(±)-Bis(1-carboxy-2-phenyl-ethanaminium) hexafluorosilicate(VI)

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The asymmetric unit of the title fluorosilicate salt, $2C_9H_{12}NO_2{}^+\cdot SiF_6{}^{2-}$, consists of a phenylalaninium cation and half of a fluorosilicate anion, the Si atom being located on an inversion center. In the crystal, all of the F atoms act as hydrogen-bond acceptors and link the cations through different graph-set motifs, forming layers developing parallel to (100).

Related literature

For applications of fluorosilicate salts, see: Katayama *et al.* (2001); Kalem (2004); Airoldi & De Farias (2000); Han *et al.* (2000); Gelmboldt (1989); Gelmboldt *et al.* (2007). For our previous work on hydrogen-bonding interactions in the crystal structures of protonated amines, see: Bouacida *et al.* (2005, 2007, 2009); Benslimane *et al.* (2007); Bouacida (2008). For a description of the Cambridge Structural Database, see: Allen (2002). For hydrogen-bond motifs, see: Etter *et al.* (1990); Bernstein *et al.* (1995); Janiak (2000).
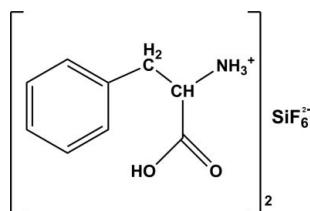
Experimental

Crystal data

$2C_9H_{12}NO_2{}^+\cdot SiF_6{}^{2-}$	b = 5.7531 (10) Å
M_r = 474.48	c = 17.000 (4) Å
Monoclinic, $P2_1/c$	β = 105.59 (2)°
a = 11.183 (2) Å	V = 1053.5 (4) Å^3
Z = 2	T = 295 K
Mo $K\alpha$ radiation	0.59 × 0.50 × 0.37 mm
μ = 0.19 mm^{-1}	

Data collection

Nonius KappaCCD diffractometer	2412 independent reflections
2412 measured reflections	1917 reflections with $I > 2\sigma(I)$

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.035	144 parameters
$wR(F^2)$ = 0.086	H-atom parameters constrained
S = 1.03	$\Delta\rho_{max}$ = 0.22 e Å^{-3}
2412 reflections	$\Delta\rho_{min}$ = −0.19 e Å^{-3}

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1—H1···F2^i	0.82	1.84	2.6456 (14)	167
N2—H2A···O2^{ii}	0.89	2.04	2.8511 (17)	151
N2—H2B···F3	0.89	1.88	2.7656 (15)	171
N2—H2C···F1^{iii}	0.89	2.01	2.8549 (15)	158

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

Data collection: *COLLECT* (Otwinowski & Minor, 1997); cell refinement: *DIRAX/LSQ* (Duisenberg *et al.*, 2003); data reduction: *EVALCCD* (Duisenberg *et al.*, 2003); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996), *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2009); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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supplementary materials

(±)-Bis(1-carboxy-2-phenylethanaminium) hexafluorosilicate(VI)

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Comment

Fluorosilicate salts involving onium cations of N– and O– containing organic bases and amino acids have practical applications as ionic liquids (Katayama *et al.*, 2001), dielectrics with cryptocrystalline structure (Kalem, 2004), layered organic-inorganic hybrid materials (Airoldi & De Farias, 2000) and chemical reagents (Han *et al.*, 2000; Gelmboldt, 1989). Their structures are commonly dominated by strong directional interactions involving F atoms and convenient hydrogen-bond donors, although the relationships in such systems can be complicated due to the presence of competitive OH and NH binding sites (Gelmboldt *et al.*, 2007). We report here the synthesis, crystal structure and hydrogen-bonded frameworks of a new hybrid compound based on fluorosilicate. The title compound (I) was prepared as part of our ongoing studies of hydrogen-bonding interactions in the crystal structures of protonated amines (Bouacida *et al.*, 2005, 2007, 2009; Benslimane *et al.*, 2007; Bouacida, 2008). From the molecular point of view, the structure is quite simple, since the individual components do not deviate from the expected geometries, with bond distances and angles lying within reported values for these species (CSD, Allen, 2002). The most attractive aspect of these structures resides in their extensive hydrogen-bonding schemes.

The asymmetric unit of (I) is built up from a (+/-)-phenylalaninium cation and half a molecule of a hexafluorosilicate anion located on an inversion center, connected by N—H⋯F hydrogen bonds (Fig. 1).

