N-[(Methylsulfanyl)methyl]benzamide

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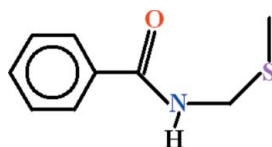Correspondence e-mail: dmntahir_uos@yahoo.com

In the title compound, $C_9H_{11}NOS$, the phenyl ring and formamide unit make a dihedral angle of 23.93 (14)°, whereas the (methylsulfanyl)methyl group is oriented at a dihedral angle of 61.31 (8)° with respect to the phenyl ring. There are intermolecular N—H⋯O hydrogen bonds, forming $C(4)$ chains along the [010] direction. These polymeric chains are linked by C—H⋯O hydrogen bonds to form polymeric sheets in the (110) plane.

Related literature

For crystal structures containing the 1-(methylsulfanyl)-methanamine grouping, see: Siddiqui *et al.* (2008); Noroozi Pesyan *et al.* (2009). For graph-set notation, see: Bernstein *et al.* (1995).

Experimental

Crystal data

$C_9H_{11}NOS$	V = 1916.67 (14) Å³
M_r = 181.25	Z = 8
Orthorhombic, $Pbca$	Mo $K\alpha$ radiation
a = 9.7841 (4) Å	μ = 0.29 mm⁻¹
b = 9.2116 (4) Å	T = 296 K
c = 21.2663 (8) Å	0.26 × 0.20 × 0.18 mm

Data collection

Bruker Kappa APEXII CCD diffractometer	9379 measured reflections
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)	2371 independent reflections
	1722 reflections with $I > 2\sigma(I)$
T_{min} = 0.932, T_{max} = 0.950	R_{int} = 0.022

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041	110 parameters
$wR(F^2)$ = 0.116	H-atom parameters constrained
S = 1.07	$\Delta\rho_{max}$ = 0.18 e Å⁻³
2371 reflections	$\Delta\rho_{min}$ = −0.26 e Å⁻³

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
N1—H1⋯O1ⁱ	0.86	2.02	2.8438 (17)	160
C8—H8B⋯O1ⁱⁱ	0.97	2.53	3.434 (2)	154

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

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2009); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WN2467).

References

Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
Bruker (2005). *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2007). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Noroozi Pesyan, N., Omidkhah, N., Maghsoodi, M. & Patrick, B. O. (2009). *Acta Cryst.* E**65**, o655.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Siddiqui, W. A., Ahmad, S., Siddiqui, H. L., Hussain, R. A. & Parvez, M. (2008). *Acta Cryst.* E**64**, o1897.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.

supplementary materials

N-[(Methylsulfanyl)methyl]benzamide

Muhammad Riaz Khan, Azim Khan, M. Nawaz Tahir, Muhammad Adeel and Saeed Ahmad

Comment

The title compound (Fig. 1) was prepared in an attempt to synthesize a different compound from benzamide and phthalic anhydride in dimethyl sulphoxide.

The crystal structures of 2-((methylsulfanyl)methyl)-1,2-benzisothiazol-3(2*H*)-one 1,1-dioxide (Siddiqui *et al.*, 2008) and 5-(2,6-dimethoxyphenoxy)-2-methylsulfanylmethyl-2*H*-tetrazole (Noroozi Pesyan *et al.*, 2009) have been published; these contain the 1-(methylsulfanyl)methanamine grouping.

Let A, B, C denote the planes defined by the phenyl ring (C1–C6), the formamide unit (O1/C7/N1) and the (methyl-sulfanyl)methane grouping (C8/S1/C9), respectively. The dihedral angles between A/B, A/C and B/C are 23.93 (14)°, 61.31 (8)° and 67.92 (13)°, respectively.

There are intermolecular N—H···O hydrogen bonds (Table 1, Fig. 2), forming C(4) chains (Bernstein *et al.*, 1995) along the [010] direction. These polymeric chains are linked by C—H···O hydrogen bonds (Table 1, Fig. 2) to form polymeric sheets in the (110) plane.

