organic compounds

N′-[1-(2,4-Difluorophenyl)ethylidene]-4-methylbenzenesulfonohydrazide

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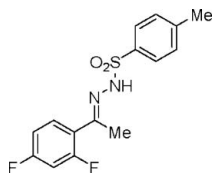Correspondence e-mail: zhenhuashang@yahoo.com.cn

The title compound, $C_{15}H_{14}F_2N_2O_2S$, was synthesized by the reaction of 1-(2,4-difluorophenyl)ethanone and 4-methyl-benzenesulfonohydrazide in ethanol under reflux. The crystal structure is stabilized mainly through intermolecular N—H···O hydrogen bonds. The C=N—N group displays a *trans* conformation.

Related literature

For related literature, see: Siemann *et al.* (2002).

Experimental

Crystal data

$C_{15}H_{14}F_2N_2O_2S$	b = 6.6082 (7) Å
M_r = 324.34	c = 16.5781 (18) Å
Monoclinic, $P2_1/n$	β = 103.924 (2)°
a = 14.1273 (15) Å	V = 1502.2 (3) Å^3
Z = 4	T = 294 (2) K
Mo $K\alpha$ radiation	$0.26 \times 0.22 \times 0.20$ mm
μ = 0.25 mm^{-1}	

Data collection

Bruker SMART CCD area-detector diffractometer	7394 measured reflections
Absorption correction: multi-scan (*SADABS*; Bruker, 1997)	2640 independent reflections
T_{min} = 0.939, T_{max} = 0.953	2252 reflections with $I > 2\sigma(I)$
	R_{int} = 0.019

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.032	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.096	
S = 1.04	$\Delta\rho_{max}$ = 0.26 e Å^{-3}
2640 reflections	$\Delta\rho_{min}$ = −0.30 e Å^{-3}
206 parameters	
1 restraint	

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···O1^i	0.878 (9)	2.154 (11)	3.007 (2)	163.7 (18)

Symmetry code: (i) $-x + 1, -y, -z + 1$.

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

References

Bruker (1997). *SADABS* (Version 2.0), *SMART* (Version 5.611), *SAINT* (Version 6.0) and *SHELXTL* (Version 5.10). Bruker AXS Inc., Madison, Wisconsin, USA.

Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

Siemann, S., Evanoff, D. P., Marrone, L., Clarke, A. J., Viswanatha, T. & Dmitrienko, G. I. (2002). *Antimicrob. Agents Chemother.* **46**, 2450–2457.

supplementary materials

N'-[1-(2,4-Difluorophenyl)ethylidene]-4-methylbenzenesulfonohydrazide

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Comment

N-arylsulfonyl hydrazones exhibit biological activities as inhibitors of metallo-beta-lactamases (Siemann *et al.*, 2002). With a view to developing this kind of potent inhibitors, The title compound was synthesized by the reaction of 1-(2,4-difluorophenyl)ethanone and 4-methylbenzenesulfonohydrazide in ethanol under reflux. There is a *trans* configuration with respect to the C=N bond [C8—N1—N2—S1=172.69 (12)°]. The crystal structure is stabilized mainly through intermolecular N—H···O hydrogen bonds.

Experimental

A solution of 1-(2,4-difluorophenyl)ethanone (1.56 g, 10 mmol), and 4-methylbenzenesulfonohydrazide (1.86 g, 10 mmol) in ethanol (20 ml) was heated under reflux for 2 h. The reaction mixture was cooled and filtered. the product was recrystallized from ethanol to afford the pure product. The title product was dissolved in 100 ml absolute ethanol and crystals suitable for X-ray analysis were grown by slow evaporation of the absolute ethanol solution at room temperature over a period of 15 d.

Refinement

Carbon-bound H atoms were positioned geometrically, with C—H = 0.93–0.96 Å, and refined in a riding model, with $U_{iso}(H) = 1.2U_{eq}$ (carrier).
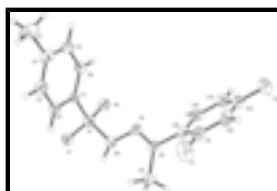
Figures

Fig. 1. The molecular structure of (I), drawn with 30% probability ellipsoids.

Fig. 2. The formation of the title compound.

