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Crystal structure of 2-cyano-1-methylpyridinium tetrafluoroborate

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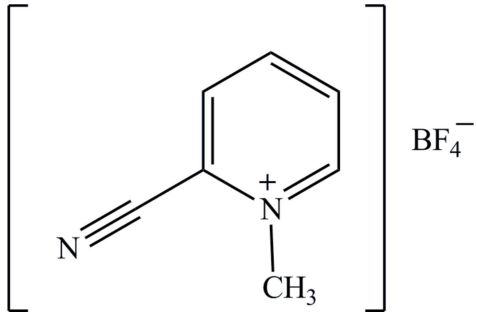
The asymmetric unit of the title salt, $C_7H_7N_2^+\cdot BF_4^-$, comprises two independent but nearly identical formula units. The solid-state structure comprises corrugated layers of cations and anions, formed by C—H···F hydrogen bonding, that are approximately parallel to (010). Further C—H···F hydrogen bonding consolidates the three-dimensional architecture. The sample was refined as a two-component non-merohedral twin.

Keywords: crystal structure; salt; C—H···F interactions.

CCDC reference: 1420782

1. Related literature

For structures of other salts of the 2-cyano-1-methylpyridinium cation, see: Koplitz *et al.* (2012); Kammer *et al.* (2013). For structures of salts of the isomeric 2-cyanoanilinium cation, see: Zhang (2009); Cui & Chen (2010).



2. Experimental

2.1. Crystal data

$C_7H_7N_2^+\cdot BF_4^-$
M_r = 205.96
Monoclinic, $P2_1$
a = 7.9704 (16) Å
b = 7.5527 (15) Å
c = 14.570 (3) Å
β = 90.312 (3)°
V = 877.1 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.15 mm^{-1}
T = 150 K
0.14 × 0.13 × 0.08 mm

2.2. Data collection

Bruker SMART APEX CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2014)
T_{min} = 0.70, T_{max} = 0.99
16120 measured reflections
4566 independent reflections
3779 reflections with $I > 2\sigma(I)$
R_{int} = 0.057

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.122
S = 1.08
4566 reflections
256 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}
Absolute structure: the absolute structure could not be determined with certainty in this light-atom structure

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

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1A···F7^{i}	0.98	2.50	3.407 (6)	154
C1—H1B···F8^{ii}	0.98	2.54	3.498 (6)	166
C1—H1C···F3^{iii}	0.98	2.47	3.214 (5)	132
C2—H2···F7^{i}	0.95	2.29	3.190 (5)	157
C3—H3···F1^{iv}	0.95	2.46	3.294 (6)	147
C5—H5···F1^{v}	0.95	2.45	3.306 (5)	149
C8—H8A···F2^{i}	0.98	2.48	3.159 (6)	126
C8—H8C···F3^{ii}	0.98	2.55	3.437 (6)	151
C9—H9···F3^{ii}	0.95	2.52	3.392 (6)	152
C9—H9···F4^{ii}	0.95	2.59	3.476 (6)	156
C10—H10···F6^{ii}	0.95	2.54	3.167 (6)	123
C12—H12···F5^{i}	0.95	2.49	3.277 (6)	141

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

Data collection: *APEX2* (Bruker, 2014); cell refinement: *SAINT* (Bruker, 2014); data reduction: *SAINT*; program(s) used to solve structure: *SHELXT* (Sheldrick, 2015*a*); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015*b*); molecular graphics: *DIAMOND* (Brandenburg & Putz, 2012); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008) and *PLATON* (Spek, 2009).

Acknowledgements

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Supporting information for this paper is available from the IUCr electronic archives (Reference: TK5380).

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supporting information

Acta Cryst. (2015). E**71**, o697–o698 [doi:10.1107/S2056989015016011]

Crystal structure of 2-cyano-1-methylpyridinium tetrafluoroborate

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S1. Comment

The asymmetric unit consists of two independent formula units. A portion of the C—H···F hydrogen bonding network which aids the packing of the several ions is shown in Fig. 1 with fuller depictions appearing in Figs 2 and 3. The solid state structure comprises corrugated layers of cations and anions formed by C—H···F hydrogen bonding between them and approximately parallel to (010). These layers are held to one another by additional C—H···F interactions.

