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Cyclohexasilanes with exocyclic organogermanium, -tin or -lead groups

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Abstract

A new class of mono- (**2**–**4**) and bis(undecamethylcyclohexasilanyl) (**5**–**9**) substituted germanium, tin and lead compounds is described. These molecules are accessible by treatment of undecamethylcyclohexasilanyl potassium (**1**) with chlorogermanes, -stannanes or plumbanes. Attempts to generate bis(cyclohexasilanyl)methanes, -plumbanes or tris(cyclohexasilanyl)-methylstannane starting from dihalomethanes, -plumbanes or methyltrichlorostannane and **1** result only in transmetallation reactions. The molecular structures of a mono- (**3**) and a dicyclohexasilanyl stannane derivative **5** were determined by single-crystal X-ray crystallography. © 2000 Elsevier Science S.A. All rights reserved.

Keywords: Undecamethylcyclohexasilanyl potassium; Undecamethylcyclohexasilanylstannanes; ^{1}H-, ^{29}Si-NMR; Mass spectroscopy; X-ray crystallography

1. Introduction

Undecamethylcyclohexasilanyl derivatives have attracted increasing interest since the first synthesis of dodecamethylcyclohexasilane in 1949 [1], due to their interesting electronic and photophysical properties [2–6]. Two types of compounds, with one (**A**) or two (**B**) cyclohexasilanyl groups have been described (Fig. 1).

In both cases, Z is either a transition metal fragment [7–9] or a main group element such as a halogen [10–12], oxygen [12,13], sulfur [12], phosphorus [12] or silicon [11]. Recently, we described the synthesis and reactivity of a variety of type **A** and **B** silicon compounds (Z = Si) [14]. However, so far type **A** or **B** derivatives, where the exocyclic Z is a different Group 14 element, are not known, with the exception of the permethylated Si$_6$ ring in itself, and other type **A** carbon substituted derivatives (Z = alkyl or aryl). Recently we communicated in a poster contribution the undecamethylcyclohexasilanyl group as a sterically demanding substituent for tin atoms [15]. In an extension to this work we now describe the syntheses and molecular structures of germanium, tin and lead derivatives with one (type **A**) or two (type **B**) undecamethylcyclohexasilanyl groups (Z = Ge, Sn, Pb; Fig. 1).

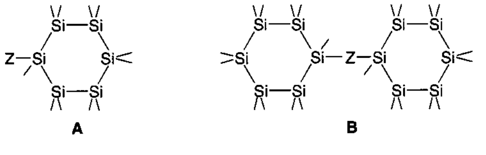

Fig. 1. Z = transition metal, O, S, PPh, SiRR′ and H, halogene (for type A).

2. Results and discussion

A convenient starting material for compounds of type **A** and **B** (as depicted in Fig. 1) is the highly reactive undecamethylcyclohexasilanyl potassium (**1**). This precursor can easily be prepared from dodecamethylcyclohexasilane and potassium *tert*-butylate

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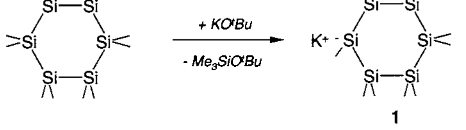
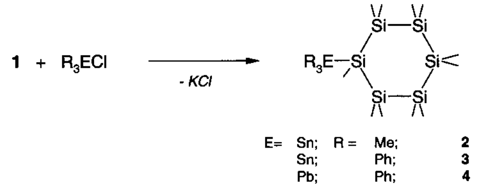
(Eq. (1)). The reaction mechanism is likely to be based on complex ring-opening–ring-closing reactions, resulting in a theoretical yield of 80% relative to the starting material cyclohexasilane [16].

