# organic compounds

## 3-Aminocarbonylpyridinium difluoro-acetate at 123 K

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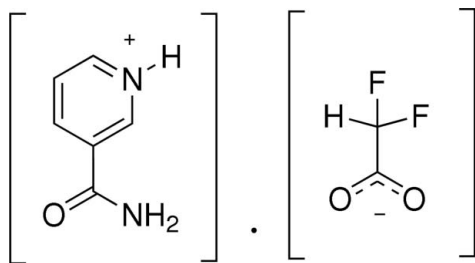Correspondence e-mail: j.bardin@strath.ac.uk

In the crystal of the title compound, $C_6H_7N_2O^+ \cdot C_2HF_2O_2^-$, the cation adopts a catemeric N—H···O hydrogen-bonded chain motif involving the carboxamide group, with two further N—H···O hydrogen bonds connecting the cations to adjacent difluoroacetate anions *via* the carboxamide and pyridinium N atoms. The carboxamide group of the nicotinamidium ion is twisted by 32.3 (6)° from the pyridine ring plane. A number of C—H···O and C—H···F interactions consolidate the packing.

## Related literature

For nicotinamide, see: Wright & King (1954); Miwa *et al.* (1999); Hino *et al.* (2001). For nicotinamide solvates, co-crystals and salts, see: Bardin *et al.* (2009); Koman *et al.* (2003); Athimoolam & Natarajan (2007a,b); Fleischman *et al.* (2003); Berry *et al.* (2008). Identification was initially made using multi-sample foil transmission X-ray powder diffraction analysis, see: Florence *et al.* (2003).



## Experimental

### Crystal data

$C_6H_7N_2O^+ \cdot C_2HF_2O_2^-$
$M_r = 218.16$
Monoclinic, $P2_1/c$
$a = 4.9888$ (2) Å
$b = 25.6147$ (12) Å
$c = 7.2006$ (4) Å
$\beta = 105.912$ (2)°
$V = 884.88$ (7) Å$^3$
$Z = 4$
Mo $K\alpha$ radiation
$\mu = 0.15$ mm$^{-1}$
$T = 123$ K
$0.30 \times 0.10 \times 0.02$ mm

### Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2002)
$T_{min} = 0.929$, $T_{max} = 0.997$
8921 measured reflections
2201 independent reflections
2087 reflections with $I > 2\sigma(I)$
$R_{int} = 0.015$

### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.034$
$wR(F^2) = 0.091$
$S = 1.05$
2201 reflections
152 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max} = 0.41$ e Å$^{-3}$
$\Delta\rho_{min} = -0.31$ e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1—H1$N$···O2$^i$ | 0.920 (12) | 2.589 (17) | 3.2275 (12) | 127.0 (13) |
| N1—H1$N$···O3$^i$ | 0.920 (12) | 1.675 (12) | 2.5921 (12) | 174.3 (17) |
| N2—H2$N$···O1$^{ii}$ | 0.895 (16) | 2.099 (15) | 2.9801 (12) | 167.9 (13) |
| N2—H3$N$···O3 | 0.863 (15) | 2.046 (15) | 2.8836 (11) | 163.2 (13) |
| C1—H1···O1$^i$ | 0.95 | 2.30 | 3.1763 (13) | 154 |
| C3—H3···F1$^{iii}$ | 0.95 | 2.53 | 3.1645 (13) | 124 |
| C4—H4···F2$^{iv}$ | 0.95 | 2.50 | 3.2885 (13) | 141 |
| C5—H5···O2$^{iv}$ | 0.95 | 2.40 | 3.2458 (13) | 149 |
| C8—H6···O2$^{ii}$ | 0.960 (14) | 2.405 (15) | 3.2403 (14) | 145.2 (11) |
| C8—H6···O2$^v$ | 0.960 (14) | 2.577 (13) | 3.3558 (13) | 138.3 (12) |

Symmetry codes: (i) $x-1, -y+\frac{3}{2}, z-\frac{1}{2}$; (ii) $x-1, y, z$; (iii) $x+1, -y+\frac{3}{2}, z+\frac{1}{2}$; (iv) $-x+1, y+\frac{1}{2}, -z+\frac{3}{2}$; (v) $-x+1, -y+1, -z+2$.

