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2-Fluoroanilinium *N*-(2-fluorophenyl)-oxamateOrhan Büyükgüngör^a and Mustafa Odabaşoğlu^{b*}

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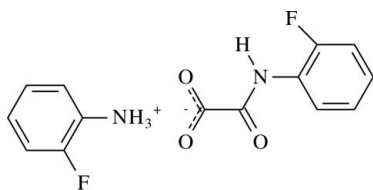
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Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å; R factor = 0.053; wR factor = 0.168; data-to-parameter ratio = 13.2.

The crystal structure of the title salt, $\text{C}_6\text{H}_7\text{FN}^+\cdot\text{C}_8\text{H}_5\text{FNO}_3^-$, exhibits intramolecular $\text{N}-\text{H}\cdots\text{O}$ and $\text{C}-\text{H}\cdots\text{O}$ and intermolecular $\text{N}-\text{H}\cdots\text{O}$ and $\text{N}-\text{H}\cdots\text{F}$ 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

 $\text{C}_6\text{H}_7\text{FN}^+\cdot\text{C}_8\text{H}_5\text{FNO}_3^-$ $M_r = 294.26$ Triclinic, $P\bar{1}$ $a = 6.7118$ (9) Å $b = 9.5998$ (14) Å $c = 11.7000$ (16) Å $\alpha = 68.346$ (11)° $\beta = 85.791$ (11)° $\gamma = 77.375$ (11)° $V = 683.69$ (18) Å³ $Z = 2$ Mo $K\alpha$ radiation $\mu = 0.12$ mm⁻¹ $T = 296$ K $0.78 \times 0.47 \times 0.09$ mm

Data collection

Stoe IPDSII diffractometer

Absorption correction: integration

 $(X\text{-RED32; Stoe \& Cie, 2002})$ $T_{\min} = 0.931, T_{\max} = 0.988$

9218 measured reflections

2684 independent reflections

2023 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.040$

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, °).

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

The authors acknowledge the Faculty of Arts and Sciences, Ondokuz Mayıs University, Turkey, for the use of the Stoe IPDSII diffractometer (purchased under grant F. 279 of the University Research Fund).

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

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supplementary materials

Acta Cryst. (2008). E64, o808 [doi:10.1107/S1600536808008891]

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

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Comment

The present work is part of a structural study of compounds of anilinium 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)°.

Experimental

The title compound was prepared according to the method described by Büyükgüngör & Odabaşođlu (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.

Refinement

H atom (for NH₃) was located in difference synthesis and refined freely. The remaining H atoms were positioned geometrically, with C—H = 0.93 Å for aromatic H and N—H = 0.86 Å for amine H, and constrained to ride on their parent atoms with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{parent})$.

Figures

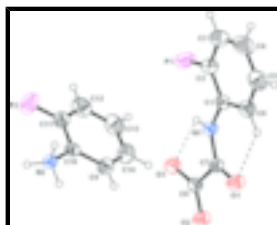


Fig. 1. The molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. The hydrogen bonds are drawn as dashed lines.

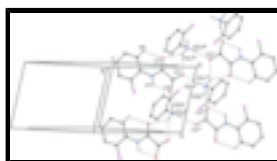


Fig. 2. A partial packing diagram of (I), showing the formation of $R_1^2(5)R_4^2(8)R_4^4(12)$ motifs. H atoms not involved in hydrogen bonds have been omitted for clarity. [Symmetry codes: (i) 1 - x, 1 - y, z; (ii) x, y - 1, z; (iii) -x, y, -z; (iv) 1 - x, -y, -z; (v) -x, -y, -z].

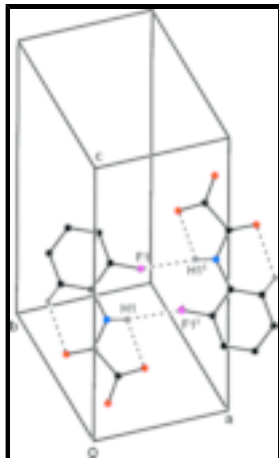
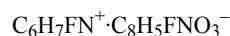


Fig. 3. A partial packing diagram of (I), showing the formation of $R_2^2(10)$ motif. H atoms not involved in hydrogen bonds have been omitted for clarity. [Symmetry code: (i) $1 - x, -y, 1 - z$].

2-Fluoroanilinium N-(2-fluorophenyl)oxamate

Crystal data



$M_r = 294.26$

Triclinic, $P\bar{1}$

Hall symbol: -P 1

$a = 6.7118$ (9) Å

$b = 9.5998$ (14) Å

$c = 11.7000$ (16) Å

$\alpha = 68.346$ (11)°

$\beta = 85.791$ (11)°

$\gamma = 77.375$ (11)°

$V = 683.69$ (18) Å³

$Z = 2$

$F_{000} = 304$

$D_x = 1.429$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 9218 reflections

