ELSEVIER

Journal of Organometallic Chemistry 499 (1995) 241–246

Journal of Organo metallic Chemistry

Syntheses, structures and properties of dihydroxypermethylcyclosilanes and permethyloxahexasilanorbornanes ☆

A. Spielberger [a], P. Gspaltl [a], H. Siegl [a], E. Hengge [a,*], K. Gruber [b]

[a] *Institute of Inorganic Chemistry, Technical University Graz, Stremayrgasse 16, A-8010 Graz, Austria*
[b] *Institute of Physical Chemistry, University of Graz, Heinrichstraße 28, A-8010 Graz, Austria*

Received 23 December 1995; in revised form 10 February 1995

Abstract

The separation of 1,3- and 1,4-dichlorodecamethylcyclohexasilanes and the isolation of 1-chloro-3-(chlorodimethylsilyl)octamethylcyclopentasilane through their hydrolysis products are shown. A crystal structure determined by X-ray diffraction of a single crystal containing decamethyl-7-oxahexasilanorbornane and 1,4-dihydroxydecamethylcyclohexasilane is discussed.

Keywords: Silicon; Crystal structure; Cyclosilanes

1. Introduction

For the formation of new cyclosilane derivatives or the syntheses of polycyclic silanes partial substituted cyclosilanes are required as starting materials. Some years ago, monosubstituted methylcyclosilanes were synthesized by reaction of antimony chloride or aluminium chloride and dodecamethylcyclohexasilane [1,2]. At forced reaction conditions higher chlorinated products are formed as well the main products being isomeric mixtures of disubstituted methylcyclosilanes [3,4]. In the case of the six-membered ring 1,3- and 1,4-dichlorodecamethylcyclohexasilane are formed [5]. Separation of these isomers is important for their use as starting materials for further syntheses. It should be mentioned that at longer reaction time the highest chlorinated product is 1,3,5-trichlorononamethylcyclohexasilane [6]. In this case no other isomers were obtained.

Wojnowsky et al. [2] have shown, that 1,4-dichlorodecamethylcyclohexasilane can form a sulphur bridge between the silicon atoms 1 and 4. Our first attempts to separate the disubstituted isomers were performed using this sulphur derivative. We repeated the experiments of Wojnowsky et al. and found that the 1,4-isomer affords the sulphur bridged compound, whereas the 1,3-isomer forms only polymeric material. The 1,4-derivative was crystallized from the solution and separation became possible. With $SbCl_3$ the 1,4-dichloride could be recovered from the sulphur compound.

Another separation was performed via substitution with tricarbonyl iron complexes. Both isomers, 1,3- and 1,4-dichlorodecamethylcyclohexasilane, reacted to form 1,3- and 1,4-bis(dicarbonylcyclopentadienylferrat)decamethylcyclohexasilanes, respectively. Separation of these derivatives was made possible by their different solubilities. The iron complex ligands were substituted by bromide [5].

2. Results and discussion

Both separation methods described above are difficult and troublesome. We report here a very effective and simple route for the separation of these isomers via the hydroxy derivatives. A mixture of the dichlorodecamethylcyclohexasilanes (**1**, **2**) was hydrolyzed under the following conditions. At a pH of ca. 9 and in the absence of oxygen solutions of triethylamine and the dichlorides mixture in n-pentane were dropped simultaneously into a water/pentane two-phase system at room temperature. **1** reacted to form decamethyl-7-oxahexasilanorbornane (**3**) and 1,4-dihydroxydecamethylcyclohexasilane (**5**). In contrast, **2** only gave the 1,3-dihydroxydecamethylcyclohexasilane (**4**).

☆ Dedicated to Professor Sakurai on occasion of his retirement.

* Corresponding author.

SSDI 0022-328X(95)00308-8

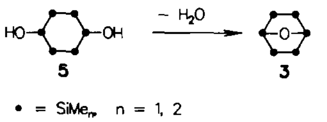

Fig. 1. Condensation reaction of 1,4-dihydroxydecamethylcyclohexasilane (**5**).

The formation of an oxygen bridge is possible only with the 1,4-dihydroxy derivative **5** probably as a result of steric factors. The situation is similar to that described above for the sulphur derivative. We were only able to isolate a crystal with a stoichiometric composition **3** : **5** = 2 : 1. The crystal structure is described below. **5** decomposed slowly at room temperature to give **3** (Fig. 1) and the reaction was becoming quantitative at the higher temperature during distillation. **4** was stable under these conditions.

