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Hydrogen-bonding in organosilanetriols. The crystal structures of tris(trimethylsilyl)silyl- and tris(trimethylsilyl)-methyl-silanetriols

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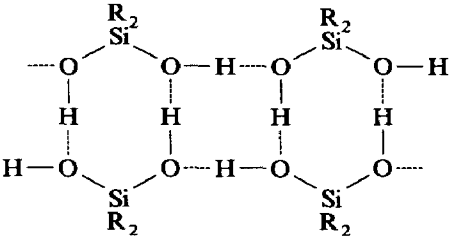
Abstract

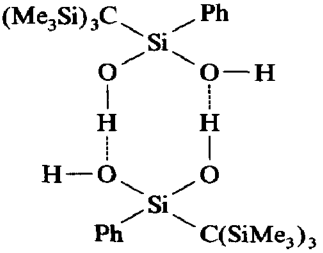
The organosilanetriols $(Me_3Si)_3CSi(OH)_3$ and $(Me_3Si)_3SiSi(OH)_3$ both crystallize as hexameric hydrogen-bonded cages with no hydrogen bonding between the cages. Both $(Me_3Si)_3CSi(OH)_3$ and $(Me_3Si)_3SiSi(OH)_3$ are remarkably thermally stable as solids, melting with decomposition only at 285–290 and 210–213°C, respectively.

Introduction

Silanediols form a variety of hydrogen-bonded structures such as ladder chains of hydrogen-bonded dimers of type I (e.g. $i\text{-}Pr_2Si(OH)_2$ [1] and $(C_6H_{11})_2Si(OH)_2$ [2]), cross-linked hydrogen-bonded chains forming a layer, e.g. $Et_2Si(OH)_2$ [3], and cyclic hydrogen-bonded hexamers linked together by further hydrogen bonding as in $Ph_2Si(OH)_2$ [4]. The very bulky diol $(Me_3Si)_3CSiPh(OH)_2$ has a structure consisting of discrete dimers as shown in II [5], and by extension we thought it possible that a bulky silanetriol might crystallize as discrete polyhedral cages, even though cyclohexylsilanetriol, studied at −60°C, was known to form infinite hydrogen-bonded sheets [6]. We show below that both $(Me_3Si)_3CSi(OH)_3$ (for a preliminary report see ref. 7) and the closely related triol $(Me_3Si)_3SiSi(OH)_3$ do, indeed, crystallize as polyhedral cages.



(I)



(II)

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Experimental

$(Me_3Si)_3CSi(OH)_3$

The triol was prepared in several steps from $(Me_3Si)_3CSiH_2I$ as described in Ref. 8, and was crystallized from heptane. A crystal of ca. 0.45 × 0.3 × 0.4 mm was used for the structure determination.

$(Me_3Si)_3SiSi(OH)_3$

A solution of $(Me_3Si)_3SiSiCl_3$ (prepared from $(Me_3Si)_3SiLi$ and $SiCl_4$ [9]) (1 g, 2.62 mmol) in anhydrous diethyl ether (20 ml) was added dropwise under nitrogen to a cooled (0 °C) stirred solution of water (0.14 g, 7.86 mmol) and aniline (0.73 g, 7.86 mmol) in dry diethyl ether (15 ml). After the addition was complete the mixture was stirred at 0 °C for a further 45 min. The solid was then filtered off and the solvent was removed under reduced pressure to leave a white solid, which was sublimed (155 °C, 0.01 mmHg) to give $(Me_3Si)_3SiSi(OH)_3$, (0.54 g, 63%), m.p. 210–213 °C (decomp.); (Found: C, 32.8; H, 9.2. $C_9H_{30}O_3Si_5$ calc: C, 33.1; H, 9.2%); ν(OH) 3402 cm^{-1}, ^1H NMR (CCl_4, 5% CH_2Cl_2): δ 0.22 (s, Me_3Si), signal due to OH not observed; ^{29}Si NMR (3/1 v/v $CDCl_3/CCl_4$): δ -10.27 (s, $SiMe_3$), -25.84 (s, $Si(OH)_3$), -139.81 (s, $\mathit{Si}(SiMe_3)$); mass spectrum (EI); m/e 308 (15% $[M-H_2O]^+$), 218(10), 131(10), 73(100 $[Me_3Si]^+$), 59(15 $[Me_2HSi]^+$); (+ve CI, ammonia); 344(40% $[M+NH_4]^+$), 94(25), 90(100 $[Me_3SiOH]^+$), 73(15 $[Me_3Si]^+$). Crystals suitable for the X-ray diffraction study were obtained by slow evaporation of a solution in CCl_4/diethyl ether. A crystal of ca. 0.4 × 0.4 × 0.4 mm was used.

