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*Acta Cryst.* (1978). **B34**, 124–128

## The Crystal and Molecular Structure of Oxobis[triphenylsilicon(IV)]

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(Received 1 July 1977; accepted 28 July 1977)

Crystals of oxobis(triphenylsilicon),  $O(\text{SiPh}_3)_2$ , are triclinic, space group  $P\bar{1}$ ,  $a = 11.301(2)$ ,  $b = 9.539(2)$ ,  $c = 8.771(2)$  Å,  $\alpha = 66.34(1)$ ,  $\beta = 114.35(1)$ ,  $\gamma = 114.24(1)^\circ$ ,  $Z = 1$ . The structure was solved by direct methods using diffractometer data and refined by full-matrix least squares to  $R = 0.0374$ . The molecules are centrosymmetric, so that the SiOSi fragment is strictly linear: principal interatomic distances are Si–O 1.616(1) Å, and Si–C 1.864(5) Å; the central  $C_6Si_2O$  part of the molecule exhibits almost exact  $3m$  symmetry.

### Introduction

The bond angles at O in the Si ethers  $O(\text{SiH}_3)_2$  (Almenningen, Bastiansen, Ewing, Hedberg & Trætterberg, 1963),  $O(\text{SiF}_3)_2$  (Airey, Glidewell, Rankin, Robiette, Sheldrick & Cruickshank, 1970), and  $O(\text{SiCl}_3)_2$  (Airey, Glidewell, Robiette & Sheldrick, 1971) are 144.1, 155.7 and 146° respectively, each markedly greater than the value of *ca* 110° expected from the VSEPR model. The structures were determined by gas-phase electron diffraction, and hence the angles quoted represent a mean over all the thermally populated vibrational states: in the case of  $O(\text{SiF}_3)_2$  [but not  $O(\text{SiH}_3)_2$ ] the possibility that the molecule has a linear ground vibrational state could not be ruled out, the observed non-linearity then being a consequence of a very low SiOSi bending frequency. It has been suggested (Glidewell, 1975) that the observed angles in these ethers are limited primarily by the non-bonded Si...Si distance. As part of a structural study of oxo and hydroxo compounds, we have investigated the

solid-state structure of a related Si ether, oxobis(triphenylsilicon),  $O(\text{SiPh}_3)_2$ , whose crystal and molecular structure is reported here.

### Experimental

#### Preparation

Oxobis[triphenylsilicon(IV)] was prepared by a method analogous to that used for the preparation of oxobis[ $\alpha$ -naphthylphenylmethylsilicon(IV)] (Sommer, Frye & Parker, 1964).

To a solution of hydroxotriphenylsilicon(IV) (2.0 g, 7.2 mmol) in dry toluene (30 cm<sup>3</sup>) was added excess powdered potassium hydroxide (7 g). The mixture was shaken vigorously for 10 min, then allowed to stand for several hours. The excess KOH was removed by filtration. A solution of chlorotriphenylsilicon(IV) (2.1 g, 7.2 mmol) in dry toluene (20 cm<sup>3</sup>) was added to the filtrate and the reaction was allowed to stand (2 h). The precipitate of KCl so formed was separated using a centrifuge. The supernatant solution was reduced in

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volume and, on standing, the product separated as a crystalline solid. Recrystallization from petrol (b.p. 60–80°C) yielded colourless crystals of oxobis[triphenylsilicon(IV)]. Yield 2.1 g (55%) (m.p. 199–200°C); identity was confirmed by mass spectrometry.

