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Crystal structure of bis(9*H*-6-aminopurin-1-ium) hexafluoridosilicate(IV) dihydrate

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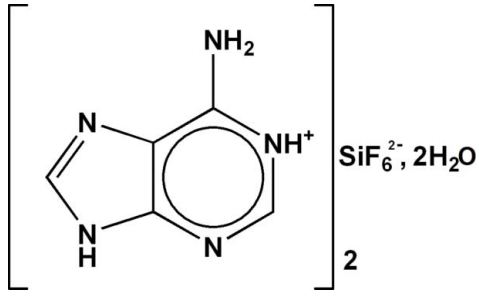
The asymmetric unit of the title compound, $2C_5H_6N_5^+\cdot SiF_6^{2-}\cdot 2H_2O$, contains one adeninium cation, half of a hexafluoridosilicate anion located on an inversion centre and one lattice water molecule. The adeninium cations are connected through N—H···N hydrogen bonds involving one H atom of the $-NH_2$ group and the H atom of the protonated N atom of the adenine ring system, forming centrosymmetric ring motifs of the type $R_2^2(10)$ and $R_2^2(8)$, respectively. The overall connection of the cation leads to the formation of planar ribbons parallel to (122). In the ribbons, slipped π–π stacking interactions, with a centroid-to-centroid distance of 3.6938 (9) Å, an interplanar distance of 3.455 Å and a slippage of 1.306 Å is observed. The hexafluoridosilicate anion and the water molecule are linked through O—H···F hydrogen bonds [ring motif $R_4^4(12)$] into chains parallel to [100]. The cationic ribbons and anionic chains are finally connected through additional N—H···O, N—H···F and O—H···F hydrogen bonds into a three-dimensional network in which layers of adeninium cations and fluoridosilicate anions alternate parallel to (001).

Keywords: crystal structure; purinium cation; hexafluoridosilicate anion; hydrogen bonding.

CCDC reference: 1038389

1. Related literature

The title compound was prepared as part of our ongoing studies of hydrogen-bonding interactions in the crystal structures of protonated amines (Bouacida *et al.*, 2005*a*,*b*,*c*; 2006; Belhouas *et al.*, 2012). For π–π stacking interactions, see: Janiak (2000).



2. Experimental

2.1. Crystal data

$2C_5H_6N_5^+\cdot SiF_6^{2-}\cdot 2H_2O$
M_r = 450.42
Triclinic, $P\overline{1}$
a = 5.7500 (7) Å
b = 7.8504 (3) Å
c = 10.0884 (6) Å
α = 79.141 (6)°
β = 84.534 (17)°
γ = 71.774 (9)°
V = 424.47 (6) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 0.24 mm^{-1}
T = 295 K
0.55 × 0.12 × 0.07 mm

2.2. Data collection

Nonius KappaCCD diffractometer
4025 measured reflections
1923 independent reflections
1757 reflections with $I > 2\sigma(I)$
R_{int} = 0.018

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.082
S = 1.03
1923 reflections
133 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1W—H1W···F2^{i}	0.85	1.88	2.7307 (14)	178
O1W—H2W···F3	0.80	1.95	2.7553 (14)	174
N1—H1···O1W	0.86	1.88	2.7059 (15)	162
N9—H9···N3^{ii}	0.86	2.13	2.9378 (17)	157
N9—H9···F1^{iii}	0.86	2.54	3.0009 (14)	115
N6—H6A···F3^{i}	0.86	1.98	2.7917 (14)	157
N6—H6A···F1^{iv}	0.86	2.61	3.2906 (15)	137
N6—H6B···N7^{v}	0.86	2.15	2.9648 (18)	159

Symmetry codes: (i) $x+1, y, z$; (ii) $-x, -y+2, -z+1$; (iii) $x, y+1, z-1$; (iv) $-x+1, -y, -z+2$; (v) $-x+2, -y+1, -z+1$.