Table 1

Fractional atomic coordinates ($\times 10^4$) for $(Me_3Si)_3CSi(OH)_3$ with estimated standard deviations in parentheses

Atom	*x*	*y*	*z*
Si(1)	1897(2)	−21(3)	5539(1)
Si(2)	3977(4)	1290(4)	5797(2)
Si(3)	3438(3)	−644(3)	5476(1)
Si(4)	2480(4)	−767(4)	6146(1)
O(1)	2223(8)	986(8)	5324(3)
O(2)	1134(8)	−1136(8)	5371(3)
O(3)	1122(7)	100(8)	5793(4)
C(1)	2941(6)	−25(6)	5772(2)
C(2)	3495(12)	2184(10)	5988(7)
C(3)	4972(10)	1437(12)	6152(4)
C(4)	4781(11)	1949(12)	5449(4)
C(5)	3477(15)	−272(18)	5064(4)
C(6)	2606(13)	−2074(11)	5512(7)
C(7)	4791(10)	−354(13)	5635(9)
C(8)	1203(11)	−1889(13)	6122(4)
C(9)	3418(14)	−1209(16)	6273(5)
C(10)	2486(20)	37(22)	6489(4)
Si(2a)	4099(5)	1393(5)	5853(2)
Si(3a)	3327(8)	−1022(6)	5681(3)
Si(4a)	2327(7)	−290(8)	6196(2)
O(1a)	2343(22)	131(20)	5138(5)
O(2a)	906(23)	−911(19)	5483(10)
O(3a)	1748(21)	774(21)	5577(15)

Crystal data

Data were measured on an Enraf–Nonius CAD4 diffractometer. The structure solution and refinement in both cases were carried out on a PDP 11/34 computer with the Enraf–Nonius structure determination package. Scattering factors for neutral atoms were taken from ref. 10.

$(Me_3Si)_3CSi(OH)_3$. $C_{10}H_{30}O_3Si_4$, *M* 310.7, rhombohedral (on hexagonal axes), *a* 15.154(1), *c* 42.079(2) Å, *U* 8368.1 Å^3, *Z* 18, D_c 1.12 g cm^{-3}, *F*(000) 1020. Cu-K_α radiation (Ni filter), λ 1.5418 Å, μ 30.0 cm^{-1}. Space group $R\bar{3}$ from successful structure refinement.

Intensities for $+h$ $-k$ $+l$ reflections with $2 < \theta < 55°$ were measured by a $\theta/2\theta$ scan with a scan width of $\Delta\theta = (0.8 + 0.15 \tan \theta)°$ and a maximum scan time of 1 min. Two standard reflections monitored every hour showed no significant variation. Data were corrected for Lorentz and polarisation (*Lp*) effects and, after averaging of any equivalent reflections, 1594 with $|F^2| > \sigma(F^2)$ were used in the structure refinement. The values of $\sigma(F^2)$ were taken as $[\sigma^2(I) + (0.02I)^2]^{1/2}/Lp$.

