organic compounds

A redetermination of 1-(2-fluorophenyl)-pyrrolidine-2,5-dione

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The crystal structure of the title compound, $C_{10}H_8FNO_2$, has been redetermined with greater precision and with a detailed investigation of the intermolecular interactions [previous report; space group: $Pca2_1$; cell: a = 11.064 (2), b = 10.271 (2), c = 8.053 (2) Å; R factor: 0,061; temperature (K): 295; Taira, Takayama & Terada (1988). *J. Chem. Soc. Perkin Trans. 2*, pp. 1439–1445]. The molecule is non-planar with the benzene and pyrrolidine rings tilted by 63.18 (12)° with respect to one another. In the crystal structure, intermolecular C—H⋯O and C—H⋯F hydrogen bonds together with C—H⋯π interactions link the molecules into a three-dimensional network. The C—H⋯O and C—H⋯F hydrogen bonds generate edge-fused $R_2^2(8)R_2^2(11)R_4^3(17)$ and $R_6^4(25)$ hydrogen-bonded rings.

Related literature

For hydrogen-bonding motif details, see: Bernstein *et al.* (1995); Etter (1990). For background, see: Taira *et al.* (1988); Rankin *et al.* (1989); Allen (2002); Malamas *et al.* (1994); Toupet *et al.* (1996); Zhang *et al.* (2007). For related structures, see: Argay & Seres (1973); Kwiatkowski *et al.* (1990); Kwiatkowski & Karolak-Wojciechowska (1990, 1992); Karolak-Wojciechowska *et al.* (1996); Argay *et al.* (1999). For details of the preparation, see: Haberle *et al.* (1989).
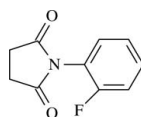
Experimental

Crystal data

$C_{10}H_8FNO_2$	a = 11.0620 (13) Å
M_r = 193.17	b = 10.2689 (13) Å
Orthorhombic, $Pca2_1$	c = 8.0586 (8) Å

V = 915.41 (18) Å³	μ = 0.11 mm⁻¹
Z = 4	T = 296 K
Mo $K\alpha$ radiation	$0.69 \times 0.56 \times 0.43$ mm

Data collection

STOE IPDS 2 diffractometer	3862 measured reflections
Absorption correction: integration	946 independent reflections
(X-$RED32$; Stoe & Cie, 2002)	826 reflections with $I > 2\sigma(I)$
T_{min} = 0.940, T_{max} = 0.967	R_{int} = 0.023

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.032	1 restraint
$wR(F^2)$ = 0.095	H-atom parameters constrained
S = 1.06	$\Delta\rho_{max}$ = 0.23 e Å⁻³
946 reflections	$\Delta\rho_{min}$ = −0.12 e Å⁻³
128 parameters	

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
C8—H8A⋯O2[i]	0.97	2.58	3.483 (3)	155
C8—H8B⋯O2[ii]	0.97	2.49	3.370 (4)	151
C4—H4⋯O2[iii]	0.93	2.68	3.417 (4)	137
C9—H9B⋯F1[iv]	0.97	2.60	3.304 (4)	129
C9—H9A⋯O1[v]	0.97	2.63	3.558 (4)	160
C3—H3⋯$Cg1$[iii]	0.93	2.97	3.724 (3)	140

Symmetry codes: (i) $x - \frac{1}{2}, -y, z$; (ii) $-x + 1, -y, z + \frac{1}{2}$; (iii) $-x + 1, -y + 1, z + \frac{1}{2}$; (iv) $-x + 1, -y, z - \frac{1}{2}$; (v) $x + \frac{1}{2}, -y, z$. $Cg1$ is the centroid of the fluorophenyl ring.

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).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ2405).