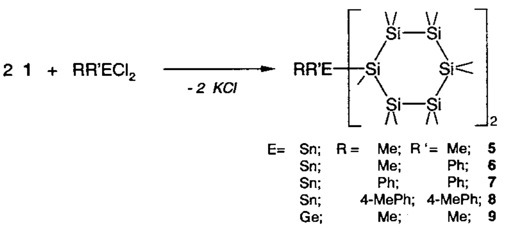


(1)

Undecamethylcyclohexasilanyl derivatives of tin(IV) are formed by reaction of **1** with organotin mono- or dichlorides at low temperatures (Eqs. (2) and (3)). A type **B** germanium compound was prepared by reacting dimethyldichlorogermane with two equivalents of **1** (**9**, Eq. (3)), a type **A** lead derivative (**4**) was synthesized by using triphenylchloroplumbane as starting material.

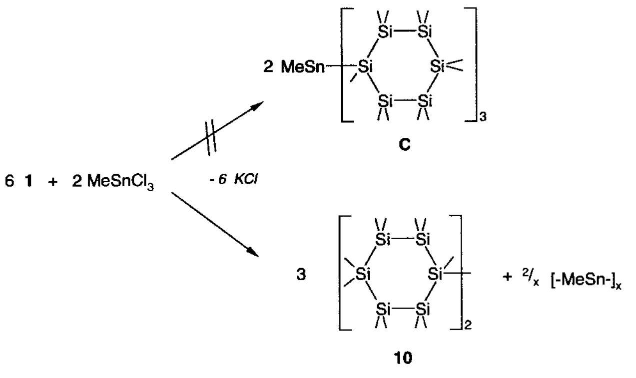


(2)



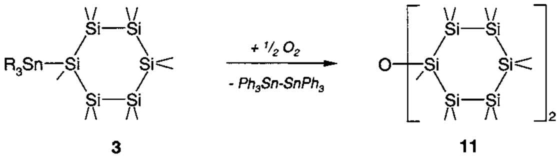
(3)

The reactions of dimethyldichloroplumbanes (Me_2PbCl_2) and dihalomethanes (CH_2X_2, X = Cl, Br, I) did not result in the formation of type **B** compounds, since transmetallation reactions result in bis(undecamethylcyclohexasilanyl) (**10**) as the only identifiable compound. Similar results were observed in the reaction of methyltrichlorostannane with three equivalents of **1**. The reaction does not afford a tin derivative of type **C** (Scheme 1), instead transmetallation was observed accompanied by the formation of polystannanes and **10**.



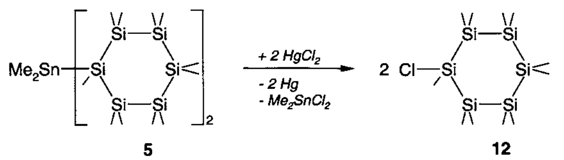
Scheme 1. Transmetallation and formation of **10**.

The yields of compounds **2**–**9** are between 30 and 70%. The tin derivatives are more air-sensitive than the corresponding silanes [11] or the germane **9**. For example, **3** reacts with dry air within 4 weeks to form hexaphenyldistannane and a siloxane [12] (**11**, Eq. (4)).



(4)

The lead derivative **4** is even more reactive towards oxygen and decomposes within minutes when exposed to air. The reaction of **5** with two equivalents of mercury dichloride yields dimethyldichlorostannane, chloroundecamethylcyclohexasilane (**12**) and elemental mercury (Eq. (5)).



(5)

Compounds **2**–**7** and **9** were characterized by NMR spectroscopy, mass spectrometry and elemental analysis. Additionally, an EDXRFS [17] (energy dispersive X-ray fluorescence spectroscopy) experiment was used to verify the germanium content of compound **9**. The connectivities of the silicon atoms of **3** and **5** were determined by ^{29}Si–^{29}Si coupling constants and independently from the $^{119/117}$Sn–^{29}Si coupling constants (Table 1). Compounds **2** and **5** were also characterized by X-ray crystallography.

Compound **3**, depicted in Fig. 2, crystallizes with two independent molecules per asymmetric unit. Both molecules display fairly similar geometrical features with the central Sn atom bound to three phenyl groups, in addition to one Si_6Me_{11} unit. The Sn atom displays a slightly distorted tetrahedral environment with angles ranging between 105.5(1) and 117.9(1)° for Sn(1) and 104.1(2) and 114.1(1)° for Sn(2). The Si–Sn bond lengths are observed at 259.3(1) pm for Sn(1) and 259.0(1) pm for Sn(2), while Sn–C bonds are observed between 214.9(4) and 216.1(4) pm. The Si_6Me_{11} rings adopt a chair conformation with Si–Si distances between 234.0(2) and 235.4(2) pm.

