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Key indicators

Single-crystal X-ray study
 $T = 293\text{ K}$
 Mean $\sigma(\text{C}-\text{C}) = 0.006\text{ \AA}$
 R factor = 0.050
 wR factor = 0.075
 Data-to-parameter ratio = 14.5

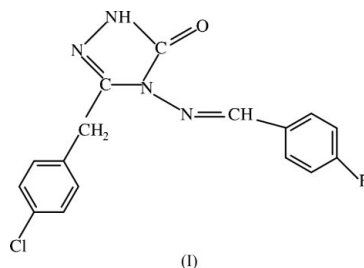
For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

3-(*p*-Chlorobenzyl)-4-(*p*-fluorobenzylidene-amino)-4,5-dihydro-1*H*-1,2,4-triazol-5-one

The title molecule, $\text{C}_{16}\text{H}_{12}\text{ClFN}_4\text{O}$, contains three planar rings. The molecules are linked by an intermolecular hydrogen bond. The *p*-chlorophenyl and *p*-fluorophenyl rings form dihedral angles of $61.2(4)$ and $12.38(10)^\circ$, respectively, with the triazole ring.

Comment

Triazole rings are typically planar 6π -electron partially aromatic systems, possessing an extensive chemistry (Kroger *et al.*, 1965; Temple, 1981). 1,2,4-Triazole and its derivatives are starting materials for the synthesis of many heterocycles (Milcent & Redeuilh, 1979; Milcent & Vicart, 1983). Compounds containing these systems have been structurally characterized as part of a study of Schiff bases of amine- and thione-substituted triazoles and their metal complexes (McCarrick *et al.*, 1999; Clark *et al.*, 1999). In addition to its extensive chemical significance, the 1,2,4-triazole nucleus is also found to be associated with diverse pharmacological properties, such as analgesic, antiasthmatic, diuretic, anti-inflammatory, fungicidal, bactericidal and pesticidal activities (Mohamed *et al.*, 1993; Grammaticakis & Champetier, 1970). Therefore, the crystal-structure determination of the title compound, (I), was carried out.



The title molecule (Fig. 1) contains three rings, a triazole ring (ring *A*; atoms N1/N2/C8/N3/C9) and two benzene rings [rings *B* (C1–C6) and *C* (C11–C16)]. The bond lengths and angles in (I) are found to be normal (Table 1). In the structure, the benzylidenamino group is almost coplanar with the triazole ring: $\text{O1}-\text{C9}-\text{N3}-\text{N4} = -1.4(5)^\circ$, $\text{N3}-\text{N4}-\text{C10}-\text{C11} = -178.1(3)^\circ$ and $\text{C8}-\text{N3}-\text{N4}-\text{C10} = -177.1(3)^\circ$. The dihedral angle between the triazole ring and benzene ring *C* is $12.38(10)^\circ$. The benzene ring *B* forms a dihedral angle of $61.2(4)^\circ$ with ring *C*. A view of the molecular packing is shown in Fig. 2.

Experimental

The corresponding amino compound, 3-*p*-chlorobenzyl-4-amino-4,5-dihydro-1,2,4-triazol-5-one (0.01 mol), was heated in an oil bath with *p*-fluorobenzaldehyde (0.01 mol, 1.05 ml) at 443–448 K for 1 h and

Received 9 January 2004
 Accepted 22 March 2004
 Online 27 March 2004

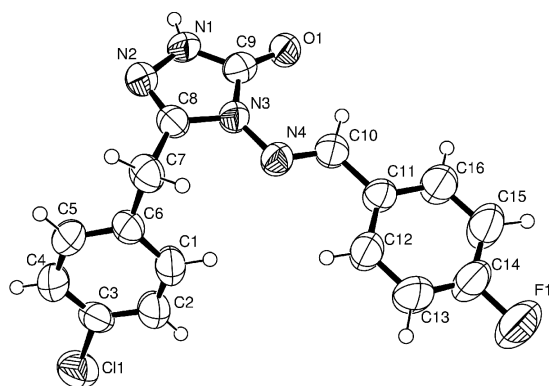


Figure 1

An *ORTEP*-3 (Farrugia, 1997) plot of (I), showing the atom-numbering scheme. Displacement ellipsoids are shown at the 50% probability level.

then allowed to cool. The solid product was recrystallized from ethanol to give the desired compound (m.p. 487–488 K). Elemental analysis calculated for $C_{16}H_{12}ClFN_4O$: C 58.10, H 3.66, N 16.94%; found: C 58.23, H 3.93, N 16.80%. Characteristic 1H NMR peaks ($COCl_2$): 4.06 (s, 2H, CH_2), 9.78 (s, 1H, NH), 7.10 (m, 6H, ArH), 7.74 (m, 2H, ArH).

