The structure of hexa(cyclohexylsesquisiloxane), $(C_6H_{11})_6Si_6O_9$

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Abstract

The structure of hexa(cyclohexylsesquisiloxane), $Cy_6Si_6O_9$, has been determined and shown to be based on a cage arrangement in which two non-planar six-membered Si_3O_3 rings are joined co-facially by Si–O–Si bridges, so generating three further Si_4O_4 rings. The structural variations in six- and eight-membered $(Si–O)_n$ rings in this and other known cage sesquisiloxanes are analysed and compared with those of analogous rings in two-dimensional diorganosiloxane heterocycles.

Key words: Silicon; Siloxane; Sesquisiloxane; Structure; X-ray diffraction; Cage compound

1. Introduction

Spherical organosesquisiloxanes, $(RSiO_{1.5})_n$, are an important class of compound. They are organometallic analogues of silicate anions $[Si_nO_{2.5n}]^{n-}$ which, apart from their intrinsic mineralogical importance, can be used after esterification [1] as pre-ceramic building blocks for the controlled formation of tailored, porous silicate lattices by sol-gel techniques [2]. Moreover, the organic groups on the periphery of the organosesquisiloxane cage have been used to link the polyhedra into three-dimensional network hybrids of inorganic and organic matrices, known generically as organolithic macromolecular materials [3]. Sesquisiloxanes in which one of the silicon vertices has been replaced by a different metal fragment have also attracted considerable attention in recent years as soluble models for heterogeneous silica-supported metal catalysts [4]. In addition, the hydridosesquisiloxanes (R = H) in general are of interest in their own right as potential photoresists [5–7], and a detailed study of the electronic structure of several symmetrical octasesquisiloxanes, including $H_8Si_8O_{12}$, has revealed important information on the mechanism of substitution reactions at silicon [8].

The sesquisiloxanes have been known since 1946, when they were first prepared by hydrolysis of organotrichlorosilanes [9,10]. The general insolubility of this class of compound has, however, held back extensive structural analysis. Indeed, of the eight organosesquisiloxane structures defined crystallographically, six [R = Me, Et, Ph, PhCH₂, vinyl, allyl) are analogous octameric species [11–13]. This list is supplemented by two further non-organometallic octamers, with R = H [14] and OMe [2]. Higher oligomers are confined to the decamer and dodecamer, $Me_{10}Si_{10}O_{15}$ [15] and $Ph_{12}Si_{12}O_{18}$ [16], and the inorganic species $(Me_3SiO)_{12}Si_{12}O_{18}$ and $(Me_3SiO)_{14}Si_{14}O_{21}$ (two isomers) [17]. As, to the best of our knowledge, no structures have been reported for smaller, potentially strained, organosilicon oligomers ($n < 8$), we have carried out a structural analysis of $Cy_6Si_6O_9$. Our structure can be used to make a direct comparison with structures of the known silicates $[Me_4N^+]_6[Si_6O_{15}^{6-}]\cdot36.5H_2O$ [18], $[Ni(en)_3^{2+}]_3[Si_6O_{15}^{6-}]\cdot26H_2O$ [19] and $(Me_3SiO)_6Si_6O_9$ [20].

2. Experimental details

2.1. Synthesis

Water (125 cm³) was added to a stirred solution of (a commercial sample of) cyclohexyltrichlorosilane (25 g, 0.11 mol) in acetone (500 cm³). The mixture was kept in a stoppered container for 4 months, after which the white crystalline solid was filtered off rinsed with

TABLE 1. Fractional atomic coordinates and thermal parameters (Å) for $(C_6H_{11})_6Si_6O_9$ (e.s.d.s. in parentheses)

