Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

(*E*)-*N*′-[4-(Methylsulfanyl)benzylidene]-furan-2-carbohydrazide monohydrate

Yu-Feng Li[a] and Fang-Fang Jian[b]*

[a]Microscale Science Institute, Department of Chemistry and Chemical Engineering, Weifang University, Weifang 261061, People's Republic of China, and [b]Microscale Science Institute, Weifang University, Weifang 261061, People's Republic of China Correspondence e-mail: liyufeng8111@163.com

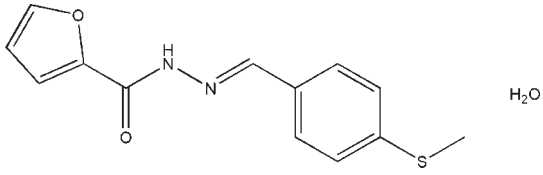
Received 14 July 2010; accepted 17 July 2010

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.005 Å; R factor = 0.052; wR factor = 0.172; data-to-parameter ratio = 14.1.

In the title compound, $C_{13}H_{12}N_2O_2S\cdot H_2O$, the dihedral angle between the aromatic rings is 35.34 (19)° and an intramolecular N—H···O hydrogen bond generates an S(5) ring. In the crystal, molecules are linked by N—H···O and O—H···O hydrogen bonds, generating (001) sheets.

Related literature

For a related structure, see: Li & Jian (2010).



Experimental

Crystal data

$C_{13}H_{12}N_2O_2S\cdot H_2O$
M_r = 278.32
Monoclinic, $P2_1/c$
a = 4.7065 (9) Å
b = 12.142 (2) Å
c = 23.979 (5) Å
β = 91.96 (3)°
V = 1369.6 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.24 mm^{-1}
T = 293 K
0.22 × 0.20 × 0.18 mm

Data collection

Bruker SMART CCD diffractometer
10766 measured reflections
2536 independent reflections
1095 reflections with $I > 2\sigma(I)$
R_{int} = 0.093

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.172
S = 0.81
2536 reflections
180 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.25 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1B···O2	0.86	2.37	2.713 (3)	104
N1—H1B···O3^{i}	0.86	2.03	2.864 (4)	162
O3—H3B···O1^{ii}	0.87 (5)	2.03 (6)	2.878 (4)	165 (4)
O3—H3C···O1	0.76 (8)	2.11 (8)	2.800 (4)	152 (8)

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

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5554).

References

Bruker (1997). *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Li, Y.-F. & Jian, F.-F. (2010). *Acta Cryst.* E**66**, o2061.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supporting information

Acta Cryst. (2010). E**66**, o2157 [https://doi.org/10.1107/S1600536810028655]

(*E*)-*N*′-[4-(Methylsulfanyl)benzylidene]furan-2-carbohydrazide monohydrate

Yu-Feng Li and Fang-Fang Jian

S1. Experimental

A mixture of 4-(methylthio)benzaldehyde (0.1 mol), and furan-2-carbohydrazide (0.1 mol) was stirred in refluxing ethanol (20 ml) for 2 h to afford the title compound (0.090 mol, yield 90%). Colourless blocks of the title compound were obtained by recrystallization from ethanol at room temperature.

S2. Refinement

H atoms were fixed geometrically and allowed to ride on their attached atoms, with C—H = 0.97 Å, and U_{iso} = 1.2–1.5U_{eq}.

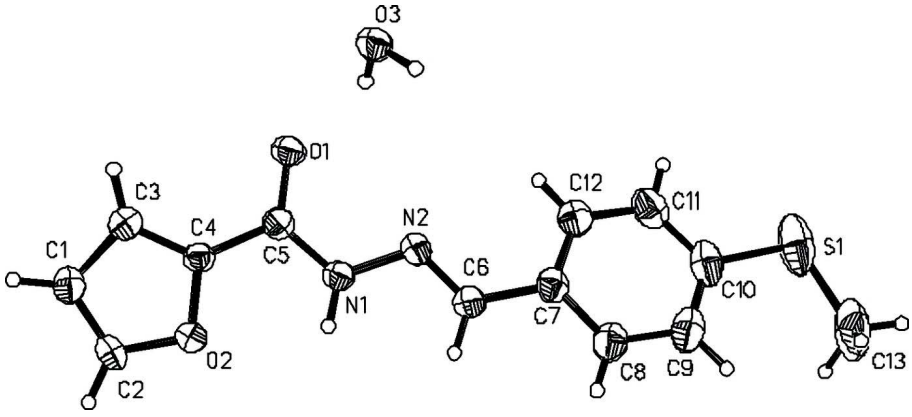


Figure 1

The structure of the title compound showing 30% probability displacement ellipsoids.

