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1-(4-Chlorobenzoyl)-3-cyclohexyl-3-methylthiourea

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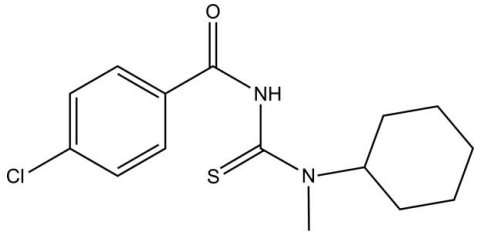
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.007 Å; R factor = 0.085; wR factor = 0.192; data-to-parameter ratio = 17.3.

In the title compound, $C_{15}H_{19}ClN_2OS$, the dihedral angle between the amide and thiourea fragments is 58.07 (17)°. The cyclohexane group adopts a chair conformation and is twisted relative to the thiourea fragment, forming a dihedral angle of 87.32 (18)°. In the crystal, N—H···S hydrogen bond links the molecules into chains running parallel to the a-axis direction.

Related literature

For related structures and background references, see: Al-abbasi & Kassim (2011); Nasir *et al.* (2011). For further synthetic details, see: Hassan *et al.* (2008).



Experimental

Crystal data

$C_{15}H_{19}ClN_2OS$
M_r = 310.83
Triclinic, $P\overline{1}$
a = 5.042 (2) Å
b = 11.368 (4) Å
c = 15.139 (6) Å
α = 69.865 (7)°
β = 82.698 (8)°
γ = 80.702 (8)°
V = 801.7 (5) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.37 mm^{-1}
T = 298 K
0.52 × 0.23 × 0.03 mm

Data collection

Bruker SMART APEX CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.906, T_{max} = 0.989
9192 measured reflections
3149 independent reflections
1935 reflections with $I > 2\sigma(I)$
R_{int} = 0.063

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.085
$wR(F^2)$ = 0.192
S = 1.10
3149 reflections
182 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.37 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···S1^{i}	0.86	2.73	3.411 (4)	137

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

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); 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*, *PARST* (Nardelli, 1995) and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5920).

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supporting information

Acta Cryst. (2011). E**67**, o1891 [doi:10.1107/S1600536811025013]

1-(4-Chlorobenzoyl)-3-cyclohexyl-3-methylthiourea

Aisha A. Al-abbasi, Bohari M. Yamin and Mohammad B. Kassim

S1. Comment

The title compound, (I), is a thiourea derivative analogous to our previously reported compounds (Al-abbasi & Kassim, 2011; Nasir *et al.*, 2011). The thiono S and the carbonyl O adopt a gauche conformation at a partially double N1—C8 bond with C7—N1—C8—S1 torsion angle of -124.4 (3)°. The dihedral angle between the mean planes of the thiourea (S1/N1/N2/C8) and the amide group (O1/N1/C1/C7/C8) is 58.07 (17)°. The cyclohexane has a chair corformation and the mean planes of (C9/C10/C11/C12/C13/C14) and the 4-chlorobenzoyl (Cl1/C1/C2/C3/C4/C5/C6/C7) fragments make an angle of 26.8 (2)°.

In the crystal, intermolecular N1—H···S1 hydrogen bond links the molecules into a one dimentional polymeric structure parallel to the *a*-axis.

S2. Experimental

The title compound was prepared according to a previously reported compound (Hassan *et al.*, 2008). Colourless plates of (I) were obtained by a slow evaporation of ethanolic solution at room temperature (yield 80%).

S3. Refinement

All H atoms were postioned geometrically with C—H bond lengths in the range 0.93 - 0.97 Å and N—H bond of 0.86 Å,.and refined in the riding model approximation with U_{iso}(H)=1.2U_{eq}(C,*N*), except for methyl group where U_{iso}(H)= 1.5U_{eq}(C).

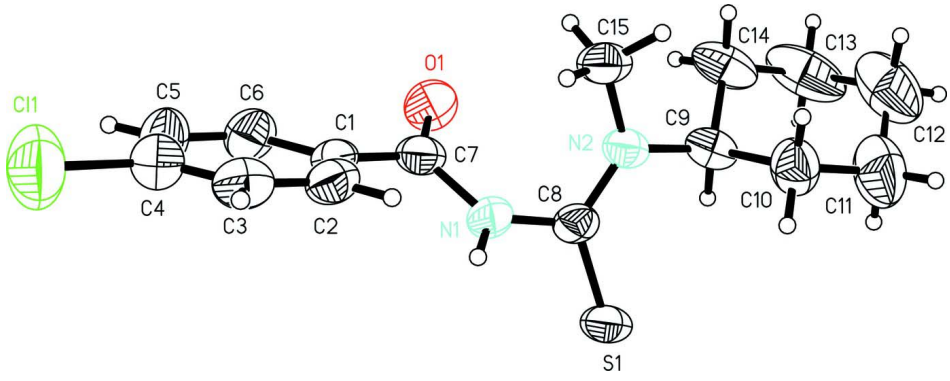

Figure 1

The molecular structure of the title compound with displacement ellipsoids drawn at the 50% probability level.