N'-[1-(2,4-Difluorophenyl)ethylidene]-4-methylbenzenesulfonohydrazide

Crystal data

$C_{15}H_{14}F_2N_2O_2S$	$F_{000} = 672$
$M_r = 324.34$	$D_x = 1.434$ Mg m^{-3}

Monoclinic, $P2_1/n$

Hall symbol: -P 2yn

$a = 14.1273\ (15)$ Å

$b = 6.6082\ (7)$ Å

$c = 16.5781\ (18)$ Å

$\beta = 103.924\ (2)°$

$V = 1502.2\ (3)$ Å^3

$Z = 4$

Melting point: 278-281 K

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å

Cell parameters from 4817 reflections

$\theta = 3.3–26.3°$

$\mu = 0.25$ mm^{-1}

$T = 294\ (2)$ K

Prism, colorless

$0.26 \times 0.22 \times 0.20$ mm

Data collection

Bruker SMART CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 294(2)$ K

φ and ω scans

Absorption correction: multi-scan (SADABS; Bruker, 1997)

$T_{min} = 0.939$, $T_{max} = 0.953$

7394 measured reflections

2640 independent reflections

2252 reflections with $I > 2\sigma(I)$

$R_{int} = 0.019$

$\theta_{max} = 25.0°$

$\theta_{min} = 2.2°$

$h = -16 \rightarrow 7$

$k = -7 \rightarrow 7$

$l = -19 \rightarrow 19$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.032$

$wR(F^2) = 0.096$

$S = 1.04$

2640 reflections

206 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

$w = 1/[\sigma^2(F_o^2) + (0.048P)^2 + 0.5147P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.001$

$\Delta\rho_{max} = 0.26$ e Å^{-3}

$\Delta\rho_{min} = -0.30$ e Å^{-3}

Extinction correction: SHELXL97 (Sheldrick, 1997), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$

Extinction coefficient: 0.0183 (16)

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, convention-al R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculat-ing 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	0.38733 (3)	0.24976 (6)	0.50714 (3)	0.04162 (16)
F1	0.65647 (12)	0.3142 (2)	0.86303 (8)	0.0899 (5)
F2	0.58129 (11)	0.9531 (2)	0.94795 (9)	0.0921 (5)
O1	0.40562 (10)	0.1220 (2)	0.44291 (7)	0.0542 (3)
O2	0.34537 (10)	0.44346 (19)	0.48774 (8)	0.0555 (4)
N1	0.49934 (10)	0.3978 (2)	0.63820 (9)	0.0428 (3)
N2	0.49483 (11)	0.2749 (2)	0.56956 (10)	0.0462 (4)
C1	0.31522 (11)	0.1159 (2)	0.56163 (10)	0.0380 (4)
C2	0.33745 (12)	−0.0836 (3)	0.58291 (11)	0.0462 (4)
H2	0.3905	−0.1457	0.5691	0.055*
C3	0.28052 (14)	−0.1892 (3)	0.62458 (12)	0.0532 (5)
H3	0.2960	−0.3229	0.6397	0.064*
C4	0.20076 (13)	−0.1012 (3)	0.64454 (12)	0.0536 (5)
C5	0.17982 (14)	0.0978 (3)	0.62269 (13)	0.0570 (5)
H5	0.1264	0.1591	0.6361	0.068*
C6	0.23639 (13)	0.2082 (3)	0.58131 (12)	0.0485 (4)
H6	0.2215	0.3426	0.5670	0.058*
C7	0.13587 (19)	−0.2225 (5)	0.68624 (17)	0.0860 (8)
H7A	0.0764	−0.2553	0.6464	0.129*
H7B	0.1212	−0.1445	0.7306	0.129*
H7C	0.1686	−0.3450	0.7083	0.129*
C8	0.58070 (12)	0.4018 (3)	0.69233 (11)	0.0423 (4)
C9	0.66954 (14)	0.2841 (3)	0.68831 (14)	0.0586 (5)
H9A	0.6558	0.1420	0.6894	0.088*
H9B	0.7218	0.3181	0.7351	0.088*
H9C	0.6882	0.3165	0.6378	0.088*
C10	0.58342 (11)	0.5477 (3)	0.76081 (10)	0.0418 (4)
C11	0.62065 (14)	0.5015 (3)	0.84319 (12)	0.0545 (5)
C12	0.62055 (16)	0.6336 (4)	0.90682 (12)	0.0636 (6)
H12	0.6454	0.5966	0.9620	0.076*
C13	0.58272 (15)	0.8206 (4)	0.88617 (13)	0.0599 (5)
C14	0.54634 (15)	0.8790 (3)	0.80576 (13)	0.0602 (5)
H14	0.5217	1.0088	0.7931	0.072*
C15	0.54702 (14)	0.7413 (3)	0.74412 (12)	0.0505 (4)
H15	0.5221	0.7795	0.6891	0.061*
H2A	0.5345 (12)	0.172 (2)	0.5714 (12)	0.055 (6)*

