organic compounds

N'-[(E)-1-(3-Fluorophenyl)ethylidene]-formohydrazide

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In the title compound, $C_9H_9FN_2O$, the dihedral angle between the fluorobenzene ring and the mean plane of the side chain is 15.59 (14)°. In the crystal, the molecules form inversion dimers linked by pairs of N—H⋯O hydrogen bonds, resulting in $R_2^2(8)$ loops. These dimers are reinforced by C—H⋯O interactions.

Related literature

For related structures, see: Shafiq et al. (2009a,b). For graph-set notation, see: Bernstein et al. (1995).
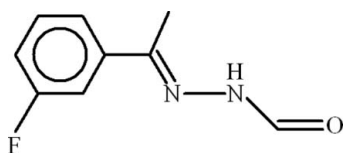
Experimental

Crystal data

$C_9H_9FN_2O$
$M_r = 180.18$
Triclinic, $P\bar{1}$
$a = 6.8466$ (5) Å
$b = 7.0258$ (6) Å
$c = 9.9419$ (8) Å
$\alpha = 70.558$ (5)°
$\beta = 81.267$ (5)°

$\gamma = 73.977$ (4)°
$V = 432.50$ (6) Å^3
$Z = 2$
Mo $K\alpha$ radiation
$\mu = 0.11$ mm^{-1}
$T = 296$ K
$0.28 \times 0.12 \times 0.10$ mm

Data collection

Bruker Kappa APEXII CCD diffractometer
Absorption correction: multi-scan (SADABS; Bruker, 2005)
$T_{min} = 0.986$, $T_{max} = 0.990$

19438 measured reflections
2124 independent reflections
1320 reflections with $I > 2\sigma(I)$
$R_{int} = 0.028$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.148$
$S = 1.00$
2124 reflections

119 parameters
H-atom parameters constrained
$\Delta\rho_{max} = 0.23$ e Å^{-3}
$\Delta\rho_{min} = -0.20$ e Å^{-3}

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
N2—H2A⋯O1[i]	0.86	2.14	2.989 (2)	168
C8—H8A⋯O1[i]	0.96	2.52	3.204 (3)	129

Symmetry code: (i) $-x, -y + 2, -z + 1$.

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 (Farrugia, 1997) and PLATON (Spek, 2009); software used to prepare material for publication: WinGX (Farrugia, 1999) and PLATON.

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supporting information

N′-[(*E*)-1-(3-Fluorophenyl)ethylidene]formohydrazide

Zahid Shafiq, Muhammad Yaqub, M. Nawaz Tahir, Mian Hasnain Nawaz and M. Saeed Iqbal

S1. Comment

Recently we have reported the crystal structures of (II) *N*′-[(1*E*)-1-(4-Chlorophenyl)ethylidene]formohydrazide (Shafiq *et al.*, 2009*a*), (III) *N*′-[(*E*)-(5-Methylfuran-2-yl)methylidene]formohydrazide (Shafiq *et al.*, 2009*b*). The title compound (I, Fig. 1) has been prepared in continuation of synthesizing various formohydrazide derivatives.

In (I), the groups A (C1—C6/F1) and B (C7/C8/N1/N2/C9) are planar with maximum r. m. s. deviations of 0.0022 and 0.0146 Å, respectively from their mean squares planes. The dihedral angle between A/B is 15.59 (14)°.

The molecules of (I) consist of dimers similar to (II) and (III) due to N–H⋯O type of intermolecular H-bondings forming $R_2^2(8)$ ring motifs (Bernstein *et al.*, 1995). The difference between (I) and (II) is the substitution of Cl and F-atom on the *para* and *meta* positions of benzene ring, respectively. Due to this change there exist two $R_2^1(7)$ ring motifs in dimers due to C–H⋯O and N—H⋯O H-bondings (Table 1).

