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2-Fluoroanilinium *N*-(2-fluorophenyl)-oxamate

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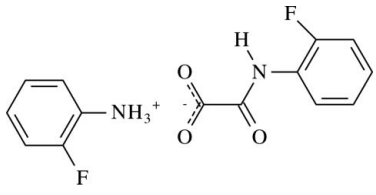
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.005 Å; R factor = 0.053; wR factor = 0.168; data-to-parameter ratio = 13.2.

The crystal structure of the title salt, $C_6H_7FN^+\cdot C_8H_5FNO_3^-$, exhibits intramolecular N—H···O and C—H···O and intermolecular N—H···O and N—H···F hydrogen-bond interactions, the intramolecular hydrogen-bond interactions generating S(6) and S(5) ring motifs. The dihedral angles between the aromatic ring and the intramolecular hydrogen-bonded rings in the anion are 2.97 (7) and 6.70 (5)°. The two aromatic rings of the title compound are oriented with a dihedral angle of 77.25 (9)°.

Related literature

For related structures see: Odabaşoğlu & Büyükgüngör (2006*a*,*b*,*c*); Büyükgüngör & Odabaşoğlu (2007). For ring motif details, see: Bernstein *et al.* (1995); Etter (1990).



Experimental

Crystal data

$C_6H_7FN^+\cdot C_8H_5FNO_3^-$
M_r = 294.26
Triclinic, $P\overline{1}$
a = 6.7118 (9) Å
b = 9.5998 (14) Å
c = 11.7000 (16) Å
α = 68.346 (11)°
β = 85.791 (11)°
γ = 77.375 (11)°
V = 683.69 (18) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.12 mm^{-1}
T = 296 K
0.78 × 0.47 × 0.09 mm

Data collection

Stoe IPDSII diffractometer
Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)
T_{min} = 0.931, T_{max} = 0.988
9218 measured reflections
2684 independent reflections
2023 reflections with $I > 2\sigma(I)$
R_{int} = 0.040

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.168
S = 0.81
2684 reflections
203 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.53 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O3	0.86	2.19	2.623 (3)	111
N1—H1···F1^{i}	0.86	2.61	3.313 (3)	140
N2—H2A···O2^{ii}	0.93 (3)	1.77 (3)	2.696 (3)	170 (3)
N2—H2B···O3^{iii}	0.90 (3)	1.86 (3)	2.753 (3)	174 (3)
N2—H2C···O2^{iv}	0.89 (3)	2.05 (3)	2.802 (3)	142 (3)
N2—H2C···O1^{iv}	0.89 (3)	2.30 (3)	3.041 (3)	141 (3)
C6—H6···O1	0.93	2.36	2.941 (3)	120

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

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2556).

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supporting information

Acta Cryst. (2008). E**64**, o808 [doi:10.1107/S1600536808008891]

2-Fluoroanilinium *N*-(2-fluorophenyl)oxamate

Orhan Büyükgüngör and Mustafa Odabaşoğlu

S1. Comment

The present work is part of a structural study of compounds of anillinium carboxylates and we report here the structure of 2-fluoroanilinium 2-(2-fluorophenylamino)-2-oxoacetate, (I).

The title compound (I) is built up from a 2-fluoroanilinium cation and 2-(2-fluorophenylamino)-2-oxoacetate anion (Fig. 1). There are two intramolecular hydrogen bonds which generate S(6)and S(5) motifs (Bernstein *et al.*, 1995; Etter, 1990), and the anions and cations of (I) are linked to each other by five intermolecular hydrogen bond interactions (Table 1.). The intermolecular hydrogen interactions, except N—H···F, generate $R_1^2(5)R_4^2(8)R_4^4(12)$ motifs (Fig. 2) and these motifs link N—H···F hydrogen bonded $R_2^2(10)$ rings (Fig. 3) forming a three dimensional network. The dihedral angles between the aromatic ring and intramolecular hydrogen bonded rings in the anion are 2.97 (7)° and 6.70 (5)°. The two aromatic rings of the title compound are oriented with a dihedral angle of 77.25 (9)°.

