

## 2-Oxo-2-phenylethyl morpholine-4-carbodithioate

Liang-Zhong Xu,\* Kai Li,  
Guan-Ping Yu, Guo-Dong Si and  
Cheng-Long YuCollege of Chemistry and Molecular  
Engineering, Qingdao University of Science and  
Technology, Qingdao 266042, People's  
Republic of China

Correspondence e-mail: qknhs@163169.net

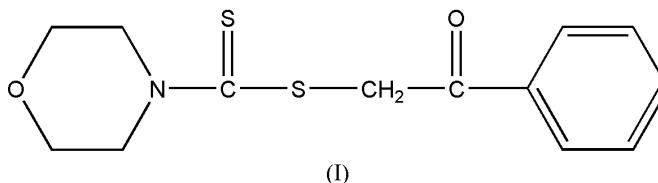
## Key indicators

Single-crystal X-ray study  
 $T = 293$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.004$  Å  
 $R$  factor = 0.027  
 $wR$  factor = 0.096  
Data-to-parameter ratio = 14.5For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.

The title compound,  $\text{C}_{13}\text{H}_{15}\text{NO}_2\text{S}_2$ , the morpholine ring adopts the usual chair conformation and the four C atoms deviate only slightly from coplanarity. The crystal structure is stabilized by some weak intramolecular  $\text{C}-\text{H}\cdots\text{S}$  hydrogen-bond interactions.

## Comment

Morpholine is a versatile chemical which is used as a solvent for resins, dyes and waxes. One of its most important uses is as a chemical intermediate in the preparation of pesticides (Li *et al.*, 1998). Due to their important biological activities, morpholines have attracted a great deal of attention in terms of their synthesis and crystal structures. In a search for new morpholine compounds with higher biological activities, the title compound, (I), was synthesized and we report here its crystal structure.



In the morpholine ring, the average  $\text{C}-\text{N}$ ,  $\text{C}-\text{C}$  and  $\text{C}-\text{O}$  bond distances [1.467 (3), 1.498 (3) and 1.418 (3) Å, respectively] are in good agreement with the values in (4-fluorobenzoyl)(1*H*-1,2,4-triazol-1-yl)methylmorpholine-4-carbodithioate (Xu *et al.*, 2004), *N*-phenyl-4-morpholinecarbothioamide and *N*-(2-tolyl)-4-morpholinecarbothioamide (Ramnathan *et al.*, 1996). The  $\text{C}1=\text{S}1$  bond length is 1.653 (3) Å, in good agreement with the mean value of 1.660 Å reported by Allen *et al.* (1987). The morpholine ring adopts the usual chair conformation and its four C atoms deviate only slightly from coplanarity, in agreement with structural data available from the Cambridge Structural Database (Version 5.14; Allen, 2002). Atoms S1, S2, N1, C1, C2, C5 and C6 are coplanar (plane *p*1). The dihedral angle between the benzene ring and *p*1 is 70.4 (6)°. The crystal structure is stabilized by some weak intramolecular  $\text{C}-\text{H}\cdots\text{S}$  hydrogen-bond interactions (Table 2).

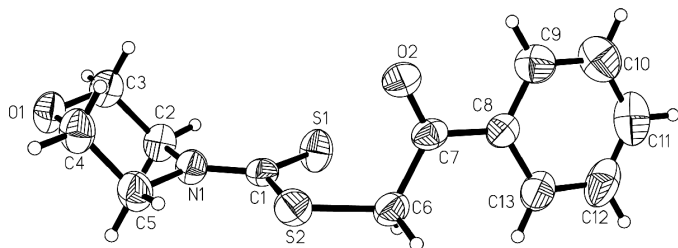
## Experimental

A mixture of 2-bromo-1-phenylethanone (3.98 g, 0.02 mol), sodium morpholine-4-carbodithioate (3.54 g, 0.02 mol) and acetone (50 ml)

Received 13 December 2004

Accepted 21 December 2004

Online 8 January 2005



**Figure 1**  
View of the title compound, (I), with displacement ellipsoids drawn at the 40% probability level.

was stirred for 1.5 h at about 273 K. The solution was then filtered, concentrated and purified by flash chromatography (silica gel, chloroform/cyclohexane 5:1) to afford the title compound (yield 4.78 g, 85%). Single crystals of the title compound suitable for X-ray measurements were obtained by recrystallization from ethyl acetate at room temperature.

