

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

Butane-1,4-diammonium hexafluoro-silicate

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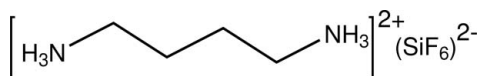
Received 24 December 2013; accepted 15 January 2014

Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.038; wR factor = 0.108; data-to-parameter ratio = 21.1.

The title compound, $[\text{NH}_3(\text{CH}_2)_4\text{NH}_3]^{2+} \cdot \text{SiF}_6^{2-}$, is a hybrid built from an organic butane-1,4-diammonium dication linked to a hexafluorosilicate mineral anion. Both ions possess inversion symmetry. In the anion the Si atom is located on an inversion center, while in the cation the center of inversion is situated at the mid-point of the central $-\text{CH}_2-\text{CH}_2-$ bond. The Si atom is surrounded by six F atoms, forming a slightly distorted SiF_6^{2-} octahedron. These octahedra are linked to the organic cations through $\text{N}-\text{H} \cdots \text{F}$ hydrogen bonds, forming a three-dimensional network.

Related literature

For background to potential physical properties, see: Ouasri *et al.* (2003); Elyoubi *et al.* (2004). For similar compounds, see: Jeghnou *et al.* (2005); Rhandour *et al.* (2011); Ouasri *et al.* (2012, 2013a,b, 2014).



Experimental

Crystal data

 $\text{C}_4\text{H}_{14}\text{N}_2^{2+} \cdot \text{SiF}_6^{2-}$ $M_r = 232.26$ Triclinic, $P\bar{1}$ $a = 5.796$ (1) Å $b = 5.889$ (1) Å $c = 7.774$ (2) Å $\alpha = 87.02$ (1)° $\beta = 82.15$ (1)° $\gamma = 61.87$ (1)° $V = 231.79$ (8) Å³ $Z = 1$ Mo $K\alpha$ radiation $\mu = 0.31$ mm⁻¹ $T = 296$ K $0.36 \times 0.32 \times 0.27$ mm

Data collection

Bruker X8 APEX diffractometer
Absorption correction: multi-scan
(*SADABS*; Bruker, 2009)
 $T_{\min} = 0.512$, $T_{\max} = 0.640$

5351 measured reflections
1285 independent reflections
1185 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.025$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.038$ $wR(F^2) = 0.108$ $S = 1.05$

1285 reflections

61 parameters

H-atom parameters constrained

 $\Delta\rho_{\max} = 0.74$ e Å⁻³ $\Delta\rho_{\min} = -0.33$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H} \cdots A$	$D-\text{H}$	$\text{H} \cdots A$	$D \cdots A$	$D-\text{H} \cdots A$
$\text{N1}-\text{H1NA} \cdots \text{F2}$	0.89	2.31	3.017 (2)	137
$\text{N1}-\text{H1NA} \cdots \text{F3}$	0.89	2.39	3.134 (2)	142
$\text{N1}-\text{H1NB} \cdots \text{F3}^{\text{i}}$	0.89	2.02	2.890 (2)	167
$\text{N1}-\text{H1NC} \cdots \text{F1}^{\text{ii}}$	0.89	2.04	2.864 (2)	154

Symmetry codes: (i) $-x, -y + 1, -z$; (ii) $x - 1, y, z$.

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT-Plus* (Bruker, 2009); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *PLATON* (Spek, 2009) and *pubCIF* (Westrip, 2010).

The authors thank the Unit of Support for Technical and Scientific Research (UATRS, CNRST) for the X-ray measurements.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IM2447).

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supporting information

Acta Cryst. (2014). E70, o174 [doi:10.1107/S1600536814001068]