S2. Experimental

The title compound was prepared according to the method described by Büyükgüngör & Odabaşoğlu (2006*b*), using 2-fluoroaniline and oxalic acid as starting materials (yield 90%). Crystals of (I) suitable for *x*-ray analysis were obtained by slow evaporation of an ethanol (95%) solution at room temperature.

S3. Refinement

H atom (for NH_3) was located in difference synthesis and refined freely. The remaining H atoms were positioned geometrically, with C—H = 0.93 for aromatic H and N—H = 0.86 Å for amine H, and constrained to ride on their parent atoms with $U_{iso}(H) = 1.2U_{eq}$(parent).

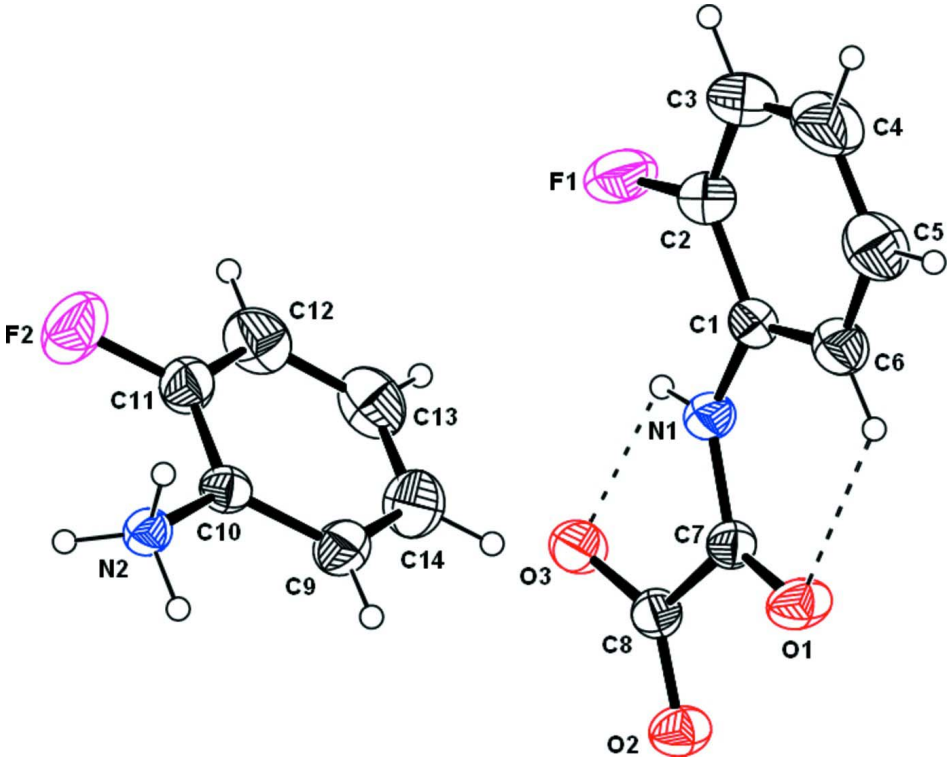

Figure 1

The molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. The hydrogen bonds are drawn as dashed lines.