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009) and *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *PLATON*.

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# supporting information

## 3-Aminocarbonylpyridinium difluoroacetate at 123 K

**Julie Bardin, Alastair J. Florence, Jean-Baptiste Arlin, Alan R. Kennedy and Li Ven Wong**

### S1. Comment

Nicotinamide ($C_6H_6N_2O$, NA) is a member of the vitamin B group that includes pyridoxine, riboflavin and thiamine and its crystal structure was first determined in 1954 (Wright & King, 1954). Additional experimental investigations of charge density (Miwa *et al.*,1999) and polymorphism (Hino *et al.*, 2001) have also been reported although the crystal structures of other polymorphic forms have not been published. A number of multicomponent crystalline forms have been investigated including a trifluoroethanol solvate (Bardin *et al.*, 2009), 3,5-dinitrosalicylate (Koman *et al.*, 2003), 2*R*,3*R*-tartrate hydrate (Athimoolam & Natarajan, 2007*a*) and trifluoroacetate (Athimoolam & Natarajan, 2007*b*) salts. Co-crystals of NA have also been reported with drugs such as carbamazepine (Fleischman *et al.*, 2003), salicylic acid and both the racemic and single enantiomer (S(+)) forms of ibuprofen (Berry *et al.*, 2008).

  The difluoroacetate (DFA⁻) salt reported here (Scheme 1) was discovered during a study of multicomponent crystal formation involving fluorinated solvents and a range of organic compounds. The ions in the NA⁺ DFA⁻ salt crystallize in space group $P2_1/c$ with one NA⁺ cation and one DFA⁻ anion in the asymmetric unit (Fig.1). The internal C1—N1—C5 angle of the pyridinium ring in NA⁺ DFA⁻ is 122.13 (9)°, in close agreement to the value reported in the NA⁺ TFA⁻ salt (122.5 (4)°) but represents a significant increase over the the non-ionized form of NA (117.59 (7)°, Miwa *et al.*, 1999). In the DFA⁻ salt, as in the TFA⁻ salt and NA structures, the carboxamide group is not coplanar with the pyridinium ring with the angle between the planes of these groups being 32.30 (6)° [16.3 (8)° and 22.16 (4)° for NA⁺ TFA⁻ and NA respectively]. The packing in NA⁺ DFA⁻ consists of hydrogen bonded chains of NA⁺ ions extending parallel to the *a*-axis *via* contact N2—H2N⋯O1 between the anti-oriented H atom of the NH₂ group and the carbonyl O-atom, O1 (Fig. 2). Each cation forms an N—H⋯O hydrogen bond to O3 on a DFA⁻ ion and this atom also accepts a second N—H⋯O hydrogen bond from a neighbouring cation in a parallel chain (Figure 3), that connect adjacent chains resulting in a two-dimensional sheet lying parallel to the *ac* plane. The remaining O-atom on the DFA⁻ ion, O2, whilst not involved in any hydrogen bonds is involved in three C—H⋯O contacts to two DFA⁻ ions and a cation. One further C—H⋯O contact is observed between O1 and C1—H1 on the pyridinium ring in a parallel chain of cations. C—H⋯F contacts are also observed (C3—H3⋯F1 and C4—H4⋯F2, see Table 2).

### S2. Experimental

The novel structure reported here was discovered during a study of multicomponent crystal formation in organic compounds using a range of fluorinated acids. Identification of the novel phase was initially made using multi-sample foil transmission X-ray powder diffraction analysis (Florence *et al.*, 2003) and a suitable single-crystal for structure determination was obtained by isothermal evaporation at 278 K of a saturated solution of nicotinamide in DFAA.

