Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

N′-(4-Chlorobenzoyl)-N-(pyrrolidin-1-yl)thiourea

Esra Kayhan,[a] Ulrich Flörke,[b]* Nevzat Külcü[a] and Hakan Arslan[a]

[a]Department of Chemistry, Faculty of Arts and Science, Mersin University, Mersin, Turkey, and [b]Department Chemie, Fakultät für Naturwissenschaften, Universität Paderborn, Warburgerstr. 100, D-33098 Paderborn, Germany

Correspondence e-mail:
uf@chemie.uni-paderborn.de

Received 18 July 2003
Accepted 21 July 2003
Online 31 July 2003

Key indicators

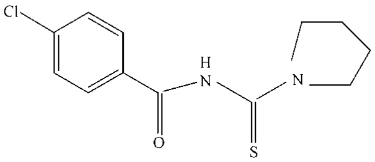
Single-crystal X-ray study
T = 150 K
Mean σ(C–C) = 0.003 Å
R factor = 0.033
wR factor = 0.081
Data-to-parameter ratio = 13.0

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

The title compound, $C_{12}H_{13}ClN_2OS$, shows a twisted conformation with respect to the carbonyl and thiocarbonyl moieties. The crystal packing is determined by strong intermolecular N—H···O interactions.

Comment

Thiourea derivatives are well known for their complexation capacity towards transition metals (Arslan *et al.*, 2003*a*). In this context, we have synthesized several new compounds, including the title compound, (I).



(I)

The molecular structure of (I) differs from that of *N*′-(2-chlorobenzoyl)-*N*-(pyrrolidin-1-yl)thiourea (Arslan *et al.*, 2003*b*) in the position of the chloro ligand. Most of the essential bond lengths, *viz*. S1—C8 = 1.668 (2) Å, C8—N1 = 1.417 (3) Å, N1—C7 = 1.359 (3) Å and C7—C6 = 1.493 (3) Å, are the same as the related bond lengths of the 2-chlorobenzoyl compound, but the C7—O1 distance of 1.238 (3) Å in (I) is slightly longer than the corresponding distance of 1.211 (3) Å. Typical for these molecular structures is the conformational twist with respect to the carbonyl and thiocarbonyl groups. The relevant torsion angles in (I) are C8—N1—C7—O1 = −5.0 (3)° and C7—N1—C8—N2 = −59.6 (3)°. The crystal packing shows the molecules stacked along [100], with strong intermolecular N1—H1···O1($x - \frac{1}{2}$, $\frac{1}{2} - y$, z) hydrogen-bond interactions, with H···O = 1.84 Å and N—H···O = 179°, and a weaker one of C11—H11*A*···O1($1 - x$, $-y$, $\frac{1}{2} + z$), with H···O = 2.42 Å and C—H···O = 141° (values normalized for N—H = 1.03 Å and C—H = 1.08 Å). However, in contrast to the packing of the 2-chlorobenzoyl compound, there are no *D*—H···S interactions and no formation of dimeric pairs of molecules.

Experimental

The title compound was prepared according to the method of Arslan *et al.* (2003*a*) by converting 4-chlorobenzoyl chloride into 4-chlorobenzoyl isothiocyanate and then condensing with pyrrolidine in CH_2Cl_2 solution at 298 K. The compound was recrystallized from ethanol.

DOI: 10.1107/S1600536803016027

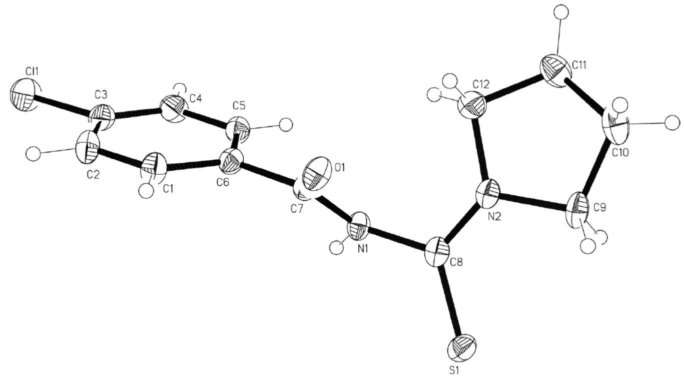


Figure 1
The molecular structure of the title compound. Displacement ellipsoids are drawn at the 50% probability level.