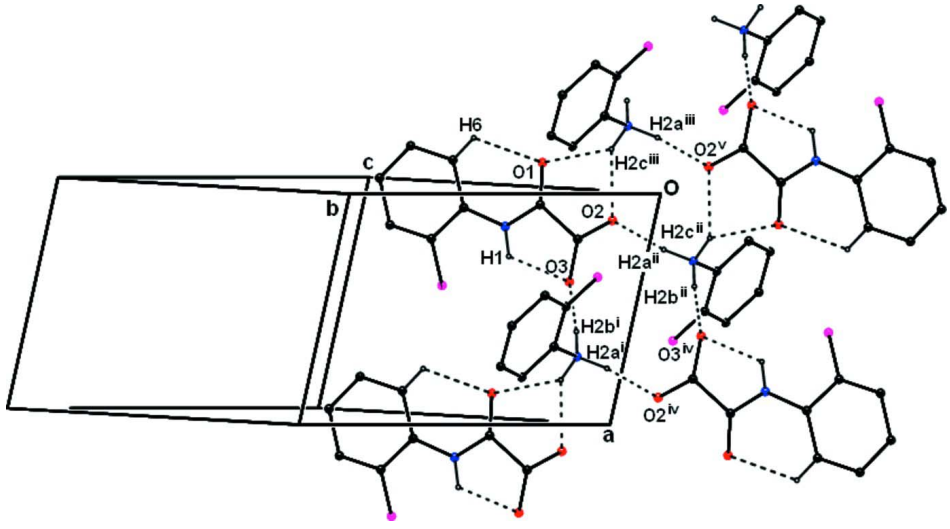

Figure 2

A partial packing diagram of (I), showing the formation of $R_1^2(5)R_4^2(8)R_4^4(12)$ motifs. H atoms not involved in hydrogen bonds have been omitted for clarity. [Symmetry codes: (i) 1 - *x*, 1 - *y*, *z*; (ii) *x*, *y* - 1, *z*; (iii) -*x*, *y*, -*z*; (iv) 1 - *x*, -*y*, -*z*; (v) -*x*, -*y*, -*z*].

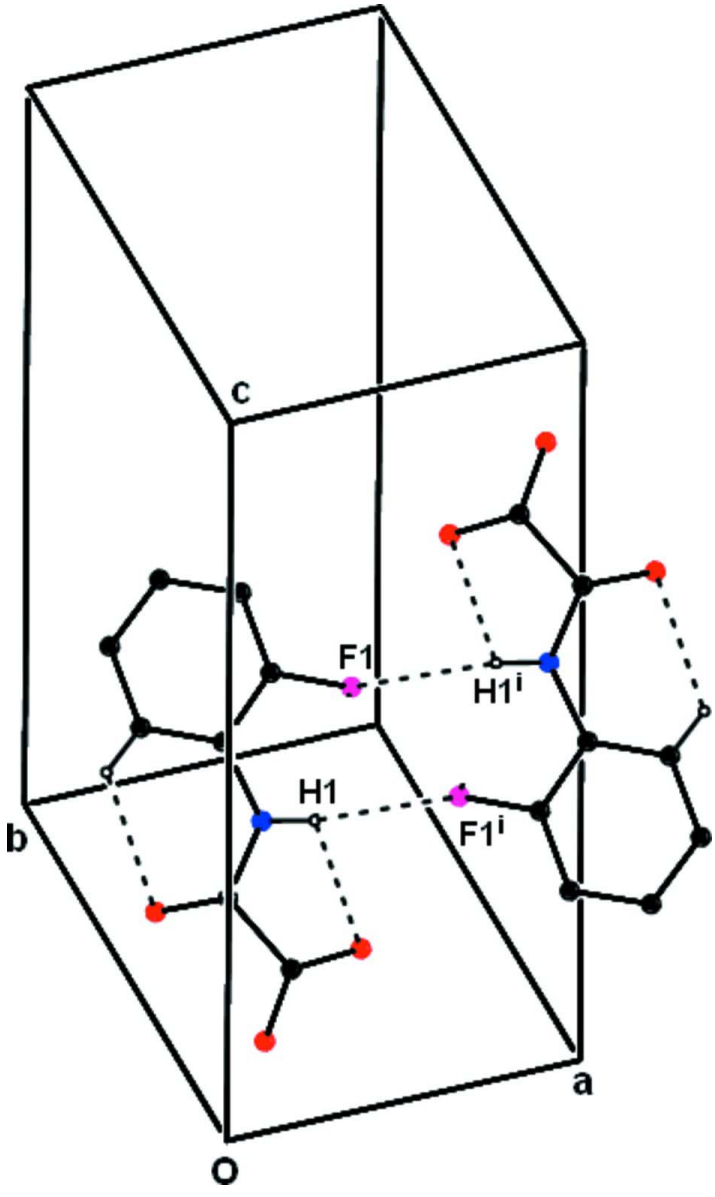

Figure 3

A partial packing diagram of (I), showing the formation of $R_2^2(10)$ motif. H atoms not involved in hydrogen bonds have been omitted for clarity. [Symmetry code: (i) 1 - *x*, -*y*, 1 - *z*].

