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Dianilinodimethylsilane

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Key indicators

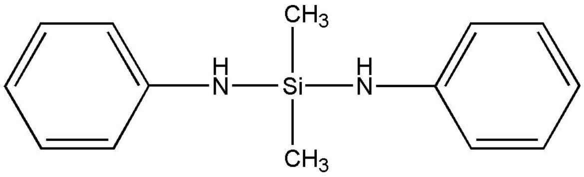
Single-crystal X-ray study
T = 183 K
Mean σ(C–C) = 0.006 Å
R factor = 0.079
wR factor = 0.155
Data-to-parameter ratio = 15.0

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

The title compound, $C_{14}H_{18}N_2Si$ or $(PhNH)_2SiMe_2$, has been prepared and its single-crystal structure determined. There are two independent molecules in the asymmetric unit. Each of them contains an N—Si—N fragment which is twisted out of the phenyl plane . Each molecule can be considered as having an approximate twofold axis.

Comment

Compounds with two amine groups have been extensively used as ligands in organometallic chemistry, as they exhibit unusual ligand geometry and different coordination behaviours. Organometallic complexes derived from such ligands are also effective catalysts for olefin polymerization (Hill & Hitchcock, 2002; Gibson *et al.*, 1998). There are many different groups which can be used as the link between the two amine groups, such as C_1, C_2 or C_n, Si, or other heteroatoms (Warren *et al.*, 1996, 1998; Male *et al.*, 1997). The title compound, (I), can be considered as a diamine as well as a silane, and has been characterized by single-crystal diffraction analysis.



(I)

There are two independent molecules in the asymmetric unit of (I), with similar geometries (Fig. 1, Table 1). The bond lengths are normal, with Si—N ranging from 1.741 (3) to 1.739 (3) Å (Allen *et al.*, 1987). The N_2SiC_2 core of the molecule is approximately tetrahedral, with N—Si—N, C—Si—C

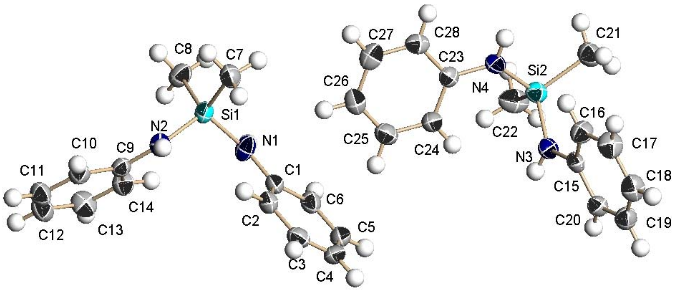


Figure 1
The asymmetric unit of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are represented by small spheres of arbitrary radii.

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and N—Si—C angles in the ranges 111.3 (2)–111.9 (2), 111.7 (2)–112.5 (2) and 103.7 (2)–112.9 (2)°, respectively. The C—N—Si angles range between 128.7 (2) and 131.4 (2)°. The dihedral angle between the C10—C9—C14 and N1—Si1—N2 planes is 61.3 (3)°, and that between the C2—C1—C6 and N1—Si1—N2 planes is 51.0 (3)°. Similarly, in the other molecule, the dihedral angle between the C15—C16—C20 and N3—Si2—N4 planes is 51.2 (3)° and that between the C23—C24–C28 and N3—Si2—N4 planes is 60.3 (4)°.