**S3. Refinement**

Aromatic H atoms bound to C were placed in idealized positions and in a riding mode, with C—H distance set to 0.95Å and $U_{iso}$ equal to 1.2 times $U_{eq}$ of the parent atom. All other H atoms were located by difference synthesis and then refined isotropically.
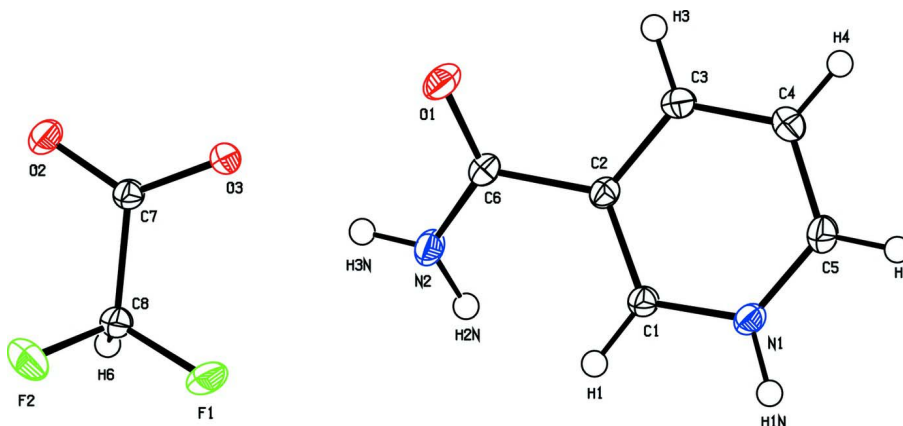
**Figure 1**

View of the title compound with the atom numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 50% probability level.
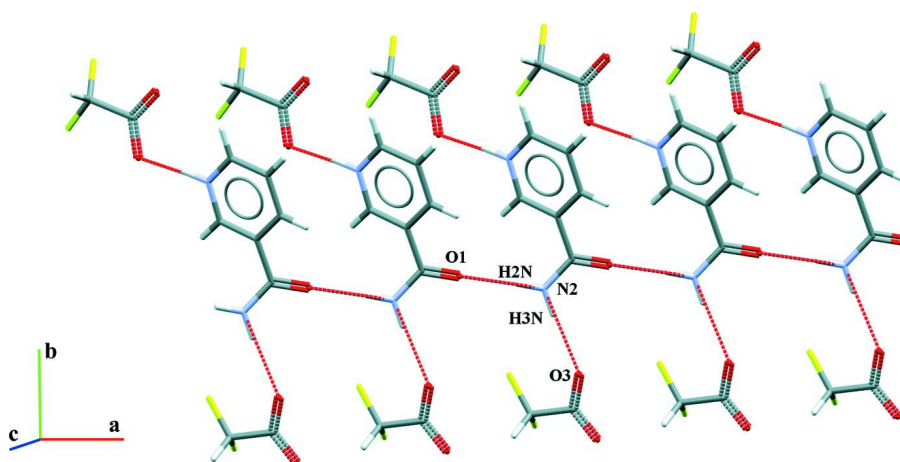
**Figure 2**