supplementary materials

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0431 (3)	0.0434 (3)	0.0385 (2)	−0.00387 (18)	0.00998 (18)	0.00020 (17)
F1	0.1256 (13)	0.0616 (8)	0.0652 (8)	0.0158 (8)	−0.0107 (8)	0.0132 (7)
F2	0.1043 (11)	0.1014 (11)	0.0727 (8)	−0.0043 (9)	0.0255 (8)	−0.0387 (8)
O1	0.0630 (8)	0.0620 (8)	0.0395 (7)	−0.0033 (7)	0.0164 (6)	−0.0056 (6)
O2	0.0586 (8)	0.0463 (7)	0.0594 (8)	−0.0007 (6)	0.0099 (6)	0.0105 (6)
N1	0.0394 (8)	0.0452 (8)	0.0445 (8)	−0.0056 (6)	0.0115 (6)	−0.0046 (6)
N2	0.0392 (8)	0.0495 (9)	0.0504 (8)	−0.0031 (7)	0.0116 (7)	−0.0085 (7)
C1	0.0353 (8)	0.0416 (9)	0.0352 (8)	−0.0032 (7)	0.0047 (6)	−0.0027 (7)
C2	0.0403 (9)	0.0465 (10)	0.0520 (10)	0.0044 (8)	0.0115 (8)	0.0046 (8)
C3	0.0504 (11)	0.0493 (10)	0.0574 (11)	−0.0014 (9)	0.0084 (9)	0.0119 (9)
C4	0.0437 (10)	0.0694 (13)	0.0464 (10)	−0.0100 (9)	0.0085 (8)	0.0055 (9)
C5	0.0441 (10)	0.0683 (13)	0.0628 (12)	0.0038 (9)	0.0212 (9)	−0.0043 (10)
C6	0.0462 (10)	0.0449 (10)	0.0550 (10)	0.0036 (8)	0.0134 (8)	−0.0021 (8)
C7	0.0658 (15)	0.112 (2)	0.0864 (17)	−0.0153 (14)	0.0298 (13)	0.0278 (15)
C8	0.0376 (9)	0.0413 (9)	0.0476 (9)	−0.0035 (7)	0.0096 (7)	0.0026 (7)
C9	0.0428 (10)	0.0622 (12)	0.0683 (13)	0.0070 (9)	0.0083 (9)	−0.0036 (10)
C10	0.0336 (8)	0.0465 (9)	0.0434 (9)	−0.0053 (7)	0.0058 (7)	0.0007 (7)
C11	0.0540 (11)	0.0508 (11)	0.0516 (11)	−0.0018 (9)	−0.0010 (9)	0.0053 (9)
C12	0.0651 (13)	0.0759 (15)	0.0427 (10)	−0.0084 (11)	−0.0010 (9)	−0.0028 (10)
C13	0.0537 (11)	0.0717 (14)	0.0557 (12)	−0.0086 (10)	0.0161 (9)	−0.0186 (11)
C14	0.0603 (12)	0.0550 (12)	0.0660 (13)	0.0094 (9)	0.0165 (10)	−0.0044 (10)
C15	0.0489 (10)	0.0549 (11)	0.0466 (10)	0.0075 (8)	0.0092 (8)	0.0026 (8)

Geometric parameters (Å, °)

S1—O2	1.4148 (13)	C6—H6	0.9300
S1—O1	1.4304 (13)	C7—H7A	0.9600
S1—N2	1.6273 (16)	C7—H7B	0.9600
S1—C1	1.7550 (17)	C7—H7C	0.9600
F1—C11	1.348 (2)	C8—C10	1.483 (2)
F2—C13	1.351 (2)	C8—C9	1.492 (3)
N1—C8	1.277 (2)	C9—H9A	0.9600
N1—N2	1.387 (2)	C9—H9B	0.9600
N2—H2A	0.878 (9)	C9—H9C	0.9600
C1—C6	1.376 (2)	C10—C11	1.375 (2)
C1—C2	1.381 (2)	C10—C15	1.382 (2)
C2—C3	1.371 (3)	C11—C12	1.369 (3)
C2—H2	0.9300	C12—C13	1.358 (3)
C3—C4	1.377 (3)	C12—H12	0.9300
C3—H3	0.9300	C13—C14	1.364 (3)
C4—C5	1.377 (3)	C14—C15	1.370 (3)
C4—C7	1.505 (3)	C14—H14	0.9300
C5—C6	1.381 (3)	C15—H15	0.9300
C5—H5	0.9300		

