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Butyl 2-(piperidine-1-carbothioylsulfanyl)acetate

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Key indicators

Single-crystal X-ray study
T = 273 K
Mean σ(C–C) = 0.005 Å
R factor = 0.049
wR factor = 0.138
Data-to-parameter ratio = 17.9

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

In the title molecule, $C_{12}H_{21}NO_2S_2$, the piperidine ring adopts a chair conformation. In the crystal structure, an intermolecular C—H···S hydrogen bond contributes to the stability.

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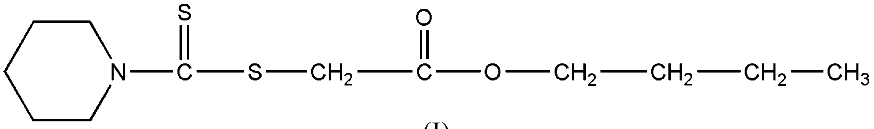
Comment

Substituted piperidines are an important class of compounds, existing as subunits in a number of biologically active compounds (Takemoto *et al.*, 1999; Nishi *et al.*, 1998). A large number of substituted piperidine derivatives have been synthesized and investigated (Chambers *et al.*, 1992). The title compound, (I), has been prepared and its crystal structure is reported here.



(I)

The bond lengths and angles are within normal ranges (Allen *et al.*, 1987) and comparable with those in a related compound, acetonyl morpholine-4-carbodithioate, (II) (Wan *et al.*, 2005). However, the side chain in (II) is oriented differently from the side chain in (I), with respect to the six-membered ring.

The piperidine ring adopts a chair conformation (Fig. 1). Atoms N1, C6, S1 and S2 are coplanar (plane $p1$), with a maximum deviation of 0.004 (2) Å for atom C6. Atoms C7, C8, O1 and O2 are essentially coplanar (plane $p2$), with a maximum deviation of 0.013 (2) Å for atom C8. The dihedral angles between $p1$ and $p2$ and atoms N1/C1/C3/C4 of the piperidine ring are 88.62 (9) and 42.04 (8)°, respectively.

There are four intramolecular hydrogen bonds (Table 1), each forming a five-membered ring. An intermolecular C—H···S hydrogen bond contributes to the stability of the crystal structure (Fig. 2).

Experimental

Sodium piperidine-1-carbodithioate (3.66 g, 0.02 mol) and butyl 2-chloroacetate (3.00 g, 0.02 mol) were dissolved in ethanol (50 ml), and the mixture was stirred for 5 h at about 273 K. The solution was then filtered and the filtrate was evaporated under reduced pressure to give the product (yield 88%). Analysis found: C 52.36, H 7.68, N 5.11%; $C_{12}H_{21}NO_2S_2$ requires: C 55.33, H 7.68, N 5.09%. Single crystals of the title compound suitable for X-ray measurements were obtained by recrystallization from ethanol at room temperature.

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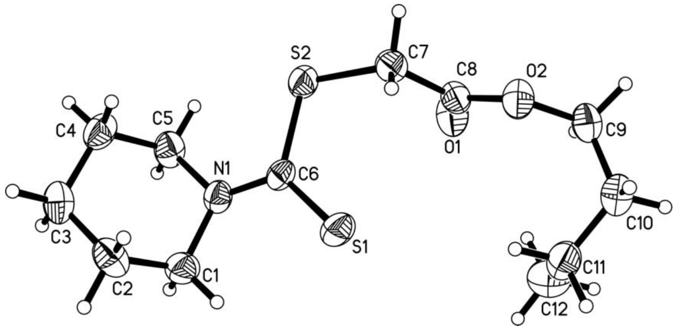

Figure 1
The molecular structure of (I), showing the atom-labeling scheme and 30% probability displacement ellipsoids.

Crystal data

$C_{12}H_{21}NO_2S_2$
$M_r = 275.42$
Monoclinic, $P2_1/n$
$a = 6.504$ (3) Å
$b = 24.123$ (12) Å
$c = 9.506$ (5) Å
$\beta = 107.513$ (7)°
$V = 1422.3$ (12) Å^3
$Z = 4$
$D_x = 1.286$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.37$ mm^{-1}
$T = 273$ (2) K
Plate, colorless
0.36 × 0.24 × 0.08 mm

Data collection

Bruker SMART APEX CCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS* in *SAINT-Plus*; Bruker, 2003)
$T_{min} = 0.879$, $T_{max} = 0.972$
8173 measured reflections
2769 independent reflections
1953 reflections with $I > 2\sigma(I)$
$R_{int} = 0.026$
$\theta_{max} = 26.1°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.049$
$wR(F^2) = 0.138$
$S = 1.02$
2769 reflections
155 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0833P)^2 + 0.0758P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.30$ e Å^{-3}
$\Delta\rho_{min} = -0.17$ e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1A···S1	0.97	2.59	3.080 (3)	112
C5—H5B···S2	0.97	2.38	2.914 (3)	114
C7—H7B···S1	0.97	2.62	3.056 (3)	108
C9—H9B···O1	0.97	2.32	2.735 (4)	105
C7—H7A···S1^{i}	0.97	2.95	3.823 (3)	151

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

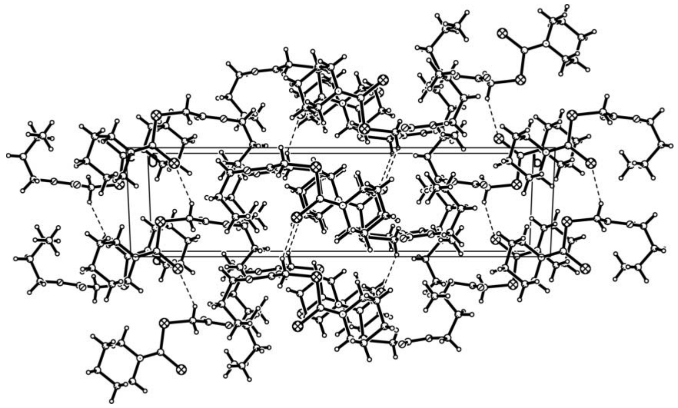

Figure 2
The crystal packing, viewed approximately down the c axis, showing the intermolecular C—H···S hydrogen bonds (dashed lines).

H atoms were placed in calculated positions (C—H = 0.97 or 0.98 Å) and refined in riding mode, with $U_{iso}(H) = xU_{eq}(C)$, where x = 1.5 for methyl and 1.2 for all other H atoms.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT-Plus* (Bruker, 2003); data reduction: *SAINT-Plus*; 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*.

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