Crystal and Molecular Structure of Cyclic α,α′-Silaalkadiynes $[-(CH_3)_2Si-C{\equiv}C-Si(CH_3)_2(CH_2)_n-]_2$, n = 1–4

Gudveig Åmdal Eliassen, Else Kloster-Jensen and Christian Rømming*

Department of Chemistry, University of Oslo, P.B. 1033, Blindern, 0315 Oslo 3, Norway

Eliassen, Gudveig Åmdal, Kloster-Jensen, Else and Rømming, Christian, 1986. Crystal and Molecular Structure of Cyclic α,α′-Silaalkadiynes $[-(CH_3)_2Si-C{\equiv}C-Si(CH_3)_2(CH_2)_n-]_2$, n = 1–4. – Acta Chem. Scand. B 40: 574–582.

The structures of the four title compounds: 3,3,5,5,8,8,10,10-octamethyl-3,5,8,10-tetrasilacyclodeca-1,6-diyne, *1*; 3,3,6,6,9,9,12,12-octamethyl-3,6,9,12-tetrasilacyclododeca-1,7-diyne, *2*; 3,3,7,7,10,10,14,14-octamethyl-3,7,10,14-tetrasilacyclotetradeca-1,8-diyne, *3*; and 3,3,8,8,11,11,16,16-octamethyl-3,8,11,16-tetrasilacyclohexadeca-1,9-diyne. *4*, have been determined from X-ray data. Full matrix least-squares refinements led to final conventional *R* values of 0.040(5251) for *1*, 0.039(2471) for *2*, 0.059(2065) for *3* and 0.029(3243) for *4*. (The number of observed reflections in parentheses.) The crystal data for the compounds are given in Table 2. The crystals of *1* contained two different conformers – chair and boat forms. The rings with an even number of methylene groups connecting silicon atoms were more strained than those with an odd number, as shown by the deviation from a staggered conformation along the polymethylene chains. Corresponding Si-C bonds in the compounds were all of equal length, but three different types were observed with weighted mean values: Si-C (methyl) 1.867(1) Å, Si-C (methylenic) 1.877(1) Å, and Si-C (acetylenic) 1.843(1) Å. The average length of the C-C triple bond was 1.217(1) Å.

Syntheses of several series of monocyclic α-silaalkynes built up from a varying number of α,α′-disilaethynyl and polymethylene units alternating along the ring chain were recently published.[1] Within each series, the polymethylene fragment varied in length. In one of these series, two α,α′-(dimethylsilyl)-ethynylic units were connected through two polymethylene bridges each with *n* CH_2 links as shown in Fig. 1. The compound with $n = 0$ was first prepared by an alternative route by Sakùrai *et al.*[2] An X-ray crystallographic analysis revealed a planar ring structure analogous to the parent compound 1,5-cyclooctadiyne.[3] The present article describes X-ray investigations of the first four homologues in the series in which one, two, three or four methylene groups, respectively, connect two silicon atoms ($n = 1,2,3,4$).

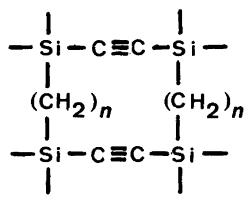

Fig. 1. Compounds *1* to *4* with $n = 1,2,3$, and 4, respectively.

Experimental

The four compounds were prepared and purified as earlier described.[1] Suitable crystals for the X-ray experiments were grown by vacuum sublimation. Details of the collection of X-ray data are given in Table 1. The standard deviations for the intensities were taken as $\sigma(I) = [C_T + (0.02\ C_N)^2]^{1/2}$, where C_T is the total number of counts and C_N is the net count. The intensities were corrected for Lorentz and polarization effects but not for absorption.

A description of the computer programs used for the structure analyses is given in Ref. 4.

*To whom correspondence should be addressed.

Table 1. Experimental details.