O2—S1—O1	120.37 (8)	C4—C7—H7C	109.5
O2—S1—N2	109.15 (8)	H7A—C7—H7C	109.5
O1—S1—N2	103.04 (8)	H7B—C7—H7C	109.5
O2—S1—C1	108.05 (8)	N1—C8—C10	113.86 (15)
O1—S1—C1	108.47 (8)	N1—C8—C9	125.63 (17)
N2—S1—C1	107.02 (8)	C10—C8—C9	120.41 (15)
C8—N1—N2	116.39 (15)	C8—C9—H9A	109.5
N1—N2—S1	115.66 (12)	C8—C9—H9B	109.5
N1—N2—H2A	121.5 (13)	H9A—C9—H9B	109.5
S1—N2—H2A	115.7 (13)	C8—C9—H9C	109.5
C6—C1—C2	120.68 (16)	H9A—C9—H9C	109.5
C6—C1—S1	120.09 (14)	H9B—C9—H9C	109.5
C2—C1—S1	119.22 (13)	C11—C10—C15	116.07 (17)
C3—C2—C1	119.32 (17)	C11—C10—C8	123.34 (16)
C3—C2—H2	120.3	C15—C10—C8	120.59 (16)
C1—C2—H2	120.3	F1—C11—C12	117.85 (17)
C2—C3—C4	121.29 (18)	F1—C11—C10	118.54 (17)
C2—C3—H3	119.4	C12—C11—C10	123.57 (19)
C4—C3—H3	119.4	C13—C12—C11	117.34 (19)
C5—C4—C3	118.46 (18)	C13—C12—H12	121.3
C5—C4—C7	120.9 (2)	C11—C12—H12	121.3
C3—C4—C7	120.6 (2)	F2—C13—C12	118.39 (19)
C4—C5—C6	121.47 (18)	F2—C13—C14	119.1 (2)
C4—C5—H5	119.3	C12—C13—C14	122.48 (19)
C6—C5—H5	119.3	C13—C14—C15	118.20 (19)
C1—C6—C5	118.78 (18)	C13—C14—H14	120.9
C1—C6—H6	120.6	C15—C14—H14	120.9
C5—C6—H6	120.6	C14—C15—C10	122.32 (18)
C4—C7—H7A	109.5	C14—C15—H15	118.8
C4—C7—H7B	109.5	C10—C15—H15	118.8
H7A—C7—H7B	109.5		
C8—N1—N2—S1	172.69 (12)	N2—N1—C8—C10	175.47 (14)
O2—S1—N2—N1	49.65 (14)	N2—N1—C8—C9	−1.0 (3)
O1—S1—N2—N1	178.69 (12)	N1—C8—C10—C11	134.53 (18)
C1—S1—N2—N1	−67.05 (14)	C9—C8—C10—C11	−48.8 (2)
O2—S1—C1—C6	−3.03 (17)	N1—C8—C10—C15	−45.1 (2)
O1—S1—C1—C6	−135.05 (14)	C9—C8—C10—C15	131.60 (19)
N2—S1—C1—C6	114.40 (15)	C15—C10—C11—F1	179.52 (17)
O2—S1—C1—C2	175.71 (13)	C8—C10—C11—F1	−0.1 (3)
O1—S1—C1—C2	43.68 (15)	C15—C10—C11—C12	1.7 (3)
N2—S1—C1—C2	−66.87 (15)	C8—C10—C11—C12	−177.86 (18)
C6—C1—C2—C3	−0.5 (3)	F1—C11—C12—C13	−178.86 (19)
S1—C1—C2—C3	−179.21 (14)	C10—C11—C12—C13	−1.1 (3)
C1—C2—C3—C4	1.0 (3)	C11—C12—C13—F2	179.59 (19)
C2—C3—C4—C5	−0.9 (3)	C11—C12—C13—C14	−0.4 (3)
C2—C3—C4—C7	176.7 (2)	F2—C13—C14—C15	−178.94 (19)
C3—C4—C5—C6	0.4 (3)	C12—C13—C14—C15	1.0 (3)
C7—C4—C5—C6	−177.3 (2)	C13—C14—C15—C10	−0.3 (3)
C2—C1—C6—C5	0.0 (3)	C11—C10—C15—C14	−1.0 (3)

| S1—C1—C6—C5 | 178.67 (14) | C8—C10—C15—C14 | 178.59 (17) |
| C4—C5—C6—C1 | 0.1 (3) | | |

Hydrogen-bond geometry (Å, °)

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
N2—H2A⋯O1[i]	0.878 (9)	2.154 (11)	3.007 (2)	163.7 (18)

Symmetry codes: (i) $-x+1, -y, -z+1$.

Fig. 1

Fig. 2

(I)