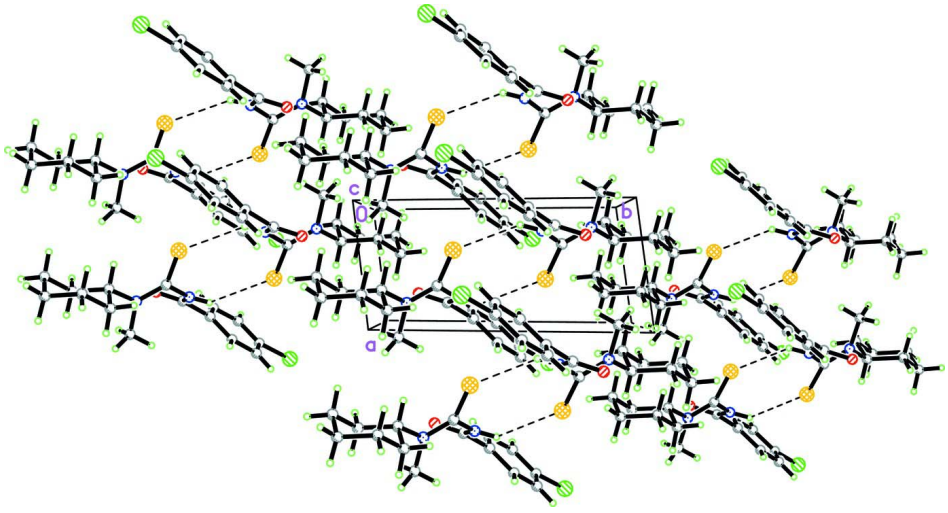

Figure 2

A packing diagram of the title compound down the *c*-axis showing the intermolecular hydrogen bonds N—H···S (*-x+1, -y + 1, -z*).

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Crystal data

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Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 5.042$ (2) Å
$b = 11.368$ (4) Å
$c = 15.139$ (6) Å
$\alpha = 69.865$ (7)°
$\beta = 82.698$ (8)°
$\gamma = 80.702$ (8)°
$V = 801.7$ (5) Å^3
$Z = 2$
$F(000) = 328$
$D_x = 1.288$ Mg m^{-3}
Melting point = 418–420 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1114 reflections
$\theta = 1.9$–26.0°
$\mu = 0.37$ mm^{-1}
$T = 298$ K
Plate, colourless
0.52 × 0.23 × 0.03 mm

