Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

1-(4-Chlorothiobenzoyl)piperidine

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
R factor = 0.030
wR factor = 0.084
Data-to-parameter ratio = 9.7

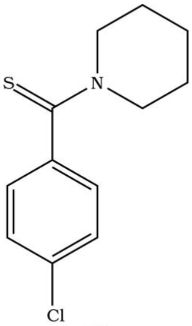
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 1 March 2007
Accepted 31 March 2007

In the title compound, $C_{12}H_{14}ClNS$, the piperidine ring adopts a chair conformation and the crystal structure is stabilized by C—H···Cl interactions.

Comment

The rapidly increasing recognition of aminosulfines (Cashman & Hanzlik, 1982) is due to their reactivity in forming heterocyclic systems with two or more heteroatoms. There are some indications of the biotransformation of thioamides (Chieli & Malvaldi, 1983) and also, in particular, the piperidine ring is a common unit in many biologically active natural products and therapeutic agents (Laschar & Dickner, 2000). Piperidine-containing entities constitute important targets for pharmaceutical research (Escolano & Amat, 2006). In the present study, the new title compound was isolated during the synthesis of the Mannich base of *p*-chlorobenzyl(1-piperidino)thionicotinamide from piperidine, *p*-chlorobenzaldehyde and thionicotinamide.



(I)

In the crystal structure, the average C—N and C—C bond distances in the piperidine ring, are in good agreement with literature values (Zhao *et al.*, 2006). The C=S distance is found to be 0.011 Å shorter than the reported value (Allen *et al.*, 1987; Xu *et al.*, 2005) (Table 1). In the piperidine ring, the plane through the four C atoms (C2, C3, C5 and C6) makes an angle of 58.8 (1)° with the plane of the phenyl ring. The chair form of the piperidine ring is confirmed by the puckering analysis [q_2 = 0.029 (4), φ_2 = 352 (9)°, q_3 = − 0.560 (4) Å; Cremer & Pople, 1975] (Fig. 1).

The crystal structure is stabilized by weak C—H···Cl interactions. This leads to a screw-related chain extending along the b axis of the unit cell (Fig. 2 and Table 2).

Experimental

A mixture of *p*-chlorobenzaldehyde (1.4 g, 0.02 mol), piperidine (1.8 ml, 0.02 mol) and thionicotinamide (1.8 g, 0.02 mol) in ethanol

(20 ml) was heated gently and stirred for 8 h. The solution was then filtered, concentrated and purified by coloum chromatography (silica gel, petroleum ether–ethyl acetate 8:2v/v). Two different compounds were obtained. One of them was the title compound (yield 2.5 g); yellow single crystals were obtained from ethyl acetate–petroleum ether at room temperature.

Crystal data

$C_{12}H_{14}ClNS$
$M_r = 239.75$
Orthorhombic, $P2_12_12_1$
$a = 6.2251$ (9) Å
$b = 7.7283$ (11) Å
$c = 25.061$ (3) Å
$V = 1205.7$ (3) Å^3
$Z = 4$
Mo $K\alpha$ radiation
$\mu = 0.46$ mm^{-1}
$T = 293$ (2) K
0.25 × 0.18 × 0.15 mm

Data collection

Nonius MACH3 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{\min} = 0.866$, $T_{\max} = 0.999$ (expected range = 0.809–0.934)
1333 measured reflections
1316 independent reflections
1046 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.004$
3 standard reflections
frequency: 60 min
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.030$
$wR(F^2) = 0.084$
$S = 1.09$
1316 reflections
136 parameters
H-atom parameters constrained
$\Delta\rho_{\max} = 0.19$ e Å^{-3}
$\Delta\rho_{\min} = -0.17$ e Å^{-3}
Absolute structure: Flack (1983), 45 Friedel pairs
Flack parameter: 0.20 (15)

Table 1
Selected bond lengths (Å).

N—C7	1.327 (4)	N—C6	1.468 (4)
N—C2	1.461 (4)	C7—S	1.669 (4)

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

D—H···A	D—H	H···A	D···A	D—H···A
C6—H6B···Cl$^{\rm i}$	0.97	2.91	3.721 (4)	142

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

All the H atoms were positioned geometrically and refined using a riding model, with C—H = 0.93–0.97 Å with $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(parent atom).

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXTL/PC* (Bruker, 2000); program(s) used to refine structure: *SHELXTL/PC*; molecular graphics: *SHELXTL/PC* and *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *SHELXTL/PC*.

VM thanks Madurai Kamaraj University for University Stipendary Research Fellowship (USRF) support. SA and SN thank the Department of Science and Technology and the University Grant Commission (UGC) for financial assistance.

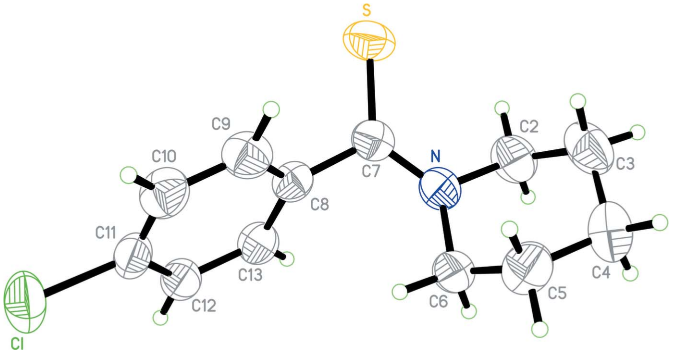


Figure 1
The molecular structure of the title compound (I) with the atom numbering scheme and 50% probability displacement ellipsoids.

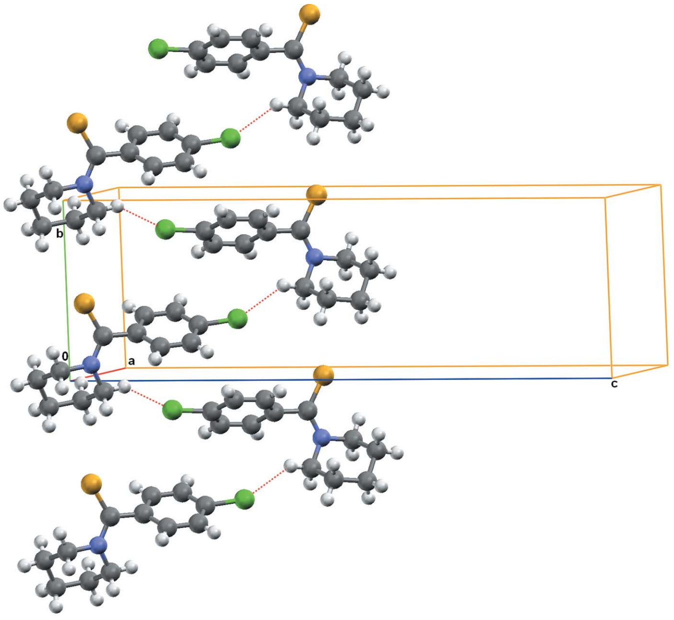


Figure 2
Aggregation of the molecules through C—H···Cl interactions (shown as dashed lines).

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