S2. Experimental

To 0.64 g (0.5 mmol) of 2-cyano-1-methylpyridinium iodide dissolved in 8.5 ml of 95% ethanol was added 1.08 g (0.55 mmol) of solid silver tetrafluoroborate with stirring. The reaction mixture was filtered to remove the precipitated AgI and the filtrate allowed to evaporate to dryness. From the resulting mass, crystals suitable for X-ray diffraction were selected.

S3. Refinement

The H-atoms attached to carbon were placed in calculated positions (C—H = 0.95 - 0.98 Å). All were included as riding contributions with isotropic displacement parameters 1.2 - 1.5 times those of the attached atoms. In the late stages of the refinement a consistent pattern of $F_o^2 >> F_c^2$ suggested twinning not yet accounted for. Use of the *TwinRotMat* routine in *PLATON* (Spek, 2009) generated the twin law -1 0 0 0 - 1 0 0 0 1, inclusion of which enabled satisfactory refinement as a 2-component twin.

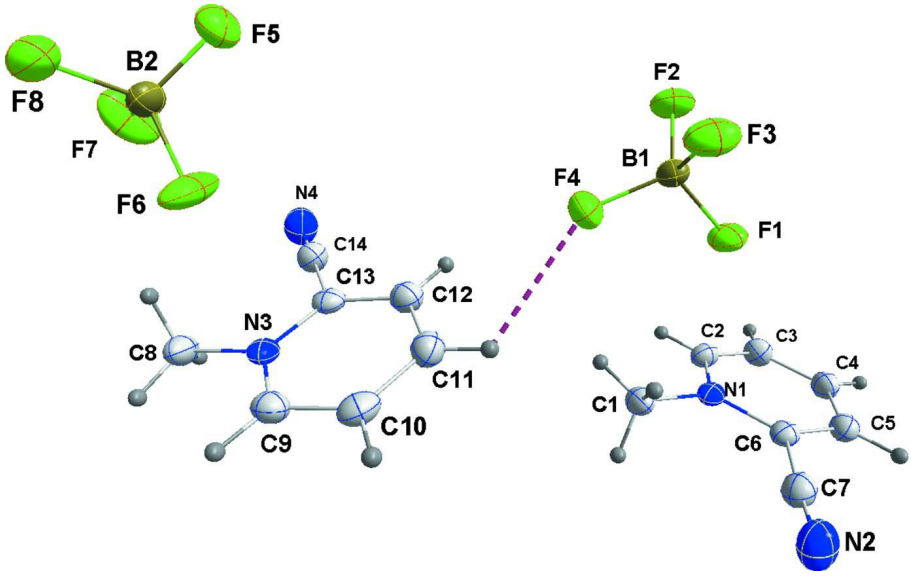

Figure 1

Perspective view of the asymmetric unit with 50% probability ellipsoids. The C—H···F interaction is shown by a dotted line.

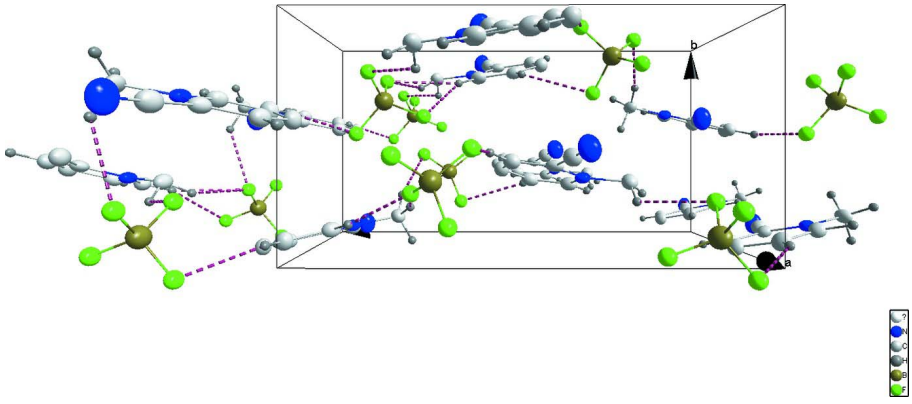

Figure 2

Packing viewed down the *a* axis showing an edge view of two corrugated layers and the C—H···F interactions (dotted lines) holding them together.

