where

$$X = ax \sin \beta, \ Y = by$$

and

$$Z = ax \cos \beta + cz.$$

The largest deviations from the planes are $-0 \cdot 009$ Å for C(3) and $-0 \cdot 005$ Å for C(5′) respectively. The dihedral angle between the six- and five-membered rings is $1 \cdot 8°$; thus the molecule as a whole is planar to within $0 \cdot 025$ Å. The planarity is the consequence of the rather strong intramolecular hydrogen bond N(2)–H(2B)$\cdots$N(2′). Details of the hydrogen bonding are given in Table 4. The mean plane through N(2), C(2), C(3), C(3′), N(2′), H(2B) is $0 \cdot 8720X + 0 \cdot 3204Y - 0 \cdot 3701Z = 3 \cdot 5162$ Å and the largest deviation from the plane is $0 \cdot 021$ Å for H(2B).

Table 4 shows that N(4′)–C(5′) and C(3′)–N(2′) in the oxadiazole ring exhibit double-bond character whereas the bond lengths in the aminopyridine unit suggest aromatic character. The methyl group attached to the oxadiazole ring does not appear to be disordered. H(7C) is nearly perpendicular to the oxadiazole ring and the torsion angles are N(4′)–C(5′)–C(6′)–H(7C) $+87 \cdot 1$, N(4′)–C(5′)–C(6′)–H(7A) $-153 \cdot 3$, N(4′)–C(5′)–C(6′)–H(7B) $-30 \cdot 9°$. Torsion angles are taken as positive if the bond in front has to be turned clockwise in order to eclipse the rear bond.

A view of the molecular packing is in Fig. 2. Discrete pairs of molecules exist which are held together by

hydrogen bonds. H(2A) is involved in the intermolecular hydrogen bonds N(2)–H(2A)$\cdots$N(1^i) and N(2^i)–H(2A^i)$\cdots$N(1), of $3 \cdot 024$ (5) Å [symmetry code: (i) $1 - x, -y, -z$]. All contacts between the pairs of molecules correspond to normal van der Waals distances.

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Octa(phenylsilasesquioxane)* Acetone Solvate

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Abstract. $C_{48}H_{40}O_{12}Si_8 \cdot C_3H_6O$, $M_r = 1091 \cdot 7$, tetragonal, $P4/n$, $a = b = 14 \cdot 608$ (4), $c = 12 \cdot 918$ (4) Å, $U = 2759 \cdot 5$ Å^3, $D_m = 1 \cdot 30$, $D_c = 1 \cdot 314$ Mg m^{-3}, $Z = 2$, $F(000) = 1136$, λ(Cu $K\alpha$) $= 1 \cdot 5418$ Å, μ(Cu $K\alpha$) = $2 \cdot 2$ mm^{-1}. The structure was refined to $R = 0 \cdot 0617$ for 1509 unique reflections. The $Ph_8Si_8O_{12}$ molecule is formed by linking six eight-membered rings and has fourfold crystallographic symmetry.

Introduction. Earlier reports suggested that octa(phenylsilasesquioxane) crystallizes in several forms: monoclinic, triclinic (Larsson, 1960a) and rhombohedral (Brown, Vogt & Prescott, 1964). As part of our general study of the structure and conformation of cyclosiloxanes, many of which seem to show polymorphism, we have begun an investigation of this system.

The compound was prepared by allowing phenyltrichlorosilane, acetone and water to stand for a few days (Brown, 1965) and recrystallized from dichloromethane and acetone as colourless plates. The pre-

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* 1,3,5,7,9,11,13,15-Octaphenylpentacyclo[9.5.1.1^{3,9}.1^{5,15}.1^{7,13}]-octasiloxane.

Table 1. *Fractional coordinates* ($\times 10^4$) *of the non-hydrogen atoms with e.s.d.'s in parentheses*

