

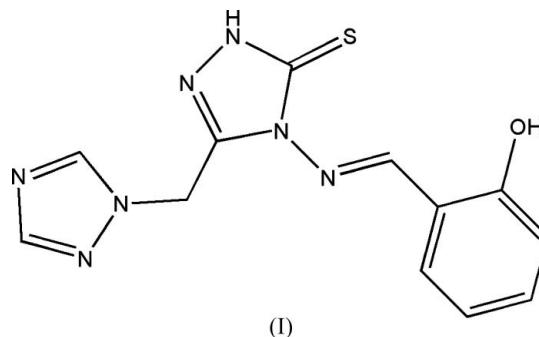
4-(2-Hydroxybenzylideneamino)-3-(1*H*-1,2,4-triazol-1-ylmethyl)-1*H*-1,2,4-triazole-5(4*H*)-thioneZhi-Qiang Hu,^a Yu-Qing Shang,^a
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Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ (C–C) = 0.002 Å
R factor = 0.031
wR factor = 0.087
Data-to-parameter ratio = 14.1For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.In the title compound, C₁₂H₁₁N₇OS, the dihedral angles made by the thione-substituted triazole ring with the other triazole ring and the benzene ring are 71.56 (2) and 47.89 (3)°, respectively. Inter- and intramolecular hydrogen-bond interactions stabilize the structure.

Comment

Recently, compounds containing the 1*H*-1,2,4-triazole group have attracted much interest because compounds containing a triazole ring system are well known as efficient fungicides in 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). 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).Bond lengths and angles of the triazole rings (Table 1) are in agreement with those in previous reports (Li *et al.*, 2005; Xu *et al.*, 2005). The molecule exists in the thione tautomeric form, with an S=C distance of 1.6762 (15) Å, which indicates substantial double-bond character (Escobar-Valderrama *et al.*, 1989). The planes C1–C3/N1/N2/N3 and C6–C12/N7/O1 make dihedral angles of 71.56 (2) and 47.89 (3)° with the thione-substituted triazole plane C4/C5/N4/N5/N6/S1. There are some intra- and intermolecular hydrogen-bond interactions which stabilize the crystal structure (Table 2).

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 2-hydroxybenzaldehyde (0.02 mol) was refluxed at 391 K for 15–20 min in glacial acetic acid. The mixture was then filtered and crystallized from ethanol to afford the title compound (5.7 g, yield 95%). Single crystals suitable for X-ray measurements were obtained by recrystallization from ethanol at room temperature.

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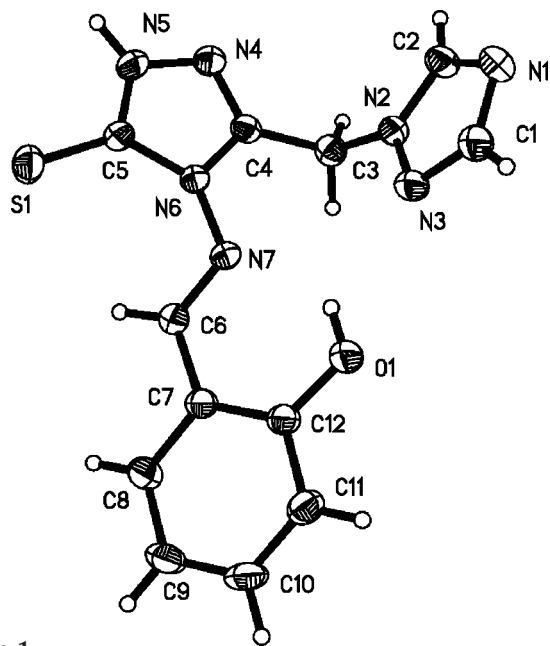


Figure 1
View of the title compound (I), with displacement ellipsoids drawn at the 40% probability level.

