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2-(Benzotriazol-1-yl)-2-(α-hydroxybenzyl)-*N*-phenylthioacrylamide

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.040
wR factor = 0.121
Data-to-parameter ratio = 17.9

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

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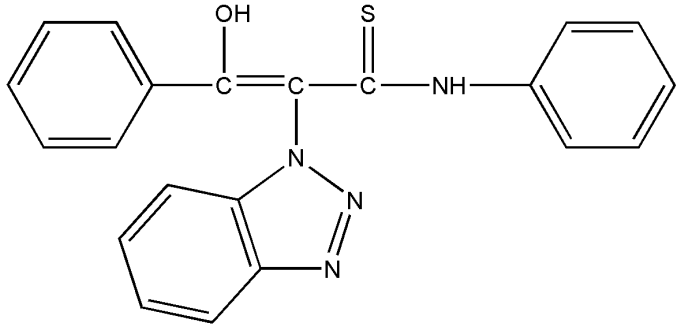
In the title compound, $C_{21}H_{16}N_4OS$, the dihedral angles between the planes of the benzotriazole and *N*-phenyl rings and the plane of the atoms that link these two rings are 79.56 (6) and 59.02 (5)°, respectively, while that between the two benzene rings is 64.12 (6)°. There are some inter- and intramolecular interactions in the crystal structure.

Comment

Triazole nuclei appear frequently in the structures of various natural products and biologically active compounds, notably thiamine (vitamin B), penicillins, and antibiotics such as micrococcin (James & Watson, 1966). Triazole derivatives have also attracted considerable attention in industry and agriculture, because of their significant biological activities (Zhang *et al.*, 2002). In this paper, we report the structure of the title compound, (I).



(I)

In compound (I) (Fig. 1), the bond lengths and angles are generally normal in the rings (Ji *et al.*, 2002). The C═S bond length in (I) is close to the typical C═S double-bond length (Table 1). Atom C2 lies in the plane of the benzotriazole ring, and atoms S1, N1, C1 and C2 are coplanar (plane *P*1). The dihedral angles formed by the benzotriazole and C16–C21 rings with *P*1 are 79.56 (6) and 59.02 (5)°, respectively. The C1—N1—C16—C17, N3—N2—C2—C1, N3—N2—C2—C3 and N2—C2—C3—C4 torsion angles are 54.8 (2), 84.17 (18), 92.39 (17), 3.6 (2)°, respectively.

The most interesting structural features of the title compound are the N—H···N inter- and intramolecular hydrogen bonds, and the strong O—H···S intramolecular interaction (Table 2). These interactions stabilize the structure of (I).

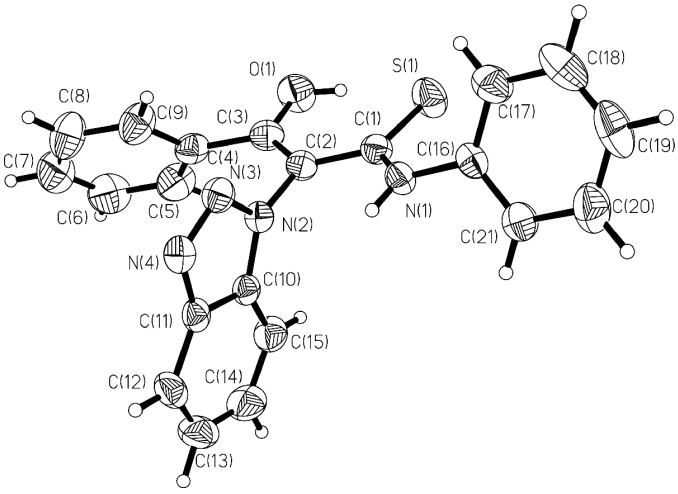


Figure 1
The structure of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 35% probability level and H atoms are shown as small spheres of arbitrary radii.

Experimental

A mixture of α-(benzotriazol-1-yl)acetophenone (1.34 g, 0.01 mol), phenyl isothiocyanate (2.42 g, 0.01 mol), potassium hydroxide (0.4 g, 0.01 mol) and dimethylsulfoxide (50 ml) was stirred for 1 h at room temperature. The solution was then filtered, concentrated and purified by flash chromatography (silica gel, chloroform:cyclohexane, 5:1) to afford the title compound (yield 3.01 g, 80%; m.p. 433–434 K). Single crystals of (I) suitable for X-ray measurements were obtained by recrystallization from cyclohexane at room temperature. ^{1}H NMR (600 MHz, acetone-d_6): 16.09 (1H, *s*, OH), 9.68 (1H, *s*, NH-Ph), 7.06–8.07 (14H, *m*, Ar). Analysis, calculated for $C_{21}H_{16}N_4OS$: C 70.76, H 4.52, N 15.72%; found: C 70.81, H 4.60, N 15.68%.

Crystal data

$C_{21}H_{16}N_4OS$
M_r = 372.45
Monoclinic, *C*2/*c*
a = 19.089 (3) Å
b = 8.4715 (11) Å
c = 23.161 (3) Å
β = 90.315 (2)°
V = 3745.4 (9) Å^3
Z = 8
D_x = 1.321 Mg m^{-3}
Mo *K*α radiation
Cell parameters from 2238 reflections
θ = 2.6–22.6°
μ = 0.19 mm^{-1}
T = 293 (2) K
Block, colourless
0.46 × 0.31 × 0.18 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.892, T_{max} = 0.966
12 321 measured reflections
4500 independent reflections
2680 reflections with $I > 2\sigma(I)$
R_{int} = 0.023
θ_{max} = 28.0°
h = −24 → 25
k = −11 → 11
l = −30 → 20

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.121
S = 1.06
4500 reflections
252 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0561P)^2 + 0.3455P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}
Extinction correction: none

Table 1
Selected geometric parameters (Å, °).

S1—C1	1.6816 (17)	N2—C2	1.4309 (19)
N1—C1	1.324 (2)	N3—N4	1.2920 (18)
N1—C16	1.428 (2)	N4—C11	1.374 (2)
N2—N3	1.3609 (17)	O1—C3	1.328 (2)
N3—N2—C2—C3	−92.39 (17)	N2—C2—C3—C4	3.6 (2)
N3—N2—C2—C1	84.17 (18)	C1—N1—C16—C17	54.8 (2)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*A*···S1	0.88 (2)	2.05 (2)	2.8752 (17)	156 (2)
N1—H1···N2	0.855 (12)	2.343 (14)	2.7188 (15)	107.0 (11)
N1—H1···N4^{i}	0.855 (12)	2.112 (12)	2.9051 (14)	154

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

H atoms on N or O atoms were located in a difference Fourier map and refined freely. All other H atoms were placed in calculated positions, with C—H = 0.93 or 0.97 Å, and included in the final cycles of refinement using a riding model, with $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1999); 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, 1999); software used to prepare material for publication: *SHELXTL*.

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