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(2*E*)-2-(4-Fluorobenzylidene)-hydrazinecarboxamide

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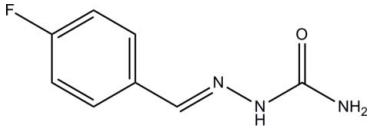
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.003 Å; R factor = 0.053; wR factor = 0.209; data-to-parameter ratio = 18.6.

In the title compound, $C_8H_8FN_3O$, the semicarbazide group is close to being planar, with a maximum deviation of 0.020 (1) Å, and subtends a dihedral angle of 16.63 (9)° with its attached fluorobenzene ring. In the crystal, molecules are linked by N—H···O hydrogen bonds, forming layers lying parallel to the *bc* plane.

Related literature

For background to semicarbazides and semicarbazones, see: Dogan *et al.* (1999); Pandeya & Dimmock (1993); Pandeya *et al.* (1998); Sriram *et al.* (2004); Yogeeswari *et al.* (2004); For further synthetic details, see: Furniss *et al.* (1978). For related structures, see: Fun *et al.* (2009*a*,*b*). For reference bond lengths, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_8H_8FN_3O$
M_r = 181.17
Monoclinic, $P2_1/c$
a = 16.522 (2) Å
b = 4.4381 (6) Å
c = 11.9457 (15) Å
β = 103.478 (3)°
V = 851.80 (19) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 296 K
0.72 × 0.18 × 0.12 mm

Data collection

Bruker APEX DUO CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.923, T_{max} = 0.987
8746 measured reflections
2418 independent reflections
1657 reflections with $I > 2\sigma(I)$
R_{int} = 0.024

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.209
S = 1.00
2418 reflections
130 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N3—H1*N3*···O1^{i}	0.88 (2)	2.07 (2)	2.8954 (19)	158 (2)
N2—H1*N2*···O1^{ii}	0.92 (2)	2.00 (2)	2.9155 (19)	179 (2)

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

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB6436).

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supporting information

Acta Cryst. (2011). E**67**, o2885–o2886 [doi:10.1107/S1600536811040797]

(2*E*)-2-(4-Fluorobenzylidene)hydrazinecarboxamide

Hoong-Kun Fun, Tze Shyang Chia, Shridhar Malladi, Arun M. Isloor and Kammasandra N. Shivananda

S1. Comment

The semicarbazides, which are the raw material of semicarbazones, have been known to possess biological activities against many of the most common species of bacteria (Dogan *et al.*, 1999). Semicarbazones are of much interest due to their wide spectrum of antibacterial activities (Pandeya & Dimmock, 1993). Recently some workers have reviewed the bioactivity of semicarbazones and they have exhibited anticonvulsant (Pandeya *et al.*, 1998; Yogeeswari *et al.*, 2004) and antitubercular (Sriram *et al.*, 2004) properties. Accordingly and by considering the biological potential of semicarbazones, herein, we have synthesized the title compound to study its crystal structure.

The molecular structure of the title compound is shown in Fig. 1. The semicarbazone group (O1/N1–N3/C8) is essentially planar with maximum deviation of 0.020 (1) Å for atom N2. This plane makes dihedral angle of 16.63 (9)° with its terminal benzene ring (C1–C6). Bond lengths (Allen *et al.*, 1987) and angles are within normal ranges and are comparable to related structures (Fun *et al.*, 2009*a*,*b*).

In the crystal structure (Fig. 2), the molecules are interconnected by N3—H1N3···O1 and N2—H1N2···O1 hydrogen bonds (Table 1) forming two-dimensional networks parallel to *bc* plane.

S2. Experimental

Semicarbazide hydrochloride (0.86 g, 7.70 mmol) and freshly recrystallized sodium acetate (0.77 g, 9.40 mmol) were dissolved in water (10 ml) following a literature procedure (Furniss *et al.*, 1978). The reaction mixture was stirred at room temperature for 10 minutes. To this, 4-fluorobenzaldehyde (0.896 g, 7.23 mmol) was added and the mixture was shaken well. A little alcohol was added to dissolve the turbidity. The mixture was shaken for a further 10 minutes and allowed to stand. The title compound crystallizes out on standing for 6 h. The separated crystals were filtered, washed with cold water and recrystallized from ethanol to yield colourless needles. Yield: 0.98 g, 75.38%. *M.p.*: 506–508 K.

