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Crystal structure of (*E*)-*N*′-(3,4-difluorobenzylidene)-4-methylbenzenesulfonohydrazide

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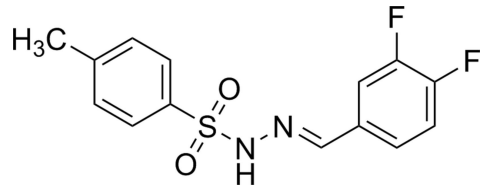
In the title compound, $C_{14}H_{12}F_2N_2O_2S$, the dihedral angle between the aromatic rings is 70.23 (8)° and the S—N—N=C torsion angle is 172.11 (11)°. In the crystal, N—H···O hydrogen bonds link the molecules into [100] *C*(4) chains, with adjacent molecules in the chain related by translational symmetry. The chains are linked by weak C—H···F and C—H···O interactions, thereby forming a three-dimensional network.

Keywords: crystal structure; sulfonylhydrazone; hydrogen bonding; biological activity.

CCDC reference: 1040450

1. Related literature

For the biological activities of sulfonamides and sulfonylhydrazones, see: El-Sayed *et al.* (2011); de Oliveira *et al.* (2011); Zhao *et al.* (2011).



2. Experimental

2.1. Crystal data

$C_{14}H_{12}F_2N_2O_2S$
M_r = 310.32
Monoclinic, $P2_1/c$
a = 5.3161 (11) Å
b = 15.388 (3) Å
c = 17.293 (4) Å
β = 97.44 (3)°
V = 1402.8 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.26 mm^{-1}
T = 173 K
0.20 × 0.18 × 0.12 mm

2.2. Data collection

Rigaku Saturn diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2009)
T_{min} = 0.950, T_{max} = 0.970
14131 measured reflections
3328 independent reflections
2729 reflections with $I > 2\sigma(I)$
R_{int} = 0.047

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.116
S = 1.04
3328 reflections
196 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.39 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2···O1^{i}	0.89 (1)	2.09 (1)	2.9747 (18)	174 (2)
C1—H1···F1^{ii}	0.95	2.47	3.345 (2)	153
C4—H4···O2^{iii}	0.95	2.57	3.473 (2)	159

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

Data collection: *CrystalClear* (Rigaku, 2009); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; 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*.

Acknowledgements

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Supporting information for this paper is available from the IUCr electronic archives (Reference: HB7491).

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supporting information

Acta Cryst. (2015). E**71**, o761 [doi:10.1107/S2056989015016205]

Crystal structure of (*E*)-*N*′-(3,4-difluorobenzylidene)-4-methylbenzenesulfonohydrazide

Yeming Wang and Hong Yan

S1. Experimental

S1.1. Synthesis and crystallization

p-Tosylhydrazine (0.372 g, 2 mmol) was added to a 50 ml refluxing ethanolic solution of 3,4-difluorobenzaldehyde (0.284 g, 2 mmol). The mixture was stirred for 3 h. After cooling, the colorless crystalline solid was isolated by filtration, washed with cold ethanol, and recrystallized from ethanol as colourless prisms.

