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2-(4-Chloroanilino)carbonylmethyl piperidine-1-carbodithioate

Fu-Hui Zhou,[a] Hong-Bin Zhao,[a]* Zhi-Gang Li,[b] Ning-Hai Hu[b] and Jing-Wei Xu[b]*

[a]The College of Chemistry, Xiangtan University, Hunan 411105, People's Republic of China, and [b]National Analytical Research Center of Electrochemistry and Spectroscopy, Changchun Institute of Applied Chemistry, Chinese Academy of Sciences, Changchun 130022, People's Republic of China

Correspondence e-mail: zhaohbhanlf@163.com, jwxu@ciac.jl.cn

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.033
wR factor = 0.093
Data-to-parameter ratio = 16.2

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

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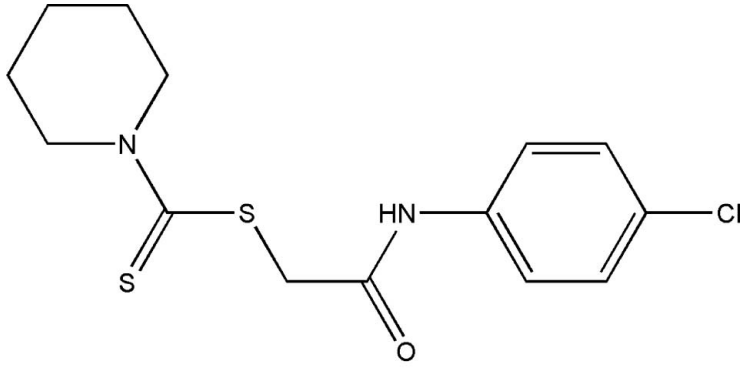
The crystal structure of the title compound, $C_{14}H_{17}ClN_2OS_2$, is stabilized by intermolecular N—H···S hydrogen bonds and π–π stacking interactions.

Comment

The title compound, (I), is a useful lubricant additive. Its molecular structure is shown in Fig. 1. The piperidine ring adopts a chair conformation. The C6—N1 bond length [1.328 (3) Å] is shorter than a C—N single bond. which can be attributed to conjugation between the lone pair electrons of N1 and the C6═S1 double bond (Zhao *et al.*, 2004).



(I)

In the crystal structure, the molecules are connected by hydrogen bonding and π–π stacking interactions. Two molecules form a dimer through intermolecular N—H···S hydrogen bonds (Table 2) that generate an $R_2^2(14)$ ring (Bernstein *et al.*, 1995). In addition, these dimers are connected by a π–π stacking interaction between the chlorobenzene rings at (*x*, *y*, *z*) and (−*x*, 1 − *y*, 1 − *z*), with a centroid–centroid distance of 3.544 Å.

Experimental

A solution of 2-chloro-*N*-(4-chlorophenyl)acetamide (80 mmol) in 30 ml ethanol (Koparır *et al.*, 2005) was added dropwise to a solution of sodium piperidine-1-carbodithioate (80 mmol) in 100 ml ethanol (Garg, 1965) at ambient temperature; the mixture was then refluxed for 5 h. The product was filtered to remove sodium chloride precipitate. The solution was removed *in vacuo* to obtain the crude product and the title compound was obtained as a white solid in 85% yield by recrystallization from ethanol. Slow evaporation of a saturated solution of absolute ethanol produced colourless crystals suitable for X-ray diffraction.

Crystal data

$C_{14}H_{17}ClN_2OS_2$
M_r = 328.87
Triclinic, $P\overline{1}$
a = 7.2299 (10) Å
b = 10.8186 (15) Å
c = 10.8659 (15) Å
α = 83.026 (2)°
β = 71.732 (2)°
γ = 71.615 (2)°
V = 765.63 (18) Å^3
Z = 2
D_x = 1.427 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.52 mm^{-1}
T = 293 (2) K
Block, colourless
0.49 × 0.31 × 0.10 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.787, T_{max} = 0.948
4301 measured reflections
2937 independent reflections
2635 reflections with $I > 2\sigma(I)$
R_{int} = 0.012
θ_{max} = 26.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.093
S = 1.04
2937 reflections
181 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.052P)^2 + 0.1916P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.40 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···S1^{i}	0.86	2.60	3.443 (2)	169

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

H atoms were positioned geometrically, C—H = 0.93 (aromatic) or 0.97 Å (CH_2) and N—H = 0.86 Å, and refined using a riding model [U_{iso}(H) = 1.2U_{eq}(C,N)].

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 2003); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

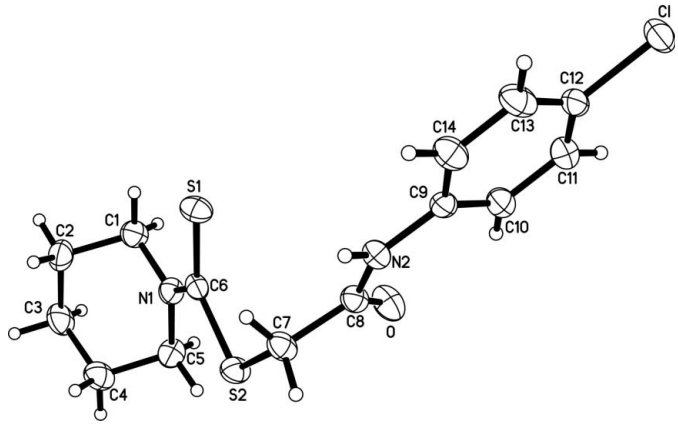


Figure 1
The molecular structure of the title compound, with the atom-labelling scheme and 30% probability displacement ellipsoids (arbitrary spheres for H atoms).

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