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1-(4-Chlorobenzylidene)-4-ethylthiosemicarbazide

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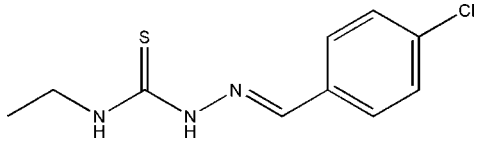
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.008 Å; R factor = 0.068; wR factor = 0.272; data-to-parameter ratio = 20.0.

In the title compound, $C_{10}H_{12}ClN_3S$, the dihedral angle between the benzene ring and the thiourea unit is 2.35 (19)°. In the crystal, inversion dimers linked by pairs of N—H···S hydrogen bonds generate $R_2^2(8)$ loops.

Related literature

For related structures, see: Li & Jian (2010); Li & Meng (2010).



Experimental

Crystal data

$C_{10}H_{12}ClN_3S$
M_r = 241.75
Monoclinic, $P2_1/c$
a = 4.6769 (10) Å
b = 26.727 (6) Å
c = 9.791 (3) Å
β = 102.59 (3)°
V = 1194.4 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.47 mm^{-1}
T = 293 K
0.22 × 0.20 × 0.18 mm

Data collection

Bruker SMART CCD diffractometer
11437 measured reflections
2723 independent reflections
1388 reflections with $I > 2\sigma(I)$
R_{int} = 0.066

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.068
$wR(F^2)$ = 0.272
S = 1.09
2723 reflections
136 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···S1^{i}	0.86	2.59	3.383 (4)	154

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

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*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5737).

References

Bruker (1997). *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Li, Y.-F. & Jian, F.-F. (2010). *Acta Cryst.* E**66**, o1399.
Li, Y.-F. & Meng, F.-Y. (2010). *Acta Cryst.* E**66**, o2685.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supporting information

Acta Cryst. (2010). E**66**, o3256 [https://doi.org/10.1107/S1600536810047446]

1-(4-Chlorobenzylidene)-4-ethylthiosemicarbazide

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S1. Experimental

A mixture of 4-ethylthiosemicarbazide (0.1 mol) and 4-chlorobenzaldehyde (0.1 mol) was stirred in refluxing ethanol (20 mL) for 2 h to afford the title compound (0.089 mol, yield 89%). Colourless bars were obtained by recrystallization from ethanol at room temperature.

S2. Refinement

H atoms were fixed geometrically and allowed to ride on their attached atoms, with C—H distances=0.97 Å, and with U_{iso}=1.2–1.5U_{eq}.

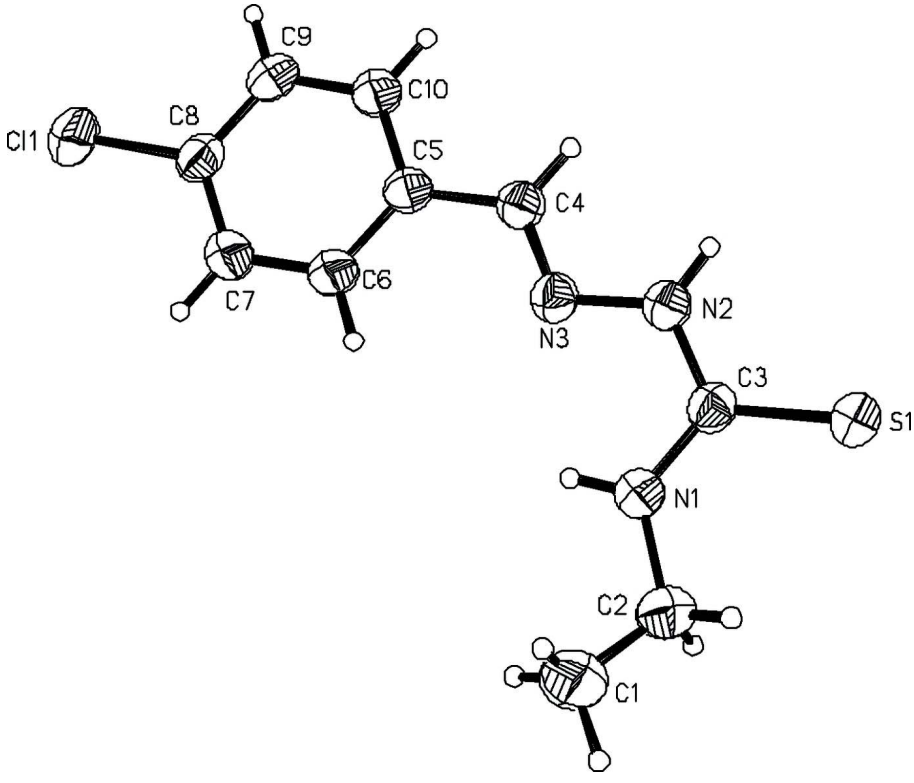

Figure 1

The structure of the title compound showing 30% probability displacement ellipsoids.

