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

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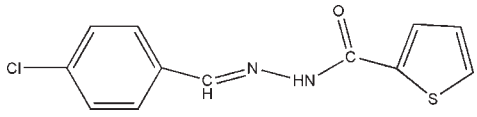
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.054; wR factor = 0.141; data-to-parameter ratio = 17.3.

In the title compound, $C_{12}H_9ClN_2OS$, the dihedral angle between the aromatic rings is 9.78 (11)°. In the crystal structure, inversion dimers linked by pairs of N—H···O hydrogen bonds occur, generating $R_2^2(8)$ loops. Weak aromatic π–π stacking [centroid–centroid separations = 3.7210 (15) and 3.8706 (15) Å] also occurs.

Related literature

For the isostructural bromo-compound and background information, see the preceding paper: Jiang (2010).



Experimental

Crystal data

$C_{12}H_9ClN_2OS$
M_r = 264.72
Monoclinic, $P2_1/n$
a = 6.0040 (12) Å
b = 16.831 (3) Å
c = 11.557 (2) Å
β = 94.38 (3)°
V = 1164.5 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.49 mm^{-1}
T = 293 K
0.21 × 0.19 × 0.18 mm

Data collection

Bruker SMART CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
T_{min} = 0.491, T_{max} = 0.728
11069 measured reflections
2660 independent reflections
1778 reflections with $I > 2\sigma(I)$
R_{int} = 0.115

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.141
S = 0.94
2660 reflections
154 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.52 e Å^{-3}
$\Delta\rho_{min}$ = −0.33 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	2.01	2.825 (2)	158

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

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*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5369).

References

Bruker (1997). *SMART*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
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**, o923 [doi:10.1107/S1600536810010615]

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

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S1. Comment

As part of our search for new Schiff base compounds (Jiang, 2010) we synthesized the title compound (I), and describe its structure here.

The molcular structure of (I) is shown in Fig. 1. In the crystal structure, molecules are linked by intermolecular N—H···O hydrogen bonds.

S2. Experimental

A mixture of thiophene-2-carbohydrazide (0.05 mol), and 4-chlorobenzaldehyde (0.05 mol) was stirred in refluxing ethanol (10 ml) for 4 h to afford the title compound (0.084 mol, yield 84%). Colourless blocks of (I) 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}).

