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5,6-Dianilino-3-(4-chlorophenyl)-3,6-dihydro-1,2,3-triazolo[4,5-*d*]pyrimidin-7-one

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In the title compound, $C_{22}H_{16}ClN_7O$, all ring atoms in the triazolopyrimidinone system are almost coplanar. The packing of the molecules in the crystal structure is mainly a result of π–π and N—H···N/O hydrogen-bonding interactions, forming a three-dimensional network.

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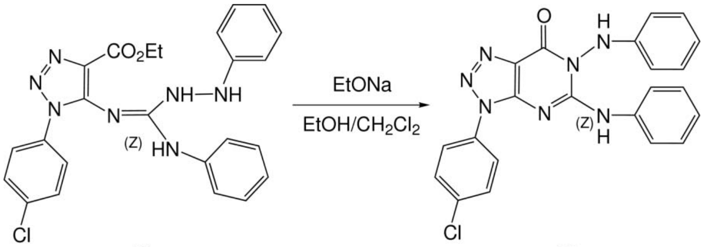
Key indicators

Single-crystal X-ray study
T = 292 K
Mean σ(C–C) = 0.003 Å
R factor = 0.050
wR factor = 0.125
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.

Comment

Among many known fused heterocycles, 8-azaguanine derivatives (1,2,3-triazolo[4,5-*d*]pyrimidin-7-one) have received much attention as a result of their broad spectrum of biological activities (Albert, 1986). We have recently synthesized some 8-azaguanines (Zhao, Xie *et al.*, 2005), but to the best of our knowledge, there is no efficient method for identifying the structure of the title compound, (2), except for X-ray diffraction. Theoretically, the cyclization of the guanidine intermediate may produce three isomers, while we obtained only one of them.



(1) (2)

The structure of (2) is shown in Fig. 1. The bond lengths and angles in the triazolopyrimidinone ring system are in good agreement with those observed for a closely related structure (Zhao, Wang & Ding, 2005). All ring atoms in the 1,2,3-triazolo[4,5-*d*]pyrimidine system are essentially coplanar, while the benzene ring (atoms C1–C6) is rotated by 11.36 (11)° about the C6—N1 bond. X-ray analysis indicates that 1,2,3-triazolo[4,5-*d*] pyrimidine is a conjugated system, in which each C and N atom is sp^2-hybridized, and ten π electrons (three from C atoms and seven from N atoms) characterize an aromatic heterocycle (Li *et al.*, 2004). There are an intramolecular N6—H6···N7 hydrogen bond and a bifurcated intermolecular N7—H7···N3^{i}/O1^{i} hydrogen bond (Table 2 and Fig. 2). There is also π–π interaction between the 4-chlorophenyl ring and its symmetry-related ring at ($1 - x$, $-y$, $1 - z$), the centroid-to-centroid distance being 3.718 Å.

Experimental

Iminophosphorane was reacted with aromatic isocyanates to give carbodiimides, which were allowed to react with primary amines and

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substituted hydrazines to provide isolable guanidine intermediates (Zhao, Xie *et al.*, 2005). The guanidine intermediate (1) (2 mmol) in a mixed solvent (CH_2Cl_2/EtOH, 1:4 *v*/*v*), treated with a catalytic amount of sodium ethoxide, cyclized smoothly at room temperature to provide only the title compound, (2). Single crystals suitable for crystallographic analysis were obtained by slow evaporation of a dichloromethane/ethanol (4:1 *v*/*v*) solution. Yield 85%, m.p. 512 K. MS *m*/*z*(%): 429 (M^+, 46), 309 (35), 172 (49), 77 (100).

