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3-(2-Fluoroanilino)isobenzofuran-1(3*H*)-one[1]

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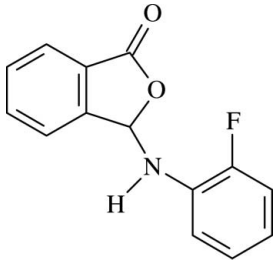
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.002 Å; R factor = 0.031; wR factor = 0.085; data-to-parameter ratio = 12.9.

The crystal structure of the title compound, $C_{14}H_{10}FNO_2$, is stabilized by N—H···O, two C—H···O and two C—H···F intermolecular hydrogen bonds. N—H···O, C—H···O and two C—H···F intermolecular hydrogen bonds generate a fused $R_2^2(10)R_2^2(12)R_2^2(15)R_3^2(13)$ ring motif. In the crystal structure, molecules are linked into chains along the b axis by C—H···O hydrogen bonds. The dihedral angle between the phthalide group and the other benzene ring is 73.51 (5)°.

Related literature

For related structures, see: Büyükgüngör & Odabaşoğlu (2006*a*,*b*); Odabaşoğlu & Büyükgüngör (2006*a*,*b*; 2007*a*,*b*). These compounds have fungicidal (Aoki *et al*., 1973; Lacova, 1973), bacterial and herbicidal (Lacova, 1973), analgesic (Elderfield, 1951), and hypotensive and vasorelaxant properties (Tsi & Tan, 1997).



Experimental

Crystal data

$C_{14}H_{10}FNO_2$
M_r = 243.23
Triclinic, $P\overline{1}$
a = 7.0942 (18) Å
b = 7.984 (2) Å
c = 10.092 (4) Å
α = 88.92 (3)°
β = 84.84 (3)°
γ = 74.93 (2)°
V = 549.7 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 296 K
0.62 × 0.52 × 0.38 mm

Data collection

Stoe IPDS-2 diffractometer
Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)
T_{min} = 0.945, T_{max} = 0.968
9227 measured reflections
2167 independent reflections
1822 reflections with $I > 2\sigma(I)$
R_{int} = 0.037

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.085
S = 1.06
2167 reflections
168 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.15 e Å^{-3}
$\Delta\rho_{min}$ = −0.12 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H15···O1^{i}	0.862 (15)	2.344 (15)	3.1627 (17)	158.7 (13)
C5—H5···O2^{ii}	0.93	2.59	3.3903 (18)	144
C8—H8···O1^{iii}	0.98	2.61	3.4022 (17)	138
C3—H3···F1^{i}	0.93	2.73	3.617 (2)	161
C13—H13···F1^{iv}	0.93	2.60	3.3275 (18)	135

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

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, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); 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: AT2436).

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[1] 3-Substituted phthalides, Part XXX.

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supplementary materials

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3-(2-Fluoroanilino)isobenzofuran-1(3*H*)-one

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Comment

As part of a continuing study of the interplay between molecular conformation and supramolecular aggregation in 3-substituted phthalides (Büyükgüngör & Odabaşoğlu, 2006*a*,b; Odabaşoğlu & Büyükgüngör, 2006*a*,b, 2007*a*,b), we now report the structure of 3-(4-butylphenylamino)isobenzofuran- 1(3*H*)-one, (I), (Fig. 1).

The crystal packing is stabilized by is stabilized by an N—H···O, two C—H···O and two C—H···F intermolecular hydrogen bonds (Table 1). In the structure, these hydrogen bond interactions generate an edge-fused $R_2^2(10)R_2^2(12)R_2^2(15)R_3^2(13)$ ring motif (Fig. 2). The hydrogen bonded motifs are link each other C5—H5···O2 chains and forming three-dimentional network (Fig. 3). The phthalide group (C1—C8/O2) is planar, the largest deviation from the mean plane being −0.026 (4) Å for atom C6. The dihedral angle between the mean planes of the phthalide group and the phenyl ring is 73.51 (5)°.