S2. Experimental

To a hot stirred solution of formic hydrazide (1.0 g, 0.017 mol) in ethanol (15 ml) was added 1-(3-fluorophenyl)ethanone (2.043 ml, 0.017 mol). The resultant mixture was then heated under reflux. The reaction mixture was refluxed about 12 h and monitored through TLC. After the completion of reaction, the mixture was cooled to room temperature. The solid was collected by suction filtration. The product obtained was washed with hot ethanol and 1,4-dioxan and dried. Colourless needles of (I) were obtained by recrystallization of the crude product in 1,4-dioxan after two days.

S3. Refinement

The H-atoms were positioned geometrically (N—H = 0.86 Å, C—H = 0.93–0.96 Å) and refined as riding with $U_{iso}(H) = 1.2U_{eq}$(carrier) or $1.5U_{eq}$(methyl C).
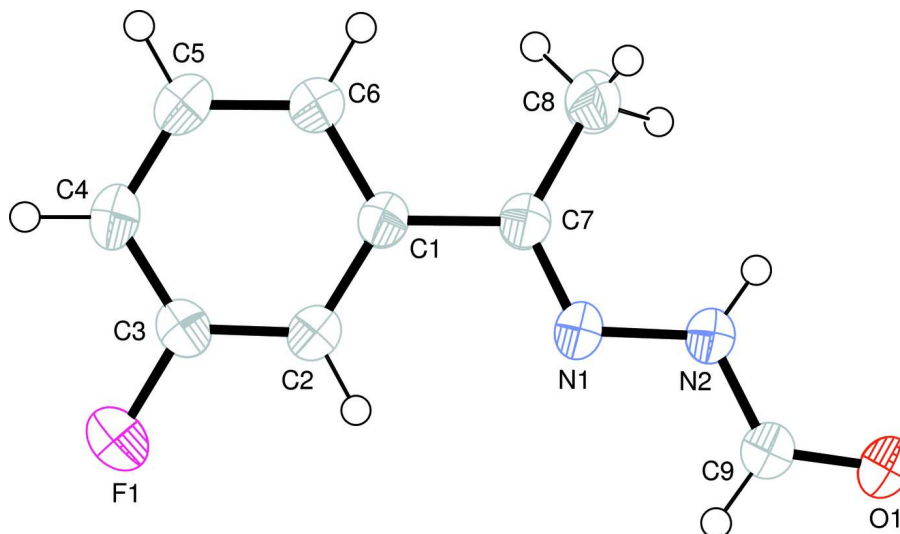
Figure 1

View of (I) with displacement ellipsoids drawn at the 50% probability level. H-atoms are shown by circles of arbitrary radius.
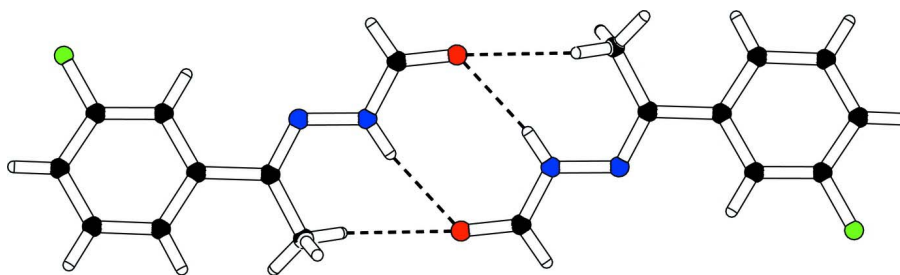
Figure 2

The partial packing of (I), which shows that molecules form dimers.

N′-[(E)-1-(3-Fluorophenyl)ethylidene]formohydrazide

Crystal data

$C_9H_9FN_2O$	$Z = 2$
$M_r = 180.18$	$F(000) = 188$
Triclinic, $P\bar{1}$	$D_x = 1.384$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 6.8466$ (5) Å	Cell parameters from 2124 reflections
$b = 7.0258$ (6) Å	$\theta = 3.1–28.3°$
$c = 9.9419$ (8) Å	$\mu = 0.11$ mm^{-1}
$\alpha = 70.558$ (5)°	$T = 296$ K
$\beta = 81.267$ (5)°	Cut needle, colourless
$\gamma = 73.977$ (4)°	$0.28 \times 0.12 \times 0.10$ mm
$V = 432.50$ (6) Å^3	

