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Crystal structure of 2-methylsulfanyl-1-(thiomorpholin-4-yl)ethanone

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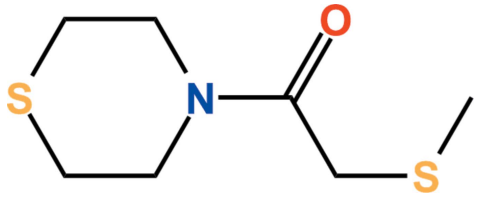
In the title compound, $C_7H_{13}NOS_2$, the thiomorpholine ring adopts a chair conformation and the bond-angle sum at the N atom is 360°. The dihedral angle between the amide group and the thiomorpholine ring (all atoms) is 36.48 (12)°. In the crystal, C—H···O and C—H···S hydrogen bonds link adjacent molecules, forming two-dimensional networks extending parellel to the (011) plane.

Keywords: crystal structure; thiomorpholine; hydrogen bonding.

CCDC reference: 1419333

1. Related literature

For further information on the synthesis, see: Kim *et al.* (2008). For related crystal structures, see: Kim *et al.* (2006); Ujam *et al.* (2010).



2. Experimental

2.1. Crystal data

$C_7H_{13}NOS_2$
M_r = 191.30
Monoclinic, $P2_1/c$
a = 15.0461 (15) Å
b = 6.1525 (6) Å
c = 10.4751 (10) Å
β = 107.581 (6)°
V = 924.40 (16) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.52 mm^{-1}
T = 173 K
0.23 × 0.18 × 0.08 mm

2.2. Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2013)
T_{min} = 0.890, T_{max} = 0.959
8512 measured reflections
2111 independent reflections
1865 reflections with $I > 2\sigma(I)$
R_{int} = 0.026

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.029
$wR(F^2)$ = 0.078
S = 1.05
2111 reflections
101 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.22 e Å^{-3}
$\Delta\rho_{min}$ = −0.27 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1B···O1^{i}	0.99	2.46	3.3490 (19)	150
C6—H6B···O1^{i}	0.99	2.59	3.4427 (18)	144
C7—H7B···O1^{ii}	0.98	2.45	3.3237 (19)	148
C3—H3A···S2^{iii}	0.99	2.88	3.8201 (15)	159

Symmetry codes: (i) $x, -y+\frac{3}{2}, z+\frac{1}{2}$; (ii) $x, y+1, z$; (iii) $x, y-1, z$.

Data collection: *APEX2* (Bruker, 2013); cell refinement: *SAINT* (Bruker, 2013); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2013* (Sheldrick, 2015); molecular graphics: *DIAMOND* (Brandenburg, 2010); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

Acknowledgements

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Supporting information for this paper is available from the IUCr electronic archives (Reference: HB7480).

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supporting information

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Crystal structure of 2-methylsulfanyl-1-(thiomorpholin-4-yl)ethanone

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S1. Comment

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S2. Experimental

Thionyl chloride (2.38 g, 20.0 mmol) was added dropwise to 2-methylthioacetic acid (2.12 g, 20.0 mmol) in the pesence of triethylamine (2.02 g, 20.0 mmol) in chloroform. The mixture was refluxed for 2 h and cooled down to room temperature. Then, thiomorpholine (2.38 g, 20.0 mmol) and triethylamine (2.02 g, 20.0 mmol) in chloroform were added dropwise to the resulting acid chloride solution, cooled by salt and ice water. The solution was stirred for 2 h, and then water was added. Organic layer was collected and water layer was extracted with chloroform. The combined organic layers dried with anhydrous sodium sulfate were evaporated to give crude oil. Column chromatography (silica gel, ethyl acetate/hexane = 20/80 (*v*/*v*), R_f 0.1) gave pure title compound (3.42 g, 89%) (Kim *et al.*, 2008). Slow evaporation of a solution in acetone/ethyl acetate gave colourless blocks.

S3. Refinement

All H-atoms were positioned geometrically and refined using a riding model with d(C—H) = 0.99 Å, U_{iso} = 1.2U_{eq}(C) for CH_2 groups and d(C—H) = 0.98 Å, U_{iso} = 1.5U_{eq}(C) for CH_3 group.

