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6-(2,4-Difluorophenyl)-3-(3-hydroxypropyl)-7*H*-1,2,4-triazolo[3,4-*b*][1,3,4]thiadiazine

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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 = 0.166
Data-to-parameter ratio = 12.8

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

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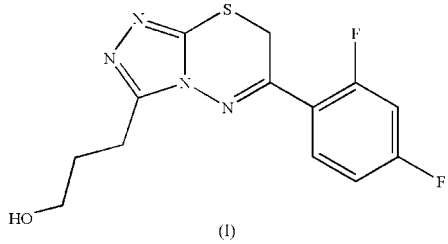
In the title compound, $C_{13}H_{12}F_2N_4OS$, the thiadiazine ring adopts a screw-boat conformation. Weak O—H···N hydrogen bonds are found in the crystal structure. These link the molecules into a zigzag chain.

Comment

1,2,4-Triazoles fused with six-membered ring systems are found to possess diverse applications in the fields of medicine, agriculture and industry. The commonly known systems are triazoles fused with pyridine, pyridazine, pyrimidine, pyrazines and triazines. A literature survey reveals that there are not many examples of triazoles fused with thiadiazines. Moreover, a large number of triazolothiazines have been shown to exhibit antimicrobial (Feng *et al.*, 1992) and diuretic (Mohan & Anjaneyulu, 1987) properties and to act as photographic couplers (Holla *et al.*, 2001). On the other hand, much attention has been paid to partially fluorinated heterocyclic compounds, because of their unique chemical, physical and biological properties (Shaaban & Fuchigami, 2002). In this paper, we report the synthesis and crystal structure of the title compound, (I).



(I)

In compound (I), the triazole and benzene rings are planar, while the six-membered thiadiazine ring is distorted from planarity and may be regarded as having a screw-boat conformation (Fig. 1). Atoms C9 and S1 deviate by 0.510 (1) and 1.119 (1) Å, respectively, from the mean plane through atoms C7, C8, N1 and N2. The bond lengths are comparable to those in related compounds (Sert *et al.*, 2003; Kou *et al.*, 2004). Likewise, the triazole bond lengths exhibit normal values (Allen *et al.*, 1987; Jin *et al.*, 2004; Table 1). Intermolecular O—H···N hydrogen bonds are found in the crystal structure and these link the molecules into a zigzag chain (Table 2 and Fig. 2).

Experimental

4-Amino-3-(3-hydroxypropyl)-5-mercapto-1,2,4-triazole, (II), was prepared from 1,4-butyrolactone and thiocarbohydrazide in a pyri-

dine solution, following the method of Xiong *et al.* (2002). The starting materials for the thiocarbohydrazide were carbon disulfide and hydrazine hydrate. To a solution of (II) (0.001 mol) in absolute ethanol was added 2-bromo-2′,4′-difluoroacetophenone (0.001 mol). The mixture was refluxed for 7 h. The solid obtained on cooling was filtered off, washed with cold water, dried and recrystallized from ethanol to give compound (I). The purified product was dissolved in 95% ethanol and kept at room temperature for 5 d whereupon single crystals of (I) were formed.

Crystal data

$C_{13}H_{12}F_2N_4OS$
M_r = 310.33
Monoclinic, $P2_1/c$
a = 16.0230 (14) Å
b = 11.0368 (10) Å
c = 7.8738 (7) Å
β = 101.174 (2)°
V = 1366.0 (2) Å^3
Z = 4
D_x = 1.509 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2391 reflections
θ = 2.3–25.1°
μ = 0.26 mm^{-1}
T = 298 (2) K
Block, colorless
0.38 × 0.28 × 0.08 mm

Data collection

Bruker APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.906, T_{max} = 0.969
7092 measured reflections
2454 independent reflections
2269 reflections with $I > 2\sigma(I)$
R_{int} = 0.024
θ_{max} = 25.2°
$h = -19 \rightarrow 18$
$k = -13 \rightarrow 10$
$l = -9 \rightarrow 8$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.062
$wR(F^2)$ = 0.166
S = 1.26
2454 reflections
191 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0803P)^2 + 0.4963P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

