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Bis(ethylamino)tetrafluorosilicon

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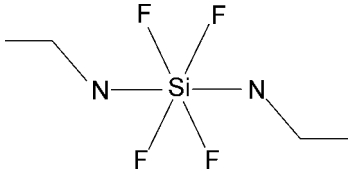
Key indicators: single-crystal X-ray study; T = 150 K; mean σ(C–C) = 0.004 Å; R factor = 0.034; wR factor = 0.042; data-to-parameter ratio = 11.2.

The title compound, $SiF_4(NH_2Et)_2$ or $C_4H_{14}F_4N_2Si$, was isolated as an intermediate in the attempted solvothermal synthesis of a nitride-containing microporous material. Its structure at 150 K displays intermolecular hydrogen bonding leading to infinite sheets of molecules (each of $\overline{1}$ point symmetry) running parallel to the *bc* plane.

Related literature

For related literature, see: Ennan & Kats (1974).



Experimental

Crystal data

$C_4H_{14}F_4N_2Si$
M_r = 194.25
Monoclinic, $P2_1/c$
a = 8.4788 (4) Å
b = 6.9602 (3) Å
c = 7.2206 (4) Å
β = 99.655 (2)°
V = 420.08 (4) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.29 mm^{-1}
T = 150 K
0.24 × 0.20 × 0.05 mm

Data collection

Nonius KappaCCD area-detector diffractometer
Absorption correction: multi-scan (*DENZO/SCALEPACK*; Otwinowski & Minor, 1997)
T_{min} = 0.93, T_{max} = 0.99
4140 measured reflections
946 independent reflections
670 reflections with $I > 3\sigma(I)$
R_{int} = 0.042

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.042
S = 1.07
670 reflections
60 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···F1^{ii}	0.81 (3)	2.15 (3)	2.938 (2)	165 (3)
N1—H2···F2^{iii}	0.81 (3)	2.29 (3)	3.091 (2)	168 (3)

Symmetry codes: (ii) $x, -y+\frac{1}{2}, z-\frac{1}{2}$; (iii) $x, -y+\frac{3}{2}, z-\frac{1}{2}$.

Data collection: *KappaCCD Server Software* (Nonius, 1997); cell refinement: *DENZO* (Otwinowski & Minor, 1997); data reduction: *DENZO*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *ATOMS* (Dowty, 2005); software used to prepare material for publication: *CRYSTALS*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: MG2025).

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supplementary materials

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Comment

The molecule is located on a centre of symmetry (Fig. 1). Intramolecular hydrogen bonds (N—H···F) link the molecules to form infinite sheets running parallel to the *bc* plane (Fig. 2). A third relatively short interaction (N1···F1 3.054 (2) Å) is apparently not a hydrogen bond and the largest N—H···F angle is *ca* 130°. The secondary C atom C1 has relatively large displacement parameters compared to its neighbours, suggesting that there may be some unresolved disorder of the ethyl group. Related adducts of silicon tetrafluoride are described in Ennan & Kats (1974).

Experimental

All manipulations were carried out in the absence of atmospheric oxygen or moisture in either an Aldrich glove bag or using standard Schlenk line techniques and house nitrogen. Starting materials were obtained from Aldrich and Et_3N was dried over 3 Å molecular sieves. 1.5996 g Si_3N_4, 1.27 ml $Et_3N{\cdot}3HF$ and 7 ml Et_3N (molar ratio 1 Si: 1 F: 12 Et_3N) were combined and stirred to homogeneity. The mixture was placed inside a 25 ml teflon-lined autoclave and heated for 12 days at 240 °C. The autoclave was then cooled by removal from the oven and the mixture washed with dry Et_3N and dried under vacuum. Products were stored in a Glovebox Technology argon-filled dry box. Powder X-ray diffraction showed Si_3N_4 and $SiF_4(NHEt_2)_2$ to be the major phases, with $SiF_4(NH_2Et)_2$ corresponding to approximately 15% of the product by reference to the observed intensities.

Refinement

The NH hydrogen atoms were located in a difference Fourier map and their coordinates and isotropic displacement parameters subsequently refined. Other hydrogen atoms were positioned geometrically after each cycle of refinement. Examination of the packing of the structure shows that there are additional pseudosymmetry operators which would correspond to the (non-standard) space group A2/a, but in order to adopt this symmetry all atoms other than Si would be required to be disordered. Examination of the original intensity data clearly shows no absences indicative of the A-centring, confirming the original symmetry assignment.