S3. Refinement

Atoms H1N2, H1N3 and H2N3 were located in a difference map and refined freely [N—H = 0.90 (2), 0.87 (2) and 0.91 (2) Å respectively]. The remaining H atoms were positioned geometrically [C—H = 0.93 Å] and refined using a riding model with U_{iso}(H) = 1.2 U_{eq}(C).

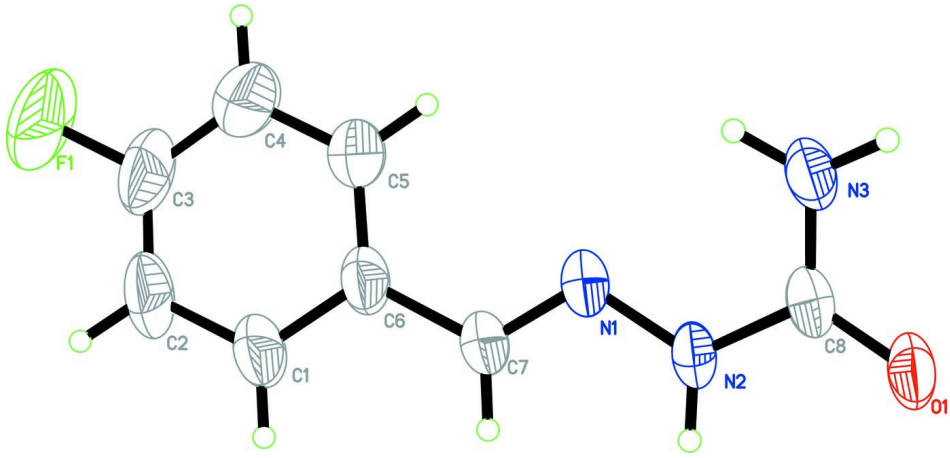

Figure 1

The molecular structure of the title compound with atom labels with 50% probability displacement ellipsoids.

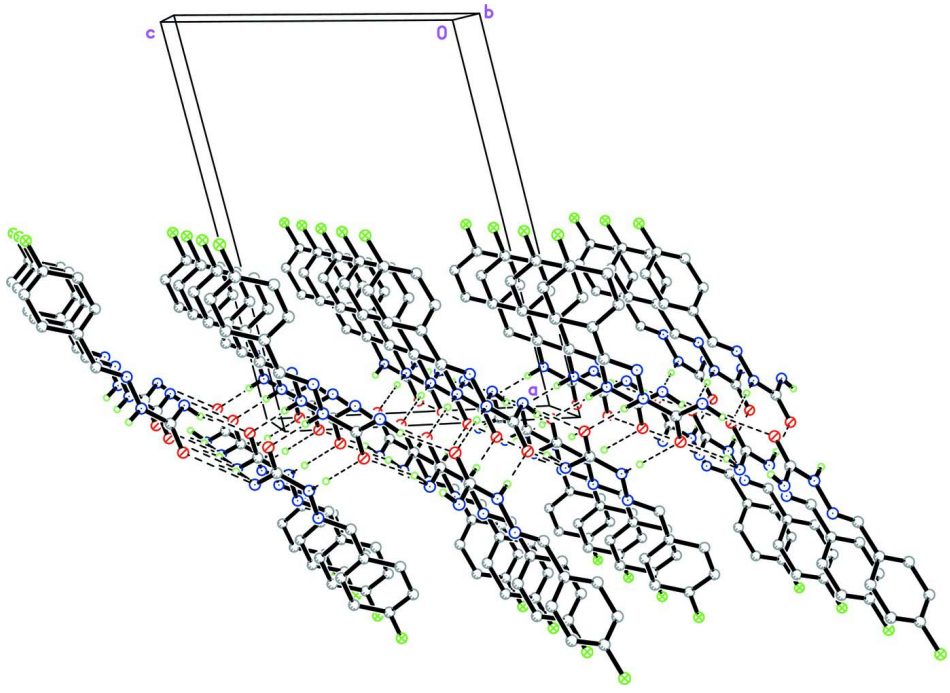

Figure 2

The crystal packing of the title compound. The dashed lines represent the hydrogen bonds.