Table 2

Intramolecular distances (Å) and angles (°) in $(Me_3Si)_3CSi(OH)_3$ with estimated standard deviations in parentheses [a]

Si(1)–O(1)	1.623(12)	Si(1)–O(2)	1.655(11)
Si(1)–O(3)	1.666(14)	Si(1)–C(1)	1.865(11)
Si(2)–C(1)	1.822(8)	Si(2)–C(2)	2.00(2)
Si(2)–C(3)	2.06(2)	Si(2)–C(4)	1.84(2)
Si(3)–C(1)	1.926(12)	Si(3)–C(5)	1.81(2)
SI(3)–C(6)	1.892(14)	Si(3)–C(7)	1.99(2)
Si(4)–C(1)	1.854(10)	Si(4)–C(8)	1.833(14)
Si(4)–C(9)	1.92(3)	Si(4)–C(10)	1.89(3)
Si(1)–O(1a)	1.79(2)	Si(1)–O(2a)	1.45(2)
Si(1)–O(3a)	1.34(4)	C(1)–Si(2a)	2.010(9)
C(1)–Si(3a)	1.91(2)	C(1)–Si(4a)	1.956(13)
O(1)...O(2)′	3.12	O(1)...O(2)″	2.93
O(3)...O(3)′	2.82		
O(1)–Si(1)–O(2)	116.8(6)	O(1)–Si(1)–O(3)	98.8(7)
O(1)–Si(1)–C(1)	115.9(5)	O(2)–Si(1)–O(3)	102.5(5)
O(2)–Si(1)–C(1)	112.5(6)	O(3)–Si(1)–C(1)	107.9(6)
C(1)–Si(2)–C(2)	110.9(6)	C(1)–Si(2)–C(3)	110.1(6)
C(1)–Si(2)–C(4)	121.5(7)	C(2)–Si(2)–C(3)	97(1)
C(2)–Si(2)–C(4)	109(1)	C(3)–Si(2)–C(4)	105.5(7)
C(1)–Si(3)–C(5)	115(1)	C(1)–Si(3)–C(6)	107.8(9)
C(1)–Si(3)–C(7)	107(1)	C(5)–Si(3)–C(6)	109(1)
C(5)–Si(3)–C(7)	113(1)	C(6)–Si(3)–C(7)	104.4(9)
C(1)–Si(4)–C(8)	114.7(7)	C(1)–Si(4)–C(9)	109.1(7)
C(1)–Si(4)–C(10)	112.2(9)	C(8)–Si(4)–C(9)	108.3(9)
C(8)–Si(4)–C(10)	108(1)	C(9)–Si(4)–C(10)	104(1)
Si(1)–C(1)–Si(2)	107.1(6)	Si(1)–C(1)–Si(3)	101.5(4)
Si(1)–C(1)–Si(4)	112.7(5)	Si(2)–C(1)–Si(3)	103.6(5)
Si(2)–C(1)–Si(4)	118.2(5)	Si(3)–C(1)–Si(4)	112.(6)
O(1a)–Si(1)–O(2a)	97(2)	O(1a)–Si(1)–O(3a)	103(3)
O(2a)–SI(1)–O(3a)	107(2)	Si(2a)–C(1)–Si(3a)	115.4(6)
Si(2a)–C(1)–Si(4a)	97.9(5)	Si(3a)–C(1)–Si(4a)	108.8(7)

[a] Symmetry elements: ′ $\bar{y}$, $x - y$, z; ″ $x - y$, x, $1 - z$.

The structure was solved by direct methods (MULTAN) [11], and refinement was by full matrix least squares. A difference map showed alternative sites for the O and Si atoms which were included in the model and their occupancies refined. Finally the O atom sites were given fixed occupancies of 80% and 20% and the Si atom sites were given fixed occupancies of 66% and 33% for major and minor orientations respectively. Hydrogen atoms were omitted. Refinement with all atoms anisotropic converged at $R = 0.13$, $R' = 0.16$, the weighting scheme was $w = 1/\sigma^2(F)$. A final difference map had peaks of up to 0.5 eÅ^{-3}.