Crystal data

$C_{16}H_{12}ClFN_4O$
 $M_r = 330.75$
 Monoclinic, $P2_1/c$
 $a = 15.219$ (2) Å
 $b = 4.7796$ (4) Å
 $c = 21.567$ (3) Å
 $\beta = 103.900$ (12)°
 $V = 1522.9$ (3) Å³
 $Z = 4$

$D_x = 1.443$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 3765 reflections
 $\theta = 1.4$ – 21.8°
 $\mu = 0.27$ mm⁻¹
 $T = 293$ (2) K
 Plate, colourless
 $0.80 \times 0.30 \times 0.03$ mm

Data collection

Stoe IPDS-2 diffractometer
 ω scans
 Absorption correction: by integration (*X-RED32*; Stoe & Cie, 2002)
 $T_{min} = 0.907$, $T_{max} = 0.992$
 17 224 measured reflections

2839 independent reflections
 959 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.146$
 $\theta_{max} = 25.5^\circ$
 $h = -18 \rightarrow 18$
 $k = -5 \rightarrow 5$
 $l = -26 \rightarrow 26$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.050$
 $wR(F^2) = 0.075$
 $S = 0.84$
 2839 reflections
 196 parameters

H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2)]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} < 0.001$
 $\Delta\rho_{max} = 0.40$ e Å⁻³
 $\Delta\rho_{min} = -0.23$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

Cl1—C3	1.748 (4)	N3—N4	1.377 (4)
F1—C14	1.362 (5)	N3—C8	1.381 (4)
O1—C9	1.249 (4)	N3—C9	1.391 (4)
N1—C9	1.335 (4)	N4—C10	1.265 (4)
N1—N2	1.389 (3)	C6—C7	1.518 (5)
N2—C8	1.282 (4)	C10—C11	1.460 (5)
N4—N3—C8	121.8 (3)	N2—C8—C7	125.0 (4)
N4—N3—C9	131.0 (3)	N3—C8—C7	122.8 (4)
C10—N4—N3	118.9 (3)	O1—C9—N1	129.4 (4)
C8—C7—C6	112.5 (3)	O1—C9—N3	127.4 (4)

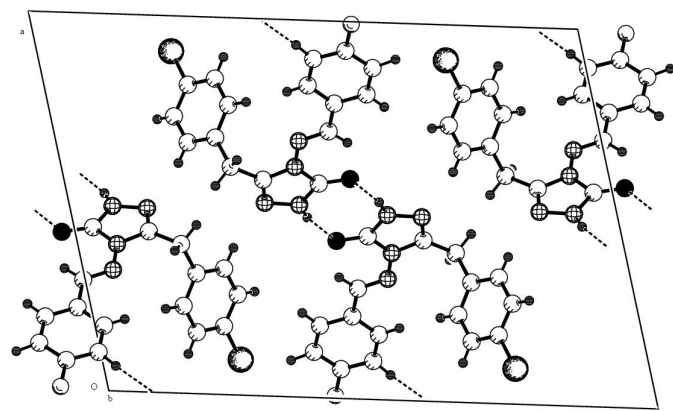


Figure 2

A view, down the b axis, of the packing. Hydrogen bonds are shown as dashed lines.

Table 2

Hydrogen-bonding geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$N1-H1A\cdots O1^i$	0.86	1.96	2.795 (4)	164

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

H atoms were positioned geometrically and treated using a riding model, with an N—H distance of 0.86 Å for atom N1 and C—H distances of 0.93 Å for the phenyl atoms and atom C10, and 0.97 Å for atom C7. The displacement parameters of the H atoms were included as $U_{iso}(H) = 1.2U_{eq}(\text{carrier atom})$. The reflections were very weak due to the thinness of the crystal. No further precaution was available to increase the intensities.

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 1990); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

References

- Clark, R. W., Squattrito, P. J., Sen, A. K. & Dubey, S. N. (1999). *Inorg Chim. Acta*, **293**, 61.
 Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
 Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
 Grammaticakis, M. P. & Champetier, M. G. (1970). *C. R. Acad. Sci. Paris*, **271**, 940.
 Kroger, C. F., Hummel, L., Mutscher, M. & Bayer, H. (1965). *Chem. Ber.* **98**, 3025.
 McCarrick, R. M., Squattrito, P. J., Singh, R. N., Honda, R. N. & Dubey, S. N. (1999). *Acta Cryst.* **C55**, 2111–2114.
 Milcent, R. & Redeuilh, C. J. (1979). *Heterocycl. Chem.* **16**, 403.
 Milcent, R. & Vicart, P. (1983). *Eur. J. Med. Chem.-Chim. Ther.* **18**, 215.
 Mohamed, E. A., El-Deen, I. M., İsmail, M. M. & Mohamed, S. M. (1993). *Indian J. Chem. Sect. B*, **32**, 933–937.
 Sheldrick, G. M. (1990). *SHELXS86*. University of Göttingen, Germany.
 Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.
 Spek, A. L. (1990). *PLATON*. University of Utrecht, The Netherlands.
 Stoe & Cie (2002). *X-AREA* and *X-RED32*. Stoe & Cie, Darmstadt, Germany.
 Temple, C. (1981). 1,2,4-Triazoles, in *The Chemistry of the Heterocyclic Compounds*, Vol. 34. New York: Wiley Interscience.