#### Data collection

Data were collected using a crystal of dimensions 0.30 × 0.28 × 0.14 mm. Unit-cell parameters were determined by least-squares fits to the reflecting positions of 25 reflections, each measured a total of 10 times during data collection using a Philips PW 1100 automatic four-circle diffractometer with graphite-monochromatized Mo  $K\alpha$  radiation. The intensities of 2842 reflections with  $3 \leq \theta \leq 25^\circ$  were measured by the  $\omega/2\theta$  scan technique, with a scan width of  $(0.90 + 0.1 \tan \theta)^\circ$  in  $\omega$ , a scan speed of  $0.05^\circ \text{ s}^{-1}$  in  $\omega$  and a maximum of two scans per reflection (a second scan was made if the total count obtained in the first scan was less than 500). Backgrounds were measured at either end of the scan range for a time equal to (scan time/2)  $\sqrt{(I_{\text{BK}}/I_{\text{INT}})}$  or a minimum of 10 s, where  $I_{\text{BK}}$  = the background intensity scaled from an initial 10 s measurement and  $I_{\text{INT}}$  = integrated peak intensity. Three standard reflections were measured every six hours during data collection, and showed only random deviations from their mean intensities. Lorentz and polarization corrections were applied to the data, but no corrections for absorption were made.

#### Crystal data

$\text{C}_{36}\text{H}_{30}\text{Si}_2\text{O}$ ,  $M_r = 534.82$ , triclinic,  $a = 11.301$  (2),  $b = 9.539$  (2),  $c = 8.771$  (2) Å (1 Å = 100 pm),  $\alpha = 66.34$  (1),  $\beta = 114.35$  (1),  $\gamma = 114.24$  (1)°,  $U = 754.2$  Å<sup>3</sup>;  $Z = 1$ ,  $D_c = 1.177$ ,  $D_m = 1.172$  g cm<sup>-3</sup>;  $F(000) = 282$ ; Mo  $K\alpha$  radiation,  $\lambda = 0.71069$  Å,  $\mu(\text{Mo } K\alpha) = 1.07$  cm<sup>-1</sup>. Space group  $P\bar{1}$  ( $C_i^1$ , No. 2) (see below): the O atom is at a centre of symmetry; the asymmetric unit consists of a  $\text{Ph}_3\text{SiO}_{1/2}$  fragment.

#### Structure solution and refinement

These were carried out using the *SHELX* program system (Sheldrick, 1976).

The  $E$  statistics suggested the space group  $P\bar{1}$ , and the structure was solved in this space group using the automatic centrosymmetric direct-methods program in *SHELX*. The  $E$  map for the most probable solution furnished positions for all non-hydrogen atoms in the asymmetric unit.

Six cycles of full-matrix least-squares refinement using 2101 independent reflections having  $F_o \geq 6\sigma(F_o)$ , with isotropic temperature parameters for all non-hydrogen atoms [78 variables, the O atom fixed at  $(\frac{1}{2}, \frac{1}{2}, \frac{1}{2})$ ] and a weighting scheme  $w = [\sigma(F)]^{-2}$ , yielded a

conventional  $R$  index ( $= \Sigma \Delta / \Sigma F_o$ , where  $\Delta = |F_o - F_c|$ ) of 0.1137, a weighted index  $R_w$  ( $= \Sigma w^{1/2} \Delta / \Sigma w^{1/2} F_o$ ) of 0.1306 and a generalized index  $R_G$  [ $= (\Sigma w \Delta^2 / \Sigma w F_o^2)^{1/2}$ ] of 0.1559. The introduction of anisotropic temperature parameters for the Si atom in three cycles of refinement (83 variables) reduced  $R_G$  to 0.1499. Three further cycles of refinement with anisotropic temperature parameters for all non-hydrogen atoms (178 variables) yielded an  $R_G$  of 0.0991. At this stage a difference synthesis furnished positions for all the H atoms. Nine cycles of refinement with the H atom positional parameters included and refined and with a common H isotropic temperature parameter (224 variables) reduced  $R_G$  to 0.0505. For this refinement the least-squares matrix was blocked such that the overall scale, the Si positional and temperature parameters, the O temperature parameters and the common temperature parameter all refined in every cycle, but the parameters for the atoms in each phenyl ring refined only once every three cycles. A further nine cycles of refinement with the matrix blocked as above, but with individual isotropic temperature parameters for the H atoms (238 variables), yielded final indices of  $R = 0.0374$ ,  $R_w = 0.0417$  and