$(Me_3Si)_3SiSi(OH)_3$. $C_9H_{30}O_3Si_5$. 1/6 CCl_4, *M* 352.4, rhombohedral, $a = b = c = 15.880(16)$ Å, $\alpha = \beta = \gamma = 102.35(10)°$, *U* 3677.2 Å^3, *Z* 6, D_c 0.96 g cm^{-3}, *F*(000) 1142. Monochromated Mo-K_α radiation, λ 0.71069 Å, μ 3.57 cm^{-1}. Space group $R\bar{3}$ from successful structure refinement. The setting used for data collection was based on hexagonal axes ($a = b = 24.691(22)$, $c = 20.822(22)$ Å). Intensities for $+h$ $-k$ $+l$ reflections with $2 < \theta < 22°$ were measured by a $\theta/2\theta$ scan with a scan width $\Delta\theta(0.8 + 0.35 \tan \theta)°$ and a maximum scan time of 1 min. Every 30 min two standard reflections were monitored and showed no significant variation. After correction for *Lp* effects but not for absorption, 1173 reflections with $|F|^2 > \sigma(F^2)$ were used for the structure analysis. The values of $\sigma(F^2)$ were calculated as $[\sigma^2(I) + (0.04I)^2]^{1/2}/Lp$. For the structure determination and refinement the data were converted to the setting based on rhombohedral axes.

The C, Si, and O atoms of the silanetriol were found by direct methods (MULTAN) [11]. Further electron density near the $\bar{3}$ inversion centre was interpreted as a disordered molecule of CCl_4. All the non-hydrogen atoms were refined by full matrix least squares with anisotropic temperature factors.

Table 3

Fractional atomic coordinates ($\times 10^4$) for $(Me_3Si)_3SiSi(OH)_3$ with estimated standard deviations in parentheses

Atom	*x*	*y*	*z*
Si(1)	2814(5)	−799(5)	2419(5)
Si(2)	1849(5)	−173(5)	1612(5)
Si(3)	2302(6)	−2348(6)	1975(7)
Si(4)	2918(7)	−229(7)	3930(6)
Si(5)	4196(6)	−310(7)	2070(7)
O(1)	2176(12)	923(10)	2042(12)
O(2)	791(10)	−514(10)	1685(9)
O(3)	1836(10)	−332(9)	543(9)
C(1)	3238(27)	−2859(20)	2189(29)
C(2)	1446(25)	−2749(23)	2520(24)
C(3)	1691(24)	−2792(19)	750(18)
C(4)	1827(22)	−118(32)	4141(21)
C(5)	3751(31)	900(24)	4335(22)
C(6)	3433(25)	−976(23)	4615(17)
C(7)	4504(23)	949(24)	2184(31)
C(8)	5124(18)	−579(26)	2844(22)
C(9)	4038(20)	−945(37)	861(20)
Cl(1)	3417(14)	3417	3417
Cl(2)	4640(11)	606(10)	4386(10)
C(10)	4592(18)	4592	4592

[a] Occupancies for C(10) and Cl(1) are 1/6 and for Cl(2) is 1/2.

Table 4

Intramolecular distances (Å) and angles (°) in $(Me_3Si)_3SiSi(OH)_3$ with estimated standard deviations in parentheses [a]

