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1,3-Dimethyl-1,3-bis(2,3,4-triphenyl-1-naphthyl)disiloxane hexane solvate

Thomas Müller,[a] Rita Meyer[a] and Michael Bolte[b]*

[a]Institut für Anorganische Chemie, J.-W.-Goethe-Universität Frankfurt, Marie-Curie-Straße 11, 60439 Frankfurt/Main, Germany, and [b]Institut für Organische Chemie, J.-W.-Goethe-Universität Frankfurt, Marie-Curie-Straße 11, 60439 Frankfurt/Main, Germany

Correspondence e-mail: bolte@chemie.uni-frankfurt.de

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The title compound, $C_{58}H_{46}OSi_2{\cdot}C_6H_{14}$, was formed in small yield as a by-product during the synthesis of 1-methyl-2,3,4,5-tetraphenylsilole.

Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.009 Å
Some non-H atoms missing
Disorder in solvent or counterion
R factor = 0.134
wR factor = 0.363
Data-to-parameter ratio = 26.1

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

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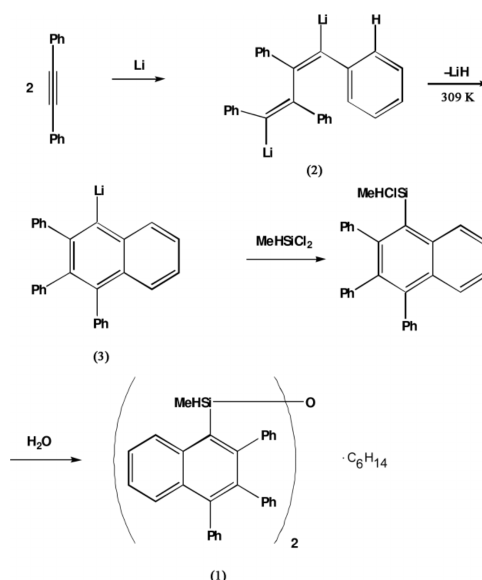
Comment

The title compound, (1), was formed in small yields (5–10%) as a by-product during the synthesis of 1-methyl-2,3,4,5-tetraphenylsilole (Schuppan, 1998; Schuppan *et al.*, 2001). A possible reaction path for the formation of (1) is shown in the scheme below.



The key step for its formation is LiH elimination from 1,4-dilithio-1,2,3,4-tetraphenylbutadiene, (2), at elevated temperature, yielding 1-lithio-2,3,4-triphenylnaphthalene, (3). Subsequent reaction with methyldichlorosilane and hydrolysis results in the formation of (1). Separation from the major product was achieved by column chromatography (hexanes–ethyl acetate = 20:1; SiO_2). Recrystallization from toluene gives colourless crystals suitable for X-ray crystallography. A search of the Cambridge Structural Database (CSD, Version 5.24 with two updates, April 2003; Allen, 2002) finds 438 structures containing the Si—O—Si fragment with four-coordinated Si and two-coordinated O atoms. Only 13 of these show an Si—O—Si angle less than 133°. This means that the angle observed in (1), 132.9 (3)°, is at the lower end of the range. The two naphthalene moieties enclose an angle of 27.07 (17)°. The dihedral angles between the naphthalene moiety C1–C10 and the attached phenyl rings are 70.69 (16), 69.31 (16) and 80.68 (15)° for C21–C26, C31–C36 and C41–

C46, respectively. For the second naphthalene moiety (C11–C20), the following dihedral angles were found: 83.39 (13)° for C51–C56, 74.53 (15)° for C61–C66, and 64.2 (2)° for C71–C76.

Experimental

Spectroscopic data: ^{1}H NMR [250 MHz, acetone [D_6], 300 K, δ(CD_3COCD_2H) = 2.1]: δ 0.38 [*d*, ^{3}J(HH) = 3.5 Hz, 6H], 4.28 [*q*, ^{3}J(HH) = 3.5 Hz, 2H], 6.4 (*m*, 2H), 6.55–7.35 (*m*, 32H), 7.50 (*m*, 2H), 8.25 (*m*, 2H); ^{13}C NMR [62.9 MHz, $CDCl_3$, 300 K, δ($CDCl_3$) = 77.0]: δ 1.3 (CH_3), 125.2 (CH), 125.6 (CH), 125.7 (CH), 126.2 (CH), 126.4 (CH), 126.5 (CH), 126.8 (CH), 126.9 (CH), 127.3 (CH), 127.6 (CH), 127.8 (CH), 128.0 (CH), 128.6 (CH), 130.3 (CH), 130.8 (CH), 130.8 (CH), 131.1 (CH), 131.3 (CH), 131.3 (CH), 131.8 (C^q), 133.5 (C^q), 136.2 (C^q), 138.2 (C^q), 139.6 (C^q), 140.2 (C^q), 140.6 (C^q), 142.3 (C^q), 147.5 (C^q); ^{29}Si NMR [79.5 MHz, $CDCl_3$, 300 K, δ(TMS) = 0.0]: δ 14.5.