The difference between the boiling points (ca. 30°C) was sufficient to enable the separation of compounds **3** and **4** (Fig. 2). It was then possible to split the Si–O bond with acetylchoride, and 1,3- and 1,4-dichlorodecamethylcyclohexasilane, respectively, were formed in quantitative yield.

A very similar reaction sequence was possible with the five-membered ring system, but separation of the 1,2- and 1,3-disubstituted products was troublesome. Five-membered ring systems were formed by the well-known ring contraction reaction of dodecamethylcyclohexasilane with $AlCl_3$ in the presence of trichloromethylsilane [1] to give (chlorodimethylsilyl)nonamethylcyclopentasilane or a mixture of 1-chloro-2-(chlorodimethylsilyl)octamethylcyclopentasilane (**6**) and 1-chloro-3-(chlorodimethylsilyl)octamethylcyclopentasilane (**7**), depending on the reaction conditions. We recently found a convenient way to split the side chain from the ring [7], but separation of the isomers was still difficult. However, with the new method using hydrolysis, we successfully separated the five-membered rings (Fig. 3).

The 1,3-substituted derivative **7** gave decamethyl-2-oxahexasilanorbornane (**8**) (Fig. 3) or 1-hydroxy-3-(hydroxydimethylsilyl) octamethylcyclopentasilane (**9**) depending on the reaction conditions. The 1,2-substituted compound could not be isolated because of the formation of polymeric structures.

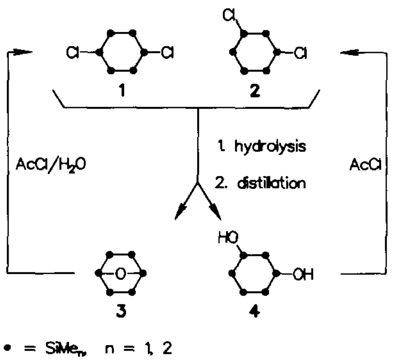

Fig. 2. Separation of 1,3- and 1,4-disubstituted decamethylcyclohexasilanes.

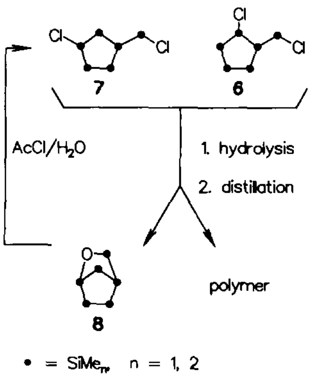

Fig. 3. Isolation of 1,3-substituted octamethylcyclopentasilane.

3. Structural analysis of 3 and 5

A single crystal (containing **3** and **5** in ratio of 2 : 1) suitable for X-ray diffraction analysis was grown in diethyl ether. The structures of **3** and **5** were determined under a cold stream of nitrogen at −186°C using a modified STOE four-circle diffractometer and graphite monochromated Mo-Kα ($\lambda = 0.71073$ Å) radiation. Detailed crystal data are given in Table 1. The crystal was mounted on the tip of a glass fibre in inert oil. Space group $P\bar{1}$ was chosen and later confirmed by successful structure solution. The structure was solved by direct methods using XS [8] and refined by the full-matrix least-squares method with SHELXL-93 [9] minimising the residuals for F^2. All hydrogen atoms were visible in difference Fourier maps and were included in the model in their calculated positions. An additional torsion angle was refined for each methyl and hydroxy group maintaining an idealised geometry. Anisotropic displacement parameters were assigned to all nonhydrogen atoms, isotropic displacement parameters were used for hydrogen atoms. An empirical volume and absorption correction (DIFABS) was performed [10]. The maximum and minimum transmission coefficients were 1.0 and 0.5, respectively.

The structure shown in Fig. 4 gives a good impression of the arrangement of the three molecules in the unit cell. They appear to be connected by an electrostatic interaction. The atoms O(1), H(1), O(2) and Si(7) are coplanar with a rms deviation of 0.03 Å. Furthermore, we observed an angle of 155.92(9)° for O · · · H–O with the two oxygen atoms O1, O2 lying 2.782 Å apart. This could be interpreted as a O(1) · · · H(1) hydrogen bond with a bond distance of 1.996 Å. To illustrate this, the two calculated hydroxy hydrogen atoms are shown.

