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1-(4-Fluoro-2-hydroxyphenyl)ethanone 4-nitrobenzoylhydrazone

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Key indicators

Single-crystal X-ray study
T = 273 K
Mean σ(C–C) = 0.004 Å
R factor = 0.067
wR factor = 0.170
Data-to-parameter ratio = 14.2

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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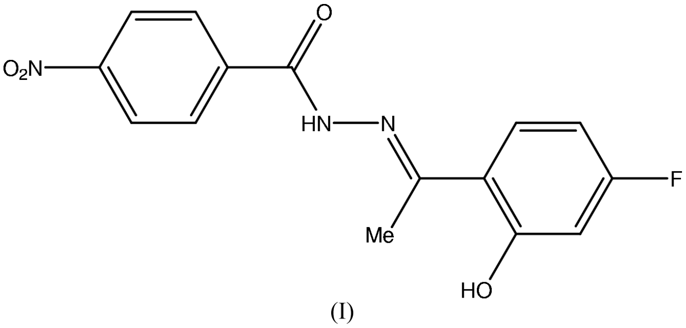
In the title compound, $C_{15}H_{12}FN_3O_4$, the planes of the nitrophenyl and 4-fluoro-2-hydroxyphenyl fragments form dihedral angles of 25.7 (1) and 10.5 (2)°, respectively, with the C—N—N—C plane of the central hydrazone linkage. In the crystal structure, molecules are linked into layers by intermolecular C—H···F and C—H···O interactions. Adjacent layers are interlinked by N—H···O and C—H···O hydrogen bonds.

Comment

Schiff bases have received considerable attention because of their structural and pharmacological properties, such as anticancer and antimicrobial activities (Bekhit *et al.*, 2004). The condensation reaction of 4-nitrobenzhydrazide with 4-fluoro-2-hydroxyacetophenone led to the formation of the title compound, (I).



(I)

The molecule of (I) is in the keto tautomeric form and is distorted from planarity (Fig. 1). The nitrophenyl fragment is planar with a maximum deviation of 0.035 (2) Å for atom O4; the 4-fluoro-2-hydroxyphenyl fragment is planar within 0.016 (2) Å. The dihedral angle between the planes of the nitrophenyl and 4-fluoro-2-hydroxyphenyl fragments is 18.1 (1)°; these fragments form dihedral angles of 25.7 (1) and

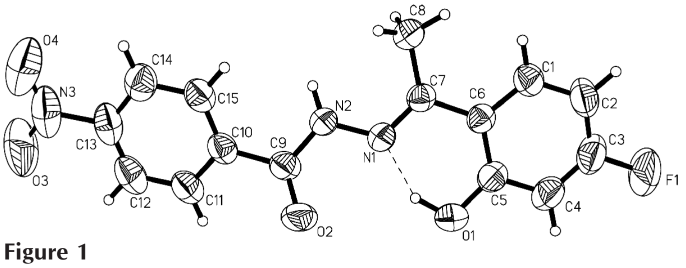


Figure 1
The molecular structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme. The dashed line indicates a hydrogen bond.

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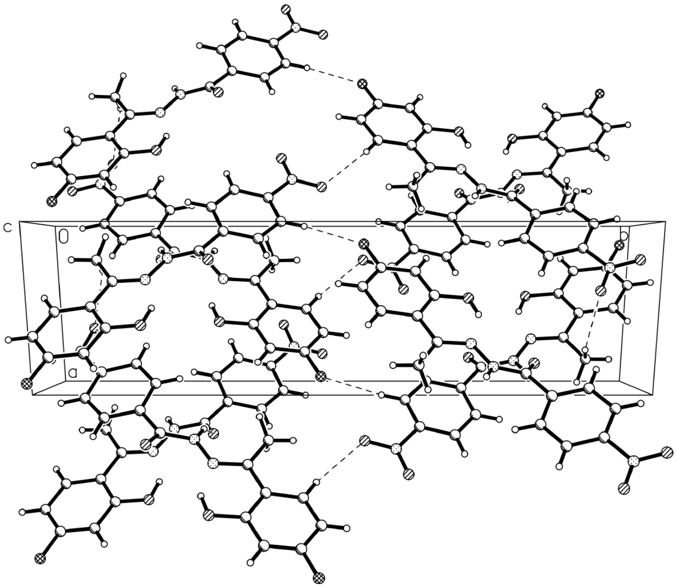

Figure 2
The packing of the molecules of (I), viewed along the *c* axis. Hydrogen bonds are shown as dashed lines.

