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4-[(2-Hydroxybenzylidene)amino]-5-(2-thienylmethyl)-2*H*-1,2,4-triazol-3(4*H*)-one

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.007 Å
R factor = 0.066
wR factor = 0.185
Data-to-parameter ratio = 14.5

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

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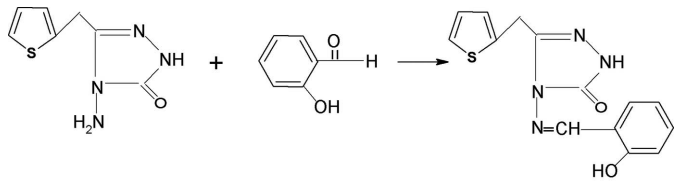
In the title compound, $C_{14}H_{12}N_4O_2S$, the triazole ring is nearly coplanar with the phenol unit, the dihedral angle being 6.60 (3)°. The crystal structure involves an intermolecular N—H···O hydrogen bond and intramolecular O—H···N and C—H···O hydrogen bonds.

Comment

Triazole ring systems are typical planar six-π-electron partially aromatic systems and 1,2,4-triazole and its derivatives are used as starting materials for the synthesis of many heterocycles (Desenko, 1995). Di- or trisubstituted 1,2,4-triazole derivatives have also been reported to show antitubercular activities (İkizler *et al.*, 1998). In a previous paper, we reported that some 1,2,4-triazol-5-one compounds have antimicrobial effects (Demirbas *et al.*, 2004). The coordination chemistry of azoles acting as ligands for the production of organometallic compounds in the context of modelling biological systems has attracted much interest (İkizler & Sancak, 1992). In this paper, we report the crystal structure of the title compound, (I).



(I)

The title compound contains three rings, *viz.* the 1,2,4-triazole ring, *A*, the thiophene ring, *B*, and the phenol ring, *C* (Fig. 1). The dihedral angles between rings *A*/*B*, *A*/*C* and *B*/*C* are 67.39 (15), 6.60 (3) and 64.76 (1)°, respectively. These values indicate that the triazole ring is nearly coplanar with the phenol group. The C8═O2 bond length (Table 1) is comparable with those of similar C═O double bonds found in 1,2,4-triazole rings (Arslan *et al.*, 2004; Ocak, Kahveci *et al.*, 2003; Ocak, Çoruh *et al.*, 2003).

In the crystal structure of (I), a strong intermolecular N—H···O hydrogen bond and intramolecular O—H···N and C—H···O hydrogen bonds are observed (Table 2 and Fig. 2).

Experimental

A mixture of 4-amino-5-(thien-2-ylmethyl)-2,4-dihydro-1,2,4-triazol-3-one (0.19 g, 0.001 mol) and salicylaldehyde (0.10 ml, 0.12 g, 0.001 mol) was warmed at 413–423 K for 1 h. The solid crude product was recrystallized from ethanol. The crystals were recrystallized several times from the same solvent and were then dried *in vacuo*

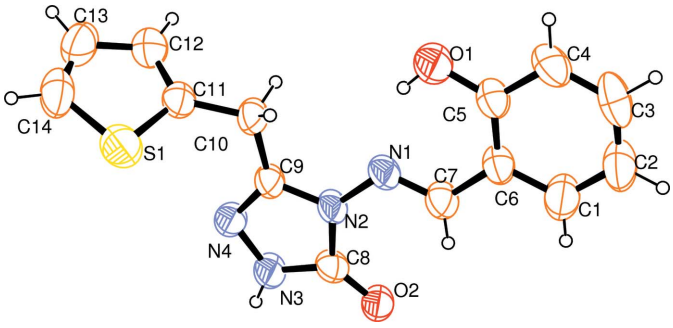


Figure 1
The molecular structure of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

(yield 0.23 g, 80.43%). Spectroscopic analysis: IR (ν, cm^{-1}): 3166 (N—H), 3045 (aromatic C—H), 1711 (C═O), 1618 (C═N), 1606 (C═C); ^{1}H NMR: δ 4.26 (*s*, tyf-CH$_2$), 7.82–7.87 (*m*, aromatic H), 7.34–7.40 (*m*, 3H), 6.91–6.97 (*m*, 4H), 9.95 (*m*, N═CH), 11.95 (*m*, NH), 10.32 (*s*, OH); MS: M^+ 300.92.

Crystal data

$C_{14}H_{12}N_4O_2S$
M_r = 300.34
Triclinic, $P\overline{1}$
a = 5.5879 (7) Å
b = 9.2167 (12) Å
c = 14.3000 (19) Å
α = 77.528 (10)°
β = 84.181 (11)°
γ = 77.290 (10)°
V = 700.36 (16) Å^3
Z = 2
D_x = 1.424 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.24 mm^{-1}
T = 293 (2) K
Plate, yellow
0.50 × 0.21 × 0.04 mm

Data collection

Stoe IPDS-2 diffractometer
ω scans
Absorption correction: integration (*X-RED*; Stoe & Cie, 2002)
T_{min} = 0.932, T_{max} = 0.993
10648 measured reflections
2752 independent reflections
1322 reflections with $I > 2\sigma(I)$
R_{int} = 0.176
θ_{max} = 26.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.066
$wR(F^2)$ = 0.185
S = 0.87
2752 reflections
190 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0823P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 1997)
Extinction coefficient: 0.025 (2)

Table 1
Selected geometric parameters (Å, °).

C5—O1	1.347 (5)	C11—S1	1.708 (4)
C8—O2	1.227 (5)	C14—S1	1.692 (6)
C9—N4	1.289 (4)	N1—N2	1.383 (4)
C9—N2	1.371 (5)	N3—N4	1.386 (5)
C9—N2—C8	108.7 (3)	C14—S1—C11	91.6 (3)

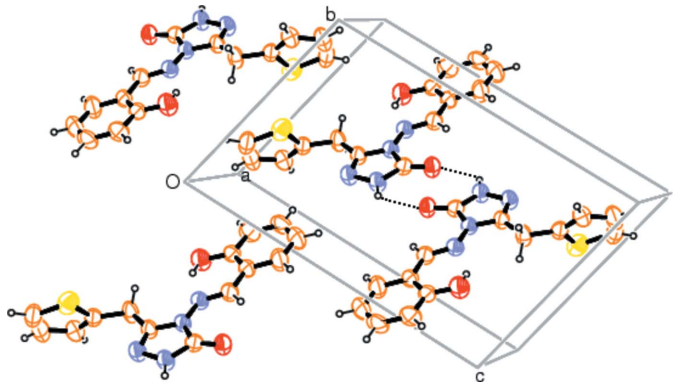


Figure 2
A packing diagram for (I), viewed approximately along the *a* axis. The N—H···O hydrogen bonds are indicated by dashed lines.

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H7···O2^{i}	0.86	1.95	2.791 (4)	167
O1—H5···N1	0.82	1.92	2.639 (4)	146
C7—H6···O2	0.93	2.28	2.946 (5)	128

Symmetry code: (i) $-x, -y+1, -z+1$.

The high value of R_{int} indicates that the overall quality of the data may be poor due to the crystal quality. All H atoms were placed in calculated positions, with C—H = 0.93–0.97 Å, N—H = 0.86 Å and O—H = 0.82 Å, and refined using a riding model, with U_{iso}(H) = 1.2U_{eq}(C,N) or 1.5U_{eq}(O).

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED* (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* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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