

Acta Crystallographica Section E

Structure Reports

Online

ISSN 1600-5368

Potassium [(1*S*)-1-azido-2-phenylethyl]-trifluoridoborateTore Lejon,^{a*} Alexey S. Gorovoy^a and Victor N. Khrustalev^b

^aDepartment of Chemistry, Faculty of Science and Technology, University of Tromsø, N-9037 Tromsø, Norway, and ^bX-Ray Structural Centre, A.N. Nesmeyanov Institute of Organoelement Compounds, Russian Academy of Sciences, 28 Vavilov Street, B-334, Moscow 119991, Russian Federation
Correspondence e-mail: tore.lejon@uit.no

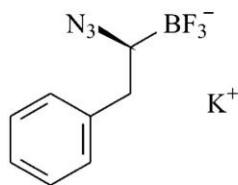
Received 27 June 2012; accepted 2 July 2012

Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.007$ Å; R factor = 0.050; wR factor = 0.081; data-to-parameter ratio = 14.3.

The title compound, $\text{K}^+\cdot\text{C}_8\text{H}_8\text{BF}_3\text{N}_3^-$, is a salt containing the chiral organic trifluoridoborate anion. The organic anions and potassium cations are tightly bound to each other by the coordination $\text{K}-\text{F}$ [2.654 (3)–3.102 (3) Å] and $\text{K}-\text{N}$ [2.951 (4)–3.338 (4) Å] interactions. Thus, the potassium cation adopts a nine-vertex coordination polyhedron, which can be described as a distorted monocapped tetragonal antiprism. In the crystal, the organic anions and potassium cations form layers parallel to (001). Weak $\text{C}-\text{H}\cdots\pi$ interactions between neighbouring phenyl rings further stabilize the crystal.

Related literature

For the Matteson homologation, see: Matteson & Kim (2002); Matteson *et al.* (2006). For related compounds, see: Matteson & Beedle (1987); Scriven & Turnbull (1988); Darses & Genet (2008); Huang *et al.* (2009).



Experimental

Crystal data

$\text{K}^+\cdot\text{C}_8\text{H}_8\text{BF}_3\text{N}_3^-$
 $M_r = 253.08$

Orthorhombic, $P2_12_12_1$
 $a = 6.052$ (2) Å

$b = 6.959$ (2) Å
 $c = 25.120$ (8) Å
 $V = 1057.9$ (6) Å³
 $Z = 4$

Mo $K\alpha$ radiation
 $\mu = 0.52$ mm⁻¹
 $T = 100$ K
 $0.15 \times 0.10 \times 0.01$ mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (SADABS; Sheldrick, 2003)
 $T_{\min} = 0.926$, $T_{\max} = 0.995$

10239 measured reflections
2075 independent reflections
1203 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.060$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.050$
 $wR(F^2) = 0.081$
 $S = 0.98$
2075 reflections
145 parameters
H-atom parameters constrained

$\Delta\rho_{\text{max}} = 0.33$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.40$ e Å⁻³
Absolute structure: Flack (1983),
828 Friedel pairs
Flack parameter: 0.00 (8)

Table 1

Weak $\text{C}-\text{H}\cdots\pi$ interactions between neighbouring phenyl rings (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{C4}-\text{H4}\cdots\text{C3}^i$	0.95	2.93	3.528 (6)	122
$\text{C4}-\text{H4}\cdots\text{C4}^i$	0.95	2.98	3.823 (6)	149
$\text{C4}-\text{H4}\cdots\text{C5}^i$	0.95	3.08	3.974 (7)	158
$\text{C4}-\text{H4}\cdots\text{C6}^i$	0.95	3.13	3.841 (7)	133
$\text{C4}-\text{H4}\cdots\text{C7}^i$	0.95	3.09	3.556 (7)	112
$\text{C4}-\text{H4}\cdots\text{C8}^i$	0.95	2.98	3.382 (6)	107

Symmetry code: (i) $x - \frac{1}{2}, -y + \frac{1}{2}, -z + 2$.

Data collection: APEX2 (Bruker, 2005); cell refinement: SAINT (Bruker, 2001); data reduction: SAINT; program(s) used to solve structure: SHELXTL (Sheldrick, 2008); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL.

Support from the Norwegian Science Council and the FORNY program is gratefully acknowledged.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV5318).