Figures

Fig. 1. Molecular structure of the title compound. Displacement ellipsoids are drawn at the 40% probability level.

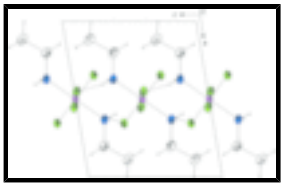

Fig. 2. Packing diagram as viewed down the *b* axis, showing the formation of infinite sheets *via* H-bonding. Displacement ellipsoids are for Si (pink), F (green), N (blue), C (white), Hydrogen atoms are shown as small white spheres. Intermolecular hydrogen bonds are indicated by dashed lines.

bis(ethylamine)tetrafluorosilicon

Crystal data

$C_4H_{14}F_4N_2Si$	F_{000} = 204
M_r = 194.25	D_x = 1.536 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 4140 reflections
a = 8.4788 (4) Å	θ = 5–28º
b = 6.9602 (3) Å	μ = 0.29 mm^{-1}
c = 7.2206 (4) Å	T = 150 K
β = 99.655 (2)º	Plate, colourless
V = 420.08 (4) Å^3	0.24 × 0.20 × 0.05 mm
Z = 2	

Data collection

Nonius KappaCCD area-detector diffractometer	670 reflections with $I > 3\sigma(I)$
Monochromator: graphite	R_{int} = 0.042
T = 150 K	θ_{max} = 27.5º
ω scans	θ_{min} = 5.7º
Absorption correction: multi-scan (DENZO/SCALEPACK; Otwinowski & Minor, 1997)	h = −10→10
T_{min} = 0.93, T_{max} = 0.99	k = −8→8
4140 measured reflections	l = −9→9
946 independent reflections	

Refinement

Refinement on F	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)]$ = 0.034	Method, part 1, Chebychev polynomial, (Watkin, 1994, *P*rince, 1982) [*w*eight] = 1.0/[A_0*T_0(x) + A_1*T_1(x) ··· + A_{n-1}]*T_{n-1}(x)] where A_i are the Chebychev coefficients listed below and x = F /Fmax Method = Robust Weighting (*P*rince, 1982) W = [*w*eight] * [1-(delta$F$/6*sigma$F$)^2]^2 A_i are: 1.21 0.411 0.931

$wR(F^2) = 0.042$	$(\Delta/\sigma)_{max} = 0.001$
$S = 1.07$	$\Delta\rho_{max} = 0.24$ e Å^{-3}
670 reflections	$\Delta\rho_{min} = -0.31$ e Å^{-3}
60 parameters	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Si1	0.5000	0.5000	0.5000	0.0222
F1	0.54851 (14)	0.26642 (17)	0.51774 (15)	0.0295
F2	0.65474 (14)	0.55391 (18)	0.66675 (15)	0.0293
N1	0.6294 (2)	0.5299 (3)	0.3080 (2)	0.0254
C1	0.7992 (3)	0.4765 (6)	0.3436 (4)	0.0634
C2	0.8842 (3)	0.5125 (4)	0.1797 (4)	0.0452
H1	0.595 (4)	0.463 (4)	0.219 (5)	0.042 (8)*
H2	0.622 (3)	0.638 (5)	0.266 (4)	0.038 (7)*
H11	0.8538	0.5527	0.4533	0.0774*
H12	0.8073	0.3365	0.3748	0.0774*
H21	0.9988	0.4730	0.2139	0.0559*
H22	0.8783	0.6524	0.1475	0.0559*
H23	0.8319	0.4361	0.0690	0.0559*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Si1	0.0300 (4)	0.0208 (4)	0.0167 (3)	0.0004 (3)	0.0061 (2)	−0.0001 (3)
F1	0.0445 (7)	0.0213 (6)	0.0249 (5)	0.0039 (5)	0.0124 (4)	0.0028 (4)
F2	0.0316 (6)	0.0361 (7)	0.0200 (6)	−0.0021 (5)	0.0035 (4)	−0.0027 (5)
N1	0.0337 (9)	0.0229 (10)	0.0206 (8)	0.0002 (7)	0.0073 (6)	0.0010 (7)
C1	0.0404 (13)	0.117 (3)	0.0363 (13)	0.0222 (16)	0.0156 (10)	0.0181 (16)
C2	0.0438 (12)	0.0520 (15)	0.0439 (14)	−0.0004 (12)	0.0199 (10)	−0.0006 (11)

