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

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Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.003 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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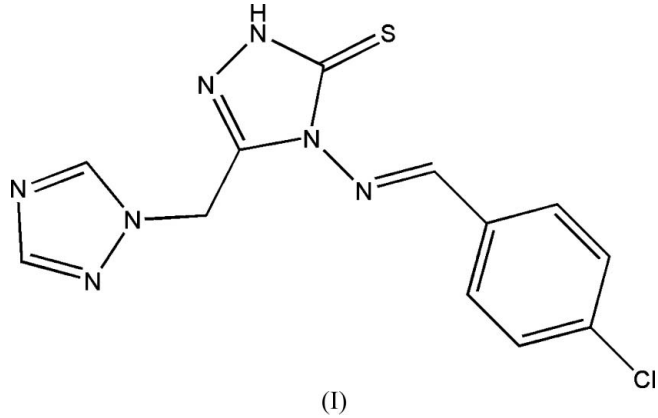
In the title compound, $C_{12}H_{10}ClN_7S$, the dihedral angles made by the plane of the thione-substituted triazole ring with the planes of the other triazole ring and the benzene ring are 73.57 (3) and 46.65 (2)°, respectively. Inter- and intramolcular hydrogen bonds and π-π stacking interactions stabilize the structure.

Comment

Recently, compounds containing a 1*H*-1,2,4-triazole group have attracted much interest because compounds containing this ring system 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)

Bond lengths and angles in the thione-substituted triazole ring and the other triazole ring (Table 1) are in agreement with the values quotes 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 1.662 (2) Å, which indicates substantial double-bond character for this bond (Allen *et al.*, 1987). The planes C10–C12/N5/N6/N7 and C1–C7/N1/Cl1 make angles of 73.57 (3) and 46.65 (2)°, respectively, with the thione-substituted triazole plane C8/C9/N2/N3/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 (C8/C9/N2/N3/N4/S1) *R*1 and the benzene ring (C2–C7) *R*2. The distance between the centroids of rings *R*1 and *R*2 (at $1-x, \frac{1}{2}+y, \frac{1}{2}-z$) is 3.532 (3) 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-chlorobenzaldehyde (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 (5.87 g, yield 92%). Single crystals suitable for X-ray measurements were obtained by recrystallization from ethanol at room temperature.

Crystal data

$C_{12}H_{10}ClN_7S$
M_r = 319.78
Monoclinic, $P2_1/c$
a = 11.090 (2) Å
b = 7.4421 (15) Å
c = 17.734 (4) Å
β = 97.693 (3)°
V = 1450.6 (5) Å^3
Z = 4
D_x = 1.464 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2083 reflections
θ = 2.3–25.2°
μ = 0.41 mm^{-1}
T = 294 (2) K
Block, yellow
0.22 × 0.20 × 0.18 mm

Data collection

Bruker SMART CCD area detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.910, T_{max} = 0.929
7928 measured reflections
2972 independent reflections
1855 reflections with $I > 2\sigma(I)$
R_{int} = 0.029
θ_{max} = 26.4°
$h = -8 \rightarrow 13$
$k = -9 \rightarrow 8$
$l = -22 \rightarrow 22$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.113
S = 1.01
2972 reflections
194 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.05P)^2 + 0.2972P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

S1—C8	1.662 (2)	N3—N4	1.376 (2)
N1—N2	1.395 (2)	N5—N6	1.354 (3)
C1—N1—N2	116.02 (18)	N5—C10—C9	112.72 (18)

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

D—H···A	D—H	H···A	D···A	D—H···A
N4—H4A···N7^{i}	0.83 (2)	2.01 (3)	2.830 (3)	169 (2)
C4—H4···S1^{ii}	0.93	2.82	3.517 (2)	133
C1—H1···S1	0.93	2.74	3.246 (2)	115

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

All H atoms were placed in calculated positions, with C—H = 0.93–0.97 Å, and refined using a riding model, with U_{iso}(H) = 1.2U_{eq}(C).

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*.

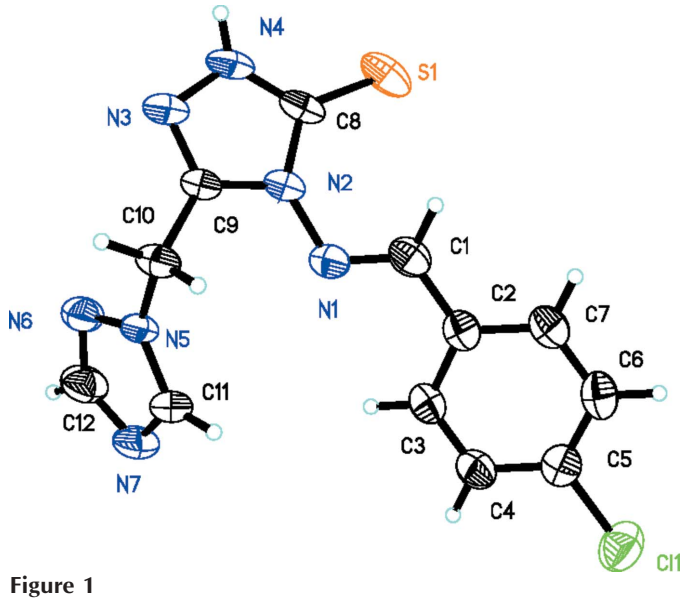


Figure 1
View of the title compound (I), with displacement ellipsoids drawn at the 40% probability level.

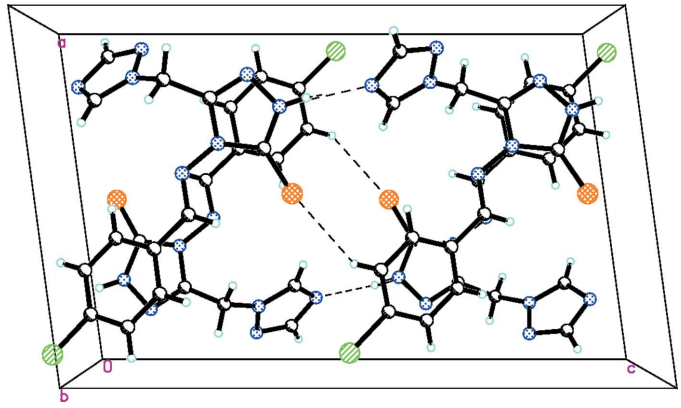


Figure 2
The packing of the title compound, viewed down the *b* axis. Hydrogen bonds are shown as dashed lines.

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