Si(1)–Si(2)	2.309(12)	Si(1)–Si(3)	2.316(11)
Si(1)–Si(4)	2.332(12)	Si(1)–Si(5)	2.387(13)
Si(2)–O(1)	1.65(2)	Si(2)–O(2)	1.69(2)
Si(2)–O(3)	1.66(2)	Si(3)–C(1)	1.85(4)
Si(3)–C(2)	1.84(4)	Si(3)–C(3)	1.88(3)
Si(4)–C(4)	1.87(4)	Si(4)–C(5)	1.87(4)
Si(4)–C(6)	1.96(4)	Si(5)–C(7)	1.91(4)
Si(5)–C(8)	1.89(4)	Si(5)–C(9)	1.90(3)
C(10)–Cl(1)	2.44(2)	C(10)–Cl(2)	2.41(2)
C(10)–Cl(2)	2.37(2)	O(1)...O(1) ^{a}	2.95(3)
O(2)...O(3) ^{e}	2.78(2)	O(2)...O(3) ^{a}	2.71(2)
Si(2)–Si(1)–Si(3)	110.8(4)	Si(2)–Si(1)–Si(4)	107.2(5)
Si(2)–Si(1)–Si(5)	104.1(5)	Si(3)–Si(1)–Si(4)	111.4(5)
Si(3)–Si(1)–Si(5)	110.7(5)	Si(4)–Si(1)–Si(5)	112.3(4)
Si(1)–Si(2)–O(1)	108.7(7)	Si(1)–Si(2)–O(2)	112.2(7)
Si(1)–Si(2)–O(3)	114.8(8)	O(1)–Si(2)–O(2)	106(1)
O(1)–Si(2)–O(3)	105(1)	O(2)–Si(2)–O(3)	109.1(8)
Si(1)–Si(3)–C(1)	111(1)	Si(1)–Si(3)–C(2)	111(1)
Si(1)–Si(3)–C(3)	112(1)	C(1)–Si(3)–C(2)	111(2)
C(1)–Si(3)–C(3)	108(2)	C(2)–Si(3)–C(3)	103(2)
Si(1)–Si(4)–C(4)	113(1)	Si(1)–Si(4)–C(5)	108(1)
Si(1)–Si(4)–C(6)	109(1)	C(4)–Si(4)–C(5)	109(2)
C(4)–Si(4)–C(6)	111(2)	C(5)–Si(4)–C(6)	106(2)
Si(1)–Si(5)–C(7)	112(1)	Si(1)–Si(5)–C(8)	110(1)
Si(1)–Si(5)–C(9)	107(1)	C(7)–Si(5)–C(8)	109(2)
C(7)–Si(5)–C(9)	109(2)	C(8)–Si(5)–C(9)	110(2)
Cl(1)–C(10)–Cl(2)	112.2(7)	Cl(1)–C(10)–Cl(2)	109.5(4)
Cl(2)–C(10)–Cl(2)	106.7(8)	Cl(2)–C(10)–Cl(2)	109(1)

[a] Symmetry element a is *y*, *z*, *x*; e is $-z$, *x*, $-y$.

The solvent CCl_4 molecule is disordered about the 3 inversion centre at $\frac{1}{2}$, $\frac{1}{2}$, $\frac{1}{2}$, with alternative sites for C(10) and Cl(1) lying on the three-fold rotation axis. This interpretation results in rather long C–Cl bond lengths and a negative temperature factor for C(10) but seems to us to account reasonably for the electron density. Hydrogen atoms were omitted. Refinement converged at $R = 0.147$, $R' = 0.166$, with a weighting scheme $w = 1/\sigma^2(F)$. A final difference map had peaks of up to 2.4 $eÅ^{-3}$ in the region of the CCl_4 molecule.

Final atom coordinates, and bond lengths and angles, together with details of the hydrogen-bonding are given in Tables 1 and 2 for $(Me_3Si)_3CSi(OH)_3$ and in Tables 3 and 4 for $(Me_3Si)_3SiSi(OH)_3$. Lists of temperature factors and structure factors are available from the authors.

Results and discussion

The molecular structures of $(Me_3Si)_3CSi(OH)_3$ and $(Me_3Si)_3SiSi(OH)_3$ and the hydrogen-bonded cages that they form are very similar, as shown in Figs. 1 and 2. Figure 1 shows the hydrogen-bonded hexamer structure and atom numbering

