3-Hydroxysalicylaldehyde 2-thienoylhydrazone

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The two independent molecules of the title compound, $C_{12}H_{10}N_2O_3S$, are approximately planar and one is stacked over the other. One of the two molecules interacts with its symmetry-related neighbors to form a hydrogen-bonded helical chain that runs along the shortest axis of the orthorhombic unit cell. The second independent molecule does not form such a chain; instead, it is merely linked to the other independent molecule by a hydrogen bond.

Key indicators

Single-crystal X-ray study
$T = 295$ K
Mean σ(C–C) = 0.004 Å
R factor = 0.045
wR factor = 0.121
Data-to-parameter ratio = 15.7

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

Comment

In the crystal structure of 3-hydroxysalicylaldehyde 2-furoyl-hydrazone, one molecule is stacked over the other across a center of inversion, and the pair of molecules is linked by hydrogen bonds into a three-dimensional network (Ali *et al.*, 2005).

(I)
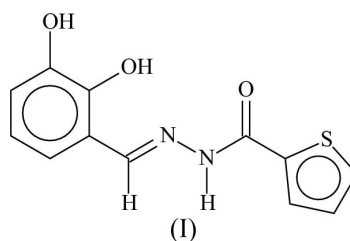
With an S atom in place of the O atom in the five-membered heterocyclic ring, the title compound, (I), crystallizes with two symmetry-independent molecules in the asymmetric unit (Fig. 1); one is stacked over the other and the primed molecule is approximately related to the unprimed molecule by a non-
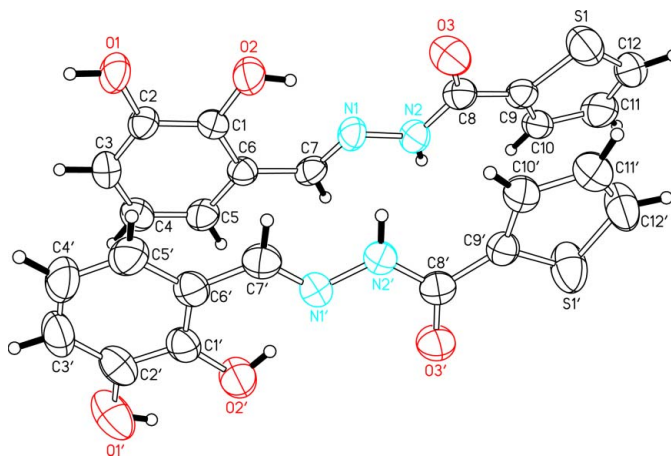

Figure 1
ORTEPII (Johnson, 1976) plot of the two independent molecules of $C_{12}H_{10}N_2O_3S$. Displacement ellipsoids are drawn at the 50% probability level, and H atoms are shown as spheres of arbitrary radii.

crystallographic twofold rotation axis. The thienyl ring is twisted by 10.1 (1)° with respect to the C7—N1—N2—C8 fragment; the twist in the primed molecule is 15.4 (2)°. The unprimed molecule propagates along the shortest axis of the unit cell, giving rise to a hydrogen-bonded helical chain (Fig. 2). The primed molecule does not form such a chain; instead, it is merely connected to the unprimed molecule by a hydrogen bond. The difference in the hydrogen bonding interactions arises from the orientation of the H atom of the 3-hydroxy substituent.

Experimental

3-Hydroxysalicyldehyde (0.21 g, 1.5 mmol) and 2-thienoylhydrazide (0.22 g, 1.5 mmol) were heated in ethanol (40 ml) for 2 h. The compound that was isolated upon removal of the solvent was recrystallized from ethanol to give pale yellow–orange crystals.

