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2,4,6-Trifluoro-1,3,5-trimethyl-2,4,6-triphenylcyclotrisilazane

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Abstract. $C_{21}H_{24}F_3N_3Si_3$, $M_r = 459{\cdot}7$, trigonal, $R3$, $a = 8{\cdot}589$ (2) Å, $\alpha = 102{\cdot}53$ (2)°, $U = 580{\cdot}2$ Å^3, $Z = 1$, $D_x = 1{\cdot}316$ Mg m^{-3}; final $R = 0{\cdot}055$ for 459 reflexions. The molecule has a crystallographic threefold axis. The central Si_3N_3 ring is almost planar with Si—N = 1·700 (6) Å, and internal angles of 110·1 (5) and 129·8 (5)° at Si and N.

Introduction. This structure was determined as part of a study of Si—N derivatives. Crystals were obtained by diffusing light petroleum ether into a solution in benzene. They were of only moderate quality and deteriorated gradually when exposed to X-rays.

Intensities of reflexions in the range $7 \leq 2\theta \leq 45°$ were measured from two crystals on a Stoe–Siemens four-circle diffractometer with monochromated Mo $K\alpha$ radiation (2349 reflexions, including many symmetry equivalents). After averaging of equivalent reflexions, assuming Friedel's law, 459 unique reflexions with $I > 2\sigma(I)$ were used for structure solution and refinement. Cell dimensions were obtained from 19 centred reflexions.

The structure was solved by direct methods. All atoms other than H were refined anisotropically. H atoms were included in the final refinement, with fixed C—H = 0·96 Å, constrained angles (CH_3 as a rigid group with H—C—H = 109·5°, phenyl H atoms on external bisectors of C—C—C angles), and isotropic thermal parameters set at 1·2 times the equivalent isotropic value for the corresponding C atom. The weighting scheme was $w^{-1} = \sigma^2(F) + 0{\cdot}001F^2$. The final value of R was 0·055, and $R_w = (\sum w\Delta^2/\sum wF_o^2)^{1/2}$ was 0·062. Refined coordinates are given in Table 1, bond lengths and angles in Table 2.*

Table 1. *Atomic coordinates* (×10^4) *and isotropic thermal parameters* (Å^2 × 10^3)

Equivalent isotropic U values were calculated from the anisotropic U_{ij} components.

	x	*y*	*z*	*U*
Si	1573 (4)	3875 (5)	3866 (4)	50 (1)
N	2464 (8)	4536 (8)	2449 (8)	54 (3)
F	−326 (7)	2986 (7)	3003 (7)	78 (2)
C(1)	1609 (12)	5616 (12)	5581 (12)	57 (4)
C(2)	362 (13)	5547 (13)	6379 (13)	77 (5)
C(3)	448 (18)	6878 (17)	7687 (15)	93 (6)
C(4)	1726 (20)	8240 (16)	8180 (14)	101 (6)
C(5)	3032 (15)	8356 (12)	7441 (13)	86 (5)
C(6)	2936 (12)	7049 (11)	6180 (11)	66 (4)
C(7)	1749 (14)	5720 (12)	1697 (13)	72 (4)

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

The prime denotes an atom generated by the threefold rotation axis.

Si—F	1·576 (6)	Si—C(1)	1·847 (11)
Si - N	1·705 (9)	Si—N′	1·696 (9)
C(1)—C(2)	1·389 (17)	C(2)—C(3)	1·395 (17)
C(3)—C(4)	1·334 (18)	C(4)—C(5)	1·399 (22)
C(5)—C(6)	1·355 (13)	C(6)—C(1)	1·393 (12)
N—C(7)	1·488 (14)		
F—Si—C(1)	104·7 (4)	F—Si—N	108·9 (4)
C(1)—Si—N	112·3 (5)	F—Si—N′	107·6 (4)
C(1)—Si—N′	113·0 (5)	N—Si—N′	110·1 (5)
Si—C(1)—C(2)	122·3 (7)	Si—C(1)—C(6)	121·1 (9)
C(2)—C(1)—C(6)	116·6 (9)	C(1)—C(2)—C(3)	120·5 (10)
C(2)—C(3)—C(4)	120·3 (14)	C(3)—C(4)—C(5)	121·4 (12)
C(4)—C(5)—C(6)	117·6 (9)	C(1)—C(6)—C(5)	123·5 (11)
Si—N—C(7)	115·5 (7)	Si—N—Si′	129·8 (5)
C(7)—N—Si′	114·2 (7)		

Because $R3$ is a polar space group, the question of absolute polarity arises. With the data from one crystal only, and not assuming Friedel's law, all parameters were refined for both polarities. The two results, as assessed by R indices, refined parameters, and molecular geometries, were insignificantly different.

