

4-(*p*-Fluorobenzylamino)-3-phenyl-4,5-dihydro-1*H*-1,2,4-triazol-5-oneŞamil Işık,^{a*} Bahattin Kahveci,^b
Erbil Açar^c and Selami Şaşmaz^b

^aDepartment of Physics, Faculty of Arts and Sciences, Ondokuz Mayıs University, Kurupelit, 55139 Samsun, Turkey, ^bKaradeniz Teknik University, Rize Art and Science Faculty, Department of Chemistry, Rize, Turkey, and ^cDepartment of Chemistry, Faculty of Arts and Sciences, Ondokuz Mayıs University, Kurupelit, 55139 Samsun, Turkey

Correspondence e-mail: samili@omu.edu.tr

Key indicators

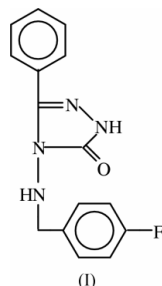
Single-crystal X-ray study
T = 293 K
Mean $\sigma(\text{C}-\text{C}) = 0.004 \text{ \AA}$
R factor = 0.043
wR factor = 0.089
Data-to-parameter ratio = 15.1

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

The molecules of the title compound, $\text{C}_{15}\text{H}_{13}\text{FN}_4\text{O}$, display the characteristic features of 1,2,4-triazole derivatives. The triazole ring is planar and the two benzene rings are nearly parallel, with a dihedral angle of $8.5(1)^\circ$. The crystal structure is stabilized by $\text{N}-\text{H}\cdots\text{O}$ and $\text{N}-\text{H}\cdots\text{N}$ intermolecular hydrogen bonds.

Comment

1,2,4-Triazole and its derivatives are starting materials for the synthesis of many heterocycles (Desenko, 1995). Apart from its extensive chemical significance, the 1,2,4-triazole nucleus is also found to be associated with diverse medicinal properties, such as analgesic, anti-asthmatic, diuretic and antifungal activities (Mohamed *et al.*, 1993). The title compound, (I), is shown in Fig. 1. Its bond lengths and angles are normal (Table 1) and agree with those in known structures (Puviarasan *et al.*, 1999; Liu *et al.*, 1999; Zhu *et al.*, 2000).



In (I), two benzene rings are planar within experimental error and are nearly parallel, with a dihedral angle of $8.5(1)^\circ$. Atom F1 lies $0.051(1) \text{ \AA}$ out of the C10–C15 ring plane. The 1,2,4-triazole ring is also planar. The maximum deviation from planarity is $0.009(1) \text{ \AA}$ for atom N1. The dihedral angles between the triazole ring and the C1–C6 and C10–C15 rings are $34.55(1)$ and $42.91(1)^\circ$, respectively.

In the crystal structure, the molecules are linked by intermolecular $\text{N}-\text{H}\cdots\text{O}$ and $\text{N}-\text{H}\cdots\text{N}$ contacts (Table 2), resulting in an infinite network structure (Fig. 2).

Experimental

The corresponding Schiff base (2.82 g, 0.01 mol) was dissolved in 40 ml of dry diglyme with gentle heating and a solution of NaBH_4 (0.003 mol) in 30 ml of dry diglyme was slowly added to the solution with constant stirring. The mixture was then allowed to cool. In order to precipitate the product, 300 ml of water was added to the solution and the mixture was allowed to stand overnight at 273–278 K. The precipitate was filtered off and washed with cold water. After drying *in vacuo*, the solid product was recrystallized from ethanol to afford the

Received 24 April 2003

Accepted 7 May 2003

Online 16 May 2003

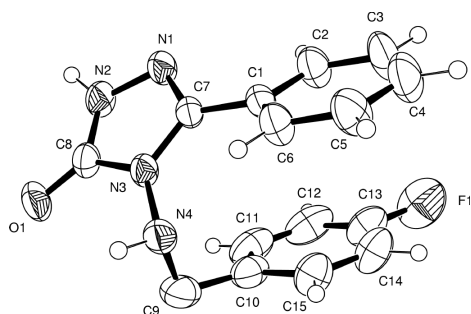


Figure 1
The structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme.

