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4-Amino-3-*p*-chlorobenzyl-4,5-dihydro-1,2,4-triazol-5-one

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.037
wR factor = 0.115
Data-to-parameter ratio = 15.6

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

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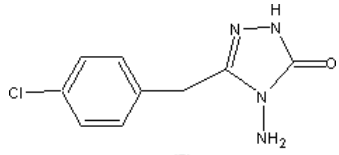
The title compound, $C_9H_9ClN_4O$, possesses a triazole ring, which is a typically planar six-π-electron partially aromatic system, and displays the characteristic features of a 1,2,4-triazole derivative. In the crystal structure, symmetry-related molecules are linked by intermolecular N—H···O and N—H···N hydrogen bonds.

Comment

Triazole ring systems are typically planar six-π-electron partially aromatic systems, and 1,2,4-triazole and its derivatives are used as starting materials for the synthesis of many heterocycles (Desenko, 1995). In addition to having extensive chemical significance (Benson, 1967; Temle, 1981), the 1,2,4-triazole nucleus is also associated with diverse pharmacological properties, such as analgesic, anti-asthmatic, diuretic, fungicidal, bactericidal, anti-inflammatory and pesticidal activities (Bennur *et al.*, 1976; Webb & Parsons, 1997; Heubach *et al.*, 1980; Mohammed *et al.*, 1993; Yüksek, 1992).

The crystal structure of the title compound, (I), is illustrated in Fig. 1 and selected bond distances are given in Table 1. The dihedral angle between the planes of the triazole and benzene rings is 65.92 (6)°. The maximum deviations from planarity are 0.0096 (9) Å for atom N2 in ring C8—N3 and 0.0078 (10) Å for atom C1 in ring C1—C6.



(I)

The chloro and triazole linkages distort the C—C bond lengths of the benzene ring, giving bond lengths in the range 1.367 (3)–1.383 (2) Å. The Cl1—C4 bond distance

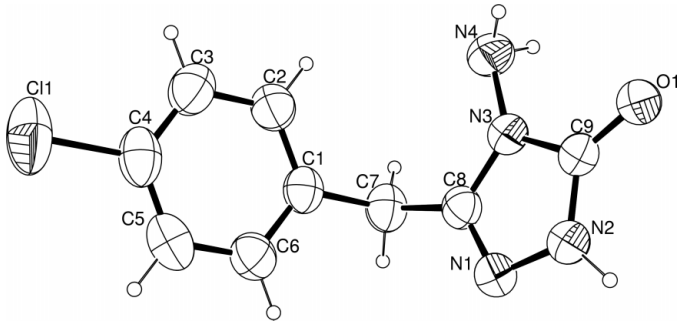


Figure 1
A view of (I), with the atom-numbering scheme and 50% probability displacement ellipsoids.

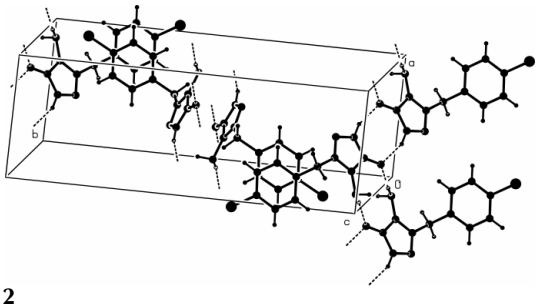


Figure 2
A packing diagram of (I), viewed approximately along the *c* axis, with the hydrogen bonds shown as dashed lines.

[1.744 (2) Å] is similar to the distance [1.735 (3) Å] found in another compound that contains a triazole and a benzene ring (Kumaran *et al.*, 1999). The C8═N1 bond length [1.292 (2) Å] is comparable to those for similar bonds in the literature (Ocak, Çoruh *et al.*, 2003; Puviarasan *et al.*, 1999; Liu *et al.*, 1999; Zhu *et al.*, 2000). The C9═O1 bond length [1.2373 (18) Å] agrees with the C═O bond length [1.234 (3) Å] found in similar structures (Ocak, Kahveci *et al.*, 2003).

In the crystal structure, symmetry-related molecules are linked by N—H···O and N—H···N hydrogen bonds; details are given in Table 2 and Fig. 2.

Experimental

Ethyl-*p*-chlorophenylacetoxycarbonyl hydrazone (2.845 g, 0.01 mol) was dissolved in water (50 ml) with stirring. Hydrazine hydrate (1.25 ml) was added, and the mixture was refluxed for 5 h. The reaction mixture precipitated a product at 273 K over a period of 12 h. The product was filtered off and then recrystallized from ethanol (m.p. 454 K; yield 2 g, 90%).

Crystal data

$C_9H_9ClN_4O$
M_r = 224.65
Monoclinic, $P2_1/c$
a = 7.1316 (6) Å
b = 21.225 (2) Å
c = 7.1164 (6) Å
β = 110.770 (6)°
V = 1007.19 (15) Å^3
Z = 4
D_x = 1.482 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 14 661 reflections
θ = 1.9–27.5°
μ = 0.36 mm^{-1}
T = 293 (2) K
Prism, colourless
0.38 × 0.36 × 0.18 mm

Data collection

Stoe IPDS-II diffractometer
ω scans
Absorption correction: by integration (*X-RED*; Stoe & Cie, 1996)
T_{min} = 0.873, T_{max} = 0.938
13 749 measured reflections
2320 independent reflections
1710 reflections with $I > 2\sigma(I)$
R_{int} = 0.042
θ_{max} = 27.5°
$h = -9 \rightarrow 9$
$k = -27 \rightarrow 27$
$l = -9 \rightarrow 9$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.115
S = 1.03
2320 reflections
149 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0752P)^2 + 0.0064P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.047 (6)

Table 1
Selected interatomic distances (Å).

O1—C9	1.2373 (18)	Cl1—C4	1.7436 (17)
N1—C8	1.292 (2)		

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H22···O1^{i}	0.92 (2)	1.93 (2)	2.8037 (17)	158.3 (18)
N4—H44A···N1^{ii}	0.90 (2)	2.65 (2)	3.327 (2)	133.6 (17)
N4—H44B···O1^{iii}	0.90 (2)	2.09 (2)	2.976 (2)	169 (2)

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

H atoms attached to atoms C2, C3, C5 and C6 were treated using a riding model, with C—H = 0.93 Å and U_{iso}(H) = 1.5U_{eq}(C). Other H atoms were refined isotropically. The calculated C—H bond lengths were in the range 0.93–0.97 Å.

Data collection: *X-AREA* (Stoe & Cie, 1996); cell refinement: *X-AREA*; data reduction: *X-RED* (Stoe & Cie, 1996); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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