References

- Bruker (2001). SAINT. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2005). APEX2. Bruker AXS Inc., Madison, Wisconsin, USA.
Darses, S. & Genet, J. P. (2008). *Chem. Rev.* **108**, 288–325.
Flack, H. D. (1983). *Acta Cryst.* **A39**, 876–881.
Huang, J., Macdonald, S. J. F., Cooper, A. W. J., Fisher, G. & Harrity, J. P. A. (2009). *Tetrahedron Lett.* **50**, 5539–5541.
Matteson, D. S. & Beedle, E. C. (1987). *Tetrahedron Lett.* **28**, 4499–4502.
Matteson, D. S. & Kim, B. (2002). *Org. Lett.* **4**, 2153–2155.
Matteson, D. S., Maliakal, D., Pharazyn, P. S. & Kim, B. (2006). *Synlett*, pp. 3501–3503.
Scriven, E. F. V. & Turnbull, K. (1988). *Chem. Rev.* **88**, 297–368.
Sheldrick, G. M. (2003). SADABS. Bruker AXS Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.

supplementary materials

Acta Cryst. (2012). E68, m1048 [doi:10.1107/S1600536812030085]

Potassium [(1*S*)-1-azido-2-phenylethyl]trifluoridoborate**Tore Lejon, Alexey S. Gorovoy and Victor N. Khurstalev****Comment**

The target compound was synthesized as part of our studies on the Matteson homologation and the synthesis of chiral amino boronates. It was accomplished using potassium or caesium bifluorides which have been shown to be useful reagents in the deprotection of boronic esters (Matteson & Kim, 2002; Matteson *et al.*, 2006). However, organotrifluoridoborates are not only used for preservation and release of the boronic acid moiety, but also find application in different synthetic transformations (Darses & Genet, 2008). Moreover, organic azides have a rich chemistry and could prove important as building blocks in click chemistry (Scriven & Turnbull, 1988; Huang *et al.*, 2009).

The title compound - potassium [(1*S*)-1-azido-2-phenyl-ethyl]-trifluoro-boranuide, C₈H₈BF₃KN₃ (**I**), is a salt containing the chiral organic trifluoridoborate anion (Figure 1). It crystallizes in the orthorhombic space group *P*2₁2₁2₁, with one crystallographically independent formula unit within the lattice cell. The asymmetrical C1 carbon atom has the *S*-configuration. The organic anions and potassium cations are tightly bound to each other by the coordination K—F [K1—F1 2.648 (3), K1—F1ⁱ 2.673 (3), K1—F2ⁱ 2.933 (3), K1—F2ⁱⁱ 2.685 (3), K1—F3ⁱⁱ 3.102 (3) and K1—F3ⁱⁱⁱ 2.654 (3) Å] and K—N [K1—N1 2.951 (4), K1—N3ⁱⁱⁱ 3.338 (4) and K1—N3^{iv} 3.020 (4) Å] interactions [symmetry codes: (i) $-x, y + 1/2, -z + 3/2$; (ii) $x, y + 1, z$; (iii) $-x + 1, y + 1/2, -z + 3/2$; (iv) $x - 1, y + 1, z$]. Thus, the potassium cation adopts the 9-vertex coordination polyhedron, which can be described as a distorted monocapped tetragonal antiprism (Figure 2).

In the crystal, the organic anions and potassium cations form the layers parallel to (001) (Figure 3). Moreover, the crystal packing is stabilized by the additional C4—H4 $\cdots\pi$ interactions between the phenyl rings, with C4 \cdots Centroid^v distance of 3.418 (3) Å [symmetry code: (v) $x - 1/2, -y + 1/2, -z + 2$].

