

Related literature. The structure determination of the title compound was undertaken to elucidate its molecular conformation in order to help explain why this compound inhibits xanthine oxidase (Henichart, Pesando, Breittmayer, Wallet, Gaydou & Puiseux-Dao, 1991). A similar compound which is not a xanthine oxidase inhibitor has already been described (Wallet, Gaydou, Tinant, Declercq, Baldy & Bonifassi, 1990). 5,7-Dihydroxy-4'-methoxyflavone presents the same hydrogen-bond pattern (Cantrel, 1986). A survey of the structures of flavones and flavanones has been reported (Cody, 1988).

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Structure of 1,3-Di-*tert*-butyl-2,4-dichloro-2,4-bis[1-(trimethylsilyl)vinyl]-1,3-diaza-2,4-disilacyclobutane*

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Abstract. $C_{18}H_{40}Cl_2N_2Si_4$, $M_r = 467.8$, monoclinic, $P2_1/c$, $a = 10.580$ (5), $b = 9.803$ (4), $c = 13.610$ (7) Å, $\beta = 104.57$ (4)°, $V = 1366$ (1) Å³, $Z = 2$, $D_x = 1.137$ g cm⁻³, $\lambda(Mo K\alpha) = 0.71073$ Å, $\mu = 4.2$ cm⁻¹, $F(000) = 504$, $T = 223$ K, $R = 0.041$, $wR = 0.038$, for 2287 observed reflections. The central four-membered ring, Si₂N₂, shows a crystallographic centre of symmetry. Surrounded by very bulky ligands, the exocyclic C(1)=C(2) double bond is preserved, and is therefore not easily accessible for further reaction.

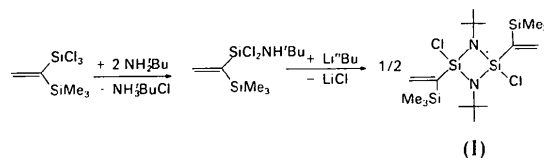
Experimental. The title compound (I) was prepared in a multiple-step procedure (Penzenstadler, 1991). Recrystallization from *n*-hexane yielded colourless crystals suitable for X-ray crystallography. Elemental analysis: calculated C 46.20, H 8.62, N 5.99, Si 24.03, Cl 15.16%; found C 45.52, H 8.82, N 5.59, Si 23.90, Cl 14.96%. A well shaped colourless

* Silaheterocycles. XIX. Part XVIII: Auner & Penzenstadler (1992).

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prism of approximate dimensions 0.51 × 0.31 × 0.31 mm was randomly mounted on a glass fibre. Preliminary examinations and data collection were carried out with Mo *K*α radiation on an Enraf-Nonius CAD-4 diffractometer equipped with a graphite monochromator. The observed extinctions (*h*0*l*: *l* = 2*n* + 1; 0*k*0: *k* = 2*n* + 1) together with the monoclinic crystal system confirmed the space group $P2_1/c$. Final cell constants were obtained by least-squares refinement of 23 automatically centred high-angle reflections ($39.9 < 2\theta < 49.1^\circ$). Data were collected using ω scans. Each reflection in the hemisphere h 12, k 11, $l \pm 16$ and $\theta_{\max} = 25.0^\circ$ was measured with a maximum scan time of 90 s. Orientation control reflections were monitored every 100 reflections. No loss of intensity of three stand-

Table 1. Fractional atomic coordinates and equivalent isotropic atomic displacement parameters (\AA^2) with e.s.d.'s in parentheses

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
Cl	0.20045 (6)	0.02811 (7)	0.38781 (4)	0.0432 (2)
Si(1)	0.10089 (6)	0.06370 (7)	0.49858 (5)	0.0280 (2)
Si(2)	0.34506 (7)	0.28754 (8)	0.57585 (5)	0.0375 (2)
N	0.0641 (2)	-0.0851 (2)	0.5522 (1)	0.0301 (6)
C(1)	0.1912 (2)	0.1986 (2)	0.5825 (2)	0.0327 (7)
C(2)	0.1315 (3)	0.2330 (3)	0.6541 (2)	0.0604 (9)
C(3)	0.4782 (3)	0.1635 (3)	0.5811 (3)	0.0672 (11)
C(4)	0.3158 (3)	0.3898 (3)	0.4578 (2)	0.0764 (11)
C(5)	0.3966 (3)	0.4048 (3)	0.6864 (2)	0.0569 (10)
C(6)	0.1410 (2)	-0.1835 (3)	0.6250 (2)	0.0428 (8)
C(7)	0.1633 (4)	-0.1265 (4)	0.7333 (2)	0.0796 (11)
C(8)	0.2719 (3)	-0.2044 (3)	0.6014 (3)	0.0732 (11)
C(9)	0.0678 (3)	-0.3159 (3)	0.6195 (3)	0.0675 (11)

