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1-(2-Chlorobenzoyl)-3-(2,3-dimethylphenyl)thiourea

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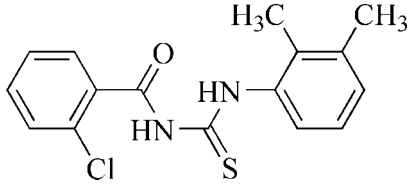
Key indicators: single-crystal X-ray study; T = 123 K; mean σ(C–C) = 0.002 Å; R factor = 0.036; wR factor = 0.090; data-to-parameter ratio = 18.3.

The dihedral angle between the two phenyl groups in the title compound, $C_{16}H_{15}ClN_2OS$, is 14.88 (4)°. An intramolecular N—H···O hydrogen bond occurs. In the crystal, pairs of N—H···S hydrogen bonds link the molecules into centrosymmetric dimers.

Related literature

For background and a related structure, see: Rauf *et al.* (2012). For a description of the Cambridge Structural Database, see: Allen *et al.* (2002).



Experimental

Crystal data

$C_{16}H_{15}ClN_2OS$
M_r = 318.81
Triclinic, $P\overline{1}$
a = 7.489 (3) Å
b = 9.338 (4) Å
c = 13.274 (5) Å
α = 65.674 (13)°
β = 69.975 (16)°
γ = 73.639 (17)°
V = 783.9 (5) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.38 mm^{-1}
T = 123 K
0.50 × 0.35 × 0.28 mm

Data collection

Rigaku/MSC Mercury CCD diffractometer
6180 measured reflections
3506 independent reflections
3373 reflections with $I > 2\sigma(I)$
R_{int} = 0.062

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.090
S = 1.08
3506 reflections
192 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 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}	0.88	2.46	3.3104 (15)	164
N2—H2···O1	0.88	2.00	2.6866 (17)	134

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

Data collection: *CrystalClear* (Molecular Structure Corporation & Rigaku, 2001); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *Yadokari-XG* (Wakita, 2001; Kabuto *et al.*, 2009).

MR is grateful to The Quaid-i-Azam University, Islamabad, for financial support for a postdoctoral fellowship.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG5274).

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supporting information

Acta Cryst. (2012). E**68**, o3483 [doi:10.1107/S1600536812048209]

1-(2-Chlorobenzoyl)-3-(2,3-dimethylphenyl)thiourea

M. Khawar Rauf, Masahiro Ebihara and Amin Badshah

S1. Comment

The background to this study has been set out in our previous work for the structural chemistry of *N*,*N*′-disubstituted thiourea (Rauf *et al.*, 2012) and their coordination chemistry. Herein, as a continuation of these crytallographic studies, the structure of the title compound (I) is described, Fig. 1. Compared to *N*-benzoyl-*N*′-phenylthioureas [Cambridge Structural Database (*Mogul* Version 1.7; Allen, 2002], the methyl substitutions at C(10) and C(11)on phenyl ring, implies no significant effect on these bond lengths. and show the molecule to exist in the thione form with typical thiourea C—S and C—O bonds, as well as shortened C—N bond lengths. The dihedral angles to the O(1) C(1) N(1) C(2) N(2)S(1) plane are 79.89 (2)° for the ring formed by C(3) to C(8) and 65.32 (2)° for the ring formed by C(9) to C(14). An intramolecular N—H···O H–bond is present (Table 1), forming a six-membered ring commonly observed in this class of compounds (Rauf *et al.*, 2012). In the crystal packing of (I), intermolecular N—H···S H–bonds link the molecules into centrosymmetric dimers (Fig.2).

S2. Experimental

Freshly prepared 2-chlorobenzoylisothiocyanate (1.98 g, 10 mmol) was dissolved in acetone (30 ml) and stirred for 30 minutes. Afterwards neat 2,3-dimethylaniline(1.21 g, 10 mmol) was added and the resulting mixture was stirred for 2 h. The reaction mixture was then poured into acidified water and stirred well. The solid product was separated and washed with deionized water and purified by recrystallization from methanol/ 1,1-dichloromethane (1:1 *v/v*) to give fine crystals of the title compound (I), with an overall yield of 95% (3.02 g). M.P; 180–181°C Anal. calcd. for C_{16} H_{15}Cl N_2 O S; C, 60.28 H, 4.74 N, 8.79 S, 10.06 Found: C, 60.22 H, 4.73 N, 8.78 S, 10.01.

