2,4,6-*tert*-butylphenyl)stannylene: synthesis and structure of a donor-free alkylarylstannylene-tungsten complex

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## Abstract

Bis(2,4,6-*tert*-butylphenyl)stannylene, R<sub>2</sub>Sn (**2**), rearranges in solution to the sterically less crowded alkylarylstannylene RR'Sn (**3**), R' = CH<sub>2</sub>-C(CH<sub>3</sub>)<sub>2</sub>-3,5-tBu<sub>2</sub>C<sub>6</sub>H<sub>3</sub>. Treatment of **3** with [(OC)<sub>5</sub>W(THF)] yields the donor-free tungsten stannylene complex [(OC)<sub>5</sub>W=SnRR'] (**4**). The molecular structure of **4** reveals a three-coordinate tin atom in a strictly planar environment and an acute CSnC angle of 91.5°. Trapping reactions of **3** with selenium, 2,3-dimethylbutadiene, and 3,5-di-*tert*-butyl-*ortho*-benzoquinone furnish the 1,3-diselenadistannetane and the [4 + 1]-cycloaddition products, respectively.

**Keywords:** Tungsten; Stannylene; Cycloaddition; Crystal structure

## 1. Introduction

Although a considerable number of heterosubstituted stannylenes of the type (RX)<sub>2</sub>Sn which alleviate their electron deficiency by means of interactions between the free-electron pairs at X and the unoccupied 5 pπ orbital of the tin centre are now known [2], exclusively alkyl- or aryl-substituted stannylenes are still very scarce. Apart from [(Me<sub>3</sub>Si)<sub>2</sub>CH]<sub>2</sub>Sn (**1**) [3], which exists in the solid state as a distannene (Lappert's distannene) with a *trans*-bent substituent arrangement [4], only one other compound in which the two C–H hydrogen atoms of **1** have been replaced by an ethylene bridge [5] has previously been structurally characterized. [2,4,6-(CF<sub>3</sub>)<sub>3</sub>C<sub>6</sub>H<sub>2</sub>]<sub>2</sub>Sn constitutes an exception and its stability presumably results from the close contacts between the fluorine atoms in the *ortho*-CF<sub>3</sub> groups with the low-valence tin atom [6]. We recently prepared bis(2,4,6-*tert*-butylphenyl)stannylene (**2**) as the first

example of a diarylstannylene without donor atom stabilization [1].

## 2. Results and discussion

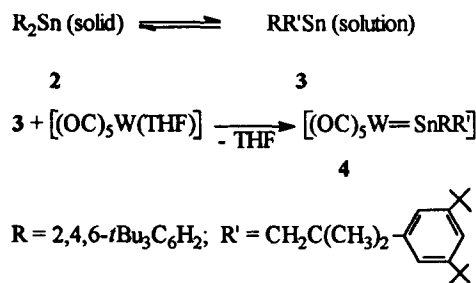
Product **2** is stable in the solid state; however, the NMR data indicate that a partial isomerization to the less sterically crowded prochiral alkylarylstannylene **3** occurs in solution (Scheme 1). Similar rearrangements had previously been observed in the transmetallation reactions of tin tetrahalides with 2,4,6-*tert*-butylphenyllithium although the resultant, isolated alkylaryl tin halides proved to be stable towards further isomerizations of this type [7].

Indirect evidence for the putative isomerization product **3** in solution was provided by its reaction with [(OC)<sub>5</sub>W(THF)] in tetrahydrofuran (THF); after expulsion of THF from the tungsten complex, pale-yellow crystals of the stannylene complex **4** were isolated. The constitution of **4** was confirmed by complete NMR spectroscopy investigations and an X-ray crystal structure analysis (Fig. 1 and Tables 1 and 2).

<sup>☆</sup> Part XIII, see [1].

Dedicated to Professor Herbert Schumann on the occasion of his 60th birthday.

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Scheme 1.