Experimental

The initial 2-[(1*S*)-1-azido-2-phenyl-ethyl]-hexahydro-3a,5,5-trimethyl-4,6-methano-1,3,2-benzodioxaborole was obtained according to the procedure described earlier (Matteson & Beedle, 1987). A saturated water solution of potassium bifluoride (2.4 g, 30 mmol) was added drop wise to a solution of 2-[(1*S*)-1-azido-2-phenyl-ethyl]-hexahydro-3a,5,5-trimethyl-4,6-methano-1,3,2-benzodioxaborole (1.0 g, 3 mmol) in methanol (25 ml) at room temperature (Figure 4). The reaction mixture was stirred for 24 h. Then, the solvents were evaporated under reduced pressure (Attention! Hydrogen fluoride). The residue was washed with pentane and re-crystallized from hot methanol to give **I** as colourless crystals. Yield is 0.6 g (77%). ¹H NMR (400 MHz, D₂O, 293 K): δ = 7.57–7.12 (m, 5H), 2.94 (dd, J = 14.2, 3.3 Hz, 1H), 2.84 (m, 1H), 2.71 (m, 1H). ¹³C NMR (101 MHz, D₂O, 293 K): δ = 141.68, 128.84, 128.51, 126.10, 35.55. ¹⁹F NMR (376 MHz, D₂O, 293 K): δ = -145.40 (s).

Refinement

The hydrogen atoms were placed in calculated positions with C—H = 0.95–1.00 Å and refined in the riding model with fixed isotropic displacement parameters [$U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$].

Computing details

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT* (Bruker, 2001); program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

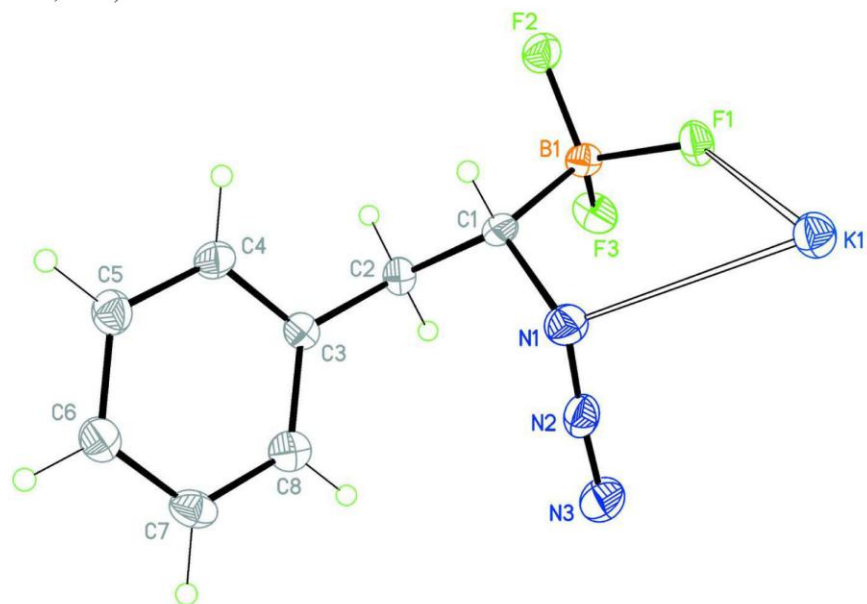


Figure 1

Independent part of the crystal structure of **I**. Displacement ellipsoids are shown at the 50% probability level.

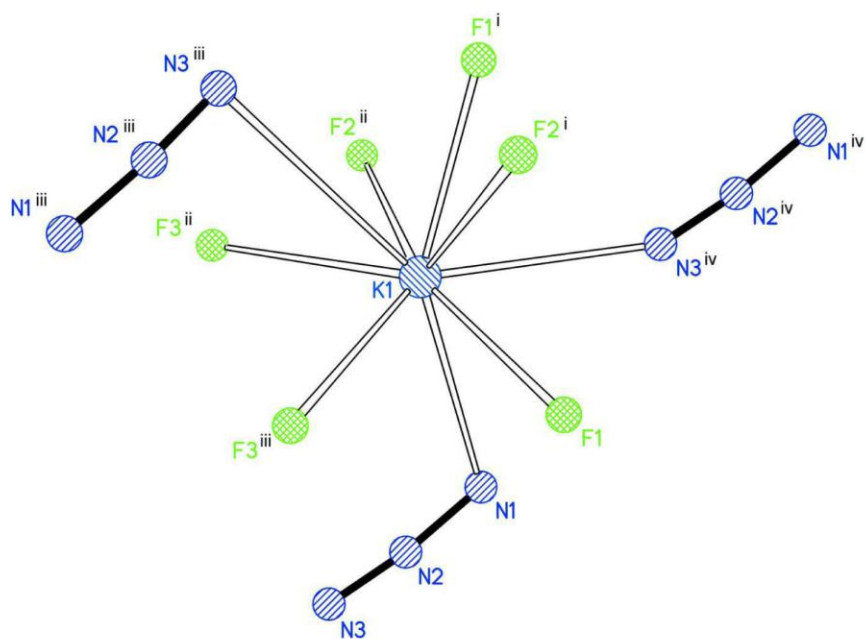


Figure 2

The coordination environment of potassium cation [symmetry codes: (i) $-x, y + 1/2, -z + 3/2$; (ii) $x, y + 1, z$; (iii) $-x + 1, y + 1/2, -z + 3/2$; (iv) $x - 1, y + 1, z$].

