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# Structure of 1,3,5-Trimethyl-2,2,4,4,6,6-hexakis(methylamino)cyclotrisilazane

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**Abstract.** $C_9H_{33}N_9Si_3$, $M_r = 351{\cdot}5$, monoclinic, $P2_1/n$, $a = 8{\cdot}737\ (2)$, $b = 14{\cdot}336\ (3)$, $c = 16{\cdot}120\ (3)$ Å, $\beta = 97{\cdot}20\ (6)°$, $V = 2003\ (4)$ Å$^3$, $Z = 4$, $D_x = 1{\cdot}1661$ g cm$^{-3}$, $\lambda$(Mo $K\alpha$) = 0·71069 Å, $\mu = 2{\cdot}39$ cm$^{-1}$, $F(000) = 768$, room temperature, final $R = 0{\cdot}0573$ for 1935 observed reflections and 191 variables, $F(hkl) > 4\sigma(F)$. The six-membered $Si_3N_3$ ring is planar with a mean value of 1·703 Å for the Si—N bond lengths. The methyl groups attached to the ring N atoms are nearly coplanar with the ring and molecular symmetry (excluding H atoms) nearly fulfills $\bar{6}2m$. The molecules are stacked in the manner of coin rolls along [101].

**Experimental.** $C_9H_{33}N_9Si_3$ was synthesized by reaction of $SiCl_4$ and $CH_3NH_2$ in *n*-pentane. Transparent crystals were grown from the vapor phase. Crystal 0·4 × 0·5 × 0·5 mm. Automated Enraf–Nonius CAD-4 diffractometer, graphite-monochromated Mo $K\alpha$ radiation. 25 centered reflections within $4 < \theta < 12°$ used for determining lattice parameters. Data corrected for Lorentz and polarization effects, absorption ignored. $2\theta_{max} = 50°$, range of *hkl*: $0 \le h \le 10$, $0 \le k \le 17$, $-19 \le l \le 19$. Two check reflections measured every 200 reflections showed no significant intensity variation over data collection. $\omega/2\theta$-scan technique. Total of 3940 reflections measured, 2857 unique (non-zero), 1935 observed with $F(hkl) > 4\sigma(F)$. Structure solved by direct methods, full-matrix least-squares refinement on $F$ of 191 parameters (*SHELX*76, Sheldrick, 1976) on a VAX11/750 computer. Anisotropic thermal parameters for non-H atoms, H atoms allowed to ride at fixed distance on C and N atoms, refined isotropically. $R = 0{\cdot}057$, unit weights, $R_{int} = 0{\cdot}012$, $(\Delta/\sigma)_{max} = 0{\cdot}001$, $\Delta\rho_{max} = 0{\cdot}24$, $\Delta\rho_{min} = -0{\cdot}26$ e Å$^{-3}$, atomic scattering factors those incorporated in *SHELX*76.

Table 1 lists atomic positional and equivalent isotropic thermal parameters, Table 2 interatomic distances and valence angles.* Fig. 1 shows a

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52644 (17 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Fractional coordinates and equivalent isotropic temperature coefficients for non-*H *atoms*

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

| | $x$ | $y$ | $z$ | $U_{eq}$ (Å$^2$) |
|---|---|---|---|---|
| Si(1) | 1·1656 (1) | 0·8154 (1) | 0·6642 (1) | 0·0476 (5) |
| Si(2) | 0·9749 (1) | 0·6372 (1) | 0·6931 (1) | 0·0482 (6) |
| Si(3) | 0·8454 (1) | 0·8338 (1) | 0·7297 (1) | 0·0506 (5) |
| N(1) | 1·0174 (5) | 0·8713 (3) | 0·7030 (3) | 0·0594 (19) |
| N(2) | 1·1212 (4) | 0·7004 (3) | 0·6592 (3) | 0·0491 (17) |
| N(3) | 0·8447 (5) | 0·7157 (3) | 0·7224 (3) | 0·0596 (19) |
| N(4) | 1·1994 (6) | 0·8551 (4) | 0·5688 (3) | 0·0785 (24) |
| N(5) | 1·3353 (5) | 0·8412 (3) | 0·7255 (3) | 0·0617 (20) |
| N(6) | 1·0299 (5) | 0·5627 (3) | 0·7742 (3) | 0·0618 (22) |
| N(7) | 0·8979 (6) | 0·5621 (3) | 0·6162 (3) | 0·0725 (25) |
| N(8) | 0·6883 (5) | 0·8796 (3) | 0·6695 (3) | 0·0657 (20) |
| N(9) | 0·8183 (6) | 0·8735 (4) | 0·8262 (3) | 0·0847 (27) |
| C(1) | 1·0344 (8) | 0·9744 (6) | 0·7043 (6) | 0·1132 (45) |
| C(2) | 1·2426 (7) | 0·6407 (4) | 0·6275 (5) | 0·0855 (32) |
| C(3) | 0·7035 (7) | 0·6715 (5) | 0·7467 (6) | 0·1142 (42) |
| C(4) | 1·0988 (10) | 0·8415 (7) | 0·4929 (5) | 0·1259 (49) |
| C(5) | 1·3574 (7) | 0·8231 (6) | 0·8144 (4) | 0·0902 (35) |
| C(6) | 1·0784 (9) | 0·5953 (5) | 0·8593 (4) | 0·0904 (34) |
| C(7) | 0·8455 (10) | 0·5920 (6) | 0·5323 (5) | 0·1106 (41) |
| C(8) | 0·6626 (8) | 0·8644 (7) | 0·5803 (4) | 0·1127 (44) |
| C(9) | 0·9274 (10) | 0·8666 (7) | 0·9009 (5) | 0·1267 (44) |

