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6-(Biphenyl-4-yl)-3-(4-ethoxyphenyl)-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 = 0.069
wR factor = 0.162
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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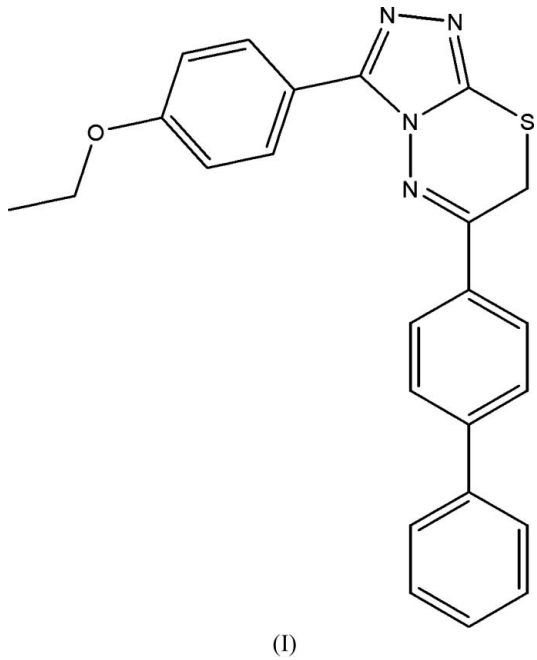
In the title compound, $C_{24}H_{20}N_4OS$, the triazole ring is planar, whereas the thiadiazine may be regarded as having a half-chair conformation.

Comment

3,6-Disubstituted 7*H*-1,2,4-triazolo[3,4-*b*][1,3,4]thiadiazines are among various heterocycles that have been of interest for over two decades due to their biological activities (Feng *et al.*, 1992; Hui *et al.*, 2000; Mohan & Anjaneyulu, 1987; Turan *et al.*, 1999; Nadkarni *et al.*, 2001). Biphenyl itself has been used as an antiseptic (Xie *et al.*, 1994), and some heterocycles with biphenyl groups are used as clinical medicines (Feng *et al.*, 2000). We have attached a biphenyl group to 7*H*-1,2,4-triazolo[3,4-*b*][1,3,4]thiadiazine in the hope of producing compounds with new biological activities. We report here the synthesis and crystal structure of the title compound, (I).



(I)

In (I), the triazole ring and the benzene rings are each essentially planar, while the thiadiazine ring is slightly distorted from planarity and may be regarded as having a half-chair conformation (Fig. 1). Both the S—C (mean distance 1.767 Å) and the C—N bond lengths are in line with those found in related complexes with conjugated triazole rings (Zou *et al.*, 2004, Allen *et al.*, 1987; Table 1). The dihedral angles between the thiadiazine ring and the triazole ring, and between the benzene rings (C1–C6 and C7–C12) are both

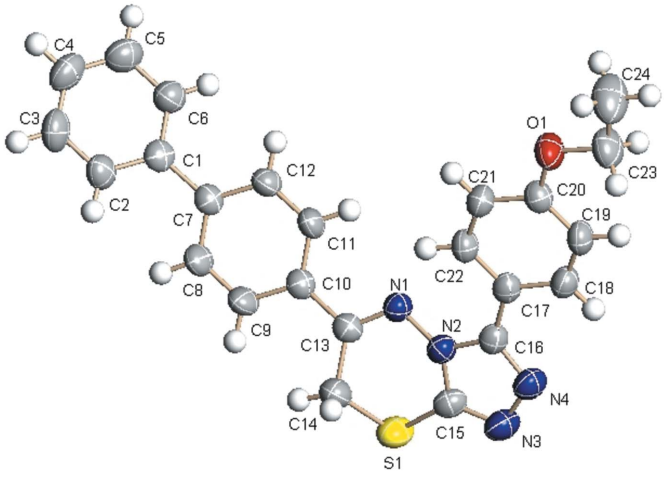


Figure 1
The molecular structure of (I), showing the atom numbering and displacement ellipsoids drawn at the 30% probability level.

15.9 (2)°, and that between the triazole ring and the third benzene ring (C17–C22) is 16.2 (1)°. The biphenyl and 4-ethoxyphenyl rings are located on the same side of the condensed heterocycle and are almost perpendicular to one another, resembling the two front claws of a crab.

Experimental

The key intermediate 4-amino-5-mercapto-3-(4-ethoxyphenyl)-1,2,4-triazole, (II), was prepared from 4-ethoxybenzoic acid hydrazide, whose starting material was 4-ethoxybenzoic acid, following the literature method of Zhang *et al.* (1990). The starting materials for the thiocarbohydrazide were carbon disulfide and hydrazine hydrate. To a solution of (II) (0.01 mol) in absolute ethanol (20 ml) was added 4-phenylbromoacetophenone (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 6 d.

Crystal data

$C_{24}H_{20}N_4OS$
M_r = 412.50
Monoclinic, $P2_1/c$
a = 13.129 (1) Å
b = 17.3638 (13) Å
c = 9.2648 (7) Å
β = 101.189 (1)°
V = 2071.9 (3) Å^3
Z = 4
D_x = 1.322 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2478 reflections
θ = 2.6–24.3°
μ = 0.18 mm^{-1}
T = 298 (2) K
Block, colorless
0.35 × 0.21 × 0.16 mm

Data collection

Bruker APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.945, T_{max} = 0.970
10919 measured reflections
3720 independent reflections
3130 reflections with $I > 2\sigma(I)$
R_{int} = 0.027
θ_{max} = 25.2°
$h = -14 \rightarrow 15$
$k = -19 \rightarrow 20$
$l = -11 \rightarrow 10$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.069
$wR(F^2)$ = 0.162
S = 1.19
3720 reflections
272 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0638P)^2 + 0.7764P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.46 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

S1—C15	1.730 (3)	N2—C16	1.377 (3)
S1—C14	1.804 (3)	N3—C15	1.299 (4)
N1—C13	1.285 (3)	N3—N4	1.391 (4)
N1—N2	1.388 (3)	N4—C16	1.314 (4)
N2—C15	1.373 (4)		
C15—S1—C14	93.94 (15)	N3—C15—N2	111.2 (3)
C15—N2—C16	104.8 (2)	N3—C15—S1	129.0 (2)
C15—N2—N1	127.8 (2)	N2—C15—S1	119.9 (2)
C16—N2—N1	125.7 (2)		

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

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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