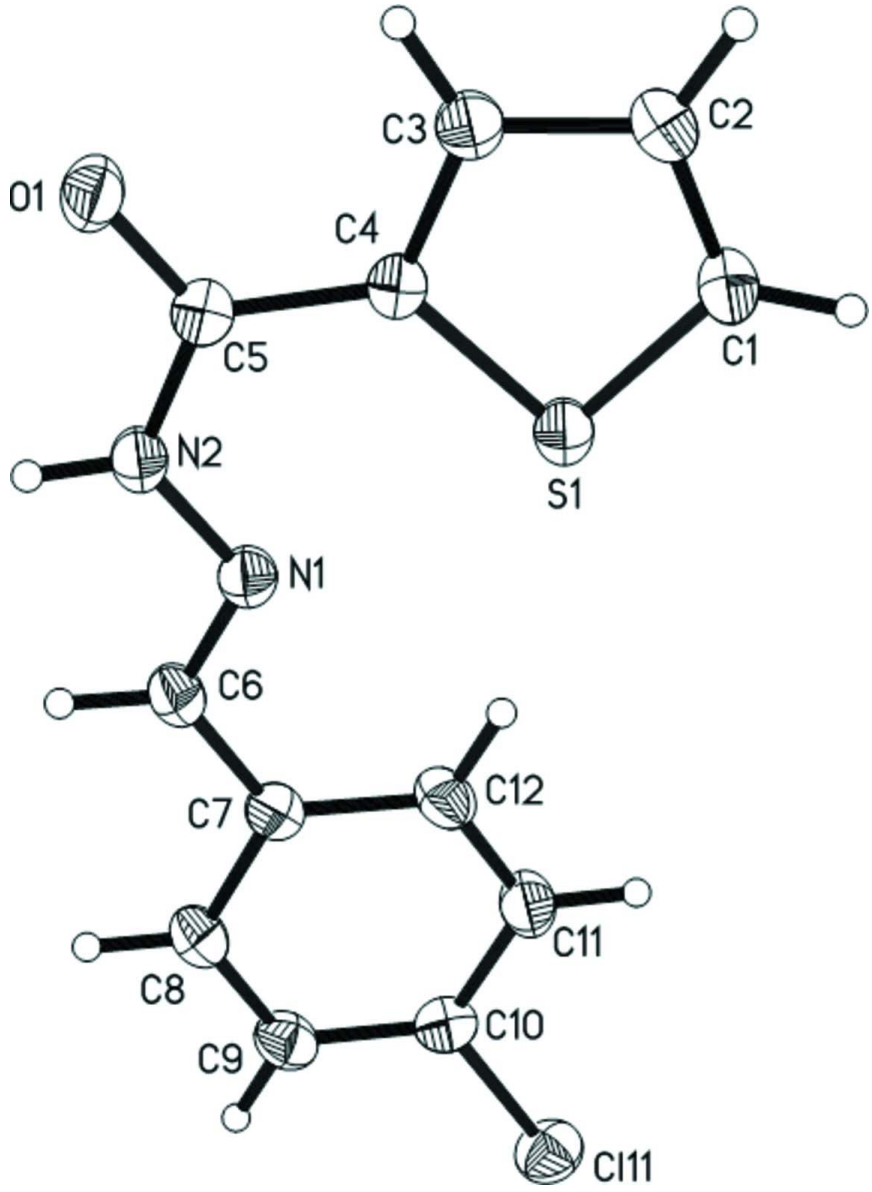

Figure 1

The molecular structure of (I) showing 30% probability displacement ellipsoids.

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

Crystal data

$C_{12}H_9ClN_2OS$
$M_r = 264.72$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 6.0040\ (12)$ Å
$b = 16.831\ (3)$ Å
$c = 11.557\ (2)$ Å
$\beta = 94.38\ (3)°$
$V = 1164.5\ (4)$ Å^3
$Z = 4$

$F(000) = 544$
$D_x = 1.510$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1778 reflections
$\theta = 3–27.5°$
$\mu = 0.49$ mm^{-1}
$T = 293$ K
Block, colorless
0.21 × 0.19 × 0.18 mm

Data collection

Bruker SMART CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)
T_{min} = 0.491, T_{max} = 0.728
11069 measured reflections
2660 independent reflections
1778 reflections with $I > 2\sigma(I)$
R_{int} = 0.115
θ_{max} = 27.5°, θ_{min} = 3.0°
$h = -7 \rightarrow 7$
$k = -21 \rightarrow 21$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.141
S = 0.94
2660 reflections
154 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
$w = 1/[\sigma^2(F_o^2) + (0.061P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.52 e Å^{-3}
$\Delta\rho_{min}$ = −0.33 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}
S1	0.13248 (10)	0.11796 (4)	0.87758 (5)	0.0454 (2)
Cl11	1.14308 (10)	0.37422 (4)	0.75642 (6)	0.0564 (2)
C6	0.4682 (4)	0.12975 (13)	0.5974 (2)	0.0415 (5)
H6A	0.4793	0.1052	0.5260	0.050*
N1	0.3130 (3)	0.10929 (11)	0.66045 (16)	0.0405 (5)
O1	−0.1503 (3)	−0.01594 (11)	0.62318 (14)	0.0543 (5)
N2	0.1660 (3)	0.05312 (11)	0.61567 (15)	0.0435 (5)
H2A	0.1869	0.0321	0.5495	0.052*
C4	−0.0422 (3)	0.05737 (13)	0.79080 (18)	0.0371 (5)
C10	0.9430 (3)	0.30429 (13)	0.7075 (2)	0.0419 (5)
C5	−0.0117 (3)	0.02976 (13)	0.67292 (19)	0.0409 (5)
C12	0.6114 (4)	0.23305 (15)	0.7390 (2)	0.0462 (6)
H12A	0.4937	0.2222	0.7844	0.055*
C3	−0.2281 (4)	0.03656 (14)	0.8471 (2)	0.0448 (5)
H3A	−0.3420	0.0043	0.8145	0.054*
C1	−0.0417 (4)	0.11356 (14)	0.9866 (2)	0.0474 (6)