TABLE 1. Crystallographic data and some experimental details for a crystal containing **3** and **5**

Crystal data	
Empirical formula	$C_{15}H_{46}O_2Si_9$
Formula weight	511.33
Crystal size (mm)	approx. 0.5 × 0.4 × 0.2
Crystal system	triclinic
Space group	$P\bar{1}$
a (Å)	9.219(9)
b (Å)	11.669(10)
c (Å)	16.167(9)
α (°)	98.46(6)
β (°)	99.75(7)
γ (°)	110.08(7)
Volume (Å^3)	1570(2)
Z	2
Density (calc.; g cm^{-3})	1.082
Absorption coeff. (mm^{-1})	0.389
$F(000)$	556
2Θ range (°)	5.7–76
Index ranges	$-5/h/15$ $-19/k/14$ $-26/l/25$
Reflections collected	15886
Independent reflections	14654 (R_{int} = 0.0279)
Observed reflections	11127 ($I > 2\sigma(I)$)
No. of parameters refined	297
Weighting scheme	$1/(\sigma^2(F_0^2)+(0.0799P)^2+0.1104P)$; $P=(F_0^2+2F_c^2)/3$
Final R indices	$R1 = 0.0444$, $wR2 = 0.1218$
R indices (all data)	$R1 = 0.0635$, $wR2 = 0.1313$
Goodness-of-fit on F^2	1.051
Largest diff. peak	0.767 e Å^{-3}
Largest diff. hole	−0.692 e Å^{-3}

The asymmetric unit comprises one molecule of compound **3** and one half molecule of **5**. It is related to the second half of the unit cell by an inversion operation with the centre of symmetry in the middle of the **5** molecule. For clarity, the atoms of only one unit are labelled in Fig. 4.

Fractional coordinates and equivalent isotropic thermal parameters for one asymmetric unit are given in Table 3; selected intramolecular angles and distances for compound **3** are shown in Table 2.

TABLE 2. Selected intramolecular angles (°) and distances (Å) with estimated standard deviations for compound **3**

Bond angles (°)		Bond length (Å)	
O(1)–Si(1)–Si(2)	100.85(8)	O(1)–Si(1)	1.695(2)
O(1)–Si(1)–Si(6)	99.67(7)	O(1)–Si(4)	1.700(2)
O(1)–Si(4)–Si(3)	98.62(9)	Si(1)–Si(2)	2.369(2)
O(1)–Si(4)–Si(5)	100.50(8)	Si(1)–Si(6)	2.353(2)
Si(1)–O(1)–Si(4)	116.21(9)	Si(2)–Si(3)	2.358(3)
Si(1)–Si(2)–Si(3)	96.08(8)	Si(3)–Si(4)	2.362(2)
Si(1)–Si(6)–Si(5)	96.16(7)	Si(4)–Si(5)	2.362(2)
Si(2)–Si(1)–Si(6)	116.83(6)	Si(5)–Si(6)	2.358(3)
Si(2)–Si(3)–Si(4)	96.39(8)		
Si(3)–Si(4)–Si(5)	118.21(5)		
Si(4)–Si(5)–Si(6)	96.50(7)		

X-ray data for compounds **3** and **5** have been deposited at the Cambridge Crystallographic Data Centre, Cambridge, UK.

4. Experimental details

4.1. General comments

Unless otherwise noted, al! reactions and manipulations were carried out under an atmosphere of nitrogen. Solvents were dried and distilled prior to use. Water was degassed for 20 min using an ultrasonic bath in vacuo (3 mbar). Melting points were determined with a Büchi melting point apparatus and are shown uncorrected. C and H analyses were performed on a Haraeus-Mikro-K1 apparatus. NMR spectra (NMR: ^1H, 300.15 MHz; ^{13}C, 75.47 MHz; ^{29}Si, 59.627 MHz) were recorded on a Bruker MSL 300 spectrometer at 20°C. Samples were dissolved in C_6D_6 or $CDCl_3$ and tetramethylsilane was used as an external standard. IR spectra were recorded with paraffin (SPEKTRANAL) and caesium bromide pellets on a Perkin Elmer 883 spectrophotometer. UV spectra from n-hexane (SPEKTRANAL) solutions

