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6-(4-Chlorophenyl)-3-(2-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) = 0.005 Å
R factor = 0.063
wR factor = 0.157
Data-to-parameter ratio = 13.4

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

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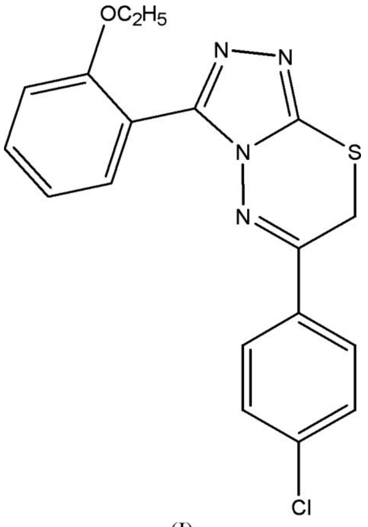
The title compound, $C_{18}H_{15}ClN_4OS$, was prepared by the reaction of 4-amino-3-(2-ethoxyphenyl)-5-mercapto-1,2,4-triazole and 2-bromo-4′-chloroacetophenone. The dihedral angle between the triazole ring and the benzene ring bearing the ethoxy substituent is 17.7 (1)°.

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 (Turan *et al.*, 1999; Invidiata *et al.*, 1996; Chadha *et al.*, 1998). The commonly known systems are triazoles fused with pyridine, pyridazine, pyrimidine, pyrazines and triazines. 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, a literature survey reveals that there are not many examples of triazoles fused with thiadiazines. Under the expectation that attaching 2-ethoxyphenyl and 4-chlorophenyl groups to 7*H*-1,2,4-triazolo[3,4-*b*][1,3,4]thiadiazines can produce some new biological activities, we have synthesized the title compound, (I).



(I)

In (I), the six-membered thiadiazine ring, N1/N2/C9/S1/C8/C7, is distorted from planarity, with an r.m.s deviation of 0.251 Å and the ring exists in a half-chair conformation; atoms C8 and S1 deviate −0.401 (2) and 0.330 (1) Å, respectively, from the mean plane (Fig. 1). Both S—C (with a mean distance 1.770 Å) and C—N bond lengths are comparable to those in related compounds 6-(2,4-difluorophenyl)-3-(3-methylphenyl)-7*H*-1,2,4-triazolo[3,4-*b*][1,3,4]thiadiazine (Xiang *et al.*,

2004) and 3-(3-hydroxypropyl)-6-(*p*-tolyl)-7*H*-1,2,4,-triazolo[3,4-*b*][1,3,4]thiadiazine (Zou *et al.*, 2004). The bond lengths in the triazole ring show normal values (Allen *et al.*, 1987; Jin *et al.*, 2004; Table 1). The dihedral angle between the triazole and C11–C16 benzene rings is 17.7 (1)° and that between the C1–C6 and C11–C16 benzene rings is 13.9 (1)°.

Experimental

4-Amino-5-mercapto-3-(2-ethoxyphenyl)-1,2,4-triazole was prepared from 2-ethoxybenzoic acid hydrazide according to the literature method of Zhang *et al.* (1990). To a solution of 4-amino-5-mercapto-3-(2-ethoxyphenyl)-1,2,4-triazole (0.001 mol) in absolute ethanol was added 2-bromo-4′-chloroacetophenone (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, whereupon colourless single crystals were formed.

Crystal data

$C_{18}H_{15}ClN_4OS$
$M_r = 370.85$
Monoclinic, $P2_1/c$
$a = 12.8542$ (9) Å
$b = 13.3382$ (11) Å
$c = 10.3438$ (11) Å
$\beta = 99.488$ (2)°
$V = 1749.2$ (3) Å^3
$Z = 4$

$D_x = 1.408$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2432 reflections
$\theta = 2.2$–24.8°
$\mu = 0.35$ mm^{-1}
$T = 298$ (2) K
Block, colorless
0.41 × 0.15 × 0.13 mm

Data collection

Bruker APEX area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
$T_{\rm min} = 0.869$, $T_{\rm max} = 0.956$
8700 measured reflections

3053 independent reflections
2402 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.033$
$\theta_{\rm max} = 25.0°$
$h = -15 \rightarrow 15$
$k = -15 \rightarrow 10$
$l = -12 \rightarrow 12$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.063$
$wR(F^2) = 0.157$
$S = 1.06$
3053 reflections
227 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0642P)^2 + 1.1848P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.38$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.28$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cl1—C1	1.741 (3)	N2—C9	1.358 (4)
S1—C9	1.734 (4)	N2—C10	1.378 (4)
S1—C8	1.806 (4)	N3—C9	1.299 (4)
N1—C7	1.287 (4)	N3—N4	1.404 (4)
N1—N2	1.392 (3)	N4—C10	1.308 (4)
C9—S1—C8	95.27 (16)	C10—N4—N3	107.5 (3)
C7—N1—N2	115.8 (3)	N1—C7—C8	123.8 (3)
C9—N2—C10	105.1 (3)	N3—C9—N2	111.1 (3)
C9—N2—N1	128.4 (3)	N3—C9—S1	128.7 (3)
C10—N2—N1	124.2 (3)	N2—C9—S1	120.1 (3)
C9—N3—N4	106.8 (3)	N4—C10—N2	109.5 (3)
N2—N1—C7—C8	−3.9 (4)	C9—S1—C8—C7	−48.5 (3)
N1—C7—C8—S1	45.2 (4)	C8—S1—C9—N2	24.8 (3)

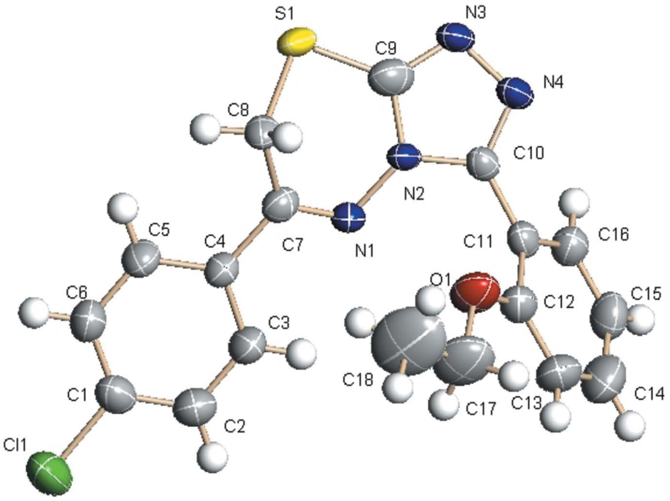


Figure 1
The molecular structure of (I), with the atom-numbering, showing displacement ellipsoids at the 30% probability level.

All H atoms were positioned geometrically and allowed to ride on their parent atoms at distances of Csp^2—H = 0.93 Å and Csp^3—H = 0.96 or 0.97 Å, with $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(parent atom) and 1.5$U_{\rm eq}$(parent methyl atom). The short C17—C18 bond and the large displacement parameter of atom C18 are probably the result of unresolved disorder or a large amplitude libration of the ethyl group around the O1—C17 bond axis.

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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