

**Jun Wan, Chun-Li Li, Xue-Mei Li  
and Shu-Sheng Zhang\***College of Chemistry and Molecular  
Engineering, Qingdao University of Science and  
Technology, 266042 Qingdao, Shandong,  
People's Republic of China

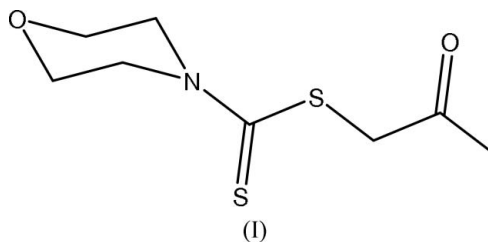
Correspondence e-mail: shushzhang@126.com

**Key indicators**Single-crystal X-ray study  
 $T = 293$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.004$  Å  
 $R$  factor = 0.034  
 $wR$  factor = 0.099  
Data-to-parameter ratio = 17.4For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.**Acetonyl morpholine-4-carbodithioate**

In the title molecule,  $\text{C}_8\text{H}_{13}\text{NO}_2\text{S}_2$ , the morpholine ring adopts a chair conformation. In the crystal structure, a short intermolecular  $\text{O} \cdots \text{S}$  interaction [ $3.214(2)$  Å] links the molecules into spiral chains along the  $b$  axis.

**Comment**

Morpholine derivatives, as an important type of fungicides, have attracted much interest because of their inward absorbent and broad-spectrum activities (Badioli *et al.*, 2002). Dialkyl-substituted dithiocarbamate salts have shown interesting biological effects as broad-range fungicides. In order to search for new morpholine compounds with high bioactivity, the title compound, (I), was synthesized. We present here its crystal structure.



The bond lengths and angles in (I) (Table 1) are within normal ranges (Allen *et al.*, 1987) and comparable with those in a related compound (Köysal *et al.*, 2004). The morpholine ring adopts a chair conformation (Fig. 1), with atoms O1 and N1 deviating by  $-0.66(2)$  and  $0.57(1)$  Å, respectively, from the mean plane through atoms C1–C4. There are three intramolecular  $\text{C}-\text{H} \cdots \text{S}$  interactions (Table 2), each forming a five-membered ring. In the crystal structure, short intermolecular contacts  $\text{O2} \cdots \text{S1}^i$  [ $3.214(2)$  Å; symmetry code: (i)  $1 - x, -\frac{1}{2} + y, -\frac{1}{2} - z$ ] link the molecules into spiral chains along the  $b$  axis (Fig. 2).

**Experimental**

1-Bromoacetone was prepared by the reaction of acetone (3.7 ml, 0.05 mol) and bromine (8.0 g, 2.6 ml) in anhydrous diethyl ether according to Xu *et al.* (2002). (4-Morpholinylcarbothioyl)sulfanylamine was prepared by reacting morpholine (3.9 ml, 0.05 mol) with carbon disulfide (3.0 ml, 0.05 mol) in ammonia (25–28%, 10 ml). The title compound was synthesized by reacting 1-bromoacetone and (4-morpholinylcarbothioyl)sulfanylamine in acetone at room temperature for 2 h. The solution was filtered and purified by flash chromatography (silica gel, petroleum ether–ethyl acetate 6:1 (v/v)). Single crystals suitable for X-ray diffraction study were obtained by slow evaporation of an ethyl acetate–petroleum ether (1:1 (v/v)) solution over a period of two weeks.

Received 22 June 2005

Accepted 30 June 2005

Online 9 July 2005

## Crystal data

$C_8H_{13}NO_2S_2$   
 $M_r = 219.31$   
 Monoclinic,  $P2_1/c$   
 $a = 15.043$  (2) Å  
 $b = 4.9119$  (7) Å  
 $c = 17.1097$  (17) Å  
 $\beta = 123.848$  (8)°  
 $V = 1050.0$  (2) Å<sup>3</sup>  
 $Z = 4$

$D_x = 1.387$  Mg m<sup>-3</sup>  
 Mo  $K\alpha$  radiation  
 Cell parameters from 2917 reflections  
 $\theta = 2.9$ – $26.0$ °  
 $\mu = 0.48$  mm<sup>-1</sup>  
 $T = 293$  (2) K  
 Block, colourless  
 $0.50 \times 0.22 \times 0.07$  mm

## Data collection

Siemens SMART 1000 CCD area-detector diffractometer  
 $\omega$  scans  
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)  
 $T_{\min} = 0.797$ ,  $T_{\max} = 0.968$   
 5540 measured reflections

