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(*E*)-4-Hydroxy-*N*′-(2-thienylmethylene)-benzohydrazide

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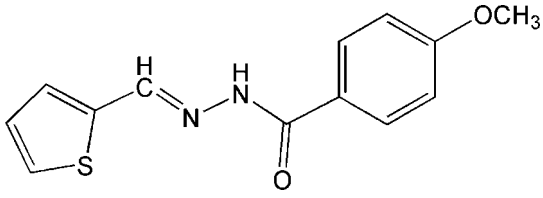
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.003 Å; R factor = 0.040; wR factor = 0.108; data-to-parameter ratio = 15.7.

In the title compound, $C_{13}H_{12}N_2O_2S$, the dihedral angle between the thiophene and benzene planes is 38.08 (11)°. Intermolecular N—H···O and C—H···O hydrogen bonds link the molecules into a two-dimensional network parallel to the *bc* plane.

Related literature

For general background, see: Belloni *et al.* (2005); Kahwa *et al.* (1986); Parashar *et al.* (1988); Santos *et al.* (2001); Tynan *et al.* (2005).



Experimental

Crystal data

$C_{13}H_{12}N_2O_2S$
M_r = 260.31
Monoclinic, $P2_1/c$
a = 11.479 (5) Å
b = 11.279 (5) Å
c = 9.934 (4) Å
β = 93.745 (8)°
V = 1283.4 (10) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.25 mm^{-1}
T = 294 (2) K
0.18 × 0.16 × 0.08 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.957, T_{max} = 0.981
7140 measured reflections
2640 independent reflections
1873 reflections with $I > 2\sigma(I)$
R_{int} = 0.031

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.040
$wR(F^2)$ = 0.108
S = 1.01
2640 reflections
168 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···O1^{i}	0.887 (9)	2.104 (11)	2.968 (2)	164 (2)
C5—H5···O1^{i}	0.93	2.48	3.292 (3)	145
C8—H8···O1^{i}	0.93	2.57	3.421 (3)	152
C11—H11···O2^{ii}	0.93	2.57	3.373 (3)	145

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

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI2442).

References

Belloni, M., Kariuki, B. M., Manickam, M., Wilkie, J. & Preece, J. A. (2005). *Cryst. Growth Des.* **5**, 1443–1449.
Bruker (1997). *SHELXTL*. Version 5.10. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (1999). *SMART* (Version 5.0) and *SAINT* (Version 4.0). Bruker AXS Inc., Madison, Wisconsin, USA.
Kahwa, I. A., Selbin, J., Hsieh, T. C.-Y. & Laine, R. A. (1986). *Inorg. Chim. Acta*, **118**, 179–185.
Parashar, R. K., Sharma, R. C., Kumar, A. & Mohan, G. (1988). *Inorg. Chim. Acta*, **151**, 201–208.
Santos, M. L. P., Bagatin, I. A., Pereira, E. M. & Ferreira, A. M. D. C. (2001). *J. Chem. Soc. Dalton Trans.* pp. 838–844.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Tynan, E., Jensen, P., Lees, A. C., Moubaraki, B., Murray, K. S. & Kruger, P. E. (2005). *CrystEngComm*, **7**, 90–95.

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supplementary materials

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(*E*)-4-Hydroxy-*N*'-(2-thienylmethylene)benzohydrazide

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Comment

In order to establish control over the preparation of crystalline solid materials so that their architecture and properties are predictable (Belloni *et al.*, 2005; Tynan *et al.*, 2005; Parashar *et al.*, 1988), the synthesis of new and designed crystal structures has become a major strand of modern chemistry. Metal complexes based on Schiff bases have attracted much attention because they can be utilized as model compounds of active centres in various proteins and enzymes (Kahwa *et al.*, 1986; Santos *et al.*, 2001). As part of an investigation of the coordination properties of Shiff bases functioning as ligands, we report the synthesis and crystal structure of the title compound, (I).

In the molecular structure of the title compound (Fig. 1), the geometric parameters are normal. The thiophene ring (C1–C4/S1) is essentially planar, with a maximum deviation from the mean plane of 0.004 (2) Å for atom C3. In the benzene ring (C7–C12), a maximum deviation of 0.012 (2) Å is observed for atom C10. The dihedral angle between the thiophene and benzene planes is 38.08 (11)°. The O1/N1/N2/C6/C7 plane makes dihedral angles of 7.06 (10) and 31.59 (1)° with the benzene and thiophen rings, respectively

Intermolecular N—H···O hydrogen bonds link the molecules into a chain along the *c* axis. The chains are cross-linked through C—H···O hydrogen bonds (Table 1) to form a two-dimensional network, as illustraed in Fig. 2.