Compound **5**, depicted in Fig. 3, displays monomeric units with no imposed crystallographic symmetry. The central Sn atom is connected to two methyl groups in addition to two Si_6Me_{11} units with Sn–Si linkages of 258.9(2) and 259.3(2) pm and Sn–C bond lengths of 216.5(9) and 217.6(9) pm. Sn(1) displays a slightly distorted tetrahedral coordination environment with angles ranging from 102.7(5)° between the two methyl

Table 1
^{29}Si- and ^{119}Sn-NMR shifts of undecamethylcyclohexasilanyl-substituted compounds **2**–**9**

Compound no.	E	R	R′	R″	^{119}Sn (ppm)	^{29}Si (ppm)/coupling constants in Hz			
						Si$_A$	Si$_B$	Si$_C$	Si$_D$
2	Sn	Me	Me	Me	−168.5	−70.8 $^{1}J(^{29}Si–^{119/117}Sn)$: 370/353	−36.8 $^{2}J(^{29}Si–^{119/117}Sn)$: 65/62	−40.4	−42.2
3	Sn	Ph	Ph	Ph	−149.5	−69.1 $^{1}J(^{29}Si–^{119/117}Sn)$: 314/300	−36.7 $^{2}J(^{29}Si–^{119/117}Sn)$: 69/66	−39.9 $^{3}J(^{29}Si–^{119/117}Sn)$: 9	−41.3 $^{1}J(^{29}Si–^{119/117}Sn)$: 6
4	Pb	Ph	Ph	Ph		−72.0 $^{1}J(^{29}Si–^{207}Pb)$: 648	−37.1	−40.2	−41.9
5	Sn	Me	Me	Si_6Me_{11}	−206.3	−70.4 $^{1}J(^{29}Si–^{119/117}Sn)$: 204/194	−40.2 $^{2}J(^{29}Si–^{119/117}Sn)$: 51	−35.8 $^{3}J(^{29}Si–^{119/117}Sn)$: 10	−42.0
6[a]	Sn	Me	Ph	Si_6Me_{11}	−191.2	−69.1 $^{1}J(^{29}Si–^{119/117}Sn)$: 204/195	−35.5	−40.3/−40.9	−42.3
7	Sn	Ph	Ph	Si_6Me_{11}	−192.3	−62.9 $^{1}J(^{29}Si–^{119/117}Sn)$: 170/163	−35.5 $^{2}J(^{29}Si–^{119/117}Sn)$: 62/59	−40.1	−42.7
8	Sn	p-MePh	p-MePh	Si_6Me_{11}	−192.3	−64.1 $^{1}J(^{29}Si–^{119/117}Sn)$: 173/167	−36.1 $^{2}J(^{29}Si–^{119/117}Sn)$: 63/60	−40.3	−42.2
9	Ge	Me	Me	Si_6Me_{11}		−76.9	−41.9	−39.6	−34.0

[a] Only NMR-spectroscopically determined.

groups, up to 113.65(6)° between the two sterically more demanding Si_6Me_{11} moieties. The Si_6 ring is in a chair conformation, with Si–Si distances in the range of 233.6(3)–235.7(3) pm.

Functionalization reactions at the tin atoms of **3** and **5** with triflic acid–lithium chloride or tetrachlorostannane are rather complex and will be described in detail in an upcoming publication.

3. Experimental

All procedures were carried out under an atmosphere of inert gas using standard Schlenk techniques. Solvents were dried by standard procedures and used after being freshly distilled [18]. All organo-substituted mono-, di- and trichlorostannanes [19] and undecamethylcyclohexasilanyl potassium [16] were prepared according to published procedures.