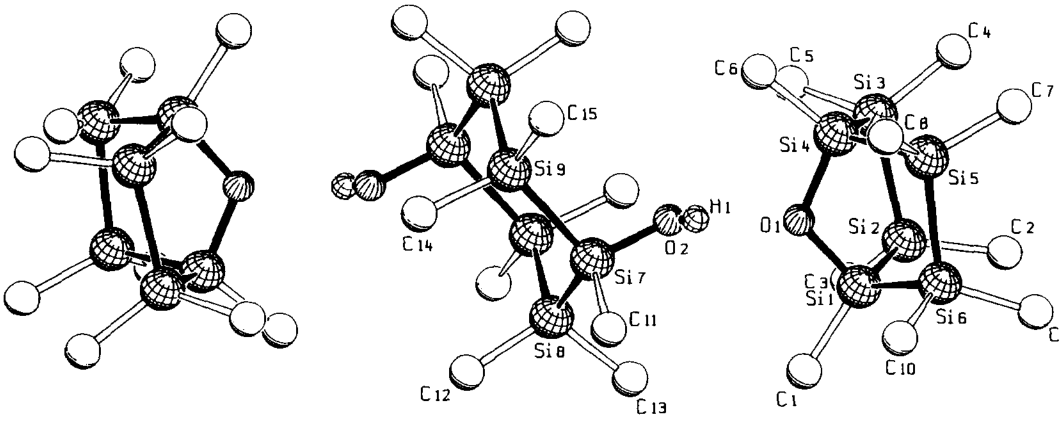

Fig. 4. X-ray sturcture and atom labelling of the three molecules (**3**–**5**–**3**) in the unit cell.

TABLE 3. Fractional atomic coordinates and equivalent isotropic thermal parameters (Å^2) for all nonhydrogen atoms of the asymmetric unit from a crystal containing compounds **3** and **5**

Atom	x	y	z	U_{eq} [a]
Si(1)	0.3853(1)	0.8104(1)	0.8618(1)	0.018(1)
Si(2)	0.1488(1)	0.8514(1)	0.8439(1)	0.023(1)
Si(3)	0.0064(1)	0.6891(1)	0.7209(1)	0.019(1)
Si(4)	0.2003(1)	0.5994(1)	0.7210(1)	0.017(1)
Si(5)	0.2323(1)	0.4831(1)	0.8259(1)	0.021(1)
Si(6)	0.3965(1)	0.6559(1)	0.9384(1)	0.020(1)
Si(7)	0.5946(1)	0.9468(1)	0.6164(1)	0.018(1)
Si(8)	0.6242(1)	1.1548(1)	0.6185(1)	0.017(1)
Si(9)	0.5837(1)	0.8346(1)	0.4810(1)	0.018(1)
O(1)	0.3656(1)	0.7321(1)	0.7610(1)	0.019(1)
O(2)	0.4227(2)	0.8850(1)	0.6439(1)	0.028(1)
C(1)	0.5719(2)	0.9541(2)	0.8879(1)	0.027(1)
C(2)	0.0428(2)	0.8341(1)	0.9340(1)	0.051(1)
C(3)	0.1940(3)	1.0127(2)	0.8218(2)	0.052(1)
C(4)	−0.1965(2)	0.5887(2)	0.7303(1)	0.032(1)
C(5)	−0.0067(2)	0.7499(2)	0.6193(1)	0.031(1)
C(6)	0.2140(2)	0.5335(2)	0.6111(1)	0.027(1)
C(7)	0.0469(3)	0.3871(3)	0.8547(1)	0.053(1)
C(8)	0.3389(3)	0.3797(2)	0.7892(2)	0.044(1)
C(9)	0.3214(3)	0.6643(2)	1.0394(1)	0.037(1)
C(10)	0.6074(2)	0.6631(2)	0.9636(1)	0.032(1)
C(11)	0.7647(2)	0.9489(2)	0.7006(1)	0.031(1)
C(12)	0.8223(2)	1.2487(2)	0.5985(1)	0.029(1)
C(13)	0.6129(2)	1.2194(2)	0.7306(1)	0.026(1)
C(14)	0.7746(2)	0.9071(2)	0.4469(1)	0.029(1)
C(15)	0.5477(2)	0.6652(2)	0.4813(1)	0.029(1)

[a] Equivalent isotropic displacement parameter U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

of the compounds were obtained in a quartz cuvette with 1 cm diameter on a Philips PU 8740 spectrometer. All reactions were monitored by GC. GC and MS data were obtained on a Hewlett-Packard 5890-II model linked with a Hewlett-Packard 5971 Mass Selective Detector (GC–MS). The gas chromatograph was equipped with a cross-linked dimethylpolysiloxane column (25 m × 0.32 mm × 0.1 μm) and helium was used as carrier gas.