Experimental

An anhydrous ethanol solution (50 ml) of thiophene-2-carbaldehyde (1.12 g, 10 mmol) was added to an anhydrous ethanol solution (50 ml) of 4-hydroxybenzohydrazide (1.52 g, 10 mmol) and the mixture was stirred at 350 K for 6 h under N_2, whereupon a yellow precipitate appeared. The product was isolated, recrystallized from anhydrous ethanol and then dried *in vacuo* to give pure compound (I) in 89% yield. Yellow single crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of an anhydrous ethanol solution.

Refinement

The N-bound H atom was located in a difference Fourier map and refined with a N—H distance restraint of 0.90 (1) Å. C-bound H atoms were included in calculated positions, with C—H = 0.93 (aromatic) or 0.96 Å (methyl), and refined using a riding model, with U_{iso}(H) = 1.2U_{eq}(C) for aromatic H or 1.5U_{eq}(C) for methyl H atoms.

Figures

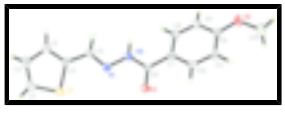

Fig. 1. The molecular structure of (I). Displacement ellipsoids are drawn at the 30% probability level.

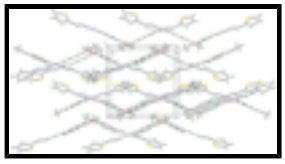

Fig. 2. The crystal packing of (I), viewed down the *c* axis. Hydrogen bonds are indicated by dashed lines.

(*E*)-4-Hydroxy-*N*'-(2-thienylmethylene)benzohydrazide

Crystal data

$C_{13}H_{12}N_2O_2S$	F_{000} = 544
M_r = 260.31	D_x = 1.347 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 2381 reflections
a = 11.479 (5) Å	θ = 2.5–26.1º
b = 11.279 (5) Å	μ = 0.25 mm^{-1}
c = 9.934 (4) Å	T = 294 (2) K
β = 93.745 (8)º	Block, yellow
V = 1283.4 (10) Å^3	0.18 × 0.16 × 0.08 mm
Z = 4	

Data collection

Bruker SMART CCD area-detector diffractometer	2640 independent reflections
Radiation source: fine-focus sealed tube	1873 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.031
T = 294(2) K	θ_{max} = 26.5º
φ and ω scans	θ_{min} = 1.8º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	h = −14→14
T_{min} = 0.957, T_{max} = 0.981	k = −12→14
7140 measured reflections	l = −8→12

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.040	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.108	$w = 1/[\sigma^2(F_o^2) + (0.0455P)^2 + 0.4301P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.01	$(\Delta/\sigma)_{max}$ = 0.004
2640 reflections	$\Delta\rho_{max}$ = 0.20 e Å^{-3}
168 parameters	$\Delta\rho_{min}$ = −0.28 e Å^{-3}
1 restraint	Extinction correction: none