NMR spectra were recorded on Bruker DRX 300 (^{29}Si: 59.63 MHz, ^{119}Sn: 111.92 MHz) and DPX 400 (^{1}H: 400.15 MHz, ^{13}C: 100.63 MHz, ^{29}Si: 79.49 MHz, ^{119}Sn: 149.21 MHz) spectrometers. The $^{1}J_{Si–Si}$-coupling constants were determined using an INEPT-INADEQUATE technique [20–22]. Mass spectra were obtained using a MAT 8200 mass spectrometer. A Cambridge Stereoscan 360 Scanning Electron Microscope with an AN-1270 EDX system by Link was used for the EDXRFS [17] experiment.

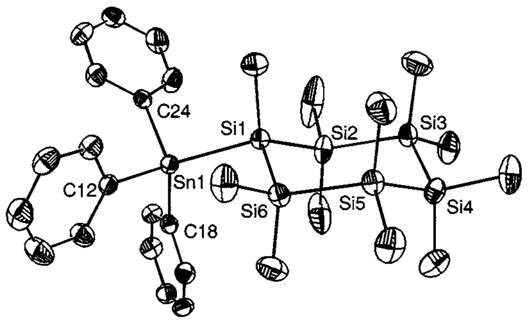


Fig. 2. Molecular structure of compound **3** showing 50% probability displacement ellipsoids (ORTEP3).

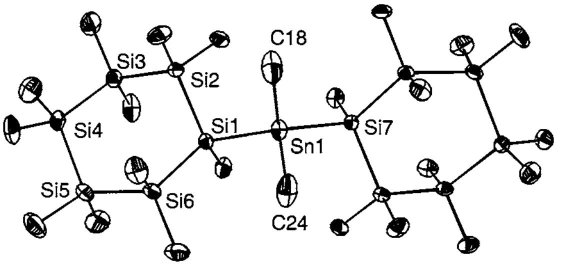


Fig. 3. Molecular structure of compound **5** showing 30% probability displacement ellipsoids (ORTEP3).

*3.1. General procedure for the preparation of mono- and bis-undecamethylcyclohexasilanyl-substituted stannanes, germanes and plumbanes **2**–**9***

A solution of 11.2 ($n = 2$) or 5.6 mmol ($n = 3$) of element chloride R_nEX_{4-n} ($n = 2$, 3) in 20 ml of tetrahydrofurane (thf) is cooled to −50°C and stirred. A solution of 11.2 mmol undecamethylcyclohexasilanyl potassium (**1**)[1] in 20 ml diethyleneglycoldimethylether (diglyme) is added dropwise over a period of 1 h. The solution is then stirred for another 12 h. The solvent is removed in vacuo, followed by addition of 50 ml of hexane. The insoluble residue is removed by filtration, and the hexane is removed by evaporation under reduced pressure.

After addition of 50 ml of ethanol the resulting solution is stored at −20°C for 24 h. During storage a solid is formed, which is isolated by filtration and recrystallized from 100 ml of acetone.

*3.1.1. (Undecamethylcyclohexasilanyl)trimethylstannane (**2**)*

Starting materials: 2.23 g (11.2 mmol) Me_3SnCl, 11.2 mmol KSi_6Me_{11}; colorless solid, m.p.: 68°C; Recrystallization from 1:5 *n*-pentane–*n*-propanol. Yield: 2.50 g (45%). ^{1}H-NMR: 0.25 [$Sn\underline{Me}_3$, $^2J(^1H-^{119/117}Sn$: 42)], 0.23, 0.17 [11 × $Si\underline{Me}$]. ^{13}C-NMR: −1.1 [$Sn\underline{Me}_3$ $^1J(^{13}C-^{119/117}Sn)$: 258/246 Hz], −3.1, −4.6, −5.0, −5.3, −6.4, −10.0 [1 × $Si\underline{Me}$]. $^{29}Si-^{29}Si$ coupling constants: $^1J(Si_A-Si_B)$: 52 Hz, $^1J(Si_B-Si_C)$: 59.5 Hz, $^1J(Si_C-Si_D)$: 60 Hz, $^2J(Si_A-Si_C)$: 6 Hz, $^2J(Si_B-Si_D)$: 7.5 Hz. MS: 333 [100%, Si_6Me_{11}], 451 [20%, $Si_6Me_{11}Sn$], 481 [5%, $M^+ - Me$], 497 [5%, M^+]; Elemental analysis: $C_{14}H_{42}Si_6Sn$, MW = 497.7 g mol^{-1}. Found (Calc.): C, 32.5 (33.8); H, 8.7 (8.5).