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supplementary materials

A redetermination of 1-(2-fluorophenyl)pyrrolidine-2,5-dione

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Comment

The crystal structures of several pyrrolidine-2,5-dione (succinimide) derivatives have been reported with respect to their biological properties, *e.g.* antiepileptic (Allen & Kennard, 1993; Argay & Seres, 1973), anticonvulsive (Argay & Seres, 1973; Kwiatkowski *et al.*, 1990; Kwiatkowski & Karolak-Wojciechowska, 1992; Karolak-Wojciechowska *et al.*, 1996), fungicidal (Taira *et al.*, 1988; Haberle *et al.*, 1989;Zhang *et al.*, 2007), antiantrogenic (Zhang *et al.*, 2007; Rankin *et al.*, 1989), and other pharmacological activities (Kwiatkowski & Karolak-Wojciechowska, 1990; Malamas *et al.*, 1994; Toupet *et al.*, 1996; Argay *et al.*, 1999; Zhang *et al.*, 2007). In each of these compounds, the ring nitrogen is substituted either by a methyl group or differently substituted phenyl and pyridine rings or a morpholinomethyl group. Against this background, we present here the crystal structure of the title compound, (I), with better precision and incorporating a detailed investigation of the intermolecular interactions.

The title compound, (I), is not planar, with a dihedral angle of 63.18 (12)° between the benzene and pyrrolidine rings [This angle is 63.5° in the previous determination (Taira *et al.*, 1988)]. The crystal packing is stabilized by C—H···O and C—H···F intermolecular hydrogen bonds and also by C—H···π interactions (Table 2). The intermolecular hydrogen bonds generate edge-fused $R_2^2(8)R_2^2(11)R_4^3(17)$ (Fig. 2) and $R_6^4(25)$ (Fig. 3) (Etter, 1990; Bernstein *et al.*, 1995) hydrogen bonded rings in a three- dimensional network (Fig. 5). In addition, C—H···π interactions form chains of molecules along the [001] direction (Table 2, Fig.4).

Experimental

Compound (I) was prepared as described by Haberle *et al.* (1989), using succinic acid anhydride and 2-fluoroaniline as starting materials. Well shaped crystals of (I) were obtained by slow evaporation of an ethanol solution [m.p.:422–423 K].

Refinement

The H atoms were included in calculated positions and refined using a riding model approximation. Constrained C—H bond lengths and isotropic U parameters: 0.93 Å and $U_{iso}(H) = 1.2U_{eq}(C)$ for Csp^2—H; 0.97 Å and $U_{iso}(H) = 1.2U_{eq}(C)$ for methylene C—H; The absolute structure could not be determined, and 745 Friedel pairs were averaged before the last refinement.
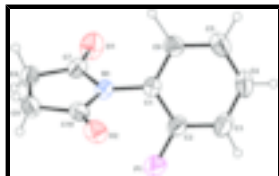
supplementary materials

Figures

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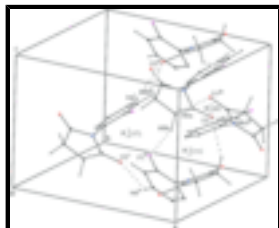

Fig. 2. Part of the crystal structure of (I), showing the formation of an $R_2^2(8)R_2^2(11)R_4^3(17)$ motifs. H atoms not involved in hydrogen bonds have been omitted for clarity. [Symmetry codes: (i) x, $1 - y$, z; (ii) $1 - x$, $1 - y$, $z + 1/2$; (iii) $x + 1/2$, $1 - y$, z; (iv) $1 - x$, $1 - y$, $z - 1/2$].
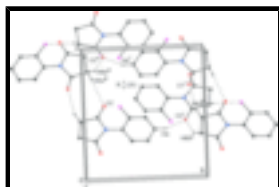

Fig. 3. Part of the crystal structure of (I), showing the formation of an $R_6^4(25)$ motif. H atoms not involved in hydrogen bonds have been omitted for clarity. [Symmetry codes: (i) $1 - x$, $1 - y$, $1 - z$; (ii) x, $y + 1$, z; (iii) $x + 1/2$, $-y$, z; (iv) $3/2 - x$, y, $z + 1/2$; (v) $x + 1/2$, $1 - y$, z].
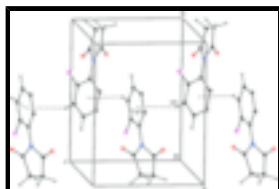

Fig. 4. Part of the crystal structure of (I), showing the C—H···π interactions along the c axis. H atoms not involved in hydrogen bonds have been omitted for clarity.
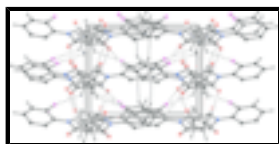
Fig. 5. A packing diagram for (I), with hydrogen bonds drawn as dashed lines. H atoms not involved in hydrogen bonds have been omitted for clarity.