As observed in compound I, all the F atoms of the hexafluorosilicate anion act as hydrogen bond acceptors and are engaged in N—H⋯F and O—H⋯F bonds with the alaninium part of the cation (Fig. 2, Table 1). Two H atoms, H2B and H2C and their symmetry related counterparts (-*x*, -*y*, 1 - *z*), of the ammonium NH_3 interact with two symmetry related fluorosilicate (-*x*, -*y*, 1 - *z*) building a $R_4^2(8)$ ring labelled **B1**, whereas H2A, the third H atom of the NH_3 and the H atom of the symmetry related (-*x*, *y* + 1/2, -*z* + 1/2) carboxylate complete a $R_3^3(10)$ graph set motif labelled **B2**. Futhermore, two symmetry related (-*x*, *y* - 1/2, 1/2 - *z*) cations and one fluorosilicate (*x*, *y* - 1, *z*) form a R_3^3 (14) ring labelled **B3**, through N2—H2C⋯F1, N2—H2B⋯O2 and O1—H⋯F2 (Etter *et al.*, 1990; Bernstein *et al.*, 1995), see Table 1, Fig. 2.

These hydrogen bonds result in the formation of layers parallel to the (1 0 0) plane. In these layers, chains of cations and anions alternate (Fig. 3). As shown in the Figure, the phenyl rings of the symmetry related layers are intercalated; however the centroid to centroid distance between the phenyl rings are too long (4.958 (1) and 4.523 (1) Å) to consider π-π interactions (Janiak, 2000).

Experimental

Crystals of compound I were grown from an aqueous solution that was obtained by dissolving 1 mmol SiO_2 and 2 mmol phenylalanine in hydrofluoric acid (HF). The solutions were slowly evaporated to dryness for a couple of weeks. Some white crystals were carefully isolated under polarizing microscope for analysis by X-ray diffraction.

Refinement

All non-H atoms were refined with anisotropic atomic displacement parameters. All H atoms were localized on Fourier maps but introduced in calculated positions and treated as riding on their parent C, N and O atoms, with C—H = 0.93,0.97, 0.98 Å, N—H = 0.89 Å and O—H = 0.82 Å with U_{iso}(H) = 1.2 or 1.5 U_{eq}(C, N or O).

Computing details

Data collection: *COLLECT* (Otwinowski & Minor, 1997); cell refinement: *DIRAX/LSQ* (Duisenberg *et al.*, 2003); data reduction: *EVALCCD* (Duisenberg *et al.*, 2003); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996), *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2009); software used to prepare material for publication: *WinGX* (Farrugia, 1999).
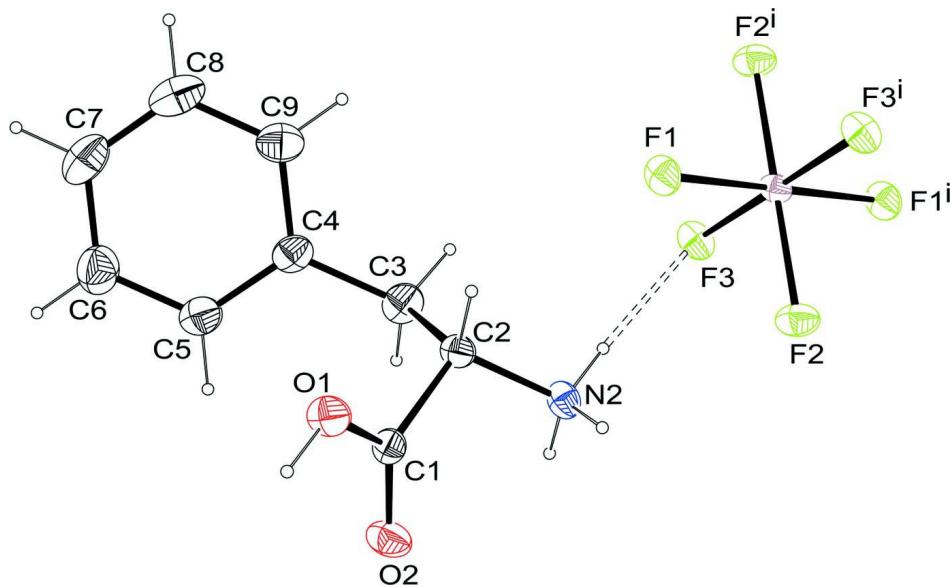
Figure 1