The NA⁺ cations form chain motifs *via* N2—H2N···O1 interactions between carboxamide groups with two further N—H···O contacts formed to the DFA⁻ anions.
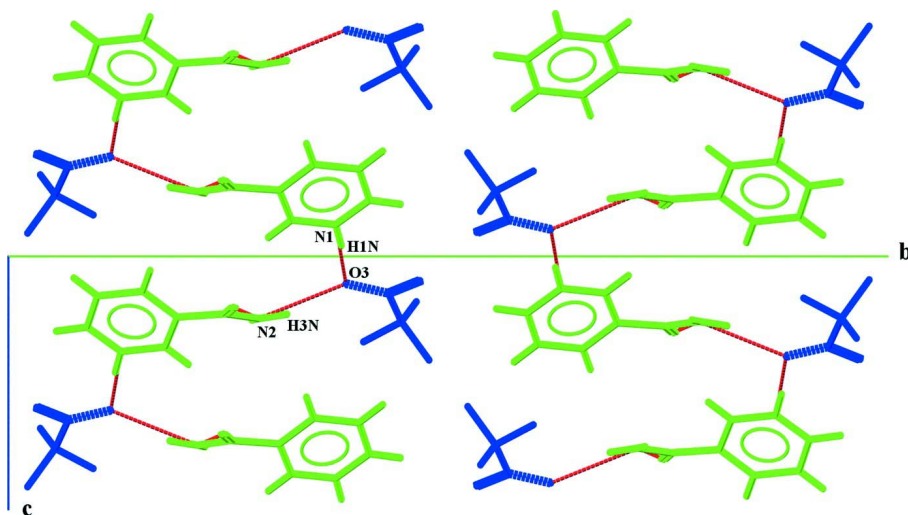
**Figure 3**

Chains of NA⁺ cations propagate down the *a*-axis with atom O3 connecting adjacent catemeric chains of cations (shown end on) *via* two N—H⋯O hydrogen bonds to N2—H3N on the carboxamide and N1—H1N on the pyridinium ring.

**3-Aminocarbonylpyridinium difluoroacetate**

*Crystal data*

$C_6H_7N_2O^+ \cdot C_2HF_2O_2^-$
$M_r = 218.16$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 4.9888$ (2) Å
$b = 25.6147$ (12) Å
$c = 7.2006$ (4) Å
$\beta = 105.912$ (2)°
$V = 884.88$ (7) Å³
$Z = 4$

$F(000) = 448$
$D_x = 1.638$ Mg m⁻³
Mo *Kα* radiation, $\lambda = 0.71073$ Å
Cell parameters from 6311 reflections
$\theta = 2.9$–28.4°
$\mu = 0.15$ mm⁻¹
$T = 123$ K
Slab, colourless
$0.30 \times 0.10 \times 0.02$ 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, 2002)
$T_{min} = 0.929$, $T_{max} = 0.997$

8921 measured reflections
2201 independent reflections
2087 reflections with $I > 2\sigma(I)$
$R_{int} = 0.015$
$\theta_{max} = 28.4°$, $\theta_{min} = 1.6°$
$h = -6 \rightarrow 6$
$k = -32 \rightarrow 34$
$l = -8 \rightarrow 9$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.034$
$wR(F^2) = 0.091$
$S = 1.05$
2201 reflections
152 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 atoms treated by a mixture of independent
　and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0508P)^2 + 0.323P]$
  where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$