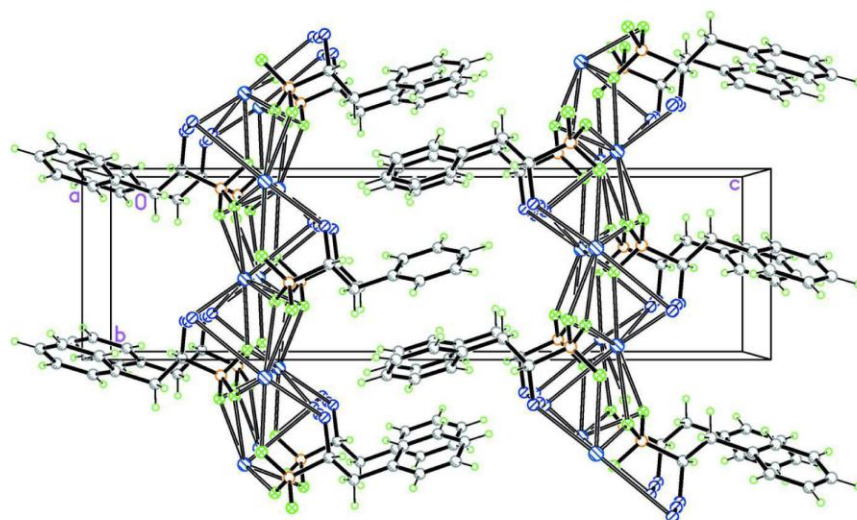
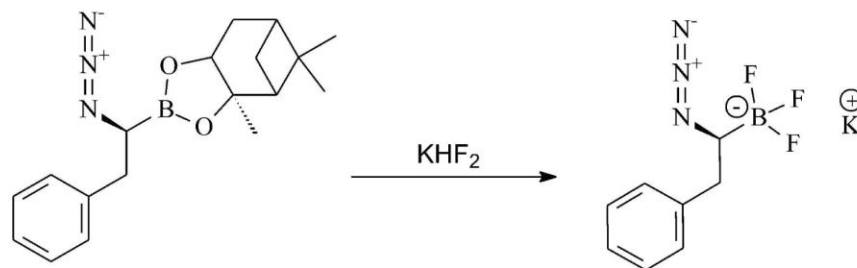


Figure 3

The layers in **I** parallel to (001).


Figure 4

Reaction of 2-[(1*S*)-1-azido-2-phenylethyl]-hexahydro-3*a*,5,5-trimethyl-4,6-methano-1,3,2-benzodioxaborole with potassium bifluoride.

Potassium [(1*S*)-1-azido-2-phenylethyl]trifluoridoborate
Crystal data
 $\text{K}^+ \cdot \text{C}_8\text{H}_8\text{BF}_3\text{N}_3^-$
 $M_r = 253.08$

 Orthorhombic, $P2_12_12_1$

Hall symbol: P 2ac 2ab

 $a = 6.052 (2) \text{ \AA}$
 $b = 6.959 (2) \text{ \AA}$
 $c = 25.120 (8) \text{ \AA}$
 $V = 1057.9 (6) \text{ \AA}^3$
 $Z = 4$
 $F(000) = 512$
 $D_x = 1.589 \text{ Mg m}^{-3}$

 Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 546 reflections

 $\theta = 3.2\text{--}18.5^\circ$
 $\mu = 0.52 \text{ mm}^{-1}$
 $T = 100 \text{ K}$

Plate, colourless

 $0.15 \times 0.10 \times 0.01 \text{ mm}$
Data collection

Bruker APEXII CCD

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

 φ and ω scans

Absorption correction: multi-scan

(SADABS; Sheldrick, 2003)

