

those about C(3)–C(4) and C(6)–C(7) which are antiperiplanar and synclinal respectively, the first favouring extension of the chain along the longer side of the rectangular cavity, the second favouring the folding of the shorter one; both push the two S atoms out of the cavity.

The molecules lie almost on planes perpendicular to the *c* axis; the crystal packing is regulated by normal van der Waals contacts.

This work was supported by CNR; computations were performed on the Univac 1100 of the Centro di Calcolo, University of Cagliari.

*Acta Cryst.* (1984). C40, 433–434

## Structure of 2,2,4,4,6,6-Hexa-*tert*-butylcyclotrisilazane, [(C<sub>4</sub>H<sub>9</sub>)<sub>2</sub>SiNH]<sub>3</sub>

BY WILLIAM CLEGG, GEORGE M. SHELDRIK AND DIETMAR STALKE

*Institut für Anorganische Chemie der Universität, Tammannstrasse 4, D-3400 Göttingen, Federal Republic of Germany*

(Received 20 September 1983; accepted 24 October 1983)

**Abstract.**  $M_r = 472.0$ , rhombohedral, space group  $R\bar{3}c$ ,  $a = 10.227(1)$ ,  $c = 49.051(7)$  Å on hexagonal axes,  $U = 4443.0$  Å<sup>3</sup>,  $Z = 6$ ,  $D_x = 1.058$  Mg m<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.71069$  Å,  $\mu = 0.17$  mm<sup>-1</sup>,  $T = 291$  K,  $F(000) = 1584$ ,  $R = 0.060$  for 736 observed reflections. The Si<sub>3</sub>N<sub>3</sub> ring is planar, with internal angles at Si and N of 104.1 (2) and 135.9 (2)°. The Si–N bond length is 1.727 (2) Å.

**Introduction.** Compounds of the type  $R_2\text{SiXY}$ , in which  $X$  and  $Y$  are the functional groups OH or NH<sub>2</sub> and  $R$  is an alkyl substituent, readily undergo condensation reactions to give chain and ring siloxanes (Si–O–Si) and silazanes (Si–N–Si); when  $R$  is *tert*-butyl (Bu), such condensation is not observed, probably because of the steric bulk of the Bu substituents (Sommer & Tyler, 1954). An indirect synthetic route is required, and has been achieved *via* the intermediates Bu<sub>2</sub>SiFOH and Bu<sub>2</sub>SiFNH<sub>2</sub> (Klingebiel, 1981; Clegg, Klingebiel & Sheldrick, 1982; Klingebiel & Vater, 1983). The cyclotrisiloxane (Bu<sub>2</sub>SiO)<sub>3</sub> contains a planar Si<sub>3</sub>O<sub>3</sub> ring (Clegg, 1982). The cyclotrisilazane (Bu<sub>2</sub>SiNH)<sub>3</sub> is found to be isostructural with it.

**Experimental.** Crystal obtained from *n*-hexane, 0.2 × 0.2 × 0.3 mm. Stoe–Siemens AED diffractometer. Unit-cell parameters from 2θ values of 38 reflections (20 < 2θ < 25°). 2049 reflections with 2θ < 55° and all indices ≥ 0. Profile analysis on-line (Clegg, 1981). No

### References

- COSTA BIZZARRI, P., DELLA CASA, C., FERRUTI, P., GHEDINI, N., PILATI, P. & SCAPINI, G. (1983). *Polymer*. In the press.  
 GOLDBERG, I. (1981). *Acta Cryst.* B37, 102–107.  
*International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press.  
 MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1980). *MULTAN80. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.  
 SHELDRIK, G. M. (1981). *SHELXTL. An Integrated System for Solving, Refining and Displaying Crystal Structures from Diffraction Data*. Univ. of Göttingen, Federal Republic of Germany.

