The five-membered chelate ring shows an envelope-like conformation with N deviating -0.439 (4) Å from the plane through Cu, O(1), P, C(1).

The crystal structure of the Cu complex contains a network of weak hydrogen bonds. The amino group and O(1), O(2) of the phosphinato group are coordinated to Cu and also form hydrogen bonds. All H atoms attached to N are involved [H(2)$\cdots$O(1)($-x$, $\frac{1}{2}+y$, $\frac{1}{2}-z$) 2·27, N$\cdots$O(1) 2·942 (5) Å, $\angle$N—H(2)$\cdots$ O(1) 124°; and H(1)$\cdots$O(2)(x, $\frac{1}{2}-y$ $-\frac{1}{2}+z$) 2·22, N(1)$\cdots$O(2) 3·177 (6) Å, $\angle$N—H(1)$\cdots$O(2) 160°].

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On the SiOSi Angle in 1,2-Di-*tert*-butyl-1,1,2,2-tetraphenyldisiloxane

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Abstract. $C_{32}H_{38}OSi_2$, $M_r = 494·83$, monoclinic, $P2_1/n$, $a = 11·783$ (2), $b = 14·259$ (2), $c = 17·358$ (2) Å, $\beta = 90·90$ (1)°, $V = 2916·0$ Å³, $Z = 4$, $D_x = 1·127$ g cm⁻³, Cu $K\alpha$, $\lambda = 1·54178$ Å, $\mu = 12·5$ cm⁻¹, $F(000) = 1063·9$, room temperature, final $R = 4·54\%$ for 3981 reflections with $|F_o| > 3\sigma$. The principal geometric parameters are: r(Si—O)$_{av} = 1·635$ (3) Å; $\angle$SiOSi $= 152·4$ (1)°; r(Si$\cdots$Si) $= 3·177$ (1) Å; r(Si—C)$_{av} = 1·879$ (5) Å. The *tert*-butyl groups are *gauche* to each other when considered with respect to the virtual Si$\cdots$Si bond. The six Si—C bonds are staggered about the Si$\cdots$Si direction with approximate $\bar{3}$ symmetry. One Si—C(butyl) bond is near the plane formed by the SiOSi moiety.

Introduction. Organodisiloxanes appear to adopt either a bent configuration with an SiOSi angle of 140–160° or a linear configuration with SiOSi equal to 180°. The value of the SiOSi angle has been determined by gas-phase electron diffraction for $O(SiH_3)_2$ (Almenningen, Bastiansen, Ewing, Hedberg & Trætteberg, 1963), for $O(SiF_3)_2$ (Airey, Glidewell, Rankin, Robiette, Sheldrick & Cruickshank, 1970) and for $O(SiCH_3)_2$ (Airey, Glidewell, Robiette & Sheldrick, 1971) to be 144·1, 155·7 and 146°, respectively. From low-temperature X-ray diffraction analyses of single crystals, the SiOSi angle has been found to have a value of 142·2° for $O(SiH_3)_2$ and 148·8° for $O[Si(CH_3)_3]_2$ (Barrow, Ebsworth & Harding, 1979). At room temperature, methylpentaphenyldisiloxane has an SiOSi angle of 158·9° (Voronkov, Nesterov, Gusev, Alexeev & Lotarev, 1981). The unexpected linear arrangement of SiOSi was observed for the first time in the crystal of $O[Si(C_6H_5)_3]_2$, where the O atom lies on a center of symmetry in a $P\bar{1}$ cell (Glidewell & Liles, 1978b). Subsequently, linear SiOSi angles were found in $O[Si(CH_2C_6H_5)_3]_2$ (Glidewell & Liles, 1981), $O[Si(CH=CH_2)_3]_2$ (Gusev, Antipin, Yufit, Struchkov, Sheludyakov, Zhun & Vlasenko, 1983) and possibly $O[Si(CH_2CH_2CF_3)_3]_2$, although a very high R factor precludes a definitive statement (Gusev, Nesterov, Alexeev, Rodzevich, Zverev, Ivanova & Sobolevskii, 1976).

The crystal structures of a number of hexaphenyl ethers of Group IVa elements ($Ph_3MOM'Ph_3$) were determined for comparison with the hexaphenyl ether of Si. For ethers of Ge and Sn where $M=M'$ (Glidewell & Liles, 1978a,c) and for mixed ethers of (Si,Ge), (Ge,Sn), and (Si,Sn) where $M \neq M'$ (Morosin & Harrah, 1981) the values for the MOM' angles were confined to the range of 135–144°, in sharp contrast to the value of 180° for the SiOSi angle in $Ph_3SiOSiPh_3$. However, for the

O[M(CH$_2$C$_6$H$_5$)$_3$]$_2$ compounds, where M = Ge or Sn, the MOM angles were found to be 180° (Glidewell & Liles, 1979a,b).