 $T_{\min} = 0.926$, $T_{\max} = 0.995$

10239 measured reflections

2075 independent reflections

 1203 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.060$
 $\theta_{\max} = 26.0^\circ$, $\theta_{\min} = 3.0^\circ$
 $h = -7 \rightarrow 7$
 $k = -8 \rightarrow 8$
 $l = -30 \rightarrow 30$
Refinement

 Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.050$
 $wR(F^2) = 0.081$
 $S = 0.98$

2075 reflections

145 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

 $w = 1/[\sigma^2(F_o^2) + (0.0191P)^2]$

 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.33 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.40 \text{ e \AA}^{-3}$

Absolute structure: Flack (1983), 828 Friedel

pairs

Flack parameter: 0.00 (8)

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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
K1	0.19352 (19)	0.92096 (16)	0.77175 (5)	0.0230 (3)
N1	0.4374 (7)	0.6992 (6)	0.85281 (14)	0.0243 (11)
N2	0.6381 (7)	0.7035 (6)	0.84172 (15)	0.0224 (11)
N3	0.8199 (7)	0.7267 (7)	0.83058 (14)	0.0296 (11)
C1	0.3397 (8)	0.5009 (6)	0.86075 (17)	0.0187 (12)
H1	0.1992	0.5194	0.8811	0.022*
C2	0.4850 (8)	0.3714 (6)	0.89471 (16)	0.0206 (13)
H2A	0.4156	0.2427	0.8965	0.025*
H2B	0.6294	0.3564	0.8766	0.025*
C3	0.5268 (8)	0.4407 (6)	0.95126 (18)	0.0167 (11)
C4	0.3648 (8)	0.4141 (7)	0.99013 (17)	0.0215 (12)
H4	0.2286	0.3546	0.9811	0.026*
C5	0.4030 (8)	0.4748 (7)	1.04210 (19)	0.0227 (13)
H5	0.2922	0.4575	1.0684	0.027*
C6	0.6016 (8)	0.5603 (7)	1.05561 (19)	0.0231 (12)
H6	0.6273	0.5998	1.0913	0.028*
C7	0.7647 (8)	0.5888 (7)	1.01697 (18)	0.0236 (12)
H7	0.9006	0.6489	1.0260	0.028*
C8	0.7250 (8)	0.5283 (6)	0.96550 (18)	0.0219 (13)
H8	0.8356	0.5469	0.9392	0.026*
F1	0.1942 (4)	0.5408 (3)	0.76697 (10)	0.0241 (7)
F2	0.0979 (4)	0.2693 (4)	0.81157 (9)	0.0250 (7)
F3	0.4491 (4)	0.3061 (4)	0.78057 (9)	0.0270 (7)
B1	0.2716 (9)	0.4042 (8)	0.8047 (2)	0.0171 (13)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
K1	0.0246 (6)	0.0215 (6)	0.0227 (6)	0.0008 (6)	-0.0020 (6)	0.0005 (6)
N1	0.021 (3)	0.028 (3)	0.024 (3)	-0.002 (2)	0.002 (2)	0.000 (2)
N2	0.031 (3)	0.017 (3)	0.019 (2)	-0.002 (2)	0.000 (2)	0.001 (2)
N3	0.025 (3)	0.036 (3)	0.029 (2)	-0.002 (3)	0.006 (2)	0.003 (2)
C1	0.017 (3)	0.017 (3)	0.022 (3)	-0.004 (2)	-0.001 (2)	0.004 (2)
C2	0.021 (3)	0.023 (3)	0.017 (3)	0.000 (2)	-0.001 (2)	0.003 (2)
C3	0.023 (3)	0.006 (3)	0.021 (3)	0.001 (2)	-0.005 (2)	0.002 (2)
C4	0.025 (3)	0.017 (3)	0.023 (3)	-0.006 (3)	-0.001 (2)	0.000 (3)
C5	0.025 (3)	0.025 (3)	0.019 (3)	0.001 (3)	0.001 (3)	-0.002 (2)

