Table 2. *Bond distances* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

Si(1)—O(1)	1.608 (1)	C(4)—C(5)	1.375 (6)
Si(1)—N(1)	1.703 (3)	C(5)—C(6)	1.386 (6)
Si(1)—N(2)	1.711 (3)	C(7)—C(8)	1.388 (4)
Si(1)—N(3)	1.709 (3)	C(7)—C(12)	1.376 (5)
F(1)—C(2)	1.361 (4)	C(8)—C(9)	1.365 (5)
F(2)—C(8)	1.366 (4)	C(9)—C(10)	1.368 (6)
F(3)—C(14)	1.368 (5)	C(10)—C(11)	1.377 (5)
N(1)—C(1)	1.400 (5)	C(11)—C(12)	1.391 (5)
N(2)—C(7)	1.401 (4)	C(13)—C(14)	1.380 (6)
N(3)—C(13)	1.394 (4)	C(13)—C(18)	1.395 (6)
C(1)—C(2)	1.386 (5)	C(14)—C(15)	1.371 (5)
C(1)—C(6)	1.386 (5)	C(15)—C(16)	1.380 (8)
C(2)—C(3)	1.360 (7)	C(16)—C(17)	1.371 (7)
C(3)—C(4)	1.382 (6)	C(17)—C(18)	1.378 (5)

N(2)—Si(1)—N(3)	108.3 (2)	N(2)—C(7)—C(12)	124.0 (3)
N(1)—Si(1)—N(3)	108.0 (2)	N(2)—C(7)—C(8)	119.8 (3)
N(1)—Si(1)—N(2)	108.0 (2)	C(8)—C(7)—C(12)	116.2 (3)
O(1)—Si(1)—N(3)	110.3 (1)	F(3)—C(8)—C(7)	116.8 (3)
O(1)—Si(1)—N(2)	111.6 (2)	C(7)—C(8)—C(9)	123.9 (4)
O(1)—Si(1)—N(1)	110.4 (1)	F(3)—C(8)—C(9)	119.2 (3)
Si(1)—N(1)—C(1)	129.4 (3)	C(8)—C(9)—C(10)	118.7 (4)
Si(1)—N(2)—C(7)	130.8 (3)	C(9)—C(10)—C(11)	119.7 (4)
Si(1)—N(3)—C(13)	128.9 (3)	C(10)—C(11)—C(12)	120.3 (4)
H(3)—N(3)—C(13)	115.5 (3)	C(7)—C(12)—C(11)	121.0 (4)
N(1)—C(1)—C(6)	123.6 (3)	N(3)—C(13)—C(18)	124.0 (3)
N(1)—C(1)—C(2)	120.1 (4)	N(3)—C(13)—C(14)	120.4 (3)
C(2)—C(1)—C(6)	116.3 (3)	C(14)—C(13)—C(18)	115.6 (4)
F(2)—C(2)—C(1)	116.8 (4)	F(1)—C(14)—C(13)	116.9 (3)
C(1)—C(2)—C(3)	124.1 (4)	C(13)—C(14)—C(15)	124.5 (4)
F(2)—C(2)—C(3)	119.2 (4)	F(1)—C(14)—C(15)	118.6 (4)
C(2)—C(3)—C(4)	118.3 (4)	C(14)—C(15)—C(16)	118.0 (4)
C(3)—C(4)—C(5)	119.9 (4)	C(15)—C(16)—C(17)	119.9 (5)
C(4)—C(5)—C(6)	120.4 (4)	C(16)—C(17)—C(18)	120.7 (4)
C(1)—C(6)—C(5)	120.9 (4)	C(13)—C(18)—C(17)	121.3 (4)

