organic compounds

2-Methylsulfanyl-1-(morpholin-4-yl-methyl)-1H-benzimidazole

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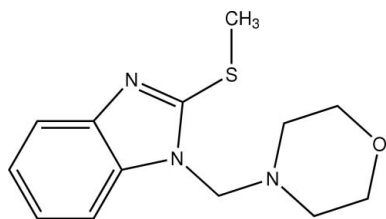Correspondence e-mail: abou_akoun@yahoo.fr

In the title compound, $C_{13}H_{17}N_3OS$, the morpholine ring has a chair conformation. The crystal structure exhibits intermolecular C—H⋯O and C—H⋯N interactions to form a consolidated array.

Related literature

For related literature, see: Dubey *et al.* (1985); Garuti *et al.* (2000); Cremer & Pople (1975).

Experimental

Crystal data

$C_{13}H_{17}N_3OS$	$\gamma = 76.251$ (2)°
$M_r = 263.36$	$V = 672.51$ (4) Å³
Triclinic, $P\bar{1}$	$Z = 2$
$a = 6.2461$ (2) Å	Mo $K\alpha$ radiation
$b = 9.7949$ (2) Å	$\mu = 0.23$ mm^{-1}
$c = 11.6966$ (5) Å	$T = 294$ K
$\alpha = 75.475$ (1)°	$0.40 \times 0.30 \times 0.30$ mm
$\beta = 84.808$ (1)°	

Data collection

Nonius KappaCCD diffractometer	3812 independent reflections
Absorption correction: none	3284 reflections with $I > 2\sigma(I)$
12818 measured reflections	$R_{int} = 0.026$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.043$	163 parameters
$wR(F^2) = 0.096$	H-atom parameters constrained
$S = 0.92$	$\Delta\rho_{max} = 0.23$ e Å^{-3}
3812 reflections	$\Delta\rho_{min} = -0.25$ e Å^{-3}

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
C7—H7⋯O14[i]	0.97	2.62	3.526 (2)	155
C4—H4⋯N3[ii]	0.97	2.64	3.574 (2)	162
C10—H102⋯N3[iii]	0.98	2.66	3.344 (2)	127

Symmetry codes: (i) $-x + 2, -y, -z$; (ii) $-x, -y, -z + 1$; (iii) $x + 1, y, z$.

Data collection: *COLLECT* (Nonius, 2001); cell refinement: *DENZO/SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO/SCALEPACK*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *CRYSTALS*.

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supplementary materials

2-Methylsulfanyl-1-(morpholin-4-ylmethyl)-1*H*-benzimidazole

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Comment

Benzimidazole derivatives are important pharmaceutical intermediates. For example, they are used in the design of anti-helmintic (Dubey *et al.*, 1985) and anti-viral (Garuti *et al.*, 2000) pharmaceutics. In order to have a better knowledge of their structure, we have embarked on a study this class of compounds. In the title compound, $C_{13}H_{17}N_3OS$ (I), the benzimidazole ring N1/C2/N3/C4—C9 is essentially planar and is connected to the morpholine ring, N11/C12/C13/O14/C15/C16, by the methylene-C10 atom. The morpholine ring has a chair conformation as judged from the puckering parameters: $Q_T = 0.571$ Å, $\theta = 179.4°$ and $\varphi = 184.6°$ (Cremer & Pople, 1975) with the C10 atom in an equatorial position. The structure is consolidated by weak intermolecular interactions (Table 1).

Experimental

Morpholine (0.6 ml, 6.09 mmol) and formaldehyde (0.5 ml, 18.27 mmol) were added to 2-methylthiobenzimidazole (1 g, 6.09 mmol) in dry ethanol (20 ml). The mixture was refluxed for 3 h, the solvent removed under vacuum, the residue diluted with water, and extracted with dichloromethane. The organic phase was dried (MgSO$_4$), filtered, and concentrated *in vacuo*. The residue was purified by chromatography (elution with ethyl acetate) to give (I) as colorless crystals (1.1 g, 69%): m. p. 352 K. ^{1}H NMR (DMSO, 300 MHz, p.p.m.) δ 2.50 (t, 4H, 2CH$_2$N), 2.67 (s, 3H, CH$_3$), 3.51 (t, 4H, 2CH$_2$O), 4.76 (s, 2H, CH$_2$, NCH$_2$N), 7.06–7.54 (m, 4H, C$_6$H$_4$). ^{13}C NMR (DMSO, 300 MHz, p.p.m.) δ 14.47 (CH$_3$), 50.50 (CH$_2$NCH$_2$), 65.08 (NCH$_2$N), 65.89 (2 CH$_2$O), 110.15, 117.44, 120.99, 121.54, 134.02, 142.87 (C4—C9), 153.35 (C=N).