*3.1.2. (Undecamethylcyclohexasilanyl)triphenylstannane (**3**)*

Starting materials: 4.31 g (11.2 mmol) Ph_3SnCl, 11.2 mmol KSi_6Me_{11}; colorless solid, m.p.: 118°C. Yield: 5.90 g (77%). ^{1}H-NMR: 0.11–0.55 (m) [11 × $Si\underline{Me}$], 7.22–7.53 (m) [3 × Ph]. ^{13}C-NMR: 140.2 [2 × C_i, $^1J(^{13}C-^{119/117}Sn)$: 398/380 Hz]; 137.9 [4 × C_o, $^2J(^{13}C-^{119/117}Sn)$: 40 Hz]; 127.8 [4 × C_m, $^3J(^{13}C-^{119/117}Sn)$: 44 Hz]; 127.8 [2 × C_p]; −7.5, −4.9, −4.1, −1.8 [22 × $Si\underline{Me}$]. MS: 73 [6%, $SiMe_3$], 273 [15%, $SnPh_2$], 334 [20%, Si_6Me_{11}], 350 [17%, $SnPh_3$], 684 [10%, M^+]. Elemental analysis: $C_{29}H_{48}Si_6Sn$, MW = 683.91 g mol^{-1}. Found (Calc.): C, 48.8 (51.0); H, 7.2 (7.0).

*3.1.3. (Undecamethylcyclohexasilanyl)triphenylplumbane (**4**)*

Starting materials: 2.65 g (5.6 mmol) Ph_3PbCl, 5.6 mmol KSi_6Me_{11}; m.p. (dec.): 75–83°C, yellow–white solid. Yield: 1.3 g (30%). ^{1}H-NMR: 0.15 (m) [3 × $SiMe_2$], 0.24 (s) [2 × $SiMe_2$], 55 (s) [1 × $Si\underline{Me}$], 7.18–7.65 (m) [3 × Ph]. Elemental analysis: $C_{29}H_{48}PbSi_6$, MW = 772.4 g mol^{-1}. No satisfactory elemental analysis could be obtained.

*3.1.4. Bis(undecamethylcyclohexasilanyl)-dimethylstannane (**5**)*

Starting materials: 1.23 g (5.6 mmol) Me_2SnCl_2, 11.2 mmol KSi_6Me_{11}; colorless solid, m.p.: 145–153°C. Yield: 3.75 g (82%). ^{1}H-NMR: 0.11–0.30 (m) [22 × $Si\underline{Me}$], 1.15 (m) [2 × Me]. ^{13}C-NMR: −7.8 [2 × $Sn\underline{Me}$]; −6.7, −5.0, −4.1, −1.8 [22 × $Si\underline{Me}$]. $^{29}Si-^{29}Si$ coupling constants: $^1J(Si_A-Si_B)$: 49 Hz, $^1J(Si_B-Si_C)$: 60 Hz, $^1J(Si_C-Si_D)$: 61.5 Hz, $^2J(Si_A-Si_C)$: 7.5 Hz, $^2J(Si_B-Si_D)$: 9.5 Hz. MS: 73 [5%, $SiMe_3$], 334 [20%, Si_6Me_{11}], 468 [30%, $Si_6Me_{11} - SnMe$], 800 [10%, $M^+ - Me$], 815 [8%, M^+]; Elemental analysis: $C_{24}H_{72}Si_{12}Sn$, MW = 816.56 g mol^{-1}. Found (Calc.): C, 36.8 (35.4); H, 9.2 (8.8).

*3.1.5. Bis(undecamethylcyclohexasilanyl)-methylphenylstannane (**6**)*

Starting materials: 1.58 g (5.6 mmol) $MePhSnCl_2$, 11.2 mmol KSi_6Me_{11}; colorless oil. Yield: 3.40 g (69%).

