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N′-(4-Cyanobenzylidene)thiophene-2-carbohydrazide

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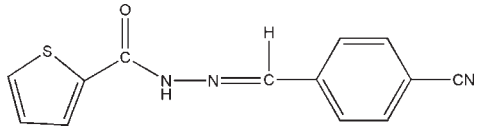
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.046; wR factor = 0.145; data-to-parameter ratio = 16.8.

The title compound, $C_{13}H_9N_3OS$, was prepared by the reaction of thiophene-2-carbohydrazide and 4-formylbenzonitrile. The dihedral angle between the benzene and thiophene rings is 11.9 (1)°. In the crystal structure, molecules are linked into centrosymmetric dimers by pairs of N—H···O hydrogen bonds.

Related literature

For related structures, see: Girgis (2006); Jiang (2010).



Experimental

Crystal data

$C_{13}H_9N_3OS$
M_r = 255.29
Monoclinic, $P2_1/n$
a = 6.3966 (13) Å
b = 16.340 (3) Å
c = 11.494 (2) Å
β = 90.66 (3)°
V = 1201.3 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.26 mm^{-1}
T = 293 K
0.21 × 0.20 × 0.18 mm

Data collection

Bruker SMART CCD diffractometer
11589 measured reflections
2746 independent reflections
1539 reflections with $I > 2\sigma(I)$
R_{int} = 0.066

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.145
S = 1.03
2746 reflections
163 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.32 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}	0.86	1.97	2.821 (2)	171

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

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH5046).

References

Bruker (1997). *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Girgis, A. S. (2006). *J. Chem. Res.* pp. 81–85.
Jiang, J.-H. (2010). *Acta Cryst.* E**66**, o922.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supporting information

Acta Cryst. (2010). E**66**, o1471 [https://doi.org/10.1107/S1600536810019215]

N′-(4-Cyanobenzylidene)thiophene-2-carbohydrazide

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S1. Comment

General background references for the title compound are included in the publication of a related structure (Jiang, 2010). As part of our search for new schiff base compounds we synthesized the title compound (I), and describe its structure here.

The molcular structure of (I) is shown in Fig. 1. The C8═N3 bond length of 1.272 (3)Å is comparable with reported values [1.281 (2) Å] (Girgis, 2006) and [1.273 (3)Å] (Jiang, 2010). In the crystal structure, molecules are linked by intermolecular N—H···O hydrogen bonds into centrosymmetric dimers.

S2. Experimental

A mixture of the thiophene-2-carbohydrazide (0.10 mol), and 4-formylbenzonitrile (0.10 mol) was stirred in refluxing ethanol (10 mL) for 4 h to afford the title compound (0.079 mol, yield 79%). Single crystals suitable for X-ray measurements were obtained by recrystallization from ethanol at room temperature.

S3. Refinement

H atoms were fixed geometrically and allowed to ride on their attached atoms, with C—H distances = 0.93-0.97 Å; N—H = 0.86Å and with U_{iso}(H) = 1.2U_{eq}(C,*N*) or 1.5U_{eq}(C_{methyl}).

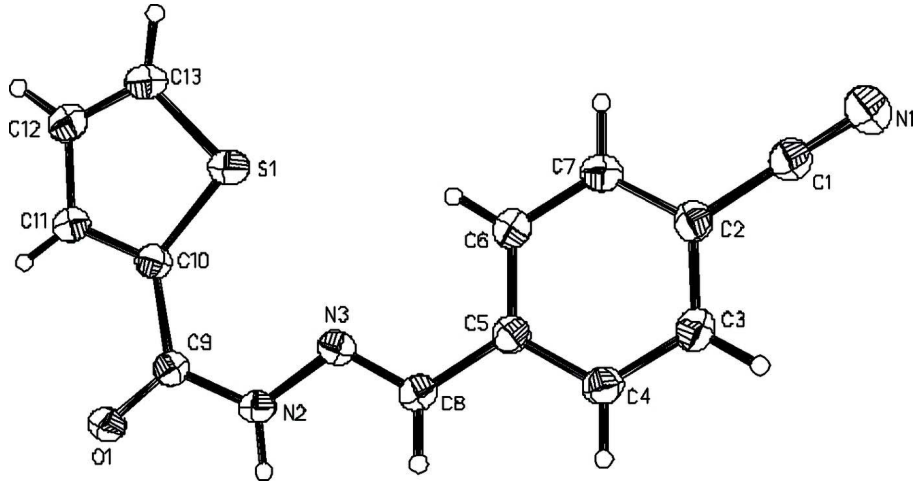

Figure 1

The structure of the title compound showing 30% probability displacement ellipsoids and the atom-numbering scheme.