H1A	−0.0129	0.1389	1.0577	0.057*
C7	0.6284 (3)	0.19098 (13)	0.63533 (18)	0.0385 (5)
C8	0.8067 (4)	0.20886 (14)	0.5689 (2)	0.0452 (6)
H8A	0.8200	0.1823	0.4992	0.054*
C11	0.7646 (4)	0.28965 (15)	0.7745 (2)	0.0499 (6)
H11A	0.7498	0.3180	0.8426	0.060*
C9	0.9633 (4)	0.26525 (15)	0.6049 (2)	0.0465 (6)
H9A	1.0814	0.2767	0.5601	0.056*
C2	−0.2269 (4)	0.06935 (15)	0.9593 (2)	0.0482 (6)
H2B	−0.3401	0.0616	1.0088	0.058*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0461 (4)	0.0519 (4)	0.0386 (4)	−0.0061 (3)	0.0064 (3)	−0.0075 (2)
Cl11	0.0523 (4)	0.0568 (5)	0.0598 (4)	−0.0115 (3)	0.0023 (3)	0.0021 (3)
C6	0.0449 (12)	0.0451 (14)	0.0355 (11)	0.0036 (10)	0.0087 (10)	−0.0003 (9)
N1	0.0414 (10)	0.0429 (11)	0.0375 (10)	−0.0030 (8)	0.0046 (8)	−0.0026 (8)
O1	0.0608 (10)	0.0597 (11)	0.0433 (9)	−0.0207 (9)	0.0103 (8)	−0.0127 (8)
N2	0.0453 (10)	0.0479 (12)	0.0381 (10)	−0.0055 (9)	0.0082 (8)	−0.0093 (9)
C4	0.0402 (10)	0.0361 (12)	0.0349 (11)	0.0006 (9)	0.0020 (9)	−0.0009 (9)
C10	0.0396 (11)	0.0387 (13)	0.0470 (13)	−0.0025 (9)	0.0017 (10)	0.0067 (10)
C5	0.0434 (11)	0.0403 (13)	0.0387 (12)	−0.0006 (10)	0.0022 (9)	−0.0013 (10)
C12	0.0448 (12)	0.0498 (15)	0.0458 (13)	−0.0024 (11)	0.0156 (10)	−0.0004 (11)
C3	0.0450 (12)	0.0451 (14)	0.0447 (13)	−0.0039 (10)	0.0056 (10)	−0.0021 (10)
C1	0.0563 (14)	0.0494 (15)	0.0373 (13)	−0.0001 (11)	0.0079 (11)	−0.0047 (10)
C7	0.0388 (11)	0.0399 (13)	0.0370 (11)	0.0033 (9)	0.0046 (9)	0.0054 (9)
C8	0.0467 (12)	0.0491 (14)	0.0413 (13)	0.0022 (11)	0.0127 (10)	0.0000 (10)
C11	0.0550 (13)	0.0530 (15)	0.0426 (13)	−0.0033 (12)	0.0102 (11)	−0.0072 (11)
C9	0.0430 (12)	0.0514 (15)	0.0465 (13)	−0.0026 (11)	0.0132 (10)	0.0059 (11)
C2	0.0498 (13)	0.0535 (15)	0.0426 (13)	0.0012 (11)	0.0129 (10)	−0.0008 (11)

Geometric parameters (Å, º)

S1—C1	1.699 (3)	C12—C11	1.365 (3)
S1—C4	1.728 (2)	C12—C7	1.402 (3)
Cl11—C10	1.745 (2)	C12—H12A	0.9300
C6—N1	1.273 (3)	C3—C2	1.408 (3)
C6—C7	1.454 (3)	C3—H3A	0.9300
C6—H6A	0.9300	C1—C2	1.355 (3)
N1—N2	1.368 (2)	C1—H1A	0.9300
O1—C5	1.242 (3)	C7—C8	1.397 (3)
N2—C5	1.356 (3)	C8—C9	1.378 (3)
N2—H2A	0.8600	C8—H8A	0.9300
C4—C3	1.379 (3)	C11—H11A	0.9300
C4—C5	1.464 (3)	C9—H9A	0.9300
C10—C9	1.369 (3)	C2—H2B	0.9300
C10—C11	1.392 (3)		

C1—S1—C4	91.38 (11)	C4—C3—H3A	123.6
N1—C6—C7	121.0 (2)	C2—C3—H3A	123.6
N1—C6—H6A	119.5	C2—C1—S1	113.05 (18)
C7—C6—H6A	119.5	C2—C1—H1A	123.5
C6—N1—N2	116.72 (19)	S1—C1—H1A	123.5
C5—N2—N1	121.65 (18)	C8—C7—C12	118.0 (2)
C5—N2—H2A	119.2	C8—C7—C6	120.3 (2)
N1—N2—H2A	119.2	C12—C7—C6	121.7 (2)
C3—C4—C5	121.4 (2)	C9—C8—C7	121.1 (2)
C3—C4—S1	110.67 (16)	C9—C8—H8A	119.5
C5—C4—S1	127.88 (17)	C7—C8—H8A	119.5
C9—C10—C11	121.4 (2)	C12—C11—C10	119.1 (2)
C9—C10—Cl11	120.03 (18)	C12—C11—H11A	120.5
C11—C10—Cl11	118.62 (18)	C10—C11—H11A	120.5
O1—C5—N2	118.6 (2)	C10—C9—C8	119.3 (2)
O1—C5—C4	120.0 (2)	C10—C9—H9A	120.4
N2—C5—C4	121.4 (2)	C8—C9—H9A	120.4
C11—C12—C7	121.2 (2)	C1—C2—C3	112.1 (2)
C11—C12—H12A	119.4	C1—C2—H2B	124.0
C7—C12—H12A	119.4	C3—C2—H2B	124.0
C4—C3—C2	112.8 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*A*···O1^{i}	0.86	2.01	2.825 (2)	158

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