	1	*2*	*3*	*4*
Diffractometer	Syntex $P\bar{1}$	Nicolet *P3/F*	Nicolet *P3/F*	Nicolet *P3/F*
Radiation	Mo*K*	Mo*K*	Mo*K*	Mo*K*
Crystal dimensions (mm)	0.1×0.3×0.3	0.2×0.3×0.3	0.1×0.2×0.3	0.2×0.3×0.4
Scan mode	θ/2θ	θ/2θ	θ/2θ	θ/2θ
Scan speed (2θ, °min^{-1})	3–6	3–6	5–10	3–6
Scan range (2θ, °)	1.7	2.2	2.2	2.0
Maximum sinθ/λ (Å^{-1})	0.76	0.76	0.54	0.76
No. of independent meas.	7326	3825	3077	4025
No. with $I > 2.5\,\sigma(I)$	6639	3013	2065	3902

Table 2. Crystal data.

Compound	$C_{14}H_{28}Si_4$[a]	$C_{16}H_{32}Si_4$[b]	$C_{18}H_{36}Si_4$[c]	$C_{20}H_{40}Si_4$[d]
M.p./°C	103–104	122–123	162–162.5	86–87
Crystal system	triclinic	monoclinic	triclinic	triclinic
a/Å	10.233(2)	10.057(2)	10.188(2)	6.302(1)
b/Å	9.556(2)	9.137(1)	11.402(2)	9.975(2)
c/Å	15.258(3)	11.975(2)	12.581(2)	10.714(2)
α°	101.55(2)		63.05(1)	89.68(2)
β°	101.71(2)	107.63(1)	68.09(1)	82.20(2)
γ°	93.07(2)		68.41(1)	72.51(2)
V/Å^3	1424.6(5)	1048.8(3)	1173.3(4)	636.0(2)
Temp./°C	−150	−133	−133	−133
Space group	$P\bar{1}$	$P2_1/c$	$P\bar{1}$	$P\bar{1}$
M	308.72	336.77	364.82	392.88
Z	3	2	2	1
F(000)	504	368	400	216
D_x/g cm^{-3}	1.079	1.066	1.033	1.026

[a] 3,3,5,5,8,8,10,10-octamethyl-3,5,8,10-tetrasilacyclodeca-1,6-diyne.
[b] 3,3,6,6,9,9,12,12-octamethyl-3,6,9,12-tetrasilacyclododeca-1,7-diyne.
[c] 3,3,7,7,10,10,14,14-octamethyl-3,7,10,14-tetrasilacyclotetradeca-1,8-diyne.
[d] 3,3,8,8,11,11,16,16-octamethyl-3,8,11,16-tetrasilacyclohexadeca-1,9-diyne.

Atomic scattering factors were those of Doyle and Turner[5] for Si and C, and of Stewart, Davidson and Simpsom[6] for H. Crystal data are given in Table 2.

Structure determination and refinement

The four structures were solved by direct methods[7] and refined by Fourier and least-squares calculations. Positions for hydrogen atoms were calculated and refined with isotropic thermal parameters. In order to reduce the influence of valence electrons, the final refinements for *1, 2*

Table 3. Figures of merit.

	1	*2*	*3*	*4*
All data				
No. of refl.	6637	3013	2065	3887
R	0.040	0.039	0.051	0.033
R_w	0.048	0.046	0.059	0.046
S'	2.11	2.24	2.51	2.41
sinθ/λ >0.4 Å^{-1}				
No. of refl.	5251	2471		3243
R	0.040	0.037		0.029
R_w	0.046	0.039		0.031
S'	1.67	1.60		1.41

$S' = [\Sigma w \Delta F^2/(n-m)]^{1/2}$.

Table 4. Atomic coordinates.