Experimental

n-Butyllithium (1 ml, 1 mmol, 1.0 *M* in hexane) was added dropwise to aniline (1 mmol) in hexane (50 ml) at 195 K. The mixture was warmed to room temperature and stirred overnight. $SiMe_2Cl_2$ (0.5 mmol) was then added to the mixture (in the molar ratio 1:2) at 195 K. The mixture was stirred for 12 h and then filtered. The filtrate was distilled at 1 mm Hg (1 mm Hg = 133.322 Pa) and 415–417 K to give a yellow liquid. Colourless crystals of (I) were obtained when this liquid was dissolved in hexane and left at 263 K for 3 d (m.p. 411 K). Spectroscopic analysis: ^{1}H NMR ($CDCl_3$, δ, p.p.m.): 0.46 (*s*, 6H, $SiMe_2$), 3.73 (*s*, 2H, NH), 6.75–6.85 (*m*, 6H, Ph), 7.14–7.25 (*m*, 4H, Ph); ^{13}C NMR ($CDCl_3$, δ, p.p.m.): 1.295 (*s*, $SiMe_2$), 119.058, 120.818, 131.771, 148.712 (*s*, Ph). Analysis, calculated for $C_{14}H_{18}N_2Si$: C 69.37, H 7.48, N 11.56%; found: C 69.41, H 7.45, N 11.50%. All reactions were performed under argon, using standard Schlenk techniques. The hexane was dried by distillation over a sodium–potassium alloy.

Crystal data

$C_{14}H_{18}N_2Si$
$M_r = 242.39$
Triclinic, $P\bar{1}$
$a = 10.384$ (2) Å
$b = 11.867$ (2) Å
$c = 12.659$ (3) Å
$\alpha = 90.980$ (3)°
$\beta = 108.339$ (3)°
$\gamma = 111.211$ (2)°
$V = 1365.2$ (5) Å^3

$Z = 4$
$D_x = 1.179$ Mg m$^{-3$}
Mo $K\alpha$ radiation
Cell parameters from 4719 reflections
$\theta = 2.3$–27.0°
$\mu = 0.15$ mm^{-1}
$T = 183$ (2) K
Block, colourless
0.30 × 0.20 × 0.10 mm

Data collection

Siemens SMART CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.956$, $T_{\max} = 0.985$
5661 measured reflections

4719 independent reflections
4310 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.022$
$\theta_{\max} = 25.0°$
$h = -12 \rightarrow 11$
$k = -14 \rightarrow 14$
$l = -15 \rightarrow 11$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.079$
$wR(F^2) = 0.155$
$S = 1.23$
4719 reflections
315 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0339P)^2 + 1.3242P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.27$ e Å^{-3}
$\Delta\rho_{\min} = -0.28$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Si1—N2	1.727 (3)	Si2—C21	1.845 (4)
Si1—N1	1.741 (3)	Si2—C22	1.850 (4)
Si1—C7	1.844 (4)	N1—C1	1.393 (4)
Si1—C8	1.848 (4)	N2—C9	1.394 (4)
Si2—N3	1.722 (3)	N3—C15	1.396 (4)
Si2—N4	1.739 (3)	N4—C23	1.394 (4)
N2—Si1—N1	111.95 (15)	N4—Si2—C21	104.88 (16)
N2—Si1—C7	105.10 (16)	N3—Si2—C22	105.08 (17)
N1—Si1—C7	112.86 (17)	N4—Si2—C22	111.68 (18)
N2—Si1—C8	111.77 (16)	C21—Si2—C22	112.5 (2)
N1—Si1—C8	103.68 (16)	C1—N1—Si1	131.4 (2)
C7—Si1—C8	111.70 (19)	C9—N2—Si1	128.7 (2)
N3—Si2—N4	111.34 (15)	C15—N3—Si2	128.9 (2)
N3—Si2—C21	111.51 (17)	C23—N4—Si2	128.8 (2)
Si1—N1—C1—C2	12.2 (5)	Si2—N3—C15—C16	26.7 (5)
Si1—N2—C9—C14	28.3 (5)	Si2—N4—C23—C24	16.3 (5)

All H atoms were initially located in a difference Fourier map. The methyl H atoms were then constrained to an ideal geometry, with C—H distances of 0.98 Å and $U_{\rm iso}(H) = 1.5U_{\rm eq}(C)$, but each group was allowed to rotate freely about its C—C bond. The positions of the amine H atoms were refined freely along with an isotropic displacement parameter (refined N—H distances fixed at 0.86 Å). All other H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H distances in the range 0.95–1.00 Å and $U_{\rm iso}(H) = 1.2U_{\rm eq}(C)$.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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