Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

4-(3-Bromobenzylideneamino)-3-(4-chlorobenzyl)-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) = 0.004 Å
R factor = 0.032
wR factor = 0.076
Data-to-parameter ratio = 14.9

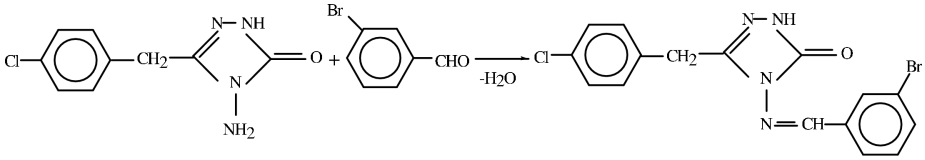
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 13 October 2004
Accepted 20 October 2004
Online 30 October 2004

The title compound, $C_{16}H_{12}BrClN_4O$, contains two benzene rings and a triazole ring which is substituted at the 1,2,4-positions. The crystal structure of (I) is stabilized by N—H···O and π–π stacking interactions.

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 addition, 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*). For these reasons, the structures of substituted 1,2,4-triazole derivatives have been a subject of interest in our laboratory. Examples include 1-acetyl-3-(*p*-chlorobenzyl)-4-(*p*-chlorobenzylidenamino)-4,5-dihydro-1*H*-1,2,4-triazol-5-one (Ocak *et al.*, 2003), 3,5-diphenyl-4-(3,4,5-trimethoxybenzylidenamino)-4*H*-1,2,4-triazole (Atalay *et al.*, 2003), and C—H···O and C—H···π interactions in 1-acetyl-3-ethyl-4,5-dihydro-1*H*-1,2,4-triazol-5-one (Çoruh *et al.*, 2003).



(I)

The molecular structure of the title compound, (I), is illustrated in Fig. 1. It consists of one 1,2,4-triazole ring (*A* C1/N2/N3/C4/N5/C6) and two benzene rings (*B* C8–C13 and *C* C15–C20). The dihedral angles between the planes of the rings are *A*/*B* = 2.83 (9), *A*/*C* = 70.66 (8) and *B*/*C* = 72.84 (8)°.

The N=C and Cl—C bond lengths agree with literature values (Liu *et al.*, 1999; Zhu *et al.*, 2000; Ocak *et al.*, 2003; Çoruh *et al.*, 2003). Similar values as in (I) for the Br—C and N—N bond have been observed in other compounds (Ünver *et al.*, 2000; Puviarasan *et al.*, 1999). Details of bond distances and angles are listed in Table 1.

The crystal structure is stabilized by C—H···O and N—H···O intra- and intermolecular hydrogen bonds (Fig. 2). Furthermore, π–π stacking is observed. Ring *A* stacks with ring *B* $(1-x, 1-y, 1-z)$, with a distance of 3.703 (2) Å between the ring centroids. There is similar π–π stacking involving ring *B* and ring *B* at $(1-x, -y, 1-z)$, with a distance of 3.931 (16) Å. A third π–π stacking is between ring *C* and ring *C* at $(1-x, 1-y, -z)$, with a distance of 3.931 (2) Å between the ring centroids.

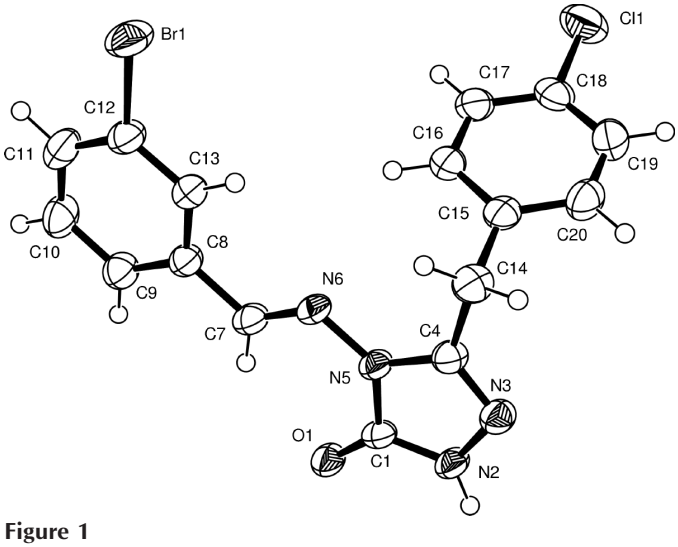


Figure 1
A view of (I) with the atom-numbering scheme and 50% probability displacement ellipsoids.

