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4-Amino-2-(*p*-chlorophenyl)-5-methyl-3,4-dihydro-2*H*-1,2,4-triazol-3-one

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.042
wR factor = 0.117
Data-to-parameter ratio = 14.2

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

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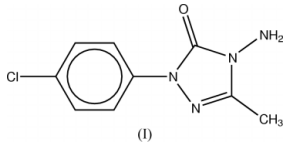
In the title compound, $C_9H_9ClN_4O$, the dihedral angle between the substituted phenyl and triazole rings is 4.86 (5)°. In the crystalline state, the molecules exist as centrosymmetrically related N—H···O hydrogen-bonded dimers.

Comment

Amino-functionalized triazole derivatives serve as starting compounds for heterocyclic syntheses. The triazole moiety possesses many pharmacological properties, *e.g.* antimicrobial (Habib *et al.*, 1997), antiviral (Ergen *et al.*, 1996), anti-HIV-1 (Invidiata *et al.*, 1996), antifungal, antimycobacterial and anticonvulsant (Gülerman *et al.*, 1997). It is also a highly potent eosinophilia inhibitor (Naito *et al.*, 1996) and is used as a fungicide (Crofton, 1996) and a herbicide (Tada *et al.*, 1995). Some triazole derivatives have been evaluated for their antibacterial activity against both Gram-positive and Gram-negative bacteria (Bs *et al.*, 1996). In view of these findings, the structure determination of the title compound, (I), was undertaken.



(I)

A perspective view of (I), including the atomic numbering scheme, is shown in Fig. 1. The bond lengths and angles in (I) are unexceptional (Table 1), and comparable with those reported for related structures (Chen *et al.*, 1998; Wang *et al.*, 1998; Thamotharan, Parthasarathi, Sunagar *et al.*, 2003; Thamotharan, Parthasarathi, Hunnur *et al.*, 2003). The sum of bond angles around N41 is 320°, which indicates a pyramidal geometry at N41. The dihedral angle between the substituted phenyl and triazole rings is 4.86 (5)°, while the corresponding angles in the chloro (Thamotharan, Parthasarathi, Sunagar *et al.*, 2003) and bromo (Thamotharan, Parthasarathi, Hunnur *et al.*, 2003) derivatives of triazole are 30.63 (9) and 8.93 (14)°, respectively. In the crystal structure, centrosymmetrically related molecules form dimers through N—H···O intermolecular hydrogen bonds (Fig. 2) and have a graph-set motif of $R_2^2(10)$ (Table 2) (Bernstein *et al.*, 1995).

Experimental

The title compound was prepared by heating 3-(4-chlorophenyl)-5-methyl-1,3,4-oxadiazolin-2-one with hydrazine hydrate in ethanol. The solid obtained, (I), was crystallized from absolute ethanol (m.p. 458–459 K).

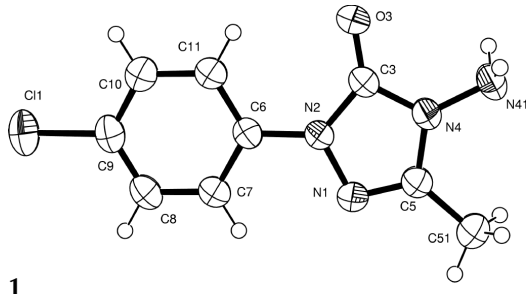


Figure 1
A view of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented by circles of arbitrary radii.

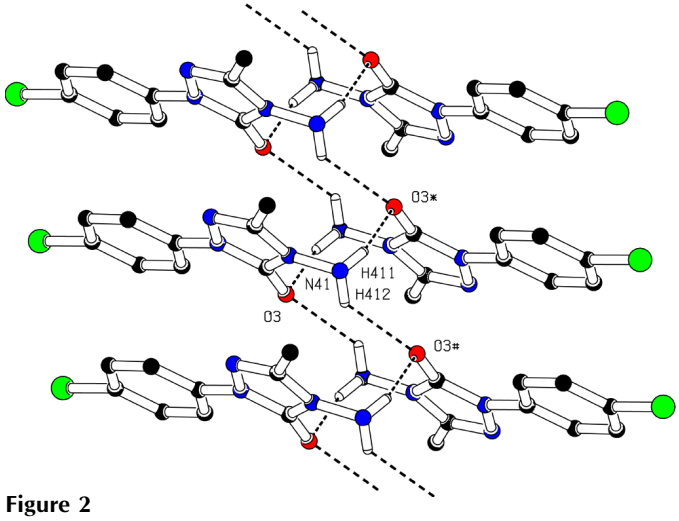


Figure 2
Linkage of molecules into dimers [symmetry codes: (*) $1-x, 2-y, 1-z$; (#) $-x, 2-y, 1-z$]. H atoms bonded to C atoms have been omitted for clarity.

Crystal data

$C_9H_9ClN_4O$
$M_r = 224.65$
Triclinic, $P\overline{1}$
$a = 4.0135\ (8)$ Å
$b = 11.691\ (2)$ Å
$c = 11.986\ (2)$ Å
$\alpha = 117.09\ (3)^\circ$
$\beta = 90.79\ (3)^\circ$
$\gamma = 91.89\ (3)^\circ$
$V = 500.17\ (16)$ Å^3
$Z = 2$
$D_x = 1.492$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4074 reflections
$\theta = 12.2$–27.9°
$\mu = 0.36$ mm^{-1}
$T = 293\ (2)$ K
Platelet, translucent colourless
0.61 × 0.30 × 0.15 mm

Data collection

Stoe IPDS diffractometer
φ scans OK?
Absorption correction: none
4391 measured reflections
2060 independent reflections
1807 reflections with $I > 2\sigma(I)$
$R_{int} = 0.024$
$\theta_{max} = 27.8^\circ$
$h = -4 \rightarrow 4$
$k = -15 \rightarrow 15$
$l = -15 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.042$
$wR(F^2) = 0.117$
$S = 1.06$
2060 reflections
145 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0674P)^2 + 0.0798P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.17$ e Å^{-3}
$\Delta\rho_{min} = -0.17$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—C5	1.3017 (19)	N4—C5	1.3687 (18)
N1—N2	1.4001 (16)	N4—C3	1.3828 (18)
N2—C3	1.3682 (18)		
C5—N1—N2	104.81 (12)	N4—N41—H412	108 (2)
C3—N2—N1	111.97 (11)	H411—N41—H412	106 (2)
C5—N4—C3	109.34 (12)	N2—C3—N4	102.89 (11)
N4—N41—H411	105.8 (16)	N1—C5—N4	110.99 (13)

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

D—H···A	D—H	H···A	D···A	D—H···A
N41—H411···O3^{i}	0.92 (2)	2.17 (2)	3.043 (3)	159 (2)
N41—H412···O3^{ii}	0.84 (3)	2.30 (3)	2.958 (2)	135 (2)

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

The amino H atoms were located from a difference Fourier map and refined freely with individual isotropic displacement parameters. The methyl H atoms were constrained to an ideal geometry (C—H = 0.96 Å), with $U_{iso}(H) = 1.5U_{eq}(C)$, but were allowed to rotate freely about the parent C—C bond. All remaining H atoms were placed in geometrically idealized positions (C—H = 0.93 Å) and constrained to ride on their parent atoms with $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *IPDS Software Package* (Stoe & Cie, 1997); cell refinement: *IPDS Software Package*; data reduction: *IPDS Software Package*; program(s) used to solve structure: *DIRDIF*99 (Beurskens *et al.*, 1999); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 (Farrugia, 1997); software used to prepare material for publication: *SHELXL*97 and *PLATON* (Spek, 2003).

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