The tricoordinated tin atom in **4** is in an exactly planar orientation with its ligands (angular sum  $360^\circ$ ). Worthy of note is the acute C–Sn–C angle of only  $91.5^\circ$  which reflects the reduced steric demands of the substituents of **3** in comparison with **2** (C–Sn–C angle of  $104^\circ$ ) [6] and which is markedly less than the also small C–Sn–C angle of  $98^\circ$  in  $[(\text{OC})_5\text{Cr}=\text{SnR}'_2]$  ( $\text{R}' = \text{CH}(\text{SiMe}_3)_2$ ) [8]. The W–Sn bond length is only slightly shorter than those of stannyltungsten compounds and is, in fact, longer than those in the base-stabilized complexes  $[(\text{OC})_5\text{WSnCl}_2(\text{THF})_x]$  in which these bond lengths are 271.7 ( $x = 1$ ) and 273.7 pm ( $x = 2$ ) [9].

The  $^{119}\text{Sn}$  NMR signal of **4** appears at 799 ppm and is thus shifted to a high field by about 300 ppm in comparison with the free stannylene. However, it does not approach the values of the donor-atom- or base-stabilized tungsten stannylene complexes which lie between 0 and  $-300$  ppm [10,12]. Also, the  $^1J(^{183}\text{W}, ^{119}\text{Sn})$  coupling constant is markedly smaller than those in the above-mentioned complexes and thus further supports the trend that these coupling constants increase significantly with increasing coordination number of tin [9,10].

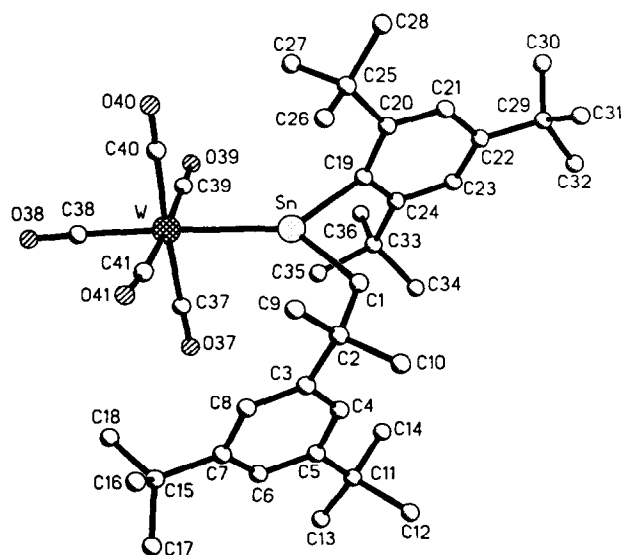
Fig. 1. Molecular structure of **4** in the crystal (hydrogen atoms omitted).