Butane-1,4-diammonium hexafluorosilicate

Ali Ouasri, Ali Rhandour, Mohamed Saadi and Lahcen El Ammari

S1. Comment

The alkanediammonium halogenometallate salts family with the general formula $(\text{NH}_3(\text{CH}_2)_n\text{NH}_3)\text{MX}_6$ where M : Sn, Si, Te and X : Cl, Br, I and F, have recently attracted the interest of many investigators due to their potential physical properties (Ouasri *et al.*, 2003; Elyoubi *et al.*, 2004). X-ray, thermal and vibrational studies of phase transitions have also been performed for highly related compounds belonging to the alkanediammonium halogenobismuthate salts family as the pentachlorobismuthate one $(\text{NH}_3(\text{CH}_2)_n\text{NH}_3)\text{BiCl}_5$ (Jeghnou *et al.*, 2005; Ouasri *et al.*, 2012; Rhandour *et al.*, 2011; Ouasri *et al.*, 2013a; Ouasri *et al.*, 2013b; Ouasri *et al.*, 2014). The aim of the present paper was to study the recently synthesized butanediammonium hexafluorosilicate $(\text{NH}_3(\text{CH}_2)_4\text{NH}_3)\text{SiF}_6$ crystal, by X-ray diffraction at room temperature.

The structure of the title compound is built up from inorganic anions linked to the organic cations through hydrogen bonds. In this structure, all atoms are in general positions, except the silicon atom (Si1 (1/2, 0, 0) which is located at a crystallographic centre of inversion of the $P\bar{1}$ space group. In addition, the centre of the bond C2—C2i is also situated on another crystallographic centre of inversion. The asymmetric unit therefore contains only one half of the organic cation and one SiF_3 moiety. The remaining atoms of the unit cell are generated by symmetry operations.

The silicon atom is surrounded by six fluorine atoms building a slightly distorted SiF_6^{2-} octahedron. SiF_6^{2-} octahedra are linked to the organic cations through N—H \cdots F hydrogen bonds producing an infinite two-dimensional layer parallel to (0 1 1) (Fig.2 and Table 1).

S2. Experimental

$(\text{NH}_3(\text{CH}_2)_4\text{NH}_3)\text{SiF}_6$ single crystals were obtained by slow evaporation, at room temperature, of an aqueous solution containing stoichiometric amounts of butane-1,4-diamine, $\text{NH}_2(\text{CH}_2)_4\text{NH}_2$, and H_2SiF_6 acid.

S3. Refinement

H atoms were located in a difference map and treated as riding with C—H = 0.97 Å, and 0.893 Å for CH_2 and N—H, respectively. Thermal parameters of all hydrogen atoms were refined with $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{methylene})$ and $U_{\text{iso}}(\text{H}) = 1.5 U_{\text{eq}}(\text{N})$ for the ammonium groups.

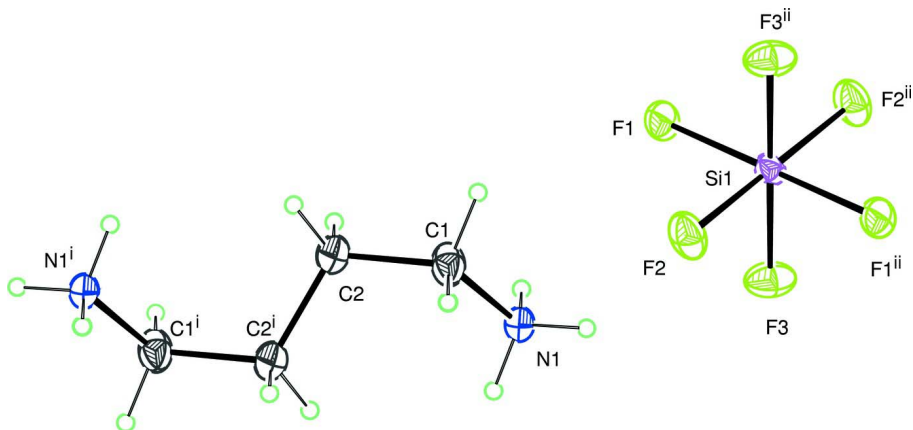


Figure 1

Molecular structure of the title compound with the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented as small circles.