Discussion. The preparation of the compound has been described (Klingebiel, Neemann & Meller, 1977). The molecule (Fig. 1) has crystallographic 3 (C_3) symmetry. The average Si—N length [1·700 (6) Å] is rather short, as expected when Si is also bonded to the very electronegative F atom.

The Si_3N_3 ring is close to planar: the N atoms lie 0·050 (5) Å from the plane of the Si atoms, and the N—Si—N—Si torsion angles are alternately +6·0 (7)

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* Lists of structure factors, anisotropic thermal parameters and H atom positional parameters have been depositied with the British Library Lending Division as Supplementary Publication No. SUP 35362 (6 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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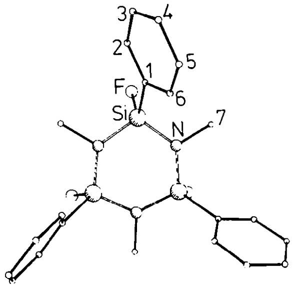

Fig. 1. Structure of the molecule, showing the atom numbering.

and −6·0 (7)°. The slight deviation from planarity is towards a chair rather than a boat conformation (as required by the crystallographic symmetry).

Previous attempts to determine the conformation of Si_3N_3 rings have been fraught with problems. $(SiCl_2.NH)_3$ (Mootz, Fayos & Zinnius, 1972) was found to be disordered in such a way that the average structure determined has a planar ring. An electron diffraction study of $(SiMe_2.NH)_3$ 'does not yield unambiguous information on the ring conformation ... except that the deviation from planarity is not very large' (Rozsondai, Hargittai, Golubinskii, Vilkov & Mastyukov, 1975); chair and boat models were found to fit the data equally well, but a twist-boat conformation was ruled out. $(SiMe_2.NSiMe_3)_3$ (Adamson & Daly, 1970) has a boat conformation but this is probably due to the bulky $SiMe_3$ substituents. Spectroscopic evidence (see references in Rozsondai *et al.*, 1975) is ambiguous and, indeed, contradictory.

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References

Adamson, G. W. & Daly, J. J. (1970). *J. Chem. Soc. A*, pp. 2724–2728.

Klingebiel, U., Neemann, J. & Meller, A. (1977). *Z. Anorg. Allg. Chem.* **429**, 63–68.

Mootz, D., Fayos, J. & Zinnius, A. (1972). *Angew. Chem.* **84**, 27–28; *Angew. Chem. Int. Ed. Engl.* **11**, 58–59.

Rozsondai, B., Hargittai, I., Golubinskii, A. V., Vilkov, L. V. & Mastyukov, V. S. (1975). *J. Mol. Struct.* **28**, 339–348.

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1,8-Dibenzoyl-2,7-dimethylnaphthalene

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Abstract. $C_{26}H_{20}O_2$, $M_r = 364$, monoclinic, $P2_1/n$, $a = 15 \cdot 024$ (3), $b = 14 \cdot 101$ (2), $c = 9 \cdot 375$ (2) Å, $\beta = 90 \cdot 42$ (1)°, $U = 1986 \cdot 1$ Å^3, $Z = 4$, $D_c = 1 \cdot 217$ Mg m^{-3}, $\mu(\text{Mo } K\alpha) = 0 \cdot 041$ mm^{-1}, $F(000) = 832$. The structure was solved from diffractometer data by direct methods and gave a final $R = 0 \cdot 072$ for 1234 observed reflections. The molecule has the *transoid* configuration for the 1,8-dibenzoyl groups.

Introduction. Although *peri* interactions in 1,8-disubstituted naphthalenes are well known (Anderson, Franck & Mandella, 1972; Herbstein, 1979), little information is available on the detailed molecular structure of compounds possessing additional neighbouring groups. White, Carnduff, Guy & Bovill (1977*a*) have discussed the parameters thought to be indicative of *peri* strain in 1,8-disubstituted naphthalenes, and concluded that the only consistent indicator of steric crowding is the C(1)–C(9)–C(8) angle $[\theta_{1-9-8}]$, which opens with respect to the normal angle in naphthalene itself (121·5°, Cruickshank, 1957). The effect of neighbouring substituents in the 2 and 7 positions on the geometry of 1,8-naphthalene derivatives has not been studied, nor have cases where rotation of the groups away from the naphthalene ring can result in geometrical isomerism. In the title compound the acyl groups cannot be expected to be coplanar with the naphthalene moiety, and two geometric isomers are possible. A single isomer of unknown configuration was obtained by synthesis (Gore, Miri & Siddiquei, 1973).

Data were collected from a crystal 0·26 × 0·32 × 0·18 mm. 3498 intensities were recorded (3·0 <

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