Table 1

Atomic parameters and equivalent isotropic displacement coefficients for **4**

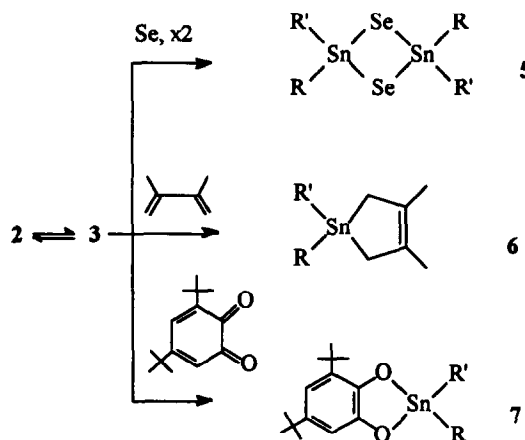
|        | $x$<br>( $\times 10^{-4}$ ) | $y$<br>( $\times 10^{-4}$ ) | $z$<br>( $\times 10^{-4}$ ) | $U_{\text{eq}}$<br>( $\times 10$ pm $^2$ ) |
|--------|-----------------------------|-----------------------------|-----------------------------|--------------------------------------------|
| W      | 2225(1)                     | 4214(1)                     | −1144(1)                    | 41(1)                                      |
| Sn     | 3584(1)                     | 3695(1)                     | 1013(1)                     | 35(1)                                      |
| C(1)   | 4308(3)                     | 4505(3)                     | 2633(4)                     | 39(2)                                      |
| C(2)   | 4135(3)                     | 5602(3)                     | 2720(4)                     | 38(2)                                      |
| C(3)   | 2994(3)                     | 6360(3)                     | 2828(4)                     | 38(2)                                      |
| C(4)   | 2569(3)                     | 6440(3)                     | 3829(4)                     | 45(2)                                      |
| C(5)   | 1567(4)                     | 7144(3)                     | 4044(4)                     | 49(2)                                      |
| C(6)   | 971(3)                      | 7790(3)                     | 3203(5)                     | 52(2)                                      |
| C(7)   | 1354(3)                     | 7753(3)                     | 2203(4)                     | 46(2)                                      |
| C(8)   | 2378(3)                     | 7021(3)                     | 2028(4)                     | 41(2)                                      |
| C(9)   | 4505(3)                     | 5609(4)                     | 1586(4)                     | 51(2)                                      |
| C(10)  | 4840(4)                     | 5911(4)                     | 3916(5)                     | 56(2)                                      |
| C(11)  | 1136(5)                     | 7244(4)                     | 5182(6)                     | 70(3)                                      |
| C(12)  | 1838(9)                     | 7661(9)                     | 6454(11)                    | 69(3)                                      |
| C(13)  | −16(10)                     | 8001(11)                    | 5248(13)                    | 81(3)                                      |
| C(14)  | 1361(9)                     | 6169(9)                     | 5364(11)                    | 62(3)                                      |
| C(12A) | 1811(16)                    | 6408(17)                    | 5940(20)                    | 132(7)                                     |
| C(13A) | 721(16)                     | 8302(17)                    | 5942(19)                    | 138(7)                                     |
| C(14A) | 84(17)                      | 7081(18)                    | 4619(21)                    | 142(7)                                     |
| C(15)  | 707(3)                      | 8487(4)                     | 1306(5)                     | 54(2)                                      |
| C(16)  | 1402(9)                     | 8770(10)                    | 860(12)                     | 69(3)                                      |
| C(17)  | −169(11)                    | 9514(11)                    | 2030(13)                    | 77(4)                                      |
| C(18)  | 181(8)                      | 7921(9)                     | 130(10)                     | 60(2)                                      |
| C(16A) | 1266(9)                     | 9203(10)                    | 1520(11)                    | 63(3)                                      |
| C(17A) | 663(19)                     | 7976(19)                    | −71(23)                     | 142(8)                                     |
| C(18A) | −424(11)                    | 9219(11)                    | 1655(14)                    | 77(4)                                      |
| C(19)  | 4347(3)                     | 2320(3)                     | 1799(4)                     | 36(2)                                      |
| C(20)  | 5378(3)                     | 1601(3)                     | 1573(4)                     | 38(2)                                      |
| C(21)  | 6031(3)                     | 1005(3)                     | 2440(4)                     | 43(2)                                      |
| C(22)  | 5732(3)                     | 1056(3)                     | 3529(4)                     | 40(2)                                      |
| C(23)  | 4693(3)                     | 1673(3)                     | 3654(4)                     | 43(2)                                      |
| C(24)  | 3985(3)                     | 2306(3)                     | 2812(4)                     | 39(2)                                      |
| C(25)  | 5765(3)                     | 1429(3)                     | 347(4)                      | 44(2)                                      |
| C(26)  | 5807(4)                     | 2373(4)                     | 214(5)                      | 52(2)                                      |
| C(27)  | 5043(4)                     | 1102(4)                     | −786(4)                     | 59(2)                                      |
| C(28)  | 6870(4)                     | 556(4)                      | 269(5)                      | 68(3)                                      |
| C(29)  | 6514(3)                     | 439(3)                      | 4491(4)                     | 47(2)                                      |
| C(30)  | 6779(5)                     | −686(4)                     | 3946(6)                     | 85(3)                                      |
| C(31)  | 7484(5)                     | 639(6)                      | 4826(6)                     | 89(4)                                      |
| C(32)  | 6101(5)                     | 755(5)                      | 5718(5)                     | 78(3)                                      |
| C(33)  | 2834(3)                     | 2875(3)                     | 3025(4)                     | 44(2)                                      |
| C(34)  | 2731(4)                     | 3345(6)                     | 4408(5)                     | 92(4)                                      |
| C(35)  | 2142(4)                     | 3714(4)                     | 2289(5)                     | 67(3)                                      |
| C(36)  | 2383(5)                     | 2084(5)                     | 2587(8)                     | 101(5)                                     |
| C(37)  | 1150(4)                     | 5196(4)                     | 36(5)                       | 55(2)                                      |
| O(37)  | 558(3)                      | 5738(3)                     | 702(4)                      | 83(2)                                      |
| C(38)  | 1134(4)                     | 4612(5)                     | −2645(5)                    | 68(3)                                      |
| O(38)  | 507(4)                      | 4879(5)                     | −3477(5)                    | 116(4)                                     |
| C(39)  | 1884(4)                     | 3027(4)                     | −1276(5)                    | 59(3)                                      |
| O(39)  | 1709(4)                     | 2361(3)                     | −1368(5)                    | 90(3)                                      |
| C(40)  | 3377(4)                     | 3254(4)                     | −2203(4)                    | 50(2)                                      |
| O(40)  | 4032(3)                     | 2749(3)                     | −2791(4)                    | 71(2)                                      |
| C(41)  | 2579(3)                     | 5346(4)                     | −1146(4)                    | 48(2)                                      |
| O(41)  | 2766(3)                     | 5963(3)                     | −1246(4)                    | 72(2)                                      |

