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1-Hydroxy-2,5-bis(triisopropylsilyl)silole

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Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.004 Å
H-atom completeness 98%
Disorder in main residue
R factor = 0.053
wR factor = 0.147
Data-to-parameter ratio = 19.8

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

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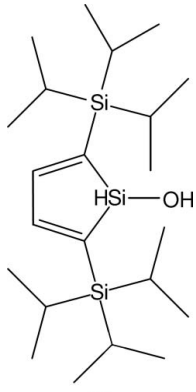
The silole ring of the title compound, $C_{22}H_{46}OSi_3$, is planar. Geometric parameters do not show unusual values. The H atom and the hydroxy group on the silole Si atom are mutually disordered.

Comment

A perspective view of the the title compound, (I), is shown in Fig. 1. Bond lengths and angles can be regarded as normal (Cambridge Structural Database, Version 5.27 plus one update; *MOGUL* Version 1.1; Allen, 2002). The silole ring is planar (r.m.s. deviation 0.003 Å) and the Si atoms of the two triisopropylsilyl residues do not deviate significantly from this plane [0.003 (3) and −0.008 (3) Å for Si1 and Si3, respectively].



(I)

The crystal packing (Fig. 2) shows centrosymmetric dimers of (I) and poses the conjecture that two molecules are connected *via* O—H···O hydrogen bonds. The O···O distance between O2 and O2′(1 − *x*, 1 − *y*, −*z*) is 2.746 (6) Å

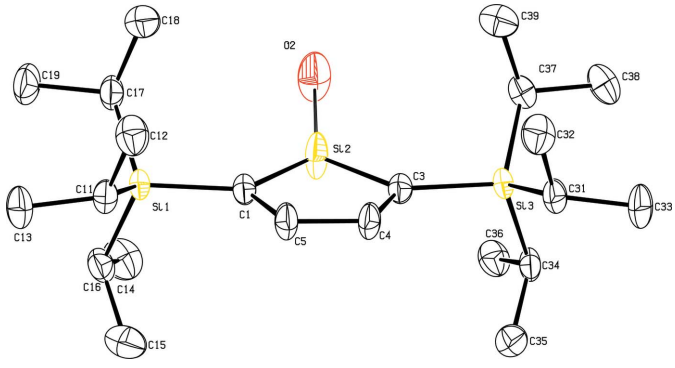


Figure 1
Perspective view of the title compound with the atom numbering; displacement ellipsoids are drawn at the 50% probability level and H atoms have been omitted for clarity. Only the major occupancy component of the disordered O atom is shown.

(O2′ is the minor disorder component of O2), but only one of these two can carry an H atom which is pointing to a neighbouring O atom. Since the hydroxy H atom was not visible in a difference map and since no other hydrogen-bond acceptor could be found, this H atom was omitted from the refinement.

Experimental

The title compound 1-hydroxy-2,5-bis(triisopropylsilyl)silole, (I), was obtained in small quantities as the only identified product of the reaction of 1,4-dilithio-1,4-bis(triisopropylsilyl)butadiene with trimethoxysilane. In this reaction, *t*-BuLi (4.72 ml, 8.018 mmol) was added to a solution of 1,4-diiodo-1,4-bis(triisopropylsilyl)butadiene (1.24 g, 2.005 mmol) in ether (50 ml) at 183 K. After stirring for 1 h at room temperature, the reaction mixture was cooled to 193 K and (0.26 ml, 2.005 mmol) trimethoxysilane was added. This reaction mixture was slowly warmed up. Aqueous work-up and column chromatography (silica, methylene chloride) gave a colourless oil. Colourless crystals of (I) were obtained from a solution of this compound in hexane at 253 K.

Crystal data

$C_{22}H_{46}OSi_3$
$M_r = 410.86$
Triclinic, $P\overline{1}$
$a = 7.8597$ (8) Å
$b = 13.5714$ (14) Å
$c = 13.6482$ (14) Å
$\alpha = 110.596$ (8)°
$\beta = 98.999$ (9)°
$\gamma = 100.074$ (8)°
$V = 1303.7$ (3) Å^3
$Z = 2$
$D_x = 1.047$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 13100 reflections
$\theta = 3.5$–25.7°
$\mu = 0.19$ mm^{-1}
$T = 173$ (2) K
Rod, colourless
0.40 × 0.20 × 0.10 mm

Data collection

Stoe IPDS-II two-circle diffractometer
ω scans
Absorption correction: multi-scan (*MULABS*; Spek, 2003; Blessing, 1995)
$T_{min} = 0.928$, $T_{max} = 0.971$
13100 measured reflections
4844 independent reflections
4178 reflections with $I > 2\sigma(I)$
$R_{int} = 0.078$
$\theta_{max} = 25.7°$
$h = -9 \rightarrow 9$
$k = -16 \rightarrow 14$
$l = -16 \rightarrow 16$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.053$
$wR(F^2) = 0.147$
$S = 1.03$
4844 reflections
245 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.083P)^2 + 0.5074P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.40$ e Å^{-3}
$\Delta\rho_{min} = -0.52$ e Å^{-3}

Table 1
Selected bond lengths (Å).

C1—C5	1.353 (3)	C3—C4	1.354 (3)
C1—Si1	1.873 (2)	C3—Si3	1.873 (2)
C1—Si2	1.877 (2)	C4—C5	1.493 (3)
Si2—C3	1.874 (2)		

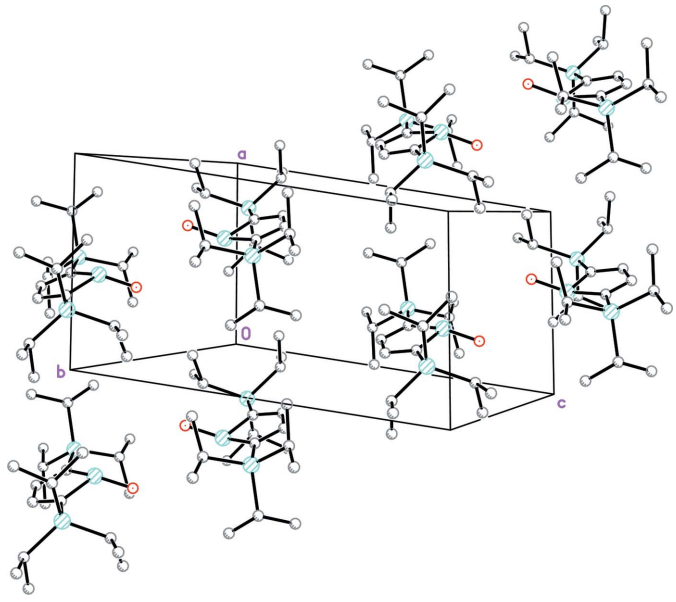


Figure 2
Packing diagram of the title compound; only the major occupancy component of the disordered O atom is shown.

H atoms bonded to C atoms were located in a difference electron-density map, but they, and the one bonded to Si, were geometrically positioned and refined with fixed individual displacement parameters [U_{iso}(H) = 1.2U_{eq}(C,Si) or 1.5U_{eq}(methyl C)] using a riding model with C—H = 0.95–0.99 Å and Si—H = 1.40 Å. The hydroxy group and the H atom bonded to the silole Si are mutually disordered. The site-occupation factors for the two positions refined to 0.537 (5) and 0.463 (5). The hydroxy H atom could not be found and therefore was not included in the refinement.

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97* and *PLATON*.

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