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N-(2-Chlorophenyl)-*N*′-(4-methoxybenzoyl)-thiourea

M. Sukeri M. Yusof and Bohari M. Yamin*

School of Chemical Sciences and Food Technology, Universiti Kebangsaan Malaysia, 43600 Bangi, Selangor, Malaysia

Correspondence e-mail: bohari@pkrisc.cc.ukm.my

Key indicators

Single-crystal X-ray study
T = 273 K
Mean σ(C–C) = 0.004 Å
R factor = 0.059
wR factor = 0.140
Data-to-parameter ratio = 16.6

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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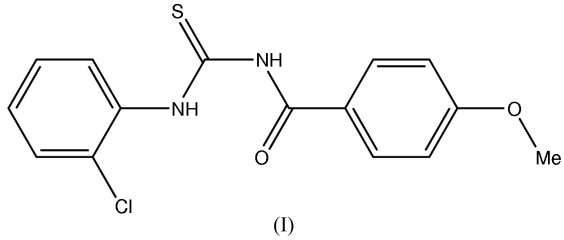
In the title compound, $C_{15}H_{13}ClN_2O_2S$, the dihedral angle between the 2-chlorophenyl and 4-methoxyphenyl groups is 54.12 (13)°. The molecule is stabilized by intermolecular C—H···S and N—H···S interactions, forming double-column chains arranged parallel to the *b* axis.

Comment

The molecular structure and dimensions of the title compound, (I), are similar to those of other benzoylthiourea derivatives, such as *N*-benzoyl-*N*′-(2-chlorophenyl)thiourea (Yusof & Yamin, 2004) and *N*-benzoyl-*N*′-phenylthiourea (Yamin & Yusof, 2003). The molecule maintains its *cis–trans* configuration with respect to the position of the 4-methoxyphenyl and 2-chlorophenyl groups relative to the S atom across the thiourea C—N bonds.



(I)

The central carbonylthiourea moiety (S1/C8/N1/N2/C9/O1), 4-methoxyphenyl (C1–C6/O2/C15) and 2-cholorophenyl (C9–C14/Cl1) fragments are each planar, the maximum deviation being 0.075 (3) Å for atom C15. The central carbonylthiourea moiety makes dihedral angles of 9.91 (10) and 44.23 (11)°, respectively, with the 4-methoxyphenyl and 2-chlorophenyl fragments. This can be compared with values of 11.96 (9) and 29.36 (8)° for the same angles in *N*-benzoyl-*N*′-(2-chlorophenyl)thiourea. However, the dihedral angle between the 4-methoxyphenyl and 2-chlorophenyl fragments of 54.12 (13)° is

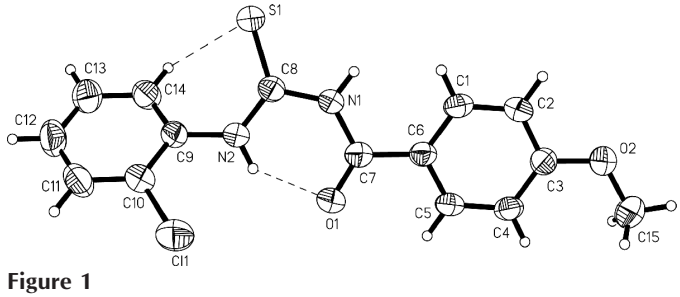


Figure 1
The molecular structure of compound (I), shown with 50% probability displacement ellipsoids. Dashed lines indicate intramolecular hydrogen bonds.

larger than the value of 38.67 (10)° for *N*-benzoyl-*N*′-(2-chlorophenyl)thiourea.

There are two intramolecular hydrogen bonds, N2—H2···O1 and C14—H14···S1 (Table 2), and as a result, two pseudo-six-membered rings (S1—C8—N2—C9—C14—H14 and O1—C7—N1—C8—N2—H2) are formed. In the crystal structure, the molecules are linked by intermolecular interactions, C1—H1*A*···S1^{i} and N1—H1···S1^{ii} [symmetry codes: (i) $\frac{3}{2}-x, y-\frac{1}{2}, \frac{1}{2}-z$; (ii) $\frac{3}{2}-x, \frac{1}{2}+y, \frac{1}{2}-z$; Table 2], forming double-column chains along the *b* axis.