Table 2. *Bond lengths* (Å) *and angles* (°)

| | | | | | |
|---|---|---|---|---|---|
| Si(1)—N(1) | 1·707 (5) | Si(2)—N(7) | 1·715 (5) | N(3)—C(3) | 1·483 (7) |
| Si(1)—N(2) | 1·695 (5) | Si(3)—N(1) | 1·702 (5) | N(4)—C(4) | 1·428 (9) |
| Si(1)—N(4) | 1·699 (6) | Si(3)—N(3) | 1·696 (5) | N(5)—C(5) | 1·446 (8) |
| Si(1)—N(5) | 1·715 (5) | Si(3)—N(8) | 1·710 (4) | N(6)—C(6) | 1·461 (8) |
| Si(2)—N(2) | 1·711 (5) | Si(3)—N(9) | 1·701 (6) | N(7)—C(7) | 1·438 (9) |
| Si(2)—N(3) | 1·709 (5) | N(1)—C(1) | 1·484 (8) | N(8)—C(8) | 1·444 (9) |
| Si(2)—N(6) | 1·709 (5) | N(2)—C(2) | 1·501 (8) | N(9)—C(9) | 1·443 (9) |

| | | | |
|---|---|---|---|
| N(2)—Si(1)—N(1) | 107·1 (2) | N(9)—Si(3)—N(8) | 101·3 (3) |
| N(4)—Si(1)—N(1) | 113·7 (3) | C(1)—N(1)—Si(1) | 113·1 (4) |
| N(4)—Si(1)—N(2) | 110·3 (3) | C(1)—N(1)—Si(3) | 113·7 (4) |
| N(5)—Si(1)—N(1) | 109·1 (3) | C(2)—N(2)—Si(1) | 113·8 (4) |
| N(5)—Si(1)—N(2) | 114·4 (3) | C(2)—N(2)—Si(2) | 113·3 (4) |
| N(5)—Si(1)—N(4) | 102·3 (3) | C(3)—N(3)—Si(2) | 113·4 (4) |
| N(3)—Si(2)—N(2) | 106·8 (2) | C(3)—N(3)—Si(3) | 113·9 (4) |
| N(6)—Si(2)—N(2) | 115·2 (2) | C(4)—N(4)—Si(1) | 124·9 (5) |
| N(6)—Si(2)—N(3) | 109·5 (3) | C(5)—N(5)—Si(1) | 122·1 (4) |
| N(7)—Si(2)—N(2) | 110·0 (2) | C(6)—N(6)—Si(2) | 122·6 (5) |
| N(7)—Si(2)—N(3) | 113·5 (3) | C(7)—N(7)—Si(2) | 122·9 (5) |
| N(3)—Si(3)—N(1) | 107·1 (2) | C(8)—N(8)—Si(3) | 121·4 (5) |
| N(7)—Si(2)—N(6) | 102·0 (3) | C(9)—N(9)—Si(3) | 126·0 (5) |
| N(8)—Si(3)—N(1) | 114·0 (3) | Si(3)—N(1)—Si(1) | 132·7 (3) |
| N(8)—Si(3)—N(3) | 110·4 (3) | Si(2)—N(2)—Si(1) | 132·5 (4) |
| N(9)—Si(3)—N(1) | 110·7 (3) | Si(3)—N(3)—Si(2) | 132·8 (3) |
| N(9)—Si(3)—N(3) | 113·4 (3) | | |