Crystal data

$C_{12}H_{10}N_2O_3S$	Mo $K\alpha$ radiation
$M_r = 262.28$	Cell parameters from 814
Orthorhombic, $P2_12_12_1$	reflections
$a = 7.994$ (1) Å	$\theta = 3.1–22.4°$
$b = 11.990$ (2) Å	$\mu = 0.27$ mm^{-1}
$c = 24.787$ (4) Å	$T = 295$ (2) K
$V = 2375.8$ (6) Å^3	Block, faint yellow–orange
$Z = 8$	$0.38 \times 0.34 \times 0.26$ mm
$D_x = 1.467$ Mg m^{-3}	

Data collection

Bruker SMART area-detector diffractometer	3383 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.024$
Absorption correction: None	$\theta_{max} = 27.2°$
10174 measured reflections	$h = -10 \rightarrow 7$
5149 independent reflections	$k = -15 \rightarrow 7$
	$l = -31 \rightarrow 31$

Refinement

Refinement on F^2	$(\Delta/\sigma)_{max} = 0.001$
$R[F^2 > 2\sigma(F^2)] = 0.045$	$\Delta\rho_{max} = 0.28$ e Å^{-3}
$wR(F^2) = 0.121$	$\Delta\rho_{min} = -0.40$ e Å^{-3}
$S = 1.03$	Extinction correction: none
5149 reflections	Absolute structure: Flack (1983),
329 parameters	2201 Friedel pairs
H-atom parameters constrained	Flack parameter: −0.05 (9)
$w = 1/[\sigma^2(F_o^2) + (0.0638P)^2]$	
where $P = (F_o^2 + 2F_c^2)/3$	

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1o···O3^i	0.85	1.82	2.668 (3)	173
O2—H2o···N1	0.85	1.92	2.656 (3)	144
N2—H2n···O3^{ii}	0.85	2.20	3.028 (3)	165
O1′—H1o′···O2′	0.85	2.24	2.679 (3)	112
O2′—H2o′···N1′	0.85	1.86	2.595 (3)	144
N2′—H2n′···O1^{iii}	0.85	2.22	3.017 (3)	156

Symmetry codes: (i) $x + \frac{1}{2}, -y + \frac{1}{2}, -z + 2$; (ii) $x + \frac{1}{2}, -y + \frac{3}{2}, -z + 2$; (iii) $x - \frac{1}{2}, -y + \frac{1}{2}, -z + 2$.
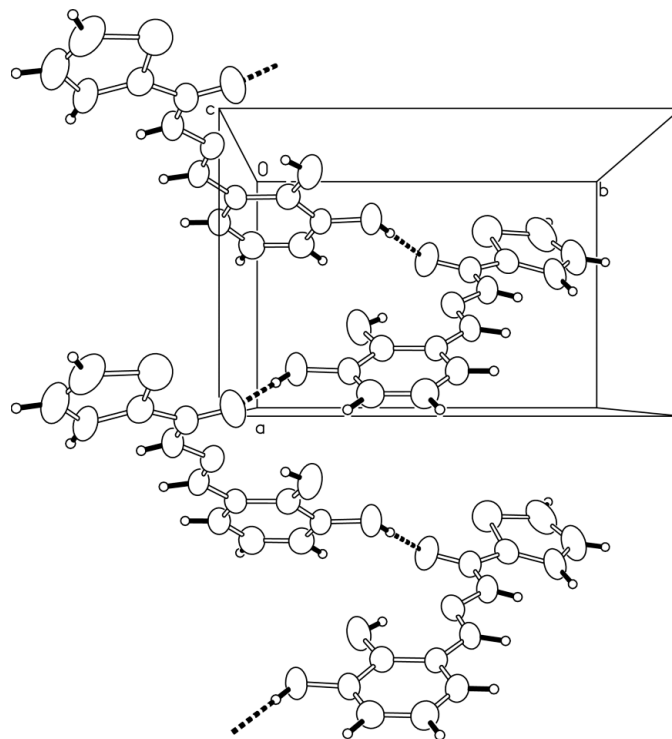

Figure 2
ORTEPII (Johnson, 1976) plot of the helical hydrogen-bonded chain that is formed by the unprimed molecule and its symmetry equivalents.

H atoms were placed at calculated positions (C—H = 0.93 Å and N—H = O—H = 0.85 Å) and were included in the refinement in the riding-model approximation, with $U_{iso}(H)$ values set to 1.2 times U_{eq} of the parent atom. The torsion angles of the hydroxy groups were refined.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

References

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