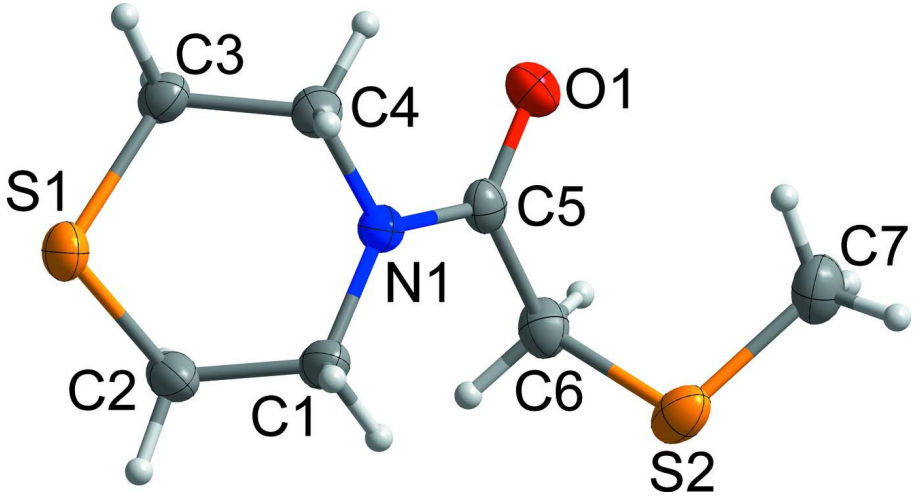

Figure 1

The asymmetric unit of the title compound with displacement ellipsoids drawn at the 50% probability level.

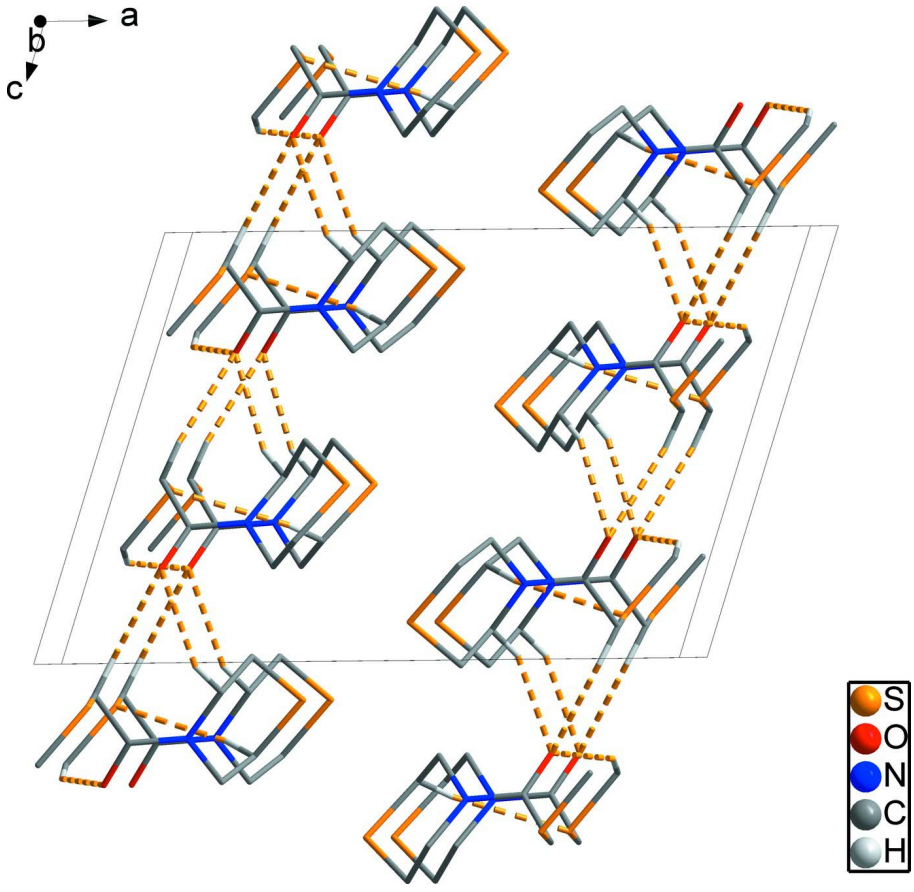

Figure 2

Crystal packing viewed along the *b* axis. The intermolecular C—H···O and C—H···S hydrogen bonds are shown as dashed lines.

