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

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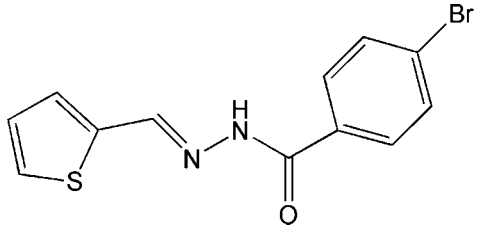
Key indicators: single-crystal X-ray study; T = 294 K; mean σ(C–C) = 0.006 Å; R factor = 0.047; wR factor = 0.148; data-to-parameter ratio = 16.0.

The title compound, $C_{12}H_9BrN_2OS$, contains planar thiophene and benzene rings [dihedral angle = 22.10 (3)°] bridged by a chain containing an N—N bond. The structure is stabilized by a weak intermolecular N—H···O hydrogen bond to form a zigzag packing arrangement.

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

$C_{12}H_9BrN_2OS$
M_r = 309.18
Orthorhombic, *Pbca*
a = 12.807 (6) Å
b = 7.803 (4) Å
c = 24.734 (11) Å
V = 2471.9 (19) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 3.48 mm^{-1}
T = 294 (2) K
0.22 × 0.20 × 0.16 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.49, T_{max} = 0.57
13066 measured reflections
2525 independent reflections
1626 reflections with $I > 2\sigma(I)$
R_{int} = 0.056

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.047
$wR(F^2)$ = 0.148
S = 1.03
2525 reflections
158 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.60 e Å^{-3}
$\Delta\rho_{min}$ = −0.72 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}	0.90 (3)	2.05 (3)	2.953 (4)	176 (4)

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

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: BG2091).

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supplementary materials

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

Z.-L. Jing, M. Yu and X. Chen

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 the 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) (Fig. 1). The geometric parameters are normal. The two main groups in the molecule are planar within experimental error (maximum deviations from the l.s. planes: 0.0069 (2) Å for S1 in the thiophen ring, and 0.0117 (3) Å in the benzene group. The dihedral angle between these two planes is 22.10 (3)°·The molecular structure is stabilized by a weak intermolecular N—H···O hydrogen bond to form a zigzag packing arrangement, as illustraed in Fig.2 and table 1.

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-bromobenzohydrazide (2.14 g, 10 mmol), and the mixture was stirred at 350 K for 6 h under N_2, whereupon a red precipitate appeared. The product was isolated, recrystallized from anhydrous ethanol and then dried *in vacuo* to give the pure compound (I) in a 92% yield. Red 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 its positional parameters were refined, with U_{iso}(H) = 1.2U_{eq}(N). 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

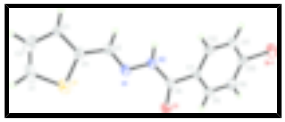

Fig. 1. The molecular structure of (I). Displacement ellipsoids are drawn at the 30% probability level.

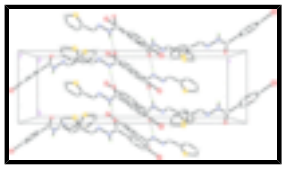

Fig. 2. The crystal packing of (I), viewed down the *b* axis. Hydrogen bonds are indicated by dashed lines.

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

Crystal data

$C_{12}H_9BrN_2OS$	F_{000} = 1232
M_r = 309.18	D_x = 1.662 Mg m^{-3}
Orthorhombic, *Pbca*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -P 2ac 2ab	Cell parameters from 3032 reflections
a = 12.807 (6) Å	θ = 2.3–24.0º
b = 7.803 (4) Å	μ = 3.48 mm^{-1}
c = 24.734 (11) Å	*T* = 294 (2) K
V = 2471.9 (19) Å^3	Block, red
Z = 8	0.22 × 0.20 × 0.16 mm

Data collection

Bruker SMART CCD area-detector diffractometer	2525 independent reflections
Radiation source: fine-focus sealed tube	1626 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.056
T = 294(2) K	θ_{max} = 26.4º
φ and ω scans	θ_{min} = 1.7º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	*h* = −16→15
T_{min} = 0.49, T_{max} = 0.57	*k* = −4→9
13066 measured reflections	*l* = −30→30