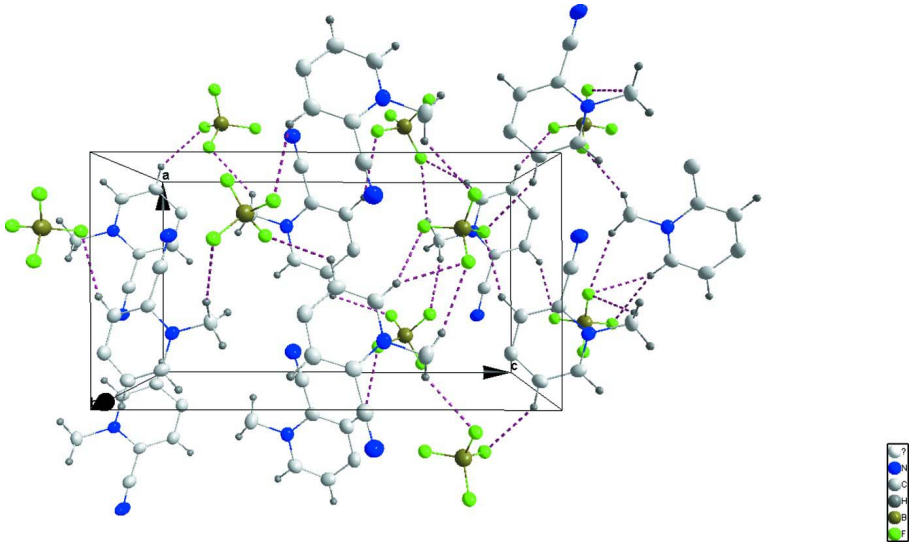

Figure 3

Packing viewed down the *b* axis providing a plan view of the corrugated sheets with C—H···F interactions shown as dotted lines.

2-Cyano-1-methylpyridinium tetrafluoroborate

Crystal data

$C_7H_7N_2^+\cdot BF_4^-$
M_r = 205.96
Monoclinic, $P2_1$
a = 7.9704 (16) Å
b = 7.5527 (15) Å
c = 14.570 (3) Å
β = 90.312 (3)°
V = 877.1 (3) Å^3
Z = 4
$F(000)$ = 416
D_x = 1.560 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 8457 reflections
θ = 2.6–29.0°
μ = 0.15 mm^{-1}
T = 150 K
Block, colourless
0.14 × 0.13 × 0.08 mm

Data collection

Bruker SMART APEX CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 8.3660 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2014)
T_{min} = 0.70, T_{max} = 0.99
16120 measured reflections
4566 independent reflections
3779 reflections with $I > 2\sigma(I)$
R_{int} = 0.057
θ_{max} = 29.3°, θ_{min} = 2.6°
h = −10→10
k = −10→10
l = −19→19

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.122
S = 1.08
4566 reflections
256 parameters
1 restraint
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
$w = 1/[\sigma^2(F_o^2) + (0.0572P)^2 + 0.091P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.33$ e Å^{-3}
$\Delta\rho_{min} = -0.27$ e Å^{-3}
Absolute structure: The absolute structure could not be determined with certainty in this light-atom structure

Special details

Experimental. The diffraction data were obtained from 3 sets of 400 frames, each of width 0.5° in ω, collected at φ = 0.00, 90.00 and 180.00° and 2 sets of 800 frames, each of width 0.45° in φ, collected at ω = -30.00 and 210.00°. The scan time was 10 sec/frame.

