# organic compounds

# (±)-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).
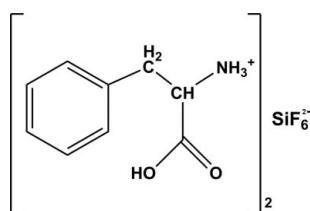


## Experimental

### 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, $P2_1/c$ | $\beta = 105.59$ (2)° |
| $a = 11.183$ (2) Å | $V = 1053.5$ (4) Å$^3$ |
| $Z = 2$ | $T = 295$ K |
| Mo $K\alpha$ radiation | $0.59 \times 0.50 \times 0.37$ mm |
| $\mu = 0.19$ 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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# supporting information

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

**Ratiba Belhouas, Sofiane Bouacida, Chaouki Boudaren, Jean-Claude Daran and Thierry Roisnel**

### S1. 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 $\pi$-$\pi$ interactions (Janiak, 2000).

### S2. 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.

**S3. 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).
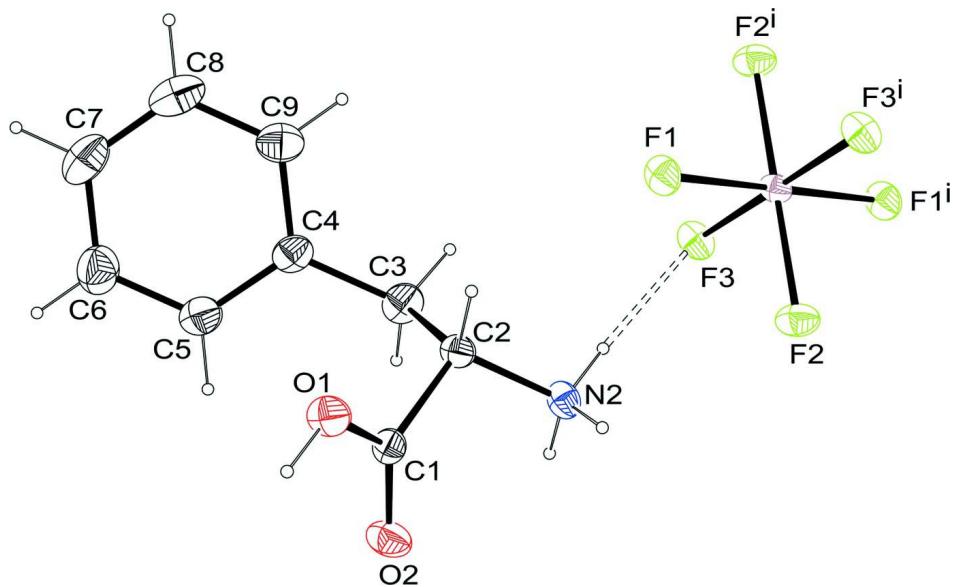


**Figure 1**

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 - $z$]
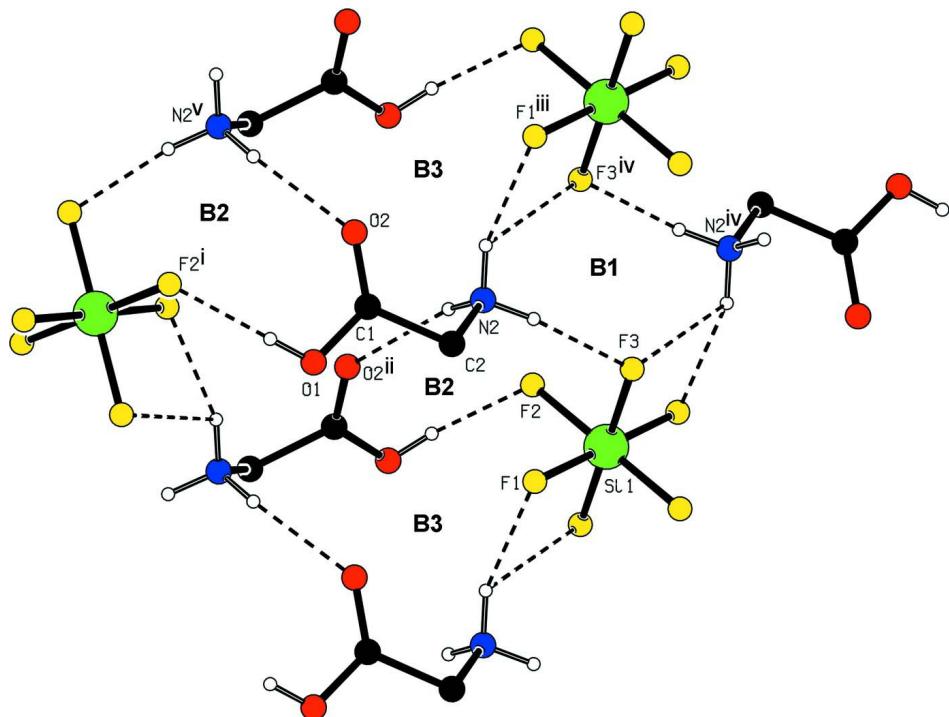
**Figure 2**

