Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

3-(6-Chloropyridin-3-ylmethyl)-7-imino-6-isobutyl-6,7-dihydro-3*H*-1,2,3-triazolo[4,5-*d*]pyrimidine

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Received 10 December 2006
Accepted 12 December 2006

Key indicators

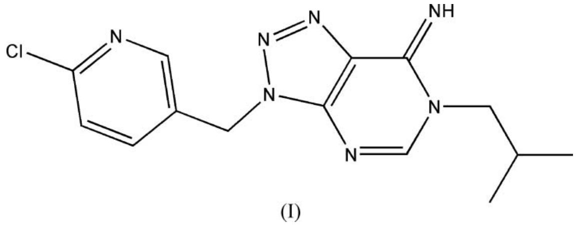
Single-crystal X-ray study
T = 297 K
Mean σ(C–C) = 0.004 Å
R factor = 0.059
wR factor = 0.155
Data-to-parameter ratio = 16.0

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

In the title compound, $C_{14}H_{16}ClN_7$, the triazole ring is almost coplanar with the pyrimidine ring. There is also evidence for significant electron delocalization in the triazolo[4,5-*d*]pyrimidinyl system. Intermolecular C—H···N and N—H···N hydrogen bonds, together with strong π–π stacking interactions, stabilize the crystal structure.

Comment

Neonicotinoids are a promising class of insecticides with excellent chemical and biological properties. 1,2,3-Triazoles have also received much attention because of their wide range of applications and biological activities, for example as anti-HIV and antimicrobial agents (Fan & Katritzky, 1996; Dehne, 1994; Abu-Orabi *et al.*, 1989). We report here the crystal structure of (I) (Fig. 1, Table 1), which was synthesized by introducing a pyridine ring into a 1,2,3-triazolo[4,5-*d*]pyrimidine molecular framework.



(I)

In (I), the C7—N4, C8—N2, C8—N7, C10—N6 and C12—N6 bonds are significantly shorter (Table 1) than a normal single C—N bond (1.47 Å; Sasada, 1984) and closer to the value for a C═N bond (1.28 Å; Wang *et al.*, 1998). This indicates significant electron delocalization in the triazolo[4,5-*d*]pyrimidinyl system. Moreover, the C12═N5 double bond is 1.270 (3) Å, which is shorter than a normal C═N double bond. The C7—C12 bond [1.436 (3) Å] is a single bond, similar to distances of 1.41–1.43 Å found in other heterocyclic compounds (Hanefeld *et al.*, 1996). Atoms C8, N2, N3, N4, C7, N7, C10, N6, C12 and N5 are coplanar, with a maximum deviation of 0.011 (2) Å, for N6, forming a fully delocalized system. The dihedral angle between the triazole and pyrimidine rings is 0.47 (11)°.

Intermolecular C—H···N and N—H···N hydrogen bonds contribute strongly to the stability of the crystal structure (Fig. 2 and Table 2). Strong π–π stacking interactions also occur between adjacent N2–N4/C7/C8 rings and again between the N2–N4/C7/C8 and N6/C10/N7/C8–C12 rings of neighboring molecules. Centroid–centroid distances are 3.400 (1) and 3.734 (1) Å, dihedral angles 0.02 (1) and 0.48 (1)°, and shortest interplanar distances 3.307 and 3.306 Å, respectively.

doi:10.1107/S1600536806053839

Experimental

Isobutylamine (1 mmol) in anhydrous acetonitrile (4 ml) was added dropwise to a solution of (*E*)-ethyl-*N*-3-((6-chloropyridin-3-yl)methyl)-5-cyano-3*H*-1,2,3-triazol-4-ylformimidate (1 mmol) in anhydrous acetonitrile (8 ml) at room temperature. The mixture was stirred until the reaction was complete (monitored by thin-layer chromatography) and the solution concentrated under vacuum. The residue was recrystallized from anhydrous ethanol to give the title compound (yield 88%). Colorless crystals of (I) suitable for X-ray structure analysis were grown from dichloromethane and petroleum ether (1:2, *v*/*v*).