S3. Refinement

Hydrogen atoms were included in calculated positions and refined as riding on their parent atom with N—H = 0.88 Å and U_{iso}(H) = 1.2U(N_{eq}), $C_{aromatic}$—H = 0.95 Å and U_{iso}(H) = 1.2U(C_{eq}) or C—H = 0.98 Å and U_{iso}(H) = 1.5U(C_{eq}), for methyl C atoms.

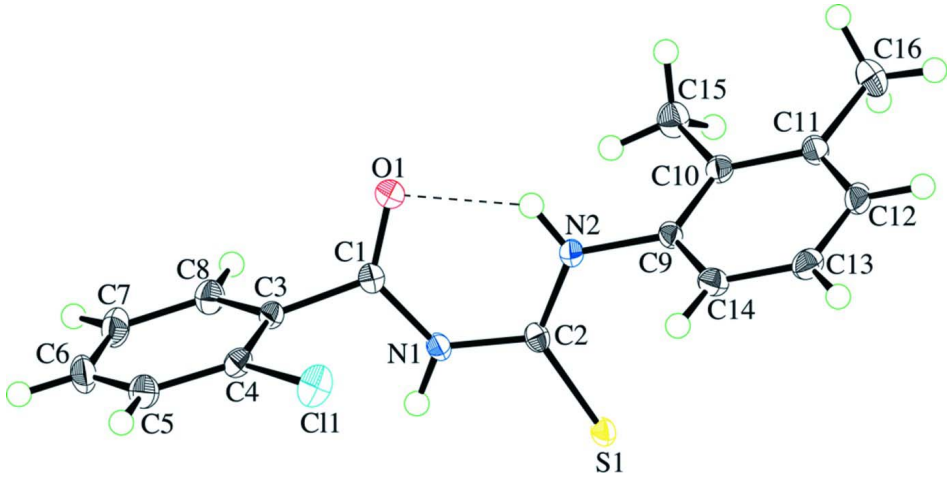

Figure 1

ORTEP of (I). Displacement ellipsoids are drawn at the 50% probability level. Hydrogen bonds shown as dashed lines.

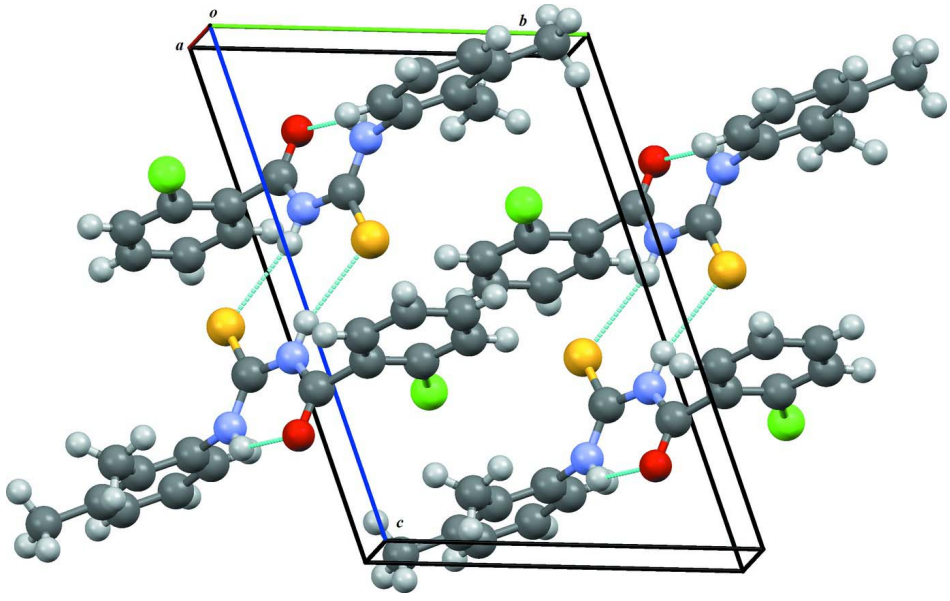

Figure 2

Packing diagram of (I). Hydrogen bonds shown as dashed lines.

