

N*-p-Bromophenyl-*N'*-(2-chlorobenzoyl)urea*Bohari M. Yamin* and
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43600 Bangi, Selangor, MalaysiaCorrespondence e-mail:
bohari@pkriscc.ukm.my**Key indicators**

Single-crystal X-ray study

 $T = 293\text{ K}$ Mean $\sigma(\text{C}-\text{C}) = 0.004\text{ \AA}$ R factor = 0.036 wR factor = 0.098

Data-to-parameter ratio = 17.3

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

In the title compound, $\text{C}_{14}\text{H}_{10}\text{BrClN}_2\text{O}_2$, the bromophenyl and chlorophenyl groups lie in *cis* and *trans* positions, respectively, across the C–N bonds with respect to the urea carbonyl O atom. In the crystal structure, the molecules form dimers *via* intermolecular hydrogen bonds involving the amino H atom that is closest to the carbonyl O atom of the benzoyl group and the urea carbonyl O atom.

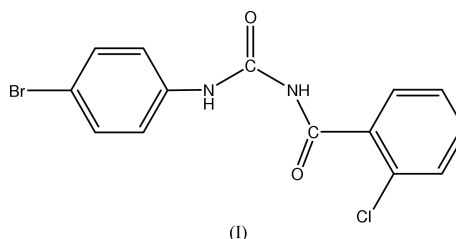
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Comment

It is known that conversion of thiocarbonyls to their carbonyl analogues can be carried out with the help of reagents such as sodium peroxide (Kalm, 1961), dimethyl selenoxide (Mikolajczyk & Luczak, 1978), dimethyl sulfoxide/iodine (Mikolajczyk & Luczak, 1975) and bismuth nitrate (Mohammadpoor-Baltrok *et al.*, 2003). In the course of our work to synthesize a series of chlorobenzoylthiourea derivatives, including *p*-bromophenylchlorobenzoylthiourea, the title compound, (I), was obtained instead.



The bond lengths and angles in (I) are in the normal ranges (Allen *et al.*, 1987) and in agreement with most urea and thiourea derivatives (Stoyanova & Peyerimhoff, 2002; Yamin & Yusof, 2003). The bromophenyl and chlorobenzoyl groups lie *cis* and *trans*, respectively, to atom O2 across the urea C–N bonds. Both aryl rings, C11/C1–C6 and Br1/C9–C14, are essentially planar, with atoms Br1 and C11 deviating by 0.046 (1) and 0.020 (1) Å, respectively, from their mean planes, and an angle between the planes of 57.12 (10)°. The central carbonyl-urea carbonyl fragment (atoms O1/O2/N1/N2/C7/C8) is also planar, with a maximum deviation of 0.039 (2)° for atom C7. The bromophenyl and chlorophenyl planes are inclined to the central carbonyl-urea plane by 7.65 (11) and 55.12 (10)°, respectively. There are two intramolecular interactions N2–H2A···O1 and C14–H14···O2, forming the two pseudo-six-membered rings O1–C7–N1–C8–N2–H2A and O2–C8–N2–C9–C14–H14, respectively. In the crystal structure, the molecules are arranged as dimers by intermolecular hydrogen bonding, N1–H1A···O2ⁱ (symmetry code as in Table 2), almost perpendicular to the *ac* plane, stacking in the *a* direction (Fig. 2).

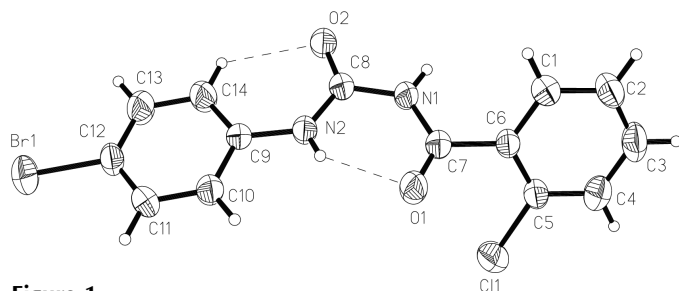


Figure 1
The molecular structure of the title compound, (I), with 50% probability displacement ellipsoids. Intramolecular hydrogen bonds are shown as dashed lines.

Experimental

A solution of 4-bromoaniline (1.411 g, 8.2 mmol) was added dropwise to 50 ml of ethanol containing an equimolar amount of 2-chlorobenzoyl chloride and ammonium thiocyanate in a two-neck round-bottomed flask. The solution was refluxed for *ca* 1 h and then cooled in ice. The orange precipitate was filtered off and washed with methanol–distilled water, then dried at room temperature. Recrystallization from DMSO yielded single crystals suitable for X-ray analysis.