Experimental

Compound (I) was prepared as described by Odabaşoğlu & Büyükgüngör (2006*a*), using phthalaldehydic acid and 2-fluoroaniline as starting materials (yield 85%, m.p. 445–446 K). Crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of a DMF-EtOH (1/1: *v*/*v*) solution at room temperature.

Refinement

Except N-bound H, all C-bound H atoms were refined using the riding model approximation with d(C—H) = 0.93 for aromatic and d(C—H) = 0.98 for methine C—H [U_{iso}(H) = 1.2U_{eq}(parent atom)]. N-bound H atom was located in Fourier difference map and refined freely due to its taking part in H-bond.

Figures

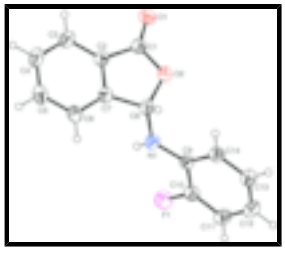

Fig. 1. A view of (I) with the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level..

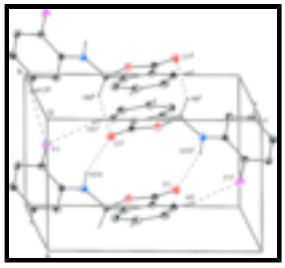

Fig. 2. Part of the crystal structure of (I), showing the formation of $R_2^2(10)R_2^2(12)R_2^2(15)R_3^2(13)$ motifs. H atoms not involved in hydrogen bonds have been omitted for clarity. [Symmetry codes: (i) $1-x$, $1-y$, $1-z$; (ii) $x-1$, y, z].

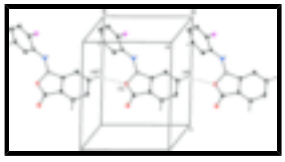

Fig. 3. Part of the crystal structure of (I), showing the formation of C6 chain along the *c* axis. H atoms not involved in hydrogen bonds have been omitted for clarity. [Symmetry code: (i) x, $y+1$, z].

3-(2-Fluoroanilino)isobenzofuran-1(3*H*)-one

Crystal data

$C_{14}H_{10}FNO_2$	$Z = 2$
$M_r = 243.23$	$F_{000} = 252$
Triclinic, $P\overline{1}$	$D_x = 1.469$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 7.0942$ (18) Å	Cell parameters from 19681 reflections
$b = 7.984$ (2) Å	$\theta = 2.0$–28.1º
$c = 10.092$ (4) Å	$\mu = 0.11$ mm^{-1}
$\alpha = 88.92$ (3)º	$T = 296$ K
$\beta = 84.84$ (3)º	Prism, colourless
$\gamma = 74.93$ (2)º	0.62 × 0.52 × 0.38 mm
$V = 549.7$ (3) Å^3	

Data collection

Stoe IPDS-2 diffractometer	2167 independent reflections
Monochromator: plane graphite	1822 reflections with $I > 2\sigma(I)$
Detector resolution: 6.67 pixels mm^{-1}	$R_{int} = 0.037$
$T = 296$ K	$\theta_{max} = 26.0$º
ω scans	$\theta_{min} = 2.0$º
Absorption correction: integration (X-RED32; Stoe & Cie, 2002)	$h = -8 \rightarrow 8$
$T_{min} = 0.945$, $T_{max} = 0.968$	$k = -9 \rightarrow 9$
9227 measured reflections	$l = -12 \rightarrow 12$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of

	independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.031$	$w = 1/[\sigma^2(F_o^2) + (0.0403P)^2 + 0.0693P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.085$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.06$	$\Delta\rho_{max} = 0.15$ e Å^{-3}
2167 reflections	$\Delta\rho_{min} = -0.12$ e Å^{-3}
168 parameters	Extinction correction: SHELXL97 (Sheldrick, 1997), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.157 (9)
Secondary atom site location: difference Fourier map	