$\Delta\rho_{max} = 0.41$ e Å$^{-3}$
$\Delta\rho_{min} = -0.31$ e Å$^{-3}$

*Special details*

**Geometry**. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

**Refinement**. Refinement on $F^2$ for ALL reflections except those flagged by the user for potential systematic errors. Weighted *R*-factors *wR* and all goodnesses 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 observed criterion of $F^2 > \sigma(F^2)$ is used only for calculating -*R*-factor-obs *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* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O1 | 0.80371 (15) | 0.74427 (3) | 0.79966 (12) | 0.0214 (2) |
| N1 | 0.11038 (17) | 0.87200 (3) | 0.62567 (12) | 0.0159 (2) |
| N2 | 0.36157 (18) | 0.71520 (3) | 0.75584 (14) | 0.0186 (3) |
| C1 | 0.19020 (19) | 0.82177 (4) | 0.64341 (14) | 0.0146 (2) |
| C2 | 0.44978 (19) | 0.80807 (4) | 0.76136 (14) | 0.0139 (2) |
| C3 | 0.6222 (2) | 0.84727 (4) | 0.86318 (15) | 0.0176 (3) |
| C4 | 0.5320 (2) | 0.89865 (4) | 0.84480 (15) | 0.0199 (3) |
| C5 | 0.2730 (2) | 0.91037 (4) | 0.72202 (15) | 0.0187 (3) |
| C6 | 0.5522 (2) | 0.75269 (4) | 0.77430 (14) | 0.0149 (2) |
| F1 | 0.12172 (14) | 0.59643 (3) | 0.66362 (11) | 0.0300 (2) |
| F2 | 0.27896 (15) | 0.52181 (3) | 0.59164 (10) | 0.0292 (2) |
| O2 | 0.73632 (16) | 0.53207 (3) | 0.90515 (13) | 0.0238 (3) |
| O3 | 0.61539 (15) | 0.61630 (3) | 0.89351 (11) | 0.0201 (2) |
| C7 | 0.56993 (19) | 0.56809 (4) | 0.85520 (14) | 0.0147 (2) |
| C8 | 0.2715 (2) | 0.55331 (4) | 0.74290 (15) | 0.0177 (3) |
| H1 | 0.06810 | 0.79550 | 0.57480 | 0.0180* |
| H1N | −0.063 (2) | 0.8784 (7) | 0.543 (2) | 0.045 (5)* |
| H2N | 0.185 (3) | 0.7234 (6) | 0.750 (2) | 0.026 (4)* |
| H3 | 0.80110 | 0.83880 | 0.94500 | 0.0210* |
| H3N | 0.422 (3) | 0.6835 (6) | 0.773 (2) | 0.027 (4)* |
| H4 | 0.64680 | 0.92550 | 0.91570 | 0.0240* |
| H5 | 0.21060 | 0.94550 | 0.70600 | 0.0220* |
| H6 | 0.171 (3) | 0.5363 (5) | 0.822 (2) | 0.017 (3)* |

*Atomic displacement parameters (Å$^2$)*

|  | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0136 (4) | 0.0203 (4) | 0.0305 (4) | 0.0040 (3) | 0.0065 (3) | 0.0053 (3) |
| N1 | 0.0135 (4) | 0.0148 (4) | 0.0183 (4) | 0.0022 (3) | 0.0027 (3) | 0.0026 (3) |
| N2 | 0.0156 (4) | 0.0124 (4) | 0.0281 (5) | 0.0020 (3) | 0.0066 (3) | 0.0021 (3) |
| C1 | 0.0130 (4) | 0.0137 (4) | 0.0168 (4) | −0.0002 (3) | 0.0034 (3) | 0.0009 (3) |
| C2 | 0.0132 (4) | 0.0132 (4) | 0.0154 (4) | 0.0012 (3) | 0.0042 (3) | 0.0020 (3) |