After  $[(\text{OC})_5\text{W}]_2\text{Sn}=\text{W}(\text{CO})_5$  [11], **4** is the second structurally elucidated tungsten stannylene complex lacking donor atoms and bases and is, at the same time, the first alkylarylstannylene complex of a transition

metal to be reported [12]. The alkyl or aryl isomerization of one of the substituents of **2** demonstrated here has also been observed in further trapping products.

Thus the reaction with selenium presumably proceeds initially to the monomeric stannaneselone which, on account of the reduced steric demands of the substituents in **3**, undergoes spontaneous head-to-tail dimerization to furnish the 1,3-diselenadistannetane **5** (Scheme 2). The species **3** participates in a [4 + 1]-cycloaddition reaction with 2,3-dimethylbutadiene to produce the 1-stannacyclopent-3-ene derivative **6**.

The reaction of **3** with 3,5-di-*tert*-butyl-*ortho*-benzoquinone is also a [4 + 1]-cycloaddition and gives rise to the 1,3,2-benzodioxastannole derivative **7**. The constitutions of the products **5**–**7** have been substantiated by elemental analyses and spectroscopic data.



Scheme 2.

### 3. Experimental section

#### 3.1. General procedure

All reactions were carried out in oven-dried glassware under an atmosphere of dry argon.

The  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra were obtained on a Bruker AM 300 spectrometer, the  $^{119}\text{Sn}$  NMR spectra on a Bruker AMX 300 spectrometer using  $\text{C}_6\text{D}_6$  as solvent. IR spectra were taken on a Bio-Rad FTS-7 spectrometer. Mass spectra were recorded on a Varian-MAT 212 instrument. Elemental analyses were performed by Analytische Laboratorien, D-51647 Gummersbach, Germany.