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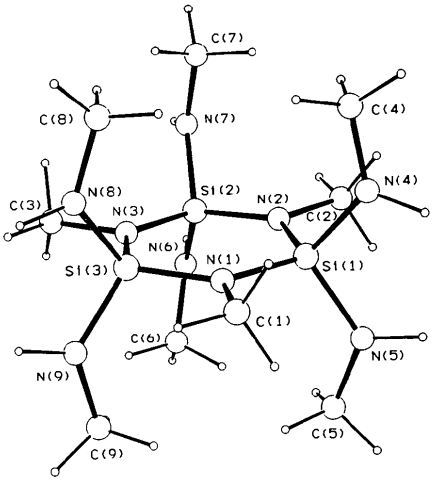


Fig. 1. The molecular structure of the title compound.

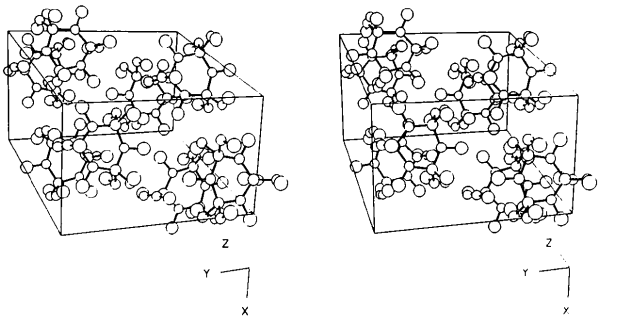


Fig. 2. Stereoplot of the unit cell. H atoms are omitted.

perspective view of the molecule, Fig. 2 crystal packing.

**Related literature.** With respect to the $Si_3N_3$ core, related silazanes are 2,2,4,4,6,6-hexaisopropylcyclotrisilazane (Klingebiel & Vater, 1983), 2,4,6-tri-*tert*-butyl-2,4,6-trifluorocyclotrisilazane (Clegg, Sheldrick & Stalke, 1984*b*) and 2,2,4,4,6,6-hexa-*tert*-butylcyclotrisilazane (Clegg, Sheldrick & Stalke, 1984*a*), all showing a planar six-membered ring in agreement with our results.

The reactions of $SiCl_4$ with $CH_3NH_2$ in the gas phase (Drake & Westwood, 1971), without solvent at room temperature (Hagen & Callaway, 1972) and in petroleum ether (Adrianov, Il'in, Talanov, Isakova & Sidorenko, 1976) have been reported in the literature. Based on an elementary analysis, Adrianov *et al.* (1976) suggested for one of their products the same composition as that of the title compound; however, no information on the molecular structure was given.

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# 9-(2,4-Cyclopentadienylidene)bicyclo[3.3.1]nonane, a Ring-Strained Pentafulvene


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**Abstract.** 9-(2,4-Cyclopentadien-1-ylidene)bicyclo-[3.3.1]nonane, $C_{14}H_{18}$, $M_r = 186{\cdot}3$, monoclinic, $P2_1/c$, $a = 10{\cdot}6088\ (11)$, $b = 12{\cdot}251\ (3)$, $c = 9{\cdot}2343\ (14)$ Å, $\beta = 111{\cdot}23\ (1)°$, $V = 1118{\cdot}7\ (6)$ Å$^3$, $Z = 4$, $D_x = 1{\cdot}106$ Mg m$^{-3}$, $\lambda$(Cu $K\alpha$) = 1·54184 Å, $\mu = 0{\cdot}425$ mm$^{-1}$, $F(000) = 408$, $T = 295$ K, $R = 0{\cdot}042$ for 1636 observations with $I > 3\sigma(I)$ (of 2300 unique data). The bicyclo[3.3.1]nonane adopts a twin-chair conformation. The bond angle of the C=C exocyclic to the cyclopentadienylidene ring is 110·95 (9)°. This bond angle is very close to the corresponding bond angle in 2-(2,4-cyclopentadien-1-ylidene)adamantane or adamantylidenefulvene. The cyclopentadienylidene ring is planar, with maximum deviation of 0·005 (2) Å.


**Experimental.** The title compound was prepared by condensing bicyclo[3.3.1]nonan-9-one and 1,3-

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