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Key indicators

Single-crystal X-ray study
 $T = 173$ K
Mean $\sigma(\text{C}-\text{C}) = 0.004$ Å
 R factor = 0.045
 wR factor = 0.117
Data-to-parameter ratio = 15.6For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.1,1-Bis(*tert*-butylamido)-3,3,5,5-tetraphenylcyclo-trisiloxane

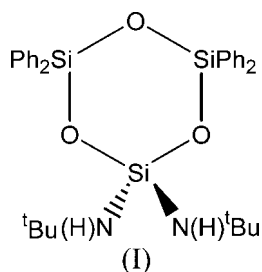
The title compound, $\text{C}_{32}\text{H}_{40}\text{N}_2\text{O}_3\text{Si}_3$ or $(\text{Ph}_2\text{SiO})_2\text{OSi}(\text{NH}^t\text{Bu})_2$, was synthesized by the *in situ* reaction of $[\text{Ph}_2\text{Si}(\text{OH})]_2\text{O}$ with two equivalents of $^t\text{BuLi}$, followed by the addition of $\text{Cl}_2\text{Si}(\text{NH}^t\text{Bu})_2$. The addition of *tert*-butylamide substituents to one of the Si centres causes a slight distortion of the planar cyclo-trisiloxane ring.

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Comment

A successful route to the preparation of polysiloxanes is the ring-opening polymerization of cyclotrisiloxanes. The relief of ring strain in the six-membered ring is thought to be the driving force for this reaction. This method has produced high molecular weight polymers, with a low molecular-weight distribution and few cyclic by-products (Beckmann *et al.*, 2002). Here, we report the synthesis and structural characterization of a new amido-substituted cyclotrisiloxane, $(\text{Ph}_2\text{SiO})_2\text{OSi}(\text{NH}^t\text{Bu})_2$, (I) (Fig. 1).



Complex (I) contains a six-membered Si_3O_3 ring. The Si centres all display distorted tetrahedral geometry. Two of the Si centres are additionally bonded to two phenyl rings, while the third, atom Si3, has two *tert*-butylamido substituents. The Si—O bond lengths fall in the narrow range 1.635 (2)–1.652 (2) Å, similar to the corresponding distances in the hexaphenylcyclo-trisiloxane structure, $(\text{Ph}_2\text{SiO})_3$ (Ziemer *et al.*, 1998). The Si_3O_3 ring in (I) is slightly distorted from planarity, with the greatest deviations being 0.186 and 0.139 Å for atoms O3 and Si3, respectively. The related *tert*-butoxy-substituted cyclotrisiloxane, $(\text{Ph}_2\text{SiO})_2\text{OSi}(\text{O}^t\text{Bu})_2$, has been reported (Beckmann *et al.*, 2002) and also shows a slight distortion from planarity in the central six-membered ring.

Experimental

To a solution of $[\text{Ph}_2\text{Si}(\text{OH})]_2\text{O}$ (1.21 g, 2.9 mmol) in tetrahydrofuran (20 ml), $^t\text{BuLi}$ (2.33 ml, 5.8 mmol; 2.5 M solution in *n*-hexanes) was added at 273 K and the mixture was stirred for 2 h. A solution of $\text{Cl}_2\text{Si}(\text{NH}^t\text{Bu})_2$ (0.71 g, 2.9 mmol) in diethyl ether (15 ml) was added

dropwise at 273 K. The cloudy reaction mixture turned to a clear colourless solution. The solution was allowed to warm to room temperature and was then stirred for 4 h. The solvent was removed under reduced pressure, producing a sticky residue. The product was extracted with diethyl ether (20 ml) and hexane was added (3 ml). Storage of the solution at 263 K yielded colourless blocks of (I) (1.2 g, 72%; m.p.: 393–395 K). Analysis, calculated for $C_{32}H_{40}Si_3N_2O_3$: C 65.70, H 6.89, N 4.78%; found: C 65.31, H 6.97, N 4.67%. Spectroscopic analysis: 1H NMR (300 MHz, 298 K, toluene- d_8 , δ , p.p.m.): 7.87, 7.18 (*m*, 20 H, C_6H_5), 2.12 (*s*, 2 H, NH), 1.17 [*s*, 18 H, $C(CH_3)_3$]; ^{13}C NMR (300 MHz, 298 K, THF- d_8 , δ , p.p.m.): 136.1–125.3 (C_6H_5), 54.4 [$C(CH_3)_3$], 38.9, [$C(CH_3)_3$]; ^{29}Si NMR (300 MHz, 298 K, toluene- d_8 , δ , p.p.m.): –34.6, –66.6 [*s*, $SiPh_2$, $Si(NH-tBu)_2$].