Primary atom site location: structure-invariant direct methods

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\mathrm{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.54698 (6)	1.09426 (5)	0.21308 (6)	0.0534 (2)
O1	0.75806 (14)	0.69128 (12)	0.27982 (12)	0.0476 (4)
O2	0.93127 (15)	0.26017 (13)	−0.06983 (15)	0.0568 (4)
N1	0.68368 (15)	0.87541 (14)	0.12216 (16)	0.0382 (4)
N2	0.72728 (15)	0.77085 (14)	0.07103 (15)	0.0389 (4)
C1	0.5263 (2)	1.2421 (2)	0.1772 (3)	0.0568 (6)
H1	0.4838	1.2932	0.2288	0.068*
C2	0.5791 (2)	1.2756 (2)	0.0653 (3)	0.0584 (6)
H2	0.5774	1.3525	0.0315	0.070*
C3	0.6370 (2)	1.18032 (19)	0.0060 (2)	0.0505 (6)
H3	0.6768	1.1879	−0.0721	0.061*
C4	0.62875 (17)	1.07586 (17)	0.07454 (19)	0.0380 (5)
C5	0.67815 (17)	0.96278 (17)	0.03968 (19)	0.0380 (5)
H5	0.7067	0.9533	−0.0451	0.046*
C6	0.76055 (17)	0.68123 (17)	0.15618 (18)	0.0351 (4)
C7	0.80166 (17)	0.57001 (16)	0.09298 (18)	0.0351 (4)
C8	0.7956 (2)	0.54827 (18)	−0.04630 (19)	0.0438 (5)
H8	0.7620	0.6043	−0.1055	0.053*
C9	0.8392 (2)	0.44441 (18)	−0.0958 (2)	0.0484 (6)
H9	0.8348	0.4311	−0.1884	0.058*
C10	0.88946 (19)	0.35946 (17)	−0.0095 (2)	0.0420 (5)
C11	0.8935 (2)	0.37771 (18)	0.1292 (2)	0.0480 (5)
H11	0.9250	0.3204	0.1882	0.058*
C12	0.8500 (2)	0.48237 (18)	0.17786 (19)	0.0452 (5)
H12	0.8531	0.4947	0.2706	0.054*
C13	0.9799 (2)	0.1678 (2)	0.0149 (3)	0.0640 (7)
H13A	1.0405	0.1999	0.0754	0.096*
H13B	1.0119	0.1074	−0.0399	0.096*
H13C	0.9199	0.1342	0.0659	0.096*
H2A	0.7329 (17)	0.7677 (17)	−0.0175 (10)	0.041 (6)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0653 (4)	0.0503 (3)	0.0464 (3)	0.0059 (3)	0.0166 (3)	−0.0037 (3)
O1	0.0741 (10)	0.0459 (8)	0.0232 (7)	0.0065 (7)	0.0065 (6)	−0.0015 (6)
O2	0.0810 (12)	0.0406 (8)	0.0481 (9)	0.0167 (8)	−0.0025 (8)	−0.0087 (7)
N1	0.0483 (10)	0.0354 (9)	0.0310 (8)	0.0028 (7)	0.0042 (7)	−0.0037 (7)
N2	0.0575 (11)	0.0368 (9)	0.0225 (8)	0.0072 (8)	0.0045 (7)	−0.0017 (7)
C1	0.0578 (15)	0.0476 (13)	0.0643 (16)	0.0100 (11)	−0.0002 (12)	−0.0160 (11)
C2	0.0701 (16)	0.0396 (12)	0.0650 (16)	0.0054 (12)	0.0004 (13)	0.0015 (11)
C3	0.0580 (14)	0.0452 (13)	0.0491 (12)	0.0020 (11)	0.0086 (11)	0.0046 (10)
C4	0.0403 (11)	0.0407 (11)	0.0326 (10)	−0.0003 (9)	−0.0010 (8)	−0.0024 (8)
C5	0.0440 (11)	0.0401 (11)	0.0303 (10)	0.0013 (9)	0.0045 (8)	−0.0013 (8)
C6	0.0411 (11)	0.0377 (10)	0.0267 (9)	−0.0037 (9)	0.0036 (8)	0.0000 (8)
C7	0.0420 (11)	0.0363 (11)	0.0271 (9)	−0.0012 (8)	0.0033 (8)	0.0010 (7)
C8	0.0644 (15)	0.0390 (11)	0.0275 (10)	0.0080 (10)	−0.0023 (9)	0.0020 (8)
C9	0.0746 (16)	0.0446 (12)	0.0256 (10)	0.0086 (11)	−0.0002 (10)	−0.0040 (9)
C10	0.0520 (13)	0.0353 (11)	0.0383 (11)	0.0003 (10)	−0.0002 (9)	−0.0047 (9)
C11	0.0658 (15)	0.0398 (12)	0.0372 (11)	0.0099 (10)	−0.0045 (10)	0.0052 (9)
C12	0.0634 (14)	0.0471 (12)	0.0248 (10)	0.0052 (11)	0.0007 (9)	0.0010 (9)
C13	0.0732 (17)	0.0440 (13)	0.0738 (17)	0.0142 (12)	−0.0031 (14)	−0.0005 (12)

Geometric parameters (Å, °)

S1—C1	1.718 (3)	C5—H5	0.93
S1—C4	1.728 (2)	C6—C7	1.493 (3)
O1—C6	1.236 (2)	C7—C12	1.391 (3)
O2—C10	1.372 (2)	C7—C8	1.402 (3)
O2—C13	1.429 (3)	C8—C9	1.377 (3)
N1—C5	1.280 (2)	C8—H8	0.93
N1—N2	1.390 (2)	C9—C10	1.386 (3)
N2—C6	1.357 (2)	C9—H9	0.93
N2—H2A	0.887 (9)	C10—C11	1.391 (3)
C1—C2	1.354 (3)	C11—C12	1.381 (3)
C1—H1	0.93	C11—H11	0.93
C2—C3	1.413 (3)	C12—H12	0.93
C2—H2	0.93	C13—H13A	0.96
C3—C4	1.367 (3)	C13—H13B	0.96
C3—H3	0.93	C13—H13C	0.96
C4—C5	1.447 (3)		
C1—S1—C4	91.51 (11)	C12—C7—C8	117.77 (18)
C10—O2—C13	118.18 (17)	C12—C7—C6	117.82 (16)
C5—N1—N2	115.03 (16)	C8—C7—C6	124.41 (17)
C6—N2—N1	119.84 (15)	C9—C8—C7	120.35 (18)
C6—N2—H2A	123.5 (13)	C9—C8—H8	119.8
N1—N2—H2A	116.6 (13)	C7—C8—H8	119.8
C2—C1—S1	112.22 (18)	C8—C9—C10	120.89 (18)