| C3 | 0.0140 (4) | 0.0181 (5) | 0.0186 (4) | 0.0002 (3) | 0.0011 (3) | 0.0005 (3) |
|---|---|---|---|---|---|---|
| C4 | 0.0196 (5) | 0.0156 (5) | 0.0223 (5) | −0.0022 (4) | 0.0023 (4) | −0.0023 (4) |
| C5 | 0.0202 (5) | 0.0139 (4) | 0.0218 (5) | 0.0017 (3) | 0.0054 (4) | 0.0009 (3) |
| C6 | 0.0144 (4) | 0.0152 (4) | 0.0149 (4) | 0.0025 (3) | 0.0039 (3) | 0.0021 (3) |
| F1 | 0.0183 (3) | 0.0226 (3) | 0.0402 (4) | 0.0048 (2) | −0.0072 (3) | 0.0045 (3) |
| F2 | 0.0318 (4) | 0.0254 (3) | 0.0267 (4) | −0.0055 (3) | 0.0020 (3) | −0.0094 (3) |
| O2 | 0.0151 (4) | 0.0154 (4) | 0.0377 (5) | 0.0025 (3) | 0.0019 (3) | 0.0020 (3) |
| O3 | 0.0158 (3) | 0.0128 (3) | 0.0276 (4) | −0.0002 (3) | −0.0007 (3) | −0.0019 (3) |
| C7 | 0.0122 (4) | 0.0140 (4) | 0.0175 (4) | −0.0006 (3) | 0.0036 (3) | 0.0005 (3) |
| C8 | 0.0146 (4) | 0.0146 (4) | 0.0216 (5) | −0.0006 (3) | 0.0011 (4) | 0.0003 (4) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| F1—C8 | 1.3676 (13) | C1—C2 | 1.3851 (14) |
| F2—C8 | 1.3642 (13) | C2—C6 | 1.5020 (14) |
| O1—C6 | 1.2364 (13) | C2—C3 | 1.3928 (14) |
| O2—C7 | 1.2272 (13) | C3—C4 | 1.3854 (15) |
| O3—C7 | 1.2721 (13) | C4—C5 | 1.3838 (15) |
| N1—C1 | 1.3425 (13) | C1—H1 | 0.9500 |
| N1—C5 | 1.3405 (13) | C3—H3 | 0.9500 |
| N2—C6 | 1.3323 (13) | C4—H4 | 0.9500 |
| N1—H1N | 0.920 (12) | C5—H5 | 0.9500 |
| N2—H2N | 0.895 (16) | C7—C8 | 1.5342 (14) |
| N2—H3N | 0.863 (15) | C8—H6 | 0.960 (14) |
| | | | |
| F1···C3ⁱ | 3.1645 (13) | C1···O1ˣⁱ | 3.1802 (13) |
| F1···O3 | 2.6133 (11) | C1···N2ⁱⁱⁱ | 3.2748 (14) |
| F1···C4ⁱ | 3.1997 (13) | C1···O1ⁱ | 3.1763 (13) |
| F2···C4ⁱⁱ | 3.2885 (13) | C1···O3ⁱ | 3.3389 (13) |
| F2···C4ⁱⁱⁱ | 3.1834 (13) | C3···N1ᵛⁱⁱ | 3.3972 (14) |
| F2···F2ⁱᵛ | 2.9535 (11) | C3···F1ᵛⁱ | 3.1645 (13) |
| F2···F2ᵛ | 3.0741 (11) | C4···F2ˣ | 3.1834 (13) |
| F2···O2 | 2.7472 (11) | C4···O3ⁱⁱⁱ | 3.4089 (13) |
| F2···C5ⁱⁱⁱ | 3.1716 (13) | C4···F2ˣⁱⁱ | 3.2885 (13) |
| F1···H4ⁱ | 2.6100 | C4···F1ᵛⁱ | 3.1997 (13) |
| F1···H3ⁱ | 2.5300 | C5···O2ⁱ | 3.3464 (14) |
| F1···H3N | 2.682 (15) | C5···C8ⁱⁱⁱ | 3.5712 (15) |
| F2···H4ⁱⁱⁱ | 2.8400 | C5···F2ˣ | 3.1716 (13) |
| F2···H5ⁱⁱⁱ | 2.8300 | C5···O3ⁱⁱⁱ | 3.3459 (13) |
| F2···H4ⁱⁱ | 2.5000 | C5···C7ⁱⁱⁱ | 3.4089 (14) |
| O1···C1ᵛⁱ | 3.1763 (13) | C5···O2ˣⁱⁱ | 3.2458 (13) |
| O1···C1ᵛⁱⁱ | 3.1802 (13) | C7···C5ˣ | 3.4089 (14) |
| O1···N2ᵛⁱⁱ | 2.9801 (12) | C7···N1ᵛⁱ | 3.2463 (13) |
| O2···C8ᵛⁱⁱ | 3.2403 (14) | C8···C5ˣ | 3.5712 (15) |
| O2···F2 | 2.7472 (11) | C8···O2ⁱˣ | 3.3558 (13) |
| O2···O2ᵛⁱⁱⁱ | 3.0848 (12) | C8···O2ˣⁱ | 3.2403 (14) |
| O2···C8ⁱˣ | 3.3558 (13) | C1···H2Nⁱⁱⁱ | 3.054 (14) |
| O2···C5ⁱⁱ | 3.2458 (13) | C1···H2N | 2.636 (15) |