1

Atom	X	Y	Z
Si1	−.39760(5)	.72274(6)	.40223(4)
Si2	.09246(5)	.76360(6)	.45721(4)
Si3	.07510(6)	.46327(6)	.30658(4)
Si4	−.41484(6)	.42316(6)	.24998(4)
Si5	.24660(5)	.10802(6)	.92574(4)
Si6	.73616(5)	.17151(6)	.98009(4)
C1	−.2132(2)	.7484(2)	.4267(2)
C2	−.0914(2)	.7580(2)	.4405(2)
C3	.1390(2)	.5772(2)	.4248(1)
C4	−.1096(2)	.4426(2)	.2803(2)
C5	−.2314(2)	.4318(2)	.2649(2)
C6	−.4524(2)	.5268(2)	.3583(1)
C7	−.4690(2)	.8316(3)	.3185(2)
C8	−.4518(2)	.7867(3)	.5122(2)
C9	.1676(2)	.8425(3)	.5805(2)
C10	.1494(2)	.8832(2)	.3866(2)
C11	.1296(2)	.5466(3)	.2174(2)
C12	.1343(2)	.2807(3)	.3004(2)
C13	−.4823(2)	.2313(2)	.2304(2)
C14	−.4871(2)	.4976(3)	.1488(2)
C15	.4301(2)	.1410(2)	.9445(2)
C16	.5517(2)	.1574(2)	.9581(2)
C17	.1988(2)	−.0850(2)	.9219(1)
C18	.1688(2)	.1515(3)	.8140(2)
C19	.1905(2)	.2285(2)	1.0203(2)
C20	.8007(2)	.3658(2)	1.0103(2)
C21	.7858(2)	.0830(3)	.8728(2)
H71	−.434	.814	.263
H72	−.449	.924	.344
H73	−.563	.809	.302
H81	−.417	.728	.556
H82	−.544	.773	.449
H83	−.419	.880	.538
H91	.143	.782	.618
H92	.141	.932	.596
H93	.260	.849	.589
H101	.106	.853	.323
H102	.238	.877	.389
H103	.138	.978	.410
H111	.086	.633	.209
H112	.111	.481	.160
H113	.215	.576	.236
H121	.109	.235	.344
H122	.225	.285	.306
H123	.095	.222	.241
H131	−.442	.191	.277
H132	−.473	.180	.173
H133	−.576	.229	.230
H141	−.446	.595	.153
H142	−.580	.515	.149
H143	−.482	.436	.094
H181	.199	.092	.766
H182	.189	.251	.816
H183	.076	.138	.803
H191	.243	.222	1.079
H192	.104	.203	1.016
H193	.199	.324	1.011
H201	.784	.406	1.061
H202	.761	.408	.961
H203	.893	.373	1.014
H211	.743	−.001	.851
H212	.878	.079	.883
H213	.764	.140	.826
H61	−.541	.517	.349
H62	−.412	.477	.403
H31	.111	.527	.464
H32	.231	.585	.434
H171	.221	−.139	.873
H172	.108	−.091	.913

2

Atom	X	Y	Z
Si1	.87550(3)	.15506(4)	.70044(3)
Si2	.68491(3)	−.15337(4)	.39315(3)
C1	1.0416(1)	.1766(2)	.6664(1)
C2	1.1508(1)	.1764(2)	.6429(1)
C3	.7884(1)	−.0095(1)	.6152(1)
C4	.7079(1)	.0170(1)	.4849(1)
C5	.9142(2)	.1201(2)	.8606(1)
C6	.7698(1)	.3247(2)	.6557(1)
C7	.5402(1)	−.1318(2)	.2531(1)
C8	.6551(2)	−.3170(2)	.4755(1)
H31	.730	−.050	.652
H32	.863	−.081	.619
H41	.628	.060	.479
H42	.756	.086	.450
H51	.965	.040	.879
H52	.828	.111	.876
H53	.955	.206	.901
H61	.740	.341	.571
H62	.815	.400	.696
H63	.679	.310	.667
H71	.557	−.059	.206
H72	.456	−.103	.271
H73	.528	−.223	.210
H81	.728	−.333	.548
H82	.659	−.400	.433
H83	.575	−.305	.497