4.2. Decamethyl-7-oxahexasilanorbornane (3) and dihydroxydecamethylcyclohexasilane (4)

One dropping funnel was filled with n-pentane (400 ml) and triethylamine (120 g, 1.19 mol), a second with n-pentane (400 ml) and (93.0 g, 0.238 mol) of an isomeric mixture of dichlorodecamethylcyclohexasilanes (**1**, **2**). A two-phase system of n-pentane (400 ml) and water (300 ml) was stirred in a flask. The pH of the water was adjusted to 9 with a few drops of the triethylamine solution. During a 5 h period the triethylamine solution and the **1**, **2** mixture were dropped into the vigorously stirred two-phase system. The cloudiness of the organic solvent disappeared and both phases were clear once the reaction was complete. The aqueous phase was withdrawn and the organic solvent removed under vacuum at room temperature. Vacuum distillation of the residue was performed with a 25-cm-long and 2.5-cm wide Vigreux column. At 95–13°C (0.02 mbar) the first fraction was collected, the second distilling at 125–135°C (0.02 mbar). By isothermal evaporation at room temperature **3** was crystallized from a diethyl ether (30 ml) solution of the first fraction. Recrystallization of the second fraction from a n-heptane (20 ml) solution lead to compound **4**. Yields: 20.8 g (26.2%) **3**; 35.3 g (42.0%) **4**. Analytical data for **3**: m.p. 81–83°C; b.p. 95°C (0.02 mbar). Anal. Found: C, 35.60; H, 9.16; Si, 50.14%. Calc. $C_{12}H_{36}OSi_6$ (334.86): C, 35.87; H, 9.03; Si, 50.32%. ^{1}H NMR (C_6D_6): δ 0.64 (6H), 0.30 (24H) ppm. ^{13}C NMR (C_6D_6): δ −2.10 (2CH$_3$), −5.58 (4CH$_3$), −6.29 (4CH$_3$) ppm. ^{29}Si NMR (C_6D_6): δ 14.80 (2Si), −42.97 (4Si) ppm. GC–MS (70 eV): selected m/e (rel. intensity) 334 (100) M$^+$, 259 (52), 245 (50), 73 (99) (%). UV: $\lambda_{max}(\varepsilon)$ 236 (4300) sh, 296 (2700), 324 (1600) nm (1 mol^{-1} cm^{-1}). IR: $\bar{\nu}$ 3375, 1243, 871, 832, 778, 752, 731, 691, 658, 636, 475, 391 cm^{-1}. Analytical data for **4**: m.p. 126–128°C. b.p. 125°C (0.02 mbar). Anal. Found: C, 34.20; H, 9.25; Si, 47.96%. Calc: $C_{10}H_{32}O_2Si_6$ (352.87): C, 34.04; H, 9.14; Si, 47.75%. ^{1}H NMR (CDCl$_3$): δ 2.74 (2H), 0.46 (6H), 0.29 bis 0.9 (24H) ppm. ^{13}C NMR (CDCl$_3$): δ −1.08, −5.64, −6.00, −6.77, −7.15, −7.81 ppm. ^{29}Si NMR (CDCl$_3$): δ 16.91 (2Si), −43.08 (1Si), −44.60 (2Si), −48.07 (1Si) ppm. GC–MS (70 eV): selected m/e (rel. intensity) 334 (26), 293 (26), 259 (19), 245 (19), 73 (100) (%). UV: λ_{max} (ε) 242 (3600), 256 (3300), 286 (700) nm (1 mol^{-1} cm^{-1}). IR: $\bar{\nu}$ 3327, 1246, 824, 799, 779, 743, 674, 655, 639, 396 cm^{-1}.

4.3. 1,4-Dihydroxydodecamethylcyclohexasilane (5)

Although **5** readily condenses at room temperature to **3** it can be detected by gaschromatographic analysis from a water-saturated solution of **3** in diethyl ether. Therefore, a mass spectrum was obtained by GC–MS analysis. Analytical data for **5**: GC–MS (70 eV): selected m/e (rel intensity) 334 (10), 277 (12), 293 (55), 259 (19), 245 (18), 73 (100) (%).

