Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

(4-Chlorobenzoyl)methyl morpholine-4-carbodithionate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean *σ*(C–C) = 0.003 Å
R factor = 0.035
wR factor = 0.088
Data-to-parameter ratio = 16.6

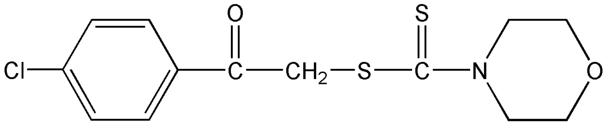
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 18 November 2004
Accepted 9 December 2004
Online 18 December 2004

The title compound, $C_{13}H_{14}ClNO_2S_2$, the morpholine crystallizes in space group $P2_1/c$. The morpholine ring adopts the usual chair conformation and the four C atoms deviate only slightly from coplanarity. There are some weak inter- and intramolecular hydrogen-bond interactions in the crystal structure, providing stabilization.

Comment

Morpholine is a versatile chemical. It is used as a solvent for resins, dyes and waxes. Its alkyl derivatives (*e.g.* *N*-methylmorpholine and *N*-ethylmorpholine) are used as catalysts for the production of polyurethane foams. The most important use is as a chemical intermediate to prepare pharmaceuticals. Drugs containing the morpholine ring have established activities that include the reduction of blood sugar and lipid levels (Yoshioka, 1995), and the amelioration of obesity and insulin resistance (Fisher & Wyvratt, 1990). Owing to their important pharmacological activities, these compounds have received a great deal of attention in respect of their syntheses and in the elucidation of their crystal structures a search of new morpholine compounds with higher pharmacological activities, the title compound, (I), was synthesized.



(I)

In the morpholine ring, the average C—N, C—C and C—O bond distances [1.472 (2), 1.506 (3) and 1.421 (2) Å, respectively] are also in good agreement with earlier reports (Ramnathan *et al.*, 1996; Yavuz *et al.*, 2004). The C5═S2 distance is 0.02 Å shorter than the mean value of 1.681 Å found in an earlier report (Allen *et al.*, 1987). The C—Cl bond length [1.740 (2) Å] is comparable to the value found by Zucco *et al.*, 1999). The morpholine ring adopts the usual chair conformation and the four C atoms deviate only slightly from coplanarity, in agreement with the structural data available from Version 5.14 of the Cambridge Structural Database (Allen, 2002). Atoms S1, S2, N1, C1, C4, C5 and C6 are coplanar (*p*1). The dihedral angle between the C8–C13 benzene ring and *p*1 is 70.4 (6)°. Weak intermolecular and intramolecular hydrogen-bond interactions stabilize the structure (Table 2).

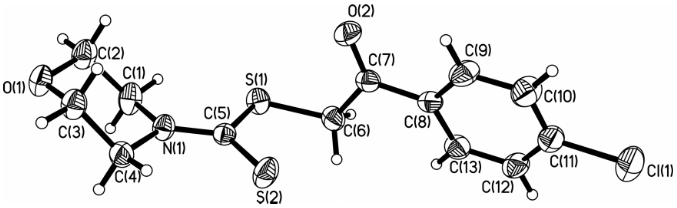


Figure 1
View of (I), with displacement ellipsoids drawn at the 35% probability level.

Experimental

A mixture of 2-bromo-1-(4-chlorophenyl)ethanone (4.67 g, 0.02 mol), sodium morpholine-4-carbodithioate (3.54 g, 0.02 mol) and acetone (50 ml) was stirred for 1 h at around 273 K. The solution was then filtered, concentrated and purified by flash chromatography (silica gel, chloroform–cyclohexane 5:1 *v*/*v*) to afford the title compound (yield 5.05 g, 80%). Single crystals suitable for X-ray measurements was obtained by recrystallization from ethyl acetate at room temperature.

Crystal data

$C_{13}H_{14}ClNO_2S_2$
$M_r = 315.82$
Monoclinic, $P2_1/c$
$a = 18.092$ (5) Å
$b = 9.471$ (2) Å
$c = 8.464$ (2) Å
$\beta = 100.127$ (8)°
$V = 1427.7$ (6) Å^3
$Z = 4$

$D_x = 1.469$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 967 reflections
$\theta = 3.1$–25.7°
$\mu = 0.56$ mm^{-1}
$T = 293$ (2) K
Block, colorless
0.48 × 0.40 × 0.32 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{\min} = 0.761$, $T_{\max} = 0.837$
7932 measured reflections

2876 independent reflections
2096 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.030$
$\theta_{\max} = 26.3°$
$h = -22 \rightarrow 20$
$k = -11 \rightarrow 11$
$l = -10 \rightarrow 6$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.035$
$wR(F^2) = 0.088$
$S = 1.06$
2876 reflections
173 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0352P)^2 + 0.3711P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.002$
$\Delta\rho_{\max} = 0.21$ e Å^{-3}
$\Delta\rho_{\min} = -0.21$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.0260 (16)

Table 1
Selected geometric parameters (Å, °).

S1—C5	1.780 (2)	N1—C4	1.469 (3)
S1—C6	1.787 (2)	N1—C1	1.475 (2)
S2—C5	1.657 (2)	O1—C2	1.416 (3)
Cl1—C11	1.740 (2)	O1—C3	1.426 (2)
N1—C5	1.341 (2)	C3—C4	1.502 (3)
C5—S1—C6	101.47 (10)	C2—O1—C3	109.12 (15)
C4—N1—C1	112.36 (16)	N1—C5—S1	113.54 (14)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1*B*···S1	0.97	2.39	2.932 (3)	115
C4—H4*A*···S2	0.97	2.57	3.068 (3)	112
C4—H4*B*···O2$^{\rm i}$	0.97	2.58	3.544 (4)	172
C6—H6*A*···S2	0.97	2.64	3.032 (2)	104

Symmetry code: (i) *x*, *y*, *z* − 1.

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 $U_{\rm iso}({\rm H}) = 1.2U_{\rm eq}({\rm C})$.

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*.

This project was supported by the National Natural Science Foundation of China (grant No. 40376023).

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