The asymmetric unit of (I) with the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms are represented as small spheres of arbitrary radii. Hydrogen bond is shown as dashed line.[Symmetry codes: (i) -*x*, 1 - *y*, 1 - *z*]
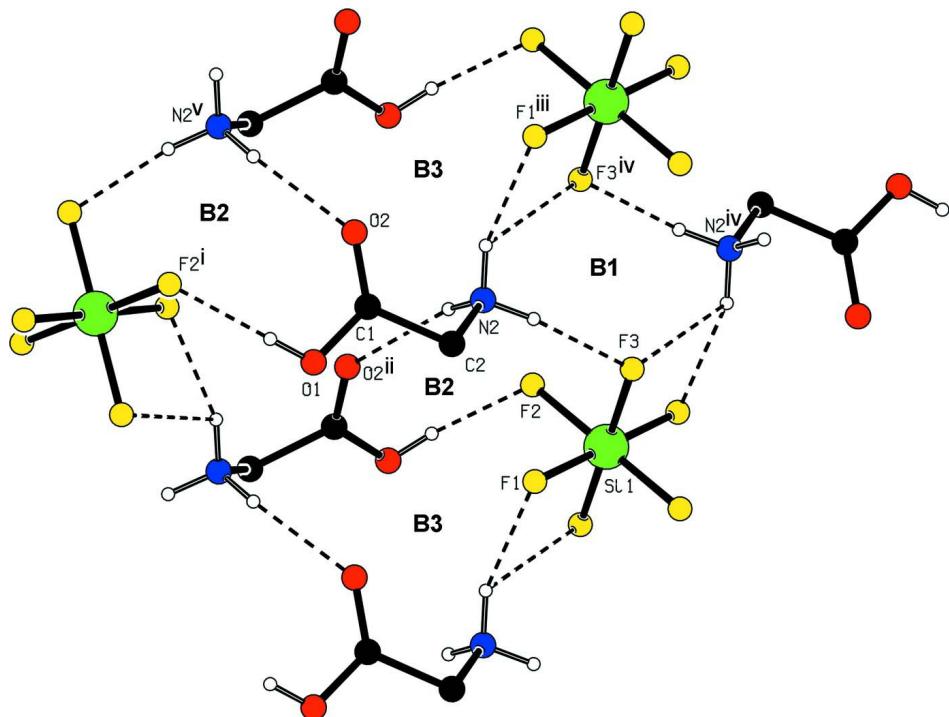
Figure 2

Partial view of compound (I), showing the formation of $R_4^2(8)$ (**B1**), $R_3^3(10)$ (**B2**) and $R_3^3(14)$ (**B3**) graph set motifs through N—H···F, N—H···O and O—H···F hydrogen bonds. The $C_6H_5CH_2$ fragment have been omitted for the sake of clarity. [Symmetry codes: (i) -*x*, *y* - 1/2, -*z* + 1/2; (ii) -*x*, *y* + 1/2, -*z* + 1/2; (iii) *x*, *y* - 1, *z*; (iv) -*x*, -*y*, -*z* + 1; (v) -*x*, *y* - 1/2, 1/2 - *z*]
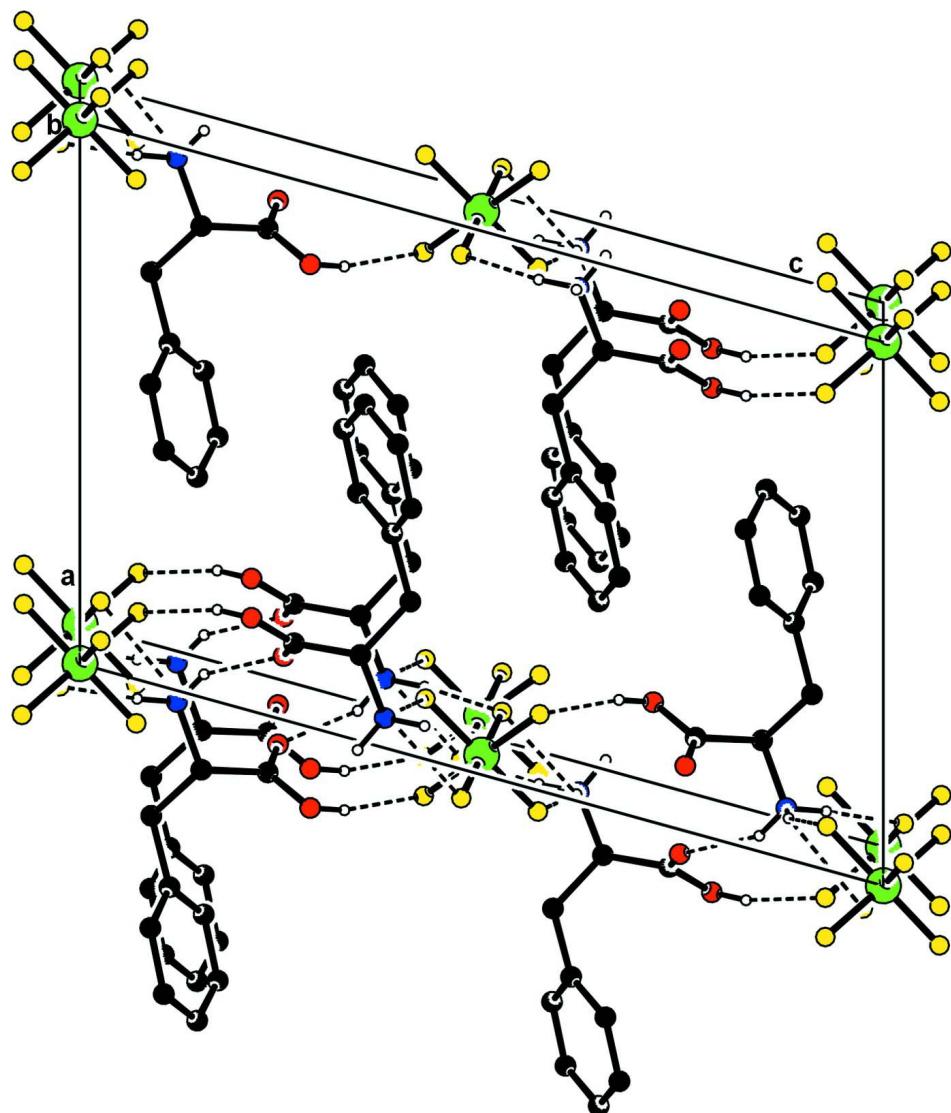
Figure 3