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: *SIR92* (Burla *et al.*, 2005); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996), *ORTEP-3 for Windows* (Farrugia, 2012) and *PLATON* (Spek, 2009); software used to prepare material for publication: *WinGX* (Farrugia, 2012).

Acknowledgements

We acknowledge MESRS and ATRST (Ministére de l'Enseignement Supérieur et de la Recherche Scientifique Algérie) for financial support *via* the PNR programme.

Supporting information for this paper is available from the IUCr electronic archives (Reference: WM5099).

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S1. Experimental

Crystals of the title compound were grown from aqueous solution by dissolving 1 mmol SiO_2 and 2 mmol adenine in hydrofluoric acid (HF). The solutions were slowly evaporated to dryness for a couple of weeks. Some colourless crystals were isolated under a polarizing microscope for X-ray diffraction analysis.

S2. Refinement

All H atoms attached to C or N atoms were fixed geometrically and treated as riding with C—H = 0.93 Å and N—H = 0.86 Å with U_{iso}(H) = 1.2U_{eq}(C or N). H atoms of the water molecule were located in difference Fourier maps and included in the subsequent refinement using restraints (O—H= 0.82 (1) Å and H···H = 1.38 (2) Å) with U_{iso}(H) = 1.5U_{eq}(O).

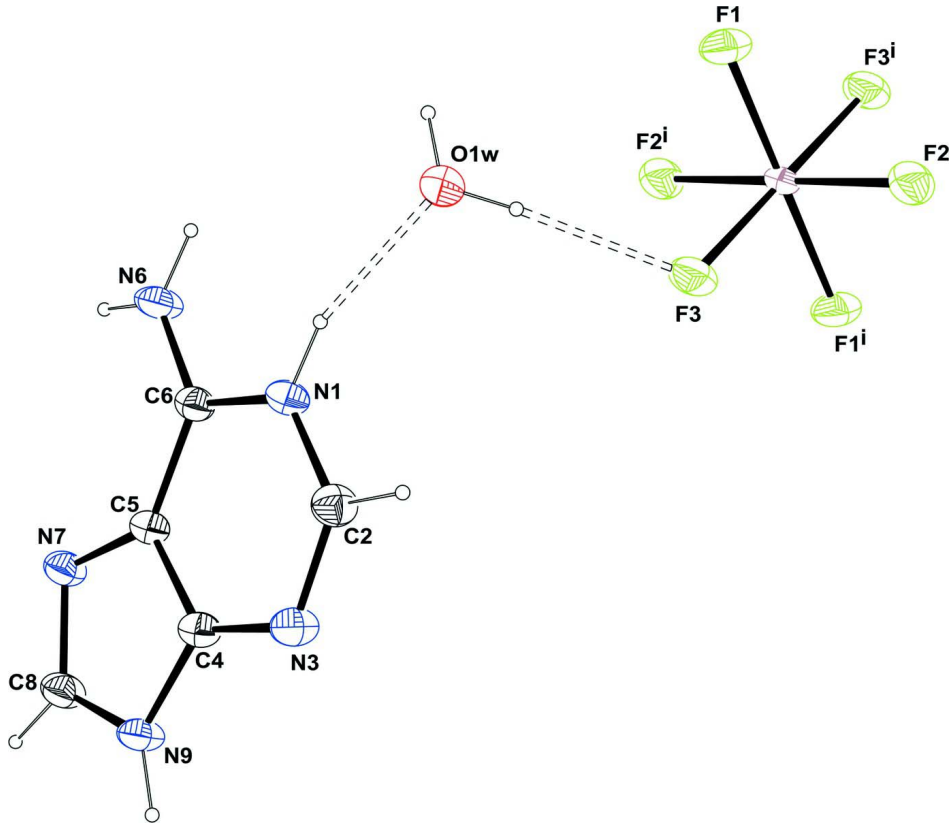

Figure 1

The principal structural units in the title compound. Displacement ellipsoids are drawn at the 30% probability level. H atoms are represented as small spheres of arbitrary radius. Hydrogen bonds are shown as dashed lines. [Symmetry code: (i) -*x*, -*y*, 2 - *z*]