Crystal data

$C_{22}H_{16}ClN_7O$
M_r = 429.87
Monoclinic, $P2_1/n$
a = 14.981 (2) Å
b = 7.6973 (12) Å
c = 17.468 (3) Å
β = 90.001 (1)°
V = 2014.3 (5) Å^3
Z = 4

D_x = 1.417 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2384 reflections
θ = 2.9–22.0°
μ = 0.22 mm^{-1}
T = 292 (2) K
Block, colourless
0.30 × 0.20 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: none
12017 measured reflections
4395 independent reflections

2595 reflections with $I > 2\sigma(I)$
R_{int} = 0.063
θ_{max} = 27.0°
$h = -16 \rightarrow 19$
$k = -9 \rightarrow 8$
$l = -22 \rightarrow 22$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.125
S = 0.90
4395 reflections
281 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0602P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.43 e Å^{-3}
$\Delta\rho_{min}$ = −0.37 e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0005 (1)

Table 1
Selected geometric parameters (Å, °).

C7—N4	1.353 (2)	C9—N5	1.421 (2)
C7—N1	1.363 (2)	C10—N4	1.313 (2)
C7—C8	1.373 (3)	C10—N5	1.395 (2)
C8—N3	1.365 (2)	N1—N2	1.386 (2)
C8—C9	1.435 (3)	N2—N3	1.303 (2)
C9—O1	1.212 (2)		
C7—N1—N2	108.98 (15)	N2—N1—C6	120.23 (16)
C7—N1—C6	130.72 (16)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N6—H6···N7	0.86	2.16	2.606 (2)	112
N7—H7···N3^i	0.86	2.38	3.065 (2)	137
N7—H7···O1^i	0.86	2.58	3.098 (2)	120

Symmetry code: (i) $-x + \frac{1}{2}, y + \frac{1}{2}, -z + \frac{3}{2}$.

All H atoms were initially located in difference Fourier maps, and they were then constrained to an ideal geometry with C—H distances of 0.93 Å, N—H distances of 0.86 Å, and U_{iso}(H) = 1.2U_{eq}(parent atom).

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2001); software used to prepare material for publication: *SHELXTL*.

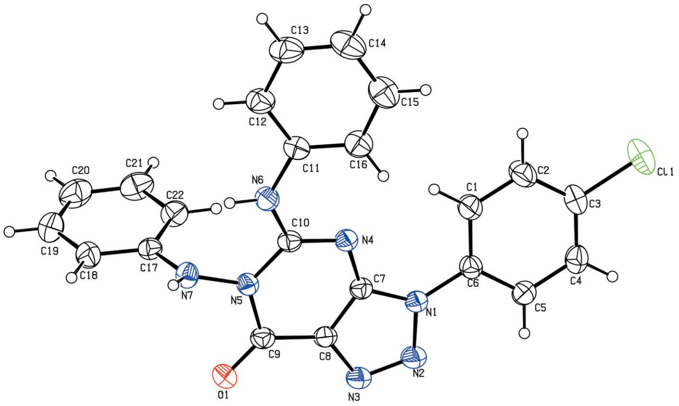

Figure 1
View of the molecule of (2), showing the atom-labelling scheme. Displacement ellipsoids are shown at the 50% probability level.

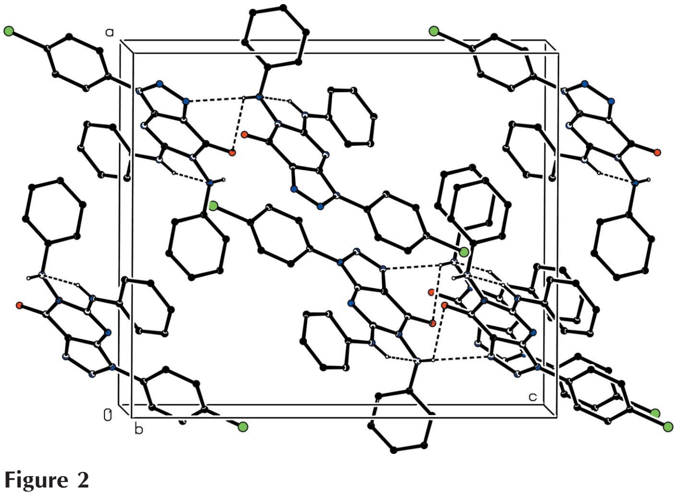

Figure 2
The crystal structure of (2), viewed along the *b* axis. The broken lines indicate hydrogen bonds. H atoms have been omitted unless they are involved in hydrogen bonding.

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