Atom	x	y	z	U_{iso} or U_{eq} (*)
Si(1)	0.5251(3)	0.2500	0.3230(1)	0.038(2)*
Si(2)	0.6201(2)	0.1703(1)	0.2174(1)	0.040(1)*
Si(3)	0.2629(3)	0.2500	0.2559(1)	0.037(2)*
Si(4)	0.3615(2)	0.1705(1)	0.1508(1)	0.037(1)*
Si(5)	0.0153(3)	−0.2500	0.1938(1)	0.036(2)*
Si(6)	0.1098(2)	−0.1707(1)	0.3060(1)	0.037(1)*
Si(7)	−0.2474(3)	−0.2500	0.2435(1)	0.036(2)*
Si(8)	−0.1491(2)	−0.1708(1)	0.3552(1)	0.038(1)*
O(1)	0.5835(5)	0.1797(3)	0.2893(2)	0.041(3)*
O(2)	0.6580(7)	0.2500	0.1929(4)	0.043(5)*
O(3)	0.2773(5)	0.1799(3)	0.2105(2)	0.039(3)*
O(4)	0.3720(7)	0.2500	0.1197(3)	0.041(5)*
O(5)	0.4994(5)	0.1429(3)	0.1744(2)	0.043(3)*
O(6)	0.3746(7)	0.2500	0.3096(3)	0.039(5)*
O(7)	0.0728(5)	−0.1797(3)	0.2317(2)	0.037(3)*
O(8)	0.1507(7)	−0.2500	0.3331(4)	0.041(5)*
O(9)	−0.2321(5)	−0.1799(3)	0.2892(2)	0.041(3)*
O(10)	−0.1398(7)	−0.2500	0.3879(4)	0.044(5)*
O(11)	−0.0109(5)	−0.1436(3)	0.3410(3)	0.043(3)*
O(12)	−0.1352(6)	−0.2500	0.1975(3)	0.038(4)*
C(1)	0.5610(10)	0.2500	0.4081(6)	0.054(3)
C(2)	0.5112(10)	0.1828(6)	0.4394(5)	0.076(3)
C(3)	0.5443(12)	0.1841(8)	0.5111(6)	0.099(4)
C(4)	0.4977(12)	0.2500	0.5408(7)	0.102(6)
C(5)	0.7530(8)	0.1080(5)	0.2137(4)	0.054(2)
C(6)	0.8502(11)	0.1337(7)	0.1715(6)	0.090(4)
C(7)	0.9637(12)	0.0838(7)	0.1719(6)	0.093(4)
C(8)	0.9293(10)	0.0084(6)	0.1571(5)	0.071(3)
C(9)	0.8314(11)	−0.0172(7)	0.1954(6)	0.094(4)
C(10)	0.7154(10)	0.0307(6)	0.1949(5)	0.076(3)
C(11)	0.1162(10)	0.2500	0.2934(5)	0.039(3)
C(12)	0.1003(8)	0.1829(5)	0.3347(4)	0.058(2)
C(13)	−0.0202(10)	0.1827(6)	0.3691(5)	0.077(3)
C(14)	−0.0285(11)	0.2500	0.4068(7)	0.086(5)
C(15)	0.2915(7)	0.1074(4)	0.0927(4)	0.041(2)
C(16)	0.3613(9)	0.1090(5)	0.0320(4)	0.058(2)
C(17)	0.3048(10)	0.0548(6)	−0.0155(5)	0.068(3)
C(18)	0.3010(9)	−0.0193(6)	0.0106(5)	0.066(3)
C(19)	0.2315(9)	−0.0216(5)	0.0706(4)	0.058(2)
C(20)	0.2881(8)	0.0304(5)	0.1181(4)	0.048(2)
C(21)	0.0521(11)	−0.2500	0.1110(5)	0.039(3)
C(22)	0.0043(8)	−0.1828(5)	0.0770(4)	0.055(2)
C(23)	0.0375(10)	−0.1825(6)	0.0076(5)	0.069(3)
C(24)	−0.0094(15)	−0.2500	−0.0268(7)	0.071(4)
C(25)	0.2393(7)	−0.1076(4)	0.3228(4)	0.041(2)
C(26)	0.2106(9)	−0.0311(6)	0.3034(5)	0.064(3)
C(27)	0.3217(10)	0.0178(6)	0.3194(5)	0.076(3)
C(28)	0.4381(8)	−0.0075(5)	0.2924(4)	0.055(2)
C(29)	0.4671(9)	−0.0826(5)	0.3110(5)	0.059(3)
C(30)	0.3584(9)	−0.1329(6)	0.2942(5)	0.066(3)
C(31)	−0.3962(11)	−0.2500	0.1961(5)	0.041(3)
C(32)	−0.4129(9)	−0.1826(5)	0.1560(4)	0.059(3)
C(33)	−0.5379(10)	−0.1837(6)	0.1159(5)	0.077(3)
C(34)	−0.5505(18)	−0.2500	0.0765(8)	0.089(5)
C(35)	−0.2164(8)	−0.1061(5)	0.4078(4)	0.043(2)
C(36)	−0.2149(9)	−0.0300(5)	0.3802(5)	0.062(3)
C(37)	−0.2702(10)	0.0246(7)	0.4238(5)	0.077(3)
C(38)	−0.2064(12)	0.0204(7)	0.4890(6)	0.093(4)
C(39)	−0.2090(12)	−0.0521(7)	0.5157(6)	0.091(4)
C(40)	−0.1525(10)	−0.1081(6)	0.4730(5)	0.074(3)