2-Methylsulfanyl-1-(thiomorpholin-4-yl)ethanone

Crystal data

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c = 10.4751 (10) Å
β = 107.581 (6)°
V = 924.40 (16) Å^3
Z = 4
$F(000)$ = 408
D_x = 1.375 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 4186 reflections
θ = 2.8–27.5°
μ = 0.52 mm^{-1}
T = 173 K
Block, colourless
0.23 × 0.18 × 0.08 mm

Data collection

Bruker APEXII CCD diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2013)
T_{min} = 0.890, T_{max} = 0.959
8512 measured reflections
2111 independent reflections
1865 reflections with $I > 2\sigma(I)$
R_{int} = 0.026
θ_{max} = 27.5°, θ_{min} = 2.8°
h = −19→19
k = −7→7
l = −13→13

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.029$
$wR(F^2) = 0.078$
$S = 1.05$
2111 reflections
101 parameters
0 restraints

Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0406P)^2 + 0.2667P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.22$ e Å^{-3}
$\Delta\rho_{min} = -0.27$ e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.57059 (2)	0.35556 (6)	0.41078 (4)	0.03060 (12)
S2	0.87043 (3)	1.06954 (6)	0.40092 (4)	0.03528 (13)
O1	0.82963 (8)	0.59419 (17)	0.21785 (11)	0.0333 (2)
N1	0.72025 (8)	0.62668 (19)	0.32412 (12)	0.0264 (3)
C1	0.68637 (10)	0.7095 (2)	0.43112 (15)	0.0315 (3)
H1A	0.6300	0.7989	0.3920	0.038*
H1B	0.7346	0.8037	0.4909	0.038*
C2	0.66298 (11)	0.5254 (3)	0.51228 (15)	0.0325 (3)
H2A	0.6437	0.5870	0.5871	0.039*
H2B	0.7193	0.4357	0.5510	0.039*
C3	0.62268 (10)	0.2963 (2)	0.28028 (15)	0.0280 (3)
H3A	0.6779	0.2023	0.3172	0.034*
H3B	0.5774	0.2148	0.2078	0.034*
C4	0.65213 (10)	0.5000 (2)	0.22214 (14)	0.0296 (3)
H4A	0.6795	0.4586	0.1507	0.036*
H4B	0.5965	0.5909	0.1812	0.036*
C5	0.80749 (9)	0.6585 (2)	0.31488 (14)	0.0243 (3)
C6	0.87815 (10)	0.7787 (2)	0.42581 (15)	0.0297 (3)
H6A	0.9416	0.7294	0.4300	0.036*
H6B	0.8679	0.7434	0.5125	0.036*
C7	0.91981 (12)	1.0968 (3)	0.26585 (18)	0.0385 (4)
H7A	0.9862	1.0593	0.2975	0.058*
H7B	0.9127	1.2471	0.2334	0.058*
H7C	0.8876	0.9988	0.1928	0.058*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0255 (2)	0.0330 (2)	0.0357 (2)	−0.00719 (13)	0.01283 (16)	−0.00026 (15)
S2	0.0262 (2)	0.0305 (2)	0.0515 (3)	−0.00455 (14)	0.01529 (18)	−0.01097 (16)

O1	0.0347 (6)	0.0363 (5)	0.0344 (6)	−0.0013 (4)	0.0187 (5)	−0.0022 (4)
N1	0.0262 (6)	0.0290 (6)	0.0263 (6)	−0.0077 (5)	0.0115 (5)	−0.0052 (5)
C1	0.0313 (8)	0.0309 (7)	0.0369 (8)	−0.0085 (6)	0.0173 (7)	−0.0101 (6)
C2	0.0323 (8)	0.0403 (8)	0.0279 (8)	−0.0093 (6)	0.0134 (6)	−0.0064 (6)
C3	0.0251 (7)	0.0268 (7)	0.0325 (8)	−0.0050 (5)	0.0092 (6)	−0.0058 (6)
C4	0.0288 (7)	0.0341 (7)	0.0248 (7)	−0.0081 (6)	0.0065 (6)	−0.0028 (6)
C5	0.0251 (7)	0.0212 (6)	0.0278 (7)	0.0011 (5)	0.0099 (6)	0.0051 (5)
C6	0.0229 (7)	0.0349 (7)	0.0300 (8)	−0.0034 (6)	0.0059 (6)	0.0037 (6)
C7	0.0374 (9)	0.0309 (8)	0.0490 (10)	−0.0021 (6)	0.0157 (8)	0.0058 (7)