| | | | |
|---|---|---|---|
| O2···N1$^{vi}$ | 3.2275 (12) | C7···H3N | 3.065 (15) |
| O2···C5$^{vi}$ | 3.3464 (14) | C7···H1N$^{vi}$ | 2.388 (14) |
| O3···F1 | 2.6133 (11) | H1···O1$^{i}$ | 2.3000 |
| O3···C5$^{x}$ | 3.3459 (13) | H1···H2N | 2.2200 |
| O3···N2 | 2.8836 (11) | H1···N2 | 2.6500 |
| O3···N1$^{vi}$ | 2.5921 (12) | H1···O1$^{xi}$ | 2.6900 |
| O3···C1$^{vi}$ | 3.3389 (13) | H1N···O3$^{i}$ | 1.675 (12) |
| O3···C4$^{x}$ | 3.4089 (13) | H1N···C7$^{i}$ | 2.388 (14) |
| O1···H2N$^{vii}$ | 2.099 (15) | H1N···O2$^{i}$ | 2.589 (17) |
| O1···H3 | 2.6400 | H2N···C1$^{x}$ | 3.054 (14) |
| O1···H1$^{vi}$ | 2.3000 | H2N···O1$^{xi}$ | 2.099 (15) |
| O1···H1$^{vii}$ | 2.6900 | H2N···C1 | 2.636 (15) |
| O2···H6$^{ix}$ | 2.577 (13) | H2N···H1 | 2.2200 |
| O2···H1N$^{vi}$ | 2.589 (17) | H3···O1 | 2.6400 |
| O2···H6$^{vii}$ | 2.405 (15) | H3···F1$^{vi}$ | 2.5300 |
| O2···H5$^{vi}$ | 2.7900 | H3N···F1 | 2.682 (15) |
| O2···H5$^{ii}$ | 2.4000 | H3N···O3 | 2.046 (15) |
| O3···H3N | 2.046 (15) | H3N···C7 | 3.065 (15) |
| O3···H1N$^{vi}$ | 1.675 (12) | H4···F2$^{x}$ | 2.8400 |
| N1···C3$^{xi}$ | 3.3972 (14) | H4···F2$^{xii}$ | 2.5000 |
| N1···C7$^{i}$ | 3.2463 (13) | H4···F1$^{vi}$ | 2.6100 |
| N1···O2$^{i}$ | 3.2275 (12) | H5···F2$^{x}$ | 2.8300 |
| N1···O3$^{i}$ | 2.5921 (12) | H5···O2$^{i}$ | 2.7900 |
| N2···O3 | 2.8836 (11) | H5···O2$^{xii}$ | 2.4000 |
| N2···O1$^{xi}$ | 2.9801 (12) | H6···O2$^{xi}$ | 2.405 (15) |
| N2···C1$^{x}$ | 3.2748 (14) | H6···O2$^{ix}$ | 2.577 (13) |
| N2···H1 | 2.6500 | | |
| | | | |
| C1—N1—C5 | 122.13 (9) | C2—C1—H1 | 120.00 |
| C1—N1—H1N | 115.7 (11) | C2—C3—H3 | 120.00 |
| C5—N1—H1N | 122.2 (11) | C4—C3—H3 | 120.00 |
| C6—N2—H2N | 120.1 (10) | C3—C4—H4 | 120.00 |
| H2N—N2—H3N | 121.9 (14) | C5—C4—H4 | 120.00 |
| C6—N2—H3N | 116.9 (10) | N1—C5—H5 | 120.00 |
| N1—C1—C2 | 120.28 (9) | C4—C5—H5 | 120.00 |
| C1—C2—C6 | 121.49 (9) | O2—C7—O3 | 126.89 (10) |
| C1—C2—C3 | 118.61 (9) | O2—C7—C8 | 116.72 (9) |
| C3—C2—C6 | 119.85 (9) | O3—C7—C8 | 116.32 (9) |
| C2—C3—C4 | 119.80 (10) | F1—C8—F2 | 106.01 (8) |
| C3—C4—C5 | 119.28 (10) | F1—C8—C7 | 111.16 (8) |
| N1—C5—C4 | 119.88 (9) | F2—C8—C7 | 109.38 (8) |
| O1—C6—C2 | 119.22 (9) | F1—C8—H6 | 107.4 (9) |
| N2—C6—C2 | 116.95 (9) | F2—C8—H6 | 109.8 (8) |
| O1—C6—N2 | 123.83 (10) | C7—C8—H6 | 112.9 (9) |
| N1—C1—H1 | 120.00 | | |
| | | | |
| C5—N1—C1—C2 | −1.18 (15) | C3—C2—C6—O1 | −30.80 (14) |
| C1—N1—C5—C4 | −0.15 (15) | C3—C2—C6—N2 | 149.70 (10) |