N′-(4-Cyanobenzylidene)thiophene-2-carbohydrazide

Crystal data

$C_{13}H_9N_3OS$
$M_r = 255.29$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 6.3966$ (13) Å
$b = 16.340$ (3) Å
$c = 11.494$ (2) Å
$\beta = 90.66$ (3)°
$V = 1201.3$ (4) Å^3
$Z = 4$
$F(000) = 528$
$D_x = 1.412$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1539 reflections
$\theta = 3.8$–$26.2°$
$\mu = 0.26$ mm^{-1}
$T = 293$ K
Block, colorless
0.21 × 0.20 × 0.18 mm

Data collection

Bruker SMART CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
11589 measured reflections
2746 independent reflections
1539 reflections with $I > 2\sigma(I)$
$R_{int} = 0.066$
$\theta_{max} = 27.5°$, $\theta_{min} = 3.1°$
$h = -8 \rightarrow 8$
$k = -21 \rightarrow 21$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.046$
$wR(F^2) = 0.145$
$S = 1.03$
2746 reflections
163 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
$w = 1/[\sigma^2(F_o^2) + (0.0682P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.23$ e Å^{-3}
$\Delta\rho_{min} = -0.32$ e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
N3	0.2706 (3)	0.38738 (12)	0.84452 (15)	0.0428 (5)
N2	0.1294 (3)	0.44128 (12)	0.88894 (14)	0.0440 (5)
H2A	0.1499	0.4603	0.9579	0.053*
O1	−0.1694 (3)	0.51089 (11)	0.87605 (12)	0.0542 (5)
C2	0.8862 (4)	0.20156 (14)	0.79598 (18)	0.0444 (6)

C9	−0.0408 (4)	0.46559 (14)	0.82840 (17)	0.0421 (6)
C4	0.7596 (4)	0.29587 (15)	0.93785 (18)	0.0463 (6)
H4A	0.7759	0.3224	1.0089	0.056*
C8	0.4290 (4)	0.37040 (14)	0.90796 (18)	0.0430 (6)
H8A	0.4457	0.3960	0.9797	0.052*
C7	0.7082 (4)	0.21428 (16)	0.72907 (19)	0.0535 (7)
H7A	0.6895	0.1858	0.6597	0.064*
C5	0.5847 (4)	0.31123 (13)	0.86961 (17)	0.0408 (6)
C10	−0.0772 (4)	0.43914 (14)	0.70730 (17)	0.0431 (6)
C3	0.9102 (4)	0.24155 (15)	0.90156 (19)	0.0487 (6)
H3B	1.0278	0.2318	0.9479	0.058*
C13	−0.0822 (5)	0.38914 (16)	0.5070 (2)	0.0600 (8)
H13A	−0.0568	0.3664	0.4344	0.072*
C6	0.5585 (4)	0.26912 (15)	0.76500 (19)	0.0505 (6)
H6A	0.4397	0.2780	0.7193	0.061*
C11	−0.2581 (4)	0.45925 (15)	0.64795 (18)	0.0514 (7)
H11A	−0.3673	0.4888	0.6802	0.062*
C12	−0.2588 (5)	0.43005 (16)	0.5330 (2)	0.0595 (7)
H12A	−0.3688	0.4380	0.4806	0.071*
S1	0.08838 (11)	0.38350 (4)	0.62050 (5)	0.0558 (3)
N1	1.1789 (4)	0.10803 (16)	0.7206 (2)	0.0703 (7)
C1	1.0483 (4)	0.14800 (17)	0.7545 (2)	0.0516 (6)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N3	0.0461 (13)	0.0436 (12)	0.0388 (9)	0.0034 (10)	0.0026 (9)	−0.0032 (8)
N2	0.0477 (13)	0.0492 (12)	0.0351 (9)	0.0055 (10)	−0.0021 (9)	−0.0080 (8)
O1	0.0571 (12)	0.0630 (12)	0.0425 (8)	0.0186 (9)	−0.0023 (8)	−0.0126 (8)
C2	0.0460 (15)	0.0440 (14)	0.0432 (11)	0.0022 (11)	0.0034 (11)	0.0055 (10)
C9	0.0471 (15)	0.0414 (13)	0.0376 (11)	0.0018 (11)	−0.0021 (11)	−0.0001 (10)
C4	0.0514 (16)	0.0490 (14)	0.0384 (11)	−0.0003 (12)	−0.0013 (11)	−0.0017 (10)
C8	0.0459 (15)	0.0463 (14)	0.0369 (11)	−0.0016 (11)	−0.0012 (10)	0.0013 (10)
C7	0.0612 (18)	0.0539 (16)	0.0452 (12)	0.0049 (13)	−0.0047 (12)	−0.0094 (11)
C5	0.0435 (15)	0.0404 (13)	0.0385 (11)	−0.0002 (11)	0.0022 (10)	0.0036 (9)
C10	0.0507 (15)	0.0401 (13)	0.0386 (11)	0.0018 (11)	0.0006 (10)	−0.0024 (9)
C3	0.0464 (16)	0.0517 (15)	0.0478 (12)	0.0026 (12)	−0.0026 (11)	0.0065 (11)
C13	0.081 (2)	0.0600 (18)	0.0391 (12)	0.0101 (16)	−0.0040 (13)	−0.0079 (11)
C6	0.0496 (16)	0.0538 (16)	0.0478 (12)	0.0066 (13)	−0.0063 (11)	−0.0027 (11)
C11	0.0591 (18)	0.0528 (16)	0.0420 (12)	0.0071 (13)	−0.0077 (11)	−0.0061 (11)
C12	0.071 (2)	0.0596 (17)	0.0477 (13)	0.0082 (15)	−0.0166 (13)	−0.0056 (12)
S1	0.0620 (5)	0.0646 (5)	0.0407 (3)	0.0109 (3)	−0.0001 (3)	−0.0088 (3)
N1	0.0686 (18)	0.0765 (17)	0.0658 (14)	0.0177 (14)	0.0068 (13)	−0.0044 (12)
C1	0.0534 (17)	0.0544 (16)	0.0471 (13)	0.0029 (13)	0.0009 (12)	0.0027 (12)