Packing view projected down the *b* axis showing the formation of layers parallel to the (1 0 0) plane. Hydrogen bonds are shown as dashed lines. H atoms not involved in hydrogen bonding have been omitted for clarity.

(±)-Bis(1-carboxy-2-phenylethanaminium) hexafluorosilicate(VI)

Crystal data
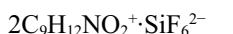
$2C_9H_{12}NO_2^+ \cdot SiF_6^{2-}$	$F(000) = 492$
$M_r = 474.48$	$D_x = 1.496$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2ybc	Cell parameters from 4769 reflections
$a = 11.183$ (2) Å	$\theta = 5.0–27.5°$
$b = 5.7531$ (10) Å	$\mu = 0.19$ mm^{-1}
$c = 17.000$ (4) Å	$T = 295$ K
$\beta = 105.59$ (2)°	Block, white
$V = 1053.5$ (4) Å^3	$0.59 \times 0.50 \times 0.37$ mm
$Z = 2$	

Data collection

Nonius KappaCCD diffractometer	2412 independent reflections
Radiation source: fine-focus sealed tube	1917 reflections with $I > 2\sigma(I)$
Graphite monochromator	$R_{int} = 0.000$
Detector resolution: 9 pixels mm^{-1}	$\theta_{max} = 27.5°$, $\theta_{min} = 5.0°$
CCD rotation images, thick slices scans	$h = -14 \rightarrow 13$
2412 measured reflections	$k = 0 \rightarrow 7$
	$l = 0 \rightarrow 22$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.035$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.086$	
$S = 1.03$	H-atom parameters constrained
2412 reflections	$w = 1/[\sigma^2(F_o^2) + (0.0329P)^2 + 0.2833P]$
144 parameters	where $P = (F_o^2 + 2F_c^2)/3$
0 restraints	$(\Delta/\sigma)_{max} = 0.001$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{max} = 0.22$ e Å^{-3}
	$\Delta\rho_{min} = -0.19$ e Å^{-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 $F^2 > \sigma(F^2)$ 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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
Si1	0.0000	0.5000	0.5000	0.02345 (13)
F1	0.07812 (8)	0.54094 (14)	0.43002 (5)	0.0345 (2)
F2	−0.11419 (8)	0.36774 (15)	0.42781 (5)	0.0371 (2)
F3	0.07100 (8)	0.24091 (13)	0.52540 (5)	0.0346 (2)
O1	0.17794 (11)	0.0905 (2)	0.21347 (6)	0.0403 (3)
H1	0.1700	0.0176	0.1709	0.060*
O2	0.10064 (12)	−0.23340 (19)	0.25348 (6)	0.0431 (3)
N2	0.05617 (12)	0.0158 (2)	0.37935 (7)	0.0307 (3)
H2A	−0.0126	0.0728	0.3455	0.046*
H2B	0.0661	0.0763	0.4289	0.046*
H2C	0.0499	−0.1381	0.3820	0.046*
C2	0.16410 (14)	0.0759 (2)	0.34897 (8)	0.0293 (3)
H2	0.1696	0.2449	0.3435	0.035*
C1	0.14352 (13)	−0.0392 (2)	0.26636 (8)	0.0289 (3)
C3	0.28085 (15)	−0.0161 (3)	0.41120 (9)	0.0410 (4)
H3A	0.2872	0.0580	0.4634	0.049*
H3B	0.2708	−0.1816	0.4182	0.049*
C4	0.40009 (15)	0.0225 (3)	0.38861 (9)	0.0374 (4)