supplementary materials

C6	0.030 (3)	0.017 (3)	0.022 (3)	-0.001 (3)	-0.004 (3)	0.001 (3)
C7	0.020 (3)	0.019 (3)	0.031 (3)	0.001 (3)	-0.007 (2)	0.000 (3)
C8	0.021 (3)	0.018 (3)	0.027 (3)	0.003 (2)	0.000 (3)	0.000 (2)
F1	0.0326 (17)	0.0189 (15)	0.0209 (16)	-0.0006 (15)	-0.0068 (16)	0.0024 (13)
F2	0.0288 (16)	0.0240 (17)	0.0221 (15)	-0.0069 (15)	-0.0006 (12)	0.0005 (15)
F3	0.0234 (16)	0.0341 (18)	0.0234 (16)	0.0050 (14)	0.0006 (13)	-0.0092 (15)
B1	0.017 (3)	0.013 (3)	0.021 (3)	0.000 (3)	0.000 (2)	-0.001 (3)

Geometric parameters (Å, °)

K1—F1	2.648 (3)	C2—H2A	0.9900
K1—F3 ⁱ	2.654 (3)	C2—H2B	0.9900
K1—F1 ⁱⁱ	2.673 (3)	C3—C8	1.392 (6)
K1—F2 ⁱⁱⁱ	2.685 (3)	C3—C4	1.396 (6)
K1—F2 ⁱⁱ	2.933 (3)	C4—C5	1.391 (6)
K1—N1	2.951 (4)	C4—H4	0.9500
K1—N3 ^{iv}	3.020 (4)	C5—C6	1.383 (6)
K1—F3 ⁱⁱⁱ	3.102 (3)	C5—H5	0.9500
K1—N3 ⁱ	3.338 (4)	C6—C7	1.399 (6)
N1—N2	1.246 (5)	C6—H6	0.9500
N1—C1	1.515 (6)	C7—C8	1.381 (6)
N2—N3	1.147 (5)	C7—H7	0.9500
C1—C2	1.521 (6)	C8—H8	0.9500
C1—B1	1.614 (7)	F1—B1	1.422 (6)
C1—H1	1.0000	F2—B1	1.420 (6)
C2—C3	1.521 (6)	F3—B1	1.410 (6)
F1—K1—F3 ⁱ	71.05 (9)	N1—C1—B1	111.4 (4)
F1—K1—F1 ⁱⁱ	107.25 (7)	C2—C1—B1	112.9 (4)
F3 ⁱ —K1—F1 ⁱⁱ	128.98 (8)	N1—C1—H1	106.4
F1—K1—F2 ⁱⁱⁱ	156.77 (10)	C2—C1—H1	106.4
F3 ⁱ —K1—F2 ⁱⁱⁱ	129.20 (9)	B1—C1—H1	106.4
F1 ⁱⁱ —K1—F2 ⁱⁱⁱ	70.42 (8)	C3—C2—C1	115.6 (4)
F1—K1—F2 ⁱⁱ	66.99 (8)	C3—C2—H2A	108.4
F3 ⁱ —K1—F2 ⁱⁱ	91.61 (8)	C1—C2—H2A	108.4
F1 ⁱⁱ —K1—F2 ⁱⁱ	47.55 (7)	C3—C2—H2B	108.4
F2 ⁱⁱⁱ —K1—F2 ⁱⁱ	117.46 (7)	C1—C2—H2B	108.4
F1—K1—N1	60.53 (10)	H2A—C2—H2B	107.4
F3 ⁱ —K1—N1	77.08 (10)	C8—C3—C4	118.9 (4)
F1 ⁱⁱ —K1—N1	148.57 (10)	C8—C3—C2	121.4 (4)
F2 ⁱⁱⁱ —K1—N1	108.83 (10)	C4—C3—C2	119.7 (4)
F2 ⁱⁱ —K1—N1	127.22 (11)	C5—C4—C3	120.0 (4)
F1—K1—N3 ^{iv}	64.93 (11)	C5—C4—H4	120.0
F3 ⁱ —K1—N3 ^{iv}	135.84 (11)	C3—C4—H4	120.0
F1 ⁱⁱ —K1—N3 ^{iv}	70.16 (10)	C6—C5—C4	120.3 (5)
F2 ⁱⁱⁱ —K1—N3 ^{iv}	93.46 (11)	C6—C5—H5	119.8
F2 ⁱⁱ —K1—N3 ^{iv}	74.82 (10)	C4—C5—H5	119.8
N1—K1—N3 ^{iv}	78.62 (11)	C5—C6—C7	120.3 (4)
F1—K1—F3 ⁱⁱⁱ	149.93 (9)	C5—C6—H6	119.9
F3 ⁱ —K1—F3 ⁱⁱⁱ	83.65 (6)	C7—C6—H6	119.9