S1.2. Refinement

S2. Results and discussion

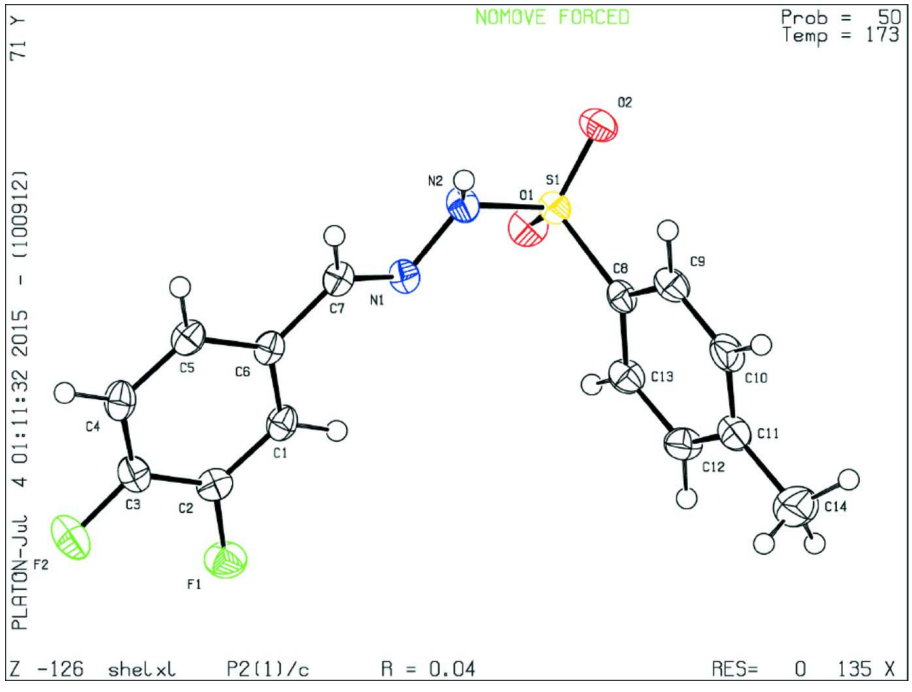

Figure 1

Displacement ellipsoid plot (50% probability level) of the title compound.

(*E*)-*N*′-(3,4-Difluorobenzylidene)-4-methylbenzenesulfonohydrazide

Crystal data

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Monoclinic, $P2_1/c$
$a = 5.3161$ (11) Å
$b = 15.388$ (3) Å
$c = 17.293$ (4) Å
$\beta = 97.44$ (3)°
$V = 1402.8$ (5) Å^3
$Z = 4$
$F(000) = 640$
$D_x = 1.469$ Mg m^{-3}
Melting point: 426 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3967 reflections
$\theta = 1.8$–27.9°
$\mu = 0.26$ mm^{-1}
$T = 173$ K
Prism, colourless
0.20 × 0.18 × 0.12 mm

Data collection

Rigaku Saturn diffractometer
Radiation source: rotating anode
Confocal monochromator
ω scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2009)
$T_{min} = 0.950$, $T_{max} = 0.970$
14131 measured reflections
3328 independent reflections
2729 reflections with $I > 2\sigma(I)$
$R_{int} = 0.047$
$\theta_{max} = 27.9°$, $\theta_{min} = 1.8°$
$h = -6 \rightarrow 6$
$k = -20 \rightarrow 19$
$l = -22 \rightarrow 22$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.116$
$S = 1.04$
3328 reflections
196 parameters
1 restraint
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
$w = 1/[\sigma^2(F_o^2) + (0.0715P)^2 + 0.0398P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.27$ e Å^{-3}
$\Delta\rho_{min} = -0.39$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.057 (4)

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
S1	0.17639 (7)	0.83591 (2)	0.34262 (2)	0.02258 (15)
F1	0.15725 (18)	0.45775 (6)	0.58632 (6)	0.0354 (3)
F2	0.52921 (19)	0.43477 (7)	0.70237 (6)	0.0403 (3)