Geometric parameters (Å, º)

N3—C8	1.272 (3)	C7—C6	1.378 (3)
N3—N2	1.365 (2)	C7—H7A	0.9300
N2—C9	1.345 (3)	C5—C6	1.394 (3)
N2—H2A	0.8600	C10—C11	1.376 (3)
O1—C9	1.239 (3)	C10—S1	1.723 (2)
C2—C7	1.382 (3)	C3—H3B	0.9300
C2—C3	1.385 (3)	C13—C12	1.349 (4)
C2—C1	1.442 (4)	C13—S1	1.693 (3)
C9—C10	1.474 (3)	C13—H13A	0.9300
C4—C3	1.378 (3)	C6—H6A	0.9300
C4—C5	1.382 (3)	C11—C12	1.405 (3)
C4—H4A	0.9300	C11—H11A	0.9300
C8—C5	1.460 (3)	C12—H12A	0.9300
C8—H8A	0.9300	N1—C1	1.133 (3)
C8—N3—N2	116.90 (18)	C6—C5—C8	120.8 (2)
C9—N2—N3	122.15 (18)	C11—C10—C9	121.4 (2)
C9—N2—H2A	118.9	C11—C10—S1	110.98 (16)
N3—N2—H2A	118.9	C9—C10—S1	127.61 (19)
C7—C2—C3	119.9 (2)	C4—C3—C2	119.9 (2)
C7—C2—C1	119.9 (2)	C4—C3—H3B	120.1
C3—C2—C1	120.2 (2)	C2—C3—H3B	120.1
O1—C9—N2	119.03 (19)	C12—C13—S1	112.96 (18)
O1—C9—C10	119.6 (2)	C12—C13—H13A	123.5
N2—C9—C10	121.3 (2)	S1—C13—H13A	123.5
C3—C4—C5	120.6 (2)	C7—C6—C5	120.1 (2)
C3—C4—H4A	119.7	C7—C6—H6A	119.9
C5—C4—H4A	119.7	C5—C6—H6A	119.9
N3—C8—C5	120.9 (2)	C10—C11—C12	112.2 (2)
N3—C8—H8A	119.6	C10—C11—H11A	123.9
C5—C8—H8A	119.6	C12—C11—H11A	123.9
C6—C7—C2	120.1 (2)	C13—C12—C11	112.5 (3)
C6—C7—H7A	119.9	C13—C12—H12A	123.7
C2—C7—H7A	119.9	C11—C12—H12A	123.7
C4—C5—C6	119.2 (2)	C13—S1—C10	91.29 (13)
C4—C5—C8	120.0 (2)	N1—C1—C2	177.8 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*A*···O1^{i}	0.86	1.97	2.821 (2)	171

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