2048 independent reflections  
 1794 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.013$   
 $\theta_{\text{max}} = 26.1$ °  
 $h = -18 \rightarrow 17$   
 $k = -5 \rightarrow 6$   
 $l = -11 \rightarrow 21$

## Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.034$   
 $wR(F^2) = 0.099$   
 $S = 1.07$   
 2048 reflections  
 118 parameters  
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0575P)^2 + 0.1984P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} < 0.001$   
 $\Delta\rho_{\text{max}} = 0.20$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.19$  e Å<sup>-3</sup>

Table 1

Selected geometric parameters (Å, °).

S1–C5	1.7754 (16)	O1–C3	1.421 (3)
S1–C6	1.7831 (18)	N1–C5	1.330 (2)
S2–C5	1.6604 (16)	N1–C4	1.471 (2)
O1–C2	1.405 (3)	N1–C1	1.473 (2)
C5–S1–C6	102.24 (8)	N1–C5–S1	113.31 (12)
N1–C5–S2	124.69 (12)	S2–C5–S1	121.99 (10)

Table 2

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C1–H1A $\cdots$ S1	0.97	2.37	2.901 (3)	114
C4–H4B $\cdots$ S2	0.97	2.57	3.048 (2)	111
C6–H6A $\cdots$ S2	0.97	2.69	3.057 (2)	103

All H atoms were located in difference Fourier maps and constrained to ride on their parent atoms, with C–H distances in the range 0.93–0.97 Å and with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ .

Data collection: SMART (Siemens, 1996); cell refinement: SAINT (Siemens, 1996); data reduction: SAINT; program(s) used to solve structure: SHELXTL (Sheldrick, 1997); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; software used

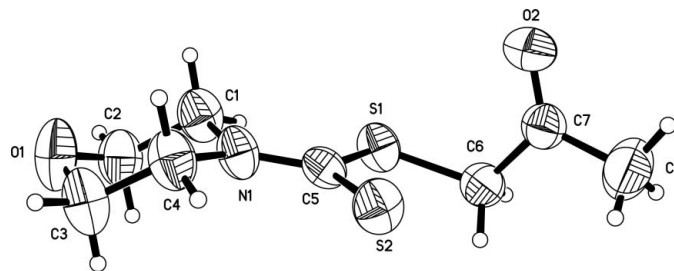


Figure 1

View of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme.

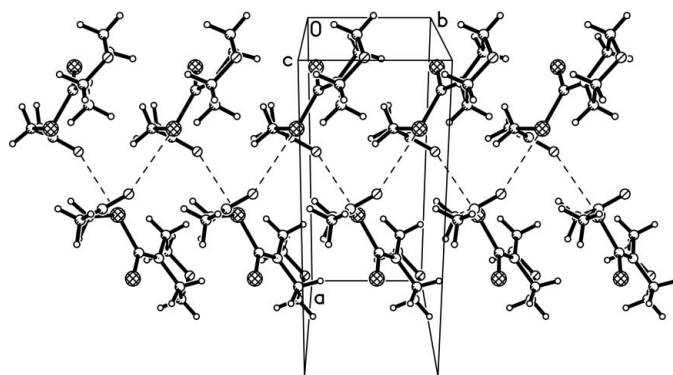


Figure 2

The crystal packing, viewed approximately down the  $c$  axis, showing the short intermolecular  $O\cdots S$  interactions (dashed lines).

to prepare material for publication: SHELXTL, PARST (Nardelli, 1995) and PLATON (Spek, 2003).

This project was supported by the Program for New Century Excellent Talents in University (No. NCET-04-0649), and the Project of Educational Administration of Shandong Province (No. J04B12).

## References

- 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.  
 Badioli, M., Ballini, R., Bartolacci, M., Bosica, G., Torregiani, E. & Marcantoni, E. (2002). *J. Org. Chem.* **67**, 8938–8942.  
 Köysal, Y., Isik, S., Septioglu, E. & Calis, U. (2004). *Acta Cryst.* **C60**, o757–o758.  
 Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.  
 Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.  
 Sheldrick, G. M. (1997). *SHELXTL*. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.  
 Siemens (1996). *SMART* and *SAINTE*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.  
 Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.  
 Xu, L. Z., Jiao, K., Zhang, S. S. & Kuang, S. P. (2002). *Bull. Kor. Chem. Soc.* **23**, 1699–1701.