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(*E*)-*N*′-[1-(Thiophen-2-yl)ethylidene]-benzohydrazide

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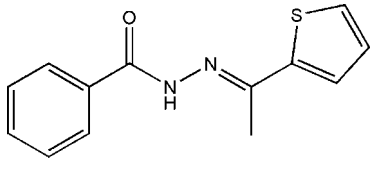
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.003 Å; R factor = 0.041; wR factor = 0.101; data-to-parameter ratio = 13.9.

The title compound, $C_{13}H_{12}N_2OS$, was obtained from the condensation reaction of 2-acetylthiophene and benzohydrazide. In the molecule, the formohydrazide fragment is approximately planar (r.m.s deviation = 0.0146 Å) and the mean plane is oriented at dihedral angles of 24.47 (11) and 28.86 (13)°, respectively, to the phenyl and thiophene rings. The thiophene and phenyl rings make a dihedral angle of 53.21 (8)°. The benzamide fragment and thiophene ring are located on the opposite sides of the C=N bond, showing an *E* conformation. Classical intermolecular N—H···O hydrogen bonds and weak C—H···O interactions are present in the crystal structure: three such bonds occur to the same O-atom acceptor.

Related literature

For applications of hydrazone derivatives in the biological field, see: Okabe *et al.* (1993). For general background to this work, see: Qiang *et al.* (2007). For a related structures, see: Xia *et al.* (2009); Shan *et al.* (2011)



Experimental

Crystal data

$C_{13}H_{12}N_2OS$ M_r = 244.31
Orthorhombic, *Pbca*
a = 9.906 (3) Å
b = 10.542 (5) Å
c = 22.870 (5) Å
V = 2388.3 (14) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.26 mm^{-1}
T = 294 K
0.32 × 0.29 × 0.28 mm

Data collection

Rigaku R-AXIS RAPID IP diffractometer
7859 measured reflections
2153 independent reflections
1552 reflections with $I > 2\sigma(I)$
R_{int} = 0.036

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.101
S = 1.03
2153 reflections
155 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1^{i}	0.86	2.50	3.340 (3)	166
C2—H2···O1^{i}	0.93	2.43	3.251 (3)	147
C13—H13A···O1^{i}	0.96	2.40	3.246 (3)	147

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

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2002); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU6116).

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supporting information

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(*E*)-*N*′-[1-(Thiophen-2-yl)ethylidene]benzohydrazide

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S1. Comment

The hydrazone derivatives has attracted our much attention because they have shown to be potential DNA damaging and mutagenic agents (Okabe *et al.*, 1993). As part of the ongoing investigation on the relationship between structure and property of hydrazone derivatives (Qiang *et al.*, 2007) the title compound has recently been prepared in our laboratory and its crystal structure is reported here.

The molecular structure of the title compound is shown in Fig. 1. In the molecule, the formohydrazide fragment is approximately co-planar [r.m.s deviation = 0.0146 Å] and the mean plane is oriented with respect to the phenyl ring and thiophene ring at 24.47 (11) and 28.86 (13)°, respectively. The N2—C8 bond length of 1.286 (2) Å shows a typical C═N double bond. The thiophene and benzamide units are located on the opposite sites of the C═N bond, showing an E configuration.

Intermolecular N—H···O and weak C—H···O hydrogen bonding is present in the crystal structure (Table 1).

S2. Experimental

Benzohydrazide (0.68 g, 5 mmol) was dissolved in ethanol (25 ml), then acetic acid (0.2 ml) was added to the ethanol solution with stirring. The solution was heated at about 333 K for several minutes until it became clear. 2-Acetylthiophene (0.63 g, 5 mmol) was then added slowly into the solution, and the mixture solution was refluxed for 6 h. After cooling to room temperature, yellow microcrystals appeared. The microcrystals were separated from the solution and washed with cold water three times. Recrystallization was performed twice with absolute methanol to obtain single crystals of the title compound.

S3. Refinement

H atoms were placed in calculated positions with C—H = 0.93 (aromatic), 0.96 Å (methyl) and N—H = 0.86 Å, and refined in riding mode with U_{iso}(H) = 1.5U_{eq}(C) for methyl H atoms and 1.2U_{eq}(C,N) for the others.

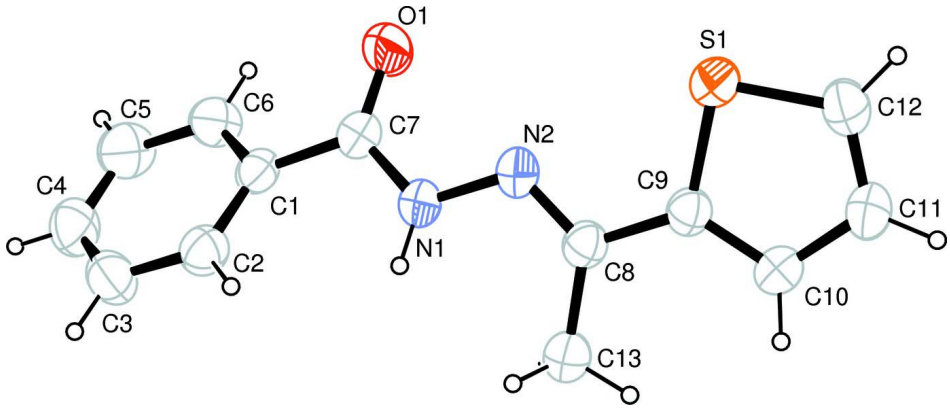

Figure 1

The molecular structure of the title compound with 40% probability displacement (arbitrary spheres for H atoms).

