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N′-[(1*E*)-(2,6-Difluorophenyl)methylidene]thiophene-2-carbohydrazide

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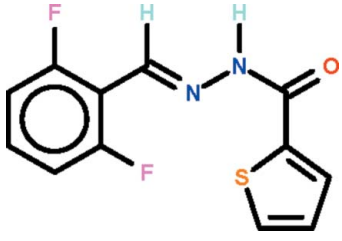
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; disorder in main residue; R factor = 0.038; wR factor = 0.108; data-to-parameter ratio = 14.4.

In the title compound, $C_{12}H_8F_2N_2OS$, the thienyl ring is disordered over two positions, with the S atom of the major component [occupancy = 75.03 (18)%] oriented away from an *ortho*-F atom of the benzene ring. The molecule is nearly planar, the dihedral angle between the thiophene and benzene rings being 6.19 (18) (in the major component) or 3.5 (6)° (in the minor component). The azomethine C=N double-bond in the molecule is of an *E* configuration. In the crystal, molecules are linked by pairs of N—H···O hydrogen bonds, generating inversion dimers.

Related literature

For a related structure, see: Alanazi *et al.* (2012).



Experimental

Crystal data

$C_{12}H_8F_2N_2OS$ M_r = 266.26
Triclinic, $P\overline{1}$
a = 6.5032 (3) Å
b = 7.7516 (4) Å
c = 11.5224 (5) Å
α = 95.184 (4)°
β = 103.344 (4)°
γ = 94.285 (4)°
V = 560.11 (5) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.30 mm^{-1}
T = 100 K
0.40 × 0.30 × 0.20 mm

Data collection

Agilent SuperNova Dual diffractometer with an Atlas detector
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2010)
T_{min} = 0.888, T_{max} = 0.942
8250 measured reflections
2594 independent reflections
2174 reflections with $I > 2\sigma(I)$
R_{int} = 0.035

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.108
S = 1.07
2594 reflections
180 parameters
24 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.33 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
N1—H1···O1^{i}	0.88 (2)	1.97 (2)	2.856 (2)	174 (2)

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

Data collection: *CrysAlis PRO* (Agilent, 2010); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: XU5416).

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Barbour, L. J. (2001). *J. Supramol. Chem.* **1**, 189–191.
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Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

supporting information

Acta Cryst. (2012). E**68**, o315 [doi:10.1107/S160053681105611X]

N′-[(1*E*)-(2,6-Difluorophenyl)methylidene]thiophene-2-carbohydrazide

Amer M. Alanazi, Adnan A. Kadi, Ali A. El-Emam and Seik Weng Ng

S1. Comment

2-Thienoylhydrazide forms a large number of Schiff base derivatives with substituted benzaldehydes; among those whose crystal structures have been reported are the 4-chloro and 4-bromo derivatives. The 4-fluoro analog is disordered in respect of the thienyl ring (Alanazi *et al.*, 2012). The azomethine double-bond in the approximately planar $C_{12}H_8F_2N_2OS$ molecule (Scheme I) is of an *E* configuration (Fig. 1). The thienyl ring is disordered over two positions, with the S atom of the major component (75.03 (18)%) oriented away from an *ortho*-F atom of the benzene ring. Two molecules are linked across a center-of-inversion by an N–H···O hydrogen bond to generate a dimer (Table 1).

S2. Experimental

2-Thienoylhydrazide (1.42 g, 0.01 mol) and 2,6-difluorobenzaldehyde (1.42 g, 0.01 mol) dissolved in ethanol (8 ml) was heated for 1 h. The product was collected and recystallized from ethanol to yield the Schiff base in 90% yield, m.p. 453–45548 K.

S3. Refinement

Carbon-bound H-atoms were placed in calculated positions [C–H 0.95 Å, U_{iso}(H) 1.2U_{eq}(C)] and were included in the refinement in the riding model approximation.

The amino H-atom was located in a difference Fourier map, and was refined freely.

