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3-Phenyl-4a,5-dihydro-1,2,4-triazolo[3,4-*b*]-[1,3]benzothiazine

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.044
wR factor = 0.146
Data-to-parameter ratio = 12.2

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

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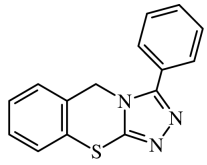
In the crystal structure of the title compound, $C_{15}H_{11}N_3S$, the thiazine ring adopts a boat conformation. The dihedral angle between the triazole and phenyl rings is 34.3 (1)°. The packing of the molecule is stabilized by π–π interactions.

Comment

Triazole derivatives exhibit antimicrobial (Habib *et al.*, 1997), antiviral (Ergen *et al.*, 1996), anti-HIV-1 (Invidiata *et al.*, 1996), antifungal, antimycobacterial and anticonvulsant (Gulerman *et al.*, 1997) activities. Triazole is also a highly potent eosinophilia inhibitor (Naito *et al.*, 1996), fungicide (Croften, 1996) and herbicide (Tada *et al.*, 1995). Some of the benzothiazine derivatives are most active against the Gram-positive *Bacillus subtilis* (Armenise *et al.*, 1991). Triazole derivatives also possesses neuroleptic activities (Grol & Rollema, 1975). The X-ray crystal structure analysis of the title compound, (I), was carried out as part of our studies on triazole derivatives.



(I)

The N—N, C—N and C—S bond distances in the fused ring system are comparable with those found in a related structure, *viz*. 3-amino-6,7-dihydro-6-hydroxy-5*H*-1,2,4-triazolo[3,4-*b*]-[1,3]thiazine (Glowka, 1994). The S1—C1 [1.736 (3) Å], C1—N1 [1.360 (4) Å] and C1—N3 [1.314 (4) Å] bond distances clearly reflect the resonance of the thiourea system (Valle *et al.*, 1970). The C1—S1—C2 angle of 97.5 (1)° suggests that the S atom uses only *p*-orbitals to form bonds with atom C1 and C2. In order to minimize the steric repulsion between the H atoms at C8 and C11, the relevant bond angles, *viz*. C8—N1—C9 and N1—C9—C10, are widened.

In the title compound, (I) (Fig. 1), the benzene ring (*A*) is planar, with a maximum deviation of 0.010 (4) Å for C5. The thiazine ring (*B*) is in a boat conformation, with puckering parameters (Cremer & Pople, 1975) q_2 = 0.547 (2), Q_3 = −0.054 (3), Q_T = 0.549 (2), Φ = 124° and θ = 96°, and asymmetry parameters (Nardelli, 1983) Δ_S(C8) = 0.024 (1) and Δ_S(N1—C1) = 0.030 (1). Atoms S1 and C8 deviate by 0.552 (1) and 0.400 (3) Å, respectively, from the weighted least-squares plane through the remaining four atoms C1, N1, C7 and C2. The dihedral angle between the benzene (*A*), triazole (*C*) and phenyl (*D*) rings are: *A*/*C* 38.5 (1) *A*/*D* 70.9 (1) and *C*/*D* 34.3 (1)°.

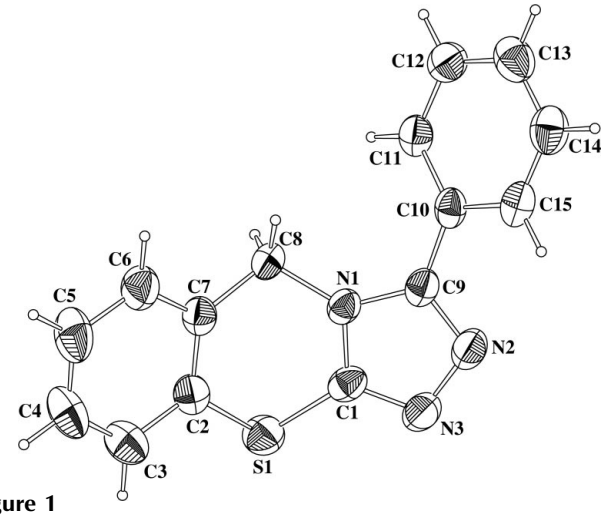


Figure 1
The molecular structure, with the atomic numbering scheme. Probability displacement ellipsoids are drawn at the 50% level.