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Crystal data

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$\beta = 69.975$ (16)°
$\gamma = 73.639$ (17)°
$V = 783.9$ (5) Å^3
$Z = 2$
$F(000) = 332$
$D_x = 1.351$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71070$ Å
Cell parameters from 2732 reflections
$\theta = 3.0$–27.5°
$\mu = 0.38$ mm^{-1}

T = 123 K
Prism, colorless

0.50 × 0.35 × 0.28 mm

Data collection

Rigaku/MSC Mercury CCD diffractometer
Radiation source: Rotating Anode
Graphite Monochromator monochromator
Detector resolution: 14.62 pixels mm^{-1}
ω scans
6180 measured reflections

3506 independent reflections
3373 reflections with $I > 2\sigma(I)$
R_{int} = 0.062
θ_{max} = 27.5°, θ_{min} = 3.5°
h = −6→9
k = −9→12
l = −10→17

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.090
S = 1.08
3506 reflections
192 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
$w = 1/[\sigma^2(F_o^2) + (0.0371P)^2 + 0.4197P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 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}
C1	0.38592 (19)	0.07516 (15)	0.27734 (11)	0.0162 (3)
O1	0.44634 (15)	0.17028 (13)	0.18262 (9)	0.0245 (2)
N1	0.20232 (16)	0.09731 (13)	0.34511 (9)	0.0170 (2)
H1	0.1791	0.0275	0.4152	0.020*
C2	0.04854 (19)	0.21635 (15)	0.31664 (11)	0.0151 (2)
S1	−0.15915 (5)	0.22091 (4)	0.41821 (3)	0.01758 (10)
N2	0.07877 (16)	0.31921 (13)	0.20967 (9)	0.0172 (2)
H2	0.1972	0.3159	0.1655	0.021*
C3	0.51000 (18)	−0.07725 (15)	0.32976 (11)	0.0157 (3)
C4	0.48935 (19)	−0.22203 (16)	0.33182 (11)	0.0175 (3)
C5	0.6119 (2)	−0.36192 (17)	0.37358 (12)	0.0234 (3)
H5	0.5988	−0.4597	0.3728	0.028*
C6	0.7535 (2)	−0.35614 (18)	0.41627 (13)	0.0269 (3)
H6	0.8371	−0.4512	0.4460	0.032*
C7	0.7751 (2)	−0.21271 (19)	0.41617 (13)	0.0258 (3)

