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1,3-Bis(2,6-diisopropylphenyl)-2,2,4,4-tetramethyl-1,3-diaza-2,4-disilacyclobutane

Syed A. A. Shah, Herbert W. Roesky, Paolo Lubini and Hans-Georg Schmidt

Institut für Anorganische Chemie der Universität Göttingen, Tammannstrasse 4, D-37077 Göttingen, Germany. E-mail: paolo@shelx.uni-ac.gwdg.de

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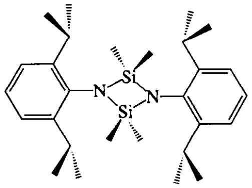
Abstract

The molecule of the title compound, $[\{(CH_3)_2CH\}_2\text{-}C_6H_3NSi(CH_3)_2]_2$, displays a four-membered silicon–nitrogen ring system, with the Si_2N_2 ring exactly planar.

Comment

In the course of our work on silicon–nitrogen and silicon–oxygen cyclic systems (Elias, Schmidt, Noltemeyer & Roesky, 1992; Elias, Roesky, Robinson & Sheldrick, 1993; Gosink *et al.*, 1994), the title compound, (I), was isolated. It crystallizes in the centrosymmetric space group $P2_1/n$ with half a molecule in the asymmetric unit so that the Si_2N_2 four-membered ring is exactly planar.



(I)

The Si—N distances are 1.748 (1) and 1.744 (1) Å, the Si—N—Si angle is 93.72 (5)° and the N—Si—N angle is 86.28 (5)°. A similar molecular geometry has been observed in related compounds with Si_2N_2 rings planar within experimental error, Si—N bond lengths in the range 1.73–1.75 Å, Si—N—Si bond angles of 93–95° and N—Si—N angles of 85–87° (Bihatsi, Hencsei & Parkanyi, 1981; Clegg, Klingebiel, Sheldrick & Vater, 1981; Szollosy, Parkanyi, Bihatsi & Hencsei, 1983; Parkanyi, Argay, Hencsei & Nagy, 1976; Parkanyi, Bihatsi & Hencsei, 1994). Owing to the steric requirements of the isopropyl groups in the *ortho* position, the phenyl groups are forced to lie in a plane perpendicular to the cyclodisilazane ring [dihedral angle 89.12 (9)°]. This conformation is characterized by intramolecular contacts between the tertiary H atoms of the isopropyl groups and the N atom [H(161)···N1 2.461 (2) Å, H(121)···N1 2.479 (2) Å]. The C—N bond length of 1.429 (2) Å is also comparable with that observed in related structures with *ortho* substituted phenyl rings in which the dihedral angle also lies around 90° (Bihatsi, Hencsei & Parkanyi, 1981; Clegg, Klingebiel, Sheldrick & Vater, 1981).

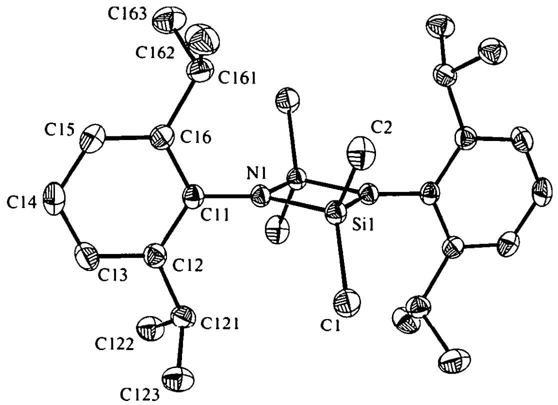


Fig. 1. Molecular structure of the title compound showing the atomic numbering scheme. Non-H atoms are represented by displacement ellipsoids at the 50% probability level. H atoms are omitted for clarity.

Experimental

The title compound was synthesized by reaction of the dilithium salt of 2,6-diisopropylaniline and dichlorodimethylsilane (molar ratio 1:1) in tetrahydrofuran. Recrystallization was from hexane. The crystal was mounted on a glass fibre according to the method described by Kottke & Stalke (1993).

Crystal data

$C_{28}H_{46}N_2Si_2$
$M_r = 466.86$
Monoclinic
$P2_1/n$
$a = 9.7355\ (19)$ Å
$b = 8.8451\ (18)$ Å
$c = 16.594\ (3)$ Å
$\beta = 99.91\ (3)°$
$V = 1407.6\ (5)$ Å^3
$Z = 2$
$D_x = 1.102$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 38 reflections
$\theta = 20\text{–}25°$
$\mu = 0.143$ mm^{-1}
$T = 150\ (2)$ K
Needle
0.60 × 0.26 × 0.26 mm
Colourless

Data collection

Stoe *P*4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
3957 measured reflections
2495 independent reflections
2263 observed reflections $[I > 2\sigma(I)]$

