N-(4-Chlorobenzoyl)-N′,N′-diethylthiourea

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Key indicators

Single-crystal X-ray study

$T = 100$ K

Mean σ(C–C) = 0.003 Å

R factor = 0.047

wR factor = 0.111

Data-to-parameter ratio = 19.0

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

In the title compound, $C_{12}H_{15}ClN_2OS$, the thiocarbonyl and carbonyl groups are twisted with respect to one another. Molecules are linked through N—H···O hydrogen bonds, forming chains along the c axis.

Comment

We have had an interest in N-aroyl-N′,N′-dialkylthioureas for their ability to selectively extract the platinum group metals (Koch, 2001). More recently, we have discovered that the Pt^{II} and Pd^{II} complexes of such ligands undergo photochemically induced cis-to-trans isomerism (Hanekom et al., 2005), which has given us cause to revisit these ligands in an attempt to exercise control over the isomerism in the associated complexes. The title compound, (I), has been previously characterized by NMR spectroscopy (Koch et al., 1991), IR spectroscopy, melting-point and CHN analysis (Mohamadou et al., 1994). In this paper, the crystal structure of (I) is reported.

(I)
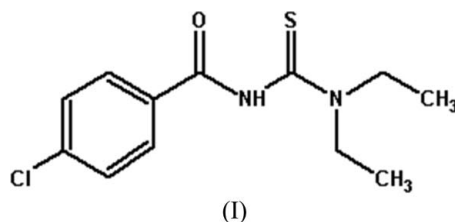
The molecular structure is shown in Fig. 1. The bond lengths (Table 1) in the thiourea group are within the limits of those found for related aroylthioureas (Cambridge Structural Database, Version 5.27; Allen 2002). As expected for this class of compounds, the thiocarbonyl and carbonyl groups are twisted with respect to one another. Molecules are linked via N—H···O hydrogen bonds, forming chains along the c axis (Fig. 2).

Experimental

Compound (I) was prepared according to the method of Douglass & Dains (1934) from 4-chlorobenzoyl chloride, NH$_4$SCN and diethylamine. Single crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of a CDCl$_3$ solution.

Crystal data

$C_{12}H_{15}ClN_2OS$	$Z = 4$
$M_r = 270.77$	$D_x = 1.408$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 6.9055$ (19) Å	$\mu = 0.45$ mm^{-1}
$b = 18.483$ (5) Å	$T = 100$ (2) K
$c = 10.028$ (3) Å	Block, colourless
$\beta = 93.871$ (5)°	$0.25 \times 0.17 \times 0.14$ mm
$V = 1277.0$ (6) Å^3	

Data collection

Bruker APEX CCD area-detector diffractometer	7901 measured reflections
ω scans	2964 independent reflections
Absorption correction: multi-scan (*SADABS*; Blessing, 1995)	2405 reflections with $I > 2\sigma(I)$
	$R_{int} = 0.043$
$T_{min} = 0.896$, $T_{max} = 0.940$	$\theta_{max} = 28.3°$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0529P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.047$	$+ 0.2862P]$
$wR(F^2) = 0.111$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.03$	$(\Delta/\sigma)_{max} = 0.001$
2964 reflections	$\Delta\rho_{max} = 0.40$ e Å^{-3}
156 parameters	$\Delta\rho_{min} = -0.35$ e Å^{-3}
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

Cl1—C4	1.742 (2)	N1—C7	1.354 (3)
S1—C8	1.663 (2)	N1—C8	1.436 (3)
O1—C7	1.228 (3)	N2—C8	1.331 (3)
N2—C8—N1	114.41 (18)	N1—C8—S1	119.21 (15)
N2—C8—S1	126.36 (17)		
C8—N1—C7—O1	−5.8 (3)	C7—N1—C8—N2	87.3 (2)
C2—C1—C7—N1	10.4 (3)	C7—N1—C8—S1	−94.0 (2)

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H5⋯O1^i	0.88	2.04	2.858 (1)	155

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

All H atoms were positioned geometrically (C—H = 0.95–0.99 Å and N—H = 0.88 Å) and constrained to ride on their parent atoms, with $U_{iso}(H) = 1.2U_{eq}(C,N)$ or $1.5U_{eq}$(methyl C).

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *X-SEED* (Barbour, 2001; Atwood & Barbour, 2003); software used to prepare material for publication: *X-SEED*.

References

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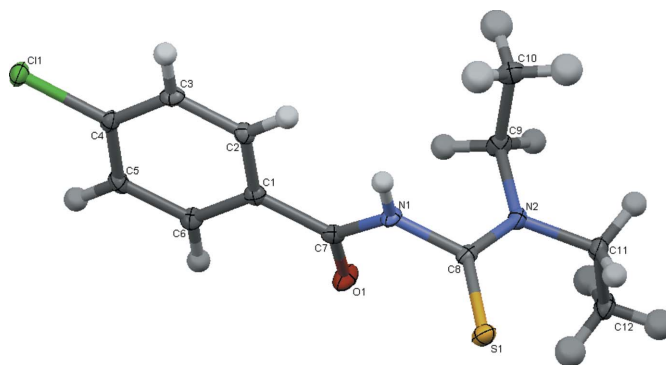Allen, F. H. (2002). *Acta Cryst.* B**58**, 380–388.

Figure 1
The molecular structure of (I), showing atom labels and 50% probability displacement ellipsoids for non-H atoms.
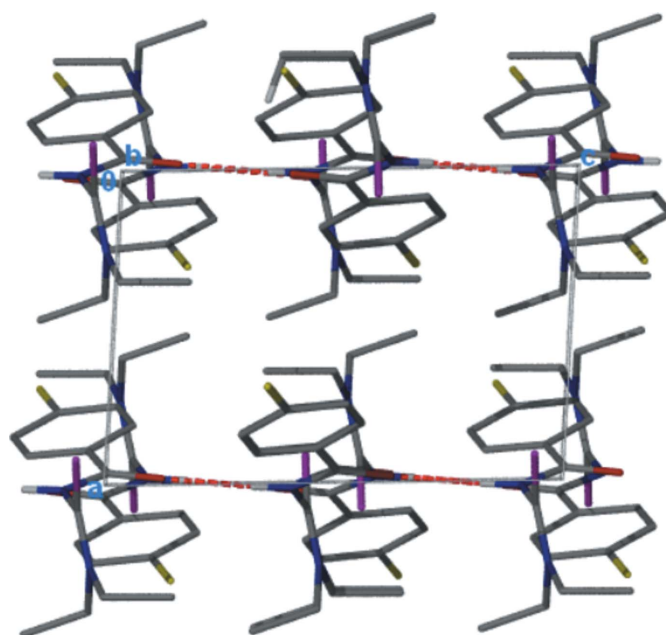

Figure 2
Packing diagram for (I), viewed along the *b* axis. H atoms have been omitted unless these are involved in hydrogen bonds (dashed lines).

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