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4-(4-Methylbenzylideneamino)-3-[(1*H*-1,2,4-triazol-1-yl)methyl]-1*H*-1,2,4-triazole-5(4*H*)-thione monohydrate

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Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.004 Å
R factor = 0.041
wR factor = 0.113
Data-to-parameter ratio = 15.3

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

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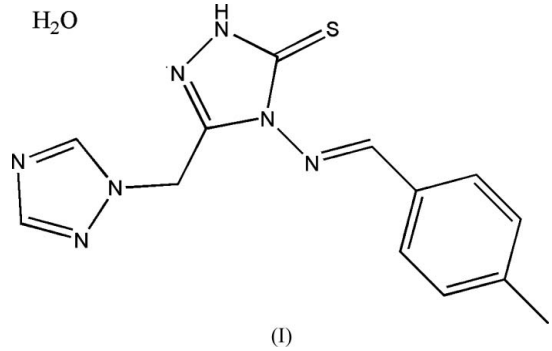
In the title compound, $C_{13}H_{15}N_7OS$, the dihedral angles made by the plane of the thione-substituted triazole ring with the planes of the other triazole ring and the benzene ring are 88.69 (3) and 2.94 (2)°, respectively. Inter- and intramolecular hydrogen-bond and π–π stacking interactions stabilize the structure.

Comment

Recently, compounds containing a 1*H*-1,2,4-triazole group have attracted much interest because they are well known as efficient fungicides in pesticides and they exhibit 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). In a search for new triazole compounds with better biological activity, the title compound, (I), was synthesized. We report here the crystal structure of (I).



(I)

Compound (I) crystallizes as a monohydrate. The bond lengths and angles are generally normal in the triazole rings (Xu *et al.*, 2005). The molecule exists in the thione tautomeric form, with an S=C distance of 1.664 (2) Å, which indicates substantial double-bond character for this bond (Allen *et al.*, 1987). The planes C11–C13/N5/N6/N7 and C1–C8 make angles of 88.69 (3) and 2.9 (2)°, respectively, with the thione-substituted triazole plane C9/C10/N2–N4/S1. The crystal structure of (I) is stabilized by weak intra- and intermolecular hydrogen bonds and π–π stacking interactions. The π–π stacking interactions involve the thione-substituted triazole ring (C9/C10/N2–N4) *R*1 and the benzene ring (C2–C5/C7/C8) *R*2. The distance between the centroids of rings *R*1 and *R*2 (at $1-x$, $-y$, $1-z$) is 3.668 (2) Å.

Experimental

A mixture of 4-amino-3-(1,2,4-triazol-1-yl)-1*H*-1,2,4-triazole-5(4*H*)-thione (0.02 mol) and 4-methylbenzaldehyde (0.02 mol) was refluxed

at 391 K for 20 min in glacial acetic acid. The mixture was then filtered and crystallized from ethanol to afford the compound 5-[(1*H*-1,2,4-triazol-1-yl)methyl]-4-(4-methylbenzylideneamino)-2*H*-1,2,4-triazole-3(4*H*)-thione (5.32 g, yield 89%). Single crystals suitable for X-ray measurements were obtained by recrystallization from ethanol at room temperature. The water in the structure is derived from the undried ethanol solvent.

Crystal data

$C_{13}H_{13}N_7S\cdot H_2O$	D_x = 1.335 Mg m^{-3}
M_r = 317.38	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 2352 reflections
a = 8.2900 (14) Å	θ = 2.8–25.0°
b = 24.778 (5) Å	μ = 0.22 mm^{-1}
c = 7.7083 (14) Å	T = 294 (2) K
β = 93.966 (3)°	Block, yellow
V = 1579.6 (5) Å^3	0.22 × 0.20 × 0.12 mm
Z = 4	

Data collection

Bruker SMART CCD area-detector diffractometer	3234 independent reflections
	2039 reflections with $I > 2\sigma(I)$
φ and ω scans	R_{int} = 0.031
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	θ_{max} = 26.4°
	$h = -10 \rightarrow 8$
T_{min} = 0.952, T_{max} = 0.974	$k = -31 \rightarrow 26$
8843 measured reflections	$l = -9 \rightarrow 8$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0501P)^2 + 0.2737P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.041	
$wR(F^2)$ = 0.113	
S = 1.02	$(\Delta/\sigma)_{max}$ = 0.004
3234 reflections	$\Delta\rho_{max}$ = 0.20 e Å^{-3}
212 parameters	$\Delta\rho_{min}$ = −0.16 e Å^{-3}
H atoms treated by a mixture of independent and constrained refinement	

Table 1
Selected geometric parameters (Å, °).