#### 3.1. [(2,4,6-Tri-*tert*-butylphenyl)(2-methyl-2(3,5-di-*tert*-butylphenyl)propyl)]stannylene-pentacarbonyltungsten(0) (**4**)

A solution of  $[\text{W}(\text{CO})_6]$  (1.08 g, 3.07 mmol) in 70 ml of THF was irradiated for 6 h with a high pressure mercury immersion lamp. This solution was added dropwise to a solution of **2** (1.43 g, 2.34 mmol) in 20 ml of THF. The reaction mixture was stirred for 14 h at

room temperature. The solvent was removed under vacuum and excess  $[\text{W}(\text{CO})_6]$  was removed by sublimation at  $50^\circ\text{C}$  (0.001 mbar). Repeated crystallization from pentane at  $4^\circ\text{C}$  yielded 0.46 g (21% yield) pale-yellow crystals of **4** (melting point (m.p.)  $116$ – $118^\circ\text{C}$ ).  $^1\text{H}$  NMR:  $\delta$  1.25 (s, 9H), 1.28 (s, 18H), 1.32 (s, 18H), 1.61 (s, 6H), 2.23 (s, 2H), 7.35 (d, 2H,  $^4J = 1.8$  Hz), 7.38 (t, 1H), 7.45 (s, 2H) ppm.  $^{13}\text{C}$  NMR:  $\delta$  30.51 ( $\text{C}_p$ ), 31.33 ( $\text{C}_p$ ), 31.73 ( $\text{C}_p$ ), 33.29 ( $\text{C}_p$ ), 35.15 ( $\text{C}_q$ ), 39.35 ( $\text{C}_q$ ), 39.49 ( $\text{C}_q$ ), 58.07 ( $\text{CH}_2$ ), 119.52, 120.70, 123.57, 149.98, 150.38, 150.87, 156.19, 199.27, 200.93 ppm.  $\text{C}_p$  and  $\text{C}_q$  refer to primary and quaternary carbon atoms respectively.  $^{119}\text{Sn}\{^1\text{H}\}$  NMR:  $\delta$  798.9 ( $^1J(^{183}\text{W}, ^{119}\text{Sn}) = 940$  Hz) ppm. IR  $\nu$ : 2064, 1945, 1920 ( $\text{CO}$ )  $\text{cm}^{-1}$ . Mass spectroscopy (MS) (CI, isobutane):  $m/z$  936 ( $\text{MH}^+$ , 100%). Anal. Found: C, 52.60; H, 6.12; Sn, 12.55.  $\text{C}_{41}\text{H}_{58}\text{O}_5\text{SnW}$  (933.50) calc.: C, 52.76; H, 6.26; Sn, 12.72%.

#### 3.2. 2,4-Bis(2,4,6-tri-*tert*-butylphenyl)-2,4-bis[2-methyl-2(3,5-di-*tert*-butylphenyl)propyl]-1,3-diselenadistannetane (**5**)

Solid selenium (0.25 g, 3.2 mmol) was added to a solution of **2** (2.0 g, 3.2 mmol) in 60 ml of *n*-hexane. The resulting suspension was stirred at room temperature for 60 h, with the colour changing from red to colourless. The mixture was filtered, concentrated and cooled to  $-30^\circ\text{C}$ . Repeated crystallization from *n*-hexane afforded 1.2 g (27%) colourless crystals of **5** (m.p.  $210^\circ\text{C}$ ).  $^1\text{H}$  NMR:  $\delta$  1.39 (s, 90H), 1.66 (s, 6H), 1.83 (s, 4H,  $\text{CH}_2$ ), 7.40–7.43 (m, 10H) ppm.  $^{13}\text{C}$  NMR:  $\delta$  31.92 ( $\text{C}_p$ ), 32.53 ( $\text{C}_p$ ), 35.02 ( $\text{C}_q$ ), 33.99 ( $\text{C}_q$ ), 44.03 ( $\text{CH}_2$ ), 119.61, 120.43, 149.91, 150.73 ppm. MS (field desorption):  $m/z$  1379 ( $\text{M}^+$ , 100%). Anal. Found: C, 62.65; H, 8.43; Se, 11.25.  $\text{C}_{72}\text{H}_{116}\text{Se}_2\text{Sn}_2$  (1377.05) calc.: C, 62.80; H, 8.49; Se, 11.46%.

