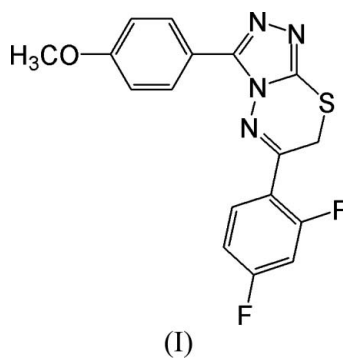


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Key indicators

Single-crystal X-ray study
 $T = 298\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.003\text{ \AA}$
 R factor = 0.050
 wR factor = 0.119
Data-to-parameter ratio = 12.3For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.6-(2,4-Difluorophenyl)-3-(4-methoxyphenyl)-
7H-1,2,4-triazolo[3,4-b][1,3,4]thiadiazineIn the title compound, $\text{C}_{17}\text{H}_{12}\text{F}_2\text{N}_4\text{OS}$, the thiadiazine ring is non-planar and adopts a half-chair conformation. The crystal packing is stabilized by $\text{C}-\text{H}\cdots\text{N}$, $\text{C}-\text{H}\cdots\text{O}$ and $\text{C}-\text{H}\cdots\text{F}$ intermolecular hydrogen-bonding interactions.Received 19 October 2005
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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 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).In compound (I), the five-membered triazole ring (N2–N4/C9,C10) and the benzene rings (C1–C6 and C11–C16) 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 0.251 Å (Fig. 1). In this half-chair conformation, atoms C8 and S1 deviate by -0.401 (2) and 0.330 (1) Å, respectively, from the plane through atoms C7, N1, N2 and C9. Both the S–C (mean 1.772 Å) and C–N bond lengths are comparable with those in related compounds (Sert *et al.*, 2003; Xiang *et al.*, 2004). In the triazole ring, the bond lengths show normal values (Allen *et al.*, 1987; Jin *et al.*, 2004; Table 1). The dihedral angle between the N2–N4/C9/C10 and C11–C16 rings is 17.7 (1)°, and that between the C1–C6 and C11–C16 rings is 13.9 (1)°. In the crystal structure, weak $\text{C}-\text{H}\cdots\text{N}$, $\text{C}-\text{H}\cdots\text{O}$

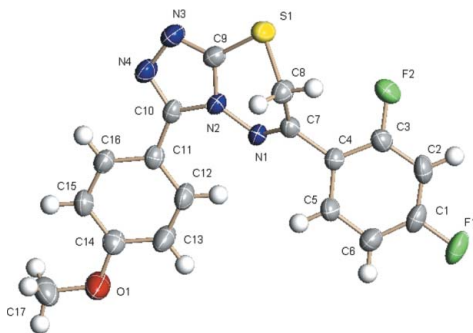


Figure 1
The molecular structure of (I), showing the atomic numbering. Displacement ellipsoids are drawn at the 30% probability level.

and C—H···F intermolecular hydrogen-bonding interactions link the molecules into a two-dimensional network (Table 2).

Experimental

4-Amino-5-mercapto-3-(4-methoxyphenyl)-1,2,4-triazole was prepared from 4-methoxybenzoic acid hydrazide, whose starting material was 4-methoxybenzoic acid, following the literature method of Zhang *et al.* (1990). To a solution of 4-amino-5-mercapto-3-(4-methoxyphenyl)-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 and colourless single crystals of (I) were formed (m.p. 454–455 K). Spectroscopic analysis: IR (KBr, ν , cm^{-1}): 3055, 3001 (Ar—H), 2922 (CH_2), 1610 ($\text{C}=\text{N}$), 1483, 1296 (N—N=C), 1176 (C—F), 834, 731 (di- and trisubstituted benzene), 691 (C—S—C); ^1H NMR (dimethylsulfoxide- d_6 , δ , p.p.m.): 7.86–7.99 (q , 3H, Ar—H), 7.50–7.57 (t , 1H, Ar—H), 7.27–7.32 (t , 1H, Ar—H), 7.03–7.13 (q , 2H, Ar—H), 4.34 (s , 2H, CH_2), 3.82 (s , 3H, OCH_3); ^{13}C NMR (dimethylsulfoxide- d_6 , p.p.m.): 166.80, 162.70, 159.10, 152.94, 151.62, 148.23, 141.93, 132.26, 129.70, 119.71, 118.31, 112.89, 105.47, 55.48, 25.95. Elemental analysis for $\text{C}_{17}\text{H}_{12}\text{F}_2\text{N}_4\text{OS}$: C 56.53, H 3.56, N 15.92%; calculated: C 56.92, H 3.38, N 15.69%.