	x	y	z
Si(1)	3924 (1)	1996 (1)	4616 (1)
Si(2)	3917 (1)	1974 (1)	2233 (1)
O(1)	4239 (2)	1878 (2)	3426 (2)
O(2)	3647 (2)	3050 (2)	4819 (2)
O(3)	3029 (2)	1345 (2)	2028 (2)
C(11)	4862 (3)	1696 (3)	5502 (3)
C(12)	4897 (4)	882 (4)	6034 (4)
C(13)	5620 (5)	675 (5)	6705 (4)
C(14)	6286 (5)	1284 (6)	6867 (5)
C(15)	6262 (4)	2116 (6)	6362 (6)
C(16)	5568 (3)	2310 (4)	5682 (5)
C(21)	4843 (3)	1592 (3)	1384 (3)
C(22)	4691 (4)	1501 (4)	331 (4)
C(23)	5376 (5)	1192 (4)	−337 (5)
C(24)	6216 (5)	979 (5)	49 (6)
C(25)	6381 (4)	1064 (5)	1078 (6)
C(26)	5705 (3)	1361 (3)	1747 (4)
C(1)	2500 (0)	2500 (0)	−1742 (13)
C(2)	1634 (19)	2200 (35)	−2241 (15)
O(4)	2500 (0)	2500 (0)	−823 (11)

Table 2. *Bond lengths* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

Si(1)−O(1)	1·613 (3)	Si(2)−O(1)	1·618 (3)
Si(1)−O(2)	1·613 (3)	Si(2)−O(3)	1·612 (3)
Si(1)−O(2')*	1·606 (3)	Si(2)−O(3')	1·610 (3)
Si(1)−C(11)	1·839 (4)	Si(2)−C(21)	1·828 (4)

O(1)−Si(1)−O(2)	109·2 (1)	O(1)−Si(2)−O(3)	109·9 (1)
O(1)−Si(1)−O(2')	108·6 (2)	O(1)−Si(2)−O(3')	108·0 (1)
O(1)−Si(1)−C(11)	110·8 (2)	O(1)−Si(2)−C(21)	109·3 (2)
O(2)−Si(1)−O(2')	109·4 (1)	O(3)−Si(2)−O(3')	109·1 (2)
O(2)−Si(1)−C(11)	108·3 (2)	O(3)−Si(2)−C(21)	108·9 (2)
O(2')−Si(1)−C(11)	110·5 (2)	O(3')−Si(2)−C(21)	111·7 (2)

Si(2)−O(1)−Si(1)	144·7 (2)	Si(2)−O(3)−Si(2')	151·6 (2)
Si(1)−O(2)−Si(1')	151·4 (2)		

C(11)−C(12)	1·373 (6)	C(21)−C(22)	1·384 (6)
C(12)−C(13)	1·400 (7)	C(22)−C(23)	1·397 (7)
C(13)−C(14)	1·335 (9)	C(23)−C(24)	1·360 (8)
C(14)−C(15)	1·379 (10)	C(24)−C(25)	1·356 (9)
C(15)−C(16)	1·372 (7)	C(25)−C(26)	1·382 (7)
C(16)−C(11)	1·387 (6)	C(26)−C(21)	1·386 (6)

C(16)−C(11)−Si(1)	120·3 (4)	C(26)−C(21)−Si(2)	122·9 (3)
C(12)−C(11)−Si(1)	123·1 (4)	C(22)−C(21)−Si(2)	120·0 (3)
C(12)−C(11)−C(16)	116·7 (4)	C(26)−C(21)−C(22)	117·0 (4)
C(13)−C(12)−C(11)	121·6 (6)	C(23)−C(22)−C(21)	121·6 (5)
C(14)−C(13)−C(12)	120·2 (6)	C(24)−C(23)−C(22)	119·6 (6)
C(15)−C(14)−C(13)	119·6 (6)	C(25)−C(24)−C(23)	119·9 (5)
C(16)−C(15)−C(14)	120·2 (7)	C(26)−C(25)−C(24)	120·9 (6)
C(11)−C(16)−C(15)	121·6 (6)	C(21)−C(26)−C(25)	121·0 (6)

* The primed atoms are related to the unprimed ones by fourfold symmetry, as indicated in Fig. 1.

liminary cell parameters and crystal system, which was found to be tetragonal, were determined from oscillation and Weissenberg photographs. Accurate cell parameters were obtained by least squares from the setting angles for 25 reflections automatically centred on a Nonius CAD-4 diffractometer. The space group was uniquely determined as $P4/n$ from the systematic absences $hk0$ for $h + k =$ odd. 2348 intensities ($3 < \theta < 60°$) were collected from a crystal $0.25 \times 0.20 \times 0.12$ mm with Ni-filtered Cu $K\alpha$ radiation and an ω–2θ scan technique. A periodic check of two standard reflections showed only minor fluctuations. The data were corrected for Lorentz and polarization effects and reduced to 1509 unique reflections with $F_o > 3\sigma(F_o)$ which were used in the analysis. No corrections were made for absorption or extinction.