In addition to van der Waals interactions, π–π interactions are possibly involved to ensure cohesion between the molecules. There is a π–π interaction between the triazole ring at (x, y, z) and the phenyl ring at ($\frac{1}{2} - x$, $-\frac{1}{2} + y$, z), the centroids of the two rings being separated by 3.531 (2) Å.

Experimental

The title compound was synthesized by irradiation (254 nm) of 4-(2-chlorobenzyl)-5-phenyl-1,2,4-triazole-3-thione, according to a literature method (Park *et al.*, 1999), using CH_3CN/NaOH as solvent. After completion of the reaction, removal of solvent and chromatographic separation on a silica-gel column by elution with a petroleum ether–ethyl acetate (1:1) mixture afforded the title compound.

Crystal data

$C_{15}H_{11}N_3S$
M_r = 265.33
Orthorhombic, *Pbca*
a = 11.5674 (15) Å
b = 10.1948 (19) Å
c = 21.433 (3) Å
V = 2527.5 (7) Å^3
Z = 8
D_x = 1.395 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 26 reflections
θ = 5–20°
μ = 0.24 mm^{-1}
T = 293 (2) K
Block, colorless
0.46 × 0.26 × 0.23 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
2197 measured reflections
2110 independent reflections
1457 reflections with $I > 2\sigma(I)$
R_{int} = 0.015
θ_{max} = 25.0°
$h = -2 \rightarrow 13$
$k = -2 \rightarrow 12$
$l = -25 \rightarrow 5$
2 standard reflections
every 200 reflections
frequency: 120 min
intensity decay: <1.5%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.146
S = 0.88
2110 reflections
173 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.1P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0065 (13)

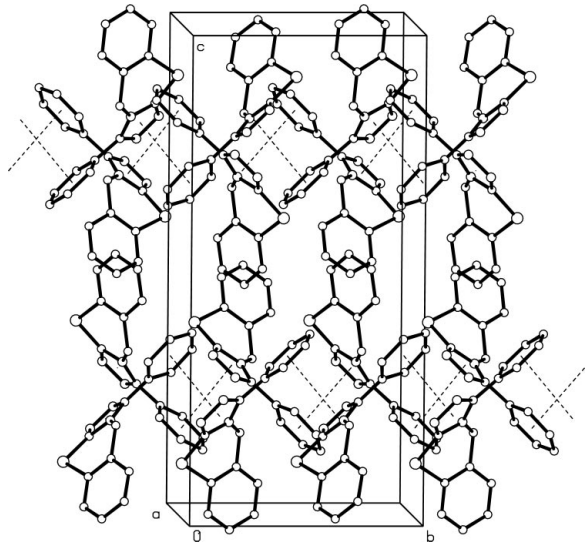


Figure 2
A packing diagram of the molecule, viewed down the a axis. π–π interactions are indicated by dashed lines.

Table 1
Selected geometric parameters (Å, °).

S1—C1	1.736 (3)	N1—C1	1.360 (4)
S1—C2	1.773 (3)	C9—C10	1.473 (4)
N1—C9	1.364 (3)	N2—N3	1.393 (4)
C1—S1—C2	97.49 (14)	N2—C9—N1	110.0 (3)
C9—N1—C8	131.6 (2)	N1—C9—C10	125.6 (2)
C1—N1—C8	123.6 (2)	C9—N2—N3	108.1 (2)

The H atoms were positioned geometrically and were treated as riding on their parent C atoms, with aromatic C—H distances of 0.93 Å and other C—H distances of 0.97 Å,

Data collection: *CAD*-4 *EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD*-4 *EXPRESS*; data reduction: *XCAD*4 (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ZORTEP* (Zsolnai, 1997) and *PLATON* (Spek, 1990); software used to prepare material for publication: *SHELXL*97 and *PARST* (Nardelli, 1995).

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