(*E*)-*N*′-[1-(Thiophen-2-yl)ethylidene]benzohydrazide

Crystal data

$C_{13}H_{12}N_2OS$
$M_r = 244.31$
Orthorhombic, *Pbca*
Hall symbol: -P 2ac 2ab
$a = 9.906$ (3) Å
$b = 10.542$ (5) Å
$c = 22.870$ (5) Å
$V = 2388.3$ (14) Å^3
$Z = 8$
$F(000) = 1024$
$D_x = 1.359$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2153 reflections
$\theta = 3.3$–25.2°
$\mu = 0.26$ mm^{-1}
$T = 294$ K
Block, yellow
0.32 × 0.29 × 0.28 mm

Data collection

Rigaku R-AXIS RAPID IP diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 10.0 pixels mm^{-1}
ω scans
7859 measured reflections
2153 independent reflections
1552 reflections with $I > 2\sigma(I)$
$R_{int} = 0.036$
$\theta_{max} = 25.2°$, $\theta_{min} = 3.3°$
$h = -10 \rightarrow 11$
$k = -11 \rightarrow 12$
$l = -27 \rightarrow 23$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.041$
$wR(F^2) = 0.101$
$S = 1.03$
2153 reflections
155 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0465P)^2 + 0.4798P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.20$ e Å^{-3}
$\Delta\rho_{min} = -0.17$ e Å^{-3}

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 $F^2 > \sigma(F^2)$ 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.61206 (5)	0.31374 (5)	0.02223 (3)	0.0507 (2)
N1	0.30582 (15)	0.50999 (16)	0.12073 (8)	0.0425 (5)
H1	0.3095	0.5895	0.1290	0.051*
N2	0.41317 (15)	0.45043 (16)	0.09296 (8)	0.0422 (4)
O1	0.19122 (15)	0.32660 (14)	0.12656 (9)	0.0679 (5)
C1	0.08229 (18)	0.51074 (18)	0.16290 (10)	0.0376 (5)
C2	0.0570 (2)	0.6393 (2)	0.15591 (10)	0.0462 (6)
H2	0.1126	0.6881	0.1321	0.055*
C3	−0.0508 (2)	0.6949 (2)	0.18432 (11)	0.0549 (6)
H3	−0.0675	0.7810	0.1794	0.066*
C4	−0.1335 (2)	0.6245 (3)	0.21977 (11)	0.0583 (7)
H4	−0.2050	0.6629	0.2393	0.070*
C5	−0.1101 (2)	0.4972 (2)	0.22631 (12)	0.0606 (7)
H5	−0.1664	0.4490	0.2501	0.073*
C6	−0.0040 (2)	0.4405 (2)	0.19801 (10)	0.0495 (6)
H6	0.0103	0.3538	0.2024	0.059*
C7	0.19588 (19)	0.4411 (2)	0.13447 (10)	0.0423 (5)
C8	0.5283 (2)	0.50629 (18)	0.09602 (10)	0.0377 (5)
C9	0.63941 (18)	0.44513 (18)	0.06501 (9)	0.0377 (5)
C10	0.7727 (2)	0.4768 (2)	0.06465 (11)	0.0488 (6)
H10	0.8080	0.5451	0.0853	0.059*
C11	0.8514 (2)	0.3953 (2)	0.02980 (11)	0.0549 (7)
H11	0.9441	0.4043	0.0249	0.066*
C12	0.7782 (2)	0.3029 (2)	0.00428 (11)	0.0509 (6)
H12	0.8141	0.2409	−0.0201	0.061*
C13	0.5569 (2)	0.6255 (2)	0.12900 (11)	0.0523 (6)
H13A	0.5023	0.6930	0.1137	0.078*
H13B	0.6506	0.6471	0.1249	0.078*
H13C	0.5362	0.6130	0.1696	0.078*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0387 (3)	0.0543 (4)	0.0590 (4)	−0.0030 (3)	−0.0008 (3)	−0.0143 (3)
N1	0.0321 (9)	0.0425 (9)	0.0529 (13)	0.0001 (8)	0.0042 (8)	−0.0043 (8)
N2	0.0328 (9)	0.0455 (9)	0.0484 (12)	0.0030 (8)	0.0026 (8)	−0.0031 (8)

