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(*E*)-*N*′-(2,4-Dichlorobenzylidene)-thiophene-2-carbohydrazide

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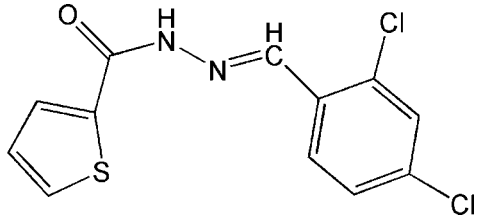
Key indicators: single-crystal X-ray study; T = 113 K; mean σ(C–C) = 0.003 Å; R factor = 0.050; wR factor = 0.097; data-to-parameter ratio = 17.4.

In the title compound, $C_{12}H_8Cl_2N_2OS$, the dihedral angle between the two ring planes is 14.30 (3)°. The molecules are linked *via* a weak intermolecular N—H···O hydrogen bond, forming an extended supramolecular structure.

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

$C_{12}H_8Cl_2N_2OS$
M_r = 299.16
Monoclinic, $P2_1/n$
a = 5.6320 (11) Å
b = 16.664 (3) Å
c = 13.077 (3) Å
β = 96.10 (3)°
V = 1220.3 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.69 mm^{-1}
T = 113 (2) K
0.10 × 0.06 × 0.04 mm

Data collection

Rigaku Saturn diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku/MSC, 2005)
T_{min} = 0.934, T_{max} = 0.973
9849 measured reflections
2911 independent reflections
2370 reflections with $I > 2\sigma(I)$
R_{int} = 0.064

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.097
S = 1.10
2911 reflections
167 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.36 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O1^{i}	0.88 (3)	1.96 (3)	2.843 (3)	177 (3)

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

Data collection: *CrystalClear* (Rigaku/MSC, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *CrystalStructure* (Rigaku/MSC, 2005); software used to prepare material for publication: *CrystalStructure*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2371).

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supplementary materials

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(*E*)-*N*'-(2,4-Dichlorobenzylidene)thiophene-2-carbohydrazide

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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 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 Schiff bases functioning as ligands, we report the synthesis and structure of the title compound, (I).

In the structure of the title molecule, (I) (Fig. 1), the geometric parameters are normal. The thiophene ring system (C9—C12/S1) is planar, with an r.m.s. deviation for fitted atoms of 0.0032 (5) Å; the benzene group (C1—C6) is also planar, with an r.m.s. deviation of 0.0053 (2) Å. The dihedral angle between these planes is 14.30 (3)°.

The molecules are linked *via* weak intermolecular N—H···O hydrogen bond, forming an extended supramolecule (Table 1). The molecules associate to form a supramolecular structure, as illustrated in Fig. 2.

Experimental

An anhydrous ethanol solution (50 ml) of thiophene-2-carbohydrazide (1.42 g, 10 mmol) was added to an anhydrous ethanol solution (50 ml) of 2,4-dichlorobenzaldehyde (1.75 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 85% 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 freely. C-bound H atoms were included in calculated positions, with C—H = 0.95 Å, and refined using a riding model, with U_{iso}(H) = 1.2U_{eq}(C).

Figures

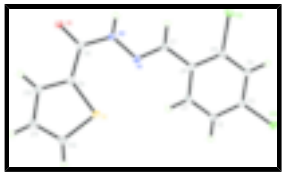

Fig. 1. The structure of the title molecule (I). Displacement ellipsoids are drawn at the 30% probability level.

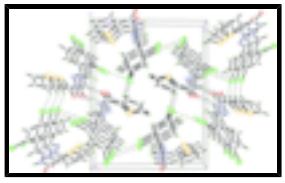

Fig. 2. The crystal packing of (I), viewed down the *c* axis. Hydrogen bonds are indicated by dashed lines.