| | | | |
|---|---|---|---|
| N1—C1—C2—C3 | 1.24 (15) | C2—C3—C4—C5 | −1.27 (15) |
| N1—C1—C2—C6 | −176.03 (9) | C3—C4—C5—N1 | 1.38 (15) |
| C1—C2—C3—C4 | −0.02 (15) | O2—C7—C8—F1 | −168.25 (9) |
| C6—C2—C3—C4 | 177.30 (9) | O2—C7—C8—F2 | −51.53 (12) |
| C1—C2—C6—O1 | 146.44 (10) | O3—C7—C8—F1 | 14.57 (12) |
| C1—C2—C6—N2 | −33.06 (14) | O3—C7—C8—F2 | 131.29 (9) |

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

*Hydrogen-bond geometry (Å, º)*

| $D$—H⋯$A$ | $D$—H | H⋯$A$ | $D$⋯$A$ | $D$—H⋯$A$ |
|---|---|---|---|---|
| N1—H1$N$⋯O2[i] | 0.920 (12) | 2.589 (17) | 3.2275 (12) | 127.0 (13) |
| N1—H1$N$⋯O3[i] | 0.920 (12) | 1.675 (12) | 2.5921 (12) | 174.3 (17) |
| N2—H2$N$⋯O1[xi] | 0.895 (16) | 2.099 (15) | 2.9801 (12) | 167.9 (13) |
| N2—H3$N$⋯O3 | 0.863 (15) | 2.046 (15) | 2.8836 (11) | 163.2 (13) |
| C1—H1⋯O1[i] | 0.95 | 2.30 | 3.1763 (13) | 154 |
| C3—H3⋯F1[vi] | 0.95 | 2.53 | 3.1645 (13) | 124 |
| C4—H4⋯F2[xii] | 0.95 | 2.50 | 3.2885 (13) | 141 |
| C5—H5⋯O2[xii] | 0.95 | 2.40 | 3.2458 (13) | 149 |
| C8—H6⋯O2[xi] | 0.960 (14) | 2.405 (15) | 3.2403 (14) | 145.2 (11) |
| C8—H6⋯O2[ix] | 0.960 (14) | 2.577 (13) | 3.3558 (13) | 138.3 (12) |

Symmetry codes: (i) $x-1$, $-y+3/2$, $z-1/2$; (vi) $x+1$, $-y+3/2$, $z+1/2$; (ix) $-x+1$, $-y+1$, $-z+2$; (xi) $x-1$, $y$, $z$; (xii) $-x+1$, $y+1/2$, $-z+3/2$.