*3.1.6. Bis(undecamethylcyclohexasilanyl)-diphenylstannane (**7**)*

Starting materials: 1.93 g (5.6 mmol) Ph_2SnCl_2, 11.2 mmol KSi_6Me_{11}; colorless solid, m.p.: 170–172°C. Yield: 3.70 g (70%). ^{1}H-NMR: 7.25–7.6 (m) [2 × SnPh], 0.54 [2 × SiMe], 0.33 [4 × $SiMe_2$], 0.14 [6 × $SiMe_2$]; Elemental analysis: $C_{34}H_{76}Si_{12}Sn$, MW = 940.72. Found (Calc.): C, 43.9 (43.4); H, 8.1 (7.5).

*3.1.7. Bis(undecamethylcyclohexasilanyl)bis-(p-methylphenyl)stannane (**8**)*

Starting materials: 2.10 g (5.6 mmol) $(p\text{-}MePh)_2SnCl_2$, 11.2 mmol KSi_6Me_{11}; colorless solid, m.p.: 146–149°C. Yield: 4.30 g (78%). ^{1}H-NMR: 7.35 (d), 7.66 (d) [2 × C_4H_4], 2.35 (s) [2 × CMe], 0.49 [2 × SiMe], 0.36 [4 × $SiMe_2$], 0.18 [6 × $SiMe_2$]. Elemental analysis: $C_{36}H_{80}Si_{12}Sn$, MW = 968.76 g mol^{-1}. Found (Calc.): C, 43.9 (44.6); H, 8.1 (8.3).

*3.1.8. Bis(undecamethylcyclohexasilanyl)-dimethylgermane (**9**)*

Starting materials: 0.97 g (5.6 mmol) Me_2GeCl_2, 11.2 mmol KSi_6Me_{11}; colorless solid, m.p.: 148–150°C. Yield: 3.65 g (85%). ^{1}H-NMR: 0.13–0.24 (m) [22 × SiMe], 1.23 [2 × GeMe]. ^{13}C-NMR: −8.7 [2 × $GeMe_2$], −6.9, −4.7, −4.3, −2.6 [22 × SiMe/$SiMe_2$]. MS: 334 [20%, Si_6Me_{11}], 753 [30%, $M^+ - Me$], 768 [3%, M^+]. Elemental analysis: $C_{24}H_{72}Si_{12}Sn$, MW = 770.46 g mol^{-1}. Found (Calc.): C, 38.1 (37.4); H, 9.2 (9.4); Ge 11.4 (7.7); Si, 88.6 (92.3) (by EDXRFS [17]).

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[1] Prepared from 4.88g (14 mmol) dodecamethylcyclohexasilane and 1.57 g (14 mmol) potassium *tert*-butylate.

Table 2
Selected bond lengths (pm) and bond angles (°) of the structures of **3** and **5**

	3	**5**
Bond lengths		
Si(1)–Sn(1)	259.31(11)	259.3(2)
Si(7)–Sn(1)		258.9(2)
Si(1)–Si(2)	234.6(2)	234.9(3)
Si(2)–Si(3)	234.0(2)	234.2(3)
Si(3)–Si(4)	234.6(2)	234.7(3)
Si(4)–Si(5)	234.5(2)	233.6(3)
Si(5)–Si(6)	234.9(2)	235.2(3)
Si(6)–Si(1)	234.7(2)	234.9(3)
Bond angles		
C(18)–Sn(1)–C(24)	105.50(14)	102.7(5)
C(18)–Sn(1)–Si(1)	117.86(10)	109.1(3)
C(24)–Sn(1)–Si(1)	107.39(10)	110.7(3)
C(12)–Sn(1)–Si(1)	112.26(10)	
C(12)–Sn(1)–C(18)	106.19(14)	
C(12)–Sn(1)–C(24)	106.98(14)	
C(18)–Sn(1)–Si(7)		109.6(3)
C(24)–Sn(1)–Si(7)		110.5(3)
Si(1)–Sn(1)–Si(7)		113.65(6)

Table 3
Summary of data collection, structure solution and refinement of **3** and **5**