C6	0.55760 (19)	−0.1169 (4)	0.32857 (12)	0.0563 (5)
H6	0.5865	−0.2308	0.2994	0.068*
C9	0.47022 (18)	0.2226 (3)	0.41237 (11)	0.0493 (4)
H9	0.4405	0.3395	0.4399	0.059*
C8	0.58322 (19)	0.2497 (4)	0.39562 (12)	0.0585 (5)
H8	0.6297	0.3835	0.4124	0.070*
C5	0.44501 (17)	−0.1453 (3)	0.34602 (11)	0.0469 (4)
H5	0.3988	−0.2791	0.3289	0.056*
C7	0.62729 (18)	0.0799 (4)	0.35423 (12)	0.0580 (5)
H7	0.7041	0.0975	0.3435	0.070*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Si1	0.0314 (3)	0.0221 (2)	0.0178 (2)	−0.0039 (2)	0.00823 (19)	−0.00049 (18)
F1	0.0451 (5)	0.0334 (4)	0.0310 (4)	−0.0022 (4)	0.0206 (4)	0.0025 (3)
F2	0.0414 (5)	0.0393 (5)	0.0281 (4)	−0.0112 (4)	0.0049 (4)	−0.0044 (3)
F3	0.0472 (5)	0.0277 (4)	0.0311 (4)	0.0050 (4)	0.0141 (4)	0.0041 (3)
O1	0.0516 (7)	0.0483 (6)	0.0232 (5)	−0.0117 (5)	0.0137 (5)	−0.0002 (4)
O2	0.0606 (8)	0.0396 (6)	0.0310 (5)	−0.0123 (5)	0.0156 (5)	−0.0066 (5)
N2	0.0388 (7)	0.0284 (6)	0.0283 (6)	0.0025 (5)	0.0147 (5)	0.0024 (5)
C2	0.0359 (8)	0.0285 (7)	0.0252 (6)	−0.0043 (6)	0.0110 (6)	−0.0002 (5)
C1	0.0274 (7)	0.0352 (8)	0.0238 (6)	0.0002 (6)	0.0065 (5)	0.0010 (5)
C3	0.0388 (9)	0.0543 (10)	0.0265 (7)	−0.0026 (7)	0.0031 (6)	0.0038 (7)
C4	0.0324 (8)	0.0468 (9)	0.0280 (7)	−0.0026 (7)	−0.0003 (6)	0.0029 (6)
C6	0.0453 (11)	0.0653 (12)	0.0589 (11)	0.0071 (9)	0.0150 (9)	−0.0007 (9)
C9	0.0484 (11)	0.0497 (10)	0.0441 (9)	−0.0052 (8)	0.0027 (8)	−0.0041 (8)
C8	0.0491 (12)	0.0588 (12)	0.0586 (11)	−0.0176 (10)	−0.0009 (9)	0.0077 (10)
C5	0.0412 (10)	0.0472 (10)	0.0502 (10)	−0.0055 (8)	0.0088 (8)	−0.0044 (7)
C7	0.0349 (10)	0.0765 (14)	0.0596 (11)	−0.0056 (10)	0.0071 (9)	0.0138 (10)

Geometric parameters (Å, º)

