Crystal structure of hexamethylguanidinium difluorotrimethylsilicate, $C_{10}H_{27}F_2N_3Si$

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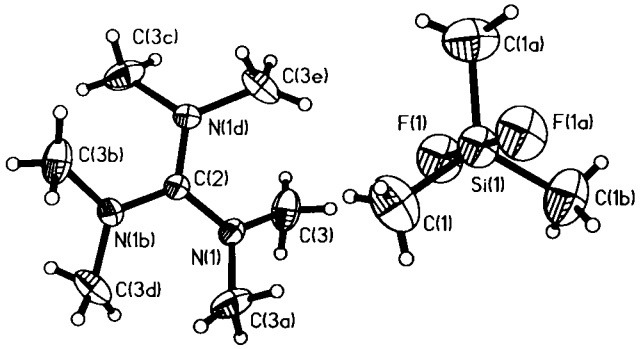

Abstract

$C_{10}H_{27}F_2N_3Si$, hexagonal, $P6_3/m$ (No. 176), $a = 7.898(1)$ Å, $c = 14.675(3)$ Å, $V = 792.8$ Å^3, $Z = 2$, $R_{gt}(F) = 0.045$, $wR_{ref}(F^2) = 0.117$, $T = 173$ K.

Source of material

Synthesis was performed according to [1]. Crystals of $[C(NMe_2)_3]^+Me_3SiF_2^-$, mp 439 K – 440 K, were grown from a concentrated solution in acetonitrile (1.0 g in 4 ml), which was cooled with liquid nitrogen, and diethylether (4 ml) was condensed to the solid. The flask was placed into a cryostate at 243 K and allowed to stand for 7 d at this temperature.

Experimental details

The symmetry determination on the diffractometer gives the Laue class *6/mmm*. In space groups belonging to this Laue class no structure solutions could be found. The best *R*-factors were found in the space group $P6_3/m$ refining the data as a merohedric twin.

In addition to this, the cation was found to be disordered. Two sets of N positions were found related by a 60° rotation, with an occupancy ratio of 1:1 (only one of the sets is shown in the figure). The C atoms of this group are not disordered.

Discussion

The structural parameters of the hexamethylguanidinium cation are in accord to already published data [2]. The CN_3 unit is planar, with ∠N1–C2–N1#6 = 119.987(7)°. The C2—N1 bond distance was found to be 134.3 (3) pm proving the iminium cation character [1], the N1—C3 distance is 153.7(4) pm. The enviroment around the nitrogen atoms is planar with the sum of the angles (∠C2–N1–C3#7 = 117.8(2)°, ∠C2–N1–C3 = 116.6(2)°, ∠C3#7–N1–C3 125.5(2)°) to be 359.9°. The trigonal-bipyramidal $Me_3SiF_2^-$ anion shows structural parameters similar to [3] with the bond lengths *d*(Si1—F1) = 176.7(2) pm and *d*(Si1—C1) = 187.1(4) pm, and angles ∠F1–Si1–F1#1 = 180.0°, ∠F1#1–Si1–C2#2 = 90.0° [4,5].

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.20 × 0.60 × 0.90 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	1.52 cm^{-1}
Diffractometer, scan mode:	Siemens P4, $\omega/2\theta$
$2\theta_{max}$:	54.9°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	1797, 634
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 469
$N(param)_{refined}$:	49
Programs:	SHELXS-97 [4], SHELXL-97 [5]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	*x*	*y*	*z*	U_{iso}
H(11)	6*h*	0.440(2)	0.449(2)	1/4	0.13(1)
H(12)	12*i*	0.350(2)	0.255(1)	0.1978(6)	0.13
H(31)	12*i*	0.438(2)	0.300(2)	0.503(1)	0.075(5)
H(32)	12*i*	0.370(3)	0.168(3)	0.587(1)	0.075
H(33)	12*i*	0.327(4)	0.337(2)	0.579(1)	0.075

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	Occ.	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Si(1)	2*d*		2/3	1/3	1/4	0.0562(6)	U_{11}	0.0236(5)	$U_{11}/2$	0	0
F(1)	4*f*		2/3	1/3	0.3704(1)	0.084(1)	U_{11}	0.0254(8)	$U_{11}/2$	0	0
C(1)	6*h*		0.4244(6)	0.3220(7)	1/4	0.073(3)	0.114(4)	0.052(2)	0.058(3)	0	0
C(2)	2*b*		0	0	1/2	0.023(1)	U_{11}	0.031(2)	$U_{11}/2$	0	0
N(1)	12*i*	0.50	0.1418(4)	0.1885(4)	0.4989(3)	0.025(1)	0.022(1)	0.041(1)	0.009(1)	–0.001(1)	0.002(1)
C(3)	12*i*		0.3350(4)	0.2380(3)	0.5460(1)	0.028(1)	0.049(1)	0.051(1)	0.004(1)	–0.009(1)	–0.005(1)

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