Crystal data

$C_{14}H_{16}ClN_7$
$M_r = 317.79$
Monoclinic, $P2_1/c$
$a = 14.3695$ (18) Å
$b = 13.1510$ (17) Å
$c = 8.3524$ (11) Å
$\beta = 93.392$ (2)°
$V = 1575.6$ (4) Å^3
$Z = 4$
$D_x = 1.340$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.25$ mm^{-1}
$T = 297$ (2) K
Plate, colorless
$0.30 \times 0.20 \times 0.04$ mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2001)
$T_{\min} = 0.929$, $T_{\max} = 0.990$
11112 measured reflections
3273 independent reflections
2439 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.037$
$\theta_{\max} = 26.5°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.059$
$wR(F^2) = 0.155$
$S = 1.05$
3273 reflections
205 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0682P)^2 + 0.4489P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.35$ e Å^{-3}
$\Delta\rho_{\min} = -0.31$ e Å^{-3}

Table 1
Selected bond lengths (Å).

C7—N4	1.358 (3)	C10—N6	1.357 (3)
C7—C12	1.436 (3)	C12—N5	1.270 (3)
C8—N2	1.345 (3)	C12—N6	1.427 (2)
C8—N7	1.368 (3)		

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

D—H⋯*A*	*D*—H	H⋯*A*	*D*⋯*A*	*D*—H⋯*A*
N5—H5*A*⋯N7$^{\rm i}$	0.88 (2)	2.40 (3)	3.238 (3)	161 (2)
C5—H5⋯N5$^{\rm ii}$	0.93	2.62	3.370 (3)	139
C10—H10⋯N4$^{\rm ii}$	0.93	2.48	3.347 (3)	155

Symmetry codes: (i) $-x+2, y-\frac{1}{2}, -z+\frac{1}{2}$; (ii) $-x+2, y+\frac{1}{2}, -z+\frac{1}{2}$.

The imine H atom, H5*A*, was located in a difference Fourier map and was refined freely with an isotropic displacement factor. Other H atoms were placed in calculated positions, with C—H distances in the range 0.93–0.97 Å and refined using a riding-model approximation, with $U_{\rm iso}$(H) = 1.2 or 1.5 times $U_{\rm eq}$(carrier atom). A rotating group model was used for the methyl groups.

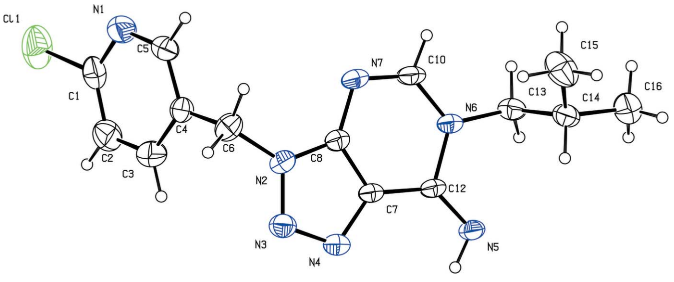


Figure 1
The molecular structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme.

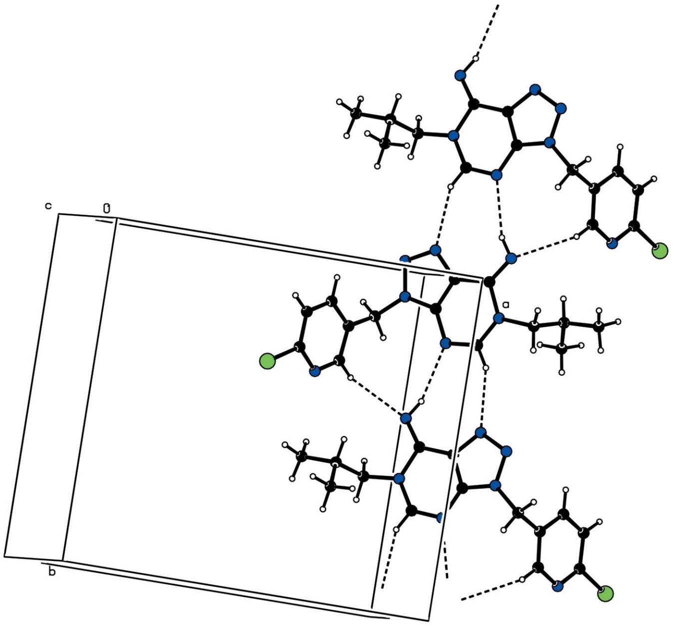


Figure 2
Part of the packing of (I). Hydrogen bonds are shown as dashed lines.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

The authors are grateful to the Natural Science Foundation of China (grant No. 20302002) for financial support.

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