rable with those in the benzyl analogue [1.613 (4) Å] (Glidewell & Liles, 1981). The bond angles about the Si atoms (Table 2) are all close to tetrahedral, whereas the angles Si—N—C show values of about 130°, which are typical for that combination of atoms (Wannagat, 1964).
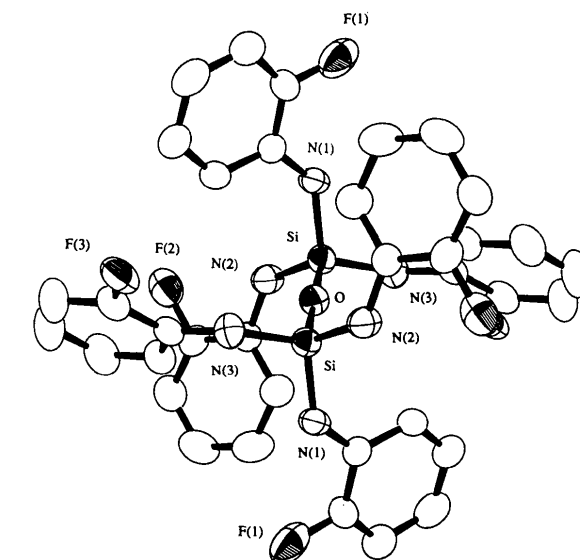

Fig. 1. Plot of the title compound showing the atomic numbering scheme with thermal ellipsoids drawn at the 50% level.

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Structure of 3,5-Dimethyl-2,2,4,4,6,6-hexakis(methylamino)-1-oxa-3,5-diaza-2,4,6-trisilacyclohexane

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Abstract. $C_8H_{30}N_8OSi_3$, $M_r = 338.6$, triclinic, $P\bar{1}$, $a = 8.984$ (6), $b = 14.899$ (20), $c = 16.561$ (22) Å, $\alpha = 113.00$ (7), $\beta = 99.63$ (7), $\gamma = 104.39$ (6)°, $V = 1888$ (4) Å³, $Z = 4$, $D_x = 1.1914$ g cm⁻³, $\lambda(Mo\ K\alpha) = 0.71069$ Å, $\mu = 2.53$ cm⁻¹, $F(000) = 736$, room temperature, $R_{int} = 0.01$, final $R = 0.060$, $wR = 0.062$ for 4928 observed reflections and 364 variables, $F(hkl) > 4\sigma(F)$. There are two independent molecules in the asymmetric unit. For each, the six-membered Si_3N_2O ring is planar. The C atoms of the methyl

Table 1. *Fractional coordinates and equivalent isotropic temperature coefficients* ($\mathring{A}^2$) *for non-H atoms*