4.4. Decamethyl-2-oxahexasilanorbornane (8)

A dropping funnel was filled with n-pentane (80 ml) and triethylamine (21.9 g, 0.217 mol), another one was charged with n-pentane (80 ml) and a mixture of 1-chloro-2-(chlorodimethylsilyl)octamethylcyclopentasilane (**6**) and 1-chloro-3-(chlorodimethylsilyl)octamethylcyclopentasilane (**7**) (16.9 g, 0.0433 mol). A two-phase system with 80 ml of n-pentane and 60 ml of water was stirred in a flask. With a few drops of the triethylamine solution the pH-value of the water was adjusted to 9. During a 3 h period the triethylamine solution and the solution of **6**, and **7** mixture were added dropwise to the vigorously stirred two phase system. The cloudiness in the organic solvent disappeared and both phases were clear after the reaction was complete. The aqueous phase was withdrawn and the organic solvent removed by vacuum evaporation at room temperature. Vacuum distillation of the residue was performed with a 25-cm long Vigreux column. At 98–110°C (0.02 mbar) a distillation fraction was collected, dissolved in diethyl ether (25 ml) and **8** was crystallized at −30°C. Yield: 3.30 g (22.8%) **8**. Analytical data for **8**: m.p. 70–73°C; b.p. 98°C (0.02 mbar). Anal. Found: C, 35.59; H, 8.99; Si, 50.03%. Calc. $C_{10}H_{30}OSi_6$ (334.86): C, 35.87; H, 9.03; Si, 50.32%. ^1H NMR (C_6D_6): δ 0.58 (3H), 0.46 (3H), 0.41 (3H), 0.40 (3H), 0.37 (3H), 0.36 (3H), 0.31 (6H), 0.25 (3H), 0.15 (3H) ppm. ^{13}C NMR (C_6D_6): δ 5.84, 5.55, −1.77, −2.99, −3.07, −4.35, −5.48, −5.91, −6.45, −14.50 ppm. ^{29}Si NMR (C_6D_6): δ 19.44, 14.91, −39.87, −42.23, −46.14, −82.88 ppm. GC–MS (70 eV): selected *m/e* (rel. intensity) 334 (52), 259 (24), 245 (24), 73 (100) (%). UV: λ_{max} (ε) 293 (800) nm (1 mol^{-1} cm^{-1}). IR: $\tilde{\nu}$ 1246, 1014, 926, 823, 804, 764, 691, 655, 608, 457 cm^{-1}.

4.5. *1-Hydroxy-3-(hydroxydimethylsilyl)octamethylcyclopentasilane (9)*

One dropping funnel was filled with 40 ml of n-pentane and 13 g (0.13 mol) of triethylamine a second one with 40 ml of n-pentane and 10 g (0.026 mol) of a mixture containing **6** and **7**. A two-phase system with 40 ml of n-pentane and 30 ml of water was stirred in a flask. With few drops of the triethylamine solution the pH value of the water was set to 11. During a 3 hour period the triethylamine solution and the solution of **6** and **7** was added dropwise to the vigorously stirred two phase system. The cloudiness in the organic solvent disappeared and both phases were clear after the reaction was complete. The aqueous phase was withdrawn and from the organic solution the formed inorganic salts were extracted three times with water (3 × 10 ml). The organic solvent and excess triethylamine were removed by vacuum evaporation at 60°C. The white oily residue was dissolved in n-pentane (60 ml) and crystallized at −70°C. Yield: 2.1 g (23.2%) **9**. Determination of the m.p. was not possible due to inter- and intramolecular condensation reactions of **9**. Anal. Found: C, 34.26; H, 9.25; Si, 47.45%. Calc. $C_{10}H_{32}O_2Si_6$ (352.87): C, 34.04; H, 9.14; Si, 47.75%. ^1H NMR ($CDCl_3$): δ 0.48 (3H), 0.32 bis 0.17 (27H) (OH hydrogen did not appear) ppm. ^{13}C NMR ($CDCl_3$): δ 4.58 ($2CH_3$), 0.47, −3.81, −4.34, −5.61, −5.86, −6.38, −6.96, −12.53 ppm. ^{29}Si NMR ($CDCl_3$): δ 22.54, 18.82, −40.15, −41.70, −45.86, −85.84 ppm.