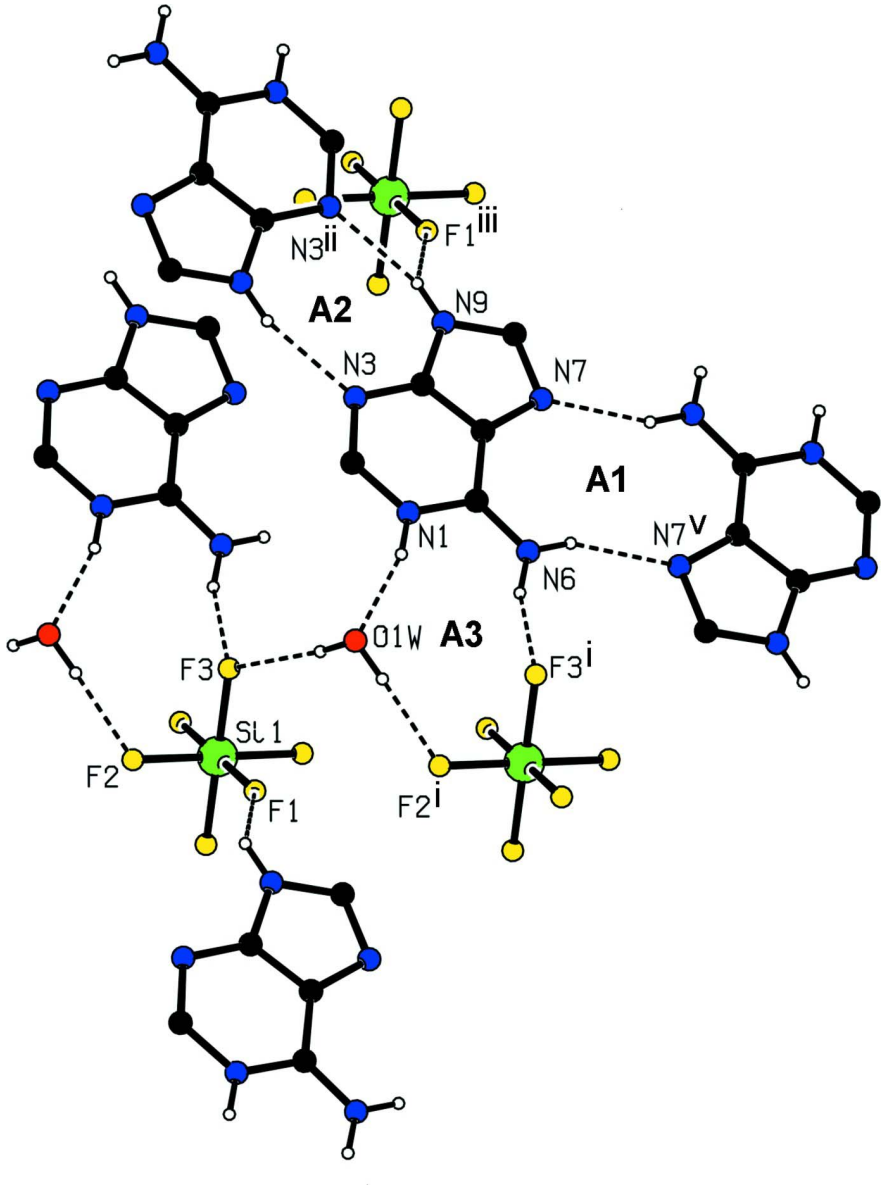

Figure 2

Partial packing view of the title compound, showing the formation of $R_2^2(10)$ (A1) and $R_2^2(8)$ (A2) rings through N—H···N, N—H···O, N—H···F and O—H···F hydrogen bonds. For the sake of clarity, H atoms not involved in hydrogen bonding have been omitted. [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.].