O1	−0.0478 (2)	0.83384 (7)	0.38043 (6)	0.0300 (3)
O2	0.2561 (2)	0.91533 (7)	0.31005 (6)	0.0308 (3)
N1	0.3852 (2)	0.73655 (8)	0.45229 (7)	0.0243 (3)
N2	0.4159 (2)	0.81054 (9)	0.40836 (7)	0.0254 (3)
C1	0.3568 (3)	0.58214 (10)	0.54189 (9)	0.0245 (3)
H1	0.2203	0.5908	0.5014	0.029*
C2	0.3516 (3)	0.51491 (10)	0.59364 (9)	0.0245 (3)
C3	0.5465 (3)	0.50210 (10)	0.65353 (9)	0.0268 (3)
C4	0.7535 (3)	0.55625 (10)	0.66269 (9)	0.0286 (3)
H4	0.8868	0.5476	0.7042	0.034*
C5	0.7641 (3)	0.62404 (10)	0.60986 (9)	0.0257 (3)
H5	0.9075	0.6614	0.6148	0.031*
C6	0.5666 (3)	0.63768 (9)	0.54969 (8)	0.0218 (3)
C7	0.5820 (3)	0.71137 (10)	0.49678 (8)	0.0233 (3)
H7	0.7391	0.7405	0.4956	0.028*
C8	0.1461 (3)	0.75599 (9)	0.26968 (8)	0.0224 (3)
C9	0.3114 (3)	0.75831 (10)	0.21365 (9)	0.0274 (3)
H9	0.4410	0.8011	0.2162	0.033*
C10	0.2849 (3)	0.69783 (11)	0.15446 (9)	0.0323 (4)
H10	0.3983	0.6992	0.1163	0.039*
C11	0.0951 (4)	0.63458 (10)	0.14931 (10)	0.0331 (4)
C12	−0.0658 (3)	0.63306 (11)	0.20640 (10)	0.0315 (4)
H12	−0.1949	0.5901	0.2041	0.038*
C13	−0.0418 (3)	0.69311 (10)	0.26667 (9)	0.0270 (3)
H13	−0.1528	0.6912	0.3055	0.032*
C14	0.0698 (5)	0.56939 (13)	0.08375 (12)	0.0565 (6)
H14A	0.1822	0.5199	0.0982	0.085*
H14B	0.1170	0.5968	0.0365	0.085*
H14C	−0.1062	0.5491	0.0739	0.085*
H2	0.573 (2)	0.8215 (12)	0.3993 (12)	0.043 (6)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0188 (2)	0.0234 (2)	0.0255 (2)	0.00121 (13)	0.00278 (14)	0.00296 (13)
F1	0.0271 (5)	0.0342 (6)	0.0442 (6)	−0.0066 (4)	0.0022 (4)	0.0015 (4)
F2	0.0390 (6)	0.0407 (6)	0.0410 (6)	0.0035 (4)	0.0041 (5)	0.0171 (4)
O1	0.0187 (6)	0.0373 (7)	0.0352 (6)	0.0019 (4)	0.0076 (5)	−0.0028 (5)
O2	0.0332 (7)	0.0237 (6)	0.0354 (6)	−0.0007 (5)	0.0037 (5)	0.0055 (4)
N1	0.0255 (7)	0.0257 (6)	0.0219 (6)	0.0010 (5)	0.0036 (5)	0.0019 (5)
N2	0.0179 (7)	0.0307 (7)	0.0274 (7)	−0.0027 (5)	0.0021 (5)	0.0064 (5)
C1	0.0213 (8)	0.0289 (8)	0.0226 (7)	0.0036 (6)	−0.0003 (6)	−0.0028 (6)
C2	0.0194 (8)	0.0261 (8)	0.0288 (8)	−0.0010 (6)	0.0054 (6)	−0.0034 (6)
C3	0.0288 (8)	0.0275 (8)	0.0250 (7)	0.0057 (6)	0.0068 (6)	0.0033 (6)
C4	0.0246 (8)	0.0355 (9)	0.0247 (7)	0.0060 (6)	−0.0013 (6)	0.0008 (6)
C5	0.0186 (7)	0.0305 (8)	0.0272 (7)	0.0005 (6)	0.0005 (6)	−0.0031 (6)
C6	0.0198 (8)	0.0255 (7)	0.0203 (7)	0.0041 (6)	0.0030 (6)	−0.0032 (5)
C7	0.0202 (8)	0.0283 (8)	0.0217 (7)	−0.0001 (6)	0.0036 (6)	−0.0022 (6)

C8	0.0204 (8)	0.0237 (7)	0.0228 (7)	0.0030 (6)	0.0010 (6)	0.0053 (5)
C9	0.0262 (9)	0.0268 (8)	0.0303 (8)	−0.0011 (6)	0.0075 (6)	0.0064 (6)
C10	0.0424 (10)	0.0314 (9)	0.0253 (8)	0.0008 (7)	0.0126 (7)	0.0057 (6)
C11	0.0471 (11)	0.0255 (8)	0.0263 (8)	0.0016 (7)	0.0036 (7)	0.0040 (6)
C12	0.0333 (9)	0.0257 (8)	0.0350 (9)	−0.0049 (7)	0.0028 (7)	0.0017 (6)
C13	0.0228 (8)	0.0281 (8)	0.0310 (8)	−0.0020 (6)	0.0067 (6)	0.0041 (6)
C14	0.0924 (18)	0.0407 (11)	0.0384 (11)	−0.0107 (11)	0.0163 (11)	−0.0076 (8)

Geometric parameters (Å, º)