Table 2  
Selected bond lengths (pm) and angles ( $^\circ$ ) (standard deviations) for **4**

| Bond lengths  |          |           |          |
|---------------|----------|-----------|----------|
| W–Sn          | 275.1(1) | Sn–C(1)   | 217.8(4) |
| Sn–C(19)      | 219.1(5) | W–C(38)   | 200.6(5) |
| W–C(37)       | 204.9(5) | W–C(39)   | 204.7(8) |
| W–C(40)       | 203.2(5) | W–C(41)   | 202.6(7) |
| Bond angles   |          |           |          |
| C(1)–Sn–C(19) | 91.5(2)  | C(1)–Sn–W | 134.0(1) |
| W–Sn–C(19)    | 124.5(1) |           |          |

### 3.3. 3,4-Dimethyl-1(2,4,6-tri-*tert*-butylphenyl)-1[2-methyl-2(3,5-di-*tert*-butylphenyl)propyl]-1-stannacyclopent-3-ene (6)

A solution of **2** (1.5 g, 2.5 mmol) in 40 ml toluene was cooled to  $-30^{\circ}\text{C}$ . After addition of 2,3-dimethylbutadiene (0.30 g, 3.6 mmol) the mixture was allowed to warm to room temperature. The solvent was removed under reduced pressure, and the remaining solid was dissolved in *n*-pentane and filtered. Crystallization at  $-25^{\circ}\text{C}$  afforded 0.93 g (55%) colourless crystals of **6** (m.p.,  $118^{\circ}\text{C}$ )  $^1\text{H}$  NMR:  $\delta$  1.27 (s, 9H), 1.39 (s, 18H), 1.45 (s, 18H), 1.54 (s, 6H), 1.58 (s, 6H), 1.73 (s, 2H), 1.78 (s, 2H), 2.14 (s, 2H),  $^2J(^{119}/^{117}\text{Sn}, ^1\text{H}) = 62$  Hz), 7.40 (t, 1H,  $^4J = 1.7$  Hz), 7.47 (d, 2H), 7.53 (s, 2H) ppm.  $^{13}\text{C}$  NMR:  $\delta$  21.52 ( $\text{C}_p$ ), 28.47 ( $\text{C}_q$ ), 30.91 ( $\text{CH}_2$ ), 31.42 ( $\text{C}_p$ ), 31.84 ( $\text{C}_p$ ), 33.04 ( $\text{C}_p$ ), 34.03 ( $\text{C}_p$ ), 35.16 ( $\text{C}_q$ ), 38.90 ( $\text{C}_q$ ), 39.09 ( $\text{C}_q$ ), 40.06 ( $\text{CH}_2$ ), 119.95, 122.89, 128.33, 130.79, 148.91, 150.19, 158.21, ppm. MS (field desorption):  $m/z$  692 ( $\text{M}^+$ , 18%). Anal. Found: C, 72.93; H, 9.76.  $\text{C}_{42}\text{H}_{68}\text{Sn}$  (691.70) calc.: C, 72.93; H, 9.90%.