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

	x	y	z	U_{eq}
Molecule A				
Si(1)	0.4714 (2)	0.3416 (1)	0.4261 (1)	0.046 (1)
Si(2)	0.5372 (2)	0.3678 (1)	0.6234 (1)	0.046 (1)
Si(3)	0.5016 (2)	0.1587 (1)	0.4661 (1)	0.052 (1)
O	0.4876 (5)	0.3892 (3)	0.5355 (2)	0.054 (2)
N(1)	0.4726 (6)	0.2189 (3)	0.3975 (3)	0.057 (3)
N(2)	0.5565 (6)	0.2478 (3)	0.5776 (3)	0.053 (3)
N(3)	0.6200 (6)	0.4106 (4)	0.3999 (4)	0.067 (3)
N(4)	0.7084 (6)	0.4537 (4)	0.7020 (3)	0.061 (3)
N(5)	0.6422 (6)	0.1000 (4)	0.4410 (4)	0.073 (3)
N(6)	0.3023 (6)	0.3454 (4)	0.3653 (3)	0.057 (3)
N(7)	0.3974 (6)	0.3844 (4)	0.6824 (3)	0.059 (3)
N(8)	0.3429 (7)	0.0556 (4)	0.4429 (4)	0.076 (3)
C(1)	0.4456 (10)	0.1565 (5)	0.2975 (4)	0.099 (5)
C(2)	0.6066 (9)	0.2106 (5)	0.6472 (4)	0.086 (5)
C(3)	0.7863 (9)	0.4177 (6)	0.4242 (6)	0.111 (7)
C(4)	0.8708 (8)	0.4523 (5)	0.6956 (5)	0.095 (5)
C(5)	0.8042 (9)	0.1553 (6)	0.4465 (6)	0.121 (4)
C(6)	0.1412 (8)	0.2909 (5)	0.3665 (5)	0.088 (5)
C(7)	0.2247 (8)	0.3504 (5)	0.6373 (4)	0.078 (5)
C(8)	0.1907 (10)	0.0603 (6)	0.4537 (7)	0.124 (6)
Molecule B				
Si(1)	0.6299 (2)	−0.2953 (1)	−0.0795 (1)	0.057 (1)
Si(2)	0.6954 (2)	−0.3264 (1)	0.0936 (1)	0.059 (1)
Si(3)	0.6876 (3)	−0.1138 (1)	0.1100 (1)	0.064 (1)
O	0.6738 (5)	−0.3465 (2)	−0.0121 (2)	0.058 (2)
N(1)	0.7140 (7)	−0.1991 (3)	0.1513 (3)	0.067 (3)
N(2)	0.6319 (6)	−0.1764 (4)	−0.0070 (3)	0.065 (3)
N(3)	0.4537 (7)	−0.3616 (5)	−0.1591 (4)	0.094 (4)
N(4)	0.5410 (6)	−0.4039 (4)	0.1106 (3)	0.061 (3)
N(5)	0.5455 (8)	−0.0615 (4)	0.1476 (4)	0.091 (4)
N(6)	0.7602 (7)	−0.2978 (4)	−0.1451 (3)	0.072 (3)
N(7)	0.8542 (7)	−0.3589 (5)	0.1300 (4)	0.090 (4)
N(8)	0.8523 (8)	−0.0066 (4)	0.1545 (4)	0.097 (4)
C(1)	0.7656 (11)	−0.1568 (5)	0.2539 (4)	0.113 (5)
C(2)	0.6063 (10)	−0.1098 (5)	−0.0539 (5)	0.107 (6)
C(3)	0.2993 (9)	−0.3561 (7)	−0.1438 (5)	0.124 (6)
C(6)	0.9356 (10)	−0.2619 (7)	−0.1041 (5)	0.120 (6)
C(4)	0.3792 (8)	−0.3942 (5)	0.0982 (5)	0.076 (4)
C(5)	0.3760 (10)	−0.1176 (6)	0.1233 (6)	0.118 (6)
C(7)	1.0090 (11)	−0.3283 (9)	0.1140 (4)	0.120 (6)
C(8)	1.0092 (11)	−0.0115 (7)	0.1460 (4)	0.116 (6)

groups attached to the rings are nearly coplanar. The molecules are stacked like a roll of coins along the *c* axis.

Experimental. The title compound was synthesized by the reaction of tetrakis(methylamino)silane, $Si(NHCH_3)_4$ (Andersch & Jansen, 1990b) and hexakis(methylamino)disiloxane (Bodegain, 1988) in sealed glass ampoules at 423 K. Transparent crystals were grown from the vapor phase, crystal used 0.7 × 0.5 × 0.2 mm. Automated Enraf–Nonius CAD-4 diffractometer, graphite-monochromated Mo $K\alpha$ radiation. 25 centered reflections within $4 < \theta < 15°$ used for determining lattice parameters. Data corrected for Lorentz and polarization effects, absorption ignored. $2\theta_{max} = 52°$, range of *hkl*: $0 \le h \le 11$, $-18 \le k \le 18$, $-20 \le l \le 20$. Two check reflections measured every 200 reflections showed no significant intensity variation during data collection. $\omega/2\theta$-scan technique. 7948 reflections measured, 6916 unique (non-zero), 4928 observed with $F(hkl) > 4\sigma(F)$. Structure solved by direct methods using *SHELXS86*

Table 2. *Bond lengths* ($\mathring{A}$) *and angles* (°)

