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6-(4-Fluorophenyl)-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) = 0.004 Å
R factor = 0.066
wR factor = 0.143
Data-to-parameter ratio = 13.7

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

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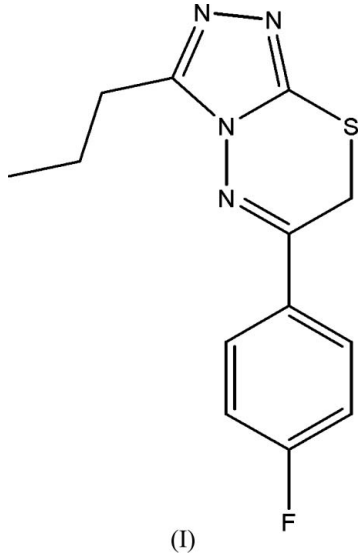
The title compound, $C_{13}H_{13}FN_4S$,was prepared by the reaction of 4-amino-5-mercapto-3-propyl-1,2,4-triazole and 2-bromo-4′-fluoroacetophenone. The bond lengths and angles show normal values. The thiadiazine ring is non-planar.

Comment

1,2,4-Triazoles fused with six-membered-ring systems have diverse applications in the fields of medicine, agriculture and industry. 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; these may exhibit antimicrobial (Feng *et al.*, 1992) and diuretic (Mohan & Anjaneyulu, 1987) properties and 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). The development of efficient methods for selective fluorination of heterocycles is, therefore, of much importance. In this paper, we report the synthesis and crystal structure of the title compound, (I).



(I)

In (I), the six-membered thiadiazine ring N1/N2/C7/C8/C9/S1 (Fig. 1) is non-planar; atoms C8 and S1 deviate from the mean plane by −0.398 (2) and 0.326 (1) Å, respectively. The S—C and C—N bond lengths (Table 1) are comparable with those observed in related compounds (Sert *et al.*, 2003; Zou *et al.*, 2004). In the triazole rings, the bond lengths and angles (Table 1) show normal values (Allen *et al.*, 1987; Jin *et al.*, 2004).

Experimental

4-Amino-5-mercapto-3-propyl-1,2,4-triazole was prepared by the reaction of propanoic 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-4-fluoroacetophenone (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 (m.p. 432–434 K). IR (KBr, cm^{-1}): 3068 (Ar–H), 2924 (CH_2), 1618, 1597, 1461 (C═C, C═N), 1282 (N—N═C), 1095 (C—F), 834 (disubstituted benzene), 686 (C—S—C). ^{1}H NMR (dimethyl sulfoxide-d_6): δ8.10 (*dd*, 2H, Ar–H), 7.43 (*dd*, 2H, A–H), 4.46 (*s*, 2H, SCH_2), 2.91 (*t*, 2H, CH_2), 1.77–1.75 (*m*, 2H, CH_2), 0.97 (*t*, 3H, CH_3). ^{13}C NMR (dimethylsulfoxide-d_6): δ 166.20, 162.87, 155.14, 153.61, 141.19, 130.47, 130.35, 129.96, 116.22, 23.18, 25.78, 19.76, 13.64. Elemental analysis found for $C_{13}H_{13}FN_4S$: C 56.49, H 4.77, N 20.38%; calculated: C 56.44, H 4.74, N 20.34%

Crystal data

$C_{13}H_{13}FN_4S$
M_r = 276.33
Monoclinic, $P2_1/c$
a = 12.5849 (9) Å
b = 14.5963 (10) Å
c = 7.2955 (5) Å
β = 101.825 (1)°
V = 1311.69 (16) Å^3
Z = 4
D_x = 1.399 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3328 reflections
θ = 2.5–24.1°
μ = 0.25 mm^{-1}
T = 298 (2) K
Block, colourless
0.29 × 0.19 × 0.11 mm

Data collection

Bruker APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.931, T_{max} = 0.968
12592 measured reflections
2374 independent reflections
2180 reflections with $I > 2\sigma(I)$
R_{int} = 0.030
θ_{max} = 25.2°
$h = -15 \rightarrow 15$
$k = -17 \rightarrow 17$
$l = -8 \rightarrow 8$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.066
$wR(F^2)$ = 0.143
S = 1.25
2374 reflections
173 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0522P)^2 + 0.9704P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.40 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 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	1.296 (4)
N1—C7	1.287 (3)	N3—N4	1.406 (4)
N1—N2	1.387 (3)	N4—C10	1.301 (4)
N2—C9	1.365 (4)		
C9—S1—C8	93.92 (14)	C10—N2—N1	124.4 (2)
C7—N1—N2	116.0 (2)	C9—N3—N4	106.3 (2)
C9—N2—C10	105.5 (2)	C10—N4—N3	108.3 (2)
C9—N2—N1	129.1 (2)		

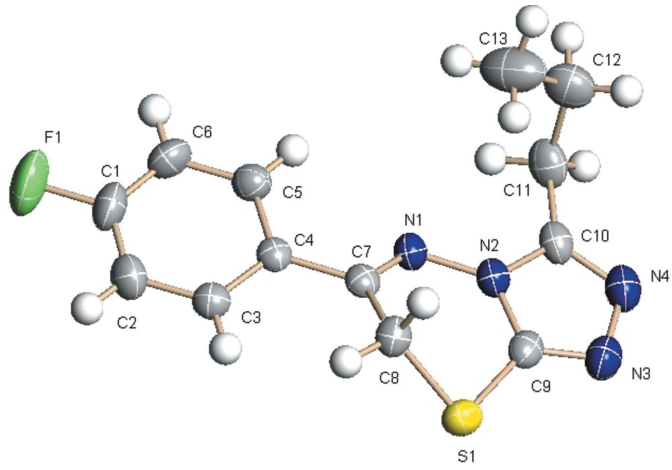


Figure 1
The molecular structure of (I), showing the atom-numbering scheme, with displacement ellipsoids drawn at the 30% probability level.

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}=1.2U_{eq}(parent atom), and Csp^3—H = 0.96 or 0.97 Å with U_{iso}=1.5U_{eq}(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 structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: SHELXLTL (Bruker, 2002); software used to prepare material for publication: *SHELXL97*.

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