Table 2. Atomic coordinates

	x	y	z
Si	0.34544 (5)	0.43422 (5)	0.38559 (6)
O	0.5	0.5	0.5
C(11)	0.3055 (2)	0.5949 (2)	0.1792 (2)
C(12)	0.1764 (2)	0.6140 (2)	0.1037 (3)
C(13)	0.1454 (3)	0.7312 (3)	-0.0507 (3)
C(14)	0.2420 (3)	0.8310 (3)	-0.1305 (3)
C(15)	0.3695 (3)	0.8158 (3)	-0.0600 (4)
C(16)	0.4019 (3)	0.6992 (3)	0.0940 (3)
C(21)	0.3264 (2)	0.2535 (2)	0.3342 (2)
C(22)	0.3547 (2)	0.1194 (2)	0.4672 (3)
C(23)	0.3386 (3)	-0.0170 (3)	0.4325 (4)
C(24)	0.2959 (3)	-0.0229 (3)	0.2653 (4)
C(25)	0.2677 (4)	0.1054 (4)	0.1326 (4)
C(26)	0.2818 (3)	0.2429 (3)	0.1664 (3)
C(31)	0.2300 (2)	0.3824 (2)	0.5146 (2)
C(32)	0.1200 (2)	0.2440 (3)	0.5230 (3)
C(33)	0.0331 (3)	0.2104 (4)	0.6147 (4)
C(34)	0.0525 (3)	0.3137 (4)	0.7001 (4)
C(35)	0.1588 (4)	0.4513 (4)	0.6942 (4)
C(36)	0.2483 (3)	0.4855 (3)	0.6048 (3)
H(12)	0.109 (2)	0.540 (2)	0.157 (3)
H(13)	0.053 (3)	0.746 (3)	-0.099 (3)
H(14)	0.217 (3)	0.909 (3)	-0.231 (4)
H(15)	0.436 (3)	0.881 (3)	-0.106 (4)
H(16)	0.481 (2)	0.686 (3)	0.134 (3)
H(22)	0.388 (2)	0.119 (3)	0.597 (3)
H(23)	0.349 (2)	-0.108 (3)	0.525 (3)
H(24)	0.290 (2)	-0.121 (3)	0.248 (3)
H(25)	0.228 (3)	0.105 (3)	0.018 (4)
H(26)	0.272 (2)	0.337 (3)	0.070 (3)
H(32)	0.108 (2)	0.170 (3)	0.470 (3)
H(33)	-0.048 (3)	0.113 (3)	0.621 (4)
H(34)	-0.005 (3)	0.284 (3)	0.768 (4)
H(35)	0.166 (3)	0.511 (4)	0.754 (4)
H(36)	0.328 (2)	0.575 (3)	0.607 (3)

$R_G = 0.0497$ . The decreases in  $R_G$  at each stage of the refinement were all significant at the 0.5% level (Hamilton, 1965).

A parallel refinement in space group  $P1$  (472 variables in the final refinement) converged to yield final indices of  $R = 0.0338$ ,  $R_w = 0.0367$  and  $R_G = 0.0424$ . The reduction in  $R_G$  compared with the refinement in

space group  $P\bar{1}$  was significant at the 0.5% level; however, we reject the structure in  $P1$  on the grounds of the unacceptably wide ranges spanned by chemically equivalent bond distances, thus: Si—O 0.045, Si—C( $j1$ ) 0.049, C( $j1$ )—C( $j2$ ) 0.072, C( $j2$ )—C( $j3$ ) 0.162, C( $j3$ )—C( $j4$ ) 0.164, and C—H 0.49 Å.

An analysis of variance showed no significant dependence on the parity group,  $\sin \theta$ , or the magnitude of  $F_o$  (Table 1).<sup>\*</sup> Complex neutral-atom scattering factors (Cromer & Mann, 1968; Doyle & Turner,

Table 3. *Geometry at silicon*

(a) Bond distances (Å)

Si—O	1.616 (1)	Si—C(21)	1.860 (2)
Si—C(11)	1.870 (2)	Si—C(31)	1.862 (3)

(b) Angles (°)

O—Si—C(11)	108.8 (1)	C(11)—Si—C(12)	109.9 (1)
O—Si—C(21)	109.7 (1)	C(11)—Si—C(13)	110.1 (1)
O—Si—C(31)	108.4 (1)	C(12)—Si—C(13)	109.9 (1)

