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6-(2,4-Difluorophenyl)-3-propyl-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) = 0.005 Å
R factor = 0.064
wR factor = 0.132
Data-to-parameter ratio = 13.0

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

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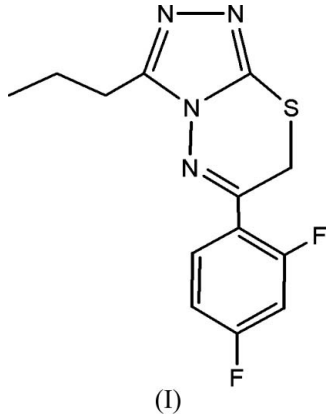
The title compound, $C_{13}H_{12}F_2N_4S$, was prepared by the reaction of propanoic acid and thiocarbohydrazide. The bond lengths in the triazole ring show normal values.

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). In recent years, much attention has been paid to partially fluorinated heterocyclic compounds, because of their unique chemical, physical and biological properties (Shaaban & Fuchigami, 2002). The development of efficient methods for the selective fluorination of heterocycles is, therefore, of much importance. In this paper, we report the synthesis and crystal structure of the title compound $C_{13}H_{12}F_2N_4S$, (I).



(I)

In (I), the five-membered triazole ring (N2–N4/C9/C10) and the benzene ring (C1–C6) are each essentially planar, while the six-membered thiadiazine ring (N1/N2/C7–C9/S1) is distorted from planarity, with an r.m.s deviation of 0.238 Å, and may be regarded as having a screw-boat conformation (Fig. 1). Both the S—C (mean 1.771 Å) and C—N bond lengths are in agreement with the values in related complexes (Sert *et al.*, 2003; Xiang *et al.*, 2004). The bond lengths in the triazole ring show normal values (Allen *et al.*, 1987; Jin *et al.*, 2004; Table 1).

Experimental

4-Amino-5-mercapto-3-propyl-1,2,4-triazole was prepared by the reaction of butyric acid and thiocarbohydrazide, following the literature method of Francesco *et al.*, 1997. To a solution of 4-amino-5-mercapto-3-propyl-1,2,4-triazole (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, 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; colourless single crystals of (I) were formed.

Crystal data

$C_{13}H_{12}F_2N_4S$
M_r = 294.33
Monoclinic, $P2_1/c$
a = 12.6930 (14) Å
b = 14.5268 (16) Å
c = 7.2715 (8) Å
β = 100.667 (2)°
V = 1317.6 (3) Å^3
Z = 4
D_x = 1.484 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1299 reflections
θ = 2.8–24.1°
μ = 0.26 mm^{-1}
T = 298 (2) K
Plate, colourless
0.21 × 0.19 × 0.08 mm

Data collection

Bruker APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.937, T_{max} = 0.969
6940 measured reflections
2375 independent reflections
1974 reflections with $I > 2\sigma(I)$
R_{int} = 0.033
θ_{max} = 25.3°
$h = -11 \rightarrow 15$
$k = -16 \rightarrow 17$
$l = -8 \rightarrow 8$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.064
$wR(F^2)$ = 0.132
S = 1.16
2375 reflections
182 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0433P)^2 + 0.8585P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

S1—C9	1.733 (3)	N2—C10	1.371 (4)
S1—C8	1.809 (3)	N3—C9	1.302 (4)
N1—C7	1.286 (3)	N3—N4	1.406 (4)
N1—N2	1.383 (3)	N4—C10	1.308 (4)
N2—C9	1.367 (4)		
C9—S1—C8	93.83 (14)	C10—N4—N3	108.2 (2)
C7—N1—N2	115.7 (2)	N1—C7—C4	117.1 (3)
C9—N2—C10	105.4 (2)	N1—C7—C8	123.4 (3)
C9—N2—N1	129.5 (2)	C7—C8—S1	112.2 (2)
C10—N2—N1	124.1 (2)	N3—C9—S1	129.5 (3)
C9—N3—N4	106.2 (3)	N2—C9—S1	119.4 (2)

All H atoms were positioned geometrically and allowed to ride on their parent atoms at distances of Csp^2—H = 0.93 Å and Csp^3—H = 0.96 or 0.97 Å, with U_{iso}(H) = 1.2U_{eq}(parent atom) or 1.5U_{eq}(methyl parent atom).

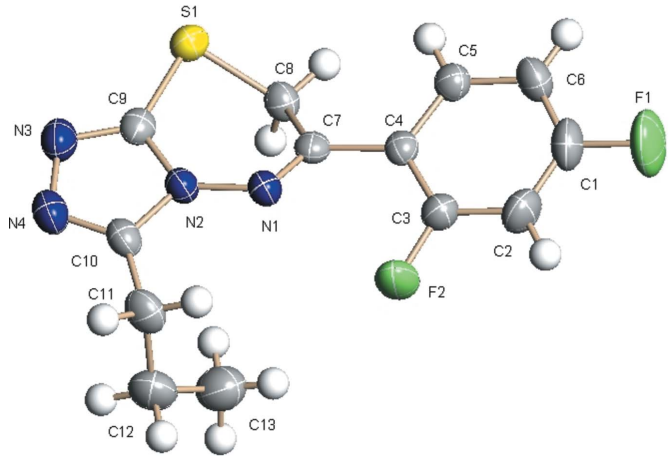


Figure 1
The molecular structure of (I) with the atom-numbering scheme, showing displacement ellipsoids at the 30% probability level.

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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