(*E*)-*N*′-[4-(Methylsulfanyl)benzylidene]furan-2-carbohydrazide monohydrate

Crystal data

$C_{13}H_{14}N_2O_3S$
M_r = 278.32
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
a = 4.7065 (9) Å
b = 12.142 (2) Å
c = 23.979 (5) Å
β = 91.96 (3)°
V = 1369.6 (5) Å^3
Z = 4
$F(000)$ = 584
D_x = 1.350 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 1095 reflections
θ = 3.1–25.5°
μ = 0.24 mm^{-1}
T = 293 K
Block, colorless
0.22 × 0.20 × 0.18 mm

Data collection

Bruker SMART CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
phi and ω scans
10766 measured reflections
2536 independent reflections
1095 reflections with $I > 2\sigma(I)$
$R_{int} = 0.093$
$\theta_{max} = 25.5°$, $\theta_{min} = 3.1°$
$h = -5 \rightarrow 5$
$k = -14 \rightarrow 14$
$l = -27 \rightarrow 29$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.052$
$wR(F^2) = 0.172$
$S = 0.81$
2536 reflections
180 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.1P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.18$ e Å^{-3}
$\Delta\rho_{min} = -0.25$ 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.

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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
C5	0.1858 (6)	0.6495 (3)	0.26977 (12)	0.0476 (8)
N2	0.5006 (6)	0.6551 (2)	0.19538 (11)	0.0543 (7)
C4	−0.0079 (6)	0.5827 (3)	0.30142 (12)	0.0469 (8)
O2	−0.0668 (5)	0.47801 (18)	0.28399 (9)	0.0576 (7)
C6	0.6577 (7)	0.5956 (3)	0.16494 (13)	0.0544 (9)
H6A	0.6664	0.5200	0.1709	0.065*
C1	−0.3067 (8)	0.5097 (3)	0.35996 (15)	0.0695 (11)
H1A	−0.4258	0.5003	0.3898	0.083*
C2	−0.2512 (7)	0.4347 (3)	0.32055 (14)	0.0647 (10)
H2B	−0.3273	0.3641	0.3186	0.078*
C8	1.0074 (7)	0.5792 (3)	0.09165 (14)	0.0686 (11)
H8A	1.0343	0.5060	0.1020	0.082*
C3	−0.1508 (8)	0.6049 (3)	0.34769 (14)	0.0620 (10)
H3A	−0.1473	0.6705	0.3677	0.074*
O1	0.2107 (5)	0.7491 (2)	0.27923 (10)	0.0628 (7)
C7	0.8238 (7)	0.6447 (3)	0.12095 (12)	0.0541 (9)

N1	0.3328 (5)	0.5959 (2)	0.23110 (10)	0.0512 (7)
H1B	0.3227	0.5254	0.2285	0.061*
C12	0.7927 (8)	0.7535 (3)	0.10515 (17)	0.0740 (11)
H12A	0.6732	0.7994	0.1247	0.089*
C11	0.9370 (9)	0.7947 (4)	0.06065 (17)	0.0823 (12)
H11A	0.9137	0.8682	0.0507	0.099*
C10	1.1155 (8)	0.7288 (4)	0.03055 (16)	0.0765 (12)
C9	1.1526 (8)	0.6220 (4)	0.04680 (16)	0.0809 (13)
H9A	1.2764	0.5771	0.0277	0.097*
O3	0.7049 (8)	0.8706 (2)	0.30372 (13)	0.0682 (8)
S1	1.2735 (3)	0.79024 (15)	−0.02691 (5)	0.1234 (7)
C13	1.4598 (13)	0.6810 (6)	−0.0588 (2)	0.161 (3)
H13A	1.5531	0.7084	−0.0910	0.241*
H13B	1.3280	0.6242	−0.0700	0.241*
H13C	1.5989	0.6515	−0.0327	0.241*
H3B	0.857 (12)	0.841 (4)	0.2908 (19)	0.12 (2)*
H3C	0.602 (17)	0.833 (7)	0.288 (3)	0.21 (4)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C5	0.0404 (18)	0.047 (2)	0.056 (2)	0.0052 (17)	0.0024 (15)	0.0034 (17)
N2	0.0518 (16)	0.0511 (18)	0.0606 (17)	−0.0064 (15)	0.0103 (14)	0.0021 (14)
C4	0.0471 (18)	0.041 (2)	0.0529 (18)	0.0025 (16)	0.0030 (15)	−0.0020 (15)
O2	0.0659 (14)	0.0447 (14)	0.0631 (14)	−0.0051 (12)	0.0166 (12)	−0.0027 (11)
C6	0.0518 (19)	0.052 (2)	0.059 (2)	−0.0016 (17)	0.0055 (17)	−0.0022 (17)
C1	0.082 (3)	0.063 (3)	0.065 (2)	0.002 (2)	0.023 (2)	0.007 (2)
C2	0.077 (3)	0.053 (2)	0.065 (2)	−0.011 (2)	0.021 (2)	0.0118 (19)
C8	0.060 (2)	0.080 (3)	0.066 (2)	−0.004 (2)	0.0084 (19)	−0.004 (2)
C3	0.069 (2)	0.055 (2)	0.062 (2)	0.0013 (19)	0.0133 (19)	−0.0036 (18)
O1	0.0614 (15)	0.0406 (15)	0.0878 (17)	−0.0035 (12)	0.0195 (13)	−0.0062 (12)
C7	0.0426 (18)	0.065 (3)	0.055 (2)	−0.0044 (18)	−0.0002 (16)	−0.0022 (18)
N1	0.0509 (16)	0.0406 (17)	0.0628 (16)	−0.0010 (13)	0.0091 (14)	0.0006 (13)
C12	0.073 (3)	0.066 (3)	0.083 (3)	−0.001 (2)	0.019 (2)	0.004 (2)
C11	0.083 (3)	0.076 (3)	0.089 (3)	−0.011 (2)	0.012 (2)	0.020 (2)
C10	0.059 (2)	0.109 (4)	0.061 (2)	−0.019 (3)	0.0024 (19)	0.009 (2)
C9	0.067 (3)	0.109 (4)	0.067 (3)	−0.004 (3)	0.017 (2)	−0.005 (3)
O3	0.0683 (18)	0.0448 (17)	0.0929 (19)	−0.0012 (15)	0.0231 (17)	−0.0016 (14)
S1	0.1001 (10)	0.1929 (17)	0.0779 (8)	−0.0324 (10)	0.0135 (7)	0.0432 (9)
C13	0.158 (5)	0.237 (8)	0.092 (4)	−0.087 (5)	0.068 (4)	−0.047 (4)