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. H-atoms attached to carbon were placed in calculated positions (C—H = 0.95 - 0.98 Å). All were included as riding contributions with isotropic displacement parameters 1.2 - 1.5 times those of the attached atoms. In the late stages of the refinement a consistent pattern of $F_o^2 >> F_c^2$ suggested twinning not yet accounted for. Use of the *TwinRotMat* routine in *PLATON* (Spek, 2009) generated the twin law -1 0 0 0 - 1 0 0 0 1, inclusion of which enabled satisfactory refinement as a 2-component twin.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.7369 (5)	0.1294 (4)	0.8819 (3)	0.0220 (7)
N2	0.3067 (5)	0.0851 (7)	0.8963 (3)	0.0416 (11)
C1	0.6868 (6)	0.1642 (6)	0.7854 (3)	0.0273 (10)
H1A	0.7826	0.2126	0.7518	0.041*
H1B	0.5943	0.2497	0.7841	0.041*
H1C	0.6504	0.0534	0.7565	0.041*
C2	0.8990 (6)	0.1331 (6)	0.9067 (3)	0.0264 (9)
H2	0.9807	0.1629	0.8620	0.032*
C3	0.9511 (6)	0.0952 (7)	0.9947 (3)	0.0299 (10)
H3	1.0671	0.0973	1.0100	0.036*
C4	0.8341 (6)	0.0543 (6)	1.0601 (3)	0.0283 (10)
H4	0.8678	0.0281	1.1213	0.034*
C5	0.6652 (6)	0.0520 (6)	1.0352 (3)	0.0274 (9)
H5	0.5819	0.0254	1.0795	0.033*
C6	0.6204 (5)	0.0884 (6)	0.9464 (3)	0.0232 (8)
C7	0.4472 (6)	0.0856 (7)	0.9165 (3)	0.0295 (10)
B1	0.7236 (7)	0.6589 (6)	0.8140 (3)	0.0242 (10)
F1	0.7151 (4)	0.5195 (3)	0.8770 (2)	0.0328 (6)
F2	0.8600 (4)	0.7654 (4)	0.8350 (2)	0.0361 (7)
F3	0.5771 (3)	0.7603 (4)	0.8200 (2)	0.0389 (7)
F4	0.7353 (4)	0.5925 (4)	0.72573 (19)	0.0446 (8)
N3	0.7497 (5)	0.3325 (5)	0.3758 (2)	0.0246 (8)
N4	1.1617 (6)	0.4550 (8)	0.3990 (3)	0.0454 (12)
C8	0.8099 (6)	0.3192 (7)	0.2807 (3)	0.0316 (10)

H8A	0.9110	0.2455	0.2790	0.047*
H8B	0.8361	0.4378	0.2575	0.047*
H8C	0.7225	0.2655	0.2421	0.047*
C9	0.5886 (6)	0.2988 (6)	0.3939 (3)	0.0292 (10)
H9	0.5148	0.2630	0.3460	0.035*
C10	0.5294 (6)	0.3159 (6)	0.4825 (3)	0.0302 (10)
H10	0.4148	0.2926	0.4954	0.036*
C11	0.6365 (6)	0.3668 (7)	0.5519 (3)	0.0310 (10)
H11	0.5962	0.3775	0.6129	0.037*
C12	0.8039 (6)	0.4028 (6)	0.5327 (3)	0.0304 (10)
H12	0.8794	0.4387	0.5798	0.036*
C13	0.8566 (6)	0.3847 (6)	0.4439 (3)	0.0255 (9)
C14	1.0267 (7)	0.4226 (7)	0.4190 (3)	0.0327 (10)
B2	0.7732 (7)	0.8421 (7)	0.3142 (3)	0.0273 (10)
F5	0.8153 (4)	0.9543 (4)	0.3856 (2)	0.0435 (8)
F6	0.6889 (5)	0.6960 (4)	0.3476 (2)	0.0500 (9)
F7	0.9153 (4)	0.7890 (6)	0.2691 (2)	0.0569 (10)
F8	0.6664 (4)	0.9294 (4)	0.2535 (2)	0.0464 (8)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0256 (18)	0.0142 (16)	0.0263 (17)	0.0013 (14)	0.0025 (15)	−0.0004 (14)
N2	0.029 (2)	0.059 (3)	0.038 (2)	0.004 (2)	0.0041 (18)	−0.002 (2)
C1	0.037 (3)	0.022 (2)	0.023 (2)	−0.0010 (18)	−0.0026 (18)	0.0022 (17)
C2	0.023 (2)	0.023 (2)	0.033 (2)	−0.0011 (16)	0.0046 (19)	−0.0008 (19)
C3	0.025 (2)	0.033 (2)	0.032 (2)	0.0022 (19)	−0.0025 (19)	−0.002 (2)
C4	0.031 (2)	0.029 (2)	0.025 (2)	0.0000 (18)	−0.0006 (18)	−0.0012 (18)
C5	0.031 (2)	0.024 (2)	0.027 (2)	0.0002 (18)	0.0039 (19)	−0.0005 (18)
C6	0.023 (2)	0.0174 (19)	0.029 (2)	0.0007 (16)	0.0041 (17)	−0.0020 (17)
C7	0.029 (2)	0.028 (2)	0.032 (2)	0.0002 (18)	0.0046 (19)	−0.001 (2)
B1	0.029 (2)	0.018 (2)	0.026 (2)	−0.0007 (19)	0.001 (2)	0.0018 (18)
F1	0.0440 (15)	0.0189 (12)	0.0355 (14)	−0.0006 (12)	−0.0028 (13)	0.0047 (10)
F2	0.0336 (15)	0.0254 (14)	0.0492 (17)	−0.0069 (12)	−0.0028 (13)	0.0005 (12)
F3	0.0311 (15)	0.0235 (13)	0.062 (2)	0.0040 (12)	0.0021 (14)	0.0036 (13)
F4	0.069 (2)	0.0343 (16)	0.0304 (14)	−0.0020 (16)	0.0062 (15)	−0.0067 (13)
N3	0.0326 (19)	0.0141 (15)	0.0270 (18)	0.0007 (14)	0.0007 (15)	−0.0004 (14)
N4	0.034 (2)	0.060 (3)	0.042 (2)	−0.008 (2)	0.000 (2)	0.005 (2)
C8	0.041 (3)	0.025 (2)	0.028 (2)	−0.004 (2)	0.004 (2)	0.0001 (19)
C9	0.031 (2)	0.023 (2)	0.034 (2)	−0.0023 (17)	−0.005 (2)	0.0003 (19)
C10	0.029 (2)	0.023 (2)	0.039 (3)	−0.0003 (18)	0.0034 (19)	0.005 (2)
C11	0.037 (2)	0.029 (2)	0.027 (2)	0.0017 (19)	0.001 (2)	0.0019 (19)
C12	0.034 (2)	0.026 (2)	0.032 (2)	0.000 (2)	−0.003 (2)	0.0016 (19)
C13	0.029 (2)	0.0147 (18)	0.033 (2)	0.0023 (16)	−0.0026 (19)	0.0026 (17)
C14	0.035 (3)	0.031 (2)	0.032 (2)	−0.001 (2)	−0.002 (2)	0.002 (2)
B2	0.033 (3)	0.023 (2)	0.026 (2)	−0.002 (2)	0.002 (2)	0.004 (2)
F5	0.061 (2)	0.0328 (16)	0.0363 (16)	0.0103 (14)	−0.0076 (16)	−0.0078 (13)
F6	0.072 (2)	0.0196 (14)	0.059 (2)	0.0019 (15)	0.0198 (17)	0.0098 (14)