(*E*)-*N*'-(2,4-Dichlorobenzylidene)thiophene-2-carbohydrazide

Crystal data

$C_{12}H_8Cl_2N_2OS$	F_{000} = 608
M_r = 299.16	D_x = 1.628 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 2966 reflections
a = 5.6320 (11) Å	θ = 1.9–28.0º
b = 16.664 (3) Å	μ = 0.69 mm^{-1}
c = 13.077 (3) Å	T = 113 (2) K
β = 96.10 (3)º	Block, yellow
V = 1220.3 (4) Å^3	0.10 × 0.06 × 0.04 mm
Z = 4	

Data collection

Rigaku Saturn diffractometer	2911 independent reflections
Radiation source: rotating anode	2370 reflections with $I > 2\sigma(I)$
Monochromator: confocal	R_{int} = 0.064
T = 113(2) K	θ_{max} = 27.9º
ω scans	θ_{min} = 2.0º
Absorption correction: multi-scan (CrystalClear, Rigaku/MSC, 2005)	h = −7→6
T_{min} = 0.934, T_{max} = 0.973	k = −21→21
9849 measured reflections	l = −15→17

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.050	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.097	$w = 1/[\sigma^2(F_o^2) + (0.0332P)^2 + 0.3458P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.10	$(\Delta/\sigma)_{max}$ = 0.001
2911 reflections	$\Delta\rho_{max}$ = 0.32 e Å^{-3}
167 parameters	$\Delta\rho_{min}$ = −0.36 e Å^{-3}

Primary atom site location: structure-invariant direct methods

Extinction correction: none

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}
Cl1	1.20133 (11)	0.60285 (4)	0.68221 (5)	0.02104 (16)
Cl2	0.84737 (12)	0.82985 (4)	0.42176 (4)	0.02250 (17)
S1	0.16921 (11)	0.87926 (4)	0.83555 (5)	0.02168 (17)
O1	−0.1664 (3)	1.00868 (11)	0.60854 (13)	0.0212 (4)
N1	0.3309 (4)	0.88243 (13)	0.63613 (15)	0.0168 (5)
N2	0.1711 (4)	0.94002 (14)	0.59904 (16)	0.0187 (5)
H2	0.164 (5)	0.956 (2)	0.535 (2)	0.042 (9)*
C1	0.6565 (4)	0.75502 (16)	0.69296 (18)	0.0186 (5)
H1	0.5405	0.7671	0.7384	0.022*
C2	0.8196 (4)	0.69461 (16)	0.71835 (18)	0.0189 (6)
H2A	0.8139	0.6645	0.7797	0.023*
C3	0.9920 (4)	0.67847 (16)	0.65305 (18)	0.0166 (5)
C4	1.0012 (4)	0.72071 (16)	0.56257 (18)	0.0175 (5)
H4	1.1207	0.7094	0.5185	0.021*
C5	0.8331 (4)	0.77977 (16)	0.53762 (17)	0.0164 (5)
C6	0.6578 (4)	0.79905 (16)	0.60175 (18)	0.0167 (5)
C7	0.4807 (4)	0.86188 (16)	0.57394 (18)	0.0173 (5)
H7	0.4771	0.8877	0.5090	0.021*
C8	−0.0114 (4)	0.96340 (16)	0.65136 (18)	0.0173 (5)
C9	−0.0280 (4)	0.93785 (15)	0.75774 (18)	0.0168 (5)
C10	−0.2147 (4)	0.96278 (17)	0.80989 (18)	0.0197 (6)
H10	−0.3423	0.9953	0.7801	0.024*
C11	−0.1964 (5)	0.93495 (17)	0.91240 (19)	0.0227 (6)
H11	−0.3099	0.9465	0.9591	0.027*
C12	0.0027 (5)	0.88965 (17)	0.9365 (2)	0.0238 (6)
H12	0.0445	0.8664	1.0022	0.029*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0206 (3)	0.0192 (4)	0.0233 (3)	0.0029 (3)	0.0022 (2)	0.0010 (3)