The present paper describes the structure of O{Si-[C(CH$_3$)$_3$](C$_6$H$_5$)$_2$}$_2$, only the second disiloxane for which a crystal structure has been reported in which all six R groups are not identical. The *tert*-butyl group is the most bulky substituent bonded to Si in disiloxane compounds that have been studied to date.

Experimental. A solution of 100 μl *tert*-butyldiphenyl-silyl trifluoroacetamide, 0·3 mg imidazole, and 200 μl dimethylformamide was heated overnight at 338 K. Upon cooling to room temperature, colorless plate-shaped crystals appeared. A suitable crystal was selected for X-ray analysis from the mother liquor. A solid-probe mass spectrum under conditions of chemical ionization with methane gas suggested a molecular weight of 494 with a small peak at m/e 495 (M + 1) and with fragments showing a loss of a methyl group (M − 15), loss of a *tert*-butyl group (M − 57), and loss of a phenyl group (M − 77, base peak). D_m not determined. Diffraction data collected from a crystal 0·18 × 0·25 × 0·02 mm in the θ–2θ mode to a 2θ value of 126° on the R3M Nicolet four-circle diffractometer with a graphite monochromator. Range of indices: h −12→12, k 0→16, l 0→20. Total number of independent reflections 4151. Standard reflections 0,0,10, 080, and 622 measured after every 60 intensities. Standards remained constant within 1·5%. Lattice parameters based on 14 centered reflections with 2θ values between 50 and 60°. No correction for absorption or extinction. Structure solved routinely by direct phase determination (Karle & Karle, 1966). The 38 H atoms were among the first 40 peaks in a difference map calculated after a least-squares refinement of the non-hydrogen atoms. Least-squares refinement performed using 3981 reflections with $|F_o| > 3\sigma(F_o)$. Coordinates for all atoms, including the 38 H atoms, were refined (on F) by a blocked cascade program in the *SHELXTL* system (Sheldrick, 1980). Anisotropic thermal parameters for the C, O and Si atoms and isotropic thermal parameters for H atoms were refined for a total of 468 parameters. Final R = 4·5% and wR = 4·8%; $w = 1/\sigma^2(F)$. Final difference electron density $|\rho| < 0·33$ e Å^{-3}; $(\Delta/\sigma)_{max}$ = 0·02; S = 1·624. Atomic scattering factors were those incorporated in *SHELXTL*.

Discussion. Coordinates and U_{eq} values are listed in Table 1 for the nonhydrogen atoms. Bond lengths and angles are shown in Table 2.*

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* Lists of structure factors, anisotropic temperature factors of the non-H atoms, coordinates of the H atoms, and selected torsional angles have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42465 (28 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Atom coordinates* ($\times 10^4$) *and thermal factors* ($\text{Å}^2 \times 10^3$)

$$U_{eq} = \tfrac{1}{3}\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Si(1)	7126 (1)	2262 (1)	−29 (1)	39 (1)
Si(2)	9497 (1)	2408 (1)	−868 (1)	37 (1)
O	8430 (1)	2136 (1)	−317 (1)	43 (1)
C(1)	6794 (2)	1221 (2)	607 (1)	49 (1)
C(2)	6986 (2)	289 (2)	186 (2)	70 (1)
C(3)	7593 (2)	1250 (2)	1312 (2)	67 (1)
C(4)	5575 (2)	1280 (2)	897 (2)	69 (1)
C(5)	7030 (2)	3370 (1)	545 (1)	43 (1)
C(6)	7934 (2)	3704 (2)	990 (1)	52 (1)
C(7)	7857 (2)	4522 (2)	1415 (1)	63 (1)
C(8)	6865 (2)	5025 (2)	1413 (1)	65 (1)
C(9)	5953 (2)	4715 (2)	984 (1)	65 (1)
C(10)	6031 (2)	3896 (2)	555 (1)	54 (1)
C(11)	6142 (2)	2387 (2)	−889 (1)	49 (1)
C(12)	6245 (2)	3186 (2)	−1346 (1)	60 (1)
C(13)	5567 (3)	3323 (2)	−1992 (2)	80 (1)
C(14)	4761 (3)	2666 (3)	−2196 (2)	95 (1)
C(15)	4639 (3)	1878 (3)	−1754 (2)	93 (1)
C(16)	5321 (2)	1739 (2)	−1114 (2)	70 (1)
C(17)	9686 (2)	1443 (2)	−1595 (1)	57 (1)
C(18)	9915 (3)	525 (2)	−1153 (2)	86 (1)
C(19)	10702 (3)	1651 (2)	−2106 (2)	91 (1)
C(20)	8608 (3)	1300 (3)	−2087 (2)	84 (1)
C(21)	9254 (2)	3594 (1)	−1305 (1)	43 (1)
C(22)	9080 (2)	3784 (2)	−2083 (1)	65 (1)
C(23)	8849 (3)	4682 (2)	−2349 (1)	78 (1)
C(24)	8793 (2)	5412 (2)	−1846 (2)	70 (1)
C(25)	8961 (2)	5257 (2)	−1077 (2)	66 (1)
C(26)	9198 (2)	4360 (2)	−812 (1)	53 (1)
C(27)	10787 (2)	2475 (1)	−229 (1)	42 (1)
C(28)	10847 (2)	2045 (2)	489 (1)	56 (1)
C(29)	11828 (2)	2082 (2)	939 (2)	73 (1)
C(30)	12765 (2)	2532 (2)	683 (2)	77 (1)
C(31)	12739 (2)	2947 (2)	−28 (2)	80 (1)
C(32)	11761 (2)	2928 (2)	−473 (1)	63 (1)