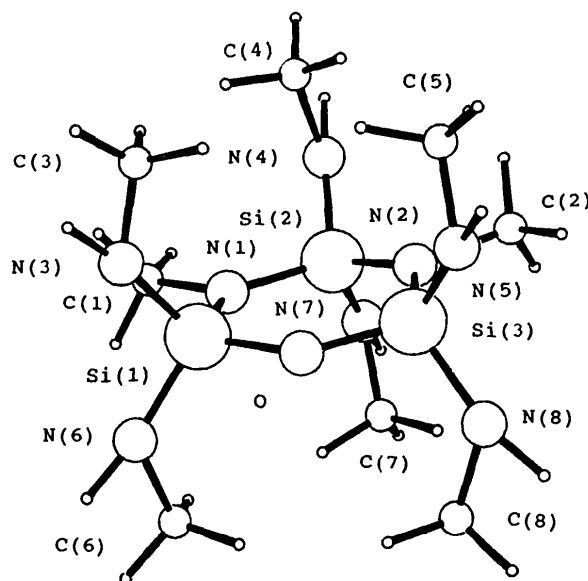|	Molecule A	Molecule B		Molecule A	Molecule B
Si(1)—O	1.632 (5)	1.629 (5)	Si(3)—N(5)	1.725 (7)	1.734 (8)
Si(1)—N(1)	1.705 (6)	1.702 (5)	Si(3)—N(8)	1.675 (6)	1.681 (6)
Si(1)—N(3)	1.700 (6)	1.651 (6)	N(1)—C(1)	1.489 (7)	1.502 (8)
Si(1)—N(6)	1.703 (6)	1.722 (6)	N(2)—C(2)	1.514 (10)	1.514 (11)
Si(2)—O	1.624 (5)	1.625 (5)	N(3)—C(3)	1.446 (10)	1.467 (11)
Si(2)—N(2)	1.713 (5)	1.706 (6)	N(4)—C(4)	1.486 (9)	1.484 (10)
Si(2)—N(4)	1.659 (5)	1.711 (6)	N(5)—C(5)	1.452 (10)	1.441 (10)
Si(2)—N(7)	1.723 (6)	1.708 (8)	N(6)—C(6)	1.481 (9)	1.473 (10)
Si(3)—N(1)	1.724 (6)	1.706 (7)	N(7)—C(7)	1.465 (8)	1.455 (12)
Si(3)—N(2)	1.694 (5)	1.704 (5)	N(8)—C(8)	1.423 (11)	1.456 (13)
O—Si(1)—N(1)	103.6 (5)	104.1 (5)	N(5)—Si(3)—N(1)	109.7 (5)	110.6 (5)
O—Si(1)—N(3)	114.3 (5)	114.7 (6)	N(5)—Si(3)—N(2)	112.6 (5)	111.7 (5)
N(1)—Si(1)—N(3)	111.6 (5)	110.0 (6)	Si(1)—O—Si(2)	141.0 (6)	140.7 (6)
N(6)—Si(1)—O	111.5 (5)	109.8 (5)	Si(1)—N(1)—Si(3)	130.0 (5)	130.3 (6)
N(6)—Si(1)—N(1)	113.6 (5)	116.9 (6)	Si(3)—N(1)—C(1)	116.8 (7)	115.1 (6)
N(6)—Si(1)—N(3)	102.7 (5)	101.8 (5)	Si(1)—N(1)—C(1)	113.3 (7)	113.8 (6)
O—Si(2)—N(2)	104.4 (4)	104.2 (5)	Si(2)—N(2)—Si(3)	129.2 (2)	129.9 (5)
N(4)—Si(2)—O	114.4 (6)	115.3 (5)	Si(3)—N(2)—C(2)	115.5 (6)	116.3 (7)
N(4)—Si(2)—N(2)	109.3 (5)	110.4 (5)	Si(2)—N(2)—C(2)	114.7 (6)	113.8 (7)
N(7)—Si(2)—O	108.1 (5)	109.6 (5)	Si(1)—N(3)—C(3)	124.3 (7)	124.6 (6)
N(7)—Si(2)—N(2)	118.4 (6)	115.8 (5)	Si(2)—N(4)—C(4)	124.5 (6)	121.8 (7)
N(7)—Si(2)—N(4)	102.7 (5)	102.1 (6)	Si(3)—N(5)—C(5)	123.6 (8)	126.1 (7)
N(1)—Si(3)—N(8)	114.3 (5)	112.5 (5)	Si(1)—N(6)—C(6)	120.7 (7)	121.8 (6)
N(2)—Si(3)—N(1)	108.9 (5)	108.9 (5)	Si(2)—N(7)—C(7)	123.5 (6)	123.6 (8)
N(2)—Si(3)—N(8)	110.9 (5)	111.7 (5)	Si(3)—N(8)—C(8)	124.5 (8)	122.2 (8)
N(5)—Si(3)—N(8)	100.4 (5)	101.4 (5)			