O1	0.0465 (9)	0.0458 (9)	0.1112 (16)	−0.0032 (7)	0.0204 (9)	−0.0110 (9)
C1	0.0319 (10)	0.0441 (11)	0.0368 (13)	−0.0032 (9)	−0.0021 (9)	−0.0031 (9)
C2	0.0422 (12)	0.0472 (12)	0.0491 (16)	−0.0032 (10)	0.0022 (11)	0.0017 (10)
C3	0.0555 (14)	0.0501 (13)	0.0591 (17)	0.0100 (11)	0.0014 (12)	−0.0064 (12)
C4	0.0485 (13)	0.0730 (17)	0.0533 (17)	0.0060 (13)	0.0099 (12)	−0.0136 (13)
C5	0.0592 (15)	0.0688 (16)	0.0538 (18)	−0.0099 (13)	0.0207 (13)	−0.0064 (12)
C6	0.0537 (13)	0.0479 (12)	0.0470 (15)	−0.0034 (11)	0.0064 (11)	−0.0016 (11)
C7	0.0344 (11)	0.0441 (12)	0.0485 (15)	−0.0015 (10)	−0.0023 (10)	−0.0020 (10)
C8	0.0345 (10)	0.0393 (10)	0.0392 (13)	0.0005 (9)	−0.0022 (10)	0.0023 (9)
C9	0.0346 (10)	0.0382 (10)	0.0402 (13)	0.0003 (9)	−0.0007 (9)	0.0029 (9)
C10	0.0375 (11)	0.0448 (12)	0.0641 (17)	−0.0057 (10)	0.0032 (11)	−0.0064 (11)
C11	0.0354 (11)	0.0549 (13)	0.0744 (19)	0.0005 (10)	0.0097 (12)	−0.0043 (13)
C12	0.0437 (12)	0.0548 (14)	0.0542 (16)	0.0068 (11)	0.0061 (11)	−0.0068 (12)
C13	0.0389 (11)	0.0533 (13)	0.0646 (18)	−0.0006 (10)	0.0028 (11)	−0.0126 (12)

Geometric parameters (Å, º)

S1—C12	1.700 (2)	C4—H4	0.9300
S1—C9	1.717 (2)	C5—C6	1.371 (3)
N1—C7	1.346 (2)	C5—H5	0.9300
N1—N2	1.389 (2)	C6—H6	0.9300
N1—H1	0.8600	C8—C9	1.459 (3)
N2—C8	1.286 (2)	C8—C13	1.493 (3)
O1—C7	1.222 (2)	C9—C10	1.361 (3)
C1—C6	1.387 (3)	C10—C11	1.407 (3)
C1—C2	1.388 (3)	C10—H10	0.9300
C1—C7	1.492 (3)	C11—C12	1.347 (3)
C2—C3	1.380 (3)	C11—H11	0.9300
C2—H2	0.9300	C12—H12	0.9300
C3—C4	1.371 (3)	C13—H13A	0.9600
C3—H3	0.9300	C13—H13B	0.9600
C4—C5	1.370 (3)	C13—H13C	0.9600
C12—S1—C9	92.23 (10)	O1—C7—C1	121.45 (18)
C7—N1—N2	118.85 (17)	N1—C7—C1	116.53 (18)
C7—N1—H1	120.6	N2—C8—C9	116.11 (18)
N2—N1—H1	120.6	N2—C8—C13	125.55 (19)
C8—N2—N1	116.57 (17)	C9—C8—C13	118.34 (17)
C6—C1—C2	118.51 (19)	C10—C9—C8	128.73 (19)
C6—C1—C7	117.01 (18)	C10—C9—S1	110.31 (16)
C2—C1—C7	124.48 (19)	C8—C9—S1	120.95 (14)
C3—C2—C1	120.0 (2)	C9—C10—C11	113.0 (2)
C3—C2—H2	120.0	C9—C10—H10	123.5
C1—C2—H2	120.0	C11—C10—H10	123.5
C4—C3—C2	120.7 (2)	C12—C11—C10	112.8 (2)
C4—C3—H3	119.6	C12—C11—H11	123.6
C2—C3—H3	119.6	C10—C11—H11	123.6
C5—C4—C3	119.6 (2)	C11—C12—S1	111.59 (17)

C5—C4—H4	120.2	C11—C12—H12	124.2
C3—C4—H4	120.2	S1—C12—H12	124.2
C4—C5—C6	120.3 (2)	C8—C13—H13A	109.5
C4—C5—H5	119.8	C8—C13—H13B	109.5
C6—C5—H5	119.8	H13A—C13—H13B	109.5
C5—C6—C1	120.8 (2)	C8—C13—H13C	109.5
C5—C6—H6	119.6	H13A—C13—H13C	109.5
C1—C6—H6	119.6	H13B—C13—H13C	109.5
O1—C7—N1	121.93 (19)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{i}	0.86	2.50	3.340 (3)	166
C2—H2···O1^{i}	0.93	2.43	3.251 (3)	147
C13—H13*A*···O1^{i}	0.96	2.40	3.246 (3)	147

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