3

Atom	X	Y	Z
Si1	.2445(2)	.4468(1)	.3073(1)
Si2	−.1936(2)	.3839(2)	.2825(1)
Si3	.1830(2)	.0540(2)	.7980(1)
Si4	−.2574(2)	−.0070(1)	.7741(1)
C1	.0795(6)	.4233(5)	.2993(5)
C2	−.0290(6)	.4078(5)	.2927(5)
C3	.0169(6)	.0304(5)	.7904(5)
C4	−.0911(6)	.0151(5)	.7838(5)
C5	.3302(6)	.2839(5)	.4170(5)
C6	.2322(6)	.2378(6)	.5458(5)
C7	.3018(6)	.1093(5)	.6374(5)
C8	−.2140(6)	.2070(5)	.3893(5)
C9	−.2281(6)	.1765(5)	.5231(5)
C10	−.2392(6)	.0306(5)	.6095(5)
C11	.1894(6)	.5861(6)	.3655(5)
C12	.3698(6)	.4916(6)	.1501(5)
C13	.2780(6)	−.1122(6)	.8981(5)
C14	.1276(7)	.1876(6)	.8628(5)
C15	−.1686(7)	.4090(6)	.1209(5)
C16	−.3525(6)	.5126(6)	.3306(6)
C17	−.4166(6)	.1150(5)	.8304(5)
C18	−.2761(6)	−.1840(5)	.8732(5)
H51	.411	.290	.426
H52	.359	.213	.379
H61	.156	.235	.538
H62	.187	.311	.586
H71	.388	.125	.652
H72	.347	.046	.608
H81	−.143	.143	.366
H82	−.302	.191	.380
H91	−.315	.243	.544
H92	−.131	.198	.532
H101	−.157	−.039	.587
H102	−.317	.021	.598
H111	.119	.556	.446
H112	.158	.680	.308
H113	.286	.598	.369
H121	.403	.418	.122
H122	.450	.512	.154
H123	.323	.568	.095
H131	.299	−.183	.867
H132	.209	−.141	.983
H133	.366	−.108	.907
H141	.086	.277	.806
H142	.223	.195	.876
H143	.074	.168	.939
H151	−.073	.350	.088
H152	−.171	.480	.079
H153	−.245	.421	.105
H161	−.349	.497	.412
H162	−.432	.528	.318
H163	−.344	.589	.285
H171	−.404	.201	.785
H172	−.420	.086	.916
H173	−.496	.111	.823
H181	−.188	−.240	.853
H182	−.364	−.198	.873
H183	−.271	−.193	.951

4

Atom	X	Y	Z
Si1	.50879(4)	.16137(2)	.74527(2)
Si2	−.08578(4)	.61447(2)	.78294(2)
C1	.2904(2)	.3337(1)	.7627(1)
C2	.1429(2)	.4465(1)	.7728(1)
C3	−.0710(2)	.6891(1)	.6220(1)
C4	.1223(2)	.7535(1)	.5910(1)
C5	.1820(2)	.7718(1)	.4502(1)
C6	.3480(2)	.8575(1)	.4217(1)
C7	−.0424(2)	.7345(1)	.9034(1)
C8	−.3581(2)	.5779(1)	.8309(1)
C9	.7089(2)	.1571(1)	.8599(1)
C10	.3678(2)	.0221(1)	.7782(1)
H31	−.207	.756	.620
H32	−.049	.613	.562
H41	.257	.695	.624
H42	.079	.846	.640
H51	.040	.817	.415
H52	.251	.677	.405
H61	.463	.831	.471
H62	.273	.955	.439
H71	.092	.756	.887
H72	−.031	.689	.981
H73	−.164	.814	.917
H81	−.383	.519	.780
H82	−.478	.663	.843
H83	−.354	.534	.917
H91	.776	.229	.851
H92	.826	.074	.857
H93	.632	.168	.946
H101	.273	.024	.731
H102	.286	.036	.865
H103	.474	−.064	.783

and *4* included only reflections with $\sin\theta/\lambda > 0.4$ Å^{-1}. The main effect of this was a lengthening of the C-C triple bond by about 0.01 Å. Since the crystals of *3* were of poorer quality and also quite small, we were unable to obtain high angle reflections; the refinement procedure for this compound must thus be performed with all data.

The final figures of merit are given in Table 3. Final atomic coordinates are listed in Table 4. Tables of observed and calculated structure factors and of thermal parameters are available from the authors. Drawings of the molecules are shown in Fig. 2 where the numbering of the atoms is also indicated. In Table 5, bond lengths and angles are presented. Estimated standard deviations were calculated from the variance-covariance matrix.