(2*E*)-2-(4-Fluorobenzylidene)hydrazinecarboxamide

Crystal data

$C_8H_8FN_3O$
$M_r = 181.17$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 16.522\ (2)$ Å
$b = 4.4381\ (6)$ Å
$c = 11.9457\ (15)$ Å
$\beta = 103.478\ (3)°$
$V = 851.80\ (19)$ Å^3
$Z = 4$
$F(000) = 376$
$D_x = 1.413$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 2666 reflections
$\theta = 2.5–29.4°$
$\mu = 0.11$ mm^{-1}

T = 296 K
Needle, colourless

0.72 × 0.18 × 0.12 mm

Data collection

Bruker APEX DUO CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.923, T_{max} = 0.987

8746 measured reflections
2418 independent reflections
1657 reflections with $I > 2\sigma(I)$
R_{int} = 0.024
θ_{max} = 29.9°, θ_{min} = 1.3°
h = −23→23
k = −6→6
l = −16→15

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.053
$wR(F^2)$ = 0.209
S = 1.00
2418 reflections
130 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
$w = 1/[\sigma^2(F_o^2) + (0.1446P)^2 + 0.0418P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 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}
F1	0.51936 (9)	1.2636 (4)	1.15848 (18)	0.1170 (6)
O1	1.01013 (7)	0.7059 (3)	0.87156 (9)	0.0508 (3)
N1	0.83497 (7)	0.8821 (3)	0.97516 (10)	0.0448 (3)
N2	0.90494 (8)	0.7354 (3)	0.96093 (11)	0.0470 (3)
N3	0.91237 (8)	1.0690 (3)	0.81619 (11)	0.0493 (4)
C1	0.71134 (11)	0.8578 (5)	1.18840 (15)	0.0637 (5)
H1A	0.7461	0.7333	1.2413	0.076*
C2	0.63885 (11)	0.9701 (6)	1.21288 (16)	0.0705 (5)
H2A	0.6244	0.9217	1.2814	0.085*
C3	0.58977 (12)	1.1518 (5)	1.1342 (2)	0.0740 (6)
C4	0.60851 (13)	1.2285 (6)	1.0325 (2)	0.0855 (7)
H4A	0.5735	1.3543	0.9805	0.103*
C5	0.68021 (11)	1.1161 (5)	1.00849 (17)	0.0669 (5)

H5A	0.6937	1.1662	0.9394	0.080*
C6	0.73235 (9)	0.9300 (4)	1.08559 (13)	0.0493 (4)
C7	0.80796 (9)	0.8014 (4)	1.06141 (13)	0.0504 (4)
H7A	0.8371	0.6560	1.1109	0.060*
C8	0.94581 (8)	0.8362 (3)	0.88136 (11)	0.0405 (3)
H1N3	0.9423 (12)	1.145 (5)	0.7725 (18)	0.064 (5)*
H1N2	0.9306 (12)	0.599 (5)	1.0133 (16)	0.061 (5)*
H2N3	0.8717 (14)	1.181 (5)	0.8357 (19)	0.074 (6)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
F1	0.0854 (10)	0.1393 (14)	0.1524 (15)	0.0374 (9)	0.0807 (10)	0.0080 (10)
O1	0.0544 (6)	0.0586 (7)	0.0499 (6)	0.0023 (4)	0.0334 (5)	−0.0022 (4)
N1	0.0447 (6)	0.0544 (7)	0.0417 (6)	0.0019 (5)	0.0228 (5)	−0.0006 (5)
N2	0.0490 (7)	0.0566 (7)	0.0446 (7)	0.0073 (5)	0.0296 (5)	0.0048 (5)
N3	0.0569 (7)	0.0541 (7)	0.0457 (7)	−0.0004 (5)	0.0299 (6)	0.0032 (5)
C1	0.0563 (9)	0.0936 (13)	0.0502 (9)	0.0081 (8)	0.0304 (7)	0.0076 (8)
C2	0.0662 (10)	0.0974 (15)	0.0620 (10)	−0.0021 (9)	0.0438 (9)	−0.0068 (10)
C3	0.0558 (10)	0.0863 (14)	0.0938 (15)	0.0098 (8)	0.0457 (10)	−0.0057 (11)
C4	0.0695 (12)	0.1019 (16)	0.0965 (17)	0.0321 (11)	0.0423 (12)	0.0232 (13)
C5	0.0631 (10)	0.0820 (12)	0.0656 (11)	0.0196 (8)	0.0353 (8)	0.0179 (9)
C6	0.0457 (7)	0.0643 (9)	0.0448 (7)	0.0019 (6)	0.0244 (6)	−0.0001 (6)
C7	0.0482 (8)	0.0673 (9)	0.0425 (8)	0.0090 (6)	0.0245 (6)	0.0085 (6)
C8	0.0461 (7)	0.0452 (7)	0.0361 (6)	−0.0067 (5)	0.0215 (5)	−0.0085 (5)