The thiophene ring is disordered over two positions in respect of four of the five atoms, with major component being 87.1 (2) %. Pairs of C–C and C–S bond distances were restrained to within 0.0 Å of each other. The temperature factors of C3′ was set to those of S1 (as were these pairs: C2′ to C1, C1′ to C2 and S1′ to C3). The anisotropic temperature factors of the disordered atoms were tightly restrained to be nearly isotropic.

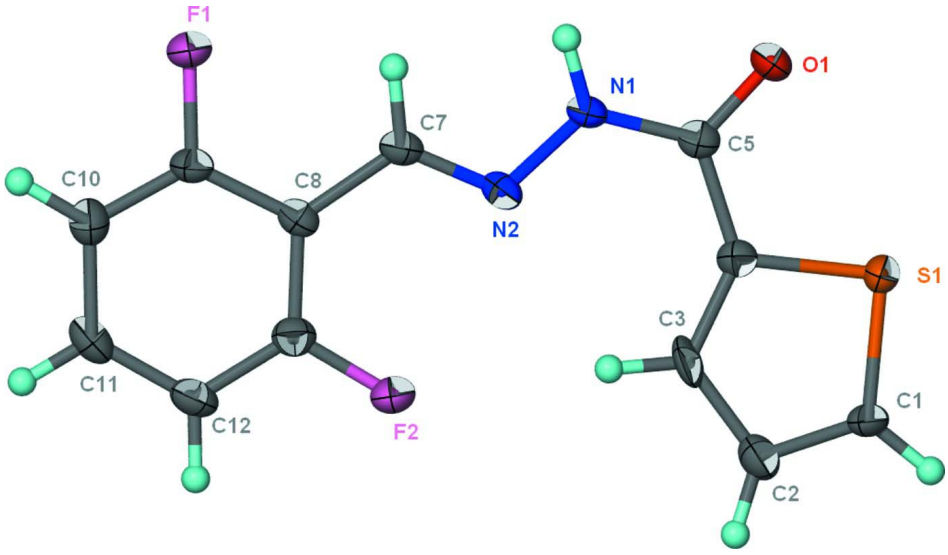

Figure 1

Thermal ellipsoid plot (Barbour, 2001) of $C_{12}H_8F_2N_2OS$ at the 70% probability level; hydrogen atoms are drawn as spheres of arbitrary radius. The disorder is not shown.

N′-[(1*E*)-(2,6-Difluorophenyl)methylidene]thiophene- 2-carbohydrazide

Crystal data

$C_{12}H_8F_2N_2OS$
$M_r = 266.26$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 6.5032$ (3) Å
$b = 7.7516$ (4) Å
$c = 11.5224$ (5) Å
$\alpha = 95.184$ (4)°
$\beta = 103.344$ (4)°
$\gamma = 94.285$ (4)°
$V = 560.11$ (5) Å^3
$Z = 2$
$F(000) = 272$
$D_x = 1.579$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3787 reflections
$\theta = 2.7$–$27.5°$
$\mu = 0.30$ mm^{-1}
$T = 100$ K
Wedge, colorless
0.40 × 0.30 × 0.20 mm