Cl2	0.0298 (4)	0.0213 (4)	0.0172 (3)	0.0001 (3)	0.0065 (3)	0.0026 (3)
S1	0.0198 (3)	0.0241 (4)	0.0212 (3)	0.0033 (3)	0.0022 (3)	0.0060 (3)
O1	0.0196 (9)	0.0229 (11)	0.0212 (9)	0.0041 (8)	0.0023 (7)	0.0066 (8)
N1	0.0159 (11)	0.0151 (12)	0.0193 (10)	−0.0005 (9)	0.0006 (8)	0.0006 (9)
N2	0.0200 (11)	0.0179 (12)	0.0183 (11)	0.0031 (9)	0.0030 (9)	0.0025 (9)
C1	0.0194 (13)	0.0189 (14)	0.0178 (12)	−0.0023 (11)	0.0034 (10)	−0.0004 (10)
C2	0.0232 (14)	0.0168 (14)	0.0166 (12)	−0.0015 (11)	0.0020 (10)	0.0017 (10)
C3	0.0157 (12)	0.0143 (14)	0.0194 (12)	0.0001 (10)	0.0004 (10)	−0.0030 (10)
C4	0.0161 (12)	0.0169 (14)	0.0198 (12)	−0.0033 (11)	0.0026 (10)	−0.0027 (10)
C5	0.0190 (13)	0.0157 (14)	0.0144 (11)	−0.0058 (11)	0.0010 (10)	−0.0006 (10)
C6	0.0152 (12)	0.0163 (14)	0.0186 (12)	−0.0008 (10)	0.0021 (10)	−0.0029 (10)
C7	0.0198 (13)	0.0141 (13)	0.0176 (12)	−0.0018 (11)	0.0003 (10)	0.0038 (10)
C8	0.0172 (12)	0.0128 (13)	0.0218 (12)	−0.0054 (10)	0.0016 (10)	−0.0001 (10)
C9	0.0147 (12)	0.0145 (14)	0.0208 (12)	−0.0024 (10)	0.0003 (10)	0.0010 (10)
C10	0.0190 (13)	0.0196 (15)	0.0201 (12)	0.0001 (11)	0.0008 (10)	0.0008 (11)
C11	0.0234 (14)	0.0238 (16)	0.0215 (13)	−0.0002 (12)	0.0050 (11)	0.0014 (11)
C12	0.0241 (14)	0.0261 (16)	0.0212 (13)	0.0004 (12)	0.0020 (11)	0.0057 (11)

Geometric parameters (Å, °)

Cl1—C3	1.740 (3)	C3—C4	1.383 (3)
Cl2—C5	1.739 (2)	C4—C5	1.381 (3)
S1—C12	1.707 (3)	C4—H4	0.9500
S1—C9	1.727 (3)	C5—C6	1.399 (3)
O1—C8	1.241 (3)	C6—C7	1.465 (4)
N1—C7	1.279 (3)	C7—H7	0.9500
N1—N2	1.369 (3)	C8—C9	1.467 (3)
N2—C8	1.351 (3)	C9—C10	1.377 (3)
N2—H2	0.88 (3)	C10—C11	1.412 (3)
C1—C2	1.379 (4)	C10—H10	0.9500
C1—C6	1.401 (3)	C11—C12	1.361 (4)
C1—H1	0.9500	C11—H11	0.9500
C2—C3	1.386 (3)	C12—H12	0.9500
C2—H2A	0.9500		
C12—S1—C9	91.51 (13)	C5—C6—C7	121.4 (2)
C7—N1—N2	114.6 (2)	C1—C6—C7	121.4 (2)
C8—N2—N1	122.0 (2)	N1—C7—C6	120.7 (2)
C8—N2—H2	116 (2)	N1—C7—H7	119.6
N1—N2—H2	121 (2)	C6—C7—H7	119.6
C2—C1—C6	121.6 (2)	O1—C8—N2	118.9 (2)
C2—C1—H1	119.2	O1—C8—C9	119.4 (2)
C6—C1—H1	119.2	N2—C8—C9	121.7 (2)
C1—C2—C3	119.1 (2)	C10—C9—C8	120.7 (2)
C1—C2—H2A	120.5	C10—C9—S1	110.85 (18)
C3—C2—H2A	120.5	C8—C9—S1	128.38 (19)
C4—C3—C2	121.4 (2)	C9—C10—C11	112.9 (2)
C4—C3—Cl1	118.03 (19)	C9—C10—H10	123.6
C2—C3—Cl1	120.56 (19)	C11—C10—H10	123.6
C5—C4—C3	118.6 (2)	C12—C11—C10	112.1 (2)

