Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

2-(1*H*-Benzotriazol-1-yl)-3-(4-chlorophenyl)-3-hydroxy-*N*-phenylprop-2-enethioamide

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Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.005 Å
R factor = 0.035
wR factor = 0.090
Data-to-parameter ratio = 11.8

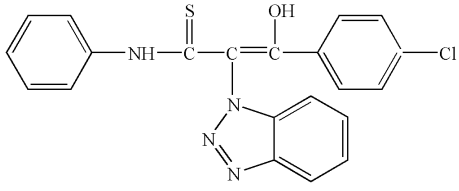
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 20 December 2004
Accepted 4 January 2005
Online 8 January 2005

The title compound, $C_{21}H_{15}ClN_4OS$, was synthesized by the reaction of 2-(1*H*-benzotriazol-1-yl)-1-(4-chlorophenyl)-ethanone with phenyl isothiocyanate and potassium hydroxide in dimethylsulfoxide. There are some inter- and intramolecular interactions in the crystal structure.

Comment

The triazole motif appears frequently in the structures of various natural products and biologically active compounds, notably thiamine (vitamin B), penicillins, antibiotics such as micrococcin (James & Watson, 1966), and many other metabolic products of fungi and primitive marine animals. Benzotriazole derivatives also exhibit better pharmacological activity than triazole compounds and have different biological activities (Zhang *et al.*, 2002). However, triazole compounds containing benzotriazole have rarely been reported. In order to search for new benzotriazole compounds with higher bioactivity, we synthesized the title compound, (I); we describe its structure here.



(I)

In the crystal structure of (I), atoms C1, O1, C15, N2, C16 and C8 are coplanar (plane *p*1). Atoms S1, N1, N2, C1, C2 and C8 also lie in a plane (plane *p*2). The dihedral angles formed by the benzene rings (C2–C7 and C16–C21) and the benzotriazole ring with planes *p*1 and *p*2 are 56.2 (1), 52.0 (2) and 75.8 (2)°, and 61.1 (3), 57.3 (1) and 79.4 (2)°, respectively. The dihedral angle between planes *p*1 and *p*2 is 5.3 (1)°.

The most interesting structural feature of the title compound is the combination of intra- and intermolecular interactions (see Table 2), which stabilize the structure.

Experimental

The title compound was prepared by the reaction of 2-(1*H*-benzotriazol-1-yl)-1-(4-chlorophenyl)ethanone (4.14 g, 0.02 mol), phenylisothiocyanate (2.24 g, 0.02 mol) and potassium hydroxide (2.24 g, 0.04 mol) in DMSO solution (30 ml) at room temperature. Single crystals of the title compound suitable for X-ray measurements were obtained by recrystallization from chloroform/ethylacetate (1:3, *v*/*v*) at room temperature.

Crystal data

$C_{21}H_{15}ClN_4OS$
M_r = 406.88
Monoclinic, *Pc*
a = 10.2362 (12) Å
b = 8.9429 (11) Å
c = 10.3855 (12) Å
β = 91.887 (2)°
V = 950.2 (2) Å^3
Z = 2
D_x = 1.422 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1173 reflections
θ = 3.0–21.1°
μ = 0.33 mm^{-1}
T = 295 (2) K
Block, yellow
0.42 × 0.35 × 0.18 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.820, T_{max} = 0.942
5920 measured reflections
3055 independent reflections
2232 reflections with $I > 2\sigma(I)$
R_{int} = 0.026
θ_{max} = 27.7°
$h = -13 \rightarrow 13$
$k = -11 \rightarrow 11$
$l = -8 \rightarrow 13$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.090
S = 1.05
3055 reflections
260 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + 0.0316P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.13 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}
Absolute structure: Flack (1983), 2239 Friedel pairs
Flack parameter: −0.06 (7)

Table 1
Selected bond lengths (Å).

S1—C1	1.686 (3)	N2—N3	1.373 (3)
Cl1—C19	1.733 (4)	N2—C8	1.426 (4)
N1—C1	1.334 (4)	N3—N4	1.285 (3)
N1—C2	1.418 (4)	N4—C10	1.377 (4)
N2—C9	1.362 (3)	O1—C15	1.320 (3)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···S1^{i}	0.86 (1)	2.70 (2)	3.398 (2)	138 (2)
O1—H2···S1	0.85 (1)	2.07 (2)	2.875 (2)	157 (4)

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

H atoms bonded to N and O were located in a difference map and were refined freely. Other H atoms were fixed geometrically and were treated as riding on the parent C atoms, with C—H distances of 0.93 Å and U_{iso}(H) = 1.2U_{eq}(parent atom).

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); 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*.

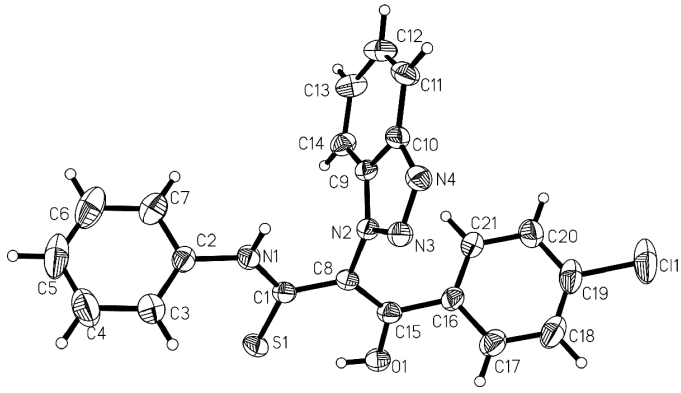


Figure 1
The structure of the title compound, showing 30% probability displacement ellipsoids and the atom-numbering scheme.

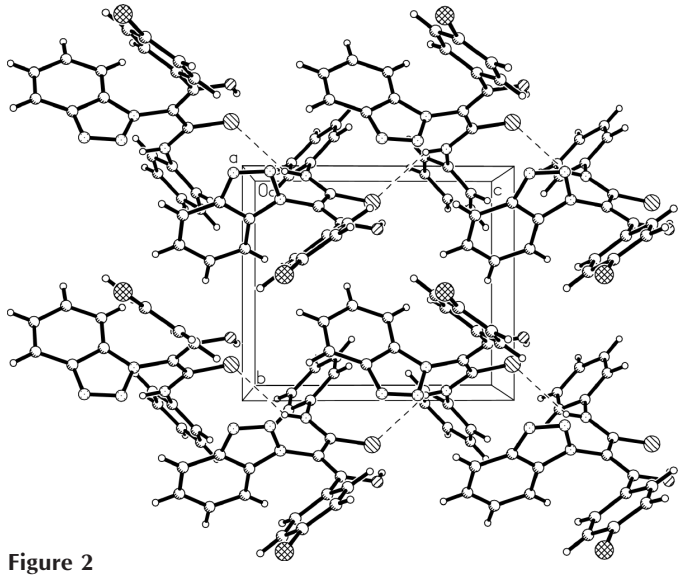


Figure 2
A packing diagram of the molecule of the title compound, viewed down the *a* axis. Hydrogen bonds are shown as dashed lines.

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