C2—C1—H1	123.9	C8—C9—H9	119.6
S1—C1—H1	123.9	C10—C9—H9	119.6
C1—C2—C3	112.2 (2)	O2—C10—C9	115.96 (18)
C1—C2—H2	123.9	O2—C10—C11	124.31 (19)
C3—C2—H2	123.9	C9—C10—C11	119.73 (19)
C4—C3—C2	113.4 (2)	C12—C11—C10	118.94 (19)
C4—C3—H3	123.3	C12—C11—H11	120.5
C2—C3—H3	123.3	C10—C11—H11	120.5
C3—C4—C5	126.82 (19)	C11—C12—C7	122.28 (18)
C3—C4—S1	110.62 (16)	C11—C12—H12	118.9
C5—C4—S1	122.56 (15)	C7—C12—H12	118.9
N1—C5—C4	121.94 (17)	O2—C13—H13A	109.5
N1—C5—H5	119.0	O2—C13—H13B	109.5
C4—C5—H5	119.0	H13A—C13—H13B	109.5
O1—C6—N2	121.84 (17)	O2—C13—H13C	109.5
O1—C6—C7	121.57 (17)	H13A—C13—H13C	109.5
N2—C6—C7	116.59 (15)	H13B—C13—H13C	109.5
C5—N1—N2—C6	165.79 (19)	O1—C6—C7—C8	173.2 (2)
C4—S1—C1—C2	0.0 (2)	N2—C6—C7—C8	−7.4 (3)
S1—C1—C2—C3	0.4 (3)	C12—C7—C8—C9	−1.6 (3)
C1—C2—C3—C4	−0.8 (3)	C6—C7—C8—C9	177.9 (2)
C2—C3—C4—C5	179.9 (2)	C7—C8—C9—C10	0.1 (4)
C2—C3—C4—S1	0.8 (3)	C13—O2—C10—C9	−177.6 (2)
C1—S1—C4—C3	−0.45 (18)	C13—O2—C10—C11	1.5 (3)
C1—S1—C4—C5	−179.62 (18)	C8—C9—C10—O2	−179.2 (2)
N2—N1—C5—C4	176.45 (17)	C8—C9—C10—C11	1.6 (4)
C3—C4—C5—N1	167.3 (2)	O2—C10—C11—C12	179.1 (2)
S1—C4—C5—N1	−13.7 (3)	C9—C10—C11—C12	−1.9 (3)
N1—N2—C6—O1	−3.2 (3)	C10—C11—C12—C7	0.3 (3)
N1—N2—C6—C7	177.48 (16)	C8—C7—C12—C11	1.4 (3)
O1—C6—C7—C12	−7.3 (3)	C6—C7—C12—C11	−178.2 (2)
N2—C6—C7—C12	172.09 (18)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2A···O1^{i}	0.887 (9)	2.104 (11)	2.968 (2)	164 (2)
C5—H5···O1^{i}	0.93	2.48	3.292 (3)	145
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Fig. 1

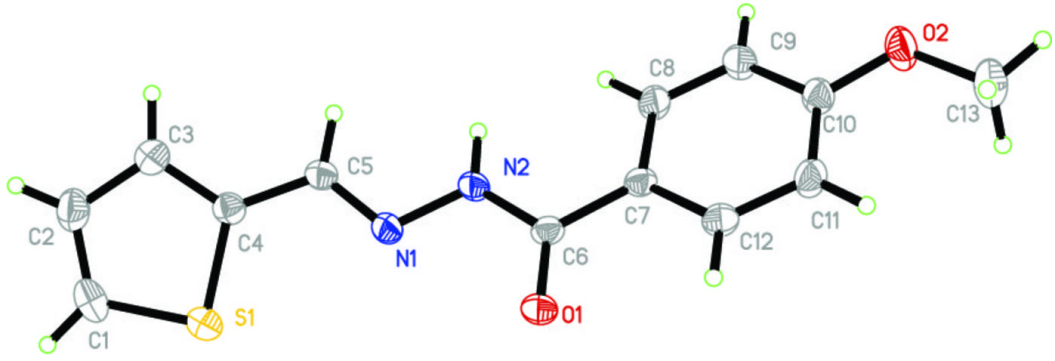

Fig. 2