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4-(*m*-Chlorobenzylamino)-3-phenyl-4,5-dihydro-1*H*-1,2,4-triazol-5-one

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The conformation of the title compound, $C_{15}H_{13}ClN_4O$, can be described in terms of four essentially planar fragments. The triazole ring is planar within 0.008 Å, and its plane, while being almost parallel to the benzene plane of the benzylamino group [dihedral angle 2.95 (14)°], forms a dihedral angle of 16.43 (12)° with the plane of its 3-phenyl substituent. The four-atom bridge (triazole)N—N(H)—C(H$_2$)—C(C$_6$H$_4$Cl), linking the triazole moiety with the chlorophenyl group, is also almost planar, the N—N—C—C torsion angle being −178.82 (13)°. Its mean plane is approximately normal to the triazole and chlorophenyl planes [dihedral angles 107.08 (10) and 108.81 (10)°, respectively]. Two independent N—H···O bonds link the molecules into infinite chains running along the *b* axis of the crystal.

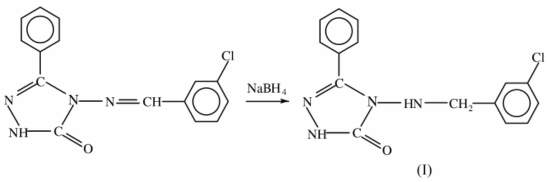
Key indicators

Single-crystal X-ray study
T = 293 K
Mean *σ*(C–C) = 0.003 Å
R factor = 0.039
wR factor = 0.126
Data-to-parameter ratio = 9.6

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

Comment

1,2,4-Triazole rings are typically planar 6*π*-electron aromatic systems, featuring an extensive chemistry (Temple, 1981; Benson, 1967). Detailed studies have been carried out on substituted 1,2,4-triazole derivatives (Cornelissen *et al.*, 1992; Kunkeler *et al.*, 1996; Chinnakali *et al.*, 1999; Fun *et al.*, 1999; Kumaran *et al.*, 1999). Their findings indicate that the 1,2,4-triazole moiety is associated with diverse pharmacological activities, such as analgesic, anti-asthmatic, diuretic, antifungal, antibacterial, pesticidal and anti-inflammatory (Bennur *et al.*, 1976; Heubach *et al.*, 1980; Sharma & Babel, 1982; Mohamed *et al.*, 1993). Furthermore, some of the complexes containing 1,2,4-triazole ligands have rather peculiar structures and specific magnetic properties (Vreugdenhil *et al.*, 1987; Albada *et al.*, 1984; Vos *et al.*, 1983; Kahn & Martinez, 1998). Taking into account the importance of the 1,2,4-triazoles, we have undertaken the X-ray diffraction study of the title compound, (I), a new triazole derivative with a benzylamino substituent.



(I)

The conformation of the molecule of (I) (Fig. 1) can be described in terms of four essentially planar fragments. The triazole ring N2/C8/N3/N4/C9 is planar within 0.008 Å, and its plane, while being almost parallel to the plane of benzene ring C1–C6 [dihedral angle 2.95 (14)°], forms a small dihedral angle of 16.43 (12)° with the plane of the triazole 3-phenyl substituent C10–C15. The aminomethylene bridge linking the

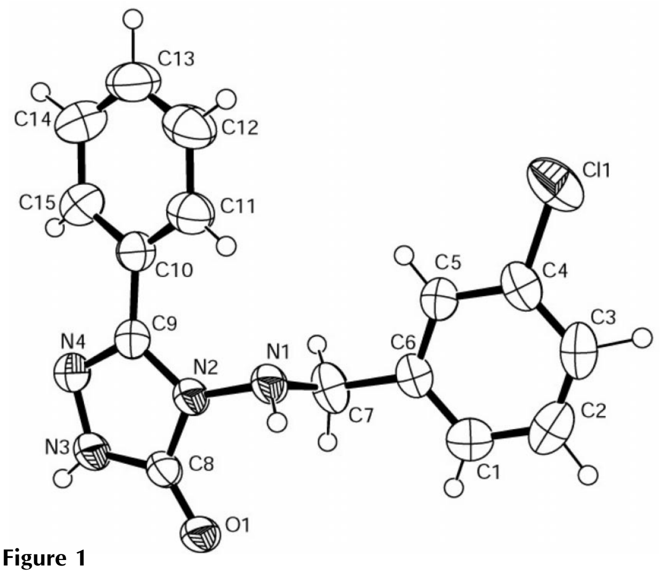


Figure 1
An *ORTEP*-3 (Farrugia, 1997) drawing of the title compound, (I), showing the atomic numbering scheme. Displacement ellipsoids of non-H atoms are drawn at the 50% probability level.

triazole moiety with the chlorophenyl group is also almost planar, the N2—N1—C7—C6 torsion angle being −178.82 (13)°. Its N2/N1/C7/C6 mean plane is approximately normal to both the triazole and chlorophenyl planes [dihedral angles 107.08 (10) and 108.81 (10)°, respectively].

There are two independent N—H···O hydrogen bonds in the structure, involving two 'active' H atoms H7 and H8 [N1—O1^{i} 2.981 (15) Å and N3—O1^{ii} 2.832 (17) Å; symmetry codes: (i) −*x*, 1 − *y*, −*z*; (ii) −*x*, −*y*, −*z*] (Table 2). Each of the two hydrogen bonds is responsible for the formation of a typical hydrogen-bonded centrosymmetric motif, with the carbonyl atom O1 acting as a single acceptor for both hydrogen bonds. Such an arrangement results in the formation of infinite chains running along the *b* axis of the crystal (Fig. 2).