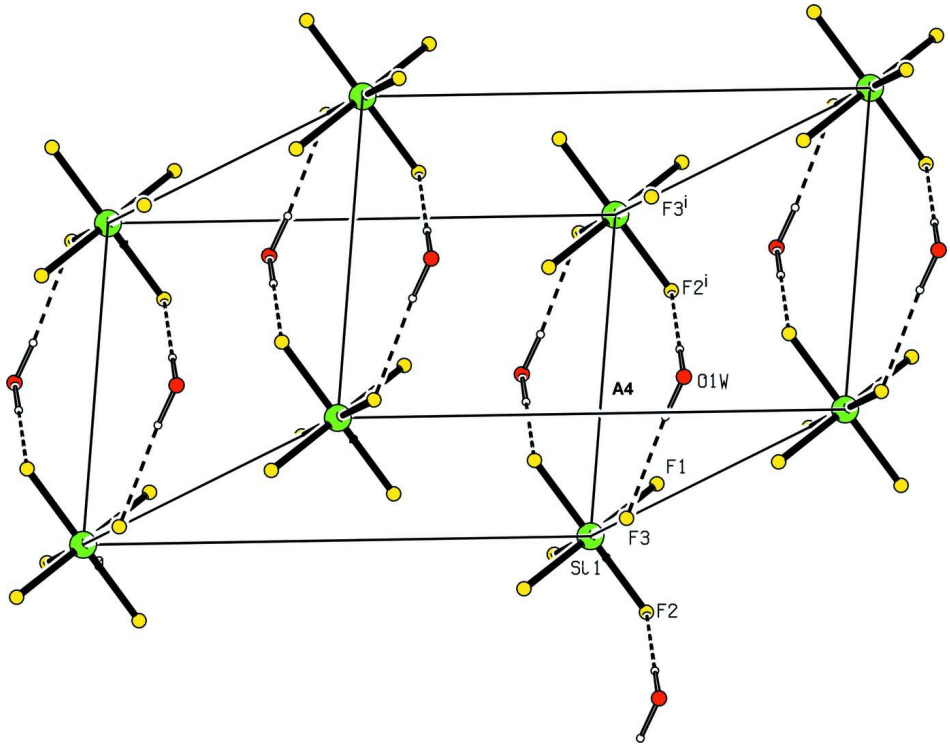

Figure 3

Partial packing view showing chains formed between water molecules and fluoridosilicate anions through O—H···F hydrogen bonds. For the sake of clarity, the cationic counterparts have been omitted. [Symmetry code: (i) $x + 1, y, z$]