Crystal data

$C_{58}H_{46}OSi_2 \cdot C_6H_{14}$
M_r = 901.30
Monoclinic, $P2_1/c$
a = 18.716 (4) Å
b = 15.533 (3) Å
c = 17.685 (4) Å
β = 96.67 (2)°
V = 5106.5 (19) Å^3
Z = 4
D_x = 1.172 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3857 reflections
θ = 1–25°
μ = 0.11 mm^{-1}
T = 173 (2) K
Plate, colourless
0.30 × 0.30 × 0.08 mm

Data collection

Siemens SMART CCD three-circle diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.967, T_{max} = 0.991
77354 measured reflections
14554 independent reflections
4271 reflections with $I > 2\sigma(I)$
R_{int} = 0.126
θ_{max} = 30.7°
$h = -25 \rightarrow 25$
$k = -21 \rightarrow 21$
$l = -24 \rightarrow 24$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.134
$wR(F^2)$ = 0.363
S = 0.97
14554 reflections
558 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.146P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ < 0.001
$\Delta\rho_{max}$ = 0.50 e Å^{-3}
$\Delta\rho_{min}$ = −0.37 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Si1—O1	1.644 (4)	O1—Si2	1.637 (4)
Si2—O1—Si1	132.9 (3)		

All H atoms were located by difference Fourier synthesis and refined with fixed individual displacement parameters [U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(C_{methyl})] using a riding model with aromatic C—H = 0.95 Å and methyl C—H = 0.98 Å. The H atoms bonded to Si were refined isotropically with an Si—H distance restrained to 1.45 (6) Å. The hexane molecule is severely disordered. The contribution of the disordered solvent to the calculated structure factors was taken into account with the *BYPASS* algorithm (van der Sluis & Spek, 1990), implemented as the SQUEEZE option in *PLATON* (Spek, 2003). The final F_o^2/F_c^2 data were calculated with the FCF routine of *PLATON* and include the disordered solvent contribution. The inclusion of such a large percentage of weak data in the refinement is probably responsible for the high R factors. This, in turn, affects the precision of the results. The crystal did not appear to be twinned.

Data collection: *SMART* (Siemens, 1995); cell refinement: *SMART*; data reduction: *SAINT* (Siemens, 1995); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XP* in *SHELXTL-Plus* (Sheldrick, 1991); software used to prepare material for publication: *PLATON* (Spek, 2003).

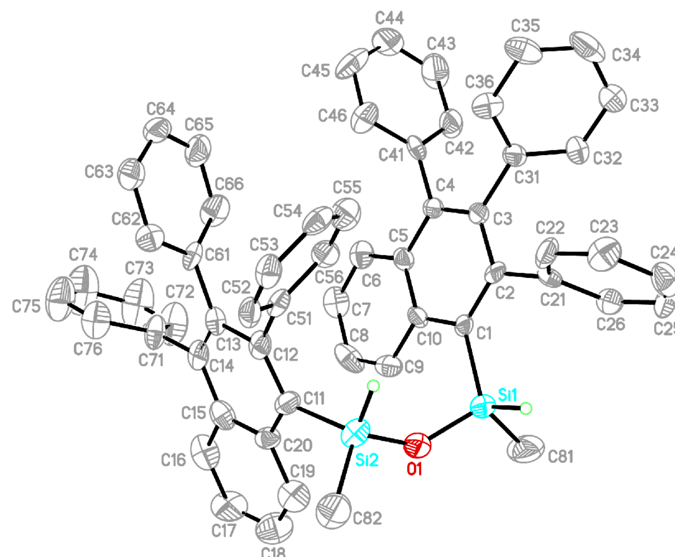


Figure 1
A perspective view of the title compound with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

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