Experimental

The title compound was prepared by adding a solution of benzamide (0.1 g, 0.078 mmol) in 3 ml of dimethyl sulphoxide (DMSO) to a solution of phthalic anhydride (0.1 g, 0.078 mmol) in DMSO (3 ml). The reaction mixture was heated to 353 K for 6 h. The organic and aqueous layers were separated and the latter was extracted with chloroform (3×15 ml). The organic layer was concentrated *in vacuo* and the residue was purified by chromatography (silica gel, EtOAc/hexane= 1:4). The title compound was obtained as a colorless crystalline solid. Yield = 0.14 g, 70%, m.p = 365 K. Crystallization from a saturated chloroform solution at ambient temperature gave colourless prisms.

Refinement

The H-atoms were positioned geometrically (N—H = 0.86 Å, C—H = 0.93–0.97 Å) and refined as riding with U_{iso}(H) = xU_{eq}(C, N), where x = 1.5 for methyl groups and x = 1.2 for all other H-atoms.

Computing details

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT* (Bruker, 2007); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2009); software used to prepare material for publication: *WinGX* (Farrugia, 1999) and *PLATON* (Spek, 2009).
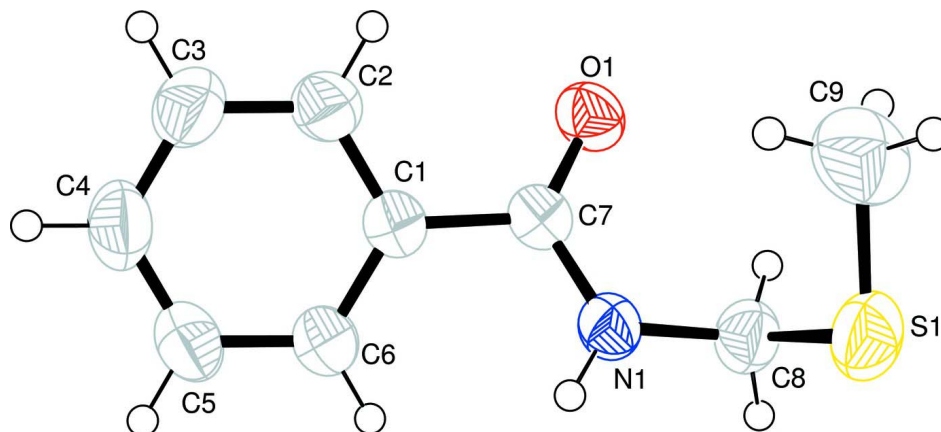
Figure 1

