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4-(*p*-Fluorobenzylideneamino)-3-methyl-4,5-dihydro-1*H*-1,2,4-triazol-5-one

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.002 Å
R factor = 0.037
wR factor = 0.107
Data-to-parameter ratio = 19.5

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

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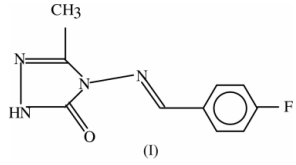
The molecule of the title compound, $C_{10}H_9FN_4O$, is almost planar. In the 1,2,4-triazole moiety, the C=N, C—N and N—N bond lengths are normal. The crystal structure is stabilized by intra- and intermolecular hydrogen bonds.

Comment

1,2,4-Triazole compounds are a relatively new group of Schiff bases; they possess significant antibacterial, anticancer, anti-inflammatory and antitoxic activity (Williams, 1972; McCarrick *et al.*, 1999; Liu *et al.*, 1999). In order to provide information about structure–activity relationships in a series of similar complexes, the crystal structure of the title compound, (I), synthesized by Kahveci & Ikizler (2000), is presented here.



(I)

A perspective view of the molecule of (I) with the atomic numbering is shown in Fig. 1. In the 1,2,4-triazole moiety, the bond lengths are normal. The C—N, C2=N3 and N2—N3 bonds are in good agreement with the values found by Işık *et al.* (2003). The other bond lengths generally agree well with those found in the crystal structures of 1,2,4-triazole derivatives (Ocak *et al.*, 2003).

In the molecule of (I), both rings are planar within experimental error and the largest deviation of 0.003 (2) Å is for atom N3 of the triazole ring. Meanwhile, atoms F1, C3 and O1 lie almost in the ring plane, with a maximum deviation of 0.016 (2) Å for atom F1. Also, the dihedral angle between the C5–C10 ring and 1,2,4-triazole ring is 6 (1)°. These results show that the compound is almost planar.

In addition, the crystal structure is stabilized by intra- and two intermolecular hydrogen bonds. Crystal packing involves C—H···F and N—H···O-type hydrogen bonds, resulting in an infinite network structure. Also, the compound has C—H···O-type intramolecular hydrogen bonds. Details of these interactions are give in Table 2.

Experimental

The corresponding *N*-amino compound (0.01 mol) was heated with 4-fluorobenzaldehyde (1.04 ml, 0.01 mol) in an oil bath at 448 K for 1 h. After cooling, the resulting solid was recrystallized from ethanol (yield; 84%) to afford the desired compound (I). M.p. 506–507 K. Calculated: C 54.54, H 4.12, N 25.44%; found: C 54.16, H:4.16, N

25.14%. IR data (KBr cm^{-1}): 3190 (N—H), 1715 (C═O), 1605, 1565 (C═N), 840 (aromatic). ^1H NMR ($CDCl_3$, δ p.p.m.): 2.40 (CH_3, *s*, 3H), 9,76 (CH, *s*, 1H), 11.90 (NH, *s*, 1H), Ar—H: 7.00–7.80 (*m*, 4H).

Crystal data

$C_{10}H_9FN_4O$	D_x = 1.415 Mg m^{-3}
M_r = 220.21	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 8026 reflections
a = 7.0012 (8) Å	θ = 1.5–29.5°
b = 13.8430 (12) Å	μ = 0.11 mm^{-1}
c = 11.1307 (11) Å	T = 293 (2) K
β = 106.572 (8)°	Prism, colourless
V = 1033.95 (18) $Å^3$	0.40 × 0.37 × 0.30 mm
Z = 4	

Data collection

Stoe IPDS-II diffractometer	R_{int} = 0.000 ??
ω scans	θ_{max} = 29.5°
Absorption correction: none	$h = -9 \rightarrow 9$
10015 measured reflections	$k = -19 \rightarrow 19$
2861 independent reflections	$l = -15 \rightarrow 15$
1657 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0664P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.037	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2)$ = 0.107	$\Delta\rho_{max}$ = 0.14 e $Å^{-3}$
S = 0.89	$\Delta\rho_{min}$ = −0.20 e $Å^{-3}$
2861 reflections	Extinction correction: *SHELXL*97
147 parameters	Extinction coefficient: 0.054 (11)
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

O1—C1	1.2332 (15)	N4—C4	1.2648 (16)
N1—C2	1.3755 (15)	N3—C2	1.2923 (14)
N1—N4	1.3824 (12)	N3—N2	1.3814 (15)
N1—C1	1.3899 (14)		
C2—N1—N4—C4	−172.87 (11)		

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O1^{i}	0.86	1.98	2.832 (1)	172
C3—H3*C*···F1^{ii}	0.96	2.45	3.175 (2)	132
C4—H4···O1	0.93	2.27	2.939 (2)	129

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

H atoms were positioned geometrically and refined using a riding model, fixing the aromatic C—H distance at 0.93 Å, the methyl C—H distances at 0.96 Å and the N—H distance at 0.86 Å. The methyl group was allowed to rotate but not to tip.

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*III (Burnett & Johnson, 1996); software used to prepare material for publication: *WinGX* (Farrugia, 1997) and *PARST* (Nardelli, 1995).

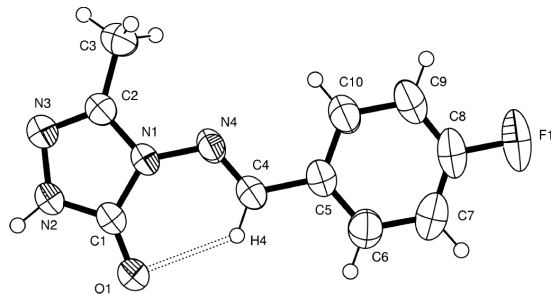


Figure 1
The structure of the title compound, showing 50% probability displacement ellipsoids and the atom-numbering scheme. The hydrogen bond is shown with dashed lines.

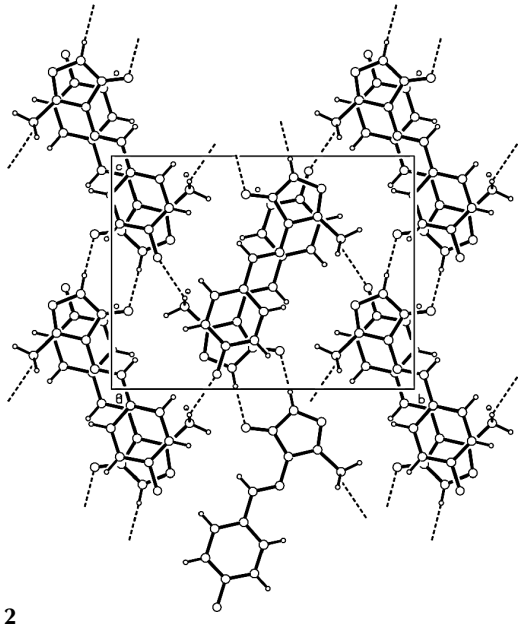


Figure 2
A packing diagram of the title compound, illustrating the hydrogen-bonding network.

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