$R_{int} = 0.0154$
$\theta_{max} = 25.05°$
$h = -11 \rightarrow 11$
$k = -10 \rightarrow 10$
$l = -19 \rightarrow 19$
3 standard reflections
frequency: 90 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0329$
$wR(F^2) = 0.0897$

$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.355$ e Å^{-3}
$\Delta\rho_{min} = -0.244$ e Å^{-3}

$S = 1.037$
2495 reflections
151 parameters
Methyl H atoms refined as rigid groups, others riding
$w = 1/[\sigma^2(F_o^2) + (0.0463P)^2 + 0.5608P]$
where $P = (F_o^2 + 2F_c^2)/3$

Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)*

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
Si1	0.53781 (4)	0.09933 (4)	0.05492 (2)	0.02092 (13)
N1	0.44462 (11)	0.09563 (12)	−0.04499 (6)	0.0201 (3)
C1	0.43712 (16)	0.16566 (18)	0.13365 (8)	0.0319 (3)
C2	0.70274 (16)	0.20907 (17)	0.07048 (9)	0.0321 (3)
C11	0.38136 (14)	0.21337 (14)	−0.09760 (7)	0.0209 (3)
C12	0.24128 (14)	0.25562 (15)	−0.09808 (8)	0.0244 (3)
C13	0.18406 (16)	0.37400 (17)	−0.14867 (9)	0.0309 (3)
C14	0.26048 (16)	0.45051 (18)	−0.19832 (9)	0.0333 (3)
C15	0.39685 (16)	0.40832 (16)	−0.19810 (9)	0.0296 (3)
C16	0.45944 (14)	0.29083 (15)	−0.14927 (8)	0.0238 (3)
C121	0.14866 (14)	0.17733 (16)	−0.04575 (8)	0.0255 (3)
C122	0.02672 (16)	0.09664 (18)	−0.09988 (9)	0.0328 (3)
C123	0.09385 (16)	0.28845 (18)	0.01226 (9)	0.0335 (3)
C161	0.61026 (15)	0.25177 (16)	−0.15315 (9)	0.0277 (3)
C162	0.70550 (17)	0.38758 (18)	−0.12837 (11)	0.0391 (4)
C163	0.62561 (19)	0.1965 (2)	−0.23888 (10)	0.0431 (4)

Table 2. *Selected geometric parameters (Å, °)*

Si1—N1$^{\rm i}$	1.7435 (11)	Si1—C1	1.8581 (15)
Si1—N1	1.7479 (12)	N1—C11	1.4286 (16)
Si1—C2	1.8559 (16)		
N1$^{\rm i}$—Si1—N1	86.28 (5)	C11—N1—Si1$^{\rm i}$	134.60 (9)
N1$^{\rm i}$—Si1—C2	115.71 (6)	C11—N1—Si1	131.67 (9)
N1—Si1—C2	115.92 (6)	Si1$^{\rm i}$—N1—Si1	93.72 (5)
N1$^{\rm i}$—Si1—C1	116.80 (6)	C12—C11—N1	120.69 (12)
N1—Si1—C1	114.98 (6)	C16—C11—N1	120.18 (12)
C2—Si1—C1	106.59 (7)		

Symmetry code: (i) $1 - x, -y, -z$.

Data were collected according to the learnt-profile method (Clegg, 1983). Corrections for background, decay, Lorentz and polarization factors were included in the data reduction using *REDU*4 (Stoe & Cie, 1990*b*). The structure was solved by direct methods and refined by least-squares techniques. Non-H atoms were refined with anisotropic displacement parameters. The positions of the H atoms were refined using a riding model. Most calculations were conducted on an IBM *RS*6000.

Data collection: *DIF*4 (Stoe & Cie, 1990*a*). Cell refinement: *DIF*4. Data reduction: *REDU*4 (Stoe & Cie, 1990*b*). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL-Plus* (Sheldrick, 1992). Software used to prepare material for publication: *SHELXL*93.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: JZ1138). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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3,3,6,6-Tetramethyl-9-(2-nitrophenyl)-3,4,5,6,9,10-hexahydroacridine-1,8(2*H*,7*H*)-dione

MARCO BRITO-ARIAS,[a] GUADALUPE RAMIREZ,[a] RUTH E. RIVAS,[a] ELIES MOLINS[b] AND WALDEMAR MANIUKIEWICZ[b]

[a]*Unidad Profesional Interdisciplinaria de Biotecnología-IPN, Av. Acueducto s/n Col. Barrio la Laguna Ticomán CP 07340, México DF, México, and* [b]*Institut de Ciència de Materials de Barcelona, Campus de la UAB, 08193 Bellaterra, Spain.*
E-mail: waldemar@icmab.es

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Abstract

The title compound, $C_{23}H_{26}N_2O_4$, consists of a partially hydrogenated acridine moiety with one 2-nitrophenyl substituent on the central ring. The acridine nucleus suf-

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