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2-Chloro-*N*-[*N*-(4-chlorobenzoyl)hydrazinocarbothioyl]benzamide

Hapipah Ali,[a] M. Sukeri Yusof,[b] Nur Ashikin Khamis,[a] A. Sahali Mardi[b] and Bohari M. Yamin[b]*

[a]Department of Chemistry, Universiti Malaya, Kuala Lumpur, Malaysia, and [b]School of Chemical Sciences and Food Technology, Universiti Kebangsaan Malaysia, 43600 Bangi, Selangor, Malaysia

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

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The title compound, $C_{15}H_{11}Cl_2N_3O_2S$, has a similar structure and similar structural dimensions to the unsubstituted *N*-(*N*-benzoylhydrazinocarbothioyl)benzamide. However, the presence of Cl atoms at the *para* and *ortho* positions in the benzamide and benzoyl groups, respectively, caused the dihedral angle between these groups to increase from 16.42 (14)° in the unsubstituted compound to 74.96 (8)°. The molecule is stabilized by intermolecular C—H···O and N—H···S interactions, forming polymeric chains parallel to the *c* axis.

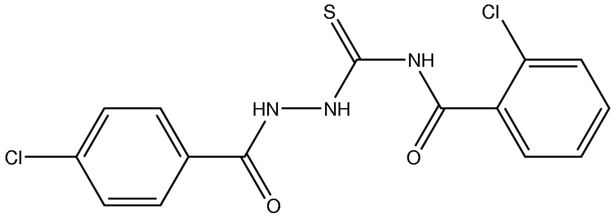
Key indicators

Single-crystal X-ray study
T = 273 K
Mean σ(C–C) = 0.003 Å
R factor = 0.040
wR factor = 0.112
Data-to-parameter ratio = 17.1

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

Comment

The reaction of 2-chlorobenzoylisothiocyanate with 4-chlorobenzhydrazide leads to the formation of the title compound, (I), a chloro-substituted relation of *N*-(*N*-benzoylhydrazinocarbothioyl)benzamide (Yusof *et al.*, 2003). The presence of Cl atoms at the *para* and *ortho* positions of the benzamide and benzoyl groups, respectively, does not change the *cis–trans* configuration with respect to their positions relative to the thiono S1 atom across C8—N2 and C8—N1, respectively (Fig. 1). The bond lengths and angles of (I) (Table 1) are within normal ranges (Allen *et al.*, 1987) and in agreement with those observed in *N*-(*N*-benzoylhydrazinocarbothioyl)benzamide.



(I)

The central thiourea moiety of (I) is planar, such that for the atom sequence N1/C8/S1/N2/N3, the maximum deviation is

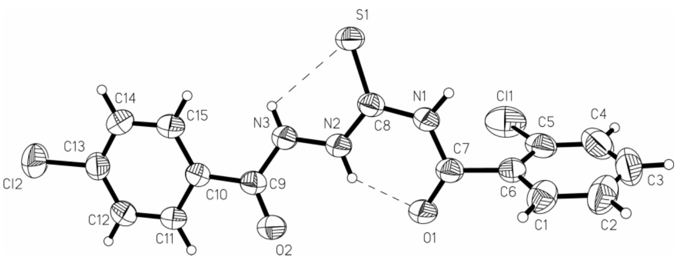


Figure 1
The molecular structure of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii. Dashed lines indicate the intramolecular hydrogen bonds.

0.012 (2) Å for atom N1, and for the chloro-benzamide Cl2/(C9–C15), the maximum deviation is 0.053 (1)° for atom Cl2. The benzoyl fragment is also planar [atom O1 is displaced by −0.873 (1) Å from the mean plane of the phenyl ring (O1/C1–C6)]. However, the dihedral angle between the central moiety and the benzoyl group of 71.57 (8)° is larger than that in *N*-(*N*-benzoylhydrazinocarbothioyl)benzamide [15.12 (11)°]. On the other hand, the dihedral angle with the chloro-benzamide group is reduced from 31.45 (12)° in *N*-(*N*-benzoylhydrazinocarbothiol)benzamide to 5.53 (3)° in (I). Similarly, the inclination between both aryl groups of 74.96 (8)° is larger than the value of 16.42 (14)° in the unsubstituted benzamide, indicating the role of steric effects on the *ortho* isomer.

There are two intramolecular hydrogen bonds in the molecule of (I), N2—H2*A*···O1 and N3—H3*A*···S1, and as a result, a six-membered ring (C7—N1—C8—N2—H2*A*—O1) and a five-membered ring (N2—C8—S1—H3*A*—N3) are formed. In the crystal structure of (I), the molecules are linked by N1—H1*A*···S1^{i} and C11—H12*A*···O2^{ii} intermolecular interactions (symmetry codes as in Table 2) into an infinite chain parallel to the *c* axis (Fig. 2).