Crystal data

$C_{14}H_{10}BrClN_2O_2$	$D_x = 1.725 \text{ Mg m}^{-3}$
$M_r = 353.60$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 2647 reflections
$a = 7.6761 (7) \text{ \AA}$	$\theta = 2.3\text{--}27.5^\circ$
$b = 17.7520 (17) \text{ \AA}$	$\mu = 3.22 \text{ mm}^{-1}$
$c = 10.3654 (10) \text{ \AA}$	$T = 293 (2) \text{ K}$
$\beta = 105.479 (2)^\circ$	Block, colourless
$V = 1361.2 (2) \text{ \AA}^3$	$0.38 \times 0.21 \times 0.16 \text{ mm}$
$Z = 4$	

Data collection

Bruker SMART APEX CCD area-detector diffractometer	3130 independent reflections
ω scans	2322 reflections with $I > 2\sigma(I)$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$R_{\text{int}} = 0.022$
$T_{\text{min}} = 0.374$, $T_{\text{max}} = 0.627$	$\theta_{\text{max}} = 27.5^\circ$
9216 measured reflections	$h = -9 \rightarrow 9$
	$k = -23 \rightarrow 22$
	$l = -13 \rightarrow 13$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.049P)^2 + 0.3187P]$
$R[F^2 > 2\sigma(F^2)] = 0.036$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.098$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 1.02$	$\Delta\rho_{\text{max}} = 0.61 \text{ e \AA}^{-3}$
3130 reflections	$\Delta\rho_{\text{min}} = -0.28 \text{ e \AA}^{-3}$
181 parameters	
H-atom parameters constrained	

Table 1

Selected geometric parameters (\AA , $^\circ$).

Br1–C12	1.900 (2)	N1–C7	1.361 (3)
Cl1–C5	1.733 (2)	N1–C8	1.402 (3)
O1–C7	1.213 (3)	N2–C8	1.338 (3)
O2–C8	1.215 (3)	N2–C9	1.406 (3)
C7–N1–C8	128.58 (19)	O2–C8–N1	119.13 (19)
C8–N2–C9	127.73 (19)	N2–C8–N1	115.48 (19)
O1–C7–N1	123.9 (2)		

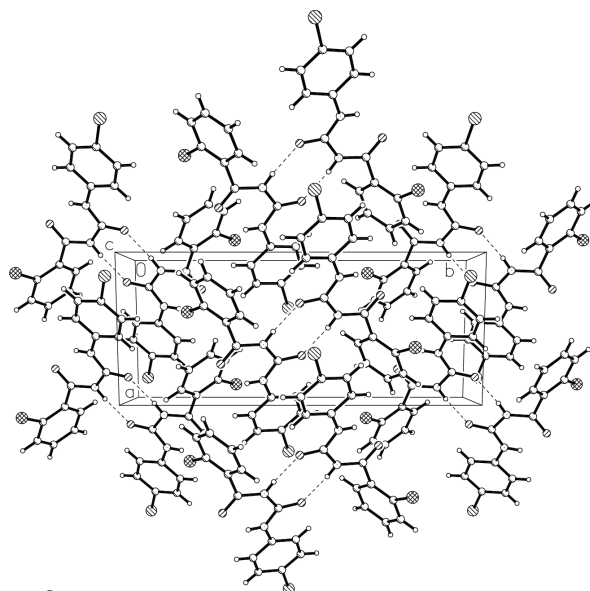


Figure 2
Packing diagram of the title compound, viewed down the c axis. The dashed lines denote $N\text{--}H\cdots O$ hydrogen bonds.

Table 2

Hydrogen-bonding geometry (\AA , $^\circ$).

$D\text{--}H\cdots A$	$D\text{--}H$	$H\cdots A$	$D\cdots A$	$D\text{--}H\cdots A$
$N1\text{--}H1A\cdots O2^i$	0.86	1.98	2.833 (3)	171
$N2\text{--}H2A\cdots O1$	0.86	1.97	2.667 (2)	137
$C14\text{--}H14A\cdots O2$	0.93	2.27	2.873 (3)	122

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

After confirming their presence in difference Fourier maps, all H atoms were fixed geometrically at ideal positions and allowed to ride on the parent C or N atoms, with $C\text{--}H = 0.97 \text{ \AA}$ and $N\text{--}H = 0.89 \text{ \AA}$.

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).

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