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3,3-Dimethyl-1,1,1,5,5,5-hexaphenyltrisiloxane

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Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.004 Å
R factor = 0.047
wR factor = 0.112
Data-to-parameter ratio = 19.4

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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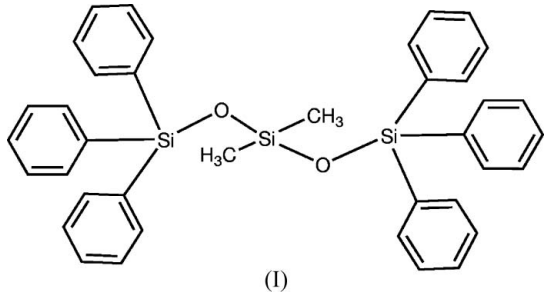
The title compound, $C_{38}H_{36}O_2Si_3$, crystallizes with one crystallographically independent molecule in a general position and one molecule in a special position with twofold rotation symmetry. The approximate twofold rotation axis of the molecule in the general position is also approximately parallel to the monoclinic b axis. The conformations of the two independent molecules are similar.

Comment

The symmetrical title compound, (I), was obtained unintentially in an attempt to synthesize the unsymmetrical compound 1,1-dimethyl-3,3,3-triphenyl-1-(2-pyridyl)disiloxane (*i.e.* with one of the two triphenylsiloxy groups substituted by a covalently bonded pyridyl group).



(I)

The unit cell contains eight equivalent molecules of (I) in general positions and four equivalent molecules located in positions with imposed crystallographic twofold rotation symmetry (Fig. 1). The two independent molecules have very similar conformations as illustrated by the quaternion fit (Mackay, 1984; Spek 2003) plot shown in Fig. 2. The approximate molecular twofold axes are approximately parallel to the monoclinic b axis.

The Si—O—Si angles range from 151.51 (10) to 164.17 (10)° (Table 1). Similar values in the range 160–163° have been reported by Graalmann *et al.* (1984) in the related siloxane structure 1,1,5,5-tetra-*tert*-butyl-1,5-dihydroxy-3,3-dimethyl-1,3,5-trisiloxane.

Molecules are stacked in planes parallel to ($\bar{1}$02) with an interplanar distance of approximately 8.3 Å (Fig. 3). Each layer contains the two types of molecules (general and special positions) in a 2:1 ratio.

Experimental

The crystal used for this study was obtained in low yield (<10%) from dimethylmethoxy(2-pyridyl)silane and triphenylsilanol. A subsequently optimized synthesis is described. Ph_3SiOH (0.915 g,

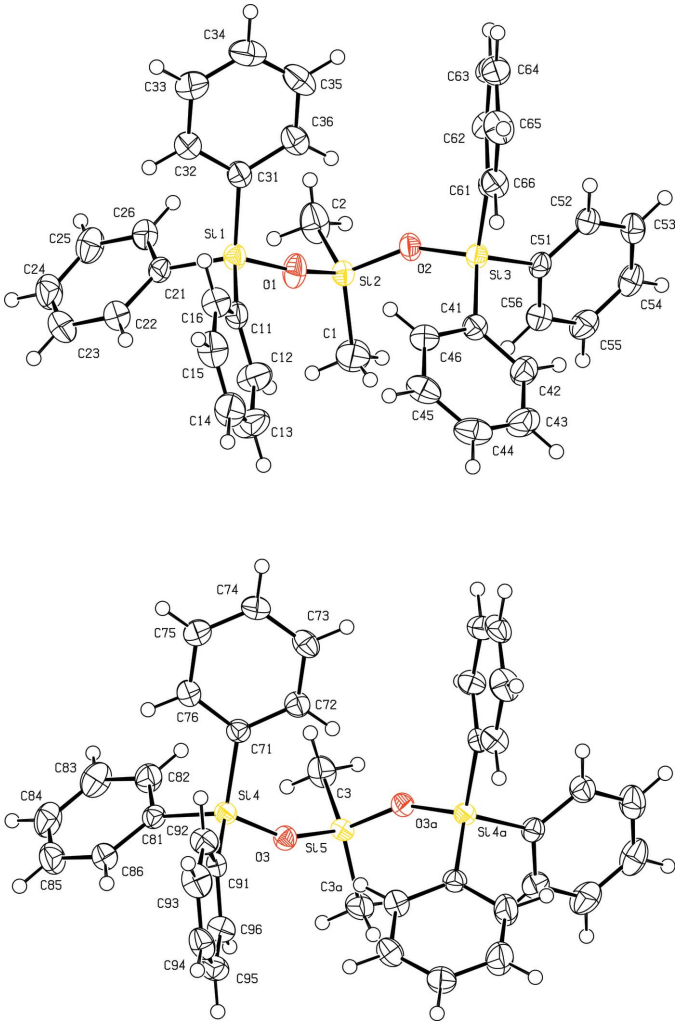


Figure 1
Views of the two independent molecules of the title compound with the atom-numbering schemes. Displacement ellipsoids for non-H atoms are drawn at the 50% probability level. [Symmetry code: (*a*) $-x, y, \frac{1}{2}-z$.]