Data collection

Bruker SMART APEX CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scan
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
$T_{min} = 0.906$, $T_{max} = 0.989$
9192 measured reflections
3149 independent reflections
1935 reflections with $I > 2\sigma(I)$
$R_{int} = 0.063$
$\theta_{max} = 26.0°$, $\theta_{min} = 1.9°$
$h = -6 \rightarrow 6$
$k = -14 \rightarrow 14$
$l = -18 \rightarrow 18$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.085$
$wR(F^2) = 0.192$
$S = 1.10$
3149 reflections
182 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
$w = 1/[\sigma^2(F_o^2) + (0.0826P)^2 + 0.0972P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.37$ e Å^{-3}
$\Delta\rho_{min} = -0.21$ 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	0.3915 (2)	0.34433 (9)	0.12184 (8)	0.0545 (4)
Cl1	1.2888 (4)	0.63599 (15)	−0.45045 (10)	0.1074 (6)
N1	0.7565 (6)	0.3373 (3)	−0.0182 (2)	0.0470 (8)
H1	0.7996	0.4111	−0.0278	0.056*
N2	0.7860 (6)	0.1587 (3)	0.1141 (2)	0.0436 (8)
O1	0.7203 (6)	0.1941 (3)	−0.0894 (2)	0.0647 (9)
C1	0.9200 (8)	0.3791 (3)	−0.1813 (3)	0.0453 (10)
C8	0.6580 (8)	0.2720 (3)	0.0727 (3)	0.0434 (10)
C7	0.7916 (8)	0.2943 (4)	−0.0951 (3)	0.0496 (10)
C2	1.1049 (8)	0.4556 (4)	−0.1799 (3)	0.0511 (10)
H2	1.1513	0.4549	−0.1221	0.061*
C9	0.6886 (8)	0.0807 (3)	0.2086 (3)	0.0500 (10)
H9	0.4977	0.1115	0.2179	0.060*
C3	1.2217 (9)	0.5326 (4)	−0.2615 (3)	0.0593 (12)
H3	1.3486	0.5823	−0.2593	0.071*
C10	0.8322 (9)	0.0976 (4)	0.2850 (3)	0.0642 (12)
H10A	0.8101	0.1863	0.2789	0.077*
H10B	1.0233	0.0699	0.2772	0.077*
C6	0.8558 (9)	0.3803 (4)	−0.2693 (3)	0.0614 (12)
H6	0.7376	0.3271	−0.2722	0.074*
C15	1.0483 (8)	0.1113 (4)	0.0754 (3)	0.0561 (11)
H15A	1.0200	0.0601	0.0393	0.084*
H15B	1.1572	0.0614	0.1262	0.084*
H15C	1.1382	0.1813	0.0354	0.084*
C4	1.1485 (10)	0.5351 (4)	−0.3466 (3)	0.0668 (13)
C5	0.9673 (10)	0.4596 (5)	−0.3505 (3)	0.0718 (14)
H5	0.9201	0.4623	−0.4086	0.086*
C14	0.7051 (9)	−0.0591 (4)	0.2195 (4)	0.0708 (14)
H14A	0.6045	−0.0679	0.1721	0.085*
H14B	0.8915	−0.0930	0.2098	0.085*
C12	0.7376 (12)	−0.1166 (6)	0.3931 (4)	0.108 (2)
H12A	0.6573	−0.1624	0.4547	0.130*

H12B	0.9254	−0.1518	0.3886	0.130*
C13	0.5919 (11)	−0.1324 (5)	0.3164 (5)	0.100 (2)
H13A	0.6101	−0.2212	0.3229	0.120*
H13B	0.4015	−0.1031	0.3240	0.120*
C11	0.7191 (12)	0.0218 (6)	0.3827 (4)	0.0969 (18)
H11A	0.8191	0.0306	0.4303	0.116*
H11B	0.5322	0.0549	0.3927	0.116*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0603 (7)	0.0371 (6)	0.0621 (7)	0.0054 (5)	−0.0001 (5)	−0.0182 (5)
Cl1	0.1343 (14)	0.1065 (12)	0.0655 (9)	−0.0256 (10)	0.0032 (9)	−0.0077 (8)
N1	0.065 (2)	0.0364 (17)	0.045 (2)	−0.0057 (15)	−0.0051 (17)	−0.0215 (15)
N2	0.0444 (19)	0.0371 (17)	0.051 (2)	0.0010 (14)	−0.0094 (16)	−0.0180 (15)
O1	0.090 (2)	0.0534 (18)	0.065 (2)	−0.0119 (16)	−0.0157 (17)	−0.0333 (16)
C1	0.049 (2)	0.043 (2)	0.046 (2)	0.0083 (18)	−0.0065 (19)	−0.0231 (19)
C8	0.051 (2)	0.038 (2)	0.050 (2)	−0.0032 (18)	−0.010 (2)	−0.0238 (19)
C7	0.053 (3)	0.043 (2)	0.057 (3)	0.0087 (19)	−0.016 (2)	−0.025 (2)
C2	0.051 (3)	0.054 (2)	0.055 (3)	0.001 (2)	−0.006 (2)	−0.030 (2)
C9	0.042 (2)	0.039 (2)	0.067 (3)	−0.0003 (17)	−0.009 (2)	−0.016 (2)
C3	0.057 (3)	0.056 (3)	0.070 (3)	−0.003 (2)	−0.006 (2)	−0.029 (2)
C10	0.078 (3)	0.057 (3)	0.054 (3)	−0.015 (2)	−0.007 (2)	−0.010 (2)
C6	0.074 (3)	0.066 (3)	0.055 (3)	−0.004 (2)	−0.015 (2)	−0.033 (2)
C15	0.050 (3)	0.056 (2)	0.069 (3)	0.011 (2)	−0.014 (2)	−0.033 (2)
C4	0.073 (3)	0.064 (3)	0.056 (3)	0.002 (3)	−0.003 (3)	−0.017 (2)
C5	0.085 (4)	0.087 (4)	0.047 (3)	0.002 (3)	−0.018 (3)	−0.028 (3)
C14	0.059 (3)	0.037 (2)	0.112 (4)	−0.002 (2)	−0.019 (3)	−0.016 (3)
C12	0.080 (4)	0.093 (5)	0.103 (5)	−0.011 (3)	0.005 (4)	0.024 (4)
C13	0.062 (3)	0.045 (3)	0.163 (6)	−0.013 (2)	−0.003 (4)	0.003 (3)
C11	0.106 (5)	0.104 (5)	0.061 (3)	−0.018 (4)	−0.004 (3)	0.000 (3)