Crystal data

$\text{C}_{17}\text{H}_{12}\text{F}_2\text{N}_4\text{OS}$
 $M_r = 358.37$
Monoclinic, $P2_1/c$
 $a = 12.9203$ (7) Å
 $b = 13.9490$ (11) Å
 $c = 8.7609$ (10) Å
 $\beta = 92.502$ (1)°
 $V = 1577.4$ (2) Å³
 $Z = 4$

$D_x = 1.509$ Mg m⁻³
Mo $K\alpha$ radiation
Cell parameters from 2351 reflections
 $\theta = 2.7$ – 24.4 °
 $\mu = 0.24$ mm⁻¹
 $T = 298$ (2) K
Block, colourless
0.30 × 0.22 × 0.13 mm

Data collection

Bruker APEX area-detector diffractometer
 φ and ω scans
Absorption correction: multi-scan (SADABS; Bruker, 2002)
 $T_{\min} = 0.931$, $T_{\max} = 0.959$
8091 measured reflections

2782 independent reflections
2387 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.026$
 $\theta_{\max} = 25.0$ °
 $h = -15 \rightarrow 15$
 $k = -16 \rightarrow 8$
 $l = -10 \rightarrow 10$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.050$
 $wR(F^2) = 0.119$
 $S = 1.11$
2782 reflections
227 parameters
H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0534P)^2 + 0.5462P]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.23 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.25 \text{ e \AA}^{-3}$

Table 1

Selected bond lengths (Å).

S1—C9	1.731 (2)	N1—N2	1.389 (2)
S1—C8	1.813 (2)	N2—C9	1.375 (3)
O1—C14	1.365 (3)	N2—C10	1.381 (3)
O1—C17	1.423 (3)	N3—C9	1.303 (3)
N1—C7	1.284 (3)	N3—N4	1.398 (3)

Table 2

Hydrogen-bond geometry (Å, °).

$D\text{—H}\cdots A$	$D\text{—H}$	$\text{H}\cdots A$	$D\cdots A$	$D\text{—H}\cdots A$
C6—H6···O1 ⁱ	0.93	2.50	3.241 (3)	137
C8—H8A···F2	0.97	2.44	2.976 (3)	115
C8—H8A···N4 ⁱⁱ	0.97	2.48	3.394 (3)	156
C12—H12···N1	0.93	2.42	3.042 (3)	124
C15—H15···F1 ⁱⁱⁱ	0.93	2.54	3.458 (3)	171
C16—H16···N4	0.93	2.54	2.862 (3)	101

Symmetry codes: (i) $-x + 2, y + \frac{1}{2}, -z + \frac{3}{2}$; (ii) $-x + 1, y + \frac{1}{2}, -z + \frac{1}{2}$; (iii) $x, y - 1, z$.

All H atoms were positioned geometrically [C—H = 0.93 (aromatic), 0.96 (methyl) and 0.97 Å (methylene)] and allowed to ride on their parent atoms, with $U_{\text{iso}} = 1.2\text{--}1.5U_{\text{eq}}$ (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: SHELXTL (Bruker, 2002); software used to prepare material for publication: SHELXL97.

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