Crystal data

$C_{32}H_{40}N_2O_3Si_3$ $Z = 4$
 $M_r = 584.93$ $D_x = 1.199$ Mg m $^{-3}$
 Monoclinic, $P2_1/n$ Mo $K\alpha$ radiation
 $a = 10.246$ (2) Å $\mu = 0.18$ mm $^{-1}$
 $b = 15.759$ (3) Å $T = 173$ (2) K
 $c = 20.087$ (4) Å Block, colourless
 $\beta = 92.94$ (3)° $0.20 \times 0.20 \times 0.10$ mm
 $V = 3239.1$ (11) Å 3

Data collection

Nonius KappaCCD area-detector diffractometer 10563 measured reflections
 φ and ω scans 5714 independent reflections
 Absorption correction: multi-scan (SCALEPACK; Otwinowski & Minor, 1997) 4160 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.040$
 $T_{min} = 0.965$, $T_{max} = 0.982$ $\theta_{max} = 25.0^\circ$

Refinement

Refinement on F^2 $w = 1/[\sigma^2(F_o^2) + (0.0481P)^2 + 1.5999P]$
 $R[F^2 > 2\sigma(F^2)] = 0.046$ where $P = (F_o^2 + 2F_c^2)/3$
 $wR(F^2) = 0.117$ $(\Delta/\sigma)_{max} = 0.001$
 $S = 1.02$ $\Delta\rho_{max} = 0.31$ e Å $^{-3}$
 5714 reflections $\Delta\rho_{min} = -0.33$ e Å $^{-3}$
 367 parameters
 H-atom parameters constrained

Table 1 Selected geometric parameters (Å, °).

Si1–O3	1.6349 (16)	Si3–O3	1.6382 (16)
Si1–O1	1.6366 (17)	Si3–O2	1.6518 (18)
Si2–O1	1.6363 (16)	Si3–N1	1.691 (2)
Si2–O2	1.6386 (16)	Si3–N2	1.694 (2)
O3–Si3–O2	104.96 (8)	O3–Si3–N2	104.95 (10)
O3–Si3–N1	112.27 (10)	O2–Si3–N2	112.79 (10)
O2–Si3–N1	110.07 (10)	N1–Si3–N2	111.56 (11)

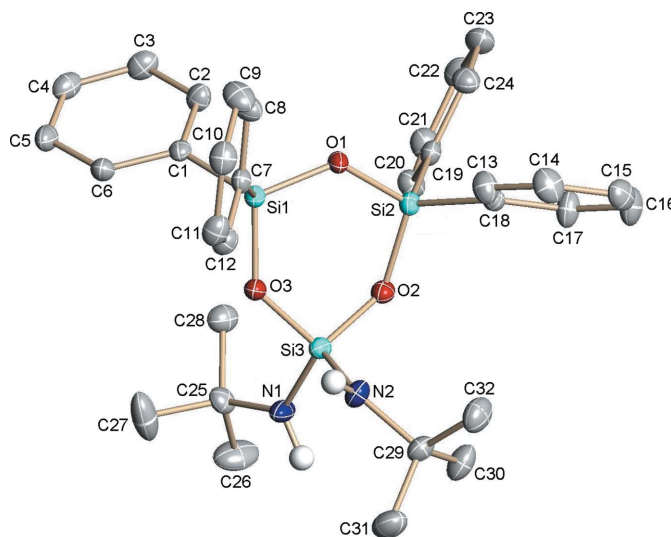


Figure 1 The molecular structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are shown at the 30% probability level. H atoms, except for those on N atoms, have been omitted for clarity.

All H atoms were refined in geometrically idealized positions with $U_{iso}(H) = 1.2-1.5U_{eq}(\text{parent atom})$. Atoms H1A and H2A were first located in an electron difference map, before being constrained to their parent N atoms. C–H distances are in the range 0.95–0.98 Å. The N1–H1A and N2–H2A distances are 0.88 Å.

Data collection: COLLECT (Nonius, 1998); cell refinement: DENZO (Otwinowski & Minor, 1997); data reduction: DENZO; program(s) used to solve structure: SHELXTL (Bruker, 2000); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL.

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