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Key indicators

Single-crystal X-ray study
$T = 293$ K
Mean σ(C–C) = 0.004 Å
R factor = 0.057
wR factor = 0.108
Data-to-parameter ratio = 15.6

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

4-(Benzoylthiocarbonyl)morpholine

The title compound, $C_{12}H_{13}NO_2S$, was synthesized from a mixture of morpholine, acetophenone and elemental sulfur dissolved in pyridine. In the crystal structure, the molecules interact through weak intermolecular $C-H\cdots O/S$ interactions, resulting in a three-dimensional network.

Comment

Thioamides have higher chemical activities than their corresponding amides in many chemical reactions as a result of the electronegativity of the S atom. Moreover, they can be synthesized and purified easily (Sosnicki *et al.*, 2001), which is an important consideration for industrial applications. Recently, thioamides have been used as fungistatic reagents (Matysiak *et al.*, 2000) and chain terminators in Sanger–DNA sequencing reactions (Schwarzer *et al.*, 2001). The Willgerodt–Kindler reaction is a more convenient procedure for preparing thioamides than other routes (Kawai *et al.*, 1999).

(I)
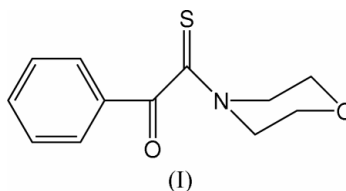
We report here the result of the reaction between morpholine, acetophenone and elemental sulfur, which led to the title compound, (I) (Fig. 1). The geometric parameters for (I) (Table 1) are normal.

There are five weak intermolecular $C-H\cdots O/S$ interactions in the crystal structure (Table 2), as shown in Fig. 2, which connect molecules, resulting in a three-dimensional network (Fig. 3).
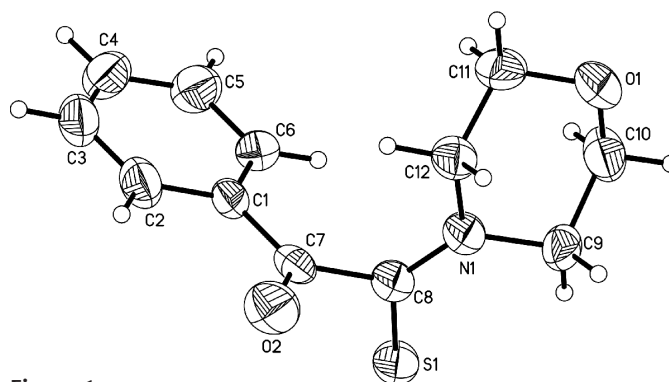

Figure 1
The molecular structure of (I), showing 50% probability displacement ellipsoids (H atoms are shown as spheres of arbitrary radii).
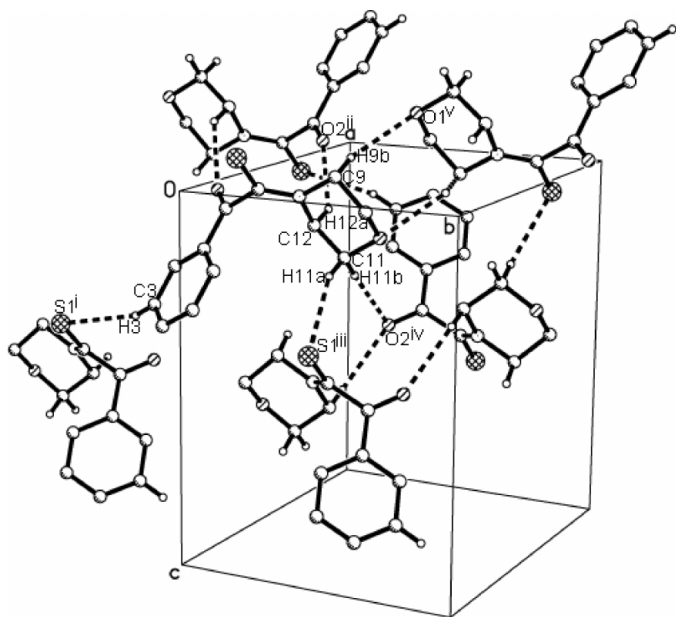
Figure 2
Weak intermolecular interactions (dashed lines) in (I) [symmetry codes: (i) $x, -\frac{1}{2} - y, \frac{1}{2} + z$; (ii) $1 - x, -y, -z$; (iii) $x, \frac{1}{2} - y, \frac{1}{2} + z$; (iv) $1 - x, \frac{1}{2} + y, \frac{1}{2} - z$; (v) $1 - x, 1 - y, -z$]. H atoms not involved in the C—H⋯O/S bonds have been omitted for clarity.
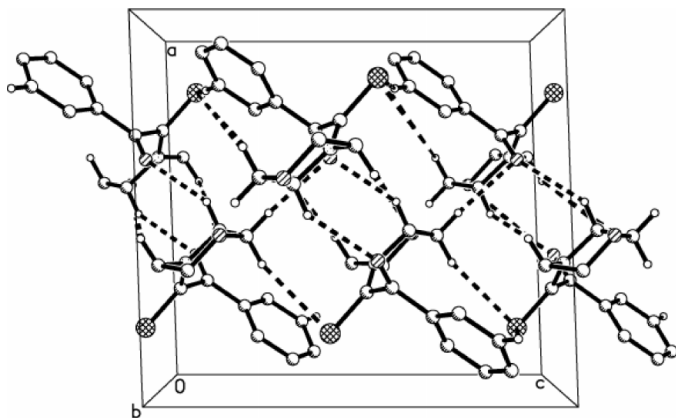

