

3-Benzyl-4-(*p*-fluorobenzylidenoamino)-
4,5-dihydro-1*H*-1,2,4-triazol-5-oneŞamil Işık,^{a*} Bahittin Kahveci,^b
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Key indicators

Single-crystal X-ray study

T = 293 K

Mean $\sigma(\text{C}-\text{C}) = 0.002 \text{ \AA}$

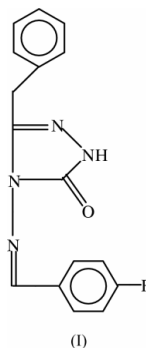
R factor = 0.047

wR factor = 0.153

Data-to-parameter ratio = 16.7

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.In the crystal structure of the title compound, $\text{C}_{16}\text{H}_{13}\text{N}_4\text{O}$, molecules are linked through intermolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds to form dimers. The dimers are held together by van der Waals interactions.

Comment

Most Schiff bases possess antibacterial, anticancer, anti-inflammatory and antitoxic activities (Williams, 1972), and sulfur-containing Schiff bases are particularly effective. For example, the 4-amino-3-methyl-1,2,4-triazole-5-thione derivative of *p*-nitrobenzaldehyde is known as a highly effective inhibitor of *Staphylococcus aureus* (Liu *et al.*, 1999). As part of our continuing study of Schiff bases of amine and 1,2,4-triazole derivatives, we have structurally characterized the title compound, (I) (Fig. 1)The bonds and angles in (I) (Table 1) are consistent with those in similar substituted triazoles (Kahveci *et al.*, 2003; Çoruh *et al.*, 2003; Işık *et al.*, 2003). The benzyl rings and 1,2,4-triazole ring are almost planar, with a maximum deviation of 0.008 (2) Å for N2. The dihedral angles between the 1,2,4-triazole ring and benzyl rings C1–C6 and C11–C16 are 179.3 (1) and 104.9 (1)°, respectively, and the dihedral angle between the benzyl rings is 104.9 (1)°.The molecules of (I) form hydrogen-bonded dimers through the $\text{N}-\text{H}\cdots\text{O}$ interactions involving triazole atoms O1 and N2, shown as dashed lines in Fig. 2. This appears to be a common feature in Schiff bases of 4-amino-3-alkyl-1,2,4-triazole-5-thiones (Sen *et al.*, 1998). The molecule also exhibits an intramolecular $\text{C}7-\text{H}7\cdots\text{O}1$ contact (Table 2).

Experimental

The corresponding amino compound (1.90 g., 0.01 mol) was heated in an oil bath with 4-fluorobenzene (1.04 ml, 0.01 mol) at 428–433 K for one hour and then allowed to cool. The solid product was recryst-

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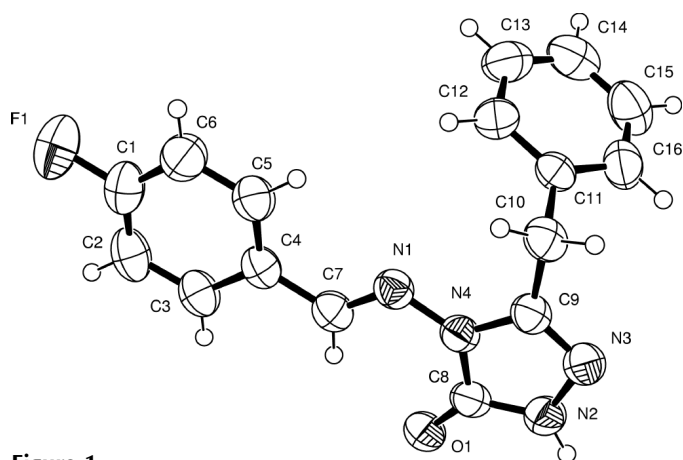


Figure 1
The structure of (I), with displacement ellipsoids drawn at the 50% probability level and the atom-numbering scheme.