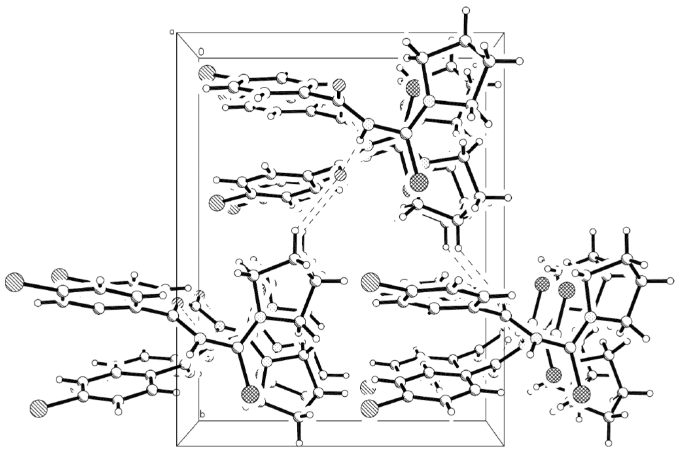


Figure 2
Packing diagram, viewed along [100]. Intermolecular hydrogen bonding is indicated by dashed lines.

Crystal data

$C_{12}H_{13}ClN_2OS$
$M_r = 268.75$
Orthorhombic, $Pna2_1$
$a = 7.8623\ (7)$ Å
$b = 14.2934\ (13)$ Å
$c = 11.3321\ (10)$ Å
$V = 1273.5\ (2)$ Å^3
$Z = 4$
$D_x = 1.402$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3606 reflections
$\theta = 2.3$–$28.2°$
$\mu = 0.45$ mm^{-1}
$T = 150\ (2)$ K
Prism, colorless
$0.40 \times 0.35 \times 0.32$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
$T_{\min} = 0.839$, $T_{\max} = 0.871$
6878 measured reflections
2054 independent reflections
1958 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.037$
$\theta_{\max} = 26.4°$
$h = -9 \rightarrow 9$
$k = -17 \rightarrow 17$
$l = -10 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.033$
$wR(F^2) = 0.081$
$S = 1.04$
2054 reflections
158 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0515P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.48$ e Å^{-3}
$\Delta\rho_{\min} = -0.15$ e Å^{-3}
Absolute structure: Flack (1983)
Flack parameter = 0.00 (6)

Table 1
Selected geometric parameters (Å, °).

Cl1—C3	1.738 (2)	N1—C8	1.417 (3)
S1—C8	1.668 (2)	N2—C8	1.325 (3)
O1—C7	1.238 (3)	C6—C7	1.493 (3)
N1—C7	1.359 (3)		
C7—N1—C8	124.76 (17)	N2—C8—N1	115.38 (19)
O1—C7—N1	123.0 (2)	N2—C8—S1	124.63 (17)
N1—C7—C6	115.21 (18)	N1—C8—S1	119.96 (16)

The H atom bonded to the N atom was refined freely. Other H atoms were placed at calculated positions, riding on their attached C atoms, with isotropic displacement parameters $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$.

Data collection: *SMART* (Bruker, 2002); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 2002); program(s) used to solve structure: *SHELXTL* (Bruker, 2002); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

References

Arslan, H., Flörke, U. & Külcü, N. (2003*a*). *J. Chem. Crystallogr.* In the press.
Arslan, H., Flörke, U. & Külcü, N. (2003*b*). *Acta Cryst.* E59, o641–o642.
Bruker (2002). *SMART* (Version 5.62), *SAINT* (Version 6.02), *SHELXTL* (Version 6.10) and *SADABS* (Version 2.03). Bruker AXS Inc., Madison, Wisconsin, USA.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.