4.6. *1,4-Dichlorodecamethylcyclohexasilane (1)*

Compound **3** (16 g, 0.048 mol) was dissolved in ice-cold acetylchloride (48 g, 0.48 mol). From a dropping funnel water (4.3 g, 0.24 mol) was added dropwise. After the addition of the water the ice bath was removed and the reaction stirred for 12 h. From the clear solution obtained excess acethylchloride and produced ethylacetate were removed by vacuum evaporation. The reaction was quantitative and **1** was identified using the literature data.

4.7. *1,3-Dichlorodecamethylcyclohexasilane (2)*

Compound **4** (18 g, 0.051 mol) was suspended in ice-cold acetylchloride (52 g, 0.51 mol) and stirred at room temperature for 12 h. From the clear solution obtained excess acethylchloride and ethylacetate produced were removed by vacuum evaporation. The reaction was quantitative and **2** was identified using the literature data.

4.8. *1-Chloro-3-(chlorodimethylsilyl)octamethylcyclopentasilan (7)*

Compound **8** (3.50 g, 0.0104 mol) was suspended in ice-cold acetylchloride (22 g, 0.28 mol). To the stirred solution water (1.0 g, 0.056 mol) was added dropwise and the ice bath removed after 1 h. After stirring for additional 12 h a clear solution was obtained and the excess acetylchloride and the ethylacetate produced were removed by vacuum evaporation. Using a 10 cm Vigreux column vacuum distillation of the residue gave semicrystalline **7** (*cis* and *trans*). Yield: 3.58 g (88%) **7**. Analytical data of mixed *cis* and *trans* isomers *7*: m.p. 59–61°C. b.p. 91°C (0.02 mbar). Anal. Found: C, 30.87; H, 7.89%. Calc: $C_{10}H_{30}Cl_2Si_6$ (389.77): C, 30.82; H, 7.76%. ^1H NMR ($CDCl_3$): δ 0.59/0.58 (6/6H), 0.38 bis 0.19 (24/24H) ppm. ^{13}C NMR ($CDCl_3$): δ 5.40 ($2/2CH_3$), −0.32/−0.71 ($1/1CH_3$) −2.93 to −6.76 ($6/6CH_3$), −12.09/−12.18 ($1/1CH_3$) ppm. ^{29}Si NMR ($CDCl_3$): δ 31.32/30.96, 19.22/18.66, −36.99/−37.15, −38.72/−38.78, −41.33/−41.95, −79.21/−80.63 ppm. GC–MS (70 eV): selected *m/e* (rel. intensity) 388 (9) M^+, 295 (10), 280 (16), 73 (100) (%). UV: λ_{max} (ε) 290 (900) nm (1 mol^{-1} cm^{-1}). IR: $\tilde{\nu}$ 1716, 1247, 841, 809, 770, 733, 696, 664, 500 cm^{-1}.

Acknowledgement

The authors thank the Fond zur Förderung der wissenschaftlichen Forschung, Vienna for financial support of project P 09751-CHE. We are also grateful to

Wacker-Chemie GmbH for gifts of several silane derivatives.

References

[1] M. Ishikawa and M. Kumada, *Synth. React. Inorg. Met.-Org. Chem.*, *1* (1971) 229.

[2] W. Wojnowski, B. Dreczewski, A. Herman, K. Peters, E.M. Peters and H.G. von Schnering, *Angew. Chem.*, *97* (1985) 978.

[3] T.J. Drahnak, R. West and J.C. Calabrese, *J. Organomet. Chem.*, *198* (1980) 55.

[4] P.K. Jenkner and E. Hengge, *J. Organomet. Chem.*, *430* (1992) 259.

[5] E. Hengge and M. Eibl, *J. Organomet. Chem.*, *428* (1992) 335.

[6] M. Eibl, U. Katzenbeißer and E. Hengge, *J. Organomet. Chem.*, *444* (1993) 29.

[7] E. Hengge, P.K. Jenkner, A. Spielberger and P. Gspaltl, *Monatsh. Chem.*, *124* (1993) 1005.

[8] SHELXTL 4.1, Siemens Crystallographic Research System, 1990.

[9] SHELXL-93 Program for crystal structure refinement. G.M. Sheldrick, University of Göttingen, 1993.

[10] N. Walker, D. Stuart, *Acta Cryst.*, *A39* (1983) 158.