significant variation for three standard reflections. No absorption corrections.  $R_{\text{int}} = 0.017$ , 1137 unique reflections, 736 with  $F > 4\sigma(F)$ . Automatic multi-solution direct methods. Blocked-cascade refinement on  $F$ ;  $w^{-1} = \sigma^2(F) + 0.0004F^2$ . Bu H atoms constrained to give C–H = 0.96 Å, H–C–H = 109.5°,  $U(\text{H}) = 1.2U(\text{C})$ ; N–H refined freely subject to space-group symmetry constraints, with isotropic  $U$ . Anisotropic thermal parameters for non-H atoms. No extinction correction. Scattering factors from *International Tables for X-ray Crystallography* (1974). 58 parameters,  $R = 0.060$ ,  $wR = 0.058$ . Slope of normal probability plot = 1.32.  $(\Delta/\sigma)_{\text{max}} = 0.006$ ,  $(\Delta/\sigma)_{\text{mean}} = 0.002$ . Largest peak in final difference map 0.35 e Å<sup>-3</sup>, largest hole –0.29 e Å<sup>-3</sup>. Programs used: *SHELXTL* (Sheldrick, 1978); diffractometer control program by WC.

**Discussion.** Atomic coordinates are given in Table 1,\* bond lengths and angles in Table 2. The molecular structure is shown in Fig. 1. (Bu<sub>2</sub>SiNH)<sub>3</sub> is isostructural with (Bu<sub>2</sub>SiO)<sub>3</sub> (Clegg, 1982), three N–H groups replacing the three O atoms. The Si<sub>3</sub>N<sub>3</sub> ring is

\* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38978 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Atomic coordinates ( $\times 10^4$ ) and equivalent isotropic thermal parameters ( $\text{\AA}^2 \times 10^3$ )
$$U = \frac{1}{3}(\text{trace of the orthogonalized } U_{ij} \text{ matrix}).$$

	x	y	z	U
Si	1807 (1)	0	7500	37 (1)
N	0	-1537 (3)	7500	40 (1)
H	0	-2155 (39)	7500	36 (15)
C(1)	2869 (3)	-26 (3)	7822 (1)	46 (1)
C(2)	4585 (3)	1034 (4)	7807 (1)	67 (2)
C(3)	2571 (4)	-1634 (3)	7878 (1)	61 (2)
C(4)	2334 (4)	482 (4)	8069 (1)	65 (2)

Table 2. Bond lengths ( $\text{\AA}$ ) and angles ( $^\circ$ )

The prime denotes a symmetry-generated atom.

Si-N	1.727 (2)	Si-C(1)	1.924 (3)
N-H	0.631 (40)	C(1)-C(2)	1.535 (4)
C(1)-C(3)	1.541 (5)	C(1)-C(4)	1.523 (5)
N-Si-N'	104.1 (2)	N-Si-C(1)	110.0 (1)
N-Si-C(1')	111.1 (1)	C(1)-Si-C(1')	110.3 (2)
Si-N-Si'	135.9 (2)	Si-N-H	112.1 (1)
Si-C(1)-C(2)	113.9 (2)	Si-C(1)-C(3)	110.5 (2)
Si-C(1)-C(4)	110.3 (3)	C(2)-C(1)-C(3)	108.0 (3)
C(2)-C(1)-C(4)	106.0 (3)	C(3)-C(1)-C(4)	108.0 (3)

strictly planar as a consequence of the crystallographic 32 ( $D_3$ ) symmetry of the molecule. Almost planar rings have been observed in other cyclotrisilazanes in which the substituents on N are not bulky (Rozsondai, Hargittai, Golubinskii, Vilkov & Mastuykov, 1975; Clegg, Noltemeyer, Sheldrick & Vater, 1980). Bulky N substituents (Adamson & Daly, 1970), including N-X-N bridges between cyclotrisilazane rings (Clegg, Noltemeyer, Sheldrick & Vater, 1981; Clegg, 1983), lead to non-planar rings, which may also be produced by other constraints, such as fusion of the ring to a planar cyclodisilazane ring (Clegg, 1980; Clegg, Klingebiel, Sheldrick, Skoda & Vater, 1980). In such cases, the ring usually approximates to a boat conformation, but deviations from the ideal boat can be large (Clegg, 1983).

The Si-N bond length and the N-Si-N and Si-N-Si angles in  $(\text{Bu}_2\text{SiNH})_3$  are similar to those in other cyclotrisilazanes.