Refinement

The H atoms were placed at calculated positions with C—H in the range of 0.96–1.01 Å, and with U_{iso}(H) = 1.13–1.5U_{eq}(carrier).
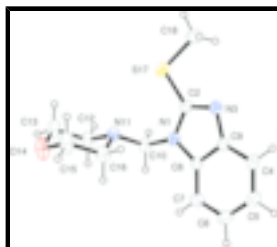
Figures

Fig. 1. The molecular structure of (I) showing the atomic labeling scheme with displacement ellipsoids drawn at the 30% probability level. H atoms are shown as spheres of arbitary radius.

2-Methylsulfanyl-1-(morpholin-4-ylmethyl)-1*H*-benzimidazole

Crystal data

$C_{13}H_{17}N_3O_1S_1$	$Z = 2$
$M_r = 263.36$	$F_{000} = 280$
Triclinic, $P\bar{1}$	$D_x = 1.300$ Mg m^{-3}
Hall symbol: -P 1	Melting point: 352 K
$a = 6.2461$ (2) Å	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$b = 9.7949$ (2) Å	Cell parameters from 3812 reflections
$c = 11.6966$ (5) Å	$\theta = 4–30º$
$\alpha = 75.475$ (1)º	$\mu = 0.23$ mm^{-1}
$\beta = 84.808$ (1)º	$T = 294$ K
$\gamma = 76.251$ (2)º	Prism, colorless
$V = 672.51$ (4) Å^3	$0.40 \times 0.30 \times 0.30$ mm

Data collection

Nonius KappaCCD diffractometer	3284 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.026$
$T = 294$ K	$\theta_{max} = 30.0º$
φ scans	$\theta_{min} = 1.8º$
Absorption correction: none	$h = -8 \rightarrow 8$
12818 measured reflections	$k = -12 \rightarrow 13$
3812 independent reflections	$l = -16 \rightarrow 16$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.043$	Method, part 1, Chebychev polynomial, [Watkin, D. (1994). Acta Cryst. A50, 411–437. *P*rince, E. (1982). Mathematical Techniques in Crystallography and Materials Science. Ne*w* York: Springer-Verlag.] [*w*eight] $= 1.0/[A_0*T_0(x) + A_1*T_1(x) \cdots + A_{n-1}]*T_{n-1}(x)]$ where A_i are the Chebychev coefficients listed below and x $= F /F$max Method = Robust Weighting (*P*rince, 1982) W $= [w$eight$] * [1-(\text{delta}F/6*\text{sigma}F)^2]^2$ A_i are: 488. 738. 431. 172. 33.5
$wR(F^2) = 0.096$	$(\Delta/\sigma)_{max} = <0.001$
$S = 0.92$	$\Delta\rho_{max} = 0.23$ e Å^{-3}
3817 reflections	$\Delta\rho_{min} = -0.25$ e Å^{-3}
163 parameters	Extinction correction: None