S1—O1	1.4319 (12)	C5—H5	0.9500
S1—O2	1.4324 (11)	C6—C7	1.466 (2)
S1—N2	1.6404 (14)	C7—H7	0.9500
S1—C8	1.7542 (15)	C8—C13	1.387 (2)
F1—C2	1.3502 (18)	C8—C9	1.390 (2)
F2—C3	1.3473 (18)	C9—C10	1.377 (2)
N1—C7	1.2762 (19)	C9—H9	0.9500
N1—N2	1.3901 (17)	C10—C11	1.396 (3)
N2—H2	0.888 (9)	C10—H10	0.9500
C1—C2	1.371 (2)	C11—C12	1.388 (2)
C1—C6	1.398 (2)	C11—C14	1.507 (2)
C1—H1	0.9500	C12—C13	1.386 (2)
C2—C3	1.381 (2)	C12—H12	0.9500
C3—C4	1.373 (2)	C13—H13	0.9500
C4—C5	1.393 (2)	C14—H14A	0.9800
C4—H4	0.9500	C14—H14B	0.9800
C5—C6	1.395 (2)	C14—H14C	0.9800
O1—S1—O2	120.29 (6)	N1—C7—C6	120.22 (13)
O1—S1—N2	107.18 (7)	N1—C7—H7	119.9
O2—S1—N2	103.48 (7)	C6—C7—H7	119.9
O1—S1—C8	107.96 (7)	C13—C8—C9	120.60 (14)
O2—S1—C8	108.65 (7)	C13—C8—S1	120.99 (12)
N2—S1—C8	108.80 (7)	C9—C8—S1	118.40 (11)
C7—N1—N2	115.66 (12)	C10—C9—C8	119.19 (14)
N1—N2—S1	115.99 (10)	C10—C9—H9	120.4
N1—N2—H2	115.6 (13)	C8—C9—H9	120.4
S1—N2—H2	119.9 (13)	C9—C10—C11	121.46 (15)
C2—C1—C6	118.81 (14)	C9—C10—H10	119.3
C2—C1—H1	120.6	C11—C10—H10	119.3
C6—C1—H1	120.6	C12—C11—C10	118.25 (15)
F1—C2—C1	120.70 (14)	C12—C11—C14	121.39 (16)
F1—C2—C3	117.91 (14)	C10—C11—C14	120.36 (16)
C1—C2—C3	121.38 (14)	C13—C12—C11	121.22 (15)
F2—C3—C4	120.86 (14)	C13—C12—H12	119.4
F2—C3—C2	118.32 (14)	C11—C12—H12	119.4
C4—C3—C2	120.81 (14)	C12—C13—C8	119.28 (14)
C3—C4—C5	118.60 (14)	C12—C13—H13	120.4

C3—C4—H4	120.7	C8—C13—H13	120.4
C5—C4—H4	120.7	C11—C14—H14A	109.5
C4—C5—C6	120.77 (14)	C11—C14—H14B	109.5
C4—C5—H5	119.6	H14A—C14—H14B	109.5
C6—C5—H5	119.6	C11—C14—H14C	109.5
C5—C6—C1	119.61 (14)	H14A—C14—H14C	109.5
C5—C6—C7	118.97 (13)	H14B—C14—H14C	109.5
C1—C6—C7	121.40 (13)		

C7—N1—N2—S1	172.11 (11)	C5—C6—C7—N1	−165.16 (14)
O1—S1—N2—N1	49.24 (12)	C1—C6—C7—N1	13.7 (2)
O2—S1—N2—N1	177.34 (10)	O1—S1—C8—C13	−12.85 (14)
C8—S1—N2—N1	−67.26 (12)	O2—S1—C8—C13	−144.84 (13)
C6—C1—C2—F1	−177.38 (13)	N2—S1—C8—C13	103.15 (13)
C6—C1—C2—C3	1.0 (2)	O1—S1—C8—C9	165.65 (11)
F1—C2—C3—F2	−1.6 (2)	O2—S1—C8—C9	33.66 (13)
C1—C2—C3—F2	179.95 (13)	N2—S1—C8—C9	−78.36 (13)
F1—C2—C3—C4	177.91 (14)	C13—C8—C9—C10	0.6 (2)
C1—C2—C3—C4	−0.5 (2)	S1—C8—C9—C10	−177.85 (12)
F2—C3—C4—C5	178.96 (13)	C8—C9—C10—C11	0.3 (2)
C2—C3—C4—C5	−0.5 (2)	C9—C10—C11—C12	−1.0 (3)
C3—C4—C5—C6	1.1 (2)	C9—C10—C11—C14	179.77 (16)
C4—C5—C6—C1	−0.6 (2)	C10—C11—C12—C13	0.7 (3)
C4—C5—C6—C7	178.21 (13)	C14—C11—C12—C13	179.94 (17)
C2—C1—C6—C5	−0.4 (2)	C11—C12—C13—C8	0.2 (2)
C2—C1—C6—C7	−179.24 (13)	C9—C8—C13—C12	−0.9 (2)
N2—N1—C7—C6	175.73 (11)	S1—C8—C13—C12	177.54 (12)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O1^{i}	0.89 (1)	2.09 (1)	2.9747 (18)	174 (2)
C1—H1···F1^{ii}	0.95	2.47	3.345 (2)	153
C4—H4···O2^{iii}	0.95	2.57	3.473 (2)	159

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