*Acta Cryst.* (1984). C40, 434-436

### Structure of 2'-Deoxy-2'-fluoroadenosine (dAf): $\text{C}_{10}\text{H}_{12}\text{N}_5\text{O}_3\text{F}$

By KAORU MORISHITA, TOSHIO HAKOSHIMA, TAKAJI FUJIWARA, KEN-ICHI TOMITA, TOSHINORI KANEYASU, SEIICHI UESUGI AND MORIO IREHARA

Faculty of Pharmaceutical Sciences, Osaka University, 1-6 Yamadaoka, Suita, Osaka 565, Japan

(Received 7 September 1983; accepted 24 October 1983)

**Abstract.**  $M_r = 269.2$ , monoclinic,  $P2_1$ ,  $a = 4.791$  (2),  $b = 10.428$  (2),  $c = 11.507$  (2)  $\text{\AA}$ ,  $\beta = 102.06$  (1) $^\circ$ ,  $V = 562.3$  (1)  $\text{\AA}^3$ ,  $Z = 2$ ,  $D_m = 1.576$  (4),  $D_x = 1.590$   $\text{Mg m}^{-3}$ , graphite-monochromated  $\text{Cu K}\alpha$ ,  $\lambda = 1.54173$   $\text{\AA}$ ,  $\mu(\text{Cu K}\alpha) = 1.15$   $\text{mm}^{-1}$ ,  $F(000) = 280$ ,  $T$

0108-2701/84/030434-03\$01.50

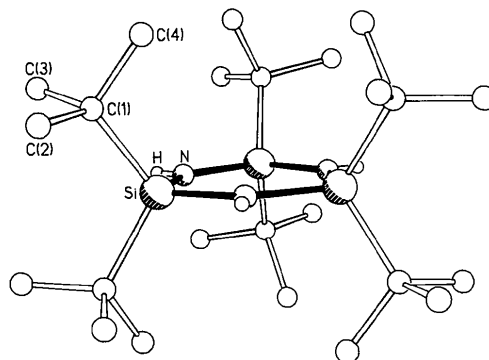


Fig. 1. The molecular structure, showing the atom-numbering scheme. *tert*-Butyl H atoms are omitted.

We thank Drs U. Klingebiel and N. Vater for supplying crystals, and the Verband der Chemischen Industrie for financial support.

#### References

- ADAMSON, G. W. & DALY, J. J. (1970). *J. Chem. Soc. A*, pp. 2724-2728.
- CLEGG, W. (1980). *Acta Cryst.* B36, 2830-2832.
- CLEGG, W. (1981). *Acta Cryst.* A37, 22-28.
- CLEGG, W. (1982). *Acta Cryst.* B38, 1648-1649.
- CLEGG, W. (1983). *Acta Cryst.* B39, 387-389.
- CLEGG, W., KLINGEBIEL, U. & SHELDRIK, G. M. (1982). *Z. Naturforsch. Teil B*, 37, 423-431.
- CLEGG, W., KLINGEBIEL, U., SHELDRIK, G. M., SKODA, L. & VATER, N. (1980). *Z. Naturforsch. Teil B*, 35, 1503-1507.
- CLEGG, W., NOLTEMEYER, M., SHELDRIK, G. M. & VATER, N. (1980). *Acta Cryst.* B36, 2461-2462.
- CLEGG, W., NOLTEMEYER, M., SHELDRIK, G. M. & VATER, N. (1981). *Acta Cryst.* B37, 986-987.
- International Tables for X-ray Crystallography* (1974). Vol. IV, pp. 99, 149. Birmingham: Kynoch Press.
- KLINGEBIEL, U. (1981). *Chem. Ber.* 114, 3366-3371.
- KLINGEBIEL, U. & VATER, N. (1983). *Chem. Ber.* 116, 3277-3282.
- ROZSONDAI, B., HARGITTAI, I., GOLUBINSKII, A. V., VILKOV, L. V. & MASTUYKOV, V. S. (1975). *J. Mol. Struct.* 28, 339-348.
- SHELDRIK, G. M. (1978). *SHELXTL. An Integrated System for Solving, Refining and Displaying Crystal Structures from Diffraction Data*. Univ. of Göttingen, Federal Republic of Germany.
- SOMMER, L. H. & TYLER, L. J. (1954). *J. Am. Chem. Soc.* 76, 1030-1033.