2-Fluoroanilinium N-(2-fluorophenyl)oxamate

Crystal data

$C_6H_7FN^+·C_8H_5FNO_3^-$
M_r = 294.26
Triclinic, $P\overline{1}$
Hall symbol: -P 1
a = 6.7118 (9) Å
b = 9.5998 (14) Å
c = 11.7000 (16) Å
α = 68.346 (11)°
β = 85.791 (11)°
γ = 77.375 (11)°
V = 683.69 (18) Å^3
Z = 2
F(000) = 304
D_x = 1.429 Mg m^{-3}
Mo *Kα* radiation, *λ* = 0.71073 Å
Cell parameters from 9218 reflections
θ = 1.9–28.1°
μ = 0.12 mm^{-1}

T = 296 K
Plate, colourless

0.78 × 0.47 × 0.09 mm

Data collection

Stoe IPDSII diffractometer
Radiation source: sealed X-ray tube, 12 x 0.4 mm long-fine focus
Plane graphite monochromator
Detector resolution: 6.67 pixels mm^{-1}
ω rotation method scans
Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)

T_{min} = 0.932, T_{max} = 0.988
9218 measured reflections
2684 independent reflections
2023 reflections with $I > 2\sigma(I)$
R_{int} = 0.040
θ_{max} = 26.0°, θ_{min} = 1.9°
h = −8→8
k = −11→11
l = −14→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.168
S = 0.81
2684 reflections
203 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 atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.1051P)^2 + 0.5375P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.53 e Å^{-3}
$\Delta\rho_{min}$ = −0.30 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.026 (7)

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}
C1	0.0927 (4)	0.1895 (3)	0.4566 (2)	0.0500 (6)
C2	0.2329 (5)	0.2000 (4)	0.5327 (3)	0.0662 (7)
C3	0.1793 (7)	0.2559 (5)	0.6239 (3)	0.0882 (10)
H3	0.2783	0.2599	0.6735	0.106*
C4	−0.0226 (7)	0.3060 (5)	0.6414 (3)	0.0958 (12)
H4	−0.0625	0.3456	0.7030	0.115*
C5	−0.1675 (6)	0.2980 (4)	0.5682 (3)	0.0826 (9)
H5	−0.3050	0.3322	0.5809	0.099*
C6	−0.1122 (4)	0.2399 (3)	0.4759 (2)	0.0619 (7)
H6	−0.2116	0.2347	0.4272	0.074*
C7	0.0672 (3)	0.1176 (3)	0.2757 (2)	0.0452 (5)
C8	0.2108 (3)	0.0541 (3)	0.1899 (2)	0.0472 (5)