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.047	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.148	$w = 1/[\sigma^2(F_o^2) + (0.074P)^2 + 2.5533P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.03	$(\Delta/\sigma)_{max}$ = 0.001
2525 reflections	$\Delta\rho_{max}$ = 0.60 e Å^{-3}
158 parameters	$\Delta\rho_{min}$ = −0.72 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 $F^2 > 2\text{sigma}(F^2)$ 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}
Br1	0.92242 (5)	1.05663 (8)	0.62759 (2)	0.0745 (3)
S1	0.70401 (13)	0.4410 (2)	0.23687 (6)	0.0801 (5)
O1	0.8747 (2)	0.4971 (4)	0.41694 (12)	0.0445 (7)
N1	0.7283 (3)	0.5996 (5)	0.34649 (13)	0.0429 (8)
N2	0.7489 (3)	0.6861 (4)	0.39387 (14)	0.0434 (8)
C1	0.6256 (6)	0.4326 (8)	0.1825 (2)	0.0818 (19)
H1	0.6381	0.3622	0.1529	0.098*
C2	0.5439 (5)	0.5367 (7)	0.1862 (2)	0.0765 (18)
H2	0.4944	0.5456	0.1588	0.092*
C3	0.5375 (3)	0.6311 (5)	0.23339 (15)	0.0386 (9)
H3	0.4846	0.7075	0.2425	0.046*
C4	0.6293 (4)	0.5892 (5)	0.26673 (17)	0.0447 (10)
C5	0.6557 (3)	0.6618 (6)	0.31812 (16)	0.0456 (10)
H5	0.6187	0.7562	0.3308	0.055*
C6	0.8247 (3)	0.6283 (5)	0.42666 (15)	0.0365 (9)
C7	0.8448 (3)	0.7303 (5)	0.47612 (15)	0.0372 (9)
C8	0.9451 (3)	0.7347 (6)	0.49632 (19)	0.0493 (11)
H8	0.9976	0.6730	0.4792	0.059*
C9	0.9682 (4)	0.8297 (6)	0.54165 (19)	0.0573 (13)
H9	1.0362	0.8340	0.5548	0.069*
C10	0.8900 (4)	0.9180 (5)	0.56724 (17)	0.0463 (11)
C11	0.7888 (3)	0.9092 (6)	0.54958 (17)	0.0475 (11)
H11	0.7358	0.9650	0.5683	0.057*
C12	0.7673 (3)	0.8164 (5)	0.50373 (17)	0.0454 (10)
H12	0.6990	0.8115	0.4910	0.054*
H2A	0.710 (3)	0.781 (3)	0.3991 (17)	0.045 (12)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.0907 (5)	0.0819 (5)	0.0510 (3)	−0.0025 (3)	−0.0156 (3)	−0.0265 (3)
S1	0.0900 (11)	0.0857 (11)	0.0647 (9)	0.0106 (9)	−0.0062 (8)	−0.0259 (8)

O1	0.0443 (16)	0.0409 (15)	0.0483 (17)	0.0057 (14)	−0.0006 (14)	−0.0114 (13)
N1	0.049 (2)	0.0407 (19)	0.0392 (19)	−0.0004 (16)	−0.0042 (16)	−0.0104 (16)
N2	0.052 (2)	0.039 (2)	0.0396 (18)	0.0076 (17)	−0.0075 (17)	−0.0123 (16)
C1	0.129 (5)	0.075 (4)	0.041 (3)	−0.031 (4)	−0.002 (3)	−0.014 (3)
C2	0.094 (4)	0.067 (4)	0.069 (4)	−0.026 (3)	−0.034 (3)	0.027 (3)
C3	0.050 (2)	0.0297 (19)	0.036 (2)	−0.0009 (18)	−0.0087 (19)	0.0031 (17)
C4	0.054 (3)	0.041 (2)	0.039 (2)	−0.004 (2)	−0.005 (2)	−0.0002 (18)
C5	0.053 (3)	0.042 (2)	0.042 (2)	0.003 (2)	−0.001 (2)	−0.0065 (19)
C6	0.039 (2)	0.034 (2)	0.036 (2)	−0.0035 (18)	0.0050 (17)	−0.0034 (17)
C7	0.042 (2)	0.035 (2)	0.035 (2)	−0.0008 (18)	−0.0033 (17)	−0.0019 (16)
C8	0.041 (2)	0.051 (3)	0.056 (3)	0.010 (2)	−0.005 (2)	−0.013 (2)
C9	0.048 (3)	0.065 (3)	0.058 (3)	0.004 (2)	−0.019 (2)	−0.018 (2)
C10	0.062 (3)	0.041 (2)	0.035 (2)	−0.001 (2)	−0.010 (2)	−0.0032 (19)
C11	0.053 (3)	0.049 (3)	0.041 (2)	0.006 (2)	0.000 (2)	−0.007 (2)
C12	0.040 (2)	0.054 (3)	0.042 (2)	0.004 (2)	−0.0024 (18)	−0.008 (2)

Geometric parameters (Å, °)