1-(2-fluorophenyl)pyrrolidine-2,5-dione

Crystal data

$C_{10}H_8FNO_2$	$F_{000} = 400$
$M_r = 193.17$	$D_x = 1.402$ Mg m^{-3}
Orthorhombic, $Pca2_1$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: P 2c -2ac	Cell parameters from 3862 reflections
$a = 11.0620\ (13)$ Å	$\theta = 2.7–27.2°$
$b = 10.2689\ (13)$ Å	$\mu = 0.11$ mm^{-1}
$c = 8.0586\ (8)$ Å	$T = 296$ K
$V = 915.41\ (18)$ Å^3	Prism, colorless
$Z = 4$	$0.69 \times 0.56 \times 0.43$ mm

Data collection

STOE IPDS 2 diffractometer	946 independent reflections
Monochromator: plane graphite	826 reflections with $I > 2\sigma(I)$
Detector resolution: 6.67 pixels mm^{-1}	$R_{int} = 0.023$
$T = 296$ K	$\theta_{max} = 26.0°$
w–scan rotation method	$\theta_{min} = 2.7°$
Absorption correction: integration (X-RED32; Stoe & Cie, 2002)	$h = -13 \rightarrow 13$
$T_{min} = 0.940$, $T_{max} = 0.967$	$k = -10 \rightarrow 12$
3862 measured reflections	$l = -9 \rightarrow 8$

Refinement

Refinement on F^2	H-atom parameters constrained
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.0609P)^2 + 0.0455P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.032$	$(\Delta/\sigma)_{max} = <0.001$
$wR(F^2) = 0.095$	$\Delta\rho_{max} = 0.23$ e Å^{-3}
$S = 1.06$	$\Delta\rho_{min} = -0.12$ e Å^{-3}
946 reflections	Extinction correction: SHELXL, $Fc^* = kFc[1 + 0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
128 parameters	Extinction coefficient: 0.021 (6)
1 restraint	
Primary atom site location: structure-invariant direct methods	
Secondary atom site location: difference Fourier map	
Hydrogen site location: inferred from neighbouring sites	

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 > 2sigma(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	0.38002 (19)	0.3133 (2)	0.4596 (3)	0.0474 (6)