C9	0.5179 (4)	0.6758 (3)	0.1045 (2)	0.0592 (7)
C10	0.3396 (3)	0.7306 (3)	0.0369 (2)	0.0447 (5)
C11	0.2088 (5)	0.6328 (3)	0.0524 (3)	0.0625 (7)
H11	0.0855	0.6678	0.0095	0.075*
C12	0.2612 (6)	0.4827 (4)	0.1318 (3)	0.0804 (9)
H12	0.1723	0.4171	0.1420	0.097*
C13	0.4415 (6)	0.4291 (4)	0.1955 (3)	0.0803 (9)
H13	0.4763	0.3270	0.2472	0.096*
C14	0.5722 (5)	0.5263 (4)	0.1834 (3)	0.0787 (9)
H14	0.6942	0.4915	0.2277	0.094*
N1	0.1682 (3)	0.1288 (2)	0.36637 (17)	0.0496 (5)
H1	0.2978	0.0935	0.3692	0.060*
N2	0.2905 (3)	0.8889 (2)	−0.04487 (19)	0.0447 (5)
O1	−0.1162 (3)	0.1563 (2)	0.25841 (17)	0.0638 (5)
O2	0.1240 (3)	0.0321 (2)	0.10956 (16)	0.0587 (5)
O3	0.3972 (3)	0.0349 (2)	0.20670 (16)	0.0651 (5)
F1	0.4339 (3)	0.1515 (3)	0.51255 (18)	0.0919 (7)
F2	0.6443 (3)	0.7710 (2)	0.0900 (2)	0.1002 (7)
H2A	0.243 (5)	0.946 (3)	0.004 (3)	0.069 (8)*
H2B	0.396 (5)	0.915 (3)	−0.094 (3)	0.064 (8)*
H2C	0.187 (5)	0.898 (3)	−0.092 (3)	0.073 (9)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0559 (14)	0.0517 (13)	0.0407 (12)	−0.0165 (11)	0.0003 (10)	−0.0118 (10)
C2	0.0678 (18)	0.0816 (19)	0.0546 (15)	−0.0240 (15)	−0.0014 (13)	−0.0257 (14)
C3	0.107 (3)	0.115 (3)	0.0628 (19)	−0.040 (2)	0.0018 (18)	−0.0461 (19)
C4	0.134 (4)	0.105 (3)	0.068 (2)	−0.035 (2)	0.020 (2)	−0.051 (2)
C5	0.086 (2)	0.090 (2)	0.075 (2)	−0.0135 (18)	0.0180 (17)	−0.0395 (18)
C6	0.0621 (16)	0.0686 (17)	0.0554 (15)	−0.0137 (13)	0.0054 (12)	−0.0239 (13)
C7	0.0433 (13)	0.0461 (12)	0.0438 (12)	−0.0093 (10)	−0.0035 (9)	−0.0128 (10)
C8	0.0418 (12)	0.0501 (13)	0.0448 (12)	−0.0068 (10)	−0.0029 (9)	−0.0124 (10)
C9	0.0528 (15)	0.0617 (16)	0.0590 (15)	−0.0124 (12)	−0.0016 (12)	−0.0166 (12)
C10	0.0465 (12)	0.0475 (12)	0.0420 (11)	−0.0057 (10)	0.0015 (9)	−0.0211 (10)
C11	0.0719 (18)	0.0591 (16)	0.0633 (16)	−0.0226 (13)	−0.0022 (13)	−0.0243 (13)
C12	0.102 (3)	0.0582 (18)	0.083 (2)	−0.0320 (17)	0.0009 (19)	−0.0194 (16)
C13	0.102 (3)	0.0503 (16)	0.0728 (19)	−0.0055 (16)	0.0036 (18)	−0.0103 (14)
C14	0.0662 (19)	0.078 (2)	0.0695 (19)	0.0046 (16)	−0.0092 (15)	−0.0104 (16)
N1	0.0409 (10)	0.0623 (12)	0.0463 (11)	−0.0105 (9)	−0.0033 (8)	−0.0198 (9)
N2	0.0391 (11)	0.0510 (11)	0.0458 (11)	−0.0096 (8)	−0.0009 (9)	−0.0192 (9)
O1	0.0404 (10)	0.0909 (14)	0.0671 (11)	−0.0012 (9)	−0.0080 (8)	−0.0420 (10)
O2	0.0498 (10)	0.0734 (12)	0.0605 (10)	−0.0046 (8)	−0.0034 (8)	−0.0369 (9)
O3	0.0402 (10)	0.0987 (15)	0.0522 (10)	−0.0088 (9)	−0.0004 (7)	−0.0254 (10)
F1	0.0653 (11)	0.1443 (18)	0.0815 (12)	−0.0250 (11)	−0.0134 (9)	−0.0540 (12)
F2	0.0742 (12)	0.0985 (15)	0.1081 (15)	−0.0317 (10)	−0.0328 (11)	−0.0010 (12)

Geometric parameters (Å, º)

