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N'-Benzoyl-N-p-bromophenylthiourea

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In the title compound, $C_{14}H_{11}BrN_2OS$, the bromophenyl and benzoyl groups lie *cis* and *trans*, respectively, to the S atom across the thiourea C—N bonds. Owing to the presence of the Br atom in the *para* position, the dihedral angle between the bromophenyl group and the central carbonylthiourea plane is increased to 20.40 (11)°, in comparison with 7.52 (9)° in its unsubstituted phenyl analogue. The molecules are linked by intermolecular contacts N—H···S and C—H···O to form linear chains parallel to the *a* axis.

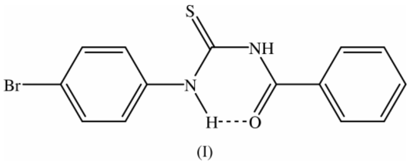
Key indicators

Single-crystal X-ray study
T = 273 K
Mean σ(C–C) = 0.004 Å
R factor = 0.033
wR factor = 0.091
Data-to-parameter ratio = 17.9

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

Comment

Benzoylthiourea derivatives adopt a *cis–trans* conformation with respect to the position of the substituent and benzoyl groups relative to the S atom across the C—N bonds (Shanmuga Sundara Raj *et al.*, 1999; Usman *et al.*, 2002; Kaminsky *et al.*, 2002). The structure and bond dimensions of the title compound, (I), are in agreement with those found in arylbenzoylthioureas, including *N*-phenyl-*N′*-benzoylthiourea, (II) (Yamin & Yusof, 2003). However, the presence of Br1 at the *para* position causes the dihedral angle between the bromophenyl plane (C9–C14/Br1) and the central thiourea fragment (S1/C8/N1/N2/C7/O1) to increase from 7.52 (9)° in (II) to 20.40 (11)° (Table 1). The phenyl (C1–C6) plane and the bromophenyl plane make an angle of 38.61 (11)°, compared to 33.3 (1)° in (II) (Yamin & Yusof, 2003). The dihedral angle between the phenyl and the central thiourea fragment is 29.93 (11)°. As in (II), there are two intramolecular hydrogen bonds, C14—H14*A*···S1 and N2—H2*A*···O1, maintaining the presence of a pseudo-six-membered ring (N1—C8—N2—H2*A*···O1—C7) (Table 2). However, the molecules are packed through weak N1—H1*B*···S1^{i} and C11—H11···O1^{ii} [symmetry codes: (i) $1-x, -y, 2-z$; (ii) $-x, -y, 2-z$] intermolecular contacts to form a linear chain parallel to the *a* axis (Fig. 2).



(I)

Experimental

A solution of *para*-bromoaniline (0.50 g, 2.9 mmol) in acetone (50 ml) was added dropwise to 50 ml of an acetone solution containing an equimolar amount of benzoyl thiocyanate in a two-neck round-bottomed flask. The solution was refluxed for about 1 h and then cooled in ice. The white precipitate which formed was filtered off and washed with ethanol–distilled water, then dried in a vacuum (yield 79%). Recrystallization from DMSO yielded single crystals suitable for X-ray analysis.

Crystal data

$C_{14}H_{11}BrN_2OS$
$M_r = 335.22$
Monoclinic, $P2_1/c$
$a = 13.846$ (3) Å
$b = 5.9486$ (14) Å
$c = 16.972$ (4) Å
$\beta = 103.311$ (4)°
$V = 1360.3$ (6) Å^3
$Z = 4$
$D_x = 1.637$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2075 reflections
$\theta = 1.5$–27.5°
$\mu = 3.16$ mm^{-1}
$T = 273$ (2) K
Block, colourless
0.33 × 0.30 × 0.22 mm