H7	0.8721	−0.2102	0.4462	0.031*
C8	0.6538 (2)	−0.07287 (17)	0.37186 (12)	0.0211 (3)
H8	0.6695	0.0255	0.3704	0.025*
Cl1	0.30905 (5)	−0.22869 (5)	0.28008 (3)	0.02652 (11)
C9	−0.07073 (18)	0.43554 (16)	0.16191 (10)	0.0155 (3)
C10	−0.06255 (19)	0.59756 (16)	0.12305 (11)	0.0166 (3)
C11	−0.2070 (2)	0.70696 (16)	0.07143 (11)	0.0188 (3)
C12	−0.3533 (2)	0.65092 (18)	0.06365 (11)	0.0206 (3)
H12	−0.4519	0.7251	0.0304	0.025*
C13	−0.3589 (2)	0.48910 (18)	0.10335 (11)	0.0213 (3)
H13	−0.4604	0.4533	0.0973	0.026*
C14	−0.2158 (2)	0.38012 (17)	0.15185 (11)	0.0191 (3)
H14	−0.2166	0.2690	0.1779	0.023*
C15	0.0930 (2)	0.65647 (18)	0.13585 (13)	0.0236 (3)
H15C	0.1687	0.5671	0.1833	0.035*
H15A	0.0342	0.7369	0.1724	0.035*
H15B	0.1773	0.7038	0.0602	0.035*
C16	−0.2044 (2)	0.88359 (18)	0.02548 (14)	0.0299 (3)
H16C	−0.3084	0.9405	−0.0127	0.045*
H16A	−0.0802	0.9059	−0.0295	0.045*
H16B	−0.2229	0.9192	0.0888	0.045*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0165 (6)	0.0149 (6)	0.0178 (6)	−0.0008 (5)	−0.0061 (5)	−0.0059 (5)
O1	0.0191 (5)	0.0235 (5)	0.0202 (5)	−0.0012 (4)	−0.0032 (4)	−0.0004 (4)
N1	0.0172 (5)	0.0141 (5)	0.0143 (5)	0.0016 (4)	−0.0037 (4)	−0.0030 (4)
C2	0.0173 (6)	0.0126 (5)	0.0161 (6)	−0.0001 (5)	−0.0061 (5)	−0.0059 (5)
S1	0.01736 (17)	0.01515 (16)	0.01454 (16)	0.00274 (11)	−0.00288 (12)	−0.00425 (12)
N2	0.0138 (5)	0.0177 (5)	0.0152 (5)	0.0010 (4)	−0.0040 (4)	−0.0031 (4)
C3	0.0140 (6)	0.0156 (6)	0.0149 (6)	0.0003 (5)	−0.0025 (5)	−0.0055 (5)
C4	0.0149 (6)	0.0193 (6)	0.0183 (6)	−0.0020 (5)	−0.0018 (5)	−0.0092 (5)
C5	0.0230 (7)	0.0174 (6)	0.0253 (7)	0.0014 (5)	−0.0021 (6)	−0.0095 (5)
C6	0.0234 (7)	0.0227 (7)	0.0270 (7)	0.0076 (6)	−0.0087 (6)	−0.0070 (6)
C7	0.0185 (7)	0.0310 (8)	0.0285 (7)	0.0027 (6)	−0.0112 (6)	−0.0111 (6)
C8	0.0191 (6)	0.0216 (7)	0.0245 (7)	−0.0018 (5)	−0.0078 (5)	−0.0094 (5)
Cl1	0.02276 (19)	0.0319 (2)	0.0347 (2)	−0.00257 (14)	−0.00959 (15)	−0.02058 (16)
C9	0.0147 (6)	0.0169 (6)	0.0111 (5)	0.0013 (5)	−0.0037 (4)	−0.0037 (5)
C10	0.0157 (6)	0.0184 (6)	0.0138 (5)	−0.0011 (5)	−0.0029 (5)	−0.0057 (5)
C11	0.0184 (6)	0.0181 (6)	0.0153 (6)	0.0013 (5)	−0.0038 (5)	−0.0046 (5)
C12	0.0162 (6)	0.0260 (7)	0.0146 (6)	0.0025 (5)	−0.0055 (5)	−0.0048 (5)
C13	0.0167 (6)	0.0297 (7)	0.0170 (6)	−0.0045 (5)	−0.0050 (5)	−0.0069 (5)
C14	0.0206 (6)	0.0184 (6)	0.0160 (6)	−0.0037 (5)	−0.0041 (5)	−0.0040 (5)
C15	0.0229 (7)	0.0225 (7)	0.0283 (7)	−0.0031 (5)	−0.0097 (6)	−0.0096 (6)
C16	0.0303 (8)	0.0173 (7)	0.0344 (8)	0.0012 (6)	−0.0100 (7)	−0.0037 (6)

Geometric parameters (Å, º)

C1—O1	1.2211 (17)	C8—H8	0.9500
C1—N1	1.3743 (17)	C9—C14	1.392 (2)
C1—C3	1.5017 (18)	C9—C10	1.395 (2)
N1—C2	1.3912 (17)	C10—C11	1.4115 (19)
N1—H1	0.8800	C10—C15	1.504 (2)
C2—N2	1.3299 (17)	C11—C12	1.389 (2)
C2—S1	1.6770 (14)	C11—C16	1.508 (2)
N2—C9	1.4385 (17)	C12—C13	1.388 (2)
N2—H2	0.8800	C12—H12	0.9500
C3—C8	1.389 (2)	C13—C14	1.385 (2)
C3—C4	1.3917 (19)	C13—H13	0.9500
C4—C5	1.390 (2)	C14—H14	0.9500
C4—Cl1	1.7367 (15)	C15—H15C	0.9800
C5—C6	1.385 (2)	C15—H15A	0.9800
C5—H5	0.9500	C15—H15B	0.9800
C6—C7	1.393 (2)	C16—H16C	0.9800
C6—H6	0.9500	C16—H16A	0.9800
C7—C8	1.393 (2)	C16—H16B	0.9800
C7—H7	0.9500		