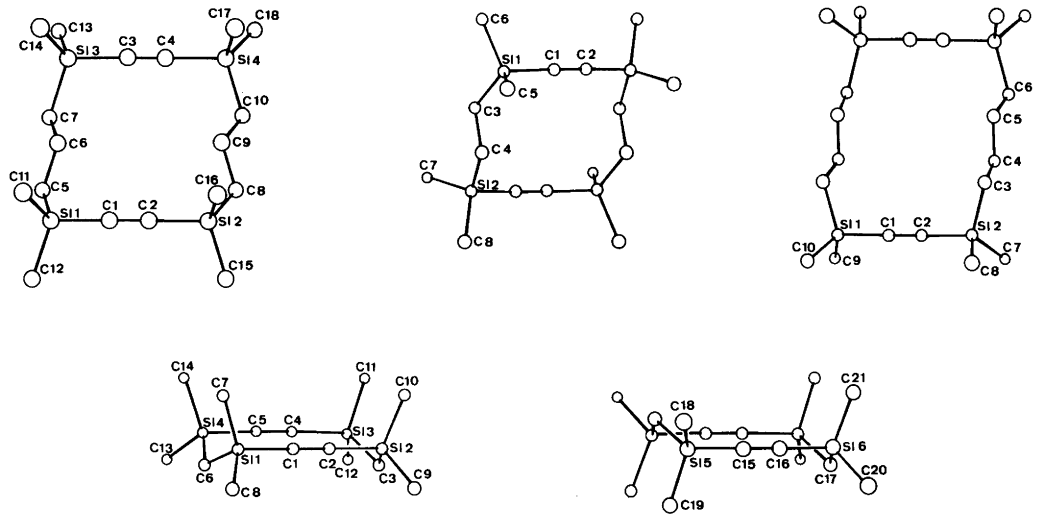

Fig. 2. Drawings of the molecules.

Results and discussion

The crystal structure of *1* exhibits some unusual features. The space group is $P\bar{1}$ and there are *three* molecules in the unit cell. Two of these are in general positions and related by a centre of symmetry; the third molecule is situated about a centre of symmetry. There are thus two crystallographically independent molecules which actually have two different conformations. One has a kind of boat arrangement (noncentrosymmetric) and the other a centrosymmetric chair structure (v. Fig. 2). However, apart from the different torsion angle about the (nearly linear) Si-C≡C-Si linkage, the structural characteristics are the same for the two conformers.

The strain imposed by the close proximity of the two acetylene groups across the rings of *1* and also of the axial methyl groups is relieved by an opening of the Si-C-Si bond angles (122°) and a slight deviation (3°) from linearity of the Si-C≡C arrangements.

Compound *2* crystallizes in the space group $P2_1/c$ with two molecules per unit cell. They are thus centrosymmetric. With an even number of methylene groups separating the silicon atoms, the conformation of the Si-C and CH_2-CH_2 bonds cannot be staggered. A conformational angle of about 60° must be distributed among the three bonds between the silicon atoms resulting in an almost 20° deviation from a staggered conformation at each of the three bonds (v. Table 6).

The crystals of *3* are triclinic, space groups $P\bar{1}$ with two molecules in general positions related by a centre of symmetry. The odd number of methylene groups between the silicon atoms results in a relaxed conformation; the three methylene carbon atoms are nearly coplanar with the two adjacent silicon atoms and their equatorial methyl carbon atoms.

The molecules of *4* are centrosymmetric, the space group being $P\bar{1}$ with one molecule in the unit cell. The number of methylene groups in the chains being even, the almost 60° torsion angle mentioned above for *2* must be distributed among the five bonds separating the silicon atoms. The conformations of the bonds along the methylene chain actually deviate by an average of 14.4° from being staggered (v. Table 6). In all four compounds, the silicon atoms in the molecule are situated in a plane. For *1b*, *2* and *4*, this is demanded by the crystal symmetry. The silicon atoms of each molecule almost form a rectangle, the Si-Si-Si angles all being less than 1° from 90°. The conformation along the nearly linear Si-C≡C-Si arrangement is eclipsed regarding the substituents on the silicon atoms except in *1b* (with chair structure) where the arrangement is staggered. Averaged bond length and angle val-

Table 5. Bond lengths (Å) and angles (°).

