Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

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

Xiao-Lan Lu,[a] Wen-Zhao Bi,[b] Yu-Qing Shang,[b] Liang-Zhong Xu[b]* and Guan-Ping Yu[b]

[a]College of Chemistry and Chemical Engineering, Ocean University of China, Qingdao 266003, People's Republic of China, and [b]College of Chemistry and Molecular Engineering, Qingdao University of Science and Technology, Qingdao 266042, People's Republic of China

Correspondence e-mail: qknhs@yahoo.com.cn

Key indicators

Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.004 Å
R factor = 0.043
wR factor = 0.112
Data-to-parameter ratio = 14.1

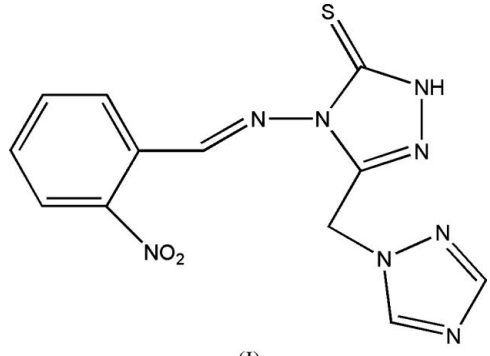
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 23 May 2006
Accepted 9 June 2006

In the title compound, $C_{12}H_{10}N_8O_2S$, the thione-substituted triazole ring forms dihedral angles of 79.86 (2) and 9.86 (3)°, respectively, with the other triazole ring and the benzene ring. Intermolecular N—H···N hydrogen bonds link the molecules into chains extended along the [101] direction. The crystal packing is further stabilized by van der Waals forces.

Comment

Recently, compounds containing the 1*H*-1,2,4-triazole group have attracted much interest because they 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) (Fig. 1).



(I)

Bond lengths and angles in the triazole rings in (I) 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 1.672 (2) Å, indicating substantial double-bond character (Escobar-Valderrama *et al.*, 1989). The mean planes C10–C12/N6/N7/N8 and C1–C7/N1 make dihedral angles of 79.86 (2) and 9.86 (3)°, respectively, with the thione-substituted triazole plane C8–C10/N3/N4/N5/S1.

Intermolecular N—H···N hydrogen bonds (Table 1) link the molecules into chains extended along the [101] direction. The crystal packing (Fig. 2) is further stabilized by van der Waals forces.

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, 93.7%). Single crystals suitable for X-ray measurements were obtained by recrystallization from ethanol at room temperature.

Crystal data

$C_{12}H_{10}N_8O_2S$
M_r = 330.34
Monoclinic, $P2_1/n$
a = 11.421 (2) Å
b = 8.1070 (14) Å
c = 16.446 (3) Å
β = 105.345 (3)°
V = 1468.5 (5) Å^3
Z = 4
D_x = 1.494 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.25 mm^{-1}
T = 294 (2) K
Block, yellow
0.18 × 0.10 × 0.06 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.950, T_{max} = 0.985
8026 measured reflections
2991 independent reflections
1894 reflections with $I > 2\sigma(I)$
R_{int} = 0.038
θ_{max} = 26.4°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.112
S = 1.03
2991 reflections
212 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0393P)^2 + 0.548P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N4—H4A···N8^{i}	0.88 (3)	1.97 (3)	2.838 (3)	171 (2)

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

The positional and isotropic displacement parameters of the H atoms attached to N4 were refined freely. All other H atoms were placed in calculated positions, with C—H = 0.93 or 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*.

References

Awad, I., Abdel-Rahman, A. & Bakite, E. (1991). *J. Chem. Technol. Biotechnol.* **51**, 483–486.
Bruker (1998). *SMART*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (1999). *SAINT* and *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.

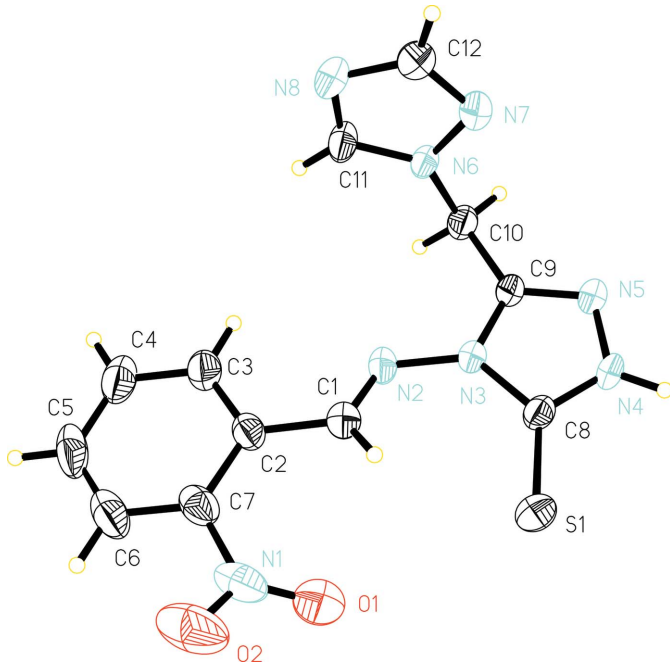


Figure 1
View of (I), with displacement ellipsoids drawn at the 40% probability level.

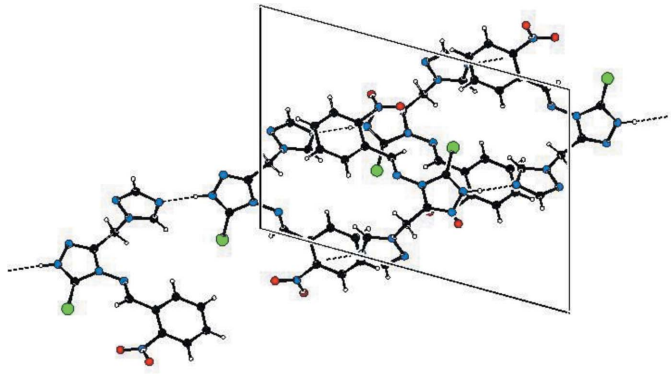


Figure 2
A packing diagram, viewed down the *b* axis. Hydrogen bonds are shown as dashed lines.

Escobar-Valderrama, J. L., Garcia-Tapia, J. H., Ramirez-Ortiz, J., Rosales, M. J., Toscano, R. A. & Valdes-Martinez, J. (1989). *Can. J. Chem.* **67**, 198–201.
Eweiss, N., Bahajaj, A. & Elsherbini, E. (1986). *J. Heterocycl. Chem.* **23**, 1451–1458.
Li, W.-H., Yu, G.-P., Liu, F.-Q., Hou, B.-R. & Yu, Z.-G. (2005). *Acta Cryst.* E**61**, o2058–o2060.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Xu, L.-Z., Yu, G.-P., Xu, F.-L. & Li, W.-H. (2005). *Acta Cryst.* E**61**, o2061–o2062.
Xu, L.-Z., Zhang, S.-S., Li, H.-J. & Jiao, K. (2002). *J. Chem. Res. Chin. Univ.* **18**, 284–286.