Experimental

The Schiff base shown in the Scheme (Kahveci & İkizler, 2000) (2.99 g, 0.01 mol) was dissolved in 40 ml of dry diglyme with gentle heating and a solution of $NaBH_4$ (0.03 mol) in 30 ml of dry diglyme was slowly added with constant stirring. After the mixture was refluxed for 8 h, it was allowed to cool. To precipitate the product, 300 ml of water was added and the mixture was allowed to stand overnight at 273–278 K. The precipitate was filtered and washed with cold water. After drying *in vacuo*, the solid product was recrystallized from an ethanol/water mixture to afford the desired compound (1.50 g, 47%). M.p: 446–447 K.

Crystal data

$C_{15}H_{13}ClN_4O$
M_r = 300.74
Triclinic, $P\bar{1}$
a = 7.1819 (10) Å
b = 7.4204 (9) Å
c = 14.3295 (19) Å
α = 88.224 (10)°
β = 80.974 (11)°
γ = 72.945 (10)°
V = 720.94 (16) Å^3
Z = 2
D_x = 1.385 Mg m^{-3}
Mo *K*α radiation
Cell parameters from 6723 reflections
θ = 2.7–29.5°
μ = 0.27 mm^{-1}
T = 293 (2) K
Rectangular, colourless
0.80 × 0.48 × 0.25 mm

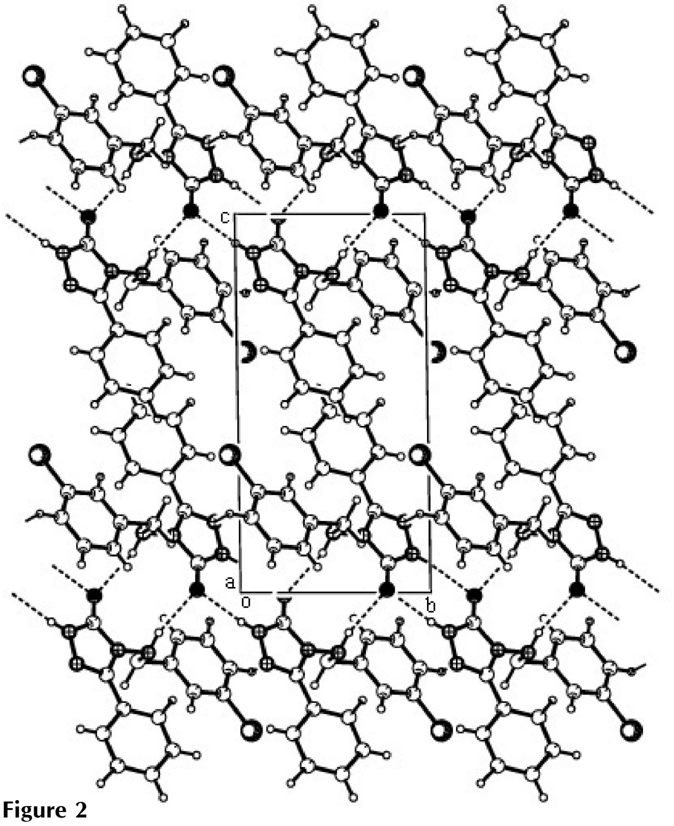


Figure 2
PLATON plot (Spek, 1997) of the crystal packing of (I), viewed down the *a* axis and showing hydrogen-bonded infinite chains running along the *b* axis.

Data collection

Stoe IPDS 2 diffractometer
φ scans
Absorption correction: none
2327 measured reflections
2327 independent reflections
1975 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.044
$\theta_{\rm max}$ = 24.8°
h = −8 → 8
k = −8 → 8
l = 0 → 16

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.126$
S = 1.07
2327 reflections
242 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.1P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max}$ = 0.18 e Å^{-3}
$\Delta\rho_{\rm min}$ = −0.27 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cl1—C4	1.736 (2)	N3—C8	1.332 (2)
O1—C8	1.2381 (19)	N3—N4	1.374 (2)
N1—N2	1.4054 (17)	N4—C9	1.299 (2)
N1—C7	1.472 (2)	C6—C7	1.504 (2)
N2—C8	1.380 (2)	C9—C10	1.467 (2)
N2—C9	1.381 (2)		
N2—N1—C7	110.79 (13)	C5—C4—Cl1	119.05 (15)
C8—N2—C9	108.47 (12)	N1—C7—C6	109.07 (15)
C8—N2—N1	124.60 (12)	O1—C8—N3	129.87 (16)
C9—N2—N1	126.84 (13)	O1—C8—N2	126.78 (13)
C8—N3—N4	113.12 (15)	N3—C8—N2	103.34 (13)
C9—N4—N3	105.32 (13)	N4—C9—N2	109.72 (14)
C3—C4—C5	121.41 (18)	N4—C9—C10	122.54 (14)
C3—C4—Cl1	119.49 (15)	N2—C9—C10	127.70 (14)

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Table 2

Hydrogen-bonding geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H7···O1^{i}	0.850 (19)	2.15 (2)	2.981 (2)	164 (2)
N3—H8···O1^{ii}	0.79 (2)	2.06 (2)	2.832 (2)	167 (2)

Symmetry codes: (i) $-x, -1-y, 2-z$; (ii) $-x, -2-y, 2-z$.

The H atoms were located in a difference map and refined isotropically (N—H = 0.79–0.85 Å and C—H = 0.87–1.04 Å).

Data collection: *X-AREA* (Stoe & Cie, 2001); cell refinement: *X-AREA*; data reduction: *X-RED* (Stoe & Cie, 2001); program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1986); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 *for Windows* (Farrugia, 1997) and *PLATON* (Spek, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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