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*]
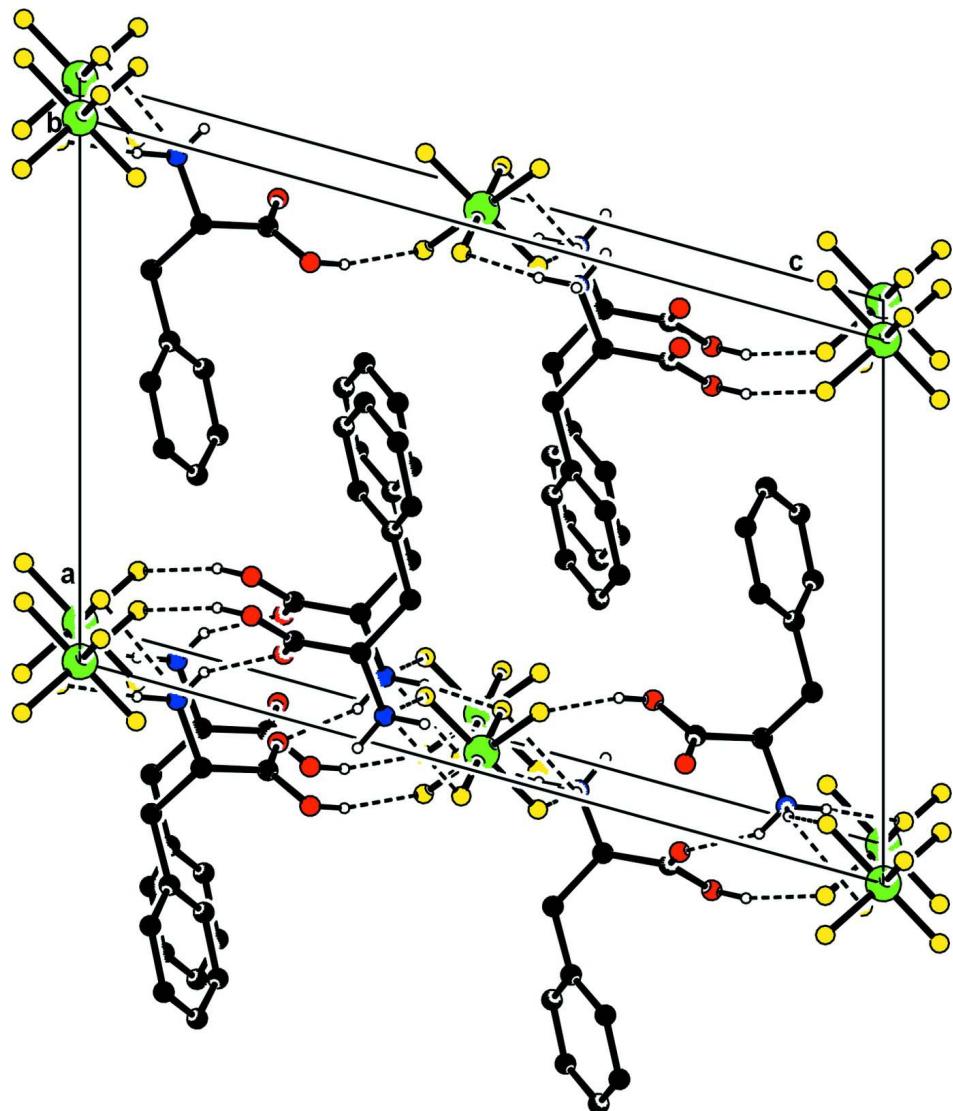
**Figure 3**

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*
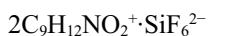
| | |
|---|---|
| $2C_9H_{12}NO_2^+ \cdot SiF_6^{2-}$ | $F(000) = 492$ |
| $M_r = 474.48$ | $D_x = 1.496$ Mg m$^{-3}$ |
| Monoclinic, $P2_1/c$ | Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å |
| Hall symbol: -P 2ybc | Cell parameters from 4769 reflections |
| $a = 11.183$ (2) Å | $\theta = 5.0$–27.5° |
| $b = 5.7531$ (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 (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| 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 |
| | | | |
| F1ⁱ—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*···O2$^{iii}$ | 0.89 | 2.04 | 2.8511 (17) | 151 |
| N2—H2*B*···F3 | 0.89 | 1.88 | 2.7656 (15) | 171 |
| N2—H2*C*···F1$^{iv}$ | 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*.