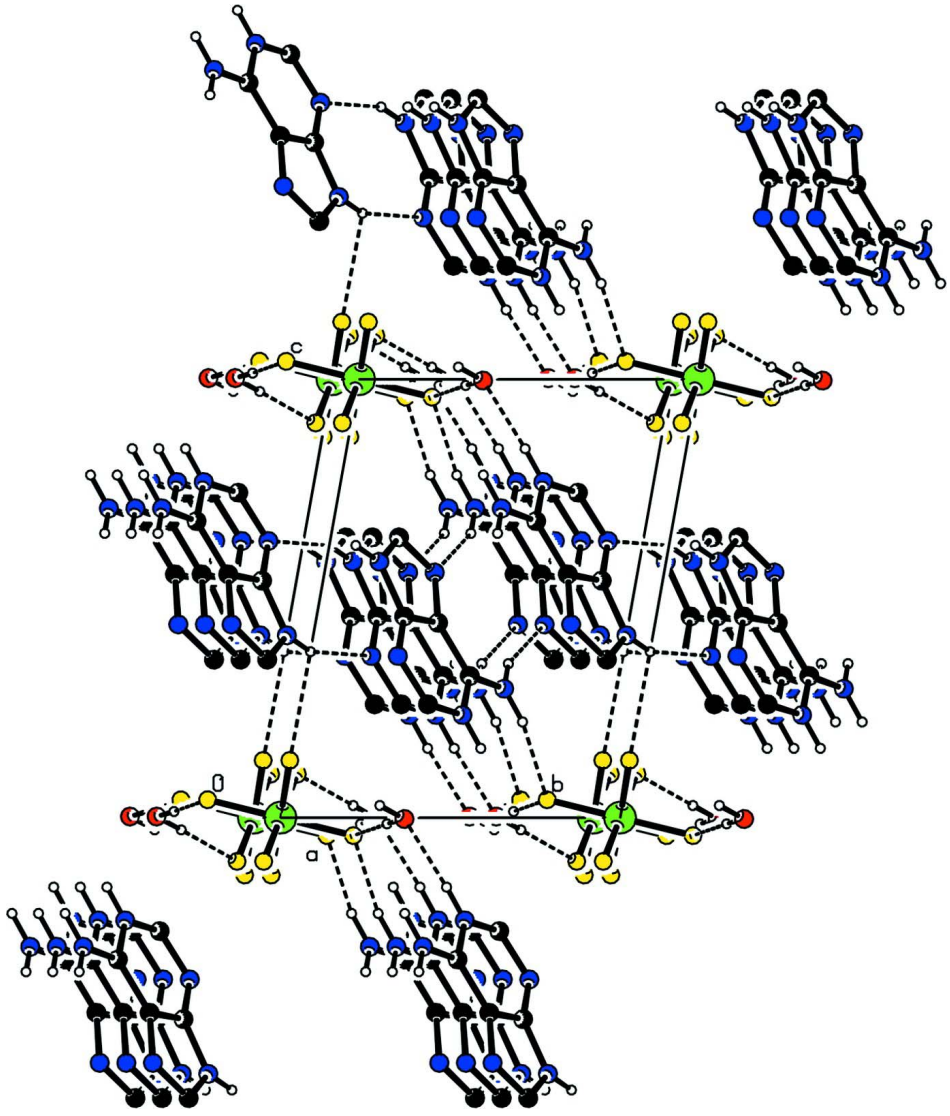

Figure 4

Packing view in a projection aproximately along [100] showing the formation of layers parallel to (001). Hydrogen bonds are shown as dashed lines. H atoms not involved in hydrogen bonding have been omitted for clarity.

Bis(9*H*-6-aminopurin-1-ium) hexafluoridosilicate(IV) dihydrate

Crystal data

$2C_5H_6N_5^+ \cdot SiF_6^{2-} \cdot 2H_2O$
$M_r = 450.42$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 5.7500\ (7)$ Å
$b = 7.8504\ (3)$ Å
$c = 10.0884\ (6)$ Å
$\alpha = 79.141\ (6)°$
$\beta = 84.534\ (17)°$
$\gamma = 71.774\ (9)°$
$V = 424.47\ (6)$ Å^3
$Z = 1$
$F(000) = 230$
$D_x = 1.762$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2981 reflections
$\theta = 5.2–27.5°$
$\mu = 0.24$ mm^{-1}
$T = 295$ K
Lath, colourless
0.55 × 0.12 × 0.07 mm