Table 4. *Distances within the phenyl rings (Å)*

(a) C—C Bond $C_{ij}$ — $C_{ik}$	Ring, $i$		
	1	2	3
1—2	1.388 (3)	1.389 (3)	1.394 (3)
2—3	1.386 (3)	1.378 (5)	1.378 (5)
3—4	1.353 (4)	1.358 (5)	1.364 (6)
4—5	1.358 (5)	1.351 (4)	1.369 (4)
5—6	1.385 (3)	1.391 (6)	1.386 (6)
6—1	1.385 (3)	1.377 (4)	1.399 (4)

(b) C—H Bond $C_{ij}$ — $H_{ij}$	Ring, $i$		
	1	2	3
$j$			
2	0.94 (2)	1.04 (3)	0.94 (3)
3	1.00 (3)	0.93 (2)	1.00 (2)
4	0.92 (3)	0.98 (4)	0.94 (4)
5	0.88 (3)	0.92 (3)	0.88 (4)
6	0.86 (3)	0.97 (2)	0.95 (2)

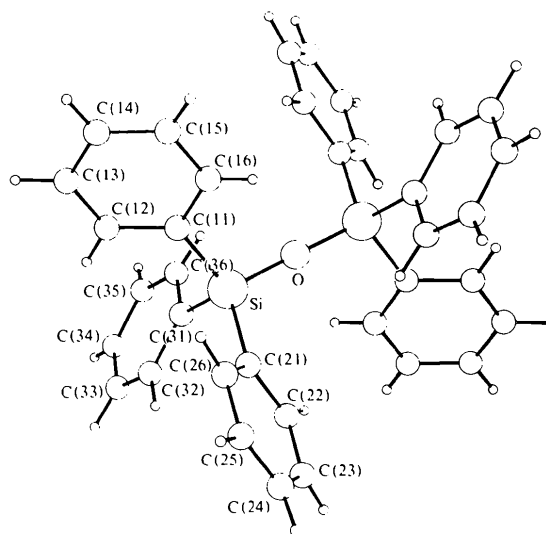


Fig. 1. A molecule of  $O(SiPh)_2$  showing the numbering of the non-hydrogen atoms.

Table 5. *Bond angles at carbon (°)*

(a) C—C—C and C—C—Si

Ring, $i$	Angle*							
	6—1—2	1—2—3	2—3—4	3—4—5	4—5—6	5—6—1	2—1—Si	6—1—Si
1	116.9 (2)	121.4 (2)	120.2 (3)	120.0 (2)	120.5 (3)	121.1 (3)	120.0 (1)	122.1 (2)
2	116.6 (2)	121.5 (3)	120.4 (2)	119.7 (4)	120.4 (3)	121.4 (2)	120.5 (2)	122.9 (2)
3	116.6 (3)	121.8 (3)	120.4 (3)	119.5 (4)	120.7 (4)	120.9 (2)	122.7 (2)	120.6 (2)

(b) C—C—H

Ring, $i$	Angle†									
	1—2—H(2)	3—2—H(2)	2—3—H(3)	4—3—H(3)	3—4—H(4)	5—4—H(4)	4—5—H(5)	6—5—H(5)	5—6—H(6)	1—6—H(6)
1	117 (1)	121 (1)	120 (1)	120 (1)	117 (2)	123 (2)	122 (2)	117 (2)	120 (1)	119 (1)
2	121 (1)	118 (1)	119 (2)	120 (2)	117 (1)	123 (1)	124 (2)	115 (2)	119 (2)	119 (2)
3	119 (1)	119 (1)	124 (2)	116 (2)	118 (2)	122 (2)	115 (2)	125 (2)	123 (2)	116 (2)

\*  $C_{ij}$ — $C_{ik}$ — $C_{il}$  or  $C_{ij}$ — $C_{ik}$ —Si.

†  $C_{ij}$ — $C_{ik}$ — $H_{ik}$ .