C2	0.4685 (2)	0.3725 (2)	0.5537 (4)	0.0595 (7)
C3	0.4684 (3)	0.5036 (3)	0.5824 (4)	0.0741 (9)
H3	0.5292	0.5423	0.6449	0.089*
C4	0.3764 (3)	0.5768 (3)	0.5169 (5)	0.0771 (9)
H4	0.3747	0.6661	0.5357	0.093*
C5	0.2876 (3)	0.5205 (3)	0.4247 (5)	0.0732 (9)
H5	0.2258	0.5715	0.3814	0.088*
C6	0.2887 (2)	0.3884 (3)	0.3953 (4)	0.0614 (7)
H6	0.2278	0.3503	0.3321	0.074*
C7	0.2954 (2)	0.0911 (2)	0.4864 (4)	0.0549 (6)
C8	0.3285 (3)	−0.0423 (3)	0.4281 (4)	0.0656 (7)
H8A	0.2674	−0.0760	0.3532	0.079*
H8B	0.3364	−0.1014	0.5213	0.079*
C9	0.4468 (3)	−0.0271 (3)	0.3403 (5)	0.0679 (8)
H9A	0.5096	−0.0756	0.3972	0.081*
H9B	0.4409	−0.0588	0.2272	0.081*
C10	0.4745 (2)	0.1146 (2)	0.3419 (4)	0.0545 (6)
N1	0.38437 (15)	0.17631 (17)	0.4328 (3)	0.0474 (5)
O1	0.20830 (18)	0.1239 (2)	0.5658 (3)	0.0823 (7)
O2	0.55827 (15)	0.1717 (2)	0.2795 (3)	0.0732 (6)
F1	0.55669 (16)	0.29716 (17)	0.6176 (3)	0.0897 (7)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0537 (11)	0.0447 (11)	0.0437 (15)	−0.0033 (9)	0.0037 (11)	−0.0015 (9)
C2	0.0654 (13)	0.0553 (13)	0.0577 (18)	−0.0068 (11)	−0.0085 (13)	0.0018 (14)
C3	0.094 (2)	0.0570 (14)	0.072 (2)	−0.0178 (14)	−0.0115 (16)	−0.0071 (15)
C4	0.112 (2)	0.0448 (13)	0.075 (2)	0.0022 (14)	0.0022 (18)	−0.0083 (14)
C5	0.0858 (19)	0.0602 (15)	0.074 (2)	0.0203 (13)	0.0016 (18)	−0.0027 (16)
C6	0.0556 (13)	0.0668 (15)	0.062 (2)	0.0064 (11)	−0.0023 (12)	−0.0096 (14)
C7	0.0585 (13)	0.0551 (13)	0.0510 (15)	−0.0114 (11)	−0.0018 (13)	0.0013 (11)
C8	0.0771 (15)	0.0497 (12)	0.0701 (19)	−0.0079 (12)	−0.0116 (16)	0.0015 (14)
C9	0.0761 (16)	0.0555 (15)	0.072 (2)	0.0138 (12)	−0.0120 (15)	−0.0074 (15)
C10	0.0517 (11)	0.0587 (14)	0.0531 (16)	0.0089 (11)	−0.0025 (12)	0.0013 (13)
N1	0.0479 (9)	0.0472 (10)	0.0472 (13)	−0.0046 (7)	0.0011 (9)	−0.0030 (9)
O1	0.0748 (12)	0.0819 (13)	0.0903 (17)	−0.0166 (10)	0.0332 (12)	−0.0054 (13)