Data collection

Agilent SuperNova Dual diffractometer with an Atlas detector
Radiation source: SuperNova (Mo) X-ray Source
Mirror monochromator
Detector resolution: 10.4041 pixels mm^{-1}
ω scan
Absorption correction: multi-scan (*CrysAlis PRO*; Agilent, 2010)
$T_{min} = 0.888$, $T_{max} = 0.942$
8250 measured reflections
2594 independent reflections
2174 reflections with $I > 2\sigma(I)$
$R_{int} = 0.035$
$\theta_{max} = 27.6°$, $\theta_{min} = 2.7°$
$h = -8 \rightarrow 8$
$k = -10 \rightarrow 10$
$l = -15 \rightarrow 14$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.038$
$wR(F^2) = 0.108$
$S = 1.07$
2594 reflections
180 parameters
24 restraints
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0585P)^2 + 0.1004P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.33$ e Å^{-3}
$\Delta\rho_{min} = -0.28$ e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
S1	0.61235 (9)	0.40630 (10)	0.12814 (6)	0.01924 (19)	0.7503 (18)
S1′	0.9967 (5)	0.2911 (5)	0.2831 (4)	0.0226 (7)	0.2497 (18)
F1	1.02233 (15)	0.19692 (13)	0.85830 (8)	0.0285 (3)	
F2	1.26733 (15)	0.14995 (13)	0.50356 (8)	0.0270 (3)	
O1	0.47384 (17)	0.48037 (15)	0.34181 (10)	0.0228 (3)	
N1	0.7178 (2)	0.37086 (17)	0.47830 (12)	0.0188 (3)	
N2	0.8961 (2)	0.28597 (16)	0.51113 (11)	0.0178 (3)	
C1	0.8196 (10)	0.3496 (7)	0.0668 (6)	0.0214 (8)	0.7503 (18)
H1A	0.8126	0.3501	−0.0165	0.026*	0.7503 (18)
C2	0.9871 (9)	0.3058 (8)	0.1414 (3)	0.0199 (7)	0.7503 (18)
H2	1.1127	0.2725	0.1205	0.024*	0.7503 (18)
C3	0.9481 (7)	0.3168 (7)	0.2667 (5)	0.0226 (7)	0.7503 (18)
H3	1.0483	0.2915	0.3357	0.027*	0.7503 (18)
C1′	0.6702 (15)	0.4020 (15)	0.1526 (9)	0.01924 (19)	0.25
H1′	0.5350	0.4436	0.1277	0.023*	0.2497 (18)
C2′	0.801 (3)	0.371 (2)	0.074 (2)	0.0214 (8)	0.25
H2′	0.7827	0.3824	−0.0092	0.026*	0.2497 (18)
C3′	0.983 (3)	0.314 (3)	0.1659 (13)	0.0199 (7)	0.25
H3′	1.1048	0.2898	0.1375	0.024*	0.2497 (18)
C4	0.7509 (2)	0.36777 (19)	0.26883 (13)	0.0172 (3)	
C5	0.6407 (2)	0.40831 (19)	0.36486 (13)	0.0176 (3)	
C7	0.9443 (2)	0.26314 (19)	0.62276 (14)	0.0176 (3)	
H7	0.8547	0.3020	0.6721	0.021*	
C8	1.1326 (2)	0.17935 (19)	0.67688 (13)	0.0167 (3)	
C9	1.1710 (2)	0.1525 (2)	0.79783 (13)	0.0190 (3)	
C10	1.3490 (3)	0.0862 (2)	0.86064 (14)	0.0225 (4)	
H10	1.3680	0.0733	0.9435	0.027*	
C11	1.4994 (3)	0.0391 (2)	0.79913 (15)	0.0229 (4)	
H11	1.6238	−0.0069	0.8401	0.027*	
C12	1.4694 (2)	0.0587 (2)	0.67816 (15)	0.0217 (3)	
H12	1.5714	0.0246	0.6356	0.026*	
C13	1.2902 (2)	0.1279 (2)	0.62060 (14)	0.0192 (3)	
H1	0.656 (3)	0.409 (3)	0.535 (2)	0.040 (6)*	

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0198 (4)	0.0239 (3)	0.0153 (3)	0.0069 (3)	0.0043 (2)	0.0045 (2)
S1′	0.0216 (14)	0.0257 (11)	0.0218 (11)	0.0042 (9)	0.0074 (10)	0.0018 (8)

