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4-(4-Nitrobenzylideneamino)-3-(1*H*-1,2,4-triazol-1-ylmethyl)-1*H*-1,2,4-triazole-5(4*H*)-thione monohydrate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.043
wR factor = 0.125
Data-to-parameter ratio = 15.7

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

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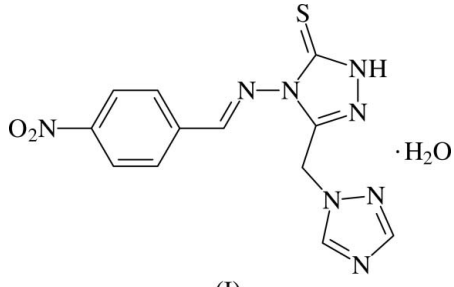
In the title compound, $C_{12}H_{10}N_8O_2S\cdot H_2O$, the thione-substituted triazole ring is coplanar with the benzene ring; it forms a dihedral angle of 86.42 (6)° with the other triazole ring. Intermolecular O—H···N and N—H···O hydrogen bonds link the molecules into sheets parallel to (010).

Comment

Recently, compounds containing the 1*H*-1,2,4-triazole group have attracted much interest because they are efficient fungicides and pesticides, and have good plant-growth regulatory activity for a wide variety of crops (Xu *et al.*, 2002). In addition, amine- and thione-substituted triazoles have been studied as anti-inflammatory and antimicrobial agents (Eweiss *et al.*, 1986; Awad *et al.*, 1991). The title compound, (I), was synthesized in a search for new triazole compounds with better biological activity. We report here the crystal structure of (I) (Fig. 1).



(I)

Bond lengths and angles in the triazole rings of (I) are in agreement with those reported previously (Li *et al.*, 2005; Xu *et al.*, 2005). The molecule exists in the thione tautomeric form; the S═C distance of 1.665 (2) Å indicates substantial double-bond character (Escobar-Valderrama *et al.*, 1989). The C1–C3,N1–N3 and C6–C12,N8 planes make dihedral angles of 86.42 (6) and 0.94 (7)°, respectively, with the thione-substituted triazole plane C4/C5/C7/N4–N6/S1.

Intermolecular O—H···N and N—H···O hydrogen bonds (Table 1) link the molecules into a two-dimensional network parallel to (010) (Fig. 2).

Experimental

A mixture of 4-amino-3-(1,2,4-triazole-1-yl)-1*H*-1,2,4-triazole-5(4*H*)-thione (5 mmol) and 2-nitrobenzaldehyde (5 mmol) was refluxed for 10–20 min in glacial acetic acid. The mixture was then filtered and crystallized from ethanol to afford the title compound (yield: 1.55 g, 94.6%). Single crystals suitable for X-ray measurements were obtained by recrystallization from ethanol at room temperature.

Crystal data

$C_{12}H_{10}N_8O_2S\cdot H_2O$
M_r = 348.37
Monoclinic, $P2_1/c$
a = 8.1883 (16) Å
b = 24.938 (5) Å
c = 7.5896 (15) Å
β = 93.56 (3)°
V = 1546.8 (5) Å^3
Z = 4
D_x = 1.496 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.24 mm^{-1}
T = 293 (2) K
Plate, colourless
0.68 × 0.68 × 0.07 mm

Data collection

Rigaku R-AXIS RAPID IP area-detector diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.853, T_{max} = 0.984
14545 measured reflections
3525 independent reflections
2454 reflections with $I > 2\sigma(I)$
R_{int} = 0.041
θ_{max} = 27.5°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.125
S = 1.04
3525 reflections
225 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0672P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1W—H2WA···N4^{i}	0.89 (4)	2.06 (4)	2.945 (3)	172 (3)
O1W—H1WA···N3^{ii}	0.88 (4)	1.88 (4)	2.747 (3)	172 (3)
N5—H5A···O1W	0.86	1.83	2.661 (2)	162

Symmetry codes: (i) $x, -y+\frac{3}{2}, z-\frac{1}{2}$; (ii) $x-1, y, z-1$.

The positional and isotropic displacement parameters of the H atoms attached to O1W were refined freely. All other H atoms were placed in calculated positions, with N—H = 0.86 Å and C—H = 0.93 or 0.97 Å, and refined using a riding model, with U_{iso}(H) = 1.2U_{eq}(C) for the aryl and methylene H atoms and 1.5U_{eq}(C) for the methyl H atoms.

Data collection: *RAPID-AUTO* (Rigaku, 2001); cell refinement: *RAPID-AUTO*; data reduction: *RAPID-AUTO*; 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: *SHELXL97*.

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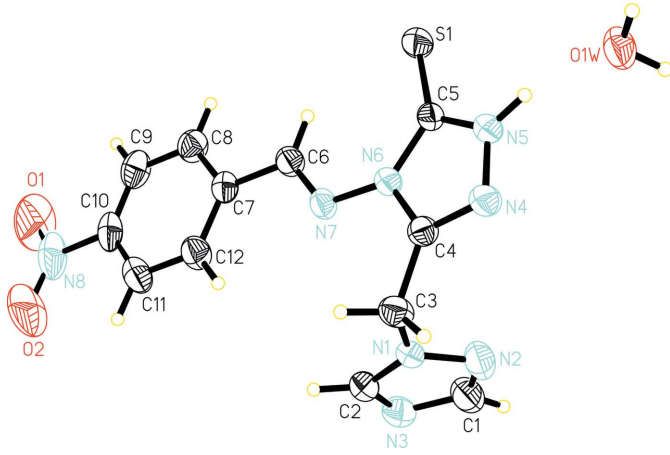

Figure 1
The molecular structure of (I), with displacement ellipsoids drawn at the 40% probability level.

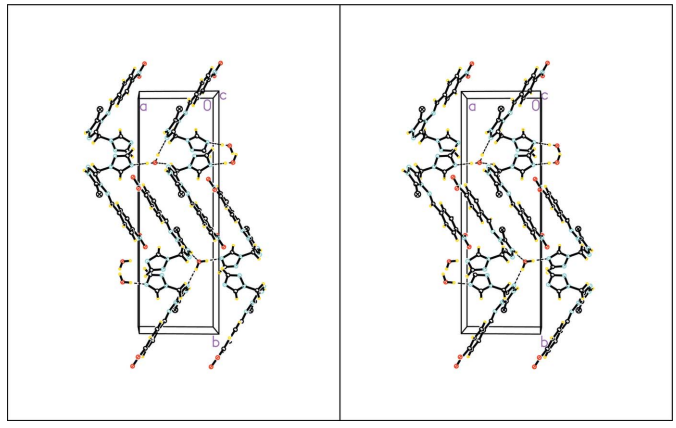

Figure 2
A stereoview of the crystal packing of (I). Hydrogen bonds are shown as dashed lines.

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