acetone, and dried. Extraction of this residue with dry pyridine removed any condensed silanol-containing products, and recrystallization of the remaining crude product afforded pure $Cy_6Si_6O_9$ (1.4 g, 10%).

2.2. X-Ray crystallography

A crystal of approximate dimensions $0.3 \times 0.3 \times 0.4$ mm^3 was used for data collection.

Crystal data: $C_{36}H_{66}O_9Si_6$, $M = 811.4$ monoclinic, $a = 10.810(1)$, $b = 18.756(3)$, $c = 21.424(3)$ Å, $\beta = 93.78(2)°$, $U = 4334.5$ Å^3, space group $P2_1/m$, $Z = 4$, $D_c = 1.24$ g cm^{-3}, $\mu(Mo\ K_\alpha) = 2.3$ cm^{-1}, $F(000) = 1752$. Data were measured at room temperature on a

TABLE 2. Selected geometric data for $(C_6H_{11})_6Si_6O_9$ (e.s.d.s. in parentheses)

Bond lengths (Å)

Si(1)–O(1)	1.649(6)	Si1–O6	1.633(8)
Si(2)–O(2)	1.645(4)	Si2–O1	1.625(6)
Si(2)–O(5)	1.630(6)	Si3–O3	1.648(6)
Si(3)–O(6)	1.612(8)	Si4–O3	1.629(6)
Si(4)–O(4)	1.641(4)	Si4–O5	1.627(6)
Si(5)–O(7)	1.648(5)	Si5–O12	1.634(8)
Si(6)–O(7)	1.626(5)	Si6–O8	1.647(4)
Si(6)–O(11)	1.628(6)	Si7–O9	1.640(6)
Si(7)–O(12)	1.611(8)	Si8–O9	1.634(6)
Si(8)–O(10)	1.643(4)	Si8–O11	1.626(6)
Si(1)–C(1)	1.839(13)	Si2–C5	1.858(10)
Si(3)–C(11)	1.825(11)	Si4–C15	1.842(8)
Si(5)–C(21)	1.844(12)	Si6–C25	1.849(8)
Si(7)–C(31)	1.845(12)	Si8–C35	1.837(9)

Bond angles (°)

O(6)–Si(1)–O(1)	109.2(3)	C1–Si1–O1	111.9(3)
C(1)–Si(1)–O(6)	108.5(5)	O1–Si1–O1'	106.2(4)
O(2)–Si(2)–O(1)	106.6(3)	C5–Si2–O5	111.7(4)
O(5)–Si(2)–O(1)	109.5(3)	O5–Si2–O2	108.3(3)
C(5)–Si(2)–O(1)	110.2(4)	O5–Si2–O2	110.4(4)
O(6)–Si(3)–O(3)	109.0(3)	C11–Si3–O3	112.2(3)
C(11)–Si(3)–O(6)	108.5(5)	O3–Si3–O3'	105.8(4)
O(4)–Si(4)–O(3)	106.1(3)	C15–Si4–O5	109.5(3)
O(5)–Si(4)–O(3)	109.8(3)	O5–Si4–O4	109.2(4)
C(15)–Si(4)–O(3)	112.0(3)	C15–Si4–O4	110.3(4)
O(12)–Si(5)–O(7)	108.7(3)	C21–Si5–O7	112.0(3)
C(21)–Si(5)–O(12)	109.0(5)	O7–Si5–O7'	106.3(4)
O(8)–Si(6)–O(7)	107.1(3)	C25–Si6–O11	109.3(3)
O(11)–Si(6)–O(7)	109.3(3)	O11–Si6–O8	108.9(4)
C(25)–Si(6)–O(7)	113.1(3)	C25–Si6–O8	109.1(4)
O(12)–Si(7)–O(9)	108.3(3)	C31–Si7–O9	112.2(3)
C(31)–Si(7)–O(12)	109.2(5)	O9–Si7–O9'	106.4(4)
O(10)–Si(8)–O(9)	106.8(3)	C35–Si8–O11	108.3(3)
O(11)–Si(8)–O(9)	109.2(3)	O11–Si8–O10	109.4(4)
C(35)–Si(8)–O(9)	112.5(3)	C35–Si8–O10	110.6(4)
Si(2)–O(1)–Si(1)	128.8(4)	Si2–O2–Si2'	130.6(5)
Si(4)–O(3)–Si(3)	129.0(4)	Si4–O4–Si4'	130.8(5)
Si(4)–O(5)–Si(2)	139.3(4)	Si3–O6–Si1	144.7(5)
Si(6)–O(7)–Si(5)	128.7(4)	Si6–O8–Si6'	129.3(5)
Si(8)–O(9)–Si(7)	129.0(4)	Si8–O10–Si8'	129.5 (5)
Si(8)–O(11)–Si(6)	139.8(4)	Si7–O12–Si5	145.2(5)