Table 2. Selected bond distances (\AA), bond angles ($^\circ$) and intermolecular contacts (\AA)

Cl—Si(1)	2.074 (1)	Cl—Si(1)—N	112.13 (7)
Si(1)—N	1.719 (2)	Cl—Si(1)—N'	112.19 (7)
Si(1)—N'	1.721 (2)	Cl—Si(1)—C(1)	106.95 (8)
Si(1)—C(1)	1.849 (2)	N—Si(1)—N'	87.64 (9)
Si(2)—C(1)	1.869 (2)	N—Si(1)—C(1)	118.78 (9)
Si(2)—C(3)	1.849 (3)	N'—Si(1)—C(1)	118.39 (10)
Si(2)—C(4)	1.853 (3)	Si(1)—N—Si(1')	92.36 (9)
Si(2)—C(5)	1.863 (3)	Si(1)—N—C(6)	134.08 (16)
N—C(6)	1.473 (3)	Si(1')—N—C(6)	133.08 (16)
C(1)—C(2)	1.331 (4)	Si(1)—C(1)—Si(2)	128.65 (12)
C(6)—C(7)	1.538 (4)	Si(1)—C(1)—C(2)	111.53 (19)
C(6)—C(8)	1.512 (4)	Si(2)—C(1)—C(2)	119.81 (19)
C(6)—C(9)	1.504 (4)		
Si(1)⋯Si(1')	2.482 (1)		

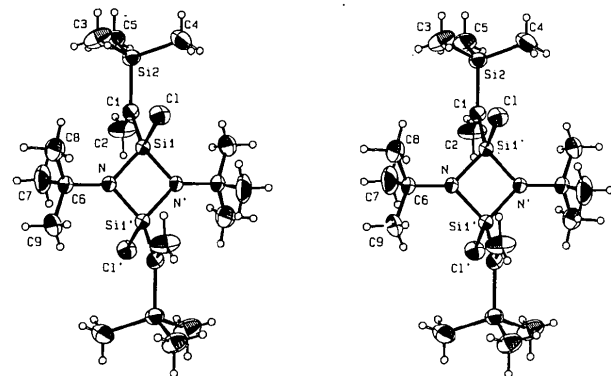


Fig. 1. ORTEPII (Johnson, 1976) stereo plot. Ellipsoids are drawn at the 50% level, and H atoms are assigned arbitrary values. The primed atoms are obtained by the symmetry operation $-x, -y, 1-z$.

ards, checked every 3600 s during the data collection, was observed. No absorption correction was applied. 168 systematically absent data together with 150 reflections with negative measured intensities were rejected from the original data set of 2710 measured reflections. After averaging, 2287 ($I > 0.01$) out of 2290 unique reflections ($R_{\text{int}} = 0.007$) were used in

the refinement. The structure was solved by direct methods and subsequent difference Fourier maps. Full-matrix least-squares refinement was carried out by minimizing $\sum w(|F_o| - |F_c|)^2$. Anisotropic atomic displacement parameters were introduced for all non-H atoms. All H atoms were located, included with individual isotropic parameters in the parameter set and refined. Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). Anomalous dispersion was considered. The refinement converged at $R = 0.041$, $wR = 0.038$ [$w = 1/\sigma^2(F_o)$], $S = 4.87$, for 198 parameters. Final shift/e.s.d. < 0.001 . Final difference Fourier syntheses showed no significant features (maximum/minimum $\Delta\rho$ $0.37/-0.27 \text{ e \AA}^{-3}$). All calculations were performed on a VAX 3100 computer with the STRUX-III system (Kiprof, Herdtweck, Schmidt, Birkhahn & Massa, 1987) including the programs ORTEPII (Johnson, 1976), PLATON (Spek, 1982), SCHAKAL (Keller, 1988), SDP (Frenz, 1988), and SHELX76 (Sheldrick, 1976). Final positional parameters are given in Table 1, selected bond angles and distances in Table 2. Fig. 1 shows a stereo plot of the molecule.*

Related literature. Wheatley (1962) and Fink (1963) first reported the existence of four-membered silazane rings.

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* Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and bond distances and angles involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55536 (22 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: SH1006]

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