F7	0.0376 (17)	0.072 (3)	0.061 (2)	0.0042 (17)	0.0157 (15)	−0.0251 (19)
F8	0.057 (2)	0.0294 (16)	0.0522 (18)	−0.0021 (14)	−0.0197 (17)	0.0099 (14)

Geometric parameters (Å, º)

N1—C2	1.340 (6)	N3—C9	1.336 (6)
N1—C6	1.360 (6)	N3—C13	1.362 (6)
N1—C1	1.484 (6)	N3—C8	1.473 (6)
N2—C7	1.157 (6)	N4—C14	1.143 (7)
C1—H1A	0.9800	C8—H8A	0.9800
C1—H1B	0.9800	C8—H8B	0.9800
C1—H1C	0.9800	C8—H8C	0.9800
C2—C3	1.376 (7)	C9—C10	1.382 (7)
C2—H2	0.9500	C9—H9	0.9500
C3—C4	1.372 (7)	C10—C11	1.375 (7)
C3—H3	0.9500	C10—H10	0.9500
C4—C5	1.392 (6)	C11—C12	1.392 (7)
C4—H4	0.9500	C11—H11	0.9500
C5—C6	1.369 (6)	C12—C13	1.370 (6)
C5—H5	0.9500	C12—H12	0.9500
C6—C7	1.446 (6)	C13—C14	1.434 (7)
B1—F4	1.384 (6)	B2—F7	1.372 (6)
B1—F2	1.385 (6)	B2—F6	1.382 (6)
B1—F1	1.398 (5)	B2—F5	1.382 (6)
B1—F3	1.400 (6)	B2—F8	1.391 (6)
C2—N1—C6	118.7 (4)	C9—N3—C13	120.6 (4)
C2—N1—C1	120.4 (4)	C9—N3—C8	119.4 (4)
C6—N1—C1	120.9 (4)	C13—N3—C8	119.9 (4)
N1—C1—H1A	109.5	N3—C8—H8A	109.5
N1—C1—H1B	109.5	N3—C8—H8B	109.5
H1A—C1—H1B	109.5	H8A—C8—H8B	109.5
N1—C1—H1C	109.5	N3—C8—H8C	109.5
H1A—C1—H1C	109.5	H8A—C8—H8C	109.5
H1B—C1—H1C	109.5	H8B—C8—H8C	109.5
N1—C2—C3	122.1 (4)	N3—C9—C10	119.9 (5)
N1—C2—H2	118.9	N3—C9—H9	120.0
C3—C2—H2	118.9	C10—C9—H9	120.0
C4—C3—C2	119.5 (4)	C11—C10—C9	120.0 (4)
C4—C3—H3	120.3	C11—C10—H10	120.0
C2—C3—H3	120.3	C9—C10—H10	120.0
C3—C4—C5	118.8 (4)	C10—C11—C12	119.9 (4)
C3—C4—H4	120.6	C10—C11—H11	120.0
C5—C4—H4	120.6	C12—C11—H11	120.0
C6—C5—C4	119.4 (4)	C13—C12—C11	118.0 (5)
C6—C5—H5	120.3	C13—C12—H12	121.0
C4—C5—H5	120.3	C11—C12—H12	121.0
N1—C6—C5	121.5 (4)	N3—C13—C12	121.5 (4)