O2	0.0589 (9)	0.0771 (12)	0.0836 (17)	0.0052 (8)	0.0204 (10)	0.0022 (11)
F1	0.0911 (12)	0.0716 (10)	0.1065 (18)	−0.0084 (8)	−0.0444 (11)	0.0065 (10)

Geometric parameters (Å, °)

C1—C6	1.373 (4)	C7—O1	1.205 (3)
C1—C2	1.380 (3)	C7—N1	1.386 (3)
C1—N1	1.424 (3)	C7—C8	1.494 (4)
C2—F1	1.347 (3)	C8—C9	1.496 (5)
C2—C3	1.366 (4)	C8—H8A	0.9700
C3—C4	1.370 (5)	C8—H8B	0.9700
C3—H3	0.9300	C9—C10	1.487 (4)

C4—C5	1.361 (4)	C9—H9A	0.9700
C4—H4	0.9300	C9—H9B	0.9700
C5—C6	1.376 (4)	C10—O2	1.206 (3)
C5—H5	0.9300	C10—N1	1.390 (3)
C6—H6	0.9300		
C6—C1—C2	118.8 (2)	N1—C7—C8	107.9 (2)
C6—C1—N1	121.5 (2)	C7—C8—C9	105.5 (2)
C2—C1—N1	119.6 (2)	C7—C8—H8A	110.6
F1—C2—C3	120.2 (2)	C9—C8—H8A	110.6
F1—C2—C1	118.1 (2)	C7—C8—H8B	110.6
C3—C2—C1	121.7 (2)	C9—C8—H8B	110.6
C2—C3—C4	118.5 (3)	H8A—C8—H8B	108.8
C2—C3—H3	120.8	C10—C9—C8	106.1 (2)
C4—C3—H3	120.8	C10—C9—H9A	110.5
C5—C4—C3	120.9 (3)	C8—C9—H9A	110.5
C5—C4—H4	119.6	C10—C9—H9B	110.5
C3—C4—H4	119.6	C8—C9—H9B	110.5
C4—C5—C6	120.4 (3)	H9A—C9—H9B	108.7
C4—C5—H5	119.8	O2—C10—N1	123.3 (2)
C6—C5—H5	119.8	O2—C10—C9	129.1 (2)
C1—C6—C5	119.6 (3)	N1—C10—C9	107.6 (2)
C1—C6—H6	120.2	C7—N1—C10	112.69 (19)
C5—C6—H6	120.2	C7—N1—C1	123.52 (19)
O1—C7—N1	123.8 (3)	C10—N1—C1	123.70 (19)
O1—C7—C8	128.3 (3)		
C6—C1—C2—F1	179.2 (3)	C8—C9—C10—O2	−176.7 (3)
N1—C1—C2—F1	−0.1 (4)	C8—C9—C10—N1	3.7 (3)
C6—C1—C2—C3	−0.9 (4)	O1—C7—N1—C10	−179.3 (3)
N1—C1—C2—C3	179.8 (3)	C8—C7—N1—C10	0.6 (3)
F1—C2—C3—C4	−179.3 (3)	O1—C7—N1—C1	−2.8 (4)
C1—C2—C3—C4	0.8 (5)	C8—C7—N1—C1	177.2 (2)
C2—C3—C4—C5	−0.3 (5)	O2—C10—N1—C7	177.6 (3)
C3—C4—C5—C6	−0.1 (5)	C9—C10—N1—C7	−2.8 (3)
C2—C1—C6—C5	0.4 (4)	O2—C10—N1—C1	1.1 (4)
N1—C1—C6—C5	179.7 (3)	C9—C10—N1—C1	−179.3 (2)
C4—C5—C6—C1	0.0 (5)	C6—C1—N1—C7	−61.5 (3)
O1—C7—C8—C9	−178.3 (3)	C2—C1—N1—C7	117.8 (3)
N1—C7—C8—C9	1.7 (3)	C6—C1—N1—C10	114.6 (3)
C7—C8—C9—C10	−3.3 (3)	C2—C1—N1—C10	−66.1 (3)

