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4,4-Dimethyl-3-oxo-*N*-phenyl-2-(1*H*-1,2,4-triazol-1-yl)pentanethioamide monohydrate

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Key indicators

Single-crystal X-ray study
T = 273 K
Mean σ(C–C) = 0.004 Å
R factor = 0.045
wR factor = 0.132
Data-to-parameter ratio = 19.8

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

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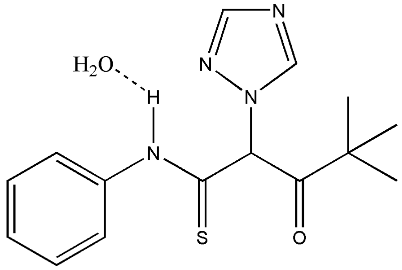
In the title compound, $C_{15}H_{18}N_4O_2S$, the dihedral angle between the planes of the phenyl and triazole rings is 69.47 (9)°. There are weak O—H···N, N—H···O and O—H···S intermolecular interactions in the crystal structure, contributing to its stability.

Comment

As an important type of fungicide, triazole compounds possessing low toxicity are highly efficient (Shi *et al.*, 1995; Xu *et al.*, 2002). At present, studies of triazole derivatives are mainly concentrated on compounds with triazole as the only active group. In order to search for new triazole compounds with higher bioactivity, the title compound, (I), was synthesized.



(I)

Compound (I) crystallizes as a monohydrate. The bond lengths and angles are generally normal in the phenyl and triazole rings (Ji *et al.*, 2002; Jian *et al.*, 2003, 2004; Xu *et al.*, 2004). The dihedral angle between the O1/C2/C5 plane and the plane of the triazole ring is 57.4 (1)°. The dihedral angle

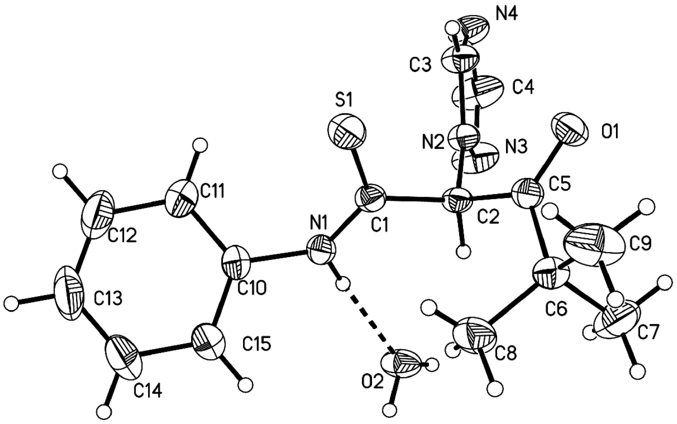


Figure 1
View of (I), with displacement ellipsoids drawn at the 30% probability level. The dashed line indicates a hydrogen bond.

between the planes of the phenyl and triazole rings is 69.47 (9)°. The crystal structure is stabilized by weak N—H···O, O—H···S and O—H···N interactions (Table 2 and Fig. 2) (Steiner, 1996; Jeffrey *et al.*, 1985).

Experimental

The title compound was prepared by reaction of 3,3-dimethyl-1-(1*H*-1,2,4-triazol-1-yl)butan-2-one (0.02 mol) with isothiocyanatobenzene (0.02 mol) at room temperature for 2 h. Single crystals suitable for X-ray measurements were obtained by recrystallization from ethanol/H_2O (3:1 *v*/*v*) at room temperature.

Crystal data

$C_{15}H_{18}N_4OS \cdot H_2O$
$M_r = 320.41$
Monoclinic, $P2_1/c$
$a = 15.465$ (4) Å
$b = 9.988$ (2) Å
$c = 11.227$ (3) Å
$\beta = 93.554$ (3)°
$V = 1730.8$ (7) Å^3
$Z = 4$

$D_x = 1.230$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1881 reflections
$\theta = 2.6$–20.8°
$\mu = 0.20$ mm^{-1}
$T = 273$ (2) K
Block, colorless
0.36 × 0.30 × 0.15 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1997)
$T_{\min} = 0.911$, $T_{\max} = 0.971$
11490 measured reflections