**Crystal data**

$C_{13}H_{15}NO_2S_2$   
 $M_r = 281.38$   
Orthorhombic, *Pbca*  
 $a = 9.5294$  (9) Å  
 $b = 8.4805$  (8) Å  
 $c = 33.362$  (3) Å  
 $V = 2696.1$  (4) Å<sup>3</sup>  
 $Z = 8$   
 $D_x = 1.386$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation  
Cell parameters from 2566 reflections  
 $\theta = 2.4$ – $20.1^\circ$   
 $\mu = 0.39$  mm<sup>-1</sup>  
 $T = 293$  (2) K  
Block, colorless  
 $0.54 \times 0.34 \times 0.18$  mm

**Data collection**

Bruker SMART 1000 CCD area-detector diffractometer  
 $\varphi$  and  $\omega$  scans  
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)  
 $T_{\min} = 0.801$ ,  $T_{\max} = 0.933$   
13423 measured reflections

2367 independent reflections  
1349 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.053$   
 $\theta_{\text{max}} = 25.0^\circ$   
 $h = -11 \rightarrow 7$   
 $k = -10 \rightarrow 9$   
 $l = -38 \rightarrow 39$

**Refinement**

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.027$   
 $wR(F^2) = 0.096$   
 $S = 0.87$   
2367 reflections  
163 parameters

H-atom parameters constrained  
 $w = 1/[\sigma^2(F_o^2) + (0.0531P)^2]$   
where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} = 0.001$   
 $\Delta\rho_{\text{max}} = 0.16$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.16$  e Å<sup>-3</sup>

**Table 1**

Selected geometric parameters (Å, °).

S1—C1	1.653 (3)	O1—C3	1.423 (3)
S2—C1	1.776 (3)	O2—C7	1.208 (3)
S2—C6	1.790 (2)	C2—C3	1.493 (3)
N1—C1	1.339 (3)	C4—C5	1.503 (4)
N1—C2	1.462 (3)	C6—C7	1.513 (3)
N1—C5	1.471 (3)	C7—C8	1.490 (3)
O1—C4	1.413 (3)		
C1—S2—C6	101.24 (12)	N1—C1—S1	124.42 (19)
C2—N1—C5	112.0 (2)	N1—C1—S2	113.49 (18)
C4—O1—C3	109.21 (18)	S1—C1—S2	122.09 (15)

**Table 2**

Hydrogen-bonding geometry (Å, °).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
C2—H2B...S1	0.97	2.57	3.065 (3)	112
C5—H5A...S2	0.97	2.38	2.928 (6)	115
C6—H6B...S1	0.97	2.63	3.024 (3)	104

All H atoms were placed in calculated positions, with C—H = 0.93 or 0.97 Å, and included in the final cycles of refinement using a riding model, with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ .

Data collection: SMART (Bruker, 1998); cell refinement: SAINT (Bruker, 1999); 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, 1999); software used to prepare material for publication: SHELXTL.

**References**

Allen, F. H. (2002). *Acta Cryst.* **B58**, 380–388.  
Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.  
Bruker (1998). SMART. Bruker AXS Inc., Madison, Wisconsin, USA.  
Bruker (1999). SAINT and SHELXTL. Bruker AXS Inc., Madison, Wisconsin, USA.  
Li, Z. C., Liu, C. L. & Liu, W. C. (1998). US Patent No. 6 020 332.  
Ramnathan, A., Sivakumar, K., Srinivasan, N., Janarthanan, N., Ramadas, K. & Fun, H.-K. (1996). *Acta Cryst.* **C52**, 1285–1288.  
Sheldrick, G. M. (1996). SADABS. University of Göttingen, Germany.  
Sheldrick, G. M. (1997). SHELXS97 and SHELXL97. University of Göttingen, Germany.  
Xu, L. Z., Zhu, C. Y., Qin, Y. Q., Yu, G. P. & Si, G. D. (2004). *Acta Cryst.* **E60**, o2099–o2100.