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 $F^2 > 2$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	0.74814 (17)	0.46754 (16)	0.55003 (12)	0.0453 (3)
C2	0.74694 (16)	0.28901 (15)	0.51978 (12)	0.0439 (3)
C3	0.71836 (19)	0.15804 (18)	0.60483 (14)	0.0538 (3)
H3	0.6942	0.1762	0.6962	0.065*
C4	0.7270 (2)	0.00029 (18)	0.54906 (16)	0.0596 (4)
H4	0.7108	−0.0911	0.6035	0.072*
C5	0.75954 (19)	−0.02397 (17)	0.41274 (16)	0.0587 (4)
H5	0.7631	−0.1315	0.3773	0.070*
C6	0.78691 (18)	0.10685 (16)	0.32784 (14)	0.0515 (3)
H6	0.8079	0.0897	0.2362	0.062*
C7	0.78179 (16)	0.26420 (15)	0.38439 (12)	0.0431 (3)
C8	0.81902 (17)	0.42464 (15)	0.32039 (12)	0.0435 (3)
H8	0.9558	0.3995	0.2829	0.052*
C9	0.73725 (17)	0.61732 (15)	0.13090 (11)	0.0420 (3)
C10	0.59784 (17)	0.69605 (16)	0.04574 (13)	0.0477 (3)
C11	0.6292 (2)	0.80564 (19)	−0.05390 (14)	0.0581 (4)
H11	0.5326	0.8517	−0.1107	0.070*
C12	0.8064 (2)	0.84704 (18)	−0.06903 (14)	0.0602 (4)
H12	0.8311	0.9218	−0.1363	0.072*
C13	0.9464 (2)	0.77698 (18)	0.01608 (13)	0.0546 (3)
H13	1.0654	0.8064	0.0069	0.065*

C14	0.91375 (18)	0.66372 (17)	0.11494 (13)	0.0485 (3)
H14	1.0107	0.6179	0.1715	0.058*
N1	0.69639 (16)	0.49475 (14)	0.22118 (10)	0.0483 (3)
O1	0.71649 (14)	0.54281 (13)	0.65565 (9)	0.0590 (3)
O2	0.79064 (13)	0.54564 (11)	0.43571 (8)	0.0492 (2)
F1	0.42053 (11)	0.65855 (12)	0.06461 (9)	0.0654 (3)
H15	0.577 (2)	0.4864 (19)	0.2323 (14)	0.057 (4)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0409 (6)	0.0482 (7)	0.0475 (7)	−0.0117 (5)	−0.0068 (5)	0.0027 (5)
C2	0.0378 (6)	0.0431 (6)	0.0504 (7)	−0.0092 (5)	−0.0063 (5)	0.0053 (5)
C3	0.0503 (7)	0.0548 (8)	0.0557 (7)	−0.0133 (6)	−0.0055 (6)	0.0134 (6)
C4	0.0532 (8)	0.0473 (8)	0.0793 (10)	−0.0159 (6)	−0.0056 (6)	0.0186 (7)
C5	0.0518 (7)	0.0411 (7)	0.0840 (10)	−0.0136 (6)	−0.0053 (7)	−0.0005 (6)
C6	0.0479 (7)	0.0468 (7)	0.0595 (8)	−0.0120 (5)	−0.0029 (5)	−0.0037 (6)
C7	0.0369 (6)	0.0411 (6)	0.0508 (7)	−0.0091 (5)	−0.0047 (5)	0.0041 (5)
C8	0.0430 (6)	0.0425 (6)	0.0456 (6)	−0.0119 (5)	−0.0039 (5)	0.0015 (5)
C9	0.0453 (6)	0.0401 (6)	0.0399 (6)	−0.0096 (5)	−0.0029 (5)	−0.0018 (5)
C10	0.0427 (6)	0.0488 (7)	0.0506 (7)	−0.0094 (5)	−0.0062 (5)	−0.0018 (5)
C11	0.0595 (8)	0.0582 (8)	0.0531 (8)	−0.0064 (6)	−0.0143 (6)	0.0099 (6)
C12	0.0686 (9)	0.0559 (8)	0.0537 (8)	−0.0138 (7)	−0.0023 (6)	0.0140 (6)
C13	0.0546 (7)	0.0535 (7)	0.0571 (8)	−0.0185 (6)	−0.0010 (6)	0.0059 (6)
C14	0.0477 (7)	0.0501 (7)	0.0491 (7)	−0.0141 (5)	−0.0088 (5)	0.0050 (5)
N1	0.0431 (6)	0.0548 (6)	0.0501 (6)	−0.0174 (5)	−0.0076 (4)	0.0097 (5)
O1	0.0645 (6)	0.0625 (6)	0.0518 (6)	−0.0194 (5)	−0.0044 (4)	−0.0071 (4)
O2	0.0594 (5)	0.0413 (5)	0.0497 (5)	−0.0176 (4)	−0.0074 (4)	0.0029 (4)
F1	0.0466 (4)	0.0767 (6)	0.0755 (6)	−0.0177 (4)	−0.0155 (4)	0.0098 (4)