O1—C1—N1	123.81 (12)	C14—C9—C10	122.26 (13)
O1—C1—C3	121.86 (12)	C14—C9—N2	117.73 (12)
N1—C1—C3	114.33 (11)	C10—C9—N2	119.95 (12)
C1—N1—C2	128.06 (11)	C9—C10—C11	117.91 (13)
C1—N1—H1	116.0	C9—C10—C15	121.91 (13)
C2—N1—H1	116.0	C11—C10—C15	120.18 (13)
N2—C2—N1	116.79 (11)	C12—C11—C10	119.49 (13)
N2—C2—S1	124.85 (10)	C12—C11—C16	119.89 (13)
N1—C2—S1	118.37 (10)	C10—C11—C16	120.61 (13)
C2—N2—C9	123.85 (11)	C13—C12—C11	121.55 (13)
C2—N2—H2	118.1	C13—C12—H12	119.2
C9—N2—H2	118.1	C11—C12—H12	119.2
C8—C3—C4	119.39 (12)	C14—C13—C12	119.64 (13)
C8—C3—C1	119.55 (12)	C14—C13—H13	120.2
C4—C3—C1	120.99 (12)	C12—C13—H13	120.2
C5—C4—C3	121.20 (13)	C13—C14—C9	119.12 (13)
C5—C4—Cl1	119.12 (11)	C13—C14—H14	120.4
C3—C4—Cl1	119.67 (11)	C9—C14—H14	120.4
C6—C5—C4	118.81 (14)	C10—C15—H15C	109.5
C6—C5—H5	120.6	C10—C15—H15A	109.5
C4—C5—H5	120.6	H15C—C15—H15A	109.5
C5—C6—C7	120.82 (14)	C10—C15—H15B	109.5
C5—C6—H6	119.6	H15C—C15—H15B	109.5
C7—C6—H6	119.6	H15A—C15—H15B	109.5
C6—C7—C8	119.76 (14)	C11—C16—H16C	109.5
C6—C7—H7	120.1	C11—C16—H16A	109.5

C8—C7—H7	120.1	H16C—C16—H16A	109.5
C3—C8—C7	119.99 (14)	C11—C16—H16B	109.5
C3—C8—H8	120.0	H16C—C16—H16B	109.5
C7—C8—H8	120.0	H16A—C16—H16B	109.5
O1—C1—N1—C2	7.9 (2)	C1—C3—C8—C7	−177.24 (12)
C3—C1—N1—C2	−172.60 (12)	C6—C7—C8—C3	1.1 (2)
C1—N1—C2—N2	2.5 (2)	C2—N2—C9—C14	−64.20 (17)
C1—N1—C2—S1	−177.20 (11)	C2—N2—C9—C10	118.53 (15)
N1—C2—N2—C9	171.32 (12)	C14—C9—C10—C11	−0.39 (19)
S1—C2—N2—C9	−8.96 (19)	N2—C9—C10—C11	176.76 (11)
O1—C1—C3—C8	74.09 (18)	C14—C9—C10—C15	179.07 (12)
N1—C1—C3—C8	−105.46 (15)	N2—C9—C10—C15	−3.78 (19)
O1—C1—C3—C4	−102.77 (16)	C9—C10—C11—C12	1.59 (19)
N1—C1—C3—C4	77.68 (16)	C15—C10—C11—C12	−177.88 (12)
C8—C3—C4—C5	−1.0 (2)	C9—C10—C11—C16	−178.78 (12)
C1—C3—C4—C5	175.84 (12)	C15—C10—C11—C16	1.75 (19)
C8—C3—C4—Cl1	179.64 (10)	C10—C11—C12—C13	−1.4 (2)
C1—C3—C4—Cl1	−3.49 (17)	C16—C11—C12—C13	178.98 (13)
C3—C4—C5—C6	1.6 (2)	C11—C12—C13—C14	−0.1 (2)
Cl1—C4—C5—C6	−179.05 (11)	C12—C13—C14—C9	1.29 (19)
C4—C5—C6—C7	−0.9 (2)	C10—C9—C14—C13	−1.1 (2)
C5—C6—C7—C8	−0.5 (2)	N2—C9—C14—C13	−178.27 (11)
C4—C3—C8—C7	−0.3 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···S1^{i}	0.88	2.46	3.3104 (15)	164
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Symmetry code: (i) −*x*, −*y*, −*z*+1.