The structure was determined by direct methods and difference synthesis and refined by full-matrix least squares. With the non-hydrogen atoms treated anisotropically and the phenyl H atoms (inserted at calculated positions, C−H = 1·08 Å) isotropically, the structure refined to $R = 0.10$. A difference map computed at this stage showed three unique peaks of significant electron density, two sited on the fourfold axes and one in a general fourfold position. These peaks were interpreted as belonging to a disordered acetone molecule and were included in the refinement with anisotropic temperature factors at the final stage. The refinement finally converged at $R = 0.0617$. The weighting scheme was $\omega = 1/[\sigma^2(F_o) + 0.0004F_o^2]$, which gave flat analyses of $\omega\Delta^2$ with $\sin\theta$ and $(F_o/F_{max})^{1/2}$. The final non-hydrogen atomic co-ordinates are given in Table 1 and the interatomic

distances and angles in Table 2.* Neutral-atom scattering factors were taken from Stewart, Davidson & Simpson (1965) for H and from Cromer & Mann (1968) for Si, O and C.

Discussion. The structure of the molecule is illustrated in Fig. 1, which also shows the atom numbering. The structure is analogous to that of octa(methylsilasesquioxane) (Larsson, 1960*b*). In the molecule, six eight-membered rings are linked together to form a cage with Si atoms at the corners of a slightly distorted cube and O atoms bridging all twelve edges. The cage has a four-fold symmetry with Si $\cdots$ Si distances in the upper and lower squares of 3·120 and 3·123 Å and a side edge of 3·078 Å. As can be seen from the x and y coordinates of Si(1) and Si(2) the upper and lower squares adopt an almost perfect eclipsed configuration. The Si−O bonds (ave. 1·613 Å) are normal. The Si−C bonds (ave.
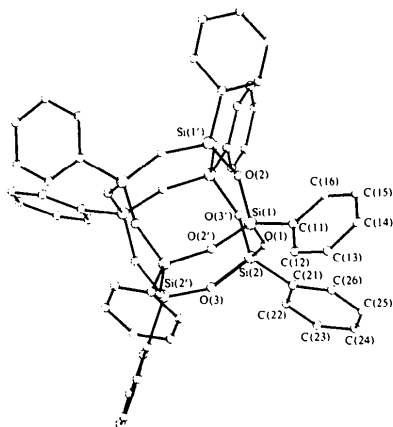
Fig. 1. The molecular structure of octa(phenylsilasesquioxane) showing the numbering scheme.
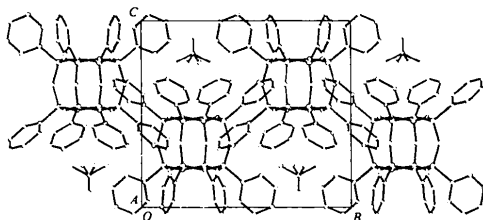

Fig. 2. The molecular packing viewed down [100].

1·833 Å) are shorter than those in the methyl derivative (ave. 1·895 Å), as expected. The angles at the Si atoms are close to tetrahedral. The average angle at the O atom (148·5°) is similar to those found in most other eight-membered siloxane ring systems (Shklover, Kalinin, Gusev, Bokii, Struchkov, Andrianov & Petrova, 1973; Carlström & Falkenberg, 1973; Söderholm & Carlström, 1977; Söderholm, 1978). An interesting feature of this crystal structure is that the acetone molecule, which is not lost by the crystals even

on prolonged exposure to air, is not involved in any specific intermolecular interaction but is trapped in a cavity formed by six silasesquioxane molecules. The packing of the silasesquioxane molecules involves a number of centrosymmetric phenyl–phenyl arrangements but these are not close, the shortest $C \cdots C$ distance being 3·752 Å. The packing of the molecules is shown in Fig. 2.

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Structure of L-γ-Carboxyglutamic Acid

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Abstract. $C_6H_9NO_6$, $M_r = 191·13$, monoclinic, $P2_1$, $a = 5·050$ (3), $b = 9·600$ (5), $c = 7·793$ (2) Å, $\beta = 99·98$ (3)°, $V = 371·4$ Å^3, $Z = 2$, $D_m = 1·70$, $D_x = 1·71$ Mg m^{-3}; refined to a conventional R value of 0·052 for 749 observed reflections. The molecule is a zwitterion: the amino group is protonated and the C_α [C(2)] and a C_γ [C(4)] carboxyl groups are partially ionized, sharing a proton in the formation of a short