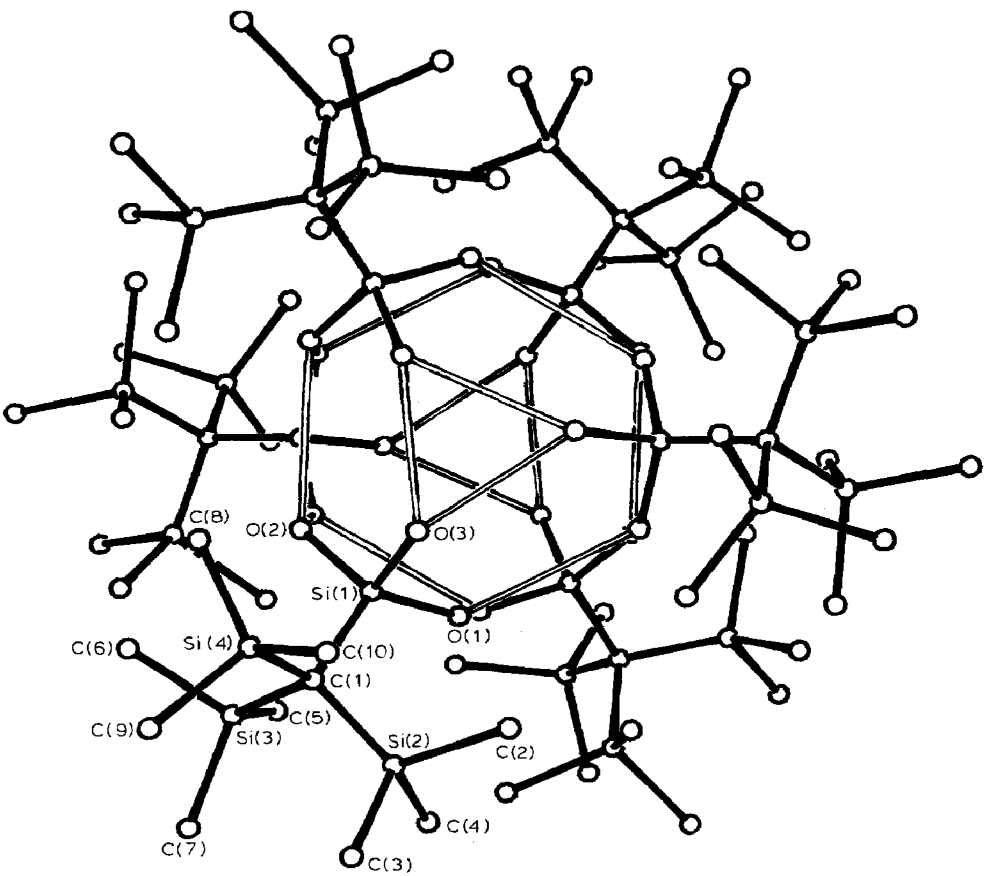

Fig. 1. The hydrogen-bonded hexamer formed from $(Me_3Si)_3CSi(OH)_3$ together with the atom numbering scheme. The open lines indicate hydrogen bonds.

scheme for $(Me_3Si)_3SiSi(OH)_3$, Fig. 2 the structure of the hydrogen-bonded hexamer and atom numbering scheme for $(Me_3Si)_3CSi(OH)_3$, and Fig. 3 the detail of the hydrogen-bonded cage in $(Me_3Si)_3SiSi(OH)_3$. (The corresponding Figure for $(Me_3Si)_3CSi(OH)_3$ can be found in ref. 7). In discussing the structure of $(Me_3Si)_3CSi(OH)_3$ only the dominant one of the two sets of alternative sites is considered.

The structure of both triols consists of discrete cages of $\bar{3}$ symmetry formed from six maximally hydrogen-bonded triol molecules (shown for $(Me_3Si)_3SiSi(OH)_3$ in Fig. 3). In both cases the cage consists of two equilateral triangles each surrounded by three 6-membered rings in a boat conformation (giving 18 faces), and six 5-membered rings in an envelope conformation (giving 12 faces), making up a total of 32 faces. The cavities in the cages are sizeable, that in $(Me_3Si)_3SiSi(OH)_3$ being slightly longer and narrower than that in $(Me_3Si)_3CSi(OH)_3$. The distance between the triangular faces is 6.7 Å in $(Me_3Si)_3CSi(OH)_3$ and 7.2 Å in $(Me_3Si)_3SiSi(OH)_3$, while the distances across the cage in $(Me_3Si)_3SiSi(OH)_3$ are 4.7 Å between $O(2)^d$ and $O(2)^c$ and 5.4 Å between $O(2)^d$ and $O(3)^c$, the corresponding distances in $(Me_3Si)_3CSi(OH)_3$ being 6.1 and 5.9 Å respectively. The O–Si–O angles in $(Me_3Si)_3SiSi(OH)_3$ are 109.1(8), 105(1), and 106(1)° for O(2)–Si(2)–O(3),