Data collection

Nonius KappaCCD diffractometer
Radiation source: fine-focus sealed tube
Detector resolution: 9 pixels mm^{-1}
CCD rotation images, thick slices scans
4025 measured reflections
1923 independent reflections
1757 reflections with $I > 2\sigma(I)$
$R_{int} = 0.018$
$\theta_{max} = 27.5°$, $\theta_{min} = 5.2°$
$h = -6 \rightarrow 7$
$k = -10 \rightarrow 10$
$l = -13 \rightarrow 13$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.030$
$wR(F^2) = 0.082$
$S = 1.03$
1923 reflections
133 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
$w = 1/[\sigma^2(F_o^2) + (0.0373P)^2 + 0.1468P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.28$ e Å^{-3}
$\Delta\rho_{min} = -0.22$ 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	0.0000	1.0000	0.02519 (13)
F1	0.16598 (16)	−0.01595 (12)	1.13081 (8)	0.0417 (2)
F2	−0.25721 (16)	0.04669 (12)	1.09945 (9)	0.0432 (2)
F3	−0.03226 (17)	0.22671 (11)	0.95904 (9)	0.0410 (2)
O1W	0.36312 (19)	0.33709 (13)	0.99720 (11)	0.0411 (3)
H1W	0.4831	0.2480	1.0279	0.062*
H2W	0.2551	0.2970	0.9859	0.062*
N1	0.4082 (2)	0.56497 (15)	0.76571 (10)	0.0304 (2)
H1	0.4261	0.4920	0.8417	0.036*
N7	0.7063 (2)	0.69851 (15)	0.44077 (11)	0.0322 (3)
N9	0.3304 (2)	0.89656 (15)	0.41590 (11)	0.0339 (3)
H9	0.2111	0.9840	0.3789	0.041*
N6	0.8149 (2)	0.42421 (16)	0.70564 (12)	0.0384 (3)
H6A	0.8298	0.3513	0.7817	0.046*
H6B	0.9376	0.4154	0.6487	0.046*
N3	0.1313 (2)	0.80786 (16)	0.63114 (11)	0.0344 (3)
C6	0.6057 (2)	0.54850 (16)	0.67661 (12)	0.0277 (3)

C4	0.3254 (2)	0.79502 (17)	0.54071 (12)	0.0287 (3)
C5	0.5581 (2)	0.67381 (16)	0.55526 (12)	0.0268 (3)
C8	0.5605 (3)	0.83344 (18)	0.36060 (14)	0.0347 (3)
H8	0.6093	0.8810	0.2748	0.042*
C2	0.1861 (3)	0.68935 (19)	0.74132 (13)	0.0334 (3)
H2	0.0632	0.6901	0.8084	0.040*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Si1	0.0258 (2)	0.0253 (2)	0.0204 (2)	−0.00695 (18)	−0.00429 (17)	0.00622 (16)
F1	0.0428 (5)	0.0461 (5)	0.0328 (4)	−0.0095 (4)	−0.0160 (4)	0.0021 (3)
F2	0.0341 (4)	0.0502 (5)	0.0354 (4)	−0.0076 (4)	0.0041 (3)	0.0054 (4)
F3	0.0494 (5)	0.0291 (4)	0.0412 (5)	−0.0126 (4)	−0.0109 (4)	0.0080 (3)
O1W	0.0376 (6)	0.0361 (5)	0.0458 (6)	−0.0123 (4)	−0.0092 (4)	0.0080 (4)
N1	0.0364 (6)	0.0310 (5)	0.0209 (5)	−0.0091 (4)	−0.0034 (4)	0.0016 (4)
N7	0.0338 (6)	0.0308 (5)	0.0266 (5)	−0.0067 (5)	−0.0001 (4)	0.0027 (4)
N9	0.0354 (6)	0.0298 (5)	0.0279 (6)	−0.0023 (5)	−0.0052 (4)	0.0050 (4)
N6	0.0354 (6)	0.0376 (6)	0.0297 (6)	−0.0022 (5)	−0.0020 (5)	0.0106 (5)
N3	0.0323 (6)	0.0355 (6)	0.0286 (6)	−0.0024 (5)	−0.0022 (4)	−0.0017 (4)
C6	0.0328 (6)	0.0260 (6)	0.0232 (6)	−0.0086 (5)	−0.0038 (5)	−0.0009 (4)
C4	0.0328 (7)	0.0265 (6)	0.0244 (6)	−0.0061 (5)	−0.0044 (5)	−0.0021 (4)
C5	0.0306 (6)	0.0247 (5)	0.0229 (6)	−0.0064 (5)	−0.0028 (5)	−0.0009 (4)
C8	0.0380 (7)	0.0332 (6)	0.0270 (6)	−0.0079 (5)	−0.0013 (5)	0.0049 (5)
C2	0.0348 (7)	0.0361 (7)	0.0271 (6)	−0.0081 (5)	0.0008 (5)	−0.0051 (5)