supplementary materials

F1 ⁱⁱ —K1—F3 ⁱⁱⁱ	101.20 (8)	C8—C7—C6	118.9 (4)
F2 ⁱⁱⁱ —K1—F3 ⁱⁱⁱ	45.66 (7)	C8—C7—H7	120.5
F2 ⁱⁱ —K1—F3 ⁱⁱⁱ	131.42 (8)	C6—C7—H7	120.5
N1—K1—F3 ⁱⁱⁱ	98.81 (10)	C7—C8—C3	121.6 (5)
N3 ^{iv} —K1—F3 ⁱⁱⁱ	136.46 (10)	C7—C8—H8	119.2
F1—K1—N3 ⁱ	127.01 (11)	C3—C8—H8	119.2
F3 ⁱ —K1—N3 ⁱ	80.25 (10)	B1—F1—K1	129.7 (3)
F1 ⁱⁱ —K1—N3 ⁱ	59.97 (9)	B1—F1—K1 ^{vii}	108.9 (3)
F2 ⁱⁱⁱ —K1—N3 ⁱ	72.92 (9)	K1—F1—K1 ^{vii}	109.06 (9)
F2 ⁱⁱ —K1—N3 ⁱ	70.44 (10)	B1—F2—K1 ^{viii}	113.1 (3)
N1—K1—N3 ⁱ	151.29 (11)	B1—F2—K1 ^{vii}	96.9 (3)
N3 ^{iv} —K1—N3 ⁱ	130.08 (7)	K1 ^{viii} —F2—K1 ^{vii}	100.87 (8)
F3 ⁱⁱⁱ —K1—N3 ⁱ	61.08 (9)	B1—F3—K1 ^{vi}	133.5 (3)
N2—N1—C1	115.6 (4)	B1—F3—K1 ^{viii}	94.0 (3)
N2—N1—K1	108.7 (3)	K1 ^{vi} —F3—K1 ^{viii}	129.14 (10)
C1—N1—K1	111.8 (3)	F3—B1—F2	107.2 (4)
N3—N2—N1	173.1 (5)	F3—B1—F1	106.7 (4)
N2—N3—K1 ^v	154.2 (4)	F2—B1—F1	106.2 (4)
N2—N3—K1 ^{vi}	94.3 (3)	F3—B1—C1	112.5 (4)
K1 ^v —N3—K1 ^{vi}	85.80 (9)	F2—B1—C1	111.0 (4)
N1—C1—C2	112.8 (4)	F1—B1—C1	112.7 (4)
F1—K1—N1—N2	101.9 (3)	N1—K1—F1—B1	9.8 (4)
F3 ⁱ —K1—N1—N2	26.6 (3)	N3 ^{iv} —K1—F1—B1	-81.1 (4)
F1 ⁱⁱ —K1—N1—N2	176.1 (3)	F3 ⁱⁱⁱ —K1—F1—B1	61.1 (4)
F2 ⁱⁱⁱ —K1—N1—N2	-100.8 (3)	N3 ⁱ —K1—F1—B1	156.4 (3)
F2 ⁱⁱ —K1—N1—N2	108.6 (3)	F3 ⁱ —K1—F1—K1 ^{vii}	-127.74 (10)
N3 ^{iv} —K1—N1—N2	169.4 (3)	F1 ⁱⁱ —K1—F1—K1 ^{vii}	-1.60 (5)
F3 ⁱⁱⁱ —K1—N1—N2	-54.8 (3)	F2 ⁱⁱⁱ —K1—F1—K1 ^{vii}	78.8 (3)
N3 ⁱ —K1—N1—N2	-12.2 (5)	F2 ⁱⁱ —K1—F1—K1 ^{vii}	-27.48 (8)
F1—K1—N1—C1	-26.9 (3)	N1—K1—F1—K1 ^{vii}	146.70 (13)
F3 ⁱ —K1—N1—C1	-102.3 (3)	N3 ^{iv} —K1—F1—K1 ^{vii}	55.89 (11)
F1 ⁱⁱ —K1—N1—C1	47.3 (4)	F3 ⁱⁱⁱ —K1—F1—K1 ^{vii}	-161.99 (14)
F2 ⁱⁱⁱ —K1—N1—C1	130.4 (3)	N3 ⁱ —K1—F1—K1 ^{vii}	-66.63 (15)
F2 ⁱⁱ —K1—N1—C1	-20.2 (3)	K1 ^{vi} —F3—B1—F2	161.9 (2)
N3 ^{iv} —K1—N1—C1	40.6 (3)	K1 ^{viii} —F3—B1—F2	2.3 (4)
F3 ⁱⁱⁱ —K1—N1—C1	176.4 (3)	K1 ^{vi} —F3—B1—F1	48.4 (5)
N3 ⁱ —K1—N1—C1	-141.0 (3)	K1 ^{viii} —F3—B1—F1	-111.2 (3)
N2—N1—C1—C2	44.7 (5)	K1 ^{vi} —F3—B1—C1	-75.8 (5)
K1—N1—C1—C2	169.7 (3)	K1 ^{viii} —F3—B1—C1	124.6 (4)
N2—N1—C1—B1	-83.6 (5)	K1 ^{viii} —F2—B1—F3	-2.8 (4)
K1—N1—C1—B1	41.5 (4)	K1 ^{vii} —F2—B1—F3	-107.8 (3)
N1—C1—C2—C3	61.4 (5)	K1 ^{viii} —F2—B1—F1	111.0 (3)
B1—C1—C2—C3	-171.2 (4)	K1 ^{vii} —F2—B1—F1	6.1 (4)
C1—C2—C3—C8	-101.8 (5)	K1 ^{viii} —F2—B1—C1	-126.1 (3)
C1—C2—C3—C4	79.0 (5)	K1 ^{vii} —F2—B1—C1	128.9 (3)
C8—C3—C4—C5	0.0 (7)	K1—F1—B1—F3	-115.8 (3)
C2—C3—C4—C5	179.2 (4)	K1 ^{vii} —F1—B1—F3	107.2 (3)
C3—C4—C5—C6	-0.5 (7)	K1—F1—B1—F2	130.0 (3)