4180 independent reflections
2527 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.033$
$\theta_{\max} = 28.1$°
$h = -20 \rightarrow 9$
$k = -13 \rightarrow 12$
$l = -14 \rightarrow 13$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.046$
$wR(F^2) = 0.132$
$S = 1.04$
4180 reflections
211 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0503P)^2 + 0.2623P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.25$ e Å^{-3}
$\Delta\rho_{\min} = -0.23$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

S1—C1	1.6567 (18)	N2—C2	1.458 (2)
N1—C1	1.328 (2)	N3—C4	1.308 (3)
N1—C10	1.425 (2)	N4—C3	1.309 (2)
N2—C3	1.331 (2)	N4—C4	1.335 (3)
N2—N3	1.346 (2)	O1—C5	1.207 (2)
C1—N1—C10	126.67 (15)	N2—C2—C1	109.14 (14)
N1—C1—C2	113.09 (15)	C1—C2—C5	112.09 (14)
N1—C1—S1	127.45 (13)	O1—C5—C2	119.09 (16)
C2—C1—S1	119.45 (14)		
C10—N1—C1—C2	−179.98 (16)	N1—C1—C2—C5	−139.77 (15)
C10—N1—C1—S1	0.9 (3)	C2—N2—C3—N4	−179.81 (18)
C3—N2—C2—C1	63.4 (2)	C1—C2—C5—O1	−109.79 (18)
N3—N2—C2—C1	−117.13 (19)	C1—C2—C5—C6	76.42 (19)
N1—C1—C2—N2	96.26 (17)	C1—N1—C10—C15	−130.2 (2)
S1—C1—C2—N2	−84.50 (17)		

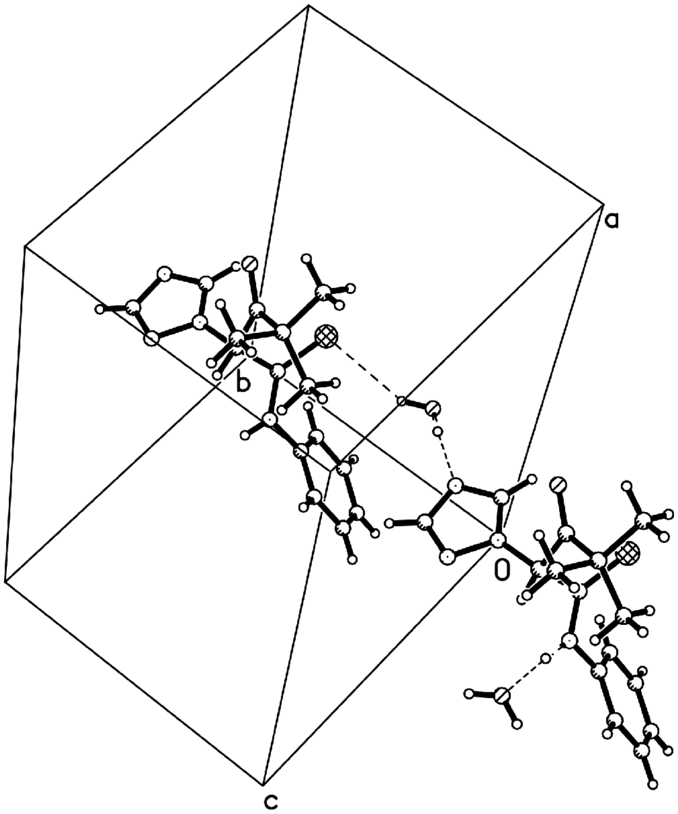


Figure 2
Intermolecular hydrogen bonds (dashed line) in the structure of (I).

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2*B*···S1$^{\rm i}$	0.850 (9)	2.649 (13)	3.4283 (17)	153 (2)
O2—H2*A*···N4$^{\rm ii}$	0.859 (9)	1.946 (9)	2.801 (2)	174 (2)
N1—H1···O2	0.878 (9)	1.939 (11)	2.802 (2)	167 (2)

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

H atoms bonded to N1 and O2 were located in a difference map and their coordinates were refined with $U_{\rm iso}({\rm H}) = 1.5U_{\rm eq}$(N or O). All other H atoms were placed in calculated positions, with C—H = 0.93 or 0.96 Å, and included in the final cycles of refinement using a riding model, with $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}$(C).

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT* program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1999); software used to prepare material for publication: *SHELXTL*.

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o164 Liang-Zhong Xu *et al.* • $C_{15}H_{18}N_4OS \cdot H_2O$ *Acta Cryst.* (2005). E**61**, o163–o165

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