1-(4-Chlorobenzylidene)-4-ethylthiosemicarbazide

Crystal data

$C_{10}H_{12}ClN_3S$
$M_r = 241.75$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 4.6769$ (10) Å
$b = 26.727$ (6) Å
$c = 9.791$ (3) Å
$\beta = 102.59$ (3)°
$V = 1194.4$ (5) Å^3
$Z = 4$
$F(000) = 504$
$D_x = 1.344$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2723 reflections
$\theta = 3.1$–27.5°
$\mu = 0.47$ mm^{-1}
$T = 293$ K
Bar, colorless
0.22 × 0.20 × 0.18 mm

Data collection

Bruker SMART CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
phi and ω scans
11437 measured reflections
2723 independent reflections
1388 reflections with $I > 2\sigma(I)$
$R_{int} = 0.066$
$\theta_{max} = 27.5°$, $\theta_{min} = 3.1°$
$h = -6 \rightarrow 5$
$k = -34 \rightarrow 34$
$l = -12 \rightarrow 12$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.068$
$wR(F^2) = 0.272$
$S = 1.09$
2723 reflections
136 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
$w = 1/[\sigma^2(F_o^2) + (0.1156P)^2 + 1.1747P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.33$ e Å^{-3}
$\Delta\rho_{min} = -0.34$ e Å^{-3}

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 $F^2 > \sigma(F^2)$ 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	1.2104 (3)	−0.01352 (5)	0.72223 (14)	0.0738 (5)
Cl1	−0.3450 (4)	0.27875 (6)	0.46099 (18)	0.1022 (6)
N2	0.7928 (8)	0.04912 (15)	0.6097 (4)	0.0637 (10)
H2A	0.7695	0.0311	0.5354	0.076*
N3	0.6207 (8)	0.09071 (14)	0.6128 (4)	0.0616 (10)

C5	0.2251 (9)	0.14199 (17)	0.4970 (5)	0.0576 (11)
C4	0.4166 (10)	0.09865 (18)	0.5041 (5)	0.0622 (11)
H4A	0.3907	0.0765	0.4291	0.075*
N1	1.0195 (10)	0.06662 (17)	0.8346 (4)	0.0789 (13)
H1A	0.8985	0.0912	0.8260	0.095*
C10	0.0157 (10)	0.15053 (19)	0.3771 (5)	0.0664 (12)
H10A	−0.0066	0.1281	0.3028	0.080*
C6	0.2528 (11)	0.1751 (2)	0.6072 (5)	0.0723 (13)
H6A	0.3933	0.1695	0.6889	0.087*
C9	−0.1627 (11)	0.1925 (2)	0.3665 (5)	0.0709 (13)
H9A	−0.3045	0.1983	0.2853	0.085*
C3	0.9998 (10)	0.03689 (17)	0.7250 (5)	0.0602 (11)
C8	−0.1287 (11)	0.22552 (19)	0.4764 (5)	0.0679 (12)
C7	0.0731 (14)	0.2164 (2)	0.5967 (6)	0.0836 (16)
H7A	0.0898	0.2382	0.6721	0.100*
C2	1.2242 (16)	0.0617 (2)	0.9669 (6)	0.096 (2)
H2B	1.1446	0.0383	1.0247	0.116*
H2C	1.4045	0.0475	0.9506	0.116*
C1	1.290 (2)	0.1061 (3)	1.0404 (9)	0.130 (3)
H1B	1.4287	0.0997	1.1264	0.196*
H1C	1.1142	0.1199	1.0606	0.196*
H1D	1.3723	0.1294	0.9851	0.196*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0781 (9)	0.0700 (8)	0.0700 (8)	0.0180 (6)	0.0089 (6)	0.0022 (6)
Cl1	0.1142 (13)	0.0852 (11)	0.1023 (12)	0.0396 (9)	0.0129 (9)	0.0139 (8)
N2	0.060 (2)	0.069 (2)	0.061 (2)	0.0125 (19)	0.0087 (17)	−0.0022 (18)
N3	0.060 (2)	0.062 (2)	0.063 (2)	0.0073 (19)	0.0145 (18)	0.0012 (17)
C5	0.054 (2)	0.062 (3)	0.055 (2)	−0.002 (2)	0.0104 (19)	0.0026 (19)
C4	0.057 (3)	0.069 (3)	0.059 (3)	0.008 (2)	0.010 (2)	−0.001 (2)
N1	0.087 (3)	0.079 (3)	0.063 (3)	0.025 (2)	0.000 (2)	−0.008 (2)
C10	0.063 (3)	0.070 (3)	0.062 (3)	0.002 (2)	0.006 (2)	−0.001 (2)
C6	0.072 (3)	0.077 (3)	0.060 (3)	0.014 (3)	−0.003 (2)	−0.004 (2)
C9	0.066 (3)	0.077 (3)	0.064 (3)	0.009 (3)	0.001 (2)	0.013 (2)
C3	0.057 (2)	0.063 (3)	0.062 (3)	0.006 (2)	0.013 (2)	0.005 (2)
C8	0.068 (3)	0.067 (3)	0.067 (3)	0.011 (2)	0.011 (2)	0.012 (2)
C7	0.098 (4)	0.079 (4)	0.068 (3)	0.025 (3)	0.005 (3)	−0.011 (3)
C2	0.111 (5)	0.085 (4)	0.077 (4)	0.012 (4)	−0.014 (3)	−0.005 (3)
C1	0.153 (7)	0.094 (5)	0.112 (6)	−0.002 (5)	−0.039 (5)	−0.022 (4)