Data collection

Bruker Kappa APEXII CCD diffractometer	Detector resolution: 7.40 pixels mm^{-1}
	ω scans
Radiation source: fine-focus sealed tube	Absorption correction: multi-scan
Graphite monochromator	(*SADABS*; Bruker, 2005)

$T_{min} = 0.986$, $T_{max} = 0.990$
19438 measured reflections
2124 independent reflections
1320 reflections with $I > 2\sigma(I)$
$R_{int} = 0.028$

$\theta_{max} = 28.3°$, $\theta_{min} = 3.1°$
$h = -9 \rightarrow 9$
$k = -9 \rightarrow 9$
$l = -13 \rightarrow 12$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.044$
$wR(F^2) = 0.148$
$S = 1.00$
2124 reflections
119 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-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0721P)^2 + 0.1041P]$
   where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.23$ e Å^{-3}
$\Delta\rho_{min} = -0.20$ 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}
F1	−0.17799 (17)	0.19897 (19)	0.11146 (13)	0.0654 (5)
O1	−0.27158 (19)	1.0743 (2)	0.48303 (16)	0.0571 (5)
N1	−0.0361 (2)	0.6861 (2)	0.32755 (15)	0.0397 (4)
N2	−0.0531 (2)	0.8255 (2)	0.40149 (15)	0.0423 (5)
C1	0.1524 (2)	0.4305 (3)	0.22398 (18)	0.0385 (5)
C2	−0.0221 (3)	0.3804 (3)	0.20534 (18)	0.0414 (5)
C3	−0.0068 (3)	0.2466 (3)	0.12916 (19)	0.0443 (6)
C4	0.1727 (3)	0.1578 (3)	0.0688 (3)	0.0600 (8)
C5	0.3447 (3)	0.2084 (4)	0.0867 (3)	0.0734 (10)
C6	0.3367 (3)	0.3417 (3)	0.1637 (2)	0.0582 (7)
C7	0.1417 (2)	0.5780 (3)	0.30417 (18)	0.0395 (5)
C8	0.3324 (3)	0.5870 (4)	0.3542 (3)	0.0689 (8)
C9	−0.2381 (3)	0.9408 (3)	0.4233 (2)	0.0462 (6)
H2	−0.14795	0.43732	0.24433	0.0496*
H2A	0.05215	0.83756	0.43242	0.0507*
H4	0.17812	0.06689	0.01769	0.0720*
H5	0.46934	0.15168	0.04599	0.0882*
H6	0.45566	0.37230	0.17538	0.0698*
H8A	0.30519	0.60432	0.44755	0.1034*
H8B	0.38093	0.70226	0.28885	0.1034*
H8C	0.43388	0.46005	0.35838	0.1034*

H9	−0.34809	0.91844	0.39133	0.0554*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
F1	0.0472 (7)	0.0812 (9)	0.0917 (9)	−0.0205 (6)	−0.0071 (6)	−0.0522 (7)
O1	0.0416 (7)	0.0611 (9)	0.0844 (10)	−0.0063 (6)	0.0031 (6)	−0.0510 (8)
N1	0.0379 (7)	0.0397 (8)	0.0486 (8)	−0.0064 (6)	−0.0014 (6)	−0.0256 (7)
N2	0.0350 (7)	0.0458 (8)	0.0558 (9)	−0.0057 (6)	−0.0034 (6)	−0.0312 (7)
C1	0.0353 (8)	0.0386 (9)	0.0457 (10)	−0.0038 (7)	−0.0042 (7)	−0.0217 (8)
C2	0.0348 (8)	0.0458 (10)	0.0482 (10)	−0.0059 (7)	0.0000 (7)	−0.0247 (8)
C3	0.0392 (9)	0.0480 (10)	0.0543 (11)	−0.0110 (8)	−0.0071 (7)	−0.0248 (9)
C4	0.0485 (11)	0.0681 (13)	0.0841 (15)	−0.0068 (9)	−0.0018 (10)	−0.0570 (12)
C5	0.0407 (10)	0.0940 (18)	0.112 (2)	−0.0058 (10)	0.0056 (11)	−0.0791 (16)
C6	0.0332 (9)	0.0723 (14)	0.0886 (15)	−0.0071 (9)	−0.0009 (9)	−0.0558 (12)
C7	0.0357 (8)	0.0408 (9)	0.0470 (10)	−0.0046 (7)	−0.0058 (7)	−0.0224 (8)
C8	0.0423 (10)	0.0802 (15)	0.1084 (18)	0.0019 (10)	−0.0197 (11)	−0.0673 (14)
C9	0.0356 (9)	0.0491 (10)	0.0638 (12)	−0.0082 (7)	−0.0003 (8)	−0.0330 (9)

