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2-[(4-Phenylthiazol-2-yl)hydrazonomethyl]phenol

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.006 Å
R factor = 0.049
wR factor = 0.097
Data-to-parameter ratio = 14.7

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

In the title compound, $C_{16}H_{13}N_3OS$, the plane of the —C—CH═N—NH—C— bridge makes dihedral angles of 7.9 (2) and 11.9 (4)° with the planes of the phenol and thiazole rings, respectively. In the molecule, there are two intramolecular interactions of type O—H···N and C—H···N. In the crystal structure, there are two intermolecular interactions of type C—H···N and N—H···O, leading to the formation of dimers.

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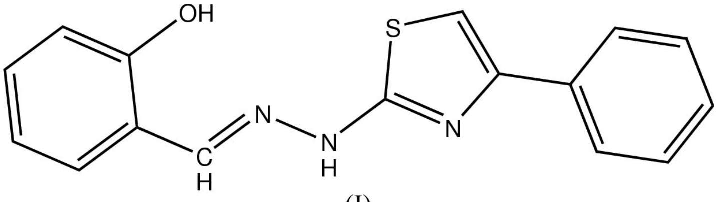
Comment

Many naturally occurring and synthetic thiazole derivatives exhibit biological activities, such as antibiotic, anti-inflammatory, anthelmintic or fungicidal properties (Metzger, 1984; Crews *et al.*, 1988; Shinagawa *et al.*, 1997 Shivarama Holla *et al.*, 2003). Since a wide spectrum of activity is shown by the thiazole ring, a large number of thiazoles substituted with different groups at various positions have been prepared (Giridhar et al., 2001). In addition, aminothiazoles have been studied extensively for a range of biological, pharmaceutical and industrial applications (Lynch *et al.*, 1999; Toplak *et al.*, 2003; Au-Alvarez *et al.*, 1999), including corrosion inhibition (Form *et al.*, 1974). 2-Amino-1,3-thiazole, the structure of which was reported in 1982 (Caranoni & Reboul, 1982), is itself listed as a thyroid inhibitor (Merck, 2001). Taking into account the importance of thiazole, the crystal structure of (I) has been determined, and the results are presented here.



(I)

The molecular structure of (I), together with the atom-labelling scheme and the intramolecular hydrogen bonding, is shown in Fig. 1. The molecule of the title compound contains of three π systems, *viz*. a thiazole ring (ring A = S1/N1/C1–C3) and two benzene rings (ring B = C4–C9 and ring C = C11–C16). The dihedral angles between the mean planes of the rings are A/B 14.9 (2), A/C 19.65 (14) and B/C 34.07 (13)°. The five-membered bridge (C1/N2/N3/C10/C11) linking rings A and C is planar, the maximum deviation from the least-squares plane being 0.034 (2) Å for atom N3. The mean plane through the bridge makes dihedral angles of 7.9 (2) and 11.9 (4)° with the planes of rings C and A, respectively. These values indicate

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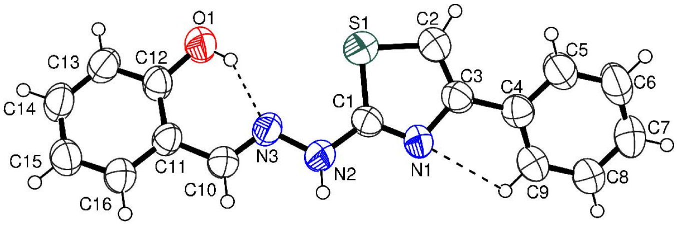

Figure 1
An *ORTEP-3* (Farrugia, 1997) drawing of (I), showing the atomic numbering scheme. Displacement ellipsoids of non-H atoms are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii. The intramolecular O—H···N and C—H···N hydrogen bonds are represented by dashed lines.

that the planes of the five-membered bridge, rings *A* and *C* are nearly coplanar.

The bond lengths and angles in (I) (Table 1) are comparable with those reported for a closely related compound, 5-bromo-2-hydroxybenzaldehyde(4-phenyl-1,3-thiazol-2-yl)hydrazone, (II) (Genç *et al.*, 2004). Though the structures of these two molecules are rather similar, compound (II) crystallizes in the centrosymmetric space group $P2_1/c$.

The hydrogen-bonding interactions in (I) and (II) are somewhat different, especially the intermolecular interactions. O—H···S intramolecular and N—H···N intermolecular interactions, which exist in (II), are not observed in (I). However, an O—H···N intramolecular interaction is common to both structures. In the molecular structure of (I), an intramolecular O1—H1···N3 hydrogen bond results in the formation of a six-membered ring fused with ring *C*, while an intramolecular C9—H9···N1 hydrogen bond leads to the formation of a five-membered ring which is fused with both ring *A* and ring *B* (Fig. 1). In addition, there are two types of intermolecular interaction, C—H···N and N—H···O, present in the crystal structure (Table 2). These intermolecular interactions linking the molecules to each other result in the formation of dimers, generating an $R_2^2(8)$ ring. This arrangement results in the formation of chains running along the *c* axis (Fig. 2). There are no other significant interactions in the crystal structure, such as π—π stacking and C—H···π, which play a role in the packing.