Geometric parameters (Å, º)

Si1—F1	1.6646 (8)	N9—C4	1.3605 (16)
Si1—F1^{i}	1.6646 (8)	N9—C8	1.3637 (19)
Si1—F2	1.6867 (9)	N9—H9	0.8600
Si1—F2^{i}	1.6867 (9)	N6—C6	1.3080 (17)
Si1—F3	1.7041 (8)	N6—H6A	0.8600
Si1—F3^{i}	1.7041 (8)	N6—H6B	0.8600
O1W—H1W	0.8476	N3—C2	1.3015 (17)
O1W—H2W	0.8046	N3—C4	1.3632 (17)
N1—C2	1.3543 (18)	C6—C5	1.4081 (16)
N1—C6	1.3681 (17)	C4—C5	1.3803 (18)
N1—H1	0.8600	C8—H8	0.9300
N7—C8	1.3154 (17)	C2—H2	0.9300
N7—C5	1.3892 (16)		
F1—Si1—F1^{i}	180.000 (1)	C4—N9—H9	126.5
F1—Si1—F2	89.86 (5)	C8—N9—H9	126.5
F1^{i}—Si1—F2	90.14 (5)	C6—N6—H6A	120.0
F1—Si1—F2^{i}	90.14 (5)	C6—N6—H6B	120.0
F1^{i}—Si1—F2^{i}	89.86 (5)	H6A—N6—H6B	120.0
F2—Si1—F2^{i}	180.0	C2—N3—C4	112.33 (12)

F1—Si1—F3	90.31 (4)	N6—C6—N1	120.97 (11)
F1^{i}—Si1—F3	89.69 (4)	N6—C6—C5	125.23 (12)
F2—Si1—F3	89.68 (5)	N1—C6—C5	113.80 (11)
F2^{i}—Si1—F3	90.32 (5)	N9—C4—N3	127.47 (12)
F1—Si1—F3^{i}	89.69 (4)	N9—C4—C5	105.20 (11)
F1^{i}—Si1—F3^{i}	90.31 (4)	N3—C4—C5	127.32 (11)
F2—Si1—F3^{i}	90.32 (5)	C4—C5—N7	111.11 (11)
F2^{i}—Si1—F3^{i}	89.68 (5)	C4—C5—C6	117.64 (12)
F3—Si1—F3^{i}	180.000 (1)	N7—C5—C6	131.23 (12)
H1W—O1W—H2W	107.5	N7—C8—N9	113.25 (12)
C2—N1—C6	123.90 (11)	N7—C8—H8	123.4
C2—N1—H1	118.1	N9—C8—H8	123.4
C6—N1—H1	118.1	N3—C2—N1	125.00 (13)
C8—N7—C5	103.37 (11)	N3—C2—H2	117.5
C4—N9—C8	107.06 (11)	N1—C2—H2	117.5

Symmetry code: (i) $-x, -y, -z+2$.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*W*—H1*W*···F2^{ii}	0.85	1.88	2.7307 (14)	178
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N9—H9···N3^{iii}	0.86	2.13	2.9378 (17)	157
N9—H9···F1^{iv}	0.86	2.54	3.0009 (14)	115
N6—H6*A*···F3^{ii}	0.86	1.98	2.7917 (14)	157
N6—H6*A*···F1^{v}	0.86	2.61	3.2906 (15)	137
N6—H6*B*···N7^{vi}	0.86	2.15	2.9648 (18)	159

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