10.5 (2)°, respectively, with the plane of the central C7—N1—N2—C9 linkage. The bond lengths observed in the structure (Table 1) show normal values (Allen *et al.*, 1987).

An intramolecular O1—H1⋯N1 hydrogen bond is observed in the molecule. In the crystal structure, the molecules are linked into layers by two intermolecular C12—H12*A*⋯F1^{iv} and C1—H1*B*⋯O3^{ii} interactions (symmetry codes are given in Table 2). Adjacent layers are interlinked by N2—H2*A*⋯O2^{i} and C8—H8*B*⋯O4^{iii} hydrogen bonds, forming a network structure (Fig. 2 and Table 2).

Experimental

An ethanol solution (80 ml) of 4-nitrobenzhydrazide (0.30 g, 1.7 mmol) was refluxed with 4-fluoro-2-hydroxyacetophenone (0.26 g, 1.7 mmol) for 2 h. The solid obtained was recrystallized from pyridine to give brown crystals.

Crystal data

$C_{15}H_{12}FN_3O_4$
M_r = 317.28
Monoclinic, $P2_1/c$
a = 7.4152 (19) Å
b = 24.532 (6) Å
c = 8.739 (2) Å
β = 113.576 (4)°
V = 1457.0 (6) Å^3
Z = 4

D_x = 1.446 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 936 reflections
θ = 1.7–26.5°
μ = 0.12 mm^{-1}
T = 273 (2) K
Block, brown
0.49 × 0.45 × 0.26 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.946, T_{max} = 0.971
15277 measured reflections

3010 independent reflections
2472 reflections with $I > 2\sigma(I)$
R_{int} = 0.024
θ_{max} = 26.5°
$h = -9 \rightarrow 9$
$k = -30 \rightarrow 30$
$l = -10 \rightarrow 10$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.067
$wR(F^2)$ = 0.170
S = 1.15
3010 reflections
212 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0658P)^2 + 0.5534P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

F1—C3	1.355 (3)	O4—N3	1.201 (4)
O1—C5	1.345 (3)	N1—C7	1.287 (3)
O2—C9	1.220 (3)	N1—N2	1.381 (2)
O3—N3	1.219 (4)	N2—C9	1.347 (3)
C7—N1—N2—C9	−165.1 (2)	N2—C9—C10—C15	−34.9 (3)
C5—C6—C7—N1	−0.3 (3)	O2—C9—C10—C11	−34.4 (3)
N1—N2—C9—O2	7.4 (3)	O3—N3—C13—C12	−2.2 (4)
N1—N2—C9—C10	−173.80 (18)	O4—N3—C13—C14	−1.1 (4)

Table 2
Hydrogen-bonding geometry (Å, °).

D—H⋯*A*	*D*—H	H⋯*A*	*D*⋯*A*	*D*—H⋯*A*
O1—H1⋯N1	0.96 (3)	1.66 (3)	2.517 (2)	147 (3)
N2—H2*A*⋯O2^{i}	0.86	2.15	2.889 (3)	144
C1—H1*B*⋯O3^{ii}	0.93	2.48	3.353 (4)	157
C8—H8*B*⋯O4^{iii}	0.96	2.57	3.328 (5)	136
C12—H12*A*⋯F1^{iv}	0.93	2.44	3.366 (3)	173

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

After their location in a difference map, all H atoms except H1 were positioned geometrically and allowed to ride on the parent C atoms, with C—H = 0.93–0.96 Å and N—H = 0.86 Å, and U_{iso}(H) = 1.2 or 1.5U_{eq}(C,N). Atom H1 was refined isotropically.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*, *PARST* (Nardelli, 1995) and *PLATON* (Spek, 2003).

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