Geometric parameters (Å, º)

F1—C3	1.355 (2)	C4—C5	1.372 (3)
O1—C9	1.223 (3)	C5—C6	1.379 (3)
N1—N2	1.380 (2)	C7—C8	1.490 (3)
N1—C7	1.278 (2)	C2—H2	0.9300
N2—C9	1.332 (3)	C4—H4	0.9300
N2—H2A	0.8600	C5—H5	0.9300
C1—C2	1.389 (3)	C6—H6	0.9300
C1—C7	1.485 (3)	C8—H8A	0.9600
C1—C6	1.388 (3)	C8—H8B	0.9600
C2—C3	1.365 (3)	C8—H8C	0.9600
C3—C4	1.364 (3)	C9—H9	0.9300
N2—N1—C7	117.88 (15)	O1—C9—N2	123.78 (19)
N1—N2—C9	117.74 (15)	C1—C2—H2	120.00
C9—N2—H2A	121.00	C3—C2—H2	120.00
N1—N2—H2A	121.00	C3—C4—H4	121.00
C6—C1—C7	120.83 (15)	C5—C4—H4	121.00
C2—C1—C7	120.92 (16)	C4—C5—H5	119.00
C2—C1—C6	118.24 (18)	C6—C5—H5	119.00
C1—C2—C3	119.28 (19)	C1—C6—H6	120.00
C2—C3—C4	123.5 (2)	C5—C6—H6	120.00
F1—C3—C2	118.80 (18)	C7—C8—H8A	109.00
F1—C3—C4	117.74 (18)	C7—C8—H8B	109.00
C3—C4—C5	117.2 (2)	C7—C8—H8C	109.00
C4—C5—C6	121.4 (2)	H8A—C8—H8B	110.00
C1—C6—C5	120.5 (2)	H8A—C8—H8C	109.00
N1—C7—C1	115.92 (14)	H8B—C8—H8C	109.00

N1—C7—C8	124.86 (19)	O1—C9—H9	118.00
C1—C7—C8	119.20 (17)	N2—C9—H9	118.00
C7—N1—N2—C9	178.74 (16)	C2—C1—C7—C8	164.42 (19)
N2—N1—C7—C1	−179.88 (14)	C6—C1—C7—N1	164.85 (17)
N2—N1—C7—C8	1.7 (3)	C6—C1—C7—C8	−16.6 (3)
N1—N2—C9—O1	−177.46 (17)	C1—C2—C3—F1	−179.92 (16)
C6—C1—C2—C3	0.1 (3)	C1—C2—C3—C4	−0.2 (3)
C7—C1—C2—C3	179.02 (17)	F1—C3—C4—C5	179.6 (2)
C2—C1—C6—C5	0.4 (3)	C2—C3—C4—C5	−0.2 (4)
C7—C1—C6—C5	−178.6 (2)	C3—C4—C5—C6	0.7 (4)
C2—C1—C7—N1	−14.1 (3)	C4—C5—C6—C1	−0.8 (4)

Hydrogen-bond geometry (Å, º)

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···O1[i]	0.86	2.14	2.989 (2)	168
C8—H8A···O1[i]	0.96	2.52	3.204 (3)	129

Symmetry code: (i) −x, −y+2, −z+1.