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5-Benzyl-4-phenyl-2,4-dihydro-1,2,4-triazole-3-thione

Mehmet Akkurt,[a] Sema Öztürk,[a]* Süleyman Servi,[b] Ahmet Cansız,[b] Memet Şekerci[b] and Canan Kazak[c]

[a]Department of Physics, Faculty of Arts and Sciences, Erciyes University, 38039 Kayseri, Turkey, [b]Department of Chemistry, Faculty of Arts and Sciences, Fırat University, 23119 Elazığ, Turkey, and [c]Department of Physics, Faculty of Arts and Sciences, Ondokuz Mayıs University, 55139 Kurupelit, Samsun, Turkey

Correspondence e-mail: ozturk@erciyes.edu.tr

Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.003 Å
R factor = 0.039
wR factor = 0.096
Data-to-parameter ratio = 17.3

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

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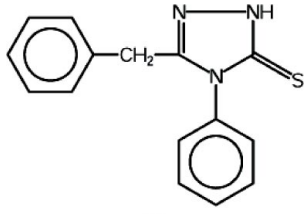
As part of structural studies of 1,2,4-triazole derivatives, the crystal structure of the title compound, $C_{15}H_{13}N_3S$, has been investigated. The structure shows a V-shape in the molecular skeleton, as found for similar compounds. The crystal structure is stabilized by an N—H···S and three C—H···π intermolecular interactions.

Comment

Derivatives of 1,2,4-triazole are known to exhibit anti-inflammatory (Unangst *et al.*, 1992; Mullican *et al.*, 1993), antiviral (Jones *et al.*, 1965), analgesic (Sughen & Yoloye, 1978), antimicrobial (Shams El-Dine & Hazzaa, 1974; Misato *et al.*, 1977; Cansız *et al.*, 2001), anticonvulsant (Stillings *et al.*, 1986) and antidepressant activities (Kane *et al.*, 1988), the last of these usually being explored by the forced swim test (Porsolt *et al.*, 1977; Vamvakides, 1990). Among the pharmacological profiles of 1,2,4-triazoles, their antimicrobial, anticonvulsant and antidepressant properties seem to be the best documented. The derivatives of 4,5-disubstituted 1,2,4-triazole are synthesized by intramolecular cyclization of 1,4-disubstitued thiosemicarbazides (Zamani *et al.*, 2003; Cansız *et al.*, 2004). In addition, there are some studies on electronic structures and the thiol–thione tautomeric equilibrium of heterocyclic thione derivatives (Aydoğan *et al.*, 2002; Charistos *et al.*, 1994). In this context, we have synthesized several new compounds, including the title compound, (I) (Fig. 1).



(I)

The N=C [1.296 (2) Å] and S=C distances [1.6717 (15) Å] are comparable to those observed in related structures (Öztürk *et al.*, 2004*a*,*b*). The title compound contains three planar rings. One is the triazole ring (ring *A*; N1, C7, N2, N3 and C8); the others are rings *B* (C1–C6) and *C* (C10–C15). The dihedral angles between these rings are 69.7 (1)° for *A*/*B*, 82.0 (1)° for *A*/*C* and 34.5 (1)° for *B*/C.

In the crystal structure of (I), the molecules are linked by N—H···S and C—H···π interactions, resulting in a network structure (Fig. 2); details of these interactions are listed in Table 2.

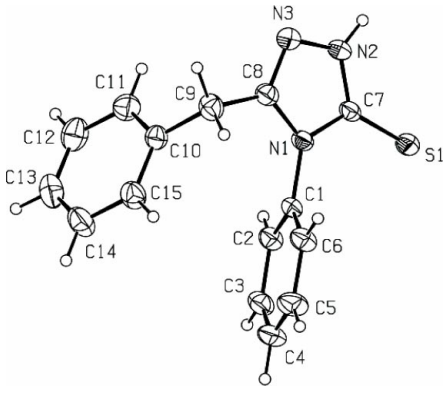


Figure 1
An *ORTEP*-3 (Farrugia, 1997) plot of the title compound, with the atom-numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 30% probability level.