1

Distance			Distance		
Si1	–C1	1.841(2)	Si1	–C6	1.872(2)
Si1	–C7	1.867(2)	Si1	–C8	1.871(2)
Si2	–C2	1.843(2)	Si2	–C3	1.865(2)
Si2	–C9	1.866(2)	Si2	–C10	1.866(2)
Si3	–C3	1.877(2)	Si3	–C4	1.842(2)
Si3	–C11	1.868(2)	Si3	–C12	1.868(2)
Si4	–C5	1.840(2)	Si4	–C6	1.872(2)
Si4	–C13	1.867(2)	Si4	–C14	1.862(2)
Si5	–C15	1.841(2)	Si5	–C17′	1.869(2)
Si5	–C18	1.865(2)	Si5	–C19	1.865(2)
Si6	–C16	1.842(2)	Si6	–C17	1.874(2)
Si6	–C20	1.870(2)	Si6	–C21	1.866(2)
C1	–C2	1.216(3)	C4	–C5	1.215(3)
C15	–C16	1.216(3)			

Angle				Angle			
C1	–Si1	–C6	108.6(1)	C1	–Si1	–C7	109.9(1)
C1	–Si1	–C8	107.8(1)	C6	–Si1	–C7	112.0(1)
C6	–Si1	–C8	109.5(1)	C7	–Si1	–C8	109.0(1)
C2	–Si2	–C3	108.9(1)	C2	–Si2	–C9	108.1(1)
C2	–Si2	–C10	108.7(1)	C3	–Si2	–C9	110.4(1)
C3	–Si2	–C10	112.1(1)	C9	–Si2	–C10	108.6(1)
C3	–Si3	–C4	109.5(1)	C3	–Si3	–C11	111.7(1)
C3	–Si3	–C12	110.1(1)	C4	–Si3	–C11	107.7(1)
C4	–Si3	–C12	108.0(1)	C11	–Si3	–C12	109.8(1)
C5	–Si4	–C6	107.7(1)	C5	–Si4	–C13	108.6(1)
C5	–Si4	–C14	109.3(1)	C6	–Si4	–C13	109.6(1)
C6	–Si4	–C14	112.1(1)	C13	–Si4	–C14	109.6(1)
C15	–Si5	–C17	109.8(1)	C15	–Si5	–C18	108.7(1)
C15	–Si5	–C19	108.4(1)	C17	–Si5	–C18	109.2(1)
C17	–Si5	–C19	111.4(1)	C18	–Si5	–C19	109.4(1)
C16	–Si6	–C17	109.0(1)	C16	–Si6	–C20	108.6(1)
C16	–Si6	–C21	108.2(1)	C17	–Si6	–C20	109.8(1)
C17	–Si6	–C21	112.0(1)	C20	–Si6	–C21	109.3(1)
Si1	–C1	–C2	176.6(2)	Si2	–C2	–C1	177.0(2)
Si1	–C3	–Si3	121.8(1)	Si3	–C4	–C5	178.3(2)
Si4	–C5	–C4	175.8(2)	Si1	–C6	–Si14	122.2(1)
Si5	–C15	–C16	177.2(2)	Si6	–C16	–C15	176.6(2)
Si5	–C17	–S16	122.0(1)				

2

Distance				Distance			
Si1	–C1	1.846(1)		Si1	–C3	1.878(1)	
Si1	–C5	1.866(1)		Si1	–C6	1.864(1)	
Si2	–C2′	1.842(1)		Si2	–C4	1.879(1)	
Si2	–C7	1.869(1)		Si2	–C8	1.865(1)	
C1	–C2	1.213(2)		C3	–C4	1.544(2)	
Angle				Angle			
C1	–Si1	–C3	105.7(1)	C1	–Si1	–C5	108.9(1)
C1	–Si1	–C6	108.9(1)	C3	–Si1	–C5	109.8(1)
C3	–Si1	–C6	112.2(1)	C5	–Si1	–C6	111.2(1)
C2	–Si2	–C4	105.8(1)	C2	–Si2	–C7	108.2(1)
C2	–Si2	–C8	109.4(1)	C4	–Si2	–C7	111.2(1)
C4	–Si2	–C8	111.5(1)	C7	–Si2	–C8	110.7(1)
Si1	–C1	–C2	173.8(1)	Si2	–C2	–C1	173.5(1)
Si1	–C3	–C4	116.1(1)	Si2	–C4	–C3	113.3(1)