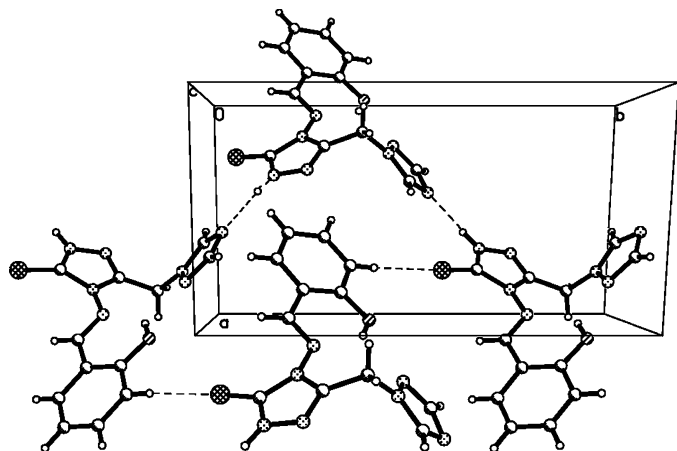


Figure 2
A packing diagram of the molecule of the title compound, viewed down the *c* axis. Hydrogen bonds are shown as dashed lines.

Crystal data

$C_{12}H_{11}N_7OS$
 $M_r = 301.34$
 Monoclinic, $P2_1/c$
 $a = 8.3335$ (9) Å
 $b = 14.9777$ (16) Å
 $c = 11.4724$ (12) Å
 $\beta = 107.990$ (2)°
 $V = 1361.9$ (3) Å³
 $Z = 4$

$D_x = 1.470$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 4407 reflections
 $\theta = 2.9$ – 26.4 °
 $\mu = 0.25$ mm⁻¹
 $T = 294$ (2) K
 Block, yellow
 $0.24 \times 0.22 \times 0.20$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{min} = 0.941$, $T_{max} = 0.951$
 7581 measured reflections

2794 independent reflections
 2360 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.019$
 $\theta_{max} = 26.4$ °
 $h = -10 \rightarrow 7$
 $k = -16 \rightarrow 18$
 $l = -14 \rightarrow 13$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.031$
 $wR(F^2) = 0.087$
 $S = 1.03$
 2794 reflections
 198 parameters
 H atoms treated by a mixture of independent and constrained refinement

$$w = 1/[\sigma^2(F_o^2) + (0.0406P)^2 + 0.4772P]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} = 0.001$
 $\Delta\rho_{max} = 0.19$ e Å⁻³
 $\Delta\rho_{min} = -0.20$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

S1–C5	1.6762 (15)	N5–C5	1.3392 (19)
N4–C4	1.2941 (19)	N6–N7	1.3981 (16)
N4–N5	1.3730 (18)	N7–C6	1.2799 (18)
C6–N7–N6	117.05 (12)	N2–C3–C4	111.71 (12)

Table 2

Hydrogen-bond geometry (Å, °).

<i>D</i> –H... <i>A</i>	<i>D</i> –H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> –H... <i>A</i>
N5–H5...N1 ⁱ	0.89 (2)	1.97 (2)	2.8389 (19)	168 (2)
O1–H1...N3	0.83 (2)	2.32 (2)	2.9324 (17)	131 (2)
O1–H1...N7	0.83 (2)	2.03 (2)	2.7051 (17)	137 (2)
C3–H3B...S1 ⁱⁱ	0.97	2.85	3.8020 (16)	168
C6–H6...S1	0.93	2.82	3.3094 (15)	114

Symmetry codes: (i) $-x + 1, y - \frac{1}{2}, -z + \frac{5}{2}$; (ii) $x, -y + \frac{1}{2}, z + \frac{1}{2}$.

H atoms bonded to C atoms were placed in calculated positions and constrained to ride on their parent atoms ($C-H = 0.93$ – 0.96 Å) with $U_{iso}(H) = 1.2U_{eq}(C)$. The positions and isotropic displacement parameters of the H atoms attached to the N and O 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.

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