supplementary materials

C4—C5—C6—C7	0.9 (7)	K1 ^{vii} —F1—B1—F2	-7.0 (4)
C5—C6—C7—C8	-0.8 (7)	K1—F1—B1—C1	8.2 (6)
C6—C7—C8—C3	0.3 (7)	K1 ^{vii} —F1—B1—C1	-128.8 (3)
C4—C3—C8—C7	0.1 (7)	N1—C1—B1—F3	86.5 (5)
C2—C3—C8—C7	-179.0 (4)	C2—C1—B1—F3	-41.7 (6)
F3 ⁱ —K1—F1—B1	95.3 (4)	N1—C1—B1—F2	-153.3 (4)
F1 ⁱⁱ —K1—F1—B1	-138.5 (4)	C2—C1—B1—F2	78.5 (5)
F2 ⁱⁱⁱ —K1—F1—B1	-58.1 (5)	N1—C1—B1—F1	-34.3 (6)
F2 ⁱⁱ —K1—F1—B1	-164.4 (4)	C2—C1—B1—F1	-162.5 (4)

Symmetry codes: (i) $-x+1, y+1/2, -z+3/2$; (ii) $-x, y+1/2, -z+3/2$; (iii) $x, y+1, z$; (iv) $x-1, y, z$; (v) $x+1, y, z$; (vi) $-x+1, y-1/2, -z+3/2$; (vii) $-x, y-1/2, -z+3/2$; (viii) $x, y-1, z$.

Hydrogen-bond geometry ($\text{\AA}, ^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C4—H4 \cdots C3 ^{ix}	0.95	2.93	3.528 (6)	122
C4—H4 \cdots C4 ^{ix}	0.95	2.98	3.823 (6)	149
C4—H4 \cdots C5 ^{ix}	0.95	3.08	3.974 (7)	158
C4—H4 \cdots C6 ^{ix}	0.95	3.13	3.841 (7)	133
C4—H4 \cdots C7 ^{ix}	0.95	3.09	3.556 (7)	112
C4—H4 \cdots C8 ^{ix}	0.95	2.98	3.382 (6)	107

Symmetry code: (ix) $x-1/2, -y+1/2, -z+2$.