Br1—C10	1.890 (4)	C4—C5	1.432 (6)
S1—C4	1.673 (5)	C5—H5	0.9300
S1—C1	1.679 (6)	C6—C7	1.482 (5)
O1—C6	1.231 (5)	C7—C8	1.379 (5)
N1—C5	1.263 (5)	C7—C12	1.379 (5)
N1—N2	1.378 (5)	C8—C9	1.376 (6)
N2—C6	1.344 (5)	C8—H8	0.9300
N2—H2A	0.90 (3)	C9—C10	1.370 (6)
C1—C2	1.328 (9)	C9—H9	0.9300
C1—H1	0.9300	C10—C11	1.369 (6)
C2—C3	1.383 (7)	C11—C12	1.374 (6)
C2—H2	0.9300	C11—H11	0.9300
C3—C4	1.473 (6)	C12—H12	0.9300
C3—H3	0.9300		
C4—S1—C1	92.2 (3)	O1—C6—C7	121.2 (4)
C5—N1—N2	115.1 (3)	N2—C6—C7	116.3 (3)
C6—N2—N1	119.2 (3)	C8—C7—C12	118.6 (4)
C6—N2—H2A	126 (3)	C8—C7—C6	118.3 (4)
N1—N2—H2A	115 (3)	C12—C7—C6	123.1 (4)
C2—C1—S1	113.1 (4)	C9—C8—C7	120.6 (4)
C2—C1—H1	123.4	C9—C8—H8	119.7
S1—C1—H1	123.4	C7—C8—H8	119.7
C1—C2—C3	115.5 (5)	C10—C9—C8	119.4 (4)
C1—C2—H2	122.3	C10—C9—H9	120.3
C3—C2—H2	122.3	C8—C9—H9	120.3
C2—C3—C4	107.9 (4)	C11—C10—C9	121.3 (4)
C2—C3—H3	126.1	C11—C10—Br1	119.2 (3)
C4—C3—H3	126.1	C9—C10—Br1	119.5 (3)
C5—C4—C3	126.7 (4)	C10—C11—C12	118.7 (4)
C5—C4—S1	122.0 (3)	C10—C11—H11	120.7
C3—C4—S1	111.3 (3)	C12—C11—H11	120.7

N1—C5—C4	121.0 (4)	C11—C12—C7	121.4 (4)
N1—C5—H5	119.5	C11—C12—H12	119.3
C4—C5—H5	119.5	C7—C12—H12	119.3
O1—C6—N2	122.5 (4)		
C5—N1—N2—C6	179.7 (4)	N2—C6—C7—C8	−148.6 (4)
C4—S1—C1—C2	−0.6 (5)	O1—C6—C7—C12	−145.9 (4)
S1—C1—C2—C3	−0.4 (7)	N2—C6—C7—C12	33.4 (6)
C1—C2—C3—C4	1.5 (6)	C12—C7—C8—C9	−3.0 (7)
C2—C3—C4—C5	177.8 (4)	C6—C7—C8—C9	179.0 (4)
C2—C3—C4—S1	−1.9 (5)	C7—C8—C9—C10	1.1 (7)
C1—S1—C4—C5	−178.3 (4)	C8—C9—C10—C11	2.1 (7)
C1—S1—C4—C3	1.4 (4)	C8—C9—C10—Br1	−177.2 (4)
N2—N1—C5—C4	177.2 (4)	C9—C10—C11—C12	−3.2 (7)
C3—C4—C5—N1	170.2 (4)	Br1—C10—C11—C12	176.1 (3)
S1—C4—C5—N1	−10.2 (6)	C10—C11—C12—C7	1.2 (7)
N1—N2—C6—O1	−2.5 (6)	C8—C7—C12—C11	1.8 (6)
N1—N2—C6—C7	178.3 (3)	C6—C7—C12—C11	179.8 (4)
O1—C6—C7—C8	32.1 (6)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2A···O1^{i}	0.90 (3)	2.05 (3)	2.953 (4)	176 (4)

Symmetry codes: (i) −*x*+3/2, *y*+1/2, *z*.

Fig. 1

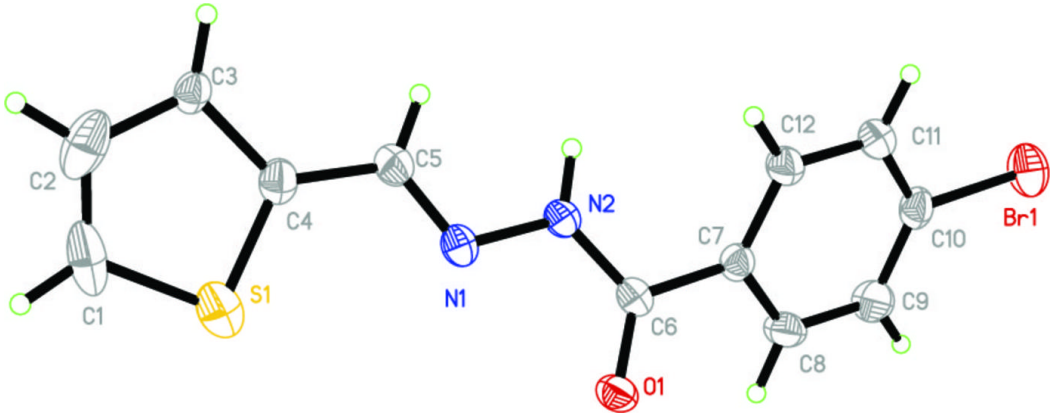

Fig. 2