Geometric parameters (Å, º)

S1—C2	1.8061 (15)	C2—H2B	0.9900
S1—C3	1.8065 (14)	C3—C4	1.517 (2)
S2—C7	1.7935 (17)	C3—H3A	0.9900
S2—C6	1.8067 (16)	C3—H3B	0.9900
O1—C5	1.2267 (17)	C4—H4A	0.9900
N1—C5	1.3592 (17)	C4—H4B	0.9900
N1—C1	1.4563 (17)	C5—C6	1.511 (2)
N1—C4	1.4622 (18)	C6—H6A	0.9900
C1—C2	1.520 (2)	C6—H6B	0.9900
C1—H1A	0.9900	C7—H7A	0.9800
C1—H1B	0.9900	C7—H7B	0.9800
C2—H2A	0.9900	C7—H7C	0.9800
C2—S1—C3	97.45 (6)	H3A—C3—H3B	107.8
C7—S2—C6	100.51 (7)	N1—C4—C3	111.87 (12)
C5—N1—C1	125.07 (12)	N1—C4—H4A	109.2
C5—N1—C4	120.24 (11)	C3—C4—H4A	109.2
C1—N1—C4	114.68 (11)	N1—C4—H4B	109.2
N1—C1—C2	111.34 (12)	C3—C4—H4B	109.2
N1—C1—H1A	109.4	H4A—C4—H4B	107.9
C2—C1—H1A	109.4	O1—C5—N1	121.49 (13)
N1—C1—H1B	109.4	O1—C5—C6	119.42 (12)
C2—C1—H1B	109.4	N1—C5—C6	119.09 (12)
H1A—C1—H1B	108.0	C5—C6—S2	111.99 (10)
C1—C2—S1	111.64 (11)	C5—C6—H6A	109.2
C1—C2—H2A	109.3	S2—C6—H6A	109.2
S1—C2—H2A	109.3	C5—C6—H6B	109.2
C1—C2—H2B	109.3	S2—C6—H6B	109.2
S1—C2—H2B	109.3	H6A—C6—H6B	107.9
H2A—C2—H2B	108.0	S2—C7—H7A	109.5
C4—C3—S1	112.52 (10)	S2—C7—H7B	109.5
C4—C3—H3A	109.1	H7A—C7—H7B	109.5
S1—C3—H3A	109.1	S2—C7—H7C	109.5
C4—C3—H3B	109.1	H7A—C7—H7C	109.5
S1—C3—H3B	109.1	H7B—C7—H7C	109.5

C5—N1—C1—C2	−115.63 (15)	C1—N1—C5—O1	−175.84 (13)
C4—N1—C1—C2	64.06 (16)	C4—N1—C5—O1	4.5 (2)
N1—C1—C2—S1	−62.22 (15)	C1—N1—C5—C6	3.2 (2)
C3—S1—C2—C1	53.57 (12)	C4—N1—C5—C6	−176.45 (12)
C2—S1—C3—C4	−52.50 (12)	O1—C5—C6—S2	92.67 (14)
C5—N1—C4—C3	117.12 (14)	N1—C5—C6—S2	−86.42 (13)
C1—N1—C4—C3	−62.59 (16)	C7—S2—C6—C5	−73.01 (11)
S1—C3—C4—N1	59.54 (14)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1*B*···O1^{i}	0.99	2.46	3.3490 (19)	150
C6—H6*B*···O1^{i}	0.99	2.59	3.4427 (18)	144
C7—H7*B*···O1^{ii}	0.98	2.45	3.3237 (19)	148
C3—H3*A*···S2^{iii}	0.99	2.88	3.8201 (15)	159

Symmetry codes: (i) *x*, −*y*+3/2, *z*+1/2; (ii) *x*, *y*+1, *z*; (iii) *x*, *y*−1, *z*.