### 3.4. 5,7-Di-*tert*-butyl-2(2,4,6-tri-*tert*-butylphenyl)-2[2-methyl-2(3,5-di-*tert*-butylphenyl)propyl]-1,3,2-benzodioxastannole (7)

A solution of **2** (2.0 g, 3.2 mmol) in 40 ml of toluene was cooled to  $-10^{\circ}\text{C}$ . After addition of 3,5-di-*tert*-butyl-*ortho*-benzoquinone (0.7 g, 3.2 mmol) it was allowed to warm to room temperature and the mixture was stirred for 2 h at this temperature. The solvent was removed under reduced pressure, and the residue was recrystallized from *n*-pentane at  $-25^{\circ}\text{C}$ , (yield, 0.7 g (26%) of colourless crystals of **7**; m.p.  $212^{\circ}\text{C}$ ).  $^1\text{H}$  NMR:  $\delta$  1.18 (s, 9H), 1.38 (s, 18H), 1.40 (s, 18H), 1.49 (s, 9H), 1.55 (s, broad, 6H), 1.70 (s, 9H), 2.35 (AB, 2H,  $J = 12.7$  Hz), 6.94 (d, 1H,  $^4J = 1.9$  Hz), 7.19 (d, 1H), 7.44 (d, 2H,  $^4J = 8.2$  Hz), 7.45 (s, 2H), 7.51 (t, 1H) ppm.  $^{13}\text{C}$  NMR:  $\delta$  30.68 ( $\text{C}_p$ ), 31.12 ( $\text{C}_p$ ), 31.78 ( $\text{C}_p$ ), 32.12 ( $\text{C}_p$ ), 32.79 ( $\text{C}_p$ ), 33.20 ( $\text{C}_p$ ), 34.46 ( $\text{C}_q$ ), 35.19 ( $\text{C}_q$ ), 37.04 ( $\text{C}_q$ ), 38.22 ( $\text{C}_q$ ), 38.73 ( $\text{C}_q$ ), 48.35 ( $\text{CH}_2$ ), 111.00, 113.29, 119.65, 120.32, 124.01, 125.09, 135.12, 139.97, 141.79, 148.88, 150.69, 151.60, 155.55 ppm. MS (field desorption):  $m/z$  830 ( $\text{M}^+$ , 22%). Anal. Found: C, 72.53; H, 9.52.  $\text{C}_{50}\text{H}_{72}\text{O}_2\text{Sn}$  (829.82) calc.: C, 72.37; H, 9.47%.

### 3.5. X-ray structure analysis of **4**

Empirical formula,  $\text{C}_{41}\text{H}_{58}\text{O}_5\text{SnW}$ ; molecular weight,  $933.5\text{ g mol}^{-1}$ ; crystal size,  $0.35 \times 0.40 \times 0.10\text{ mm}^3$ ; triclinic; space group,  $P\bar{1}$ ;  $a = 1475.0(2)$ ,  $b = 1521.5(2)$  and  $c = 1134.9(1)\text{ pm}$ ;  $\alpha = 108.56(1)$ ,  $\beta = 106.47(1)$  and  $\gamma = 61.36(1)^{\circ}$ ;  $V = 20924(4)\text{ nm}^3$ ,  $D_{\text{calc}} = 1.481\text{ g cm}^{-3}$ ,  $\lambda(\text{Mo K}\alpha) = 71.073\text{ pm}$ ;  $T = 296(2)$

K; data collection mode, Wyckoff scan;  $2\theta_{\text{max}} = 55^{\circ}$ ; number of unique reflections, 9612; number observed ( $F > 3\sigma(F)$ ), 8474; data-to-parameter ratio, 19.8, linear absorption coefficient,  $3.38\text{ mm}^{-1}$ . The structure was solved by direct phase determination using the SHELXTL PLUS program system and refined by the full-matrix least squares techniques. Hydrogen atoms were placed in calculated positions, and all other atoms were refined anisotropically. The methyl carbon atoms of the *meta-tert*-butyl groups are disordered occupying two positions each with an occupancy factor of 0.5.  $R = 0.035$ ;  $R_w = 0.034$ . Further details of the crystal structure determination may be obtained from the Fachinformationszentrum Karlsruhe, D-76344 Eggenstein-Leopoldshafen, Germany, upon quoting the depository number CSD-401 881.

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