3

Distance				Distance			
Si1	–C1	1.842(6)		Si1	–C5	1.871(5)	
Si1	–C11	1.862(5)		Si1	–C12	1.866(6)	
Si2	–C2	1.853(6)		Si2	–C8	1.888(5)	
Si2	–C15	1.848(6)		Si2	–C16	1.862(6)	
Si3	–C3	1.850(6)		Si3	–C7	1.877(5)	
Si3	–C13	1.874(6)		Si3	–C14	1.859(6)	
Si4	–C4	1.857(6)		Si4	–C10	1.863(6)	
Si4	–C17	1.856(6)		Si4	–C18	1.868(5)	
C1	–C2	1.219(7)		C3	–C4	1.213(7)	
C5	–C6	1.518(7)		C6	–C7	1.514(8)	
C8	–C9	1.517(7)		C9	–C1	1.540(7)	
Angle				Angle			
C1	–Si1	–C5	107.4(2)	C1	–Si1	–C11	108.4(2)
C1	–Si1	–C12	109.0(2)	C5	–Si1	–C11	110.3(3)
C5	–Si1	–C12	111.4(3)	C11	–Si1	–C12	110.3(3)
C2	–Si2	–C8	108.5(2)	C2	–Si2	–C15	107.8(3)
C2	–Si2	–C16	107.4(3)	C8	–Si2	C15	111.1(3)
C8	–Si2	–C16	110.7(3)	C15	–Si2	–C16	111.2(3)
C3	–Si3	–C7	108.2(2)	C3	–Si3	–C13	107.9(3)
C3	–Si3	–C14	108.4(3)	C7	–Si3	–C13	110.8(3)
C7	–Si3	–C14	109.9(3)	C13	–Si3	–C14	111.4(3)
C4	–Si4	–C10	107.8(2)	C4	–Si4	–C17	107.9(2)
C4	–Si4	–C18	109.3(2)	C10	–Si4	–C17	110.3(2)
C10	–Si4	–C18	111.6(2)	C17	–Si4	–C18	109.8(2)
Si1	–C1	–C2	179.3(5)	Si2	–C2	–C1	179.9(5)
Si3	–C3	–C4	179.1(5)	Si4	–C4	–C3	179.5(5)
Si1	–C5	–C6	114.4(4)	C5	–C6	–C7	115.2(4)
Si3	–C7	–C6	115.5(4)	Si2	–C8	–C9	114.2(3)
C8	–C9	–C10	114.2(4)	Si4	–C10	–C9	114.3(4)

4

Distance				Distance			
Si1	–C1	1.842(1)		Si1	–C6′	1.875(1)	
Si1	–C9	1.866(1)		Si1	–C10	1.872(1)	
Si2	–C2	1.844(1)		Si2	–C3	1.879(1)	
Si2	–C7	1.865(1)		Si2	–C8	1.865(1)	
C1	–C2	1.220(1)		C3	–C4	1.539(1)	
C4	–C5	1.529(1)		C5	–C6	1.539(1)	
Angle				**Angle**			
C1	–Si1	–C6	107.0(0)	C1	–Si1	–C9	108.6(0)
C1	–Si1	–C10	108.2(0)	C6	–Si1	–C9	111.6(0)
C6	–Si1	–C10	110.6(0)	C9	–Si1	–C10	110.7(0)
C2	–Si2	–C3	106.4(0)	C2	–Si2	–C7	109.3(0)
C2	–Si2	–C8	108.3(0)	C3	–Si2	–C7	111.1(0)
C3	–Si2	–C8	111.9(0)	C7	–Si2	–C8	109.7(0)
Si1	–C1	–C2	178.5(1)	Si2	–C2	–C1	177.6(1)
Si2	–C3	–C4	113.8(1)	C3	–C4	–C5	113.8(1)
C4	–C5	–C6	113.3(1)	Si1′	–C6	–C5	114.7(1)

Table 6. Averaged structural data with sample standard deviations in patentheses if more than two measurements.