Primary atom site location: structure-invariant direct
methods

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

	x	y	z	$U_{iso}*/U_{eq}$
H4	0.0870	−0.1337	0.4686	0.0597*
H5	0.2946	−0.3677	0.4625	0.0673*
H6	0.6429	−0.4009	0.3691	0.0660*
H7	0.8112	−0.2040	0.2879	0.0591*
H101	0.9009	0.0258	0.2412	0.0582*
H102	0.8445	0.1724	0.2873	0.0590*
H121	1.1010	0.1571	0.0820	0.0683*
H122	1.0136	0.3003	0.1296	0.0674*
H131	1.1036	0.3597	−0.0774	0.0902*
H132	0.8395	0.4284	−0.0455	0.0896*
H151	0.5774	0.3214	−0.0837	0.0965*
H152	0.6497	0.1739	−0.1392	0.0961*
H161	0.5522	0.1153	0.0655	0.0701*
H162	0.8054	0.0401	0.0394	0.0708*
H181	0.1456	0.3821	0.4361	0.0934*
H182	0.1018	0.5284	0.3327	0.0928*
H183	0.0064	0.3947	0.3217	0.0930*
N1	0.58426 (17)	0.08468 (11)	0.29036 (9)	0.0419
C2	0.3987 (2)	0.17950 (13)	0.31895 (11)	0.0409
N3	0.24286 (17)	0.11681 (11)	0.37395 (10)	0.0434
C4	0.2341 (2)	−0.14769 (15)	0.43270 (12)	0.0498
C5	0.3558 (3)	−0.28335 (16)	0.42638 (14)	0.0572
C6	0.5646 (3)	−0.30343 (16)	0.37240 (14)	0.0576
C7	0.6637 (2)	−0.18902 (15)	0.32349 (13)	0.0499
C8	0.5408 (2)	−0.05237 (13)	0.33039 (10)	0.0399
C9	0.3294 (2)	−0.03035 (13)	0.38288 (10)	0.0400
C10	0.7913 (2)	0.11818 (16)	0.23991 (12)	0.0488
N11	0.76733 (17)	0.20686 (12)	0.12018 (9)	0.0427
C12	0.9767 (2)	0.24619 (18)	0.07720 (13)	0.0550
C13	0.9562 (3)	0.3378 (2)	−0.04718 (15)	0.0732
O14	0.8918 (2)	0.26370 (17)	−0.12346 (10)	0.0819
C15	0.6858 (3)	0.2283 (3)	−0.08343 (15)	0.0769
C16	0.6983 (3)	0.13381 (18)	0.04037 (13)	0.0556
S17	0.38074 (7)	0.36544 (4)	0.27562 (4)	0.0558
C18	0.1258 (3)	0.42494 (17)	0.35116 (16)	0.0639

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0372 (5)	0.0433 (5)	0.0423 (5)	−0.0112 (4)	−0.0017 (4)	−0.0026 (4)
C2	0.0416 (6)	0.0404 (6)	0.0394 (6)	−0.0092 (5)	−0.0060 (5)	−0.0053 (5)
N3	0.0404 (5)	0.0425 (5)	0.0457 (6)	−0.0097 (4)	−0.0006 (4)	−0.0073 (4)
C4	0.0508 (7)	0.0506 (7)	0.0472 (7)	−0.0193 (6)	0.0000 (5)	−0.0029 (5)

C5	0.0712 (9)	0.0439 (7)	0.0551 (8)	−0.0213 (7)	−0.0070 (7)	0.0004 (6)
C6	0.0679 (9)	0.0407 (7)	0.0585 (8)	−0.0043 (6)	−0.0095 (7)	−0.0061 (6)
C7	0.0469 (7)	0.0476 (7)	0.0496 (7)	−0.0029 (5)	−0.0037 (5)	−0.0077 (5)
C8	0.0400 (6)	0.0414 (6)	0.0359 (5)	−0.0095 (5)	−0.0054 (4)	−0.0028 (4)
C9	0.0402 (6)	0.0417 (6)	0.0365 (5)	−0.0102 (5)	−0.0038 (4)	−0.0042 (4)
C10	0.0376 (6)	0.0604 (8)	0.0453 (7)	−0.0167 (6)	−0.0063 (5)	0.0003 (6)
N11	0.0387 (5)	0.0477 (6)	0.0412 (5)	−0.0144 (4)	−0.0016 (4)	−0.0054 (4)
C12	0.0493 (7)	0.0644 (9)	0.0542 (8)	−0.0273 (7)	0.0019 (6)	−0.0070 (6)
C13	0.0788 (11)	0.0854 (12)	0.0571 (9)	−0.0430 (10)	0.0057 (8)	0.0004 (8)
O14	0.0874 (9)	0.1174 (11)	0.0467 (6)	−0.0465 (8)	0.0116 (6)	−0.0125 (7)
C15	0.0796 (12)	0.1112 (15)	0.0468 (8)	−0.0419 (11)	−0.0058 (8)	−0.0105 (9)
C16	0.0552 (8)	0.0686 (9)	0.0499 (7)	−0.0265 (7)	−0.0015 (6)	−0.0146 (7)
S17	0.0609 (2)	0.04020 (18)	0.0633 (2)	−0.01433 (15)	−0.00462 (17)	−0.00330 (14)
C18	0.0739 (10)	0.0477 (8)	0.0681 (10)	−0.0049 (7)	−0.0021 (8)	−0.0182 (7)