	3	**5**
Empirical formula	$C_{29}H_{48}Si_6Sn$	$C_{24}H_{72}Si_{12}Sn$
Formula weight	683.905	816.59
Space group	Triclinic, $P\bar{1}$	Triclinic, $P\bar{1}$
Crystal color, habit	Colorless, plate	Colorless, plate
Crystal size (mm)	0.40 × 0.15 × 0.05	0.40 × 0.32 × 0.04
a (pm)	869.24(2)	984.03(4)
b (pm)	1801.95(5)	1494.30(6)
c (pm)	2499.16(6)	1769.12(7)
α (°)	76.648(1)	100.881(1)
β (°)	80.601(1)	98.369(1)
γ (°)	88.405(1)	100.544(1)
V	3757.4(2)	2467.4(2)
Z	4	2
D_{calc} (g cm^{-3})	1.209	1.099
Linear absorption coefficient (mm^{-1})	0.887	0.823
Radiation	Mo–K$_\alpha$	Mo–K$_\alpha$
T (K)	150(2)	150(2)
θ Range for data collection	1.16–28.27	1.19–25.00
Independent reflections (R_{int})	16 414 [0.0270]	8270 [0.0370]
Number of parameters	650	334
Goodness-of-fit on F^2	1.125	1.114
R_1, wR_2 [$I>2\sigma(I)$]	0.0527, 0.0877	0.0711, 0.1549
R_1, wR_2 (all data)	0.0771, 0.0973	0.1067, 0.1970

*3.2. Reaction of **3** with dry oxygen*

A total of 2.4 g (3.5 mmol) of **3** were placed in a one-necked flask fitted with a drying tube ($CaCl_2$). After 4 weeks the residue was analyzed by NMR spectroscopy. ^{119}Sn-NMR: −144 ppm, $^{1}J(^{119}Sn–^{117}Sn)$: 4420 Hz [23]. ^{29}Si-NMR: 6.3, −43.7, −42.5, −42.2 ppm [12].

*3.3. Reaction of **5** with mercury dichloride*

A total of 2.5 g (9.2 mmol) mercury dichloride and 20 ml of thf were placed in a Schlenk tube. A solution of 3.75 g (4.6 mmol) **5** in 50 ml of thf was added slowly at room temperature over a period of 2 h. After an additional 4 h of stirring the reaction was complete and the solvent was then removed in vacuo. Sublimation in vacuo yielded 2.90 g of **11** (83%). The physical and spectroscopic data of **11** are given in [10].

*3.4. Crystallography of **3** and **5***

The crystals were mounted on the diffractometer as previously described [24]. Intensity data were collected with graphite monochromated Mo–K$_\alpha$ radiation ($\lambda = 0.71073$ Å), using a Siemens SMART system, complete with three-circle goniometer and CCD detector operating at −54°C. An absorption correction was applied utilizing the program SADABS [25]. The crystal structure was solved by direct methods, as included in the SHELXTL-PLUS program package [26]. Missing atoms were located in subsequent difference Fourier maps and included in the refinement. Remaining hydrogen atoms were placed geometrically and refined using a riding model with U_{iso} constrained at 1.2 for non-methyl groups, and 1.5 for methyl groups times U_{eq} of the carrier carbon atom. The structure was refined by full-matrix least-squares refinement on F^2 (SHELXL-93) [27] (Tables 2 and 3). Scattering factors were those provided with the SHELXL program system. All non-hydrogen atoms, with the exception of some disordered or restrained positions were refined anisotropically. Disorder was handled by including split positions for the affected groups, and included in the refinement of the respective occupancies. A set of restraints was applied to aid in modeling the disorder.

4. Supplementary material

Crystallographic data for the structural analysis have been deposited with the Cambridge Crystallographic Data Centre, CCDC no. 101462 for compounds **3** and **5**. Copies of this information may be obtained free of charge from: The Director, CCDC, 12 Union Road, Cambridge, CB2 1EZ, UK (Fax: +44-1223-336033; e-mail: deposit@ccdc.cam.ac.uk or www: http://www.ccdc.cam.ac.uk).

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