Figure 3
The three-dimensional structure of (I). Dashed lines indicate the weak intermolecular C—H⋯O/S interactions. H atoms not involved in the C—H⋯O/S bonds have been omitted for clarity.

Experimental

A mixture of elemental sulfur (2.6 g, 0.08 mol), morpholine (6.1 ml, 0.07 mol) and acetophenone (6 g, 0.05 mol) in anhydrous pyridine (30 ml) was refluxed with stirring until the acetophenone had almost disappeared (monitored by thin-layer chromatography). To the cooled mixture was added iced water, and then the organic phase was extracted with toluene. The solvent was removed *in vacuo* and the title compound was separated on a column of silica gel with petroleum ether/ethyl acetate as eluants. Yellow crystals of (I) suitable for X-ray structure analysis were obtained by recrystallizing the crude product from acetone.

Crystal data

$C_{12}H_{13}NO_2S$	$D_x = 1.357$ Mg m^{-3}
$M_r = 235.29$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 551 reflections
$a = 10.715$ (2) Å	$\theta = 2.8–18.8°$
$b = 9.200$ (1) Å	$\mu = 0.27$ mm^{-1}
$c = 11.692$ (2) Å	$T = 293$ (2) K
$\beta = 92.02$ (4)°	Block, yellow
$V = 1151.8$ (3) Å^3	$0.30 \times 0.20 \times 0.20$ mm
$Z = 4$	

Data collection

Bruker SMART APEX CCD diffractometer	2264 independent reflections
	1578 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.044$
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)	$\theta_{max} = 26.0°$
	$h = -13 \rightarrow 10$
$T_{min} = 0.94$, $T_{max} = 0.95$	$k = -11 \rightarrow 11$
6029 measured reflections	$l = -14 \rightarrow 13$

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.057$	$w = 1/[\sigma^2(F_o^2) + (0.04P)^2]$
$wR(F^2) = 0.108$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.10$	$(\Delta/\sigma)_{max} < 0.001$
2264 reflections	$\Delta\rho_{max} = 0.32$ e Å^{-3}
145 parameters	$\Delta\rho_{min} = -0.26$ e Å^{-3}

Table 1
Selected torsion angles (°).

O2—C7—C8—N1	−83.7 (3)	S1—C8—N1—C9	−1.5 (4)
C1—C7—C8—N1	99.0 (3)	C7—C8—N1—C12	−7.1 (4)
O2—C7—C8—S1	93.9 (2)	S1—C8—N1—C12	175.67 (19)
C1—C7—C8—S1	−83.4 (2)		

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
C3—H3⋯S1^i	0.93	2.90	3.781 (3)	159
C12—H12a⋯O2^{ii}	0.97	2.59	3.401 (3)	141
C11—H11a⋯S1^{iii}	0.97	2.97	3.890 (3)	159
C11—H11b⋯O2^{iv}	0.97	2.54	3.500 (3)	170
C9—H9b⋯O1^v	0.97	2.57	3.467 (3)	154

Symmetry codes: (i) $x, -\frac{1}{2} - y, \frac{1}{2} + z$; (ii) $1 - x, -y, -z$; (iii) $x, \frac{1}{2} - y, \frac{1}{2} + z$; (iv) $1 - x, \frac{1}{2} + y, \frac{1}{2} - z$; (v) $1 - x, 1 - y, -z$.

All H atoms were placed in calculated positions, with C—H distances 0.93–0.97 Å, and included in the refinement as riding, with $U_{iso}(H) = 1.2U_{eq}$(carrier atom).

Data collection: *SMART* (Bruker, 2000); cell refinement: *SMART*; data reduction: *SAINT* (Bruker, 2000); program(s) used to solve structure: *SHELXTL* (Bruker, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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