Geometric parameters (Å, °)

C1—O1	1.2077 (15)	C8—O2	1.4923 (15)
C1—O2	1.3487 (16)	C8—H8	0.9800
C1—C2	1.4656 (18)	C9—C10	1.3861 (17)
C2—C7	1.3752 (18)	C9—C14	1.3906 (17)
C2—C3	1.3844 (18)	C9—N1	1.3922 (16)
C3—C4	1.374 (2)	C10—C11	1.3617 (19)
C3—H3	0.9300	C10—F1	1.3633 (15)
C4—C5	1.383 (2)	C11—C12	1.376 (2)
C4—H4	0.9300	C11—H11	0.9300
C5—C6	1.3801 (19)	C12—C13	1.372 (2)
C5—H5	0.9300	C12—H12	0.9300
C6—C7	1.3801 (18)	C13—C14	1.3785 (18)
C6—H6	0.9300	C13—H13	0.9300
C7—C8	1.4972 (17)	C14—H14	0.9300
C8—N1	1.3982 (16)	N1—H15	0.862 (15)
O1—C1—O2	121.89 (12)	O2—C8—H8	109.1

O1—C1—C2	129.46 (12)	C7—C8—H8	109.1
O2—C1—C2	108.65 (11)	C10—C9—C14	116.10 (11)
C7—C2—C3	121.87 (12)	C10—C9—N1	119.13 (11)
C7—C2—C1	108.46 (11)	C14—C9—N1	124.71 (11)
C3—C2—C1	129.68 (12)	C11—C10—F1	119.38 (11)
C4—C3—C2	117.46 (13)	C11—C10—C9	123.86 (12)
C4—C3—H3	121.3	F1—C10—C9	116.76 (11)
C2—C3—H3	121.3	C10—C11—C12	118.79 (12)
C3—C4—C5	120.66 (13)	C10—C11—H11	120.6
C3—C4—H4	119.7	C12—C11—H11	120.6
C5—C4—H4	119.7	C13—C12—C11	119.35 (13)
C6—C5—C4	121.90 (13)	C13—C12—H12	120.3
C6—C5—H5	119.1	C11—C12—H12	120.3
C4—C5—H5	119.1	C12—C13—C14	121.16 (13)
C5—C6—C7	117.30 (13)	C12—C13—H13	119.4
C5—C6—H6	121.4	C14—C13—H13	119.4
C7—C6—H6	121.4	C13—C14—C9	120.65 (12)
C2—C7—C6	120.81 (12)	C13—C14—H14	119.7
C2—C7—C8	109.45 (11)	C9—C14—H14	119.7
C6—C7—C8	129.68 (12)	C9—N1—C8	122.50 (11)
N1—C8—O2	111.83 (10)	C9—N1—H15	117.7 (10)
N1—C8—C7	114.81 (10)	C8—N1—H15	117.0 (10)
O2—C8—C7	102.60 (9)	C1—O2—C8	110.74 (9)
N1—C8—H8	109.1		
O1—C1—C2—C7	176.95 (12)	C14—C9—C10—C11	3.59 (19)
O2—C1—C2—C7	−2.39 (13)	N1—C9—C10—C11	−173.92 (12)
O1—C1—C2—C3	−3.4 (2)	C14—C9—C10—F1	−176.78 (11)
O2—C1—C2—C3	177.27 (12)	N1—C9—C10—F1	5.71 (17)
C7—C2—C3—C4	0.32 (18)	F1—C10—C11—C12	177.82 (12)
C1—C2—C3—C4	−179.31 (12)	C9—C10—C11—C12	−2.6 (2)
C2—C3—C4—C5	−1.2 (2)	C10—C11—C12—C13	0.1 (2)
C3—C4—C5—C6	0.8 (2)	C11—C12—C13—C14	1.1 (2)
C4—C5—C6—C7	0.4 (2)	C12—C13—C14—C9	0.0 (2)
C3—C2—C7—C6	0.89 (18)	C10—C9—C14—C13	−2.25 (18)
C1—C2—C7—C6	−179.41 (10)	N1—C9—C14—C13	175.10 (12)
C3—C2—C7—C8	−176.47 (11)	C10—C9—N1—C8	−172.37 (11)
C1—C2—C7—C8	3.23 (13)	C14—C9—N1—C8	10.35 (18)
C5—C6—C7—C2	−1.22 (18)	O2—C8—N1—C9	78.83 (14)
C5—C6—C7—C8	175.54 (12)	C7—C8—N1—C9	−164.80 (10)
C2—C7—C8—N1	−124.33 (12)	O1—C1—O2—C8	−178.85 (11)
C6—C7—C8—N1	58.62 (17)	C2—C1—O2—C8	0.55 (12)
C2—C7—C8—O2	−2.79 (12)	N1—C8—O2—C1	124.86 (11)
C6—C7—C8—O2	−179.84 (12)	C7—C8—O2—C1	1.30 (12)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H15···O1^{i}	0.862 (15)	2.344 (15)	3.1627 (17)	158.7 (13)

C5—H5···O2^{ii}	0.93	2.59	3.3903 (18)	144
C8—H8···O1^{iii}	0.98	2.61	3.4022 (17)	138
C3—H3···F1^{i}	0.93	2.73	3.617 (2)	161
C13—H13···F1^{iv}	0.93	2.60	3.3275 (18)	135