C1—C6	1.386 (4)	C9—F2	1.337 (3)
C1—C2	1.385 (4)	C9—C14	1.374 (4)
C1—N1	1.399 (3)	C9—C10	1.375 (3)
C2—C3	1.356 (5)	C10—C11	1.377 (3)
C2—F1	1.361 (4)	C10—N2	1.445 (3)
C3—C4	1.363 (5)	C11—C12	1.378 (4)
C3—H3	0.9300	C11—H11	0.9300
C4—C5	1.375 (5)	C12—C13	1.365 (5)
C4—H4	0.9300	C12—H12	0.9300
C5—C6	1.381 (4)	C13—C14	1.379 (5)
C5—H5	0.9300	C13—H13	0.9300
C6—H6	0.9300	C14—H14	0.9300
C7—O1	1.214 (3)	N1—H1	0.8600
C7—N1	1.349 (3)	N2—H2A	0.93 (3)
C7—C8	1.536 (3)	N2—H2B	0.90 (3)
C8—O3	1.244 (3)	N2—H2C	0.89 (3)
C8—O2	1.244 (3)		
C6—C1—C2	117.3 (2)	C14—C9—C10	122.3 (3)
C6—C1—N1	125.0 (2)	C9—C10—C11	118.4 (2)
C2—C1—N1	117.7 (2)	C9—C10—N2	119.9 (2)
C3—C2—F1	119.5 (3)	C11—C10—N2	121.7 (2)
C3—C2—C1	123.4 (3)	C12—C11—C10	119.8 (3)
F1—C2—C1	117.0 (2)	C12—C11—H11	120.1
C2—C3—C4	118.7 (3)	C10—C11—H11	120.1
C2—C3—H3	120.7	C13—C12—C11	120.9 (3)
C4—C3—H3	120.7	C13—C12—H12	119.5
C3—C4—C5	120.0 (3)	C11—C12—H12	119.5
C3—C4—H4	120.0	C12—C13—C14	120.1 (3)
C5—C4—H4	120.0	C12—C13—H13	120.0
C6—C5—C4	121.1 (3)	C14—C13—H13	120.0
C6—C5—H5	119.5	C9—C14—C13	118.5 (3)
C4—C5—H5	119.5	C9—C14—H14	120.8
C5—C6—C1	119.5 (3)	C13—C14—H14	120.8
C5—C6—H6	120.3	C7—N1—C1	129.4 (2)
C1—C6—H6	120.3	C7—N1—H1	115.3
O1—C7—N1	125.5 (2)	C1—N1—H1	115.3
O1—C7—C8	121.8 (2)	C10—N2—H2A	106.5 (18)
N1—C7—C8	112.65 (19)	C10—N2—H2B	111.4 (18)
O3—C8—O2	128.1 (2)	H2A—N2—H2B	116 (3)
O3—C8—C7	116.8 (2)	C10—N2—H2C	106.7 (19)
O2—C8—C7	115.1 (2)	H2A—N2—H2C	108 (3)
F2—C9—C14	119.3 (3)	H2B—N2—H2C	108 (3)
F2—C9—C10	118.4 (2)		
C6—C1—C2—C3	0.2 (4)	F2—C9—C10—C11	−179.6 (2)

N1—C1—C2—C3	−179.6 (3)	C14—C9—C10—C11	2.2 (4)
C6—C1—C2—F1	−179.5 (2)	F2—C9—C10—N2	−1.7 (4)
N1—C1—C2—F1	0.7 (4)	C14—C9—C10—N2	−179.9 (3)
F1—C2—C3—C4	179.0 (3)	C9—C10—C11—C12	−1.8 (4)
C1—C2—C3—C4	−0.7 (5)	N2—C10—C11—C12	−179.7 (3)
C2—C3—C4—C5	0.6 (6)	C10—C11—C12—C13	0.0 (5)
C3—C4—C5—C6	−0.2 (6)	C11—C12—C13—C14	1.6 (5)
C4—C5—C6—C1	−0.3 (5)	F2—C9—C14—C13	−178.8 (3)
C2—C1—C6—C5	0.2 (4)	C10—C9—C14—C13	−0.6 (5)
N1—C1—C6—C5	−179.9 (3)	C12—C13—C14—C9	−1.3 (5)
O1—C7—C8—O3	171.7 (2)	O1—C7—N1—C1	−2.4 (4)
N1—C7—C8—O3	−6.3 (3)	C8—C7—N1—C1	175.6 (2)
O1—C7—C8—O2	−6.5 (3)	C6—C1—N1—C7	6.0 (4)
N1—C7—C8—O2	175.5 (2)	C2—C1—N1—C7	−174.2 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O3	0.86	2.19	2.623 (3)	111
N1—H1···F1^{i}	0.86	2.61	3.313 (3)	140
N2—H2*A*···O2^{ii}	0.93 (3)	1.77 (3)	2.696 (3)	170 (3)
N2—H2*B*···O3^{iii}	0.90 (3)	1.86 (3)	2.753 (3)	174 (3)
N2—H2*C*···O2^{iv}	0.89 (3)	2.05 (3)	2.802 (3)	142 (3)
N2—H2*C*···O1^{iv}	0.89 (3)	2.30 (3)	3.041 (3)	141 (3)
C6—H6···O1	0.93	2.36	2.941 (3)	120

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