The principal focus of this investigation was the determination of the SiOSi geometry for the reasons presented in the *Introduction*. In the present molecule the SiOSi angle has a value of 152·4 (1)° (see Fig. 1).

Gibbs (1982) applied computational quantum chemistry to the disiloxy group in silica polymorphs in which the SiOSi angle also displays a broad continuum of values. He found that the barrier to linearity is small, about 3 kT at room temperature, which indicates that a relatively small amount of energy is expended in deforming the SiOSi angle even to 180° from its minimum value of about 140°.

The SiO distances in the present molecule, 1·638 (1) and 1·633 (1) Å, have values larger than the 1·613 (4) Å observed in the linear disiloxane molecules. Fig. 2 shows the values of SiO distances, along with the corresponding SiOSi angles, that have been determined for seven disiloxane molecules in the crystalline state at various temperatures, and, in addition (for the point at the extreme left), values for hexaphenylcyclotrisiloxane (Tomlins, Lydon, Akrigg & Sheldrick, 1985). The least-squares line calculated through the points, for which weights inversely proportional to the standard deviations were used, is represented by $r = 1\cdot6957 - 0\cdot000455\theta$ (θ in degrees). The weighted sum of least-squares deviations from this line is 10^3 smaller than from a weighted line with zero slope. There appears to be a trend toward a decrease in the Si—O

distance with increasing values for the SiOSi angle. A similar trend is consistent for O[*M*Ph$_3$]$_2$ and O[*M*CH$_2$-Ph$_3$]$_2$, where M = Ge and Sn, for the limited number of such compounds studied.

The overall geometry of the present molecule is similar to that of O[SiPh$_3$]$_2$, even though the SiOSi angle is bent and two phenyl groups have been replaced by *tert*-butyl groups. For comparative purposes Fig. 3 is drawn in an orientation as near as possible to that of O[SiPh$_3$]$_2$ in Fig. 1 of Glidewell & Liles (1978*b*). The four phenyl groups common to both molecules are rotated similarly. It is interesting to note that the two butyl groups are not in the *trans* position with respect to the Si···Si direction, but assume the *gauche* conformation. The plane of the SiOSi moiety is not between the planes containing the Si(2)···Si(1)C(1) and Si(1)···Si(2)C(17) atoms where C(1) and C(17) are the central atoms of the *tert*-butyl groups, but rather between the planes containing Si(2)···Si(1)C(1) and Si(1)···Si(2)C(27). The dihedral angle between planes SiOSi and Si···SiC(1) is only 13·9°. All the bond angles about the Si atom in O[SiPh$_3$]$_2$ have values

Table 2. Bond lengths (Å) and angles (°)