Bond lengths (Å)	1	2	3	4	Weighted mean
Si-C(H_3)	1.867(2)	1.866(2)	1.862(8)	1.867(3)	1.867
Si-C(H_2)	1.872(4)	1.879	1.877(10)	1.877	1.877
Si-C≡	1.842(1)	1.844	1.851(6)	1.843	1.843
C≡C	1.216(1)	1.213	1.216(4)	1.220	1.217
Bond angles (°)	**1**	**2**	**3**	**4**	
Si-C≡C	176.9(8)	173.7	179.5(3)	178.1	
Si-C-Si	122.0(2)				
Si-C(H_2)-C(H_2)		114.7	114.6(6)	114.3	
-C(H_2)-C(H_2)-C(H_2)			114.7(7)	113.6	
≡C-Si-C(H_2)	109.0(7)	105.8	108.0(5)	106.7	
≡C-Si-C(H_3)	108.7(8)	108.9(5)	103.3(6)	108.6(5)	
C(H_3)-Si-C(H_3)	109.3(4)	111.0	110.7(8)	110.2	
C(H_2)-Si-C(H_3) eq.	109.8(4)	111.9	111.2(4)	111.4	
C(H_2)-Si-C(H_3) ax.	111.9(3)	110.5	110.3(3)	111.4	
Torsion angles (°)	**1**	**2**	**3**	**4**	
C(H_3)(eq)-Si-C(H_2)-Si	178.0(8)				
C(H_3)(eq)-Si-C(H_2)-C(H_2)		160.9	177.3(9)	166.3	
Si-C(H_2)-(CH_2)-Si		157.4			
Si-C(H_2)-C(H_2)-C(H_2)			178.4(9)	162.3	
C(H_2)-C(H_2)-C(H_2)-C(H_2)				170.7	

ues are listed in Table 6. The figures in parentheses are standard deviations of the sample measurements.

It can be seen from Table 6 that there are three significantly different Si-C bond lengths, the weighted mean of Si-C (methyl), Si-C (methylenic) and Si-C (acetylenic) being 1.867 Å, 1.877 Å and 1.843 Å, respectively. The mean C≡C bond length is 1.217 Å. The strain imposed on *2* and *4* is also evident from the bond angles. The deviation from linearity of the Si-C≡C-Si arrangements is larger for *2* than for *1* and also larger for *4* than for *3*. The deviation of the ≡C-Si-CH_2-angle from tetrahedral is also quite large (3–4°) in *2* and *4*. The packing of the molecules in the crystals is governed by normal van der Waals' interactions between hydrogen atoms of neighbouring molecules.

References

1. Kloster-Jensen, E. and Eliassen, G. Å. *Angew. Chem. 97* (1985) 587; *Angew. Chem. Int. Ed. Engl. 24* (1985) 565.
2. Sakùrai, H., Nakadaira, Y., Hosomi, A. and Eriyama, Y. *J. Am. Chem. Soc. 105* (1983) 3359.
3. a. Rømming, C. *Unpublished details*; b. Kloster-Jensen, E. and Wirz, J. *Angew. Chem. 85* (1973) 723; *Angew. Chem. Int. Ed. Engl. 12* (1973) 671; c. *Helv. Chim. Acta 58* (1975) 162.
4. Groth, P. *Acta Chem. Scand. 27* (1973) 1837.
5. Doyle, P. A. and Turner, P. S. *Acta Crystallogr. A 24* (1968) 390.
6. Stewart, R. F., Davidson, E. R. and Simpson, W. T. *J. Chem. Phys. 42* (1965) 3175.
7. Gilmore, C. J. *J. Appl. Crystallogr. 17* (1984) 42.

Received February 14, 1986.