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2-(1*H*-Benzo-1,2,3-triazol-1-yl)-4,4-dimethyl-3-oxo-*N*-phenylpentanethioamide monohydrate

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Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.006 Å
R factor = 0.062
wR factor = 0.151
Data-to-parameter ratio = 13.5

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

The title compound, $C_{19}H_{22}N_4O_2S\cdot H_2O$, was synthesized in order to search for new benzotriazole compounds with high bioactivity. There are some intermolecular hydrogen-bond interactions in the crystal structure, providing stabilization.

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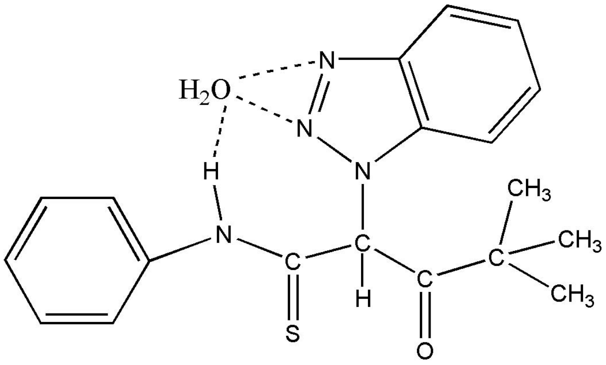
Comment

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). In order to search for new benzotriazole compounds with higher bioactivity, we synthesized the title compound, (I), and describe its structure here.



(I)

The title compound crystallizes as a monohydrate. Bond lengths and angles in the benzotriazole system are in good agreement with those quoted in a previous report (Xu *et al.*, 2005). The C═S bond length is shorter than the typical C═S bond length [1.68 Å; Allen *et al.*, 1987]. The S1/N1/C1/C2 group is planar and nearly coplanar with the phenyl ring (dihedral angle: 4.4 (1)°). The dihedral angles formed by the benzotriazole ring system with the phenyl ring is 82.6 (2)°.

The most interesting structural feature of the title compound is the presence of intermolecular hydrogen-bond interactions (Table 2), which stabilize the structure.

Experimental

The title compound was prepared by reaction of 1-(1*H*-benzo[1,2,3]triazol-1-yl)-3,3-dimethylbutan-2-one (4.34 g, 0.02 mol),

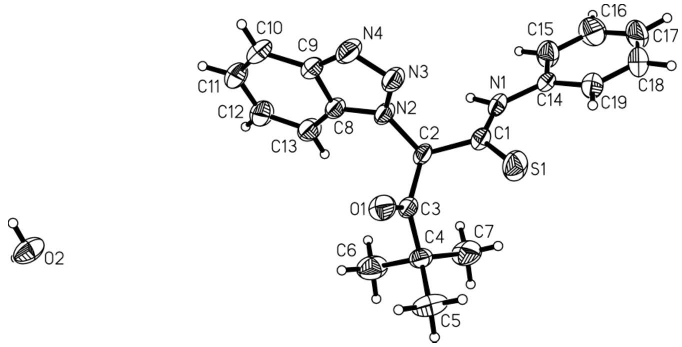


Figure 1
View of the title compound, (I), with displacement ellipsoids drawn at the 40% probability level.

phenyl isothiocyanate (2.24 g, 0.02 mol) and potassium hydroxide (2.24 g, 0.04 mol) in dimethylsulfoxide at room temperature. Single crystals suitable for X-ray measurements were obtained by recrystallization from ethanol–water (*v*/*v* = 3:1) at room temperature.

Crystal data

$C_{19}H_{20}N_4OS{\cdot}H_2O$
$M_r = 370.47$
Triclinic, $P\overline{1}$
$a = 5.7572$ (14) Å
$b = 11.545$ (3) Å
$c = 14.778$ (4) Å
$\alpha = 78.575$ (4)°
$\beta = 81.022$ (3)°
$\gamma = 81.362$ (4)°
$V = 943.7$ (4) Å^3
$Z = 2$
$D_x = 1.304$ Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 1048 reflections
$\theta = 2.3$–22.3°
$\mu = 0.19$ mm^{-1}
$T = 295$ (2) K
Block, yellow
0.26 × 0.18 × 0.16 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.943$, $T_{\max} = 0.970$
5114 measured reflections
3274 independent reflections
1619 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.148$
$\theta_{\max} = 25.0°$
$h = -6 \rightarrow 6$
$k = -13 \rightarrow 7$
$l = -17 \rightarrow 17$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.062$
$wR(F^2) = 0.151$
$S = 0.80$
3274 reflections
242 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0587P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.23$ e Å^{-3}
$\Delta\rho_{\min} = -0.30$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

S1—C1	1.632 (3)	N3—N4	1.295 (4)
N1—C1	1.331 (4)	O1—C3	1.198 (4)
N1—C14	1.407 (4)		
C1—N1—C14	132.8 (3)	N1—C1—S1	128.3 (3)
N4—N3—N2	108.2 (3)	O1—C3—C4	121.6 (3)
N1—C1—C2	111.7 (3)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2*B*···N4$^{\rm i}$	0.85	2.10	2.926 (4)	163
O2—H2*A*···N3$^{\rm ii}$	0.85	2.02	2.866 (4)	176
N1—H1···O2$^{\rm iii}$	0.89 (4)	1.93 (4)	2.766 (4)	155 (3)

Symmetry codes: (i) $1 - x, 1 - y, -z$; (ii) $1 + x, y - 1, z$; (iii) $x, 1 + y, z$.

All H atoms were placed in calculated positions. H atoms bonded to C and O atoms were constrained to ride on their parent atom (C—H = 0.93–0.96 Å and O—H = 0.85 Å), with $U_{\rm iso}$ values of $1.2U_{\rm eq}$(C) for the aryl and CH H atoms and 1.5 $U_{\rm eq}$(C,O) for the CH_3 and water H atoms. The position and isotropic displacement parameter of the NH H atom were refined freely. The crystals showed a weak diffracting ability, which could account for the rather high $R_{\rm int}$ and low goodness-of-fit.

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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