<sup>\*</sup> Lists of structure factors, thermal parameters and Table I have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 32974 (15 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 6. *Least-squares planes*

Least-squares planes are defined in terms of orthogonal Cartesian coordinates by the equation:  $AX + BY + CZ + 1 = 0$ ;  $X$ ,  $Y$  and  $Z$  are related to the cell (ångström) coordinates by:  $X = x + y \cos \gamma + z \cos \beta$ ,  $Y = y \sin \gamma - z \sin \beta \cos \alpha^*$ ,  $Z = z \sin \beta \sin \alpha^*$ . The superscript (i) refers to the symmetry position  $1 - x$ ,  $1 - y$ ,  $1 - z$ .

Plane	$j$	$A$	$B$	$C$	Atom distances from the plane ( $\text{Å} \times 10^3$ )						
<i>(a) Planes through Si, Si<sup>i</sup>, O, C(j1), C(j1)<sup>i</sup></i>											
1	1	0.2476	-0.1637	-0.1491							
2	2	0.8455	0.7474	-1.740							
3	3	0.1332	-0.3381	0.1555							
<i>(b) Planes through phenyl ring<sup>†</sup></i>											
					C(j1)	C(j2)	C(j3)	C(j4)	C(j5)	C(j6)	Si
4	1	0.0474	-0.1584	-0.1010	2	-2	2	-1	1	-2	25
5	2	-0.3878	-0.1515	0.0088	-2	-3	6	-2	-3	6	25
6	3	-0.3020	0.1601	-0.4923	3	2	-3	-2	7	-8	70
Dihedral angles between planes ( $^\circ$ )											
Planes		1-2	1-3	2-3	1-4	2-5	3-6				
Angles		59.8	60.3	59.9	35.0	58.1	47.8				

<sup>†</sup> Planes are calculated from the carbon positions only.

Table 7. *Selected non-bonded distances (Å)*

<i>(a) Intramolecular</i>						
Si...Si <sup>i</sup>	3.232					
Si...C(12)	2.845	Si...C(22)	2.830	Si...C(32)	2.866	
Si...C(16)	2.854	Si...C(26)	2.853	Si...C(36)	2.842	
Si...H(12)	2.89	Si...H(22)	2.95	Si...H(32)	2.95	
Si...H(16)	2.92	Si...H(26)	2.95	Si...H(36)	2.87	
O...C(11)	2.839	O...C(21)	2.845	O...C(31)	2.825	
O...H(16)	2.95			O...H(36)	2.895	
C(11)...H(26)	2.81	C(21)...H(32)	2.86	C(31)...H(12)	2.89	
<i>(b) Intermolecular distances (Å) less than 3 Å</i>						
C(12)...H(24) <sup>ii</sup>	2.86	C(23)...H(33) <sup>iii</sup>	2.91	C(35)...H(26) <sup>iv</sup>	2.91	

Superscripts refer to the following symmetry positions:

- (i)  $1 - x, 1 - y, 1 - z$  (ii)  $x, 1 + y, z$  (iii)  $\bar{x}, \bar{y}, 1 - z$  (iv)  $x, y, 1 + z$ .

1968) were employed for all atoms. In the final refinement, 238 parameters were varied, comprising 102 positional coordinates, 120 anisotropic temperature-factor components, 15 isotropic temperature factors and one overall scale factor: all parameter shifts were less than  $0.045\sigma$ ; a difference synthesis revealed no residual electron density greater than  $+0.20$  or less than  $-0.17 e \text{ Å}^{-3}$ .

The results from the final least-squares cycle are given in Table 2: these, together with the full-covariance matrix, were used to calculate the bond lengths and angles, which are given with e.s.d.'s in Tables 3, 4 and 5. No correction for thermal motion has been applied to the bond lengths. Least-squares planes were calculated for each of the three C-Si-O-Si<sup>i</sup>-C<sup>i</sup> fragments and for each of the three

phenyl rings: these, the deviations from them and the dihedral angles between them are given in Table 6. Some non-bonded distances are listed in Table 7. Fig. 1 is a perspective view of the molecule showing the numbering scheme (H atoms are numbered according to the C atoms to which they are bonded).