Geometric parameters (Å, º)

C5—O1	1.235 (4)	C3—H3A	0.9300
C5—N1	1.344 (4)	C7—C12	1.380 (5)
C5—C4	1.454 (4)	N1—H1B	0.8600
N2—C6	1.280 (4)	C12—C11	1.378 (5)
N2—N1	1.385 (3)	C12—H12A	0.9300

C4—C3	1.344 (4)	C11—C10	1.381 (6)
C4—O2	1.364 (4)	C11—H11A	0.9300
O2—C2	1.360 (4)	C10—C9	1.364 (6)
C6—C7	1.461 (4)	C10—S1	1.754 (4)
C6—H6A	0.9300	C9—H9A	0.9300
C1—C2	1.344 (5)	O3—H3B	0.87 (5)
C1—C3	1.406 (5)	O3—H3C	0.75 (8)
C1—H1A	0.9300	S1—C13	1.777 (6)
C2—H2B	0.9300	C13—H13A	0.9600
C8—C7	1.384 (4)	C13—H13B	0.9600
C8—C9	1.394 (5)	C13—H13C	0.9600
C8—H8A	0.9300		

O1—C5—N1	123.6 (3)	C8—C7—C6	119.4 (3)
O1—C5—C4	120.5 (3)	C5—N1—N2	119.6 (3)
N1—C5—C4	115.9 (3)	C5—N1—H1B	120.2
C6—N2—N1	114.4 (3)	N2—N1—H1B	120.2
C3—C4—O2	109.7 (3)	C11—C12—C7	120.6 (4)
C3—C4—C5	131.2 (3)	C11—C12—H12A	119.7
O2—C4—C5	119.0 (3)	C7—C12—H12A	119.7
C2—O2—C4	106.9 (2)	C12—C11—C10	121.3 (4)
N2—C6—C7	121.0 (3)	C12—C11—H11A	119.4
N2—C6—H6A	119.5	C10—C11—H11A	119.4
C7—C6—H6A	119.5	C9—C10—C11	118.4 (4)
C2—C1—C3	107.1 (3)	C9—C10—S1	125.1 (4)
C2—C1—H1A	126.4	C11—C10—S1	116.5 (4)
C3—C1—H1A	126.4	C10—C9—C8	120.9 (4)
C1—C2—O2	109.6 (3)	C10—C9—H9A	119.6
C1—C2—H2B	125.2	C8—C9—H9A	119.6
O2—C2—H2B	125.2	H3B—O3—H3C	96 (6)
C7—C8—C9	120.6 (4)	C10—S1—C13	104.4 (3)
C7—C8—H8A	119.7	S1—C13—H13A	109.5
C9—C8—H8A	119.7	S1—C13—H13B	109.5
C4—C3—C1	106.7 (3)	H13A—C13—H13B	109.5
C4—C3—H3A	126.7	S1—C13—H13C	109.5
C1—C3—H3A	126.7	H13A—C13—H13C	109.5
C12—C7—C8	118.2 (3)	H13B—C13—H13C	109.5
C12—C7—C6	122.3 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*B*···O2	0.86	2.37	2.713 (3)	104
N1—H1*B*···O3^{i}	0.86	2.03	2.864 (4)	162
O3—H3*B*···O1^{ii}	0.87 (5)	2.03 (6)	2.878 (4)	165 (4)
O3—H3*C*···O1	0.76 (8)	2.11 (8)	2.800 (4)	152 (8)

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