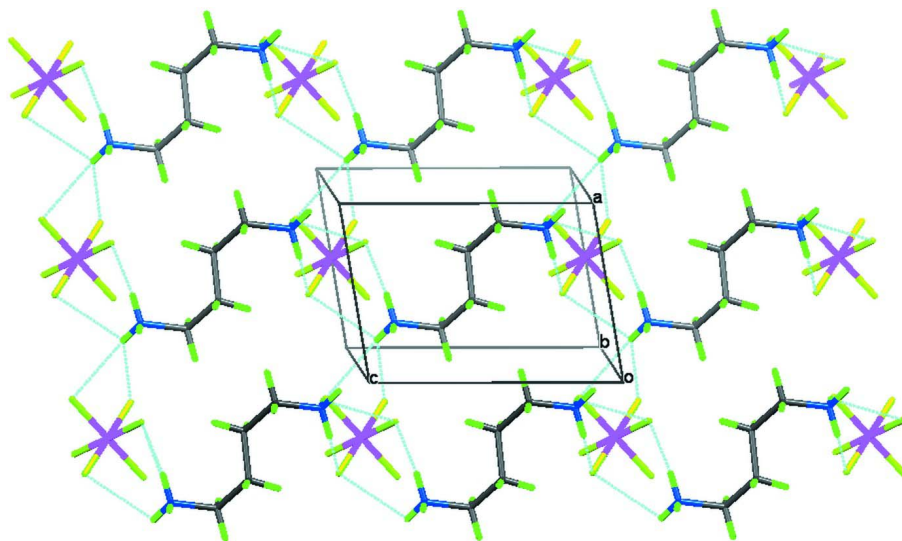


Figure 2

Three dimensional plot of the title compound, showing inorganic sheets linked through N–H⋯F hydrogen bonds to the organic layers (dashed lines).

Butane-1,4-diammonium hexafluorosilicate

Crystal data

$C_4H_{14}N_2^{2+} \cdot SiF_6^{2-}$
 $M_r = 232.26$
 Triclinic, $P\bar{1}$
 Hall symbol: $-P\ 1$
 $a = 5.796$ (1) Å
 $b = 5.889$ (1) Å
 $c = 7.774$ (2) Å
 $\alpha = 87.02$ (1)°
 $\beta = 82.15$ (1)°
 $\gamma = 61.87$ (1)°
 $V = 231.79$ (8) Å³

$Z = 1$
 $F(000) = 120$
 $D_x = 1.664$ Mg m⁻³
 Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
 Cell parameters from 1285 reflections
 $\theta = 3.9$ – 29.6 °
 $\mu = 0.31$ mm⁻¹
 $T = 296$ K
 Block, colourless
 $0.36 \times 0.32 \times 0.27$ mm

Data collection

Bruker X8 APEX
diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
 φ and ω scans
Absorption correction: multi-scan
(*SADABS*; Bruker, 2009)
 $T_{\min} = 0.512$, $T_{\max} = 0.640$

5351 measured reflections
1285 independent reflections
1185 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.025$
 $\theta_{\max} = 29.6^\circ$, $\theta_{\min} = 3.9^\circ$
 $h = -7 \rightarrow 8$
 $k = -8 \rightarrow 8$
 $l = -10 \rightarrow 10$

Refinement

Refinement on F^2
Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.038$
 $wR(F^2) = 0.108$
 $S = 1.05$
1285 reflections
61 parameters
0 restraints
Primary atom site location: structure-invariant
direct methods

Secondary atom site location: difference Fourier
map
Hydrogen site location: inferred from
neighbouring sites
H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0549P)^2 + 0.127P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.74 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.33 \text{ e } \text{\AA}^{-3}$

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against all reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on all data will be even larger.

