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4-(*m*-Chlorobenzylamino)-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) = 0.005 Å
R factor = 0.062
wR factor = 0.205
Data-to-parameter ratio = 22.2

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

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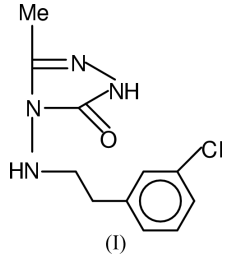
The title compound, $C_{10}H_{11}ClN_4O$, displays the characteristic features of 1,2,4-triazole derivatives. The crystal structure is stabilized by N—H···O intermolecular hydrogen bonds.

Comment

In recent years, various 1,2,4-triazoles and 4,5-dihydro-1*H*-1,2,4-triazol-5-ones have been found to exhibit pharmacological activities. In this connection, several articles have been devoted to the synthesis and biological activities of some 4-arylidenamino-4,5-dihydro-1*H*-1,2,4-triazol-5-ones (Kahveci & Ikizler, 2000*a*,*b*).



(I)

An *ORTEP*III (Burnett & Johnson, 1996) plot of the title structure, (I), is shown in Fig. 1. All bond distances and angles are normal (Table 1) and agree with those in 1,2,4-triazole derivatives (Ocak *et al.*, 2003; Puviarasan *et al.*, 1999; Işık *et al.*, 2003; Wang *et al.*, 1998; Liu *et al.*, 1999).

The 1,2,4-triazole ring is planar, the maximum deviation from planarity being −0.010 (2) Å for atom C10. The dihedral angle between the triazole ring and the six-membered ring is 56.00 (9)°.

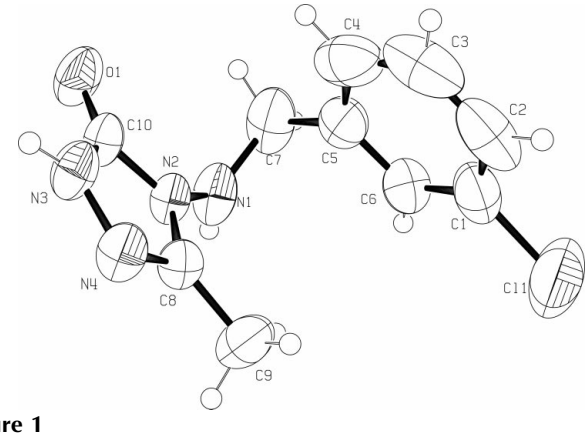


Figure 1
An *ORTEP*III (Burnett & Johnson, 1996) drawing of the title compound, showing the atomic numbering scheme. Displacement ellipsoids of non-H atoms are shown at the 50% probability level.

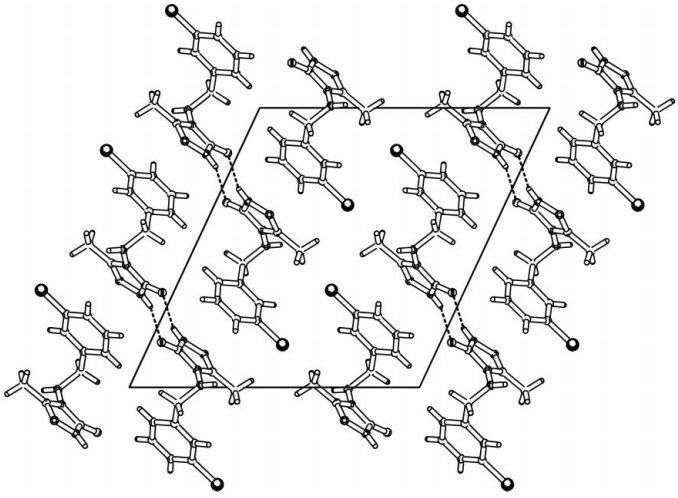


Figure 2
PLATON/*PLUTON* plot (Spek, 1997) of the crystal packing, viewed down the *b* axis.

In the crystal structure, the molecules are linked by N—H···O intermolecular hydrogen bonds (Table 2) and form chains along the *b* axis (Fig. 2).

Experimental

The corresponding Schiff base, 3-methyl-4-(*m*-chlorobenzalimine)-4,5-dihydro-1*H*-1,2,4-triazol-5-one (2.37 g, 0.01 mol) in warm dry diglyme (40 ml) was added to a solution of $NaBH_4$ (0.03 mol) in dry diglyme (30 ml). The mixture was refluxed for 8 h. After cooling, 200 ml of water was added to the medium and the mixture allowed to stand overnight at 273–278 K. The resulting precipitate was filtered off and washed with cold water. After drying *in vacuo*, the product was recrystallized from water to give (I) (1.21 g, 51%). Calculated: C 50.32, H 4.65, N 23.47%; found: C 49.87, H 5.01, N 23.17%. M.p: 443–444 K. IR (cm^{-1}): ν_{NH} 3280 and 3090, $\nu_{C=O}$ 1680, $\nu_{C=N}$ 1600, $\nu_{aromatic}$ 790 and 740; ^1H NMR ($CDCl_3$, p.p.m.): δ 1.96 (*s*, CH_3), 4.18 (*d*, CH_2), 4.80 (*t*, NNH), 10.40 (*s*, NH), 7.20–7.60 (*m*, 4H).

Crystal data

$C_{10}H_{11}ClN_4O$
M_r = 238.68
Monoclinic, $P2_1/c$
a = 14.3423 (15) Å
b = 5.8708 (3) Å
c = 15.2730 (14) Å
β = 114.963 (7)°
V = 1165.86 (17) Å^3
Z = 4
D_x = 1.360 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 14 391 reflections
θ = 1.7–29.3°
μ = 0.31 mm^{-1}
T = 293 (2) K
Prism, colourless
0.60 × 0.46 × 0.28 mm

Data collection

Stoe IPDS–II diffractometer
ω scans
Absorption correction: by integration (*X-RED*32; Stoe & Cie, 2002)
T_{min} = 0.839, T_{max} = 0.923
14 391 measured reflections
3218 independent reflections
1519 reflections with $I > 2\sigma(I)$
R_{int} = 0.087
θ_{max} = 29.4°
$h = -19 \rightarrow 19$
$k = -7 \rightarrow 8$
$l = -20 \rightarrow 21$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.063
$wR(F^2)$ = 0.205
S = 0.86
3218 reflections
145 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1356P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.51 e Å^{-3}
$\Delta\rho_{min}$ = −0.43 e Å^{-3}

Table 1
Selected interatomic distances (Å).

O1—C10	1.234 (3)	N2—C8	1.356 (3)
C10—N3	1.330 (3)	N2—N1	1.392 (2)
C10—N2	1.379 (3)	N4—C8	1.293 (3)
N3—N4	1.375 (3)	C8—C9	1.480 (3)
C1—Cl1	1.746 (3)		

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3A···O1^{i}	0.86	1.94	2.752 (2)	158

Symmetry code: (i) $2-x, y-\frac{1}{2}, \frac{5}{2}-z$.

H atoms were positioned geometrically and refined using a riding model, fixing the aromatic C—H distances at 0.93 Å, methylene C—H distances at 0.97 Å, methyl C—H distances at 0.96 Å and N—H distances at 0.86 Å. U_{iso}(H) values were calculated as $1.5U_{eq}$ (methyl group) of the parent atom.

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED*32 (Stoe & Cie, 2002); 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, 1999), *PARST* (Nardelli, 1995) and *PLATON* (Spek, 1997).

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