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Key indicators

Single-crystal X-ray study
 $T = 298$ K
Mean $\sigma(\text{C}-\text{C}) = 0.003$ Å
 R factor = 0.031
 wR factor = 0.080
Data-to-parameter ratio = 13.6

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

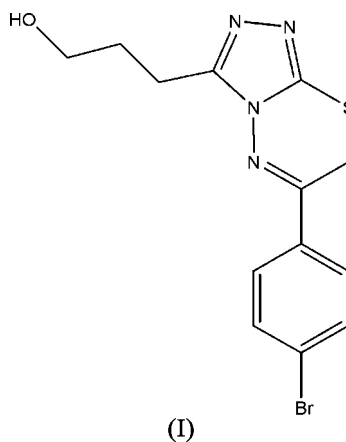
3-[6-(4-Bromophenyl)-7H-1,2,4-triazolo[3,4-b]- [1,3,4]thiadiazin-3-yl]propanol

The title compound, $\text{C}_{13}\text{H}_{13}\text{BrN}_4\text{OS}$, crystallizes with two molecules in the asymmetric unit. Intermolecular $\text{O}-\text{H}\cdots\text{N}$ hydrogen bonds enhance the stability of the crystal structure.

Received 12 January 2006
Accepted 23 February 2006

Comment

3,6-Disubstituted-7H-1,2,4-triazolo[3,4-b][1,3,4]thiadiazines are among various heterocycles that have received considerable attention over the past two decades as potentially biologically active reagents (Zhou *et al.*, 2006). Compound (I) was synthesized as part of our continuing research on this class of compounds and we report its structure here.



In (I), the five-membered triazole ring and the benzene ring are planar, while the six-membered thiadiazine ring is slightly distorted and may be regarded as having a half-chair conformation, atoms C8 and C7 (C20 and C21) being the out-of-plane atoms. The two independent molecules in the asymmetric unit have normal values for their bond lengths and angles (Fig. 1 and Table 1); these indicate that the five-membered triazole rings are conjugated (Allen *et al.*, 1987; Jin *et al.*, 2004). $\text{O}-\text{H}\cdots\text{N}$ hydrogen bonds link the molecules into pairs (Table 2).

Experimental

The key intermediate 4-amino-5-mercapto-3-(3-hydroxypropyl)-1,2,4-triazole, (II), was prepared from 1,4-butyrolactone and thiocarbonylhydrazide in pyridine solution, following the method of Xiong *et al.* (2002). The starting materials for the thiocarbonylhydrazide were carbon disulfide and hydrazine hydrate. To a solution of (II) (0.01 mol) in absolute ethanol (20 ml) was added 2-bromo-4'-bromoacetophenone (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

$C_{13}H_{13}BrN_4OS$
 $M_r = 353.24$
 Monoclinic, $P2_1/c$
 $a = 6.9002$ (4) Å
 $b = 15.740$ (1) Å
 $c = 25.533$ (1) Å
 $\beta = 91.132$ (1)°
 $V = 2772.6$ (3) Å³
 $Z = 8$

$D_x = 1.693$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 5420 reflections
 $\theta = 2.6$ – 25.0 °
 $\mu = 3.12$ mm⁻¹
 $T = 298$ (2) K
 Block, colorless
 $0.43 \times 0.26 \times 0.18$ mm

Data collection

Bruker SMART APEX area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Bruker, 2002)
 $T_{\min} = 0.392$, $T_{\max} = 0.579$
 14310 measured reflections

4895 independent reflections
 3926 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.024$
 $\theta_{\text{max}} = 25.0$ °
 $h = -8 \rightarrow 5$
 $k = -18 \rightarrow 18$
 $l = -26 \rightarrow 30$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.031$
 $wR(F^2) = 0.080$
 $S = 1.01$
 4895 reflections
 361 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0454P)^2 + 0.4483P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.002$
 $\Delta\rho_{\text{max}} = 0.28$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.43$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

| | | | |
|-----------|-------------|------------|-------------|
| S1–C9 | 1.738 (2) | S2–C22 | 1.735 (2) |
| S1–C8 | 1.811 (3) | S2–C21 | 1.825 (2) |
| N1–C7 | 1.295 (3) | N5–C20 | 1.289 (3) |
| N1–N2 | 1.385 (2) | N5–N6 | 1.391 (2) |
| N2–C9 | 1.365 (3) | N6–C22 | 1.368 (3) |
| N2–C10 | 1.381 (3) | N6–C23 | 1.374 (3) |
| N3–C9 | 1.296 (3) | N7–C22 | 1.301 (3) |
| N3–N4 | 1.407 (3) | N7–N8 | 1.409 (3) |
| N4–C10 | 1.306 (3) | N8–C23 | 1.299 (3) |
| C9–S1–C8 | 94.20 (12) | C22–S2–C21 | 93.63 (10) |
| C9–N2–C10 | 105.15 (19) | C22–N6–C23 | 105.40 (17) |
| C9–N2–N1 | 129.34 (18) | C22–N6–N5 | 128.67 (17) |
| C10–N2–N1 | 125.13 (18) | C23–N6–N5 | 125.48 (17) |
| N3–C9–N2 | 111.1 (2) | N7–C22–N6 | 110.42 (19) |
| N3–C9–S1 | 129.12 (19) | N7–C22–S2 | 130.25 (17) |
| N2–C9–S1 | 119.69 (17) | N6–C22–S2 | 119.30 (16) |

Table 2

Hydrogen-bond geometry (Å, °).

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|---|-------|-------------|-------------|---------------|
| O2–H2 ⁱ ···N3 ⁱ | 0.82 | 2.16 | 2.956 (3) | 164 |
| O1–H1 ⁱⁱ ···N7 ⁱⁱ | 0.82 | 2.11 | 2.911 (3) | 166 |

Symmetry codes: (i) $x + 1, y, z$; (ii) $x - 1, y, z$.

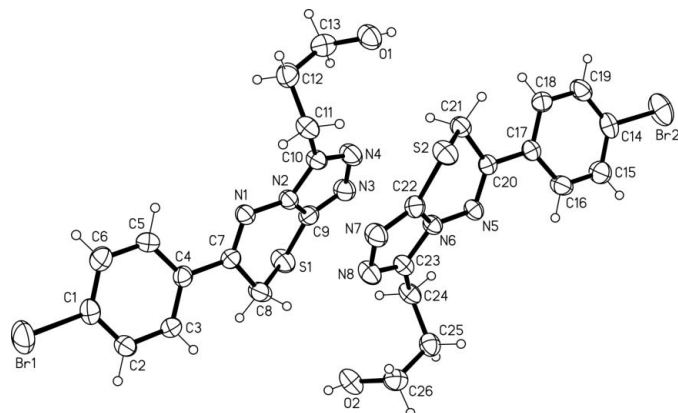


Figure 1

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

All H atoms were positioned geometrically and allowed to ride on their parent atoms, with C–H = 0.93 and 0.97 Å, O–H = 0.82 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C}, \text{O})$.

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

This work was supported by the Zhejiang Provincial Natural Science Foundation of China (No. M203149).

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