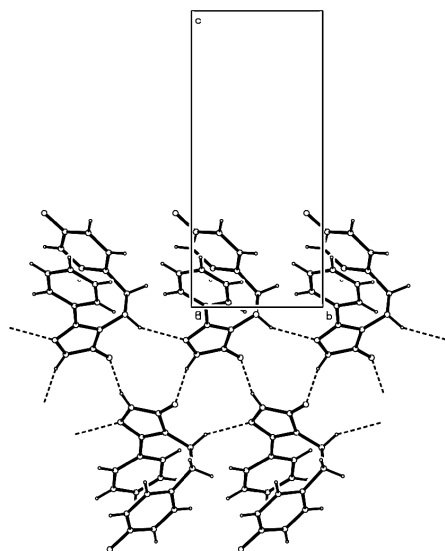


Figure 2
A packing diagram of (I), illustrating the hydrogen-bonding network.

desired compound (1.46 g, 51%). Calculated: C 63.37, H 4.61, N 19.71%; found: C 63.31, H 5.00, N 19.64%. IR data (KBr cm^{-1}): 3290, 3090 (N–H), 1710 (C=O), 1510 (C=N), 830, 740, 670 (aromatic). ^1H NMR (CDCl_3 , δ p.p.m.): 4.18 (*d*, CH_2), 5.22 (*t*, NNH), 10.78 (*s*, NH), 6.80–7.30 (*m*, 3H), 7.40 (*m*, 2H), 7.86 (*m*, 2H).

Crystal data

$\text{C}_{15}\text{H}_{13}\text{FN}_4\text{O}$
 $M_r = 284.29$
Monoclinic, $P2_1/c$
 $a = 15.4560$ (19) Å
 $b = 6.2183$ (7) Å
 $c = 14.8165$ (18) Å
 $\beta = 108.347$ (2)°
 $V = 1351.6$ (3) Å³
 $Z = 4$

$D_x = 1.397$ Mg m⁻³
Mo $K\alpha$ radiation
Cell parameters from 7012 reflections
 $\theta = 2.8$ – 18.9°
 $\mu = 0.10$ mm⁻¹
 $T = 293$ (2) K
Prismatic, colourless
 $0.42 \times 0.17 \times 0.16$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
 φ and ω scans
Absorption correction: none
7084 measured reflections
2992 independent reflections

1141 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.052$
 $\theta_{\text{max}} = 28.1^\circ$
 $h = -20 \rightarrow 15$
 $k = -8 \rightarrow 7$
 $l = -18 \rightarrow 19$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.043$
 $wR(F^2) = 0.089$
 $S = 0.75$
2992 reflections
198 parameters

H atoms treated by a mixture of independent and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.0243P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.17$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.15$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

C7–N1	1.305 (2)	N2–C8	1.342 (3)
C7–N3	1.369 (2)	N2–N1	1.390 (2)
N3–C8	1.384 (2)	F1–C13	1.364 (3)
N3–N4	1.397 (2)	N4–C9	1.474 (3)
O1–C8	1.234 (2)		
N3–C7–C1	125.0 (2)	N3–N4–C9	111.17 (19)
C8–N3–N4	124.57 (18)	N4–C9–C10	109.5 (2)

Table 2

Hydrogen-bonding geometry (Å, °).

$D\cdots H\cdots A$	$D\cdots H$	$H\cdots A$	$D\cdots A$	$D\cdots H\cdots A$
N2–H2N \cdots O1 ⁱ	0.90 (2)	1.89 (2)	2.768 (2)	164 (2)
N4–H4N \cdots N1 ⁱⁱ	0.94 (2)	2.28 (2)	3.153 (3)	154 (2)

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

Only H atoms attached to N were located in a difference Fourier map and refined isotropically. Other H atoms were positioned geometrically and refined using a riding model, fixing the aromatic CH C–H distance at 0.93 Å and the CH₂ C–H distance at 0.97 Å.

Data collection: SMART (Bruker, 1998); cell refinement: SMART; data reduction: SAINT (Bruker, 1998); 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).

References

- Burnett, M. N. & Johnson, C. K. (1996). ORTEPIII. Report ORNL-6895. Oak Ridge National Laboratory, Tennessee, USA.
Bruker (1998). SMART and SAINT. Bruker AXS Inc., Madison, Wisconsin, USA.
Desenko, S. M. (1995). *Khim. Geterotsikl. Soed.* pp. 2–24.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Liu, Y. F., Chantrapromma, S., Shanmuga Sundara Raj, S., Fun, H.-K., Zhang, Y. H., Xie, F. X., Tian, Y. P. & Ni, Y. Y. D. (1999). *Acta Cryst.* **C55**, 93–94.
Mohamed, E. A., El-Deen, I. M., Ismail, M. M. & Mohamed, S. M. (1993). *Indian J. Chem. Sect. B*, **32**, 933–937.
Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
Puviarasan, K., Govindasamy, L., Shanmuga Sundara Raj, S., Velmurugan, D., Jayanthi, G. & Fun, H.-K. (1999). *Acta Cryst.* **C55**, 951–953.
Sheldrick, G. M. (1997). SHELXS97 and SHELXL97. University of Göttingen, Germany.
Zhu, D. R., Xu, Y., Liu, Y.-J., Song, Y., Zhang, Y. & You X.-Z. (2000). *Acta Cryst.* **C56**, 242–243.