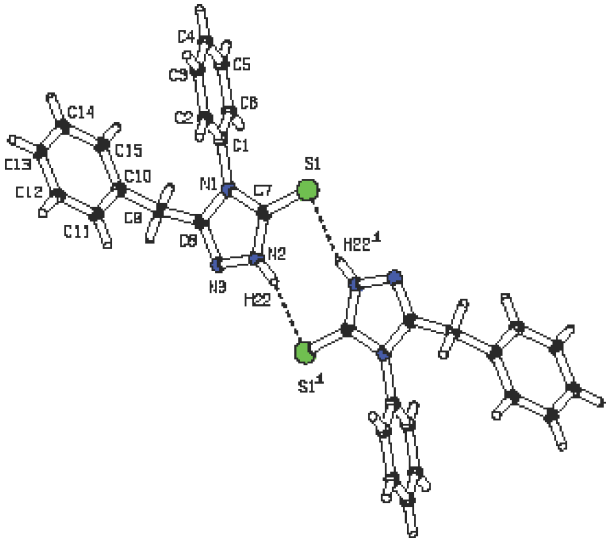


Figure 2
A view of the hydrogen-bonded dimer in the crystal structure of (I). Dashed lines indicate hydrogen bonds. [Symmetry code: (i) $1-x$, $-y$, $-z$].

Experimental

A stirred mixture of 1-benzyl-4-phenylthiosemicarbazide (1 mmol) and sodium hydroxide (40 mg, 1 mmol, as a 2*N* solution) was refluxed for 4 h. After cooling, the solution was acidified with hydrochloric acid and the precipitate was filtered off and crystallized from an ethanol–dioxane mixture (yield 79%; m.p. 470 K). IR (cm^{-1}): 2560 (SH), 1606 (C═N), 1535, 1260, 1050, 950 (N—C═S, amide I, II, III and IV bands); ^{1}H NMR: δ 3.80 (*s*, 2H, $-CH_2$), 7.45–7.10 (*m*, 10H, Ar-H), 13.92 (*s*, 1H, SH or NH).

Crystal data

$C_{15}H_{13}N_3S$
M_r = 267.35
Monoclinic, $P2_1/n$
a = 7.0467 (5) Å
b = 17.6802 (13) Å
c = 11.2725 (7) Å
β = 104.355 (5)°
V = 1360.56 (17) Å^3
Z = 4
D_x = 1.305 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2998 reflections
θ = 2.2–27.2°
μ = 0.23 mm^{-1}
T = 296 K
Prism, colorless
0.45 × 0.35 × 0.25 mm

Data collection

Stoe IPDS-II diffractometer
ω scans
21 276 measured reflections
2998 independent reflections
2023 reflections with $I > 2\sigma(I)$
R_{int} = 0.109
θ_{max} = 27.1°
$h = -8 \rightarrow 9$
$k = -22 \rightarrow 22$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.096
S = 0.89
2998 reflections
173 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0538P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.012 (2)

Table 1
Selected geometric parameters (Å, °).

S1—C7	1.6717 (15)	N2—N3	1.372 (2)
N1—C1	1.439 (2)	N2—C7	1.338 (2)
N1—C7	1.379 (2)	N3—C8	1.296 (2)
N1—C8	1.381 (2)		
C1—N1—C7	125.22 (13)	N1—C7—N2	102.86 (13)
C1—N1—C8	126.65 (13)	S1—C7—N2	128.90 (12)
C7—N1—C8	108.11 (13)	S1—C7—N1	128.23 (12)
N3—N2—C7	114.01 (13)	N1—C8—C9	124.56 (16)
N2—N3—C8	104.21 (13)	N1—C8—N3	110.81 (14)
N1—C1—C2	119.66 (14)	N3—C8—C9	124.63 (16)
N1—C1—C6	119.33 (14)		
C7—N1—C1—C6	110.66 (18)	C1—N1—C7—S1	−2.6 (2)
C8—N1—C1—C6	−70.4 (2)	N2—N3—C8—C9	−179.59 (16)
C1—N1—C8—C9	0.7 (3)	C8—C9—C10—C15	120.92 (19)
C7—N1—C8—C9	179.79 (16)	C8—C9—C10—C11	−61.2 (2)
C8—N1—C7—S1	178.35 (12)		

Table 2
Hydrogen-bonding geometry (Å, °).

*Cg*1 and *Cg*2 denote the centroids of the triazole and benzyl rings.

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H22···S1^{i}	0.86	2.45	3.293 (2)	166
C4—H4···*Cg*1^{ii}	0.93	2.95	3.715 (2)	141
C12—H12···*Cg*1^{iii}	0.93	2.98	3.796 (2)	147
C14—H14···*Cg*2^{iv}	0.93	2.99	3.603 (2)	125

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

All H atoms were positioned geometrically and refined using a riding model, with aromatic C—H distances of 0.93 Å, methylene C—H distances of 0.97 Å and a triazole N—H distance of 0.86 Å. U_{iso}(H) values were set at 1.2U_{eq} of the carrier atom.

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED*32 (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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