3.3 mmol) in tetrahydrofuran (20 ml) was treated with $SiMe_2Cl_2$ (0.21 g, 1.65 mmol) in the presence of pyridine (1 ml). The reaction mixture was dried *in vacuo* and the residue was extracted with hexanes (20 ml). After concentration of the extract, pure colorless crystals of the title compund were formed (0.51 g, 0.84 mmol, 51%).

Crystal data

$C_{38}H_{36}O_2Si_3$
M_r = 608.94
Monoclinic, $C2/c$
a = 47.7051 (5) Å
b = 7.6270 (1) Å
c = 33.1200 (5) Å
β = 124.1246 (6)°
V = 9975.7 (2) Å^3
Z = 12
D_x = 1.216 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 11325 reflections
θ = 1.0–27.5°
μ = 0.18 mm^{-1}
T = 150 K
Plate, colorless
0.30 × 0.27 × 0.09 mm

Data collection

Nonius KappaCCD diffractometer
ω and φ scans
Absorption correction: none
43681 measured reflections
11325 independent reflections
7490 reflections with $I > 2\sigma(I)$
R_{int} = 0.066
θ_{max} = 27.4°
$h = -60 \rightarrow 61$
$k = -9 \rightarrow 9$
$l = -40 \rightarrow 42$

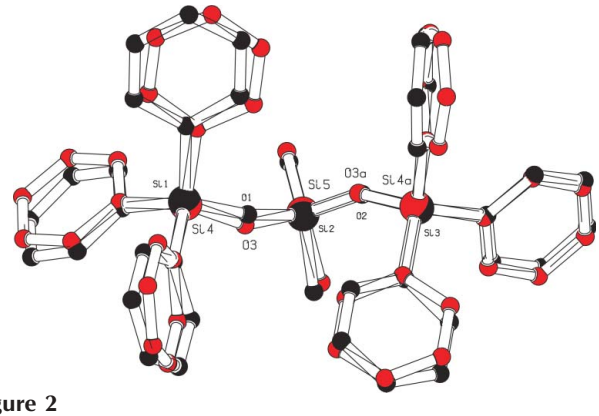


Figure 2
Quaternion fit of the two crystallographically independent molecules, illustrating their conformational similarity. The molecule with exact rotation symmetry is shown in red. H atoms have been omitted.

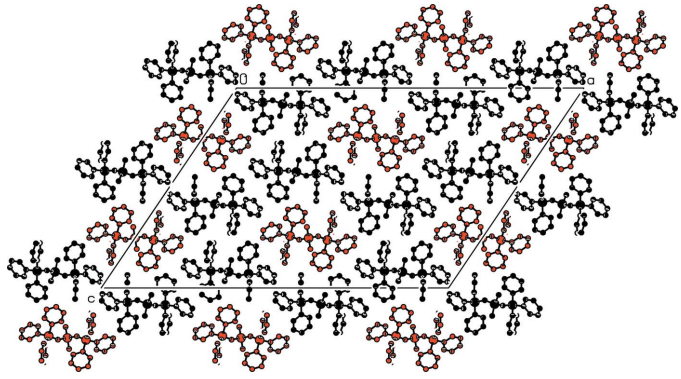


Figure 3
Molecular packing plot, projected down the *b* axis. The molecules on special positions are in red. H atoms have been omitted.

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.112
S = 1.02
11325 reflections
585 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0388P)^2 + 6.4055P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.36 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Si1—O1	1.619 (2)	Si3—O2	1.6312 (17)
Si2—O1	1.611 (2)	Si4—O3	1.6268 (17)
Si2—O2	1.6209 (17)	Si5—O3	1.6303 (16)
Si2—C1	1.834 (3)	Si5—C3	1.834 (2)
Si2—C2	1.831 (3)		
O1—Si2—O2	107.83 (9)	C3—Si5—C3^{i}	112.76 (11)
O1—Si2—C1	108.35 (12)	O3—Si5—O3^{i}	108.13 (8)
O1—Si2—C2	109.76 (12)	O3—Si5—C3	109.67 (11)
O2—Si2—C1	108.65 (13)	O3^{i}—Si5—C3^{i}	109.67 (11)
O2—Si2—C2	109.02 (11)	Si1—O1—Si2	164.17 (13)
C1—Si2—C2	113.09 (13)	Si2—O2—Si3	151.51 (10)
O3—Si5—C3^{i}	108.25 (10)	Si4—O3—Si5	153.62 (10)
O3^{i}—Si5—C3	108.25 (10)		

Symmetry code: (i) $-x, y, -z+\frac{1}{2}$.

H-atoms were placed in calculated positions, with C—H distances 0.95 (aromatic) and 0.98 Å (CH_3), and with U_{iso}(H) = 1.2U_{eq}(C), and refined as riding, with a rigid rotator model for CH_3.

Data collection: *COLLECT* (Hooft, 1998); cell refinement: *DENZO* (Otwinowski & Minor, 1997); data reduction: *DENZO*; program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *PLATON*.

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