Data collection

Bruker SMART APEX CCD area-detector
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.421$, $T_{\max} = 0.542$
7554 measured reflections
3081 independent reflections
2284 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.026$
$\theta_{\max} = 27.5°$
$h = -12 \rightarrow 17$
$k = -7 \rightarrow 7$
$l = -21 \rightarrow 22$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.033$
$wR(F^2) = 0.091$
$S = 0.91$
3081 reflections
172 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0511P)^2 + 0.2498P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.41$ e Å^{-3}
$\Delta\rho_{\min} = -0.48$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Br1—C12	1.900 (2)	O1—C7	1.220 (3)
S1—C8	1.661 (3)	C7—N1	1.372 (3)
N2—C8	1.325 (3)	C8—N1	1.394 (3)
N2—C9	1.418 (3)		
C8—N2—C9	132.1 (2)	N2—C8—N1	114.3 (2)
C13—C12—Br1	119.47 (19)	N2—C8—S1	127.57 (19)
O1—C7—N1	122.1 (2)	N1—C8—S1	118.15 (18)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*A*···O1	0.86	1.87	2.606 (3)	143
C14—H14*A*···S1	0.93	2.64	3.246 (3)	123
N1—H1*B*···S1$^{\rm i}$	0.86	2.72	3.534 (2)	158
C11—H11*A*···O1$^{\rm ii}$	0.93	2.51	3.421 (3)	165

Symmetry codes: (i) $1 - x, -y, 2 - z$; (ii) $-x, -y, 2 - z$.

After their location in a difference map, all H atoms were included in the refinement in geometrically determined positions and made to ride on the parent C or N atoms, with C—H = 0.97 Å and N—H = 0.89 Å.

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, 1990).

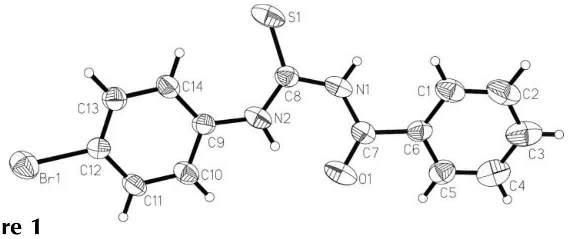


Figure 1
The molecular structure of the title compound, shown with 50% probability displacement ellipsoids.

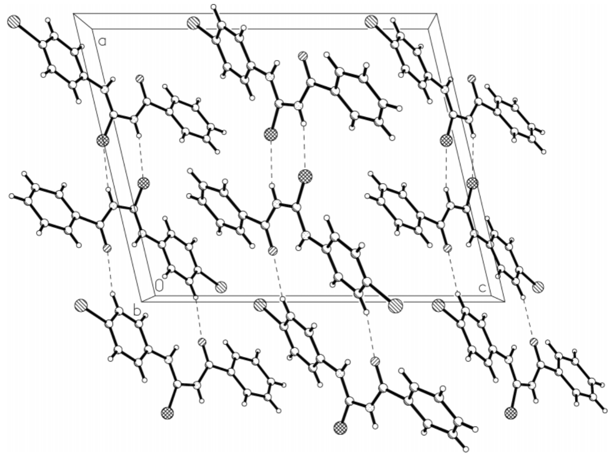


Figure 2
Packing diagram of (I), viewed down the *b* axis. Dashed lines indicate N—H···S and C—H···O hydrogen bonds.

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References

Kaminsky, W., Golberg, K. I. & West, D. X. (2002). *J. Mol. Struct.* **605**, 9–15.
Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
Shanmuga Sundara Raj, S., Puviarasan, K., Velmurugan, D. & Fun, H.-K. (1999). *Acta Cryst.* C**55**, 1318–1320.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXTL*. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.
Siemens (1996). *SMART* and *SAINT*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Spek, A. L. (1990). *Acta Cryst.* A**46**, C-34.
Usman, A., Razak, I. A., Satar, S., Kadir, M. A., Yamin, B. M & Fun, H.-K. (2002). *Acta Cryst.* E**58**, o656–o658.
Yamin, B. M. & Yusof, M. S. (2003). *Acta Cryst.* E**59**, o151–o152.