Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

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

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Received 5 October 2004
Accepted 11 October 2004
Online 22 October 2004

In the title compound, $C_{15}H_{13}BrN_2O_2S$, the 4-methoxybenzoyl and 2-bromophenyl fragments are located *trans* and *cis*, respectively, with respect to the thione group across the C—N bonds. The central carbonylthiourea (N_2C_2SO) moiety makes dihedral angles with the 4-methoxybenzoyl and 2-bromophenyl fragments of 10.57 (12) and 46.88 (13)°, respectively.

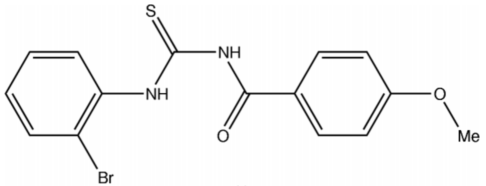
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.042
wR factor = 0.100
Data-to-parameter ratio = 15.2

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

Comment

In the title compound, (I), the 4-methoxybenzoyl and 2-bromophenyl groups are located at *trans* and *cis* positions, respectively, about the thione group across the C8—N1 and C8—N2 bonds. The bond lengths and angles are comparable with other thioureas, such as *N*′-benzoyl-*N*-(*p*-bromophenyl)thiourea (Yamin & Yusof, 2003*a*) and *N*-benzoyl-*N*′-phenylthiourea (Yamin & Yusof, 2003*b*). The central carbonylthiourea (C7/N1/C8/S1/N2/O1), 4-methoxyphenyl (C1–C6/O2/C15) and 2-bromophenyl (C9–C14/Br1) moieties are all planar. The maximum deviation is 0.066 (4) Å for atom C15 from the mean plane of the 4-methoxyphenyl group. The dihedral angles between the central carbonylthiourea moiety and both the 4-methoxyphenyl and 2-bromophenyl fragments are 10.57 (12) and 46.88 (13)°, respectively. The inclination between the aryl fragments of 57.42 (16)°, is larger than the values of 38.61 (11) and 33.3 (1)° in *N*′-benzoyl-*N*-(*p*-bromophenyl)thiourea and *N*-benzoyl-*N*′-phenylthiourea, respectively. There are two intramolecular interactions, N2—H2*A*···O1 and C14—H14*A*···S1. As a result, two pseudo-six-membered rings (N2—C8—N1—C7—O1···H2*A* and H14*A*···S1—C8—N2—C9—C14) are formed. The crystal structure is stabilized by weak intermolecular interactions [C1—H1*B*···S1^{i} and N1—H1A···S1^{ii}; symmetry code: (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], with molecules arranged as dimers parallel to the *ac* face (Fig. 2).



(I)

Experimental

A solution of 2-bromoaniline (1.50 g, 8.77 mmol) in acetone (50 ml) was added dropwise to an acetone solution (50 ml) containing an equimolar amount of 4-methoxybenzoyl 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. Recrystallization from methanol yielded single crystals suitable for X-ray analysis (yield 76%, 2.44 g, 6.66 mmol; m.p. 433–435 K). ^{1}H NMR: δ 2.50 (CH$_3$, *s*), 11.61 (NH, *s*), 12.65 (NH, *s*), 7.06–8.02 (C_6H_4 rings); ^{13}C{^{1}H} NMR δ: 55.81 (CH$_3$), 180.76 (CS), 167.95 (CO), 114.09–163.57 (C_6H_4 rings).

Crystal data

$C_{15}H_{13}BrN_2O_2S$
$M_r = 365.24$
Monoclinic, $P2_1/n$
$a = 13.576$ (2) Å
$b = 4.0362$ (6) Å
$c = 27.495$ (4) Å
$\beta = 98.621$ (3)°
$V = 1489.6$ (4) Å^3
$Z = 4$

$D_x = 1.629$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1580 reflections
$\theta = 1.5$–$26.0°$
$\mu = 2.91$ mm^{-1}
$T = 293$ (2) K
Slab, colourless
$0.30 \times 0.13 \times 0.09$ mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.471$, $T_{max} = 0.772$
7392 measured reflections