View of the title compound, with displacement ellipsoids drawn at the 50% probability level. H-atoms are shown as small circles of arbitrary radius.
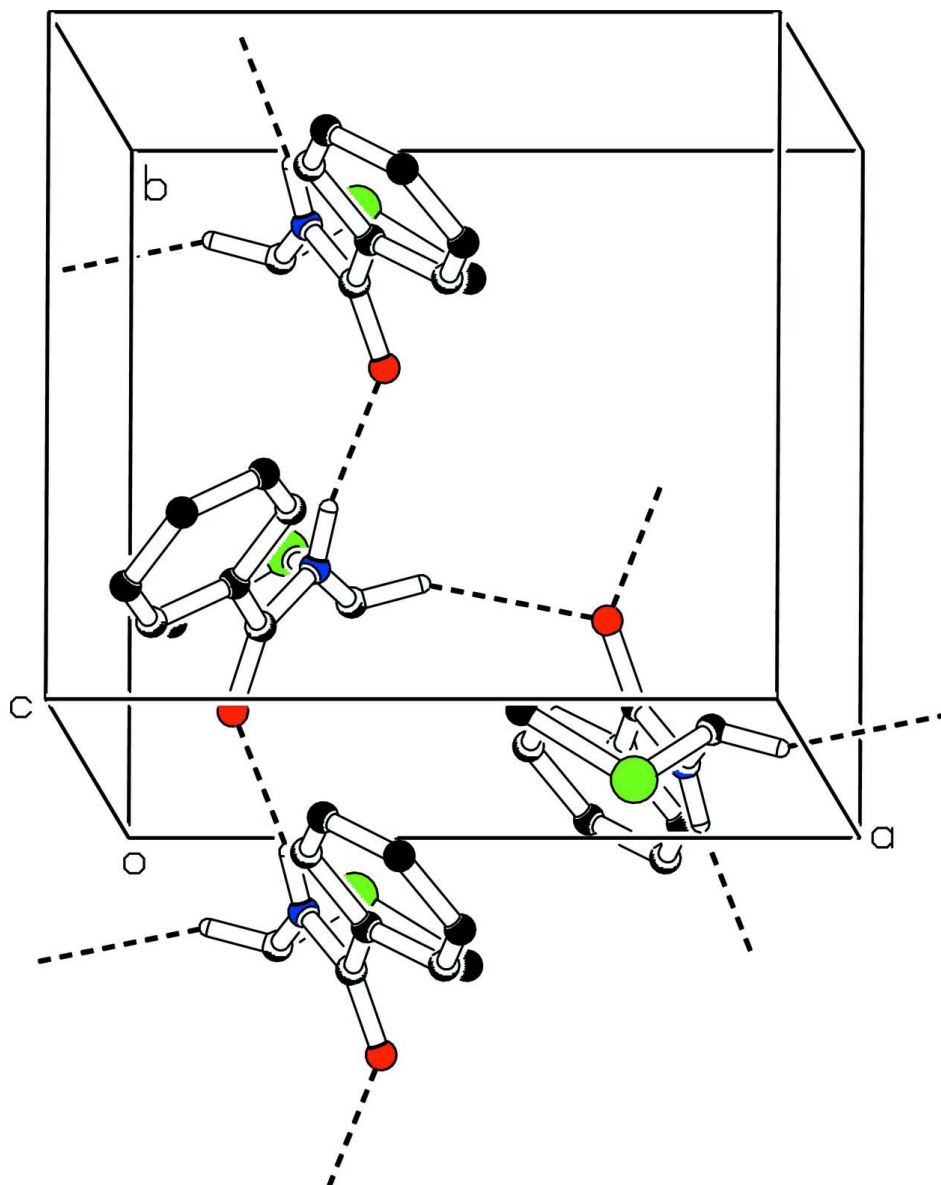
Figure 2

Packing diagram of the title compound (*PLATON*: Spek, 2009) showing that molecules form one dimensional polymeric chains along [010] and are interlinked. H atoms not involved in hydrogen bonding have been omitted for clarity.

N-[(methylsulfanyl)methyl]benzamide

Crystal data

$C_9H_{11}NOS$	$F(000) = 768$
$M_r = 181.25$	$D_x = 1.256$ Mg m^{-3}
Orthorhombic, *Pbca*	Mo *Kα* radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2ac 2ab	Cell parameters from 1722 reflections
$a = 9.7841$ (4) Å	$\theta = 1.9–28.3°$
$b = 9.2116$ (4) Å	$\mu = 0.29$ mm^{-1}
$c = 21.2663$ (8) Å	$T = 296$ K
$V = 1916.67$ (14) Å^3	Prism, colorless
$Z = 8$	$0.26 \times 0.20 \times 0.18$ mm