F1	0.0271 (5)	0.0443 (6)	0.0201 (5)	0.0167 (5)	0.0120 (4)	0.0075 (4)
F2	0.0266 (5)	0.0396 (6)	0.0208 (5)	0.0157 (4)	0.0119 (4)	0.0080 (4)
O1	0.0185 (6)	0.0310 (6)	0.0209 (6)	0.0117 (5)	0.0054 (5)	0.0045 (5)
N1	0.0168 (6)	0.0247 (7)	0.0175 (6)	0.0095 (5)	0.0071 (5)	0.0026 (5)
N2	0.0135 (6)	0.0196 (7)	0.0208 (7)	0.0053 (5)	0.0043 (5)	0.0026 (5)
C1	0.0245 (12)	0.0234 (14)	0.0196 (10)	0.0062 (10)	0.0103 (8)	0.0032 (10)
C2	0.0207 (8)	0.0267 (10)	0.0157 (18)	0.0046 (7)	0.0111 (13)	0.0002 (14)
C3	0.0216 (14)	0.0257 (11)	0.0218 (11)	0.0042 (9)	0.0074 (10)	0.0018 (8)
C1′	0.0198 (4)	0.0239 (3)	0.0153 (3)	0.0069 (3)	0.0043 (2)	0.0045 (2)
C2′	0.0245 (12)	0.0234 (14)	0.0196 (10)	0.0062 (10)	0.0103 (8)	0.0032 (10)
C3′	0.0207 (8)	0.0267 (10)	0.0157 (18)	0.0046 (7)	0.0111 (13)	0.0002 (14)
C4	0.0167 (7)	0.0182 (7)	0.0172 (7)	0.0026 (6)	0.0050 (6)	0.0018 (6)
C5	0.0168 (7)	0.0174 (7)	0.0192 (7)	0.0029 (6)	0.0055 (6)	0.0009 (6)
C7	0.0157 (7)	0.0175 (7)	0.0210 (7)	0.0035 (6)	0.0071 (6)	0.0014 (6)
C8	0.0148 (7)	0.0153 (7)	0.0202 (8)	0.0031 (6)	0.0041 (6)	0.0016 (6)
C9	0.0187 (7)	0.0210 (8)	0.0194 (8)	0.0056 (6)	0.0076 (6)	0.0015 (6)
C10	0.0239 (8)	0.0248 (8)	0.0184 (8)	0.0057 (7)	0.0024 (6)	0.0047 (6)
C11	0.0165 (7)	0.0216 (8)	0.0298 (9)	0.0063 (6)	0.0018 (7)	0.0053 (7)
C12	0.0172 (7)	0.0217 (8)	0.0286 (8)	0.0060 (6)	0.0089 (6)	0.0033 (6)
C13	0.0197 (8)	0.0186 (7)	0.0205 (8)	0.0027 (6)	0.0069 (6)	0.0032 (6)

Geometric parameters (Å, º)

S1—C4	1.7269 (16)	C1′—C2′	1.40 (2)
S1—C1	1.727 (6)	C1′—H1′	0.9500
S1′—C3′	1.360 (16)	C2′—C3′	1.52 (3)
S1′—C4	1.725 (3)	C2′—H2′	0.9500
F1—C9	1.3624 (17)	C3′—H3′	0.9500
F2—C13	1.3500 (18)	C4—C5	1.474 (2)
O1—C5	1.2425 (18)	C7—C8	1.466 (2)
N1—C5	1.354 (2)	C7—H7	0.9500
N1—N2	1.3702 (18)	C8—C9	1.395 (2)
N1—H1	0.88 (2)	C8—C13	1.397 (2)
N2—C7	1.284 (2)	C9—C10	1.377 (2)
C1—C2	1.311 (8)	C10—C11	1.385 (2)
C1—H1A	0.9500	C10—H10	0.9500
C2—C3	1.519 (7)	C11—C12	1.386 (2)
C2—H2	0.9500	C11—H11	0.9500
C3—C4	1.374 (5)	C12—C13	1.374 (2)
C3—H3	0.9500	C12—H12	0.9500
C1′—C4	1.378 (10)		

C4—S1—C1	91.0 (2)	C5—C4—S1′	127.47 (18)
C3′—S1′—C4	88.3 (9)	C3—C4—S1	111.5 (3)
C5—N1—N2	123.04 (13)	C5—C4—S1	114.07 (11)
C5—N1—H1	118.9 (14)	O1—C5—N1	118.90 (13)
N2—N1—H1	117.9 (14)	O1—C5—C4	119.41 (14)
C7—N2—N1	113.74 (13)	N1—C5—C4	121.68 (14)