Hydrogen-bond geometry (Å, °)

D—H···A	D—H	H···A	D···A	D—H···A
C8—H8A···O2[i]	0.97	2.58	3.483 (3)	155
C8—H8B···O2[ii]	0.97	2.49	3.370 (4)	151
C4—H4···O2[iii]	0.93	2.68	3.417 (4)	137
C9—H9B···F1[iv]	0.97	2.60	3.304 (4)	129
C9—H9A···O1[v]	0.97	2.63	3.558 (4)	160

C3—H3···Cg1[iii]   0.93   2.97   3.724 (3)   140

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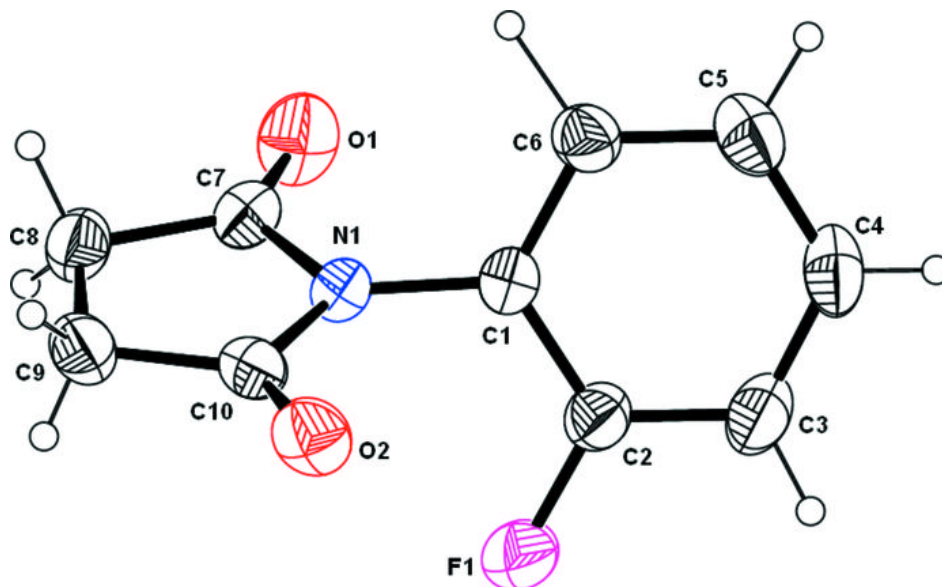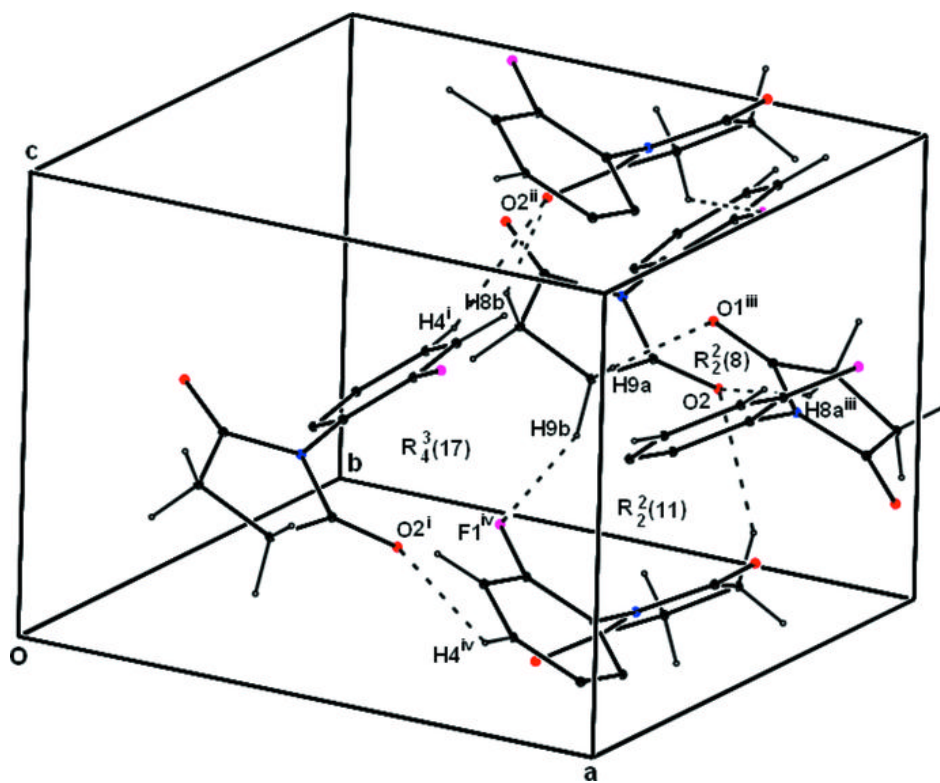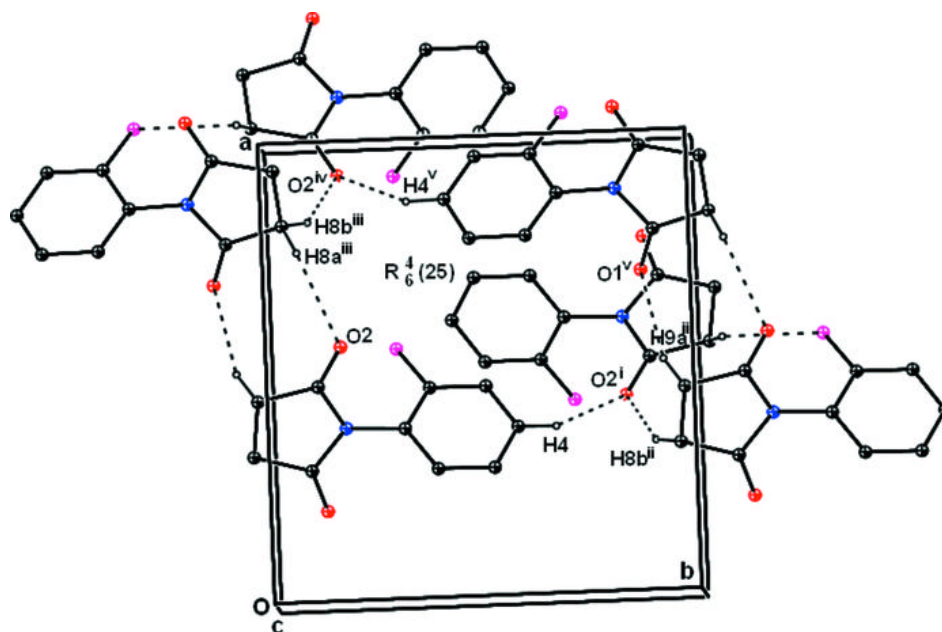
Fig. 1

Fig. 2

Fig. 3

Fig. 4

Fig. 5