$\theta = 1.9$ – 28.1 °

$\mu = 0.12$ mm⁻¹

$T = 296$ K

Plate, colourless

$0.78 \times 0.47 \times 0.09$ mm

Data collection

Stoe IPDSII
diffractometer

Monochromator: plane graphite

Detector resolution: 6.67 pixels mm⁻¹

$T = 296$ K

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 $I > 2\sigma(I)$

$R_{\text{int}} = 0.040$

$\theta_{\max} = 26.0$ °

$\theta_{\min} = 1.9$ °

$h = -8 \rightarrow 8$

$k = -11 \rightarrow 11$

$l = -14 \rightarrow 14$

Refinement

Refinement on F^2

Hydrogen site location: inferred from neighbouring sites

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

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

H atoms treated by a mixture of independent and constrained refinement

$$w = 1/[\sigma^2(F_o^2) + (0.1051P)^2 + 0.5375P]$$

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

$$(\Delta/\sigma)_{\max} < 0.001$$

$$\Delta\rho_{\max} = 0.53 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.30 \text{ e } \text{\AA}^{-3}$$

Extinction correction: SHELXL97 (Sheldrick, 2008),

$$F_c^* = kF_c[1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$$

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 (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
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*

supplementary materials

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 (\AA^2)

	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 (\AA , $^\circ$)

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)

supplementary materials

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, °)

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
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) $-x, -y+1, -z$.

Fig. 1

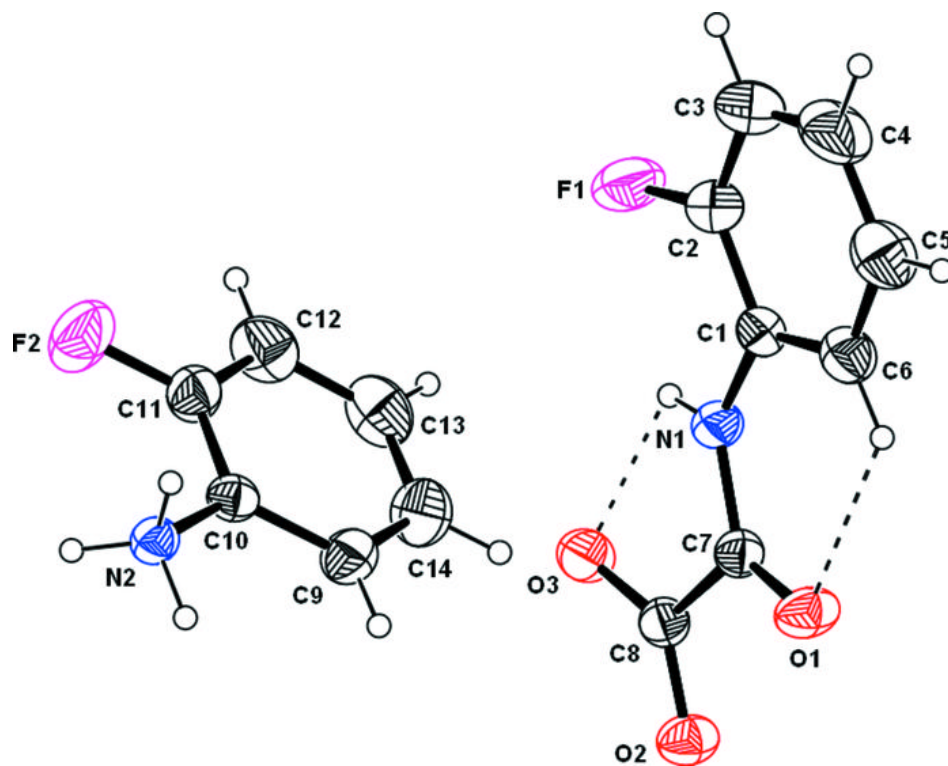


Fig. 2

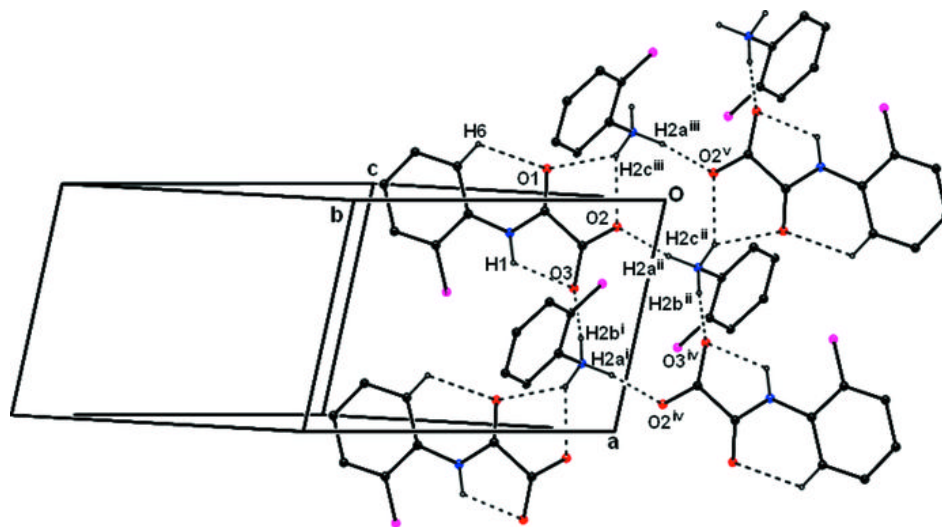


Fig. 3