CAD4 automatic four-circle diffractometer in the range $2 \le \theta \le 24°$. A total of 7018 reflections were collected of which 4372 were unique with $I \ge 3\sigma(I)$. Data were corrected for Lorentz and polarization effects but not for absorption. The structure was solved by direct methods and refined using the SHELX suite of programs [21,22]. The asymmetric unit consisted of two unique halves of the molecule (refined in blocks) and the remainder of each molecule was generated by reflection through a mirror plane. Interestingly, a similarly unusual way of generating the asymmetric unit was also observed in $(PhCH_2)_8Si_8O_{12}$, in which the contents of the unit cell $(P\bar{1})$ were generated by two independent half-molecules sitting on either side of the centre of symmetry [11]. In both independent halves of the title compound, a number of atoms (Si1, Si3, O2, O4, O6, C1, C4, C11, and C14 in molecule 1, and Si5, Si7, O8, O10, O12, C21, C24, C31 and C34 in molecule 2) were sited on the mirror planes with appropriate occupancies (0.5). In the final least squares cycles, all the silicon and oxygen atoms were allowed to vibrate anisotropically. Hydrogen atoms were included at calculated positions in all cases except for those bonded to carbon atoms lying on mirror planes. In these cases

the relevant hydrogens with half site-occupancy were located in the penultimate difference Fourier and refined at fixed distances (1.08 Å) from the parent atoms. Final residuals after 14 cycles of blocked matrix least squares were $R = R_w = 0.0861$ for unit weights. Maximum final shift/e.s.d. was 0.040. The max and min residual denitites were 0.31 and -0.24 e $Å^{-3}$, respectively. Final fractional atomic coordinates and isotropic thermal parameters, bond distances and angles are given in Tables 1 and 2, respectively. A complete list of bond lengths and angles and tables of anisotropic temperature factors and hydrogen atom coordinates have been deposited at the Cambridge Crystallographic Data Centre. The asymmetric unit is shown in Fig. 1, along with the labelling scheme used.