Si(1)–O	1·633 (1)	Si(1)–C(1)	1·895 (2)
Si(1)–C(5)	1·873 (2)	Si(1)–C(11)	1·884 (2)
Si(2)–O	1·638 (1)	Si(2)–C(17)	1·883 (2)
Si(2)–C(21)	1·874 (2)	Si(2)–C(27)	1·869 (2)
C(1)–C(2)	1·536 (3)	C(1)–C(3)	1·533 (3)
C(1)–C(4)	1·531 (3)	C(5)–C(6)	1·390 (3)
C(5)–C(10)	1·396 (3)	C(6)–C(7)	1·384 (3)
C(7)–C(8)	1·371 (4)	C(8)–C(9)	1·370 (4)
C(9)–C(10)	1·389 (3)	C(11)–C(12)	1·394 (3)
C(11)–C(16)	1·389 (3)	C(12)–C(13)	1·381 (4)
C(13)–C(14)	1·376 (5)	C(14)–C(15)	1·369 (5)
C(15)–C(16)	1·376 (4)	C(17)–C(18)	1·540 (3)
C(17)–C(19)	1·528 (4)	C(17)–C(20)	1·533 (5)
C(21)–C(22)	1·389 (3)	C(21)–C(26)	1·390 (3)
C(22)–C(23)	1·386 (4)	C(23)–C(24)	1·361 (4)
C(24)–C(25)	1·365 (4)	C(25)–C(26)	1·385 (3)
C(27)–C(28)	1·390 (3)	C(27)–C(32)	1·389 (3)
C(28)–C(29)	1·385 (4)	C(29)–C(30)	1·358 (4)
C(30)–C(31)	1·369 (4)	C(31)–C(32)	1·378 (4)
O–Si(1)–C(1)	107·2 (1)	O–Si(1)–C(5)	108·7 (1)
C(1)–Si(1)–C(5)	109·6 (1)	O–Si(1)–C(11)	109·8 (1)
C(1)–Si(1)–C(11)	113·9 (1)	C(5)–Si(1)–C(11)	107·5 (1)
O–Si(2)–C(17)	108·6 (1)	O–Si(2)–C(21)	109·7 (1)
C(17)–Si(2)–C(21)	114·0 (1)	O–Si(2)–C(27)	106·9 (1)
C(17)–Si(2)–C(27)	109·3 (1)	C(21)–Si(2)–C(27)	108·1 (1)
Si(1)–O–Si(2)	152·4 (1)	Si(1)–C(1)–C(2)	111·5 (2)
Si(1)–C(1)–C(3)	108·3 (1)	C(2)–C(1)–C(3)	108·0 (2)
Si(1)–C(1)–C(4)	110·6 (2)	C(2)–C(1)–C(4)	110·5 (2)
C(3)–C(1)–C(4)	107·7 (2)	Si(1)–C(5)–C(6)	122·2 (2)
Si(1)–C(5)–C(10)	121·2 (2)	C(6)–C(5)–C(10)	116·6 (2)
C(5)–C(6)–C(7)	121·9 (2)	C(6)–C(7)–C(8)	120·1 (2)
C(7)–C(8)–C(9)	119·6 (2)	C(8)–C(9)–C(10)	120·3 (2)
C(5)–C(10)–C(9)	121·4 (2)	Si(1)–C(11)–C(12)	118·0 (2)
Si(1)–C(11)–C(16)	125·2 (2)	C(12)–C(11)–C(16)	116·8 (2)
C(11)–C(12)–C(13)	121·6 (2)	C(12)–C(13)–C(14)	120·0 (3)
C(13)–C(14)–C(15)	119·5 (3)	C(14)–C(15)–C(16)	120·3 (3)
C(11)–C(16)–C(15)	121·8 (3)	Si(2)–C(17)–C(18)	108·0 (2)
Si(2)–C(17)–C(19)	110·4 (2)	C(18)–C(17)–C(19)	108·8 (2)
Si(2)–C(17)–C(20)	111·4 (2)	C(18)–C(17)–C(20)	107·6 (2)
C(19)–C(17)–C(20)	110·7 (2)	Si(2)–C(21)–C(22)	126·1 (2)
Si(2)–C(21)–C(26)	117·9 (2)	C(22)–C(21)–C(26)	115·9 (2)
C(21)–C(22)–C(23)	122·0 (2)	C(22)–C(23)–C(24)	120·3 (2)
C(23)–C(24)–C(25)	119·7 (2)	C(24)–C(25)–C(26)	120·0 (2)
C(21)–C(26)–C(25)	122·1 (2)	Si(2)–C(27)–C(28)	122·6 (2)
Si(2)–C(27)–C(32)	120·8 (2)	C(28)–C(27)–C(32)	116·5 (2)
C(27)–C(28)–C(29)	121·3 (2)	C(28)–C(29)–C(30)	120·6 (2)
C(29)–C(30)–C(31)	119·5 (3)	C(30)–C(31)–C(32)	120·2 (3)
C(27)–C(32)–C(31)	121·8 (2)		