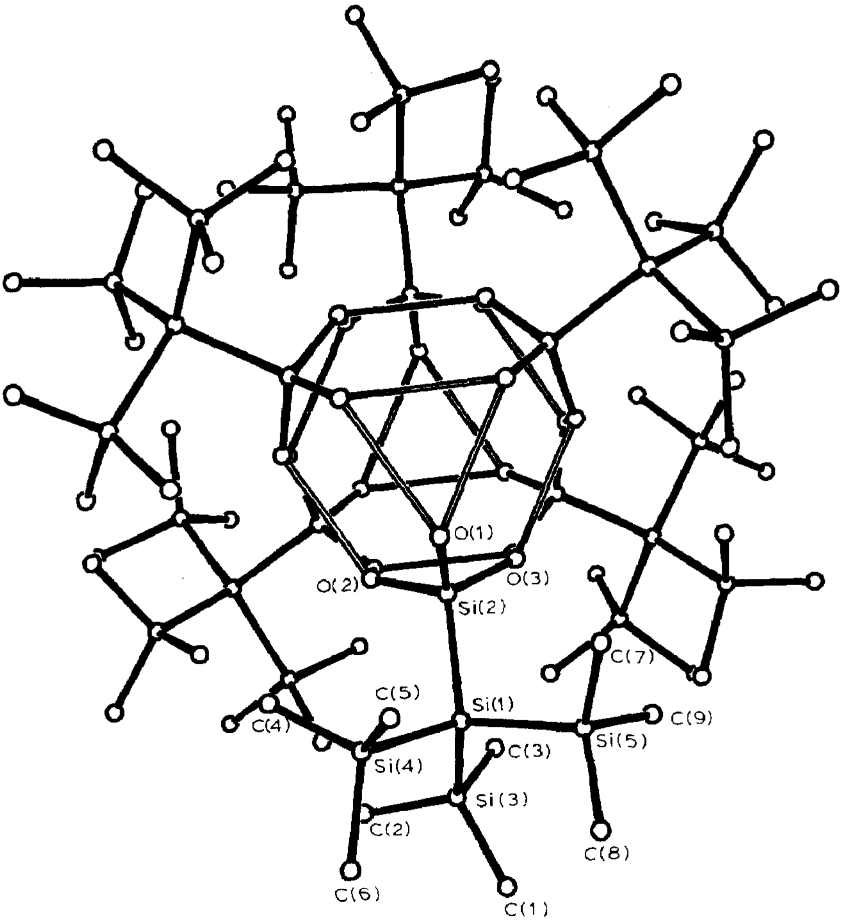

Fig. 2. The hydrogen-bonded hexamer formed from (Me Si)$_3$SiSi(OH)$_3$ together with the atom numbering scheme.

O(1)–Si(2)–O(3), and O(1)–Si(2)–O(2), respectively, i.e. not far from the tetrahedral, but significant distortion of these angles from tetrahedral is necessary to close the cage in (Me Si)$_3$CSi(OH)$_3$, the corresponding angles being 116.8(6), 98.8(7), and 102.5(5)° respectively. This difference is presumably associated with the fact that the Si–Si bonds in (Me$_3$Si)$_3$SiSi(OH)$_3$ are longer than the central C–Si bonds in (Me$_3$Si)$_3$CSi(OH)$_3$. This distortion in the O–Si–O angles in (Me$_3$Si)$_3$CSi(OH)$_3$ is reflected in the O–Si–C angles, which range from 107.9(6) (O(3)–Si(1)–C(1)), to 112.5(6) (O(2)–Si(1)–C(1)), to 115.9(5)° O(1)–Si(1)–C(1)).

A feature of the geometry of the (Me$_3$Si)$_3$C–Si(1) system is the closing of the Me–Si–Me angles from tetrahedral to a mean of 107(4)° (and the correspondingly high mean value of the C(1)–Si–Me angles of 112.0(42)°), similar to that for other tris(trimethylsilyl)methylsilicon compounds [5]. There are, however, marked variations (seemingly real) in the values of the separate Me–Si–Me angles, which range from 97(1) to 113(1)°, and in the C(1)–Si–Me angles, which range from 107.0(1) to a remarkable 121.5(7)°. However, in contrast with the usual pattern, the Si–Me bonds (mean length 1.92(9) Å) appear to be longer than the C–SiMe$_3$ bonds (mean