S1—C9	1.733 (3)	N2—C9	1.372 (3)
S1—C8	1.812 (3)	N3—C9	1.298 (4)
N1—C7	1.277 (4)	N3—N4	1.404 (4)
N1—N2	1.393 (3)	N4—C10	1.303 (4)
N2—C10	1.363 (4)	C7—C8	1.505 (4)
C9—S1—C8	93.56 (13)	N3—C9—N2	110.6 (2)
C10—N2—C9	105.8 (2)	N3—C9—S1	129.0 (2)
C10—N2—N1	124.6 (2)	N2—C9—S1	120.3 (2)
C9—N2—N1	129.1 (2)		
C1—C6—C7—N1	137.2 (3)	N4—C10—C11—C12	−13.2 (5)
C5—C6—C7—N1	−47.1 (4)	N2—C10—C11—C12	166.4 (3)
C1—C6—C7—C8	−50.0 (4)	C10—C11—C12—C13	175.7 (3)
C5—C6—C7—C8	125.7 (3)	C11—C12—C13—O1	60.4 (4)

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···N4^{i}	0.82	2.02	2.819 (4)	166

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

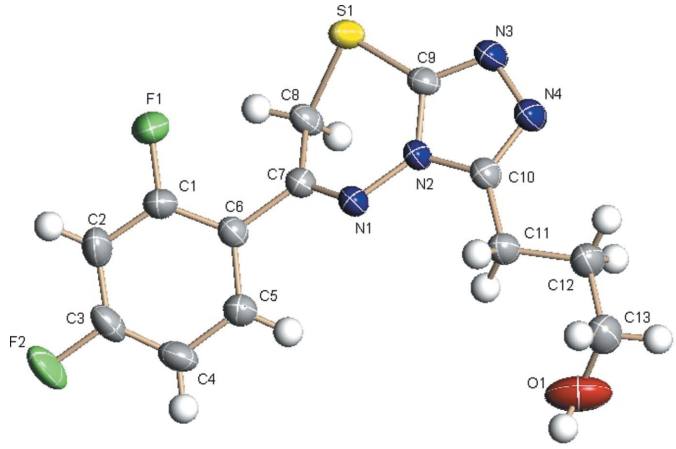


Figure 1
The molecular structure of (I) with the atom numbering, showing displacement ellipsoids drawn at the 30% probability level.

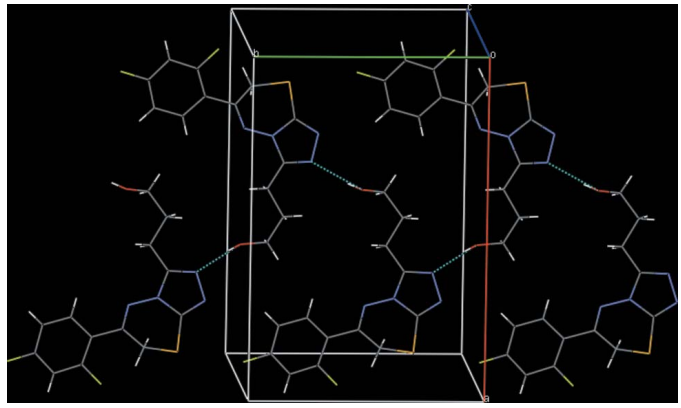


Figure 2
A zigzag chain formed by hydrogen-bonding interactions (shown as dashed lines).

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_{iso}(H) = 1.2U_{eq}(C), Csp^3—H = 0.97 Å with U_{iso}(H) = 1.5U_{eq}(C) and O—H = 0.82 Å with U_{iso}(H) = 1.5U_{eq}(O). The poor quality of the crystals may be responsible for the rather high R factor.

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: *SHELXTL* (Bruker, 2002); software used to prepare material for publication: *SHELXL97*.

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