Geometric parameters (Å, º)

S1—C8	1.687 (4)	C10—H10A	0.9700
Cl1—C4	1.739 (5)	C10—H10B	0.9700
N1—C8	1.391 (5)	C6—C5	1.365 (6)
N1—C7	1.391 (5)	C6—H6	0.9300
N1—H1	0.8600	C15—H15A	0.9600
N2—C8	1.321 (4)	C15—H15B	0.9600
N2—C9	1.470 (5)	C15—H15C	0.9600
N2—C15	1.474 (5)	C4—C5	1.370 (6)
O1—C7	1.221 (4)	C5—H5	0.9300
C1—C2	1.381 (5)	C14—C13	1.507 (7)
C1—C6	1.406 (5)	C14—H14A	0.9700
C1—C7	1.474 (5)	C14—H14B	0.9700
C2—C3	1.370 (6)	C12—C11	1.515 (8)
C2—H2	0.9300	C12—C13	1.524 (8)

C9—C10	1.520 (6)	C12—H12A	0.9700
C9—C14	1.530 (5)	C12—H12B	0.9700
C9—H9	0.9800	C13—H13A	0.9700
C3—C4	1.375 (6)	C13—H13B	0.9700
C3—H3	0.9300	C11—H11A	0.9700
C10—C11	1.524 (6)	C11—H11B	0.9700

C8—N1—C7	126.1 (3)	N2—C15—H15A	109.5
C8—N1—H1	117.0	N2—C15—H15B	109.5
C7—N1—H1	117.0	H15A—C15—H15B	109.5
C8—N2—C9	120.4 (3)	N2—C15—H15C	109.5
C8—N2—C15	122.6 (3)	H15A—C15—H15C	109.5
C9—N2—C15	116.5 (3)	H15B—C15—H15C	109.5
C2—C1—C6	118.3 (4)	C5—C4—C3	120.9 (4)
C2—C1—C7	123.2 (4)	C5—C4—Cl1	119.9 (4)
C6—C1—C7	118.5 (4)	C3—C4—Cl1	119.2 (4)
N2—C8—N1	116.8 (3)	C6—C5—C4	120.3 (4)
N2—C8—S1	125.5 (3)	C6—C5—H5	119.9
N1—C8—S1	117.8 (3)	C4—C5—H5	119.9
O1—C7—N1	121.5 (4)	C13—C14—C9	110.6 (4)
O1—C7—C1	124.2 (4)	C13—C14—H14A	109.5
N1—C7—C1	114.4 (3)	C9—C14—H14A	109.5
C3—C2—C1	121.6 (4)	C13—C14—H14B	109.5
C3—C2—H2	119.2	C9—C14—H14B	109.5
C1—C2—H2	119.2	H14A—C14—H14B	108.1
N2—C9—C10	111.3 (3)	C11—C12—C13	110.4 (5)
N2—C9—C14	113.4 (4)	C11—C12—H12A	109.6
C10—C9—C14	110.9 (4)	C13—C12—H12A	109.6
N2—C9—H9	107.0	C11—C12—H12B	109.6
C10—C9—H9	107.0	C13—C12—H12B	109.6
C14—C9—H9	107.0	H12A—C12—H12B	108.1
C2—C3—C4	119.0 (4)	C14—C13—C12	111.1 (4)
C2—C3—H3	120.5	C14—C13—H13A	109.4
C4—C3—H3	120.5	C12—C13—H13A	109.4
C9—C10—C11	110.8 (4)	C14—C13—H13B	109.4
C9—C10—H10A	109.5	C12—C13—H13B	109.4
C11—C10—H10A	109.5	H13A—C13—H13B	108.0
C9—C10—H10B	109.5	C12—C11—C10	111.0 (5)
C11—C10—H10B	109.5	C12—C11—H11A	109.4
H10A—C10—H10B	108.1	C10—C11—H11A	109.4
C5—C6—C1	120.0 (4)	C12—C11—H11B	109.4
C5—C6—H6	120.0	C10—C11—H11B	109.4
C1—C6—H6	120.0	H11A—C11—H11B	108.0

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*

N1—H1···S1^{i}	0.86	2.73	3.411 (4)	137

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