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	-0.1629 (3)	0.2744 (4)	0.3650 (2)	0.0394 (4)
H1A	0.0138	0.2407	0.3801	0.047*
H1B	-0.1739	0.1160	0.3876	0.047*
C2	-0.3593 (4)	0.4783 (4)	0.4953 (2)	0.0435 (4)
H2A	-0.3573	0.6396	0.4663	0.052*
H2B	-0.3024	0.4297	0.6094	0.052*
N1	-0.2086 (2)	0.3508 (2)	0.18267 (15)	0.0269 (3)
H1NA	-0.0891	0.2252	0.1110	0.040*
H1NB	-0.1946	0.4939	0.1603	0.040*
H1NC	-0.3695	0.3792	0.1675	0.040*
Si1	0.5000	0.0000	0.0000	0.02400 (16)
F1	0.35813 (19)	0.26912 (18)	0.12542 (14)	0.0381 (3)
F2	0.3234 (2)	-0.1206 (2)	0.12259 (14)	0.0426 (3)
F3	0.2526 (2)	0.1424 (2)	-0.12459 (15)	0.0445 (3)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0304 (7)	0.0470 (9)	0.0266 (7)	-0.0069 (7)	-0.0029 (5)	0.0022 (6)
C2	0.0368 (9)	0.0649 (12)	0.0278 (7)	-0.0233 (8)	0.0014 (6)	-0.0109 (7)
N1	0.0262 (6)	0.0287 (6)	0.0236 (5)	-0.0117 (5)	0.0007 (4)	-0.0032 (4)
Si1	0.0193 (3)	0.0203 (3)	0.0312 (3)	-0.00873 (19)	0.00039 (18)	-0.00429 (18)
F1	0.0318 (5)	0.0310 (5)	0.0487 (6)	-0.0135 (4)	0.0061 (4)	-0.0182 (4)
F2	0.0412 (6)	0.0386 (5)	0.0505 (6)	-0.0251 (5)	0.0122 (4)	-0.0037 (4)
F3	0.0405 (6)	0.0350 (5)	0.0593 (7)	-0.0143 (4)	-0.0245 (5)	0.0034 (5)

Geometric parameters (\AA , $^\circ$)

C1—N1	1.4853 (19)	N1—H1NB	0.8900
C1—C2	1.512 (2)	N1—H1NC	0.8900
C1—H1A	0.9700	Si1—F2 ⁱⁱ	1.6757 (10)
C1—H1B	0.9700	Si1—F2	1.6757 (10)
C2—C2 ⁱ	1.519 (4)	Si1—F1	1.6886 (9)
C2—H2A	0.9700	Si1—F1 ⁱⁱ	1.6886 (9)
C2—H2B	0.9700	Si1—F3	1.6908 (10)
N1—H1NA	0.8900	Si1—F3 ⁱⁱ	1.6908 (10)
N1—C1—C2	112.70 (13)	H1NB—N1—H1NC	109.5
N1—C1—H1A	109.1	F2 ⁱⁱ —Si1—F2	180.0
C2—C1—H1A	109.1	F2 ⁱⁱ —Si1—F1	89.09 (5)
N1—C1—H1B	109.1	F2—Si1—F1	90.91 (5)
C2—C1—H1B	109.1	F2 ⁱⁱ —Si1—F1 ⁱⁱ	90.91 (5)
H1A—C1—H1B	107.8	F2—Si1—F1 ⁱⁱ	89.09 (5)
C1—C2—C2 ⁱ	114.65 (19)	F1—Si1—F1 ⁱⁱ	180.0
C1—C2—H2A	108.6	F2 ⁱⁱ —Si1—F3	91.03 (6)
C2 ⁱ —C2—H2A	108.6	F2—Si1—F3	88.98 (6)
C1—C2—H2B	108.6	F1—Si1—F3	89.04 (6)
C2 ⁱ —C2—H2B	108.6	F1 ⁱⁱ —Si1—F3	90.96 (6)
H2A—C2—H2B	107.6	F2 ⁱⁱ —Si1—F3 ⁱⁱ	88.97 (6)
C1—N1—H1NA	109.5	F2—Si1—F3 ⁱⁱ	91.02 (6)
C1—N1—H1NB	109.5	F1—Si1—F3 ⁱⁱ	90.96 (6)
H1NA—N1—H1NB	109.5	F1 ⁱⁱ —Si1—F3 ⁱⁱ	89.04 (6)
C1—N1—H1NC	109.5	F3—Si1—F3 ⁱⁱ	180.0
H1NA—N1—H1NC	109.5		

Symmetry codes: (i) $-x-1, -y+1, -z+1$; (ii) $-x+1, -y, -z$.Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1NA \cdots F2	0.89	2.31	3.017 (2)	137
N1—H1NA \cdots F3	0.89	2.39	3.134 (2)	142

N1—H1NB···F3 ⁱⁱⁱ	0.89	2.02	2.890 (2)	167
N1—H1NC···F1 ^{iv}	0.89	2.04	2.864 (2)	154

Symmetry codes: (iii) $-x, -y+1, -z$; (iv) $x-1, y, z$.