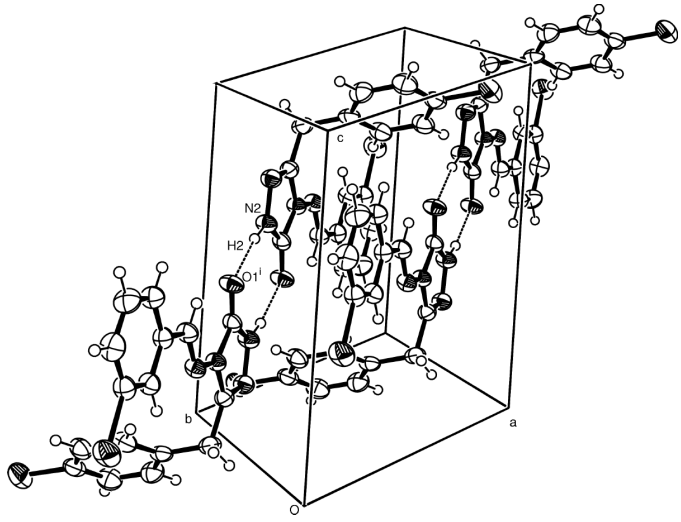


Figure 2
The hydrogen bonding (dashed lines) observed in the title compound.

Experimental

3-*p*-Chlorobenzyl-4-amino-4,5-dihydro-1*H*-1,2,4-triazol-5-one (2.25 g, 0.01 mol) was heated in an oil bath with 3-bromobenzaldehyde (1.16 ml, 0.01 mol) at 433–443 K for 1 h and then allowed to cool. The solid product was recrystallized from DMSO–water (1:5) to give the title compound, (I) (yield: 3.21 g, 82%; m.p. 513–514 K). Elemental analysis calculated for $C_{16}H_{12}BrClN_4O$: C 49.10, H 3.09, N 14.31%; found: C 48.84, H 3.06, N 14.34%. ^{1}H NMR (DMSO-d_6): δ 4.12 (*s*, CH_2, 2H), 9.64 (*s*, CH, 1H), 11.96 (*s*, NH, 1H), 7.20–8.00 (*m*, 8H, aromatic H). IR (ν_{max}/cm^{-1}): 3180 (NH), 1720(C═O), 1579, 1570 (C═N) 2220 (CN).

Crystal data

$C_{16}H_{12}BrClN_4O$
M_r = 391.66
Triclinic, $P\overline{1}$
a = 8.6832 (7) Å
b = 9.4024 (8) Å
c = 10.7392 (9) Å
α = 81.761 (7)°
β = 73.297 (7)°
γ = 69.777 (6)°
V = 787.07 (11) Å^3
Z = 2
D_x = 1.653 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 13558 reflections
θ = 0.0–28.9°
μ = 2.79 mm^{-1}
T = 293 (2) K
Plate, colourless
0.46 × 0.24 × 0.09 mm

Data collection

Stoe IPDS-2 diffractometer
ω scans
Absorption correction: by integration (*X-RED*32; Stoe & Cie, 2002)
T_{min} = 0.429, T_{max} = 0.774
13 505 measured reflections
3090 independent reflections
2483 reflections with $I > 2\sigma(I)$
R_{int} = 0.063
θ_{max} = 26.0°
$h = -10 \rightarrow 10$
$k = -11 \rightarrow 11$
$l = -13 \rightarrow 13$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.077
S = 1.05
3090 reflections
208 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.042P)^2 + 0.0465P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.50 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cl1—C18	1.742 (3)	N2—N3	1.384 (3)
O1—C1	1.225 (3)	N3—C4	1.286 (3)
Br1—C12	1.902 (2)	C4—N5	1.382 (3)
C1—N2	1.336 (3)	N6—C7	1.264 (3)
C1—N5	1.402 (3)		
O1—C1—N2	129.9 (2)	C11—C12—Br1	119.50 (19)
O1—C1—N5	127.5 (2)	C19—C18—Cl1	119.9 (2)
C7—N6—N5	119.19 (18)	C17—C18—Cl1	118.7 (2)
C13—C12—Br1	118.1 (2)		

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

D—H···A	D—H	H···A	D···A	D—H···A
C7—H7···O1	0.93	2.22	2.906 (3)	130
N2—H2···O1^{i}	0.86	1.97	2.818 (2)	170
C9—H9···*Cg*3^{ii}	0.95	2.82	3.751 (3)	166

Symmetry codes: (i) $-x, 1-y, 1-z$; (ii) $1-x, 1-y, 1-z$. Note: *Cg*3 is the centroid of ring *C* (atoms C15–C20).

H atoms were positioned geometrically and refined using a riding model, with distances 0.93 Å for aromatic C—H, 0.97 Å for methylene C—H and 0.86 Å for N—H. U_{iso} (H) was set equal to 1.2U_{eq} 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) and *PARST* (Nardelli, 1995).

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