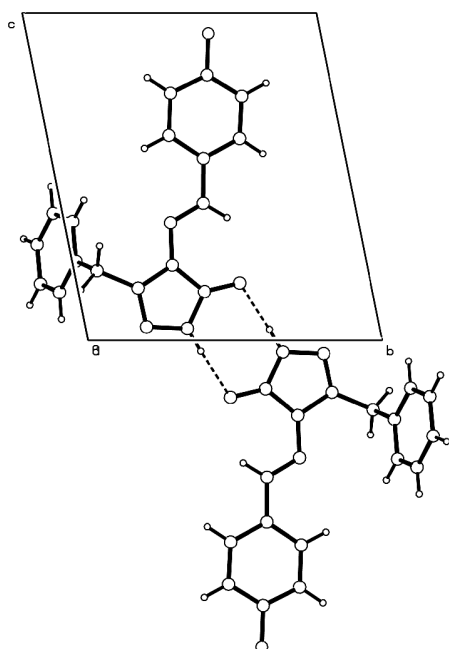


Figure 2
Dimer formation in the crystal structure of (I). Intermolecular N—H...O hydrogen bonds are shown as dashed lines.

tallized from ethanol (yield; 96%) to give the desired compound (I) (m.p. = 466–467 K). Calculated: C 64.86, H 4.42, N 18.91%; found: C 64.83, H 4.26, N 18.35%. IR data (KBr/cm⁻¹): 3250 (N—H), 1720 (C=O), 1620, 1600 (C=N), 700, 680, 840 (aromatic). ¹H NMR (CDCl₃, δ/p.p.m.): 4.18 (CH₂, s, 2H), 9.78 (CH, s, 1H), 10.10 (NH, s, 1H); Ar—H: 7.04–7.40 (m, 7H), 7.70 (d, 2H).

Crystal data

C₁₆H₁₃FN₄O
M_r = 296.30
Triclinic, *P* $\bar{1}$
a = 7.0290 (15) Å
b = 10.141 (2) Å
c = 11.205 (3) Å
α = 96.40 (2)°
β = 102.944 (17)°
γ = 108.394 (16)°
V = 724.2 (3) Å³

Z = 2
D_x = 1.359 Mg m⁻³
Mo Kα radiation
Cell parameters from 8077 reflections
θ = 0.0–29.5°
μ = 0.10 mm⁻¹
T = 293 (2) K
Prism, colourless
0.75 × 0.46 × 0.22 mm

Data collection

Stoe IPDS2 diffractometer
Rotation method scans
Absorption correction: none
3550 measured reflections
3550 independent reflections

2341 reflections with *I* > 2σ(*I*)
θ_{max} = 29.3°
h = -9 → 0
k = -12 → 12
l = -14 → 15

Refinement

Refinement on F²
R[F² > 2σ(F²)] = 0.047
wR(F²) = 0.153
S = 1.00
3550 reflections
212 parameters
H atom parameters constrained

w = 1/[σ²(F_o²) + (0.1093P)²]
where P = (F_o² + 2F_c²)/3
(Δ/σ)_{max} < 0.001
Δρ_{max} = 0.17 e Å⁻³
Δρ_{min} = -0.17 e Å⁻³
Extinction correction: SHELXL97
Extinction coefficient: 0.028 (10)

Table 1

Selected geometric parameters (Å, °).

F1—C1	1.3598 (19)	N2—N3	1.3777 (19)
O1—C8	1.2314 (18)	N3—C9	1.2975 (19)
N1—C7	1.2730 (19)	N4—C9	1.3859 (18)
N1—N4	1.3878 (16)	N4—C8	1.3890 (19)
N2—C8	1.3459 (19)		
C7—N1—N4	117.68 (13)		

Table 2

Hydrogen-bonding geometry (Å, °).

D—H...A	D—H	H...A	D...A	D—H...A
N2—H2N...O1 ⁱ	0.86	1.97	2.799 (2)	161
C7—H7...O1	0.93	2.19	2.893 (2)	132

Symmetry code: (i) -2 - x, -1 - y, 2 - z.

H atoms were positioned geometrically and refined isotropically using a riding model, fixing the aromatic C—H distance at 0.93 Å, the methylene C—H distance at 0.97 Å and the N—H distance at 0.86 Å.

Data collection: X-Area (Stoe & Cie, 2002); cell refinement: X-Area; data reduction: X-Red (Stoe & Cie, 2002); program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEPIII (Burnett & Johnson, 1996); software used to prepare material for publication: WinGX (Farrugia, 1997) and PARST (Nardelli, 1995).

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