N1—C6—C7	116.7 (4)	N3—C13—C14	117.6 (4)
C5—C6—C7	121.7 (4)	C12—C13—C14	120.9 (5)
N2—C7—C6	177.1 (5)	N4—C14—C13	179.1 (6)
F4—B1—F2	111.1 (4)	F7—B2—F6	109.9 (4)
F4—B1—F1	109.9 (4)	F7—B2—F5	110.0 (4)
F2—B1—F1	109.5 (4)	F6—B2—F5	110.0 (4)
F4—B1—F3	108.5 (4)	F7—B2—F8	109.7 (4)
F2—B1—F3	108.8 (4)	F6—B2—F8	107.8 (4)
F1—B1—F3	109.1 (4)	F5—B2—F8	109.5 (4)
C6—N1—C2—C3	0.7 (7)	C13—N3—C9—C10	−0.4 (7)
C1—N1—C2—C3	−177.6 (4)	C8—N3—C9—C10	−178.0 (4)
N1—C2—C3—C4	−0.9 (8)	N3—C9—C10—C11	−0.3 (7)
C2—C3—C4—C5	0.2 (8)	C9—C10—C11—C12	0.7 (8)
C3—C4—C5—C6	0.7 (7)	C10—C11—C12—C13	−0.3 (7)
C2—N1—C6—C5	0.2 (6)	C9—N3—C13—C12	0.7 (7)
C1—N1—C6—C5	178.5 (4)	C8—N3—C13—C12	178.4 (4)
C2—N1—C6—C7	−179.9 (4)	C9—N3—C13—C14	−178.6 (4)
C1—N1—C6—C7	−1.6 (6)	C8—N3—C13—C14	−0.9 (7)
C4—C5—C6—N1	−0.9 (7)	C11—C12—C13—N3	−0.3 (7)
C4—C5—C6—C7	179.2 (5)	C11—C12—C13—C14	178.9 (5)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1*A*···F7^{i}	0.98	2.50	3.407 (6)	154
C1—H1*B*···F8^{ii}	0.98	2.54	3.498 (6)	166
C1—H1*C*···F3^{iii}	0.98	2.47	3.214 (5)	132
C2—H2···F7^{i}	0.95	2.29	3.190 (5)	157
C3—H3···F1^{iv}	0.95	2.46	3.294 (6)	147
C5—H5···F1^{v}	0.95	2.45	3.306 (5)	149
C8—H8*A*···F2^{i}	0.98	2.48	3.159 (6)	126
C8—H8*C*···F3^{ii}	0.98	2.55	3.437 (6)	151
C9—H9···F3^{ii}	0.95	2.52	3.392 (6)	152
C9—H9···F4^{ii}	0.95	2.59	3.476 (6)	156
C10—H10···F6^{ii}	0.95	2.54	3.167 (6)	123
C12—H12···F5^{i}	0.95	2.49	3.277 (6)	141

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