Geometric parameters (Å, º)

F1—C3	1.3568 (19)	C1—H1A	0.9300
O1—C8	1.2393 (16)	C2—C3	1.355 (3)
N1—C7	1.2661 (18)	C2—H2A	0.9300
N1—N2	1.3714 (16)	C3—C4	1.365 (3)
N2—C8	1.3634 (17)	C4—C5	1.376 (2)
N2—H1N2	0.90 (2)	C4—H4A	0.9300
N3—C8	1.3333 (18)	C5—C6	1.379 (2)
N3—H1N3	0.87 (2)	C5—H5A	0.9300
N3—H2N3	0.91 (2)	C6—C7	1.4621 (19)
C1—C2	1.390 (2)	C7—H7A	0.9300
C1—C6	1.389 (2)		
C7—N1—N2	115.71 (12)	C3—C4—C5	118.7 (2)
C8—N2—N1	119.96 (12)	C3—C4—H4A	120.6
C8—N2—H1N2	118.4 (12)	C5—C4—H4A	120.6
N1—N2—H1N2	120.3 (12)	C4—C5—C6	120.79 (17)
C8—N3—H1N3	115.9 (13)	C4—C5—H5A	119.6
C8—N3—H2N3	120.3 (14)	C6—C5—H5A	119.6
H1N3—N3—H2N3	120 (2)	C5—C6—C1	118.85 (14)
C2—C1—C6	120.56 (17)	C5—C6—C7	122.08 (14)

C2—C1—H1A	119.7	C1—C6—C7	119.06 (15)
C6—C1—H1A	119.7	N1—C7—C6	121.99 (14)
C3—C2—C1	118.24 (16)	N1—C7—H7A	119.0
C3—C2—H2A	120.9	C6—C7—H7A	119.0
C1—C2—H2A	120.9	O1—C8—N3	123.66 (12)
F1—C3—C2	118.27 (19)	O1—C8—N2	119.18 (13)
F1—C3—C4	118.9 (2)	N3—C8—N2	117.15 (12)
C2—C3—C4	122.86 (16)		

C7—N1—N2—C8	−170.19 (13)	C4—C5—C6—C7	178.57 (19)
C6—C1—C2—C3	−0.3 (3)	C2—C1—C6—C5	0.3 (3)
C1—C2—C3—F1	−179.4 (2)	C2—C1—C6—C7	−178.40 (17)
C1—C2—C3—C4	0.0 (4)	N2—N1—C7—C6	−177.87 (13)
F1—C3—C4—C5	179.6 (2)	C5—C6—C7—N1	8.7 (3)
C2—C3—C4—C5	0.2 (4)	C1—C6—C7—N1	−172.65 (16)
C3—C4—C5—C6	−0.1 (4)	N1—N2—C8—O1	178.19 (12)
C4—C5—C6—C1	−0.1 (3)	N1—N2—C8—N3	−3.3 (2)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H1*N*3···O1^{i}	0.88 (2)	2.07 (2)	2.8954 (19)	158 (2)
N2—H1*N*2···O1^{ii}	0.92 (2)	2.00 (2)	2.9155 (19)	179 (2)

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