Si1—F1^i	1.6711 (9)	C2—H2	0.9800
Si1—F1	1.6711 (9)	C3—C4	1.500 (2)
Si1—F3^i	1.6895 (8)	C3—H3A	0.9700
Si1—F3	1.6895 (8)	C3—H3B	0.9700
Si1—F2	1.6952 (8)	C4—C5	1.380 (3)
Si1—F2^i	1.6952 (9)	C4—C9	1.391 (2)
O1—C1	1.3036 (17)	C6—C7	1.377 (3)
O1—H1	0.8200	C6—C5	1.379 (3)
O2—C1	1.2121 (17)	C6—H6	0.9300
N2—C2	1.4760 (18)	C9—C8	1.377 (3)
N2—H2A	0.8900	C9—H9	0.9300
N2—H2B	0.8900	C8—C7	1.371 (3)
N2—H2C	0.8900	C8—H8	0.9300
C2—C1	1.5135 (19)	C5—H5	0.9300
C2—C3	1.537 (2)	C7—H7	0.9300
F1^i—Si1—F1	180.0	O2—C1—O1	125.37 (13)

F1ⁱ—Si1—F3ⁱ	90.38 (4)	O2—C1—C2	121.67 (13)
F1—Si1—F3ⁱ	89.62 (4)	O1—C1—C2	112.95 (12)
F1ⁱ—Si1—F3	89.62 (4)	C4—C3—C2	114.97 (12)
F1—Si1—F3	90.38 (4)	C4—C3—H3A	108.5
F3ⁱ—Si1—F3	180.0	C2—C3—H3A	108.5
F1ⁱ—Si1—F2	90.90 (5)	C4—C3—H3B	108.5
F1—Si1—F2	89.10 (4)	C2—C3—H3B	108.5
F3ⁱ—Si1—F2	90.00 (4)	H3A—C3—H3B	107.5
F3—Si1—F2	90.00 (4)	C5—C4—C9	118.37 (17)
F1ⁱ—Si1—F2ⁱ	89.10 (4)	C5—C4—C3	120.33 (15)
F1—Si1—F2ⁱ	90.90 (5)	C9—C4—C3	121.26 (16)
F3ⁱ—Si1—F2ⁱ	90.00 (4)	C7—C6—C5	120.05 (19)
F3—Si1—F2ⁱ	90.00 (4)	C7—C6—H6	120.0
F2—Si1—F2ⁱ	180.0	C5—C6—H6	120.0
C1—O1—H1	109.5	C8—C9—C4	120.73 (18)
C2—N2—H2A	109.5	C8—C9—H9	119.6
C2—N2—H2B	109.5	C4—C9—H9	119.6
H2A—N2—H2B	109.5	C7—C8—C9	120.13 (18)
C2—N2—H2C	109.5	C7—C8—H8	119.9
H2A—N2—H2C	109.5	C9—C8—H8	119.9
H2B—N2—H2C	109.5	C6—C5—C4	120.84 (17)
N2—C2—C1	106.69 (11)	C6—C5—H5	119.6
N2—C2—C3	107.57 (11)	C4—C5—H5	119.6
C1—C2—C3	112.16 (13)	C8—C7—C6	119.84 (19)
N2—C2—H2	110.1	C8—C7—H7	120.1
C1—C2—H2	110.1	C6—C7—H7	120.1
C3—C2—H2	110.1		
N2—C2—C1—O2	−39.96 (18)	C4—C9—C8—C7	0.8 (3)
C3—C2—C1—O2	77.58 (18)	C7—C6—C5—C4	0.5 (3)
N2—C2—C1—O1	140.88 (13)	C9—C4—C5—C6	1.0 (2)
C3—C2—C1—O1	−101.58 (15)	C3—C4—C5—C6	−176.65 (15)
N2—C2—C3—C4	177.95 (13)	C9—C8—C7—C6	0.8 (3)
C1—C2—C3—C4	60.93 (18)	C5—C6—C7—C8	−1.4 (3)
C2—C3—C4—C5	−92.09 (19)	C4—C3—C2—C1	60.93 (18)
C2—C3—C4—C9	90.30 (18)	C4—C3—C2—N2	177.95 (13)
C5—C4—C9—C8	−1.7 (2)	C3—C2—C1—O1	−101.58 (15)
C3—C4—C9—C8	175.98 (15)	C3—C2—C1—O2	77.58 (18)

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

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···F2ⁱⁱ	0.82	1.84	2.6456 (14)	167
N2—H2*A*···O2ⁱⁱⁱ	0.89	2.04	2.8511 (17)	151
N2—H2*B*···F3	0.89	1.88	2.7656 (15)	171
N2—H2*C*···F1ⁱᵛ	0.89	2.01	2.8549 (15)	158

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