Experimental

A solution of phenacyl chloride (1.55 g, 10 mmol) and thiosemicarbazone (1.95 g, 10 mmol) in absolute ethanol (50 ml) was maintained at 313–323 K, with continuous stirring. The reaction was complete after *ca* 2 h. After cooling to room temperature, the target product, (I), precipitated by neutralization with the addition of aqueous ammonia (5%), was filtered off, washed with copious cold ethanol and dried in air. Shiny crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of a solution in ethanol (yield: 1.92 g, 65%; m.p 473 K). IR (ν, cm^{-1}): 3245 (OH), 3115 (NH), 1624 (C=N, azomethine), 1609 (C=N, thiazol), 659 (C—S—C, thiazole); ^{1}H NMR (CDCl$_3$, TMS): δ 6.34 (*s*, 1H, thiazole), 6.6–7.6 (*m*, 9H, aromatics), 7.8 (*s*, 1H, H—C=N), 10.20 (*s*, 1H, NH, D$_2$O exchangeable), 11.74 (*br*, 1H, OH, D$_2$O exchangeable).

Crystal data

$C_{16}H_{13}N_3OS$
$M_r = 295.35$
Orthorhombic, *Pbca*
$a = 8.5129$ (13) Å
$b = 30.047$ (4) Å
$c = 11.1308$ (18) Å
$V = 2847.1$ (7) Å^3
$Z = 8$
$D_x = 1.378$ Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 9629 reflections
$\theta = 1.8$–$26.1°$
$\mu = 0.23$ mm^{-1}
$T = 293$ K
Plate, pink
0.50 × 0.22 × 0.03 mm

Data collection

Stoe IPDS-2 diffractometer
ω scans
Absorption correction: by integration (*X-RED32*; Stoe & Cie, 2002)
$T_{\min} = 0.839$, $T_{\max} = 0.977$
16710 measured reflections
2803 independent reflections
1028 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.139$
$\theta_{\max} = 26.1°$
$h = -10 \rightarrow 10$
$k = -36 \rightarrow 36$
$l = -13 \rightarrow 13$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.049$
$wR(F^2) = 0.097$
$S = 0.91$
2803 reflections
191 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0255P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.17$ e Å^{-3}
$\Delta\rho_{\min} = -0.16$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0014 (2)

Table 1
Selected geometric parameters (Å, °).

S1—C1	1.720 (3)	N2—N3	1.385 (3)
S1—C2	1.727 (4)	N3—C10	1.292 (4)
O1—C12	1.375 (4)	C2—C3	1.343 (5)
N1—C1	1.296 (4)	C3—C4	1.475 (4)
N1—C3	1.392 (4)	C10—C11	1.442 (4)
N2—C1	1.374 (4)		
C1—S1—C2	87.56 (19)	C3—C2—S1	111.5 (3)
C1—N1—C3	109.6 (3)	C2—C3—N1	114.5 (3)
C1—N2—N3	117.1 (3)	C2—C3—C4	127.3 (3)
C10—N3—N2	116.4 (3)	N1—C3—C4	118.1 (3)
N1—C1—N2	122.4 (3)	N3—C10—C11	120.7 (4)
N1—C1—S1	116.8 (3)	O1—C12—C13	117.5 (3)
N2—C1—S1	120.8 (3)	O1—C12—C11	121.8 (3)
C1—N2—N3—C10	179.4 (3)	N1—C3—C4—C5	−167.9 (3)
C3—N1—C1—N2	−177.7 (3)	N2—N3—C10—C11	−175.9 (3)
N3—N2—C1—N1	−172.2 (3)	N3—C10—C11—C16	−174.6 (3)
N3—N2—C1—S1	10.6 (4)	N3—C10—C11—C12	7.2 (5)
N1—C3—C4—C9	14.3 (5)	C10—C11—C12—O1	−3.4 (6)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N3	0.82	1.93	2.650 (4)	145
C9—H9···N1	0.93	2.51	2.848 (5)	101
C13—H13···N1$^{\rm i}$	0.93	2.60	3.501 (5)	164
N2—H2*A*···O1$^{\rm ii}$	0.86	2.30	3.052 (4)	146

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

H atoms were positioned geometrically and treated using a riding model, fixing the bond lengths at 0.93, 0.86 and 0.82 Å for CH, NH and OH groups, respectively, and with $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}$(carrier atom).

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON* (Spek, 2003).

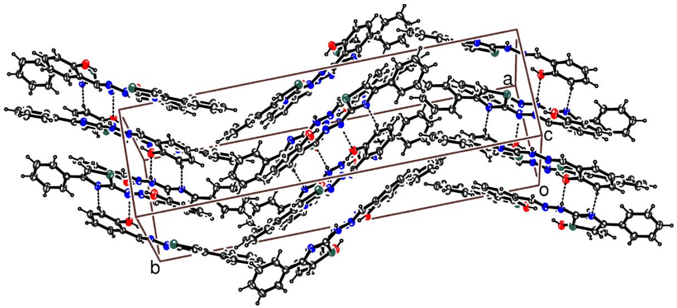

Figure 2
The crystal packing of (I), showing the C—H···N and N—H···O hydrogen-bonded (dashed lines) dimers.

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