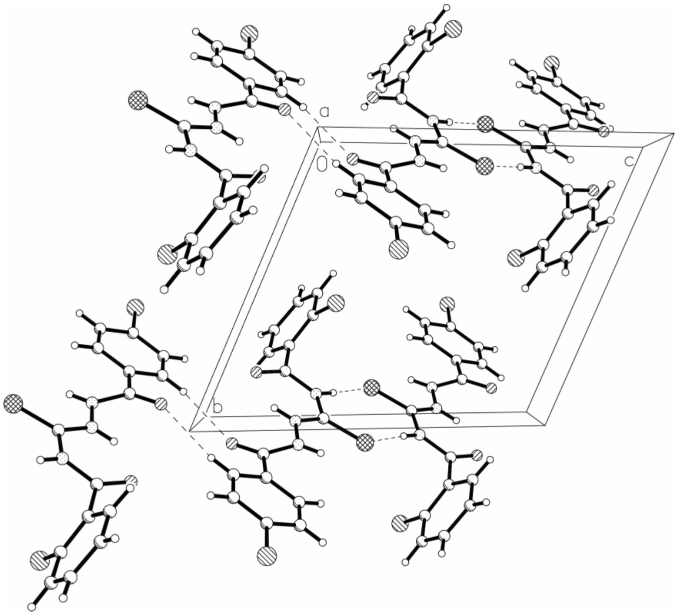


Figure 2
A packing diagram for (I), viewed down the *a* axis. Dashed lines denote C—H···O interactions and N—H···S hydrogen bonds.

Experimental

A solution of 4-chlorobenzhydrazide (1.87 g, 0.011 mol) in acetone (50 ml) was added dropwise to an acetone solution containing an equimolar quantity of 2-chlorobenzoylisothiocyanate in a tri-neck round-bottomed flask. The solution was refluxed for about 1 h and then cooled on ice. The white precipitate which formed was filtered off and washed with ethanol–distilled water, then dried in a vacuum (80% yield). Recrystallization from ethyl acetate yielded single crystals of (I) suitable for X-ray analysis.

Crystal data

$C_{15}H_{11}Cl_2N_3O_2S$
$M_r = 368.24$
Triclinic, $P\bar{1}$
$a = 7.498$ (1) Å
$b = 10.449$ (2) Å
$c = 11.861$ (2) Å
$\alpha = 110.37$ (1)°
$\beta = 104.62$ (1)°
$\gamma = 96.57$ (1)°
$V = 821.7$ (3) Å^3
$Z = 2$
$D_x = 1.488$ Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 936 reflections
$\theta = 1.9$–27.0°
$\mu = 0.53$ mm^{-1}
$T = 273$ (2) K
Block, colourless
0.48 × 0.37 × 0.17 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.783$, $T_{max} = 0.914$
9062 measured reflections
3549 independent reflections
3216 reflections with $I > 2\sigma(I)$
$R_{int} = 0.015$
$\theta_{max} = 27.0°$
$h = -9 \rightarrow 9$
$k = -13 \rightarrow 13$
$l = -15 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.112$
$S = 1.05$
3549 reflections
208 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0581P)^2 + 0.2898P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.54$ e Å^{-3}
$\Delta\rho_{min} = -0.45$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cl1—C5	1.744 (2)	N1—C8	1.384 (2)
Cl2—C13	1.7350 (19)	N2—C8	1.317 (2)
S1—C8	1.6743 (17)	N2—N3	1.375 (2)
N1—C7	1.371 (2)		
C7—N1—C8—N2	3.0 (3)	N2—N3—C9—O2	4.9 (3)
C7—N1—C8—S1	−176.17 (15)	N2—N3—C9—C10	−174.80 (15)
C8—N2—N3—C9	−161.68 (17)	N3—N2—C8—N1	−178.28 (15)
C8—N1—C7—O1	−8.5 (3)	N3—N2—C8—S1	0.8 (3)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2*A*···O1	0.86	1.92	2.613 (2)	136
N3—H3*A*···S1	0.86	2.71	2.9925 (17)	101
N1—H1*A*···S1^{i}	0.86	2.52	3.3649 (17)	170
C11—H11*A*···O2^{ii}	0.93	2.42	3.329 (2)	166

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

After their location in the difference map, all H atoms were fixed geometrically in ideal positions and allowed to ride on their parent atoms, with C—H = 0.93 Å and N—H = 0.86 Å, and with $U_{iso}(H) = 1.2U_{eq}$(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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