Geometric parameters (Å, °)

Si1—F1	1.6766 (12)	N1—H2	0.81 (3)
Si1—F1^{i}	1.6766 (12)	C1—C2	1.506 (3)
Si1—F2	1.6679 (11)	C1—H11	1.000
Si1—F2^{i}	1.6679 (11)	C1—H12	1.000
Si1—N1	1.9183 (17)	C2—H21	1.000
Si1—N1^{i}	1.9183 (17)	C2—H22	1.000
N1—C1	1.467 (3)	C2—H23	1.000
N1—H1	0.81 (3)		
F1—Si1—F1^{i}	180.	C1—N1—H1	102 (2)
F1—Si1—F2	90.56 (6)	Si1—N1—H2	110.6 (20)
F1^{i}—Si1—F2	89.44 (6)	C1—N1—H2	108.4 (20)

F1—Si1—F2^{i}	89.44 (6)	H1—N1—H2	104 (3)
F1^{i}—Si1—F2^{i}	90.56 (6)	N1—C1—C2	113.9 (2)
F2—Si1—F2^{i}	180.	N1—C1—H11	108.357
F1—Si1—N1	89.86 (6)	C2—C1—H11	108.357
F1^{i}—Si1—N1	90.14 (6)	N1—C1—H12	108.357
F2—Si1—N1	91.23 (6)	C2—C1—H12	108.357
F2^{i}—Si1—N1	88.77 (6)	H11—C1—H12	109.467
F1—Si1—N1^{i}	90.14 (6)	C1—C2—H21	109.467
F1^{i}—Si1—N1^{i}	89.86 (6)	C1—C2—H22	109.467
F2—Si1—N1^{i}	88.77 (6)	H21—C2—H22	109.476
F2^{i}—Si1—N1^{i}	91.23 (6)	C1—C2—H23	109.467
N1—Si1—N1^{i}	180.	H21—C2—H23	109.476
Si1—N1—C1	120.61 (15)	H22—C2—H23	109.476
Si1—N1—H1	110 (2)		

Symmetry codes: (i) −*x*+1, −*y*+1, −*z*+1.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···F1^{ii}	0.81 (3)	2.15 (3)	2.938 (2)	165 (3)
N1—H2···F2^{iii}	0.81 (3)	2.29 (3)	3.091 (2)	168 (3)

Symmetry codes: (ii) *x*, −*y*+1/2, *z*−1/2; (iii) *x*, −*y*+3/2, *z*−1/2.

Fig. 1

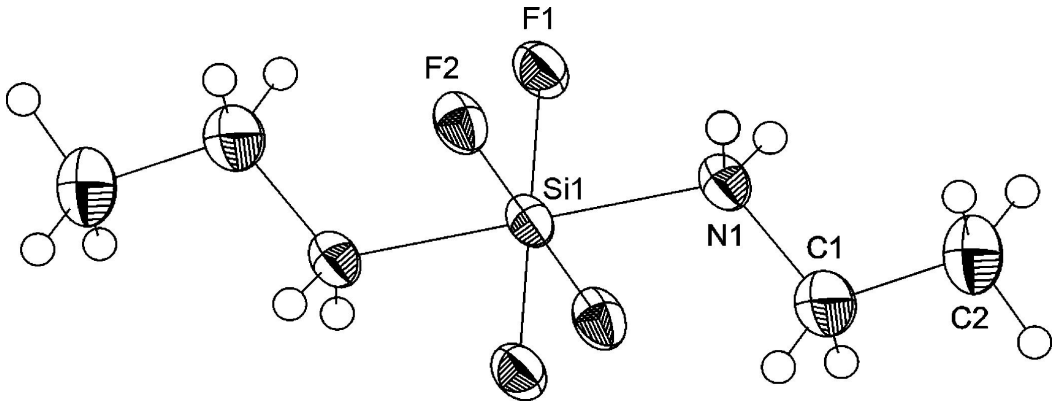

Fig. 2