Data collection

Bruker Kappa APEXII CCD diffractometer	9379 measured reflections
Radiation source: fine-focus sealed tube	2371 independent reflections
Graphite monochromator	1722 reflections with $I > 2\sigma(I)$
Detector resolution: 7.50 pixels mm^{-1}	$R_{int} = 0.022$
ω scans	$\theta_{max} = 28.3°$, $\theta_{min} = 1.9°$
Absorption correction: multi-scan	$h = -12 \rightarrow 12$
(*SADABS*; Bruker, 2005)	$k = -12 \rightarrow 12$
$T_{min} = 0.932$, $T_{max} = 0.950$	$l = -27 \rightarrow 28$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.041$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.116$	
$S = 1.07$	H-atom parameters constrained
2371 reflections	$w = 1/[\sigma^2(F_o^2) + (0.0531P)^2 + 0.2845P]$
110 parameters	where $P = (F_o^2 + 2F_c^2)/3$
0 restraints	$(\Delta/\sigma)_{max} < 0.001$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{max} = 0.18$ e Å^{-3}
	$\Delta\rho_{min} = -0.26$ e Å^{-3}

Special details

Geometry. Bond distances, angles *etc*. have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	$U_{iso}*/U_{eq}$
S1	0.20155 (6)	0.43391 (6)	−0.09248 (2)	0.0717 (2)
O1	0.14747 (12)	0.17375 (11)	0.05060 (5)	0.0537 (4)
N1	0.25653 (14)	0.38548 (15)	0.03218 (5)	0.0493 (4)
C1	0.15800 (14)	0.34302 (14)	0.13470 (7)	0.0427 (4)
C2	0.04865 (18)	0.28086 (18)	0.16623 (8)	0.0562 (5)
C3	0.0203 (2)	0.3207 (2)	0.22745 (9)	0.0710 (7)
C4	0.1003 (2)	0.4214 (2)	0.25775 (9)	0.0709 (7)
C5	0.2082 (2)	0.4838 (2)	0.22691 (8)	0.0648 (6)
C6	0.23778 (18)	0.44502 (17)	0.16551 (7)	0.0525 (5)
C7	0.18659 (14)	0.29393 (15)	0.06896 (7)	0.0424 (4)
C8	0.29981 (18)	0.3490 (2)	−0.03071 (7)	0.0559 (6)
C9	0.0467 (2)	0.3318 (3)	−0.08796 (11)	0.0905 (9)
H1	0.27659	0.46996	0.04670	0.0592*
H2	−0.00569	0.21226	0.14612	0.0674*
H3	−0.05341	0.27902	0.24832	0.0852*
H4	0.08116	0.44708	0.29912	0.0851*
H5	0.26188	0.55253	0.24731	0.0778*

H6	0.31140	0.48753	0.14487	0.0630*
H8A	0.29434	0.24449	−0.03582	0.0670*
H8B	0.39487	0.37666	−0.03557	0.0670*
H9A	0.00311	0.34980	−0.04829	0.1358*
H9B	0.06706	0.23019	−0.09169	0.1358*
H9C	−0.01321	0.36045	−0.12147	0.1358*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0957 (4)	0.0699 (4)	0.0496 (3)	−0.0010 (3)	−0.0003 (2)	0.0110 (2)
O1	0.0662 (7)	0.0395 (6)	0.0553 (6)	−0.0005 (5)	−0.0022 (5)	−0.0059 (5)
N1	0.0605 (8)	0.0426 (7)	0.0448 (7)	−0.0021 (6)	0.0027 (6)	−0.0046 (5)
C1	0.0465 (8)	0.0375 (7)	0.0440 (7)	0.0072 (6)	−0.0033 (6)	0.0001 (6)
C2	0.0588 (10)	0.0498 (9)	0.0600 (9)	−0.0024 (7)	0.0056 (8)	−0.0074 (7)
C3	0.0765 (13)	0.0686 (11)	0.0679 (12)	−0.0042 (10)	0.0239 (9)	−0.0067 (9)
C4	0.0883 (14)	0.0736 (12)	0.0509 (10)	0.0057 (11)	0.0117 (9)	−0.0129 (9)
C5	0.0733 (12)	0.0685 (11)	0.0526 (10)	−0.0033 (9)	−0.0067 (8)	−0.0134 (9)
C6	0.0541 (9)	0.0566 (9)	0.0468 (8)	−0.0028 (7)	−0.0045 (7)	−0.0024 (7)
C7	0.0445 (8)	0.0372 (7)	0.0454 (8)	0.0059 (6)	−0.0057 (6)	−0.0001 (6)
C8	0.0568 (10)	0.0632 (10)	0.0476 (9)	0.0011 (8)	0.0064 (7)	−0.0031 (8)
C9	0.0826 (15)	0.0997 (17)	0.0893 (15)	0.0017 (13)	−0.0291 (12)	0.0092 (12)