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

Fig. 1

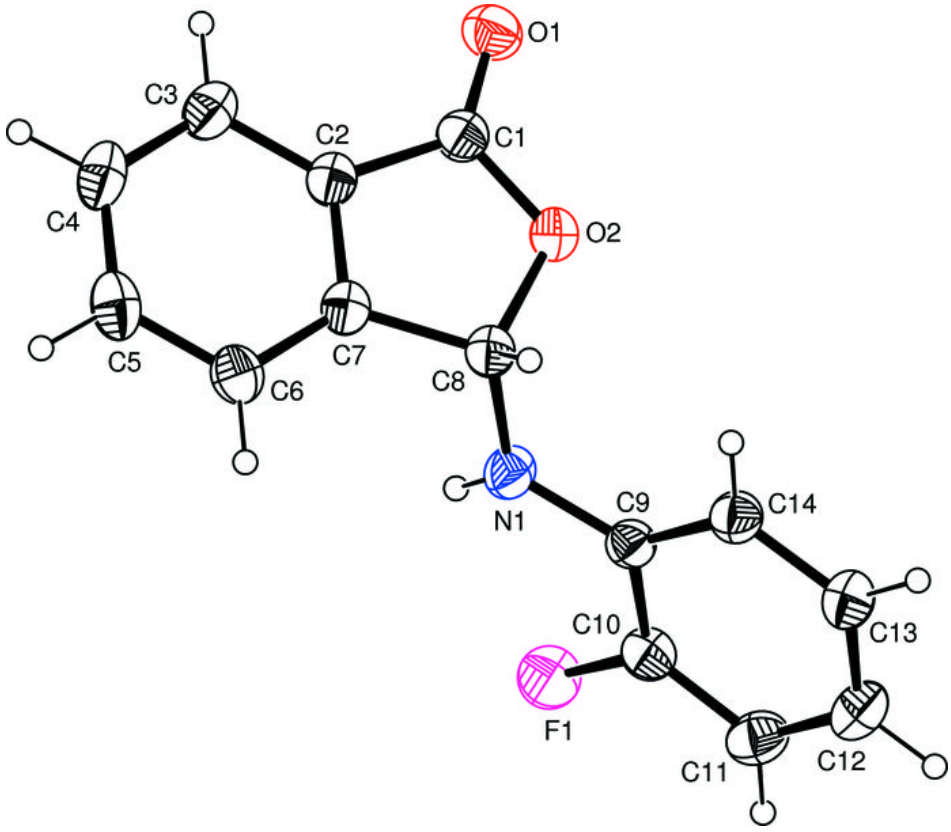

Fig. 2

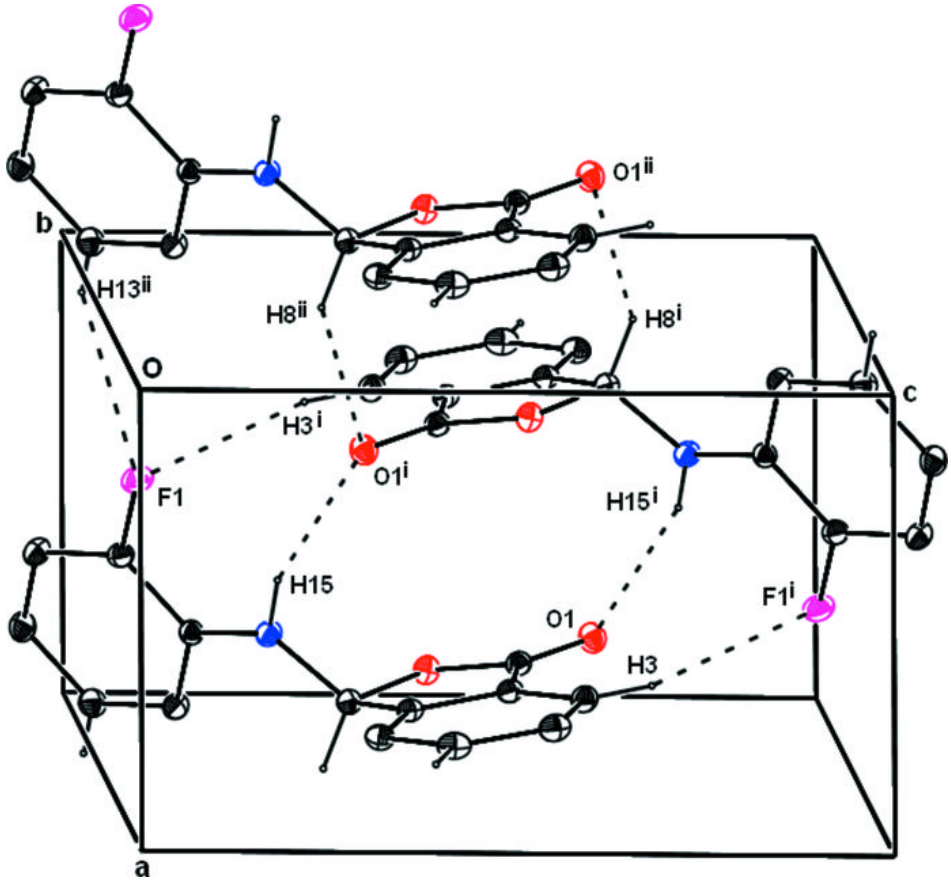

Fig. 3