3. Results and discussion

In a notable paper published in 1965, Brown and Vogt [10] first described the preparation of $(C_6H_{11})_6Si_6O_9$, which is formed as a minor product upon controlled hydrolysis of $C_6H_{11}SiCl_3$ in acetone. Two other condensed sesquisiloxanes derivatives, containing 7-, and 8-silicon centres, $Cy_7Si_7O_9(OH)_3$ and
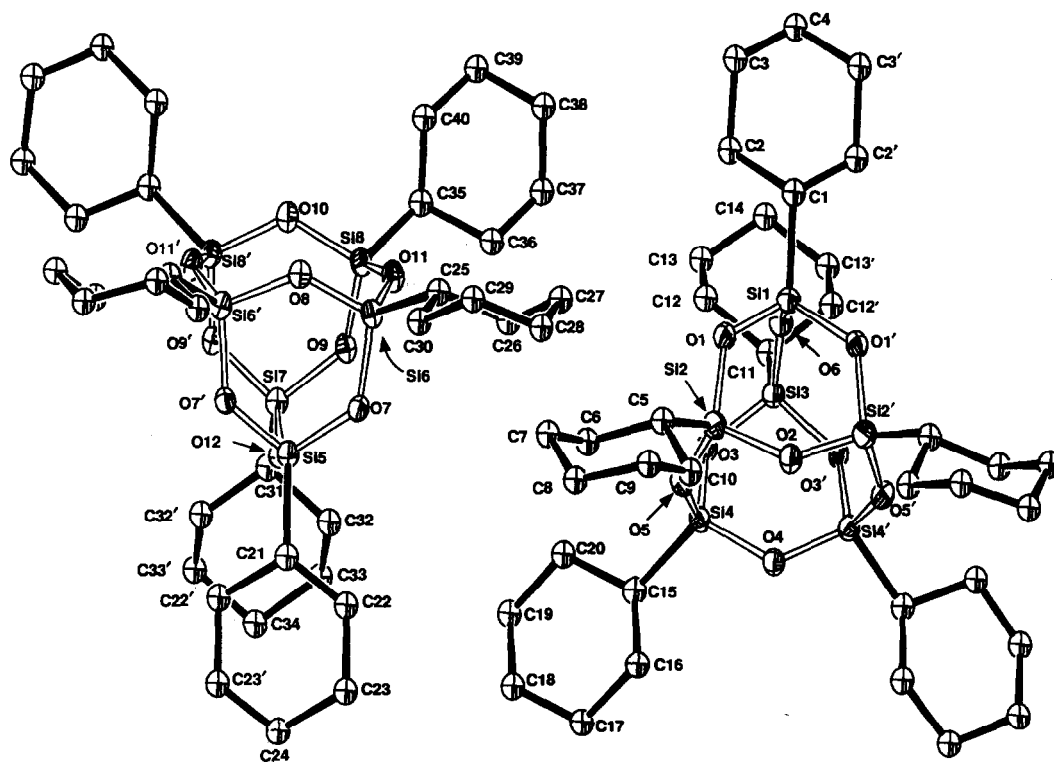

Fig. 1. The asymmetric unit of $Cy_6Si_6O_9$, which comprises two independent molecular halves. Primed atoms are related to their unprimed counterparts by mirror planes through Si1, Si3, O2, O4, O6, C1, C4, C11, and C14 in molecule 1, and Si5, Si7, O8, O10, O12, C21, C24, C31 and C34 in molecule 2. Thermal ellipsoids are at the 30% probability level.

$Cy_8Si_8O_{11}(OH)_2$, have also been identified as products of this reaction. Subsequently, Feher structurally characterized these other two products [23], both of which contain silanol groups, and extensively developed the chemistry of one of them [24]. We have used a slightly modified synthetic procedure but an identical separation method to those used by Feher [23], and by carrying out the reaction for a comparatively short reaction time, we have maximized the proportion of $(C_6H_{11})_6Si_6O_9$ and minimized the yield of octasesquisiloxane in the initial solid product, so facilitating purification of the hexamer albeit at the expense of a reduced yield. Physical and spectroscopic data were in accord with those reported previously [10,23].

The hexa-sesquisiloxane $Cy_6Si_6O_9$ adopts a cage structure in which two six-membered Si_3O_3 rings are joined co-facially by Si–O–Si bridges, generating three further Si_4O_4 heterocycles (Fig. 1). The bond length and angle data for the Si_3O_3 rings [mean Si–O: 1.640(6) Å; mean Si–O–Si angle: 129.5(5)°] are virtually unchanged from the parent $Ph_6Si_3O_3$ [1.640(6) Å; 132–133°] [25], indeed the data available for such rings, except for $Me_6Si_3O_3$, show a remarkable geometric consistency [Si–O, 1.635–1.654 Å; Si–O–Si, 125–135°] [26]. The Si_3O_3 rings in the two iso-structural silicate anions [cat]$_n$[Si$_6$O$_{15}^{6-}$] [cat = Et$_4$N$^+$, n = 6; Ni(en)$_3^{2+}$, n = 3] [18,19] also follow these geometric trends (Table 3) although the spread of Si–O bond distances and Si–O–Si angles is somewhat wider, possibly due to distortions brought about by extensive hydrogen-bonding within these lattices. However, in contrast to $R_6Si_3O_3$ species where the Si_3O_3 ring is invariably near-planar (maximum observed deviation from mean plane: 0.1 Å [26]), the two Si_3O_3 units in $Cy_6Si_6O_9$ each adopt a chair conformation (Fig. 2). This conformation is also apparent in [Et$_4$N$^+$]$_6$[Si$_6$O$_{15}^{6-}$] [18], although in [Ni(en)$_3^{2+}$]$_3$[Si$_6$O$_{15}^{6-}$] the components of the six-membered ring are described as "lying approxi-