Fig. 2. Relationship of the Si–O distance to the SiOSi angle in disiloxanes in the crystalline state. The vertical bars represent one standard deviation (+ and –) for the distances while the horizontal bars represent one standard deviation for the angles. (*a*) Hexaphenylcyclotrisiloxane (Tomlins *et al.*, 1985); (*b*) O(SiH$_3$)$_2$ (Barrow *et al.*, 1979); (*c*) O[Si(CH$_3$)$_3$]$_2$ (Barrow *et al.*, 1979); (*d*) O{Si[C(CH$_3$)$_3$](C$_6$H$_5$)$_2$}$_2$ (present paper); (*e*) methyl-pentaphenyldisiloxane (Voronkov *et al.*, 1981); (*f*) O(SiPh$_3$)$_2$ (Glidewell & Liles, 1978*b*); (*g*) O[Si(CH=CH$_2$)$_3$]$_2$ (Gusev *et al.*, 1983); (*h*) O[Si(CH$_2$Ph)$_3$]$_2$ (Glidewell & Liles, 1981).
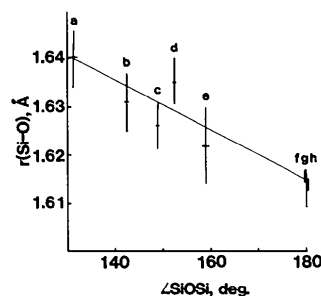

Fig. 1. Stereodiagram of [*t*-BuPh$_2$]SiOSi[*t*-BuPh$_2$]. The ellipsoids are represented at the 50% probability level.
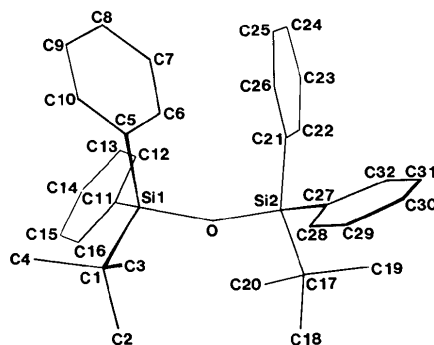

Fig. 3. The labeling of atoms on a stick diagram drawn with the experimentally determined coordinates.

between 108·4 and 110·1°. In the present molecule, the spread of values is somewhat larger, 106·9 to 114·0°, with the two highest values associated with the two butyl groups, i.e. C(1)Si(1)C(11) 113·9 (1) and C(17)-Si(2)C(21) 114·0 (1)°. The nearest intramolecular nonbonded approaches are between substituents on the same Si atom, e.g. C(10)···C(11) 3·31, C(5)···C(13) 3·36, C(18)···C(27) 3·36 and C(21)···C(32) 3·40 Å. C atoms of substituents on the different Si atoms are not as closely spaced. The minimum distances are between phenyl groups, C(6)···C(26) at 3·59 and C(12)···C(21) at 3·61 Å. All other C···C distances between moieties on the separate Si atoms are greater than 3·69 Å. The closest approach between different molecules in the cell is C(20)···C(24)(1·5 − x, 0·5 + y, −0·5 − z) at 3·59 Å.

The phenyl groups are distorted from perfect hexagons in the same manner as has been already observed in other Si compounds, e.g. in O[SiPh$_3$]$_2$ and in [OSiPh$_2$]$_3$ (Tomlins et al., 1985). The interior angle of the four phenyl groups at the C—Si bond has an average value of 116·5 (2)° in the present molecule. The two adjacent interior angles average to 121·7 (2)°, whereas the remaining angles are near 120°.

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Perhexiline [2-(2,2-Dicyclohexylethyl)piperidine] Maleate

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Abstract. Perhexiline maleate, $C_{19}H_{36}N^+.C_4H_3O_4^-$, is a vasodilator and calcium-blocking agent. $M_r = 393·6$, $P2_1/a$, $a = 18·354$ (2), $b = 19·659$ (2), $c = 6·247$ (1) Å, $\beta = 90·96$ (1)°, $V = 2253·7$ (4) Å^3, $Z = 4$, $D_x = 1·16$ g cm^{-3}, Cu $K\alpha$, $\lambda = 1·5418$ Å, $\mu = 5·432$ cm^{-1}, $F(000) = 864$, room temperature, final $R = 0·053$ for 3678 observed intensities and 409 parameters. The maleate group is planar with an internal hydrogen bond between the two carboxyl groups. Each of the three six-membered rings of perhexiline maleate is in the chair conformation, but with no apparent overall symmetry to the molecule. The two saturated cyclohexane rings provide a strongly hydrophobic area and the positively charged —NH$_2^+$— group forms two hydrogen bonds to maleate oxygen atoms 4·49 Å apart. This suggests that similar hydrogen-bond acceptors may occur in the biological perhexiline receptor.

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