C5—C4—H4	120.7	C12—C11—H11	123.9
C3—C4—H4	120.7	C10—C11—H11	123.9
C4—C5—C6	122.1 (2)	C11—C12—S1	112.6 (2)
C4—C5—Cl2	116.99 (19)	C11—C12—H12	123.7
C6—C5—Cl2	120.9 (2)	S1—C12—H12	123.7
C5—C6—C1	117.2 (2)		
C7—N1—N2—C8	174.2 (2)	C5—C6—C7—N1	−175.6 (2)
C6—C1—C2—C3	1.5 (4)	C1—C6—C7—N1	5.4 (4)
C1—C2—C3—C4	−1.0 (4)	N1—N2—C8—O1	−171.5 (2)
C1—C2—C3—Cl1	179.94 (19)	N1—N2—C8—C9	9.6 (4)
C2—C3—C4—C5	−0.3 (4)	O1—C8—C9—C10	0.8 (4)
Cl1—C3—C4—C5	178.72 (18)	N2—C8—C9—C10	179.6 (2)
C3—C4—C5—C6	1.3 (4)	O1—C8—C9—S1	−177.0 (2)
C3—C4—C5—Cl2	−178.62 (19)	N2—C8—C9—S1	1.9 (4)
C4—C5—C6—C1	−0.8 (4)	C12—S1—C9—C10	−0.6 (2)
Cl2—C5—C6—C1	179.06 (19)	C12—S1—C9—C8	177.3 (2)
C4—C5—C6—C7	−180.0 (2)	C8—C9—C10—C11	−177.7 (2)
Cl2—C5—C6—C7	−0.1 (3)	S1—C9—C10—C11	0.4 (3)
C2—C1—C6—C5	−0.6 (4)	C9—C10—C11—C12	0.1 (4)
C2—C1—C6—C7	178.5 (2)	C10—C11—C12—S1	−0.5 (3)
N2—N1—C7—C6	−178.0 (2)	C9—S1—C12—C11	0.7 (2)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O1^{i}	0.88 (3)	1.96 (3)	2.843 (3)	177 (3)

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

Fig. 1

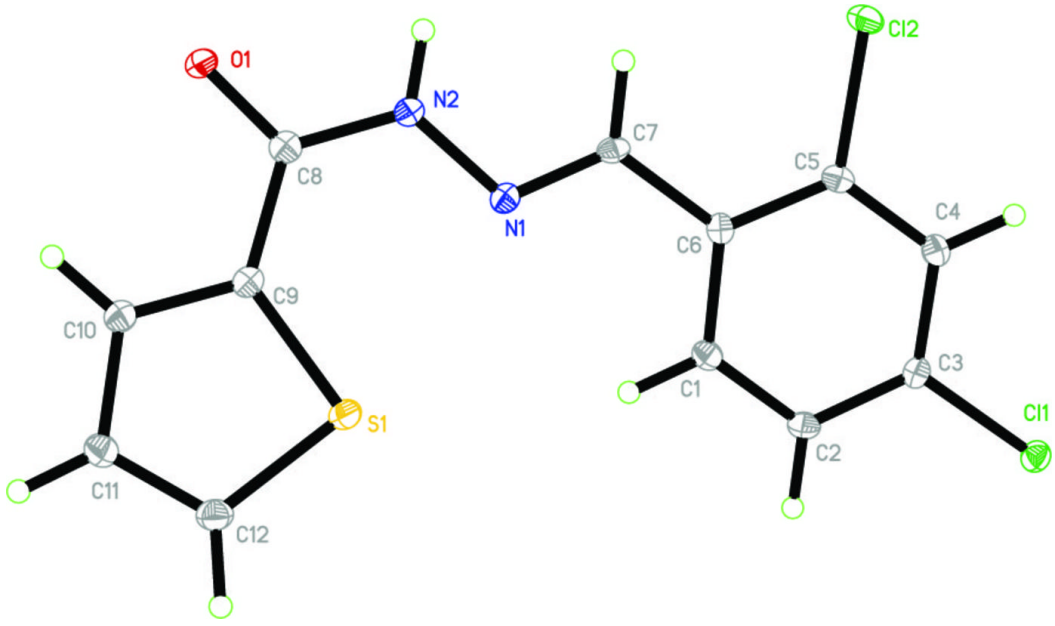

Fig. 2