Fig. 2. The sesquisiloxane cage of $Cy_6Si_6O_9$, highlighting the crown configuration of the Si_4O_4 rings (top of molecule) and the chair configuration adopted by the Si_3O_3 rings (left and right edges of molecule).

mately in one plane" [19]. In the title compound, as in siloxanes in general, the bond angles about silicon (105.8–112.2°) show only minor deviations from the ideal tetrahedral value.

The three eight-membered Si_4O_4 rings in $Cy_6Si_6O_9$ adopt a crown configuration, with the four oxygen atoms lying above an Si_4 plane. The two oxygen atoms that are solely part of the Si_4O_4 ring are significantly nearer to the Si_4 plane than the two oxygens that are also integral to the Si_3O_3 rings. For example, O5 and O6 lie 0.32, 0.25 Å above the Si1–Si4 plane, respectively, while the corresponding displacements for O1

TABLE 3. Comparative geometric data for organosesquisiloxanes and related compounds

	$R_3Si_3O_6$				$R_4Si_4O_8$				Ref.
	d(Si–O) (Å)		Si–O–Si (°)		d(Si–O) (Å)		Si–O–Si (°)		
	Range	Mean	Range	Mean	Range	Mean	Range	Mean	
$Cy_6Si_6O_9$	1.625(6)–1.649(6)	1.640	128.7(4)–130.8(5)	129.5	1.611(8)–1.634(8)	1.625	139.3(4)–145.2(5)	142.3	This work
$Si_6O_{15}^{6-}$ [a]	1.62(2)–1.67(2)	1.64	128(1)–132(2)	130.7	1.61(3)–1.66(3)	1.63	138(2)–146(1)	141	18
$Si_6O_{15}^{6-}$ [b]	1.63(2)–1.66(2)	1.65	128–136	132	1.60(2)–1.68(2)	1.64		137.7	19
$R_8Si_8O_{12}$ [c]					1.60–1.63	1.61	145.0–150.7	148.1	12
$(MeO)_8Si_8O_{12}$						1.604(2)		148.2(22)	2
$Ph_{12}Si_{12}O_{18}$					1.609(5)–1.618(5)	1.614	149.9(4)–158.1(5)	153.6	16
$Ph_6Si_3O_3$		1.64(1)		131.8(8)					25
$Ph_8Si_4O_4$						1.615	152.3(6)–167.4(2)	159.8	29 [d]

[a] [Et$_4$N$^+$] counterions. [b] [Ni(en)$_3^{2+}$] counterions. [c] Average of data for 7 structures. [d] Data taken from ref. 29, and are identical within experimental error to data quoted in ref. 30.

and O3 are 0.65 Å (Fig. 2). The Si–O bonds are somewhat shorter in this part of the molecule (mean 1.625 Å) and are associated with slighter more open Si–O–Si angles (mean 142.3°), a feature we have commented on previously [27,28]. In comparison, the data for the Si_4O_4 rings in the two silicate anions discussed earlier are consistent with the trends noted in the Si_3O_3 heterocycles, that is the mean data are virtually identical to those in the title organometallic species, but a wider spread of values within the rings is observed (Table 3).

It is noteworthy that eight-membered Si_4O_4 rings in higher sesquisiloxanes, where the heterocycle is fused to other Si_4O_4 ($R_8Si_8O_{12}$) or larger rings ($R_{10}Si_{10}O_{15}$ etc.), contain yet larger Si–O–Si angles (148–153°) and correspondingly shorter Si–O bonds (ca. 1.61 Å) (Table 3), a feature consistent with the relationship between these two parameters discussed earlier. These data approach those for the parent $Ph_8Si_4O_4$ (mean Si–O 1.615 Å; mean Si–O–Si angle, 159.8°) [29], which has been variously described as adopting a shallow boat [29] or planar [30] structure. Collectively, our results reflect the increasing strain induced in Si_4O_4 rings when fused to the other siloxane rings of decreasing nuclearity, and particularly when attached to an Si_3O_3 ring, as in the title compound.

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