Geometric parameters (Å, º)

S1—C8	1.8060 (17)	C5—C6	1.384 (2)
S1—C9	1.786 (2)	C2—H2	0.9300
O1—C7	1.2347 (17)	C3—H3	0.9300
N1—C7	1.3384 (19)	C4—H4	0.9300
N1—C8	1.4426 (19)	C5—H5	0.9300
N1—H1	0.8600	C6—H6	0.9300
C1—C6	1.386 (2)	C8—H8A	0.9700
C1—C7	1.496 (2)	C8—H8B	0.9700
C1—C2	1.386 (2)	C9—H9A	0.9600
C2—C3	1.381 (3)	C9—H9B	0.9600
C3—C4	1.374 (3)	C9—H9C	0.9600
C4—C5	1.369 (3)		
C8—S1—C9	100.62 (10)	C4—C3—H3	120.00
C7—N1—C8	123.03 (14)	C3—C4—H4	120.00
C8—N1—H1	118.00	C5—C4—H4	120.00
C7—N1—H1	118.00	C4—C5—H5	120.00
C2—C1—C7	118.13 (13)	C6—C5—H5	120.00
C2—C1—C6	119.07 (14)	C1—C6—H6	120.00
C6—C1—C7	122.78 (13)	C5—C6—H6	120.00
C1—C2—C3	120.08 (16)	S1—C8—H8A	109.00
C2—C3—C4	120.47 (18)	S1—C8—H8B	109.00
C3—C4—C5	119.86 (18)	N1—C8—H8A	109.00
C4—C5—C6	120.31 (17)	N1—C8—H8B	109.00
C1—C6—C5	120.20 (16)	H8A—C8—H8B	108.00

O1—C7—N1	122.59 (14)	S1—C9—H9A	109.00
O1—C7—C1	120.58 (13)	S1—C9—H9B	109.00
N1—C7—C1	116.83 (12)	S1—C9—H9C	109.00
S1—C8—N1	114.66 (12)	H9A—C9—H9B	109.00
C1—C2—H2	120.00	H9A—C9—H9C	109.00
C3—C2—H2	120.00	H9B—C9—H9C	109.00
C2—C3—H3	120.00		
C9—S1—C8—N1	−73.54 (15)	C2—C1—C7—O1	−23.5 (2)
C8—N1—C7—O1	−3.9 (2)	C2—C1—C7—N1	157.05 (14)
C8—N1—C7—C1	175.55 (13)	C6—C1—C7—O1	155.07 (15)
C7—N1—C8—S1	105.09 (16)	C6—C1—C7—N1	−24.4 (2)
C6—C1—C2—C3	0.0 (2)	C1—C2—C3—C4	−0.4 (3)
C7—C1—C2—C3	178.63 (15)	C2—C3—C4—C5	0.6 (3)
C2—C1—C6—C5	0.0 (2)	C3—C4—C5—C6	−0.5 (3)
C7—C1—C6—C5	−178.51 (15)	C4—C5—C6—C1	0.2 (3)

Hydrogen-bond geometry (Å, º)

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O1[i]	0.86	2.02	2.8438 (17)	160
C8—H8*B*···O1[ii]	0.97	2.53	3.434 (2)	154

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