Geometric parameters (Å, °)

H4—C4	0.969	N1—C10	1.4473 (15)
H5—C5	0.976	C2—N3	1.3083 (16)
H6—C6	0.972	C2—S17	1.7426 (13)
H7—C7	0.970	N3—C9	1.3947 (16)
H101—C10	0.994	C4—C5	1.381 (2)
H102—C10	0.982	C4—C9	1.3934 (17)
H121—C12	1.014	C5—C6	1.389 (2)
H122—C12	0.977	C6—C7	1.382 (2)
H131—C13	1.008	C7—C8	1.3935 (18)
H132—C13	1.009	C8—C9	1.3969 (17)
H151—C15	0.998	C10—N11	1.4506 (16)
H152—C15	1.009	N11—C12	1.4612 (16)
H161—C16	0.977	N11—C16	1.4573 (18)
H162—C16	1.001	C12—C13	1.504 (2)
H181—C18	0.983	C13—O14	1.419 (2)
H182—C18	0.959	O14—C15	1.420 (2)
H183—C18	0.982	C15—C16	1.508 (2)
N1—C2	1.3753 (16)	S17—C18	1.7943 (17)
N1—C8	1.3891 (15)		
C2—N1—C8	106.01 (10)	C12—N11—C16	109.85 (11)
C2—N1—C10	127.25 (11)	H121—C12—N11	111.3
C8—N1—C10	126.47 (11)	H121—C12—H122	106.5
N1—C2—N3	113.89 (11)	N11—C12—H122	107.8
N1—C2—S17	120.33 (9)	H121—C12—C13	110.9
N3—C2—S17	125.69 (10)	N11—C12—C13	110.13 (13)
C2—N3—C9	104.42 (10)	H122—C12—C13	110.1
H4—C4—C5	121.8	C12—C13—H132	107.4
H4—C4—C9	120.9	C12—C13—H131	109.4
C5—C4—C9	117.30 (13)	H132—C13—H131	112.1
H5—C5—C4	119.0	C12—C13—O14	110.91 (14)
H5—C5—C6	119.1	H132—C13—O14	109.3
C4—C5—C6	121.91 (13)	H131—C13—O14	107.7

H6—C6—C5	118.9	C13—O14—C15	109.89 (14)
H6—C6—C7	119.2	H152—C15—H151	113.6
C5—C6—C7	121.89 (14)	H152—C15—O14	106.7
H7—C7—C6	121.5	H151—C15—O14	106.8
H7—C7—C8	122.4	H152—C15—C16	109.4
C6—C7—C8	116.07 (13)	H151—C15—C16	109.1
C7—C8—N1	132.06 (12)	O14—C15—C16	111.21 (14)
C7—C8—C9	122.65 (12)	C15—C16—N11	109.90 (13)
N1—C8—C9	105.28 (10)	C15—C16—H162	108.5
C8—C9—N3	110.40 (10)	N11—C16—H162	110.9
C8—C9—C4	120.17 (12)	C15—C16—H161	108.5
N3—C9—C4	129.42 (12)	N11—C16—H161	109.6
N1—C10—H101	108.4	H162—C16—H161	109.4
N1—C10—H102	109.7	C2—S17—C18	98.85 (7)
H101—C10—H102	109.1	S17—C18—H181	107.5
N1—C10—N11	111.62 (10)	S17—C18—H183	109.7
H101—C10—N11	110.4	H181—C18—H183	111.4
H102—C10—N11	107.6	S17—C18—H182	105.0
C10—N11—C12	109.15 (10)	H181—C18—H182	112.1
C10—N11—C16	112.30 (11)	H183—C18—H182	110.9

Hydrogen-bond geometry (Å, °)

D—H⋯*A*	*D*—H	H⋯*A*	*D*⋯*A*	*D*—H⋯*A*
C7—H7⋯O14[i]	0.97	2.62	3.526 (2)	155
C4—H4⋯N3[ii]	0.97	2.64	3.574 (2)	162
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Symmetry codes: (i) −*x*+2, −*y*, −*z*; (ii) −*x*, −*y*, −*z*+1; (iii) *x*+1, *y*, *z*.

Fig. 1