S1—C9	1.664 (2)	N3—N4	1.372 (2)
N1—C1	1.269 (3)	N5—N6	1.351 (3)
N1—N2	1.394 (2)		
C1—N1—N2	119.51 (17)	C10—N3—N4	104.05 (16)
C10—N2—C9	108.34 (15)	N5—C11—C10	109.92 (17)

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1A···N3^{i}	0.86 (2)	2.06 (3)	2.912 (3)	171
O1—H1B···N7^{ii}	0.86 (2)	1.95 (3)	2.800 (2)	173
N4—H4A···O1^{iii}	0.90 (2)	1.81 (2)	2.699 (2)	167
C1—H1···S1	0.93	2.51	3.255 (2)	137

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

All C-bound H atoms were placed in calculated positions, with C—H = 0.93–0.97 Å, and included in the final cycles of refinement using a riding model, with U_{iso}(H) values of $1.2U_{eq}$(C) for the aryl and methylene H atoms, and $1.5U_{eq}$(C) for the methyl H atoms. The position and isotropic displacement parameters of the N-bound and O-bound H atoms were refined freely.

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*.

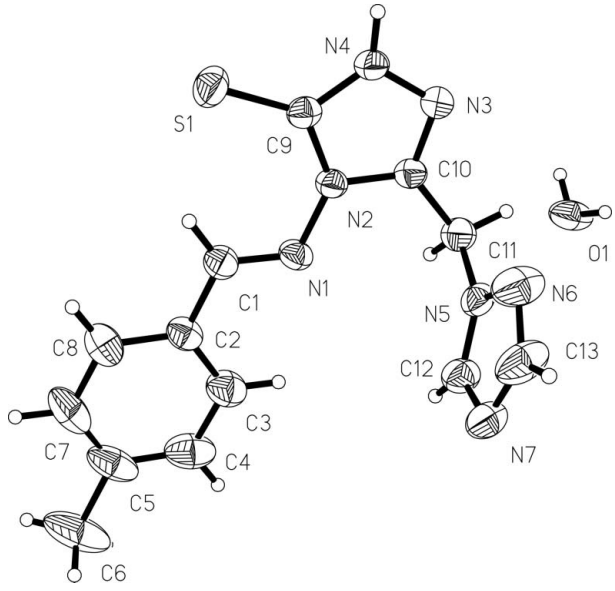


Figure 1
View of (I), with displacement ellipsoids for non-H atoms drawn at the 40% probability level.

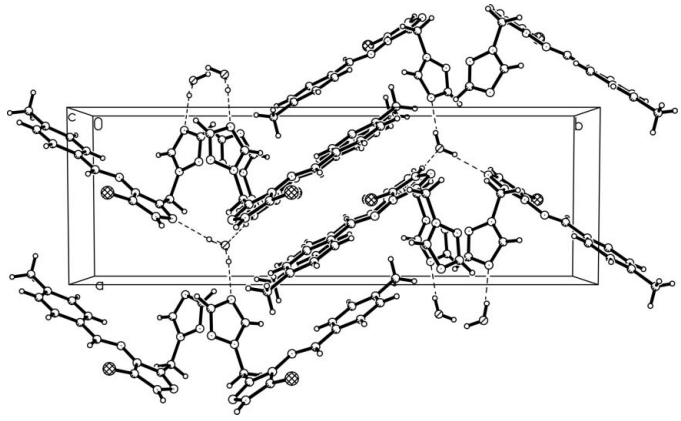


Figure 2
A packing diagram of the title compound, viewed down the *c* axis. Hydrogen bonds are shown as dashed lines.

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