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Key indicators

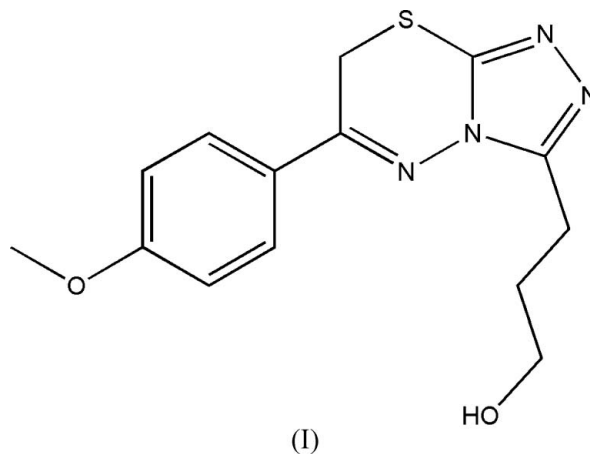
Single-crystal X-ray study
T = 298 K
Mean σ (C–C) = 0.004 Å
R factor = 0.062
wR factor = 0.137
Data-to-parameter ratio = 13.4For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.3-[6-(4-Methoxyphenyl)-7*H*-1,2,4-triazolo-
[3,4-*b*][1,3,4]thiadiazin-3-yl]propan-1-ol

In the title compound, C₁₄H₁₆N₄O₂S, the six-membered thiadiazine ring adopts a distorted boat conformation. O—H···N hydrogen bonds link the molecules into centrosymmetric dimers and enhance the stability of the crystal structure.

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Comment

As potentially biologically active reagents, 3,6-disubstituted-7*H*-1,2,4-triazolo[3,4-*b*][1,3,4]thiadiazines have received considerable attention over the past two decades (Zhou *et al.*, 2006; Nadkarni *et al.*, 2001). Triazoles fused with thiadiazines have been shown to exhibit antimicrobial (Feng *et al.*, 1992) and diuretic properties (Mohan & Anjaneyulu, 1987) and to act as photographic couplers (Holla *et al.*, 2001). In this paper, we report the synthesis and crystal structure of the title compound, (I).



In (I) (Fig. 1 and Table 1), the five-membered triazole ring is conjugated. The six-membered thiadiazine ring adopts a distorted boat conformation. O—H···N hydrogen bonds (Table 2) link the molecules into pairs around a center of symmetry (Fig. 2), enhancing the stability of the crystal structure.

Experimental

Carbon disulfide (13 ml) and hydrazine hydrate (24 ml) mixed with water (75 ml) were refluxed for 1 h at 363 K to form thiocarbonylhydrazide. 1,4-Butyrolactone (0.01 mol) and thiocarbonylhydrazide (0.01 mol) were refluxed in pyridine (40 ml) for 4 h to obtain 4-amino-5-mercapto-3-(3-hydroxypropyl)-1,2,4-triazole, (II), following the method of Xiong *et al.* (2002). To a solution of (II) (0.01 mol) in absolute ethanol (20 ml), was added 2-bromo-1-(4-methoxyphenyl)-ethanone (0.01 mol). The mixture was refluxed for 7 h. The solid

obtained on cooling was filtered, washed with cold water, dried and recrystallized from ethanol to give (I). The purified product was dissolved in 95% ethanol and single crystals were obtained after 4 d.

Crystal data

$C_{14}H_{16}N_4O_2S$
 $M_r = 304.38$
 Monoclinic, $P2_1/c$
 $a = 7.6166$ (7) Å
 $b = 12.8582$ (12) Å
 $c = 15.9198$ (13) Å
 $\beta = 111.228$ (4)°
 $V = 1453.3$ (2) Å³

$Z = 4$
 $D_x = 1.391$ Mg m⁻³
 Mo $K\alpha$ radiation
 $\mu = 0.23$ mm⁻¹
 $T = 298$ (2) K
 Block, colorless
 $0.25 \times 0.17 \times 0.16$ mm

Data collection

Bruker APEX area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Bruker, 2002)
 $T_{\min} = 0.944$, $T_{\max} = 0.959$

7493 measured reflections
 2556 independent reflections
 2288 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.031$
 $\theta_{\text{max}} = 25.0^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.062$
 $wR(F^2) = 0.137$
 $S = 1.20$
 2556 reflections
 191 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0504P)^2 + 0.734P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 0.24$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.31$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

S1—C10	1.731 (3)	N2—C10	1.367 (3)
S1—C9	1.808 (3)	N3—C10	1.297 (3)
N1—C8	1.283 (3)	N3—N4	1.403 (3)
N1—N2	1.393 (3)	N4—C11	1.299 (3)
N2—C11	1.365 (3)	C8—C9	1.510 (4)
C10—S1—C9	95.36 (13)	C5—C8—C9	118.9 (2)
C8—N1—N2	116.2 (2)	C8—C9—S1	113.99 (19)
C11—N2—C10	105.4 (2)	N3—C10—N2	110.7 (2)
C11—N2—N1	124.2 (2)	N3—C10—S1	128.8 (2)
C10—N2—N1	129.8 (2)	N2—C10—S1	120.42 (19)
C10—N3—N4	106.3 (2)	N4—C11—N2	109.4 (2)
C11—N4—N3	108.2 (2)	N4—C11—C12	127.0 (2)
N1—C8—C5	116.6 (2)	N2—C11—C12	123.6 (2)
N1—C8—C9	124.3 (2)		

Table 2

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$O2-H2\cdots N4^i$	0.82	2.08	2.892 (3)	173

Symmetry code: (i) $-x + 2, -y + 1, -z + 1$.