Fig. 1. Perspective view of the title molecule with atomic numbering scheme.
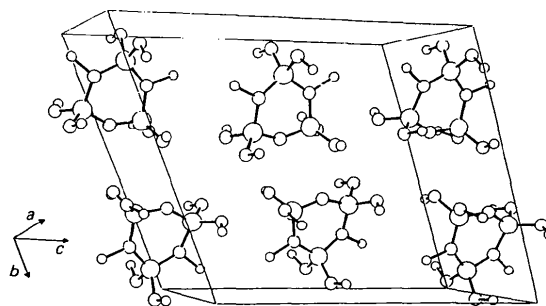

Fig. 2. Crystal packing of the title molecule.

(Sheldrick, 1986), full-matrix least-squares refinement on F of 364 parameters using *SHELX76* (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. $R = 0.060$, $wR = 0.062$, S was not calculated, $(\Delta/\sigma)_{max} = 0.001$, $(\Delta\rho)_{max} = 0.426$, $(\Delta\rho)_{min} = -0.386$ e Å^{-3}, unit weights; atomic scattering factors as incorporated in *SHELX76*. Table 1* lists atomic positional and equivalent isotropic thermal parameters. Interatomic distances and valence angles are in Table 2. Fig. 1 shows a perspective view of the molecule and Fig. 2 shows the crystal packing.

Related literature. The title compound can be regarded as a heterosilazane analog of 1,3,5-trimethyl-2,2,4,4,6,6-hexakis(methylamino)cyclotri-

silazane (Andersch & Jansen, 1990a) in which one methylamino group is substituted by an O atom with the six-membered ring being planar. A related siloxazane with respect to the Si_3N_2O core has been reported: 2,2,4,4,6,6-hexamethylcyclotrisiloxazane (Andrianov, Haiduc & Khananashvili, 1963a,b) has been synthesized by co-ammonolysis of 1,3-dichloro-tetramethyldisiloxane. An alternative synthesis is by a substitution reaction in the six-membered ring (Wannagat, Bodgusch & Höfler, 1967).

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Structure of [(4,6-Dimethyl-2-pyrimidinyl)thio]acetic Acid Hydrate

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Abstract. $C_8H_{10}N_2O_2S.H_2O$, $M_r = 216.26$, monoclinic, $P2_1/c$, $a = 7.652$ (1), $b = 10.741$ (1), $c = 13.267$ (1) Å, $\beta = 100.80$ (1)°, $V = 1071.7$ Å^3, $Z = 4$, $D_x = 1.341$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 2.739$ cm^{-1}, $F(000) = 456$, $T = 293$ K, final $R = 0.047$ for 1569 unique observed $[I > 3\sigma(I)]$ reflections. The S—C bond lengths are 1.758 (3) and 1.789 (3) Å. The thiopyrimidine ring is planar. The crystal cohesion is due to hydrogen bonds involving water molecules.

Experimental. A colorless needle-like crystal (0.15 × 0.22 × 0.59 mm) of the title compound was synthe-

sized by a reported method (Boarland, McOmie & Timms, 1952) and recrystallized from H_2O. Reflection data were collected on an Enraf–Nonius CAD-4 diffractometer with graphite-monochromatized Mo $K\alpha$ radiation. Cell dimensions were refined by 25 accurately centered reflections in the range $22 < 2\theta < 32°$. Intensities were measured using an ω–2θ scan of 0.79 to 8.24° min^{-1}; scan width (0.45 + 0.35tanθ)°; θ range 1–25° ($h = 0$ to 9, $k = 0$ to 13, $l = -16$ to 16). Three standard reflections monitored periodically showed no significant variation in intensity. Of the 2000 independent reflections measured ($R_{int} = 0.018$), 1569 with $I > 3\sigma(I)$ were used for the calculations. Lp and ψ-scan absorption corrections

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