Geometric parameters (Å, º)

S1—C3	1.673 (5)	C10—H10A	0.9300
Cl1—C8	1.733 (5)	C6—C7	1.379 (7)
N2—C3	1.357 (6)	C6—H6A	0.9300
N2—N3	1.377 (5)	C9—C8	1.373 (7)

N2—H2A	0.8600	C9—H9A	0.9300
N3—C4	1.283 (6)	C8—C7	1.361 (7)
C5—C10	1.374 (6)	C7—H7A	0.9300
C5—C6	1.379 (7)	C2—C1	1.386 (9)
C5—C4	1.457 (6)	C2—H2B	0.9700
C4—H4A	0.9300	C2—H2C	0.9700
N1—C3	1.322 (6)	C1—H1B	0.9600
N1—C2	1.439 (7)	C1—H1C	0.9600
N1—H1A	0.8600	C1—H1D	0.9600
C10—C9	1.389 (7)		

C3—N2—N3	119.4 (4)	N1—C3—N2	116.2 (4)
C3—N2—H2A	120.3	N1—C3—S1	124.1 (4)
N3—N2—H2A	120.3	N2—C3—S1	119.7 (4)
C4—N3—N2	116.6 (4)	C7—C8—C9	120.3 (5)
C10—C5—C6	119.3 (4)	C7—C8—Cl1	120.2 (4)
C10—C5—C4	119.3 (4)	C9—C8—Cl1	119.5 (4)
C6—C5—C4	121.4 (4)	C8—C7—C6	120.2 (5)
N3—C4—C5	120.6 (4)	C8—C7—H7A	119.9
N3—C4—H4A	119.7	C6—C7—H7A	119.9
C5—C4—H4A	119.7	C1—C2—N1	114.7 (6)
C3—N1—C2	126.2 (5)	C1—C2—H2B	108.6
C3—N1—H1A	116.9	N1—C2—H2B	108.6
C2—N1—H1A	116.9	C1—C2—H2C	108.6
C5—C10—C9	120.2 (5)	N1—C2—H2C	108.6
C5—C10—H10A	119.9	H2B—C2—H2C	107.6
C9—C10—H10A	119.9	C2—C1—H1B	109.5
C5—C6—C7	120.3 (5)	C2—C1—H1C	109.5
C5—C6—H6A	119.9	H1B—C1—H1C	109.5
C7—C6—H6A	119.9	C2—C1—H1D	109.5
C8—C9—C10	119.7 (4)	H1B—C1—H1D	109.5
C8—C9—H9A	120.2	H1C—C1—H1D	109.5
C10—C9—H9A	120.2		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*A*···S1^{i}	0.86	2.59	3.383 (4)	154

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