## Discussion

In the space group  $P\bar{1}$ , the  $O(\text{SiPh}_3)_2$  molecule is centrosymmetric: the possibility that the O atom is disordered is eliminated both by the difference map and by the dimensions of the thermal ellipsoid of the O. Consequently, the SiOSi fragment is strictly linear: the dihedral angles between the planes defined by

$C(j1)-Si-O-Si^i-C(j1)^i$  are 59.8, 60.3 and 59.9°, so that the central  $C_3SiOSiC_3$  core of the molecule has almost perfect  $\bar{3}m(D_{3d})$  symmetry. [The  $P1$  refinement led to  $\angle SiOSi = 176.8(7)^\circ$ .]

The Si—O distance of 1.616(1) Å is intermediate between those found in  $O(SiH_3)_2$  [1.634(2) Å] (Almenningen, Bastiansen, Ewing, Hedberg & Trætterberg, 1963), and  $O(SiCl_3)_2$  [1.592(10) Å] (Airey, Glidewell, Robiette & Sheldrick, 1971), so that no correlation of  $r(Si-O)$  with  $\angle SiOSi$  is possible, but it is almost identical with the distances found in a number of lanthanoid disilicates,  $Ln_2Si_2O_7$ , whether linear or markedly non-linear at O (Smolin & Shepelev, 1970; Felsche, 1971; Smolin, Shepelev & Titov, 1972).

The geometry adopted by the SiOSi bridges in disilicate ions has generally been discussed in terms of Si—O  $\pi$  bonding (Cruickshank, 1961): a low  $\pi$ -bond order in the terminal Si—O bonds leads to a high  $\pi$ -bond order in the bridging Si—O bonds, which requires an opening of the SiOSi angle. If this reasoning is transferred to neutral  $O(SiX_3)_2$  species, then a low Si—X  $\pi$ -bond order should be accompanied by a high Si—O  $\pi$ -bond order and a wide SiOSi angle, so that the widest SiOSi angle should be observed in  $O(SiH_3)_2$ , where the Si—H  $\pi$ -bond order must be negligible. However, this molecule has the lowest SiOSi angle observed in this series; furthermore, the possibility of a linear ground state accompanied by a large-amplitude bending vibration was conclusively eliminated (Almenningen, Bastiansen, Ewing, Hedberg & Trætterberg, 1963). Hence, the validity of the  $\pi$ -bonding rationalization in neutral  $O(SiX_3)_2$  is to be doubted.

The linear SiOSi bridge found here for  $O(SiPh_3)_2$  is unlikely to be a result of intermolecular forces, unless the bridge is unusually easily deformed, since in the C and Ge analogues the  $MOM$  angles ( $M = C, Ge$ ) are 128 and 135.2° respectively (Glidewell & Liles, 1978*a,b*), again both markedly greater than the VSEPR prediction. We suggest that  $O(SiH_3)_2$ ,  $O(CPh_3)_2$  and  $O(GePh_3)_2$  have non-linear ground states in which the observed angles at O reflect the non-bonded distances  $M \cdots M$  ( $M = Si, C, Ge$ ) (Glidewell, 1975), and that  $O(SiPh_3)_2$  and possibly  $O(SiF_3)_2$  have linear ground states; the reason for the marked difference in geometry between  $O(SiH_3)_2$  and  $O(SiPh_3)_2$  is not understood.

The Si—C distances span the range 1.860(2)–

1.870(2) Å [mean 1.864(5) Å], very similar to those found in  $Ph_4Si$  [1.872(7) Å] (Glidewell & Sheldrick, 1971), and  $Ph_3SiC(N_2)Ph$  [1.860(13) Å] (Glidewell & Sheldrick, 1972). The internal angles at C(11), C(21) and C(31) are 116.9(2), 116.6(2) and 116.6(3)° respectively, typical of phenyl rings bound to Si (Domenicano, Vaciano & Coulson, 1975).

No short intramolecular contacts occur (Table 7).

We thank Drs P. G. Owston and M. McPartlin (Polytechnic of North London) for the use of the diffractometer, and the SRC for support.

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