2891 independent reflections
2095 reflections with $I > 2\sigma(I)$
$R_{int} = 0.028$
$\theta_{max} = 26.0°$
$h = -15 \rightarrow 16$
$k = -4 \rightarrow 4$
$l = -33 \rightarrow 33$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.042$
$wR(F^2) = 0.100$
$S = 1.03$
2891 reflections
190 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0489P)^2 + 0.0218P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.33$ e Å^{-3}
$\Delta\rho_{min} = -0.22$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

S1—C8	1.662 (3)	N1—C8	1.378 (4)
O1—C7	1.229 (4)	N2—C8	1.338 (4)
O1—C7—N1	120.9 (3)	N2—C8—S1	125.9 (2)
N2—C8—N1	115.6 (3)	N1—C8—S1	118.5 (2)

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

D—H⋯*A*	*D*—H	H⋯*A*	*D*⋯*A*	*D*—H⋯*A*
N2—H2*A*⋯O1	0.86	1.94	2.640 (3)	138
C14—H14*A*⋯S1	0.93	2.84	3.224 (3)	106
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 positioned geometrically and allowed to ride on their parent atoms, with C—H = 0.93–0.96 Å and N—H = 0.86 Å, and $U_{iso}(H) = 1.5U_{eq}(C)$ for CH$_3$, $1.2U_{eq}(C)$ for CH and $1.2U_{eq}(N)$ for NH groups.

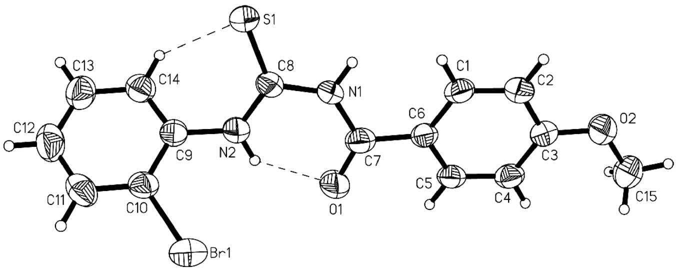


Figure 1
The molecular structure of the title compound, (I), shown with 50% probability displacement ellipsoids. Dashed lines indicate hydrogen bonds.

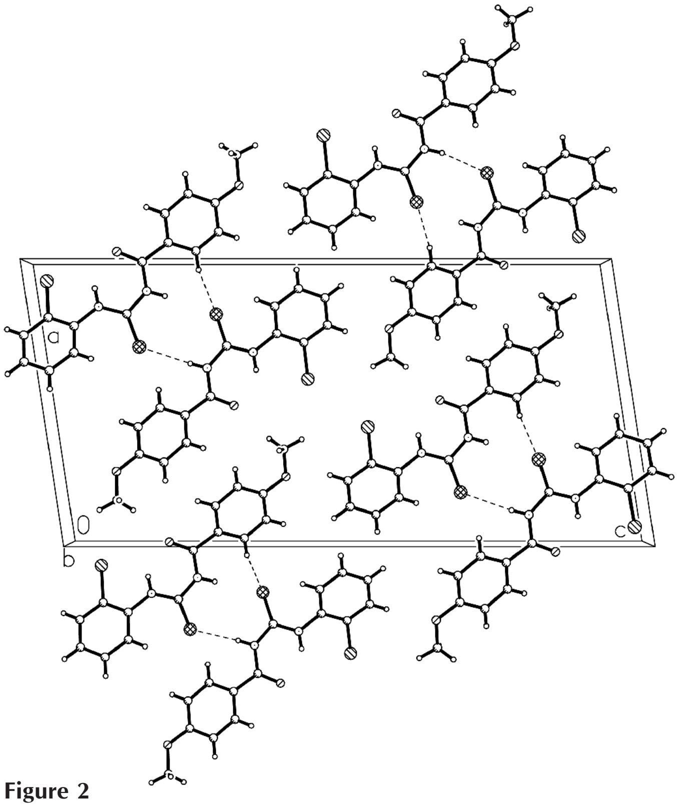


Figure 2
Packing diagram of compound (I). The dashed lines indicate N—H⋯S and C—H⋯S hydrogen bonds.

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).

The authors thank the Malaysian Government and Universiti Kebangsaan Malaysia for the research grant IRPA No. 09-02-02-0163.

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