Experimental

A solution of 2-(chlorophenyl)aniline (2.00 g, 0.016 mol) in acetone (50 ml) was added dropwise to an acetone solution (50 ml) containing an equimolar amount of 4-methoxyphenylbenzoyl isothiocyanate in a two-necked round-bottomed flask. The solution was refluxed for about 2 h and then cooled in ice. The white precipitate was filtered off and washed with ethanol–distilled water, then dried in a vacuum (yield 87%). Recrystallization from ethanol yielded single crystals suitable for X-ray analysis.

Crystal data

$C_{15}H_{13}ClN_2O_2S$
M_r = 320.78
Monoclinic, $P2_1/n$
a = 13.438 (3) Å
b = 3.9893 (9) Å
c = 27.727 (6) Å
β = 99.508 (4)°
V = 1466.0 (5) Å^3
Z = 4
D_x = 1.453 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2167 reflections
θ = 1.5–27.0°
μ = 0.41 mm^{-1}
T = 273 (2) K
Block, colourless
0.22 × 0.19 × 0.16 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.916, T_{max} = 0.938
7879 measured reflections
3162 independent reflections
2649 reflections with $I > 2\sigma(I)$
R_{int} = 0.024
θ_{max} = 27.0°
$h = -13 \rightarrow 17$
$k = -5 \rightarrow 5$
$l = -35 \rightarrow 27$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.059
$wR(F^2)$ = 0.140
S = 1.19
3162 reflections
191 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0589P)^2 + 0.3777P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cl1—C10	1.731 (3)	N1—C8	1.386 (3)
S1—C8	1.662 (2)	N2—C8	1.331 (3)
N1—C7	1.377 (3)	N2—C9	1.415 (3)
C9—N2—C8—N1	−179.3 (2)	C7—N1—C8—S1	177.4 (2)

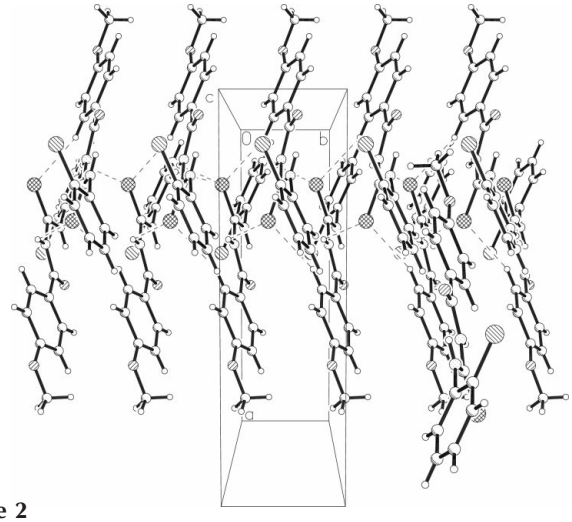


Figure 2
Packing diagram of (I), viewed down the *c* axis. Dashed lines indicate the C—H···S hydrogen bonds.

Table 2
Hydrogen-bonding geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O1	0.86	1.93	2.638 (3)	138
C14—H14···S1	0.93	2.80	3.209 (3)	107
C1—H1*B*···S1^{i}	0.93	2.80	3.686 (3)	160
N1—H1*A*···S1^{ii}	0.86	2.87	3.478 (3)	129

Symmetry codes: (i) $\frac{3}{2}-x, y-\frac{1}{2}, \frac{1}{2}-z$; (ii) $\frac{3}{2}-x, \frac{1}{2}+y, \frac{1}{2}-z$.

After their location in a difference map, all H atoms were placed geometrically in ideal positions and allowed to ride on the parent atoms, with C—H = 0.93–0.96 Å and N—H = 0.86 Å, and U_{iso}(H) = 1.5U_{eq}(methyl C) or 1.2U_{eq}(C,N).

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL, PARST* (Nardelli, 1995) and *PLATON* (Spek, 2003).

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