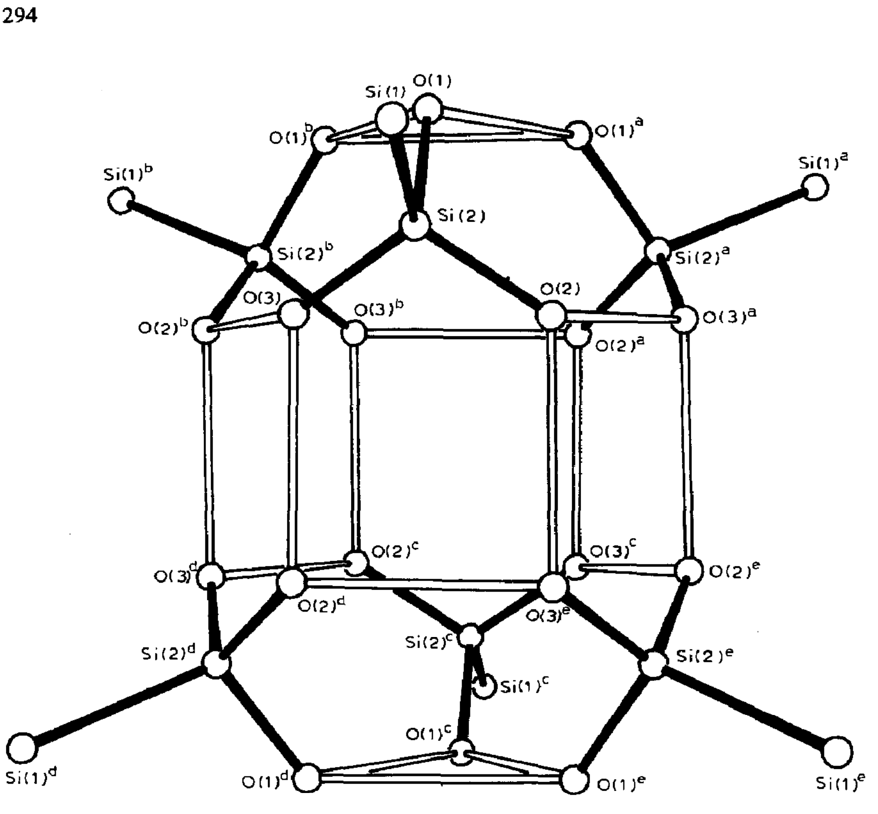

Fig. 3. Detail of the hydrogen-bonded cage formed by $(Me_3Si)_3SiSi(OH)_3$. Symmetry elements: $^{a}\,y, z, x$; $^{b}\,z, x, y$; $^{c}\,\bar{x}, \bar{y}, \bar{z}$; $^{d}\,\bar{y}, \bar{z}, \bar{x}$; $^{e}\,\bar{z}, \bar{x}, \bar{y}$.

length 1.86(6) Å), but this may simply reflect the uncertainty in the data as indicated by the large standard deviations.

The cage structure is very different from that found in the only other silanetriol, $C_6H_{11}Si(OH)_3$, for which the crystal structure is known. In that case the molecules are arranged in a head-to-head and tail-to-tail fashion, with the cyclohexyl groups forming a hydrophobic double sheet and the silanetriol groups forming a hydrophilic double sheet [6].

The carbon-centered silanetriol $(Me_3Si)_3CSi(OH)_3$ is remarkably thermally stable, melting (with decomposition) only at 285–290 ° C. The silicon-centered analogue, $(Me_3Si)_3SiSi(OH)_3$, is also fairly stable, and melts with decomposition at ca. 210–213 ° C. The decomposition products from $(Me_3Si)_3SiSi(OH)_3$ were predominantly water, $(Me_3Si)_3SiH$, and $(Me_3Si)_3SiOH$ (or an isomer) as judged by linked GLC-mass spectrometry. When a sample of $(Me_3Si)_3CSi(OH)_3$ was kept at 315 ° C in a sealed tube for 1 h [8] the decomposition products appeared to be $(Me_3Si)_3CH$ and $(Me_3Si)_2CHSi(OH)_2(OSiMe_3)$ (or an isomer) together with an unidentified material and water.

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