C2—C1—S1	116.3 (5)	N2—C7—C8	122.42 (14)
C2—C1—H1A	121.9	N2—C7—H7	118.8
S1—C1—H1A	121.9	C8—C7—H7	118.8
C1—C2—C3	109.0 (5)	C9—C8—C13	114.10 (14)
C1—C2—H2	125.5	C9—C8—C7	119.41 (14)
C3—C2—H2	125.5	C13—C8—C7	126.42 (14)
C4—C3—C2	112.2 (4)	F1—C9—C10	117.74 (14)
C4—C3—H3	123.9	F1—C9—C8	117.47 (14)
C2—C3—H3	123.9	C10—C9—C8	124.78 (14)
C4—C1′—C2′	115.3 (11)	C9—C10—C11	117.90 (15)
C4—C1′—H1′	122.3	C9—C10—H10	121.0
C2′—C1′—H1′	122.3	C11—C10—H10	121.0
C1′—C2′—C3′	96.1 (16)	C10—C11—C12	120.42 (15)
C1′—C2′—H2′	131.9	C10—C11—H11	119.8
C3′—C2′—H2′	131.9	C12—C11—H11	119.8
S1′—C3′—C2′	129.6 (16)	C13—C12—C11	119.10 (15)
S1′—C3′—H3′	115.2	C13—C12—H12	120.5
C2′—C3′—H3′	115.2	C11—C12—H12	120.5
C3—C4—C1′	103.4 (5)	F2—C13—C12	117.98 (14)
C3—C4—C5	134.2 (3)	F2—C13—C8	118.33 (14)
C1′—C4—C5	121.8 (4)	C12—C13—C8	123.67 (15)
C1′—C4—S1′	110.5 (5)		

C5—N1—N2—C7	179.73 (14)	C3—C4—C5—O1	167.6 (3)
C4—S1—C1—C2	−0.8 (5)	C1′—C4—C5—O1	−2.3 (6)
S1—C1—C2—C3	0.4 (7)	S1′—C4—C5—O1	172.3 (2)
C1—C2—C3—C4	0.4 (7)	S1—C4—C5—O1	−6.89 (19)
C4—C1′—C2′—C3′	−1.0 (16)	C3—C4—C5—N1	−11.6 (4)
C4—S1′—C3′—C2′	−4.0 (19)	C1′—C4—C5—N1	178.5 (5)
C1′—C2′—C3′—S1′	4 (2)	S1′—C4—C5—N1	−6.9 (3)
C2—C3—C4—C1′	−4.4 (7)	S1—C4—C5—N1	173.89 (12)
C2—C3—C4—C5	−175.6 (3)	N1—N2—C7—C8	−178.28 (13)
C2—C3—C4—S1′	155 (3)	N2—C7—C8—C9	−177.48 (14)
C2—C3—C4—S1	−1.0 (5)	N2—C7—C8—C13	5.7 (3)
C2′—C1′—C4—C3	1.9 (12)	C13—C8—C9—F1	−178.95 (13)
C2′—C1′—C4—C5	174.5 (10)	C7—C8—C9—F1	3.9 (2)
C2′—C1′—C4—S1′	−0.9 (13)	C13—C8—C9—C10	1.8 (2)
C2′—C1′—C4—S1	−157 (4)	C7—C8—C9—C10	−175.39 (14)
C3′—S1′—C4—C3	−19 (3)	F1—C9—C10—C11	179.29 (14)
C3′—S1′—C4—C1′	2.6 (10)	C8—C9—C10—C11	−1.4 (2)
C3′—S1′—C4—C5	−172.6 (9)	C9—C10—C11—C12	0.0 (2)
C3′—S1′—C4—S1	6.6 (9)	C10—C11—C12—C13	1.0 (2)
C1—S1—C4—C3	1.0 (3)	C11—C12—C13—F2	178.28 (14)
C1—S1—C4—C1′	23 (3)	C11—C12—C13—C8	−0.6 (2)
C1—S1—C4—C5	176.8 (2)	C9—C8—C13—F2	−179.60 (13)
C1—S1—C4—S1′	−2.4 (3)	C7—C8—C13—F2	−2.7 (2)
N2—N1—C5—O1	177.55 (13)	C9—C8—C13—C12	−0.7 (2)
N2—N1—C5—C4	−3.2 (2)	C7—C8—C13—C12	176.23 (15)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O1^{i}	0.88 (2)	1.97 (2)	2.856 (2)	174 (2)

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