All H atoms were positioned geometrically and allowed to ride on their parent atoms at distances of $Csp^2-H = 0.93$ Å with $U_{\text{iso}} = 1.2U_{\text{eq}}(\text{parent atom})$, $Csp^3-H = 0.97$ Å with $U_{\text{iso}} = 1.5U_{\text{eq}}(\text{parent atom})$ and $O-H = 0.82$ Å with $U_{\text{iso}} = 1.5U_{\text{eq}}(\text{parent atom})$.

Data collection: SMART (Bruker, 2002); cell refinement: SAINT (Bruker, 2002); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine

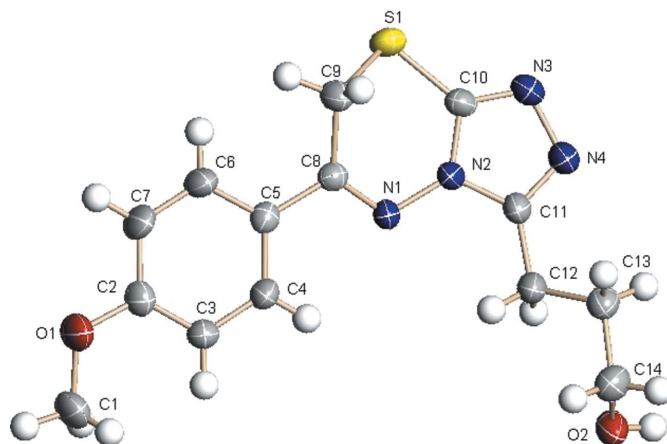


Figure 1

The molecular structure of (I) with the atom numbering, showing displacement ellipsoids at the 30% probability level.

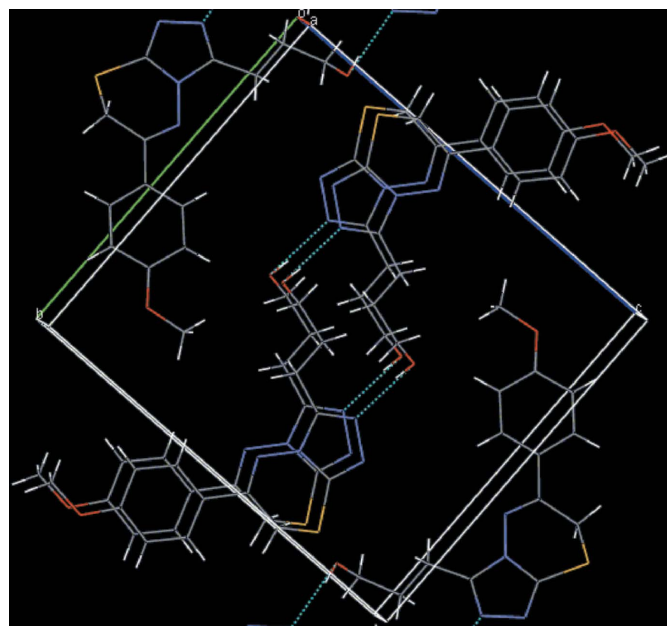


Figure 2

Packing diagram for (I), showing the hydrogen-bonded (dashed lines) dimers.

structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL (Bruker, 2002); software used to prepare material for publication: SHELXL97.

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References

- Bruker (2002). SMART (Version 5.62), SAINT (Version 6.02), SHELXTL (Version 6.10) and SADABS (Version 2.03). Bruker AXS Inc., Madison, Wisconsin, USA.
 Feng, X. M., Chen, R. & Yang, W. D. (1992). *Chem. J. Chin. Univ.* **13**, 187–194.
 Holla, B. S., Akberali, P. M. & Shivananda, M. K. (2001). *Il Farmaco*, **56**, 919–927.

- Mohan, J. & Anjaneyulu, G. S. R. (1987). *Pol. J. Chem.* **61**, 547–551.
- Nadkarni, B. A., Kamat, V. R. & Khadse, B. G. (2001). *Arzneim. Forsch.* **51**, 569–573.
- Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
- Xiong, Y., Zhang, L. X., Zhang, A. J. & Xu, D. J. (2002). *Synth. Commun.* **32**, 3455–3459.
- Zhou, S.-N., Zhang, L.-X., Jin, J.-Y., Xiao, H.-P. & Zhang, A.-J. (2006). *Acta Cryst. E* **62**, o605–o606.