

## (*E*)-*N'*-(3-Fluorobenzylidene)-3-nitrobenzohydrazide

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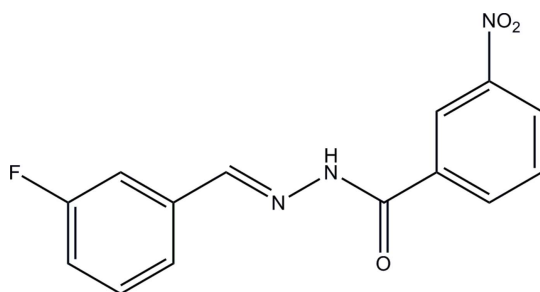
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 Key indicators: single-crystal X-ray study;  $T = 298$  K; mean  $\sigma(\text{C}-\text{C}) = 0.005$  Å;  $R$  factor = 0.080;  $wR$  factor = 0.218; data-to-parameter ratio = 12.6.

In the title compound,  $\text{C}_{14}\text{H}_{10}\text{FN}_3\text{O}_3$ , the molecule exists in a *trans* conformation with respect to the methyldene unit. The dihedral angle between the benzene rings is  $5.1(2)^\circ$ . In the crystal, molecules are linked through  $\text{N}-\text{H}\cdots\text{O}$  hydrogen bonds, forming chains along the  $c$  axis.

### Related literature

For the syntheses and crystal structures of hydrazone compounds, see: Hashemian *et al.* (2011); Lei (2011); Shalash *et al.* (2010). For the crystal structures of similar compounds, reported recently by the author, see: Li (2011*a,b*).



### Experimental

#### Crystal data

|   |   |
|---|---|
| $\text{C}_{14}\text{H}_{10}\text{FN}_3\text{O}_3$ | $V = 1313.5(5) \text{ \AA}^3$             |
| $M_r = 287.25$                                    | $Z = 4$                                   |
| Monoclinic, $P2_1/c$                              | Mo $K\alpha$ radiation                    |
| $a = 11.823(2) \text{ \AA}$                       | $\mu = 0.11 \text{ mm}^{-1}$              |
| $b = 12.813(3) \text{ \AA}$                       | $T = 298 \text{ K}$                       |
| $c = 8.7020(17) \text{ \AA}$                      | $0.17 \times 0.17 \times 0.15 \text{ mm}$ |
| $\beta = 94.855(2)^\circ$                         |   |

#### Data collection

|   |  |
|---|--|
| Bruker SMART CCD area-detector diffractometer               | 9452 measured reflections              |
| Absorption correction: multi-scan (SADABS; Sheldrick, 1996) | 2429 independent reflections           |
| $T_{\min} = 0.981$ , $T_{\max} = 0.983$                     | 1438 reflections with $I > 2\sigma(I)$ |
|   | $R_{\text{int}} = 0.075$               |

#### Refinement

|                                 |  |
|---------------------------------|--|
| $R[F^2 > 2\sigma(F^2)] = 0.080$ | H atoms treated by a mixture of independent and constrained refinement |
| $wR(F^2) = 0.218$               | $\Delta\rho_{\text{max}} = 0.59 \text{ e \AA}^{-3}$                    |
| $S = 1.02$                      | $\Delta\rho_{\text{min}} = -0.22 \text{ e \AA}^{-3}$                   |
| 2429 reflections                |  |
| 193 parameters                  |  |
| 1 restraint                     |  |

Table 1

Hydrogen-bond geometry (Å, °).

| $D-\text{H}\cdots A$                   | $D-\text{H}$ | $\text{H}\cdots A$ | $D\cdots A$ | $D-\text{H}\cdots A$ |
|--|--------------|--------------------|-------------|----------------------|
| $\text{N2}-\text{H2}\cdots\text{O3}^i$ | 0.90 (1)     | 1.96 (1)           | 2.846 (4)   | 172 (5)              |

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

Data collection: SMART (Bruker, 1998); cell refinement: SAINT (Bruker, 1998); 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.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RZ2708).

### References

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## supporting information

*Acta Cryst.* (2012). E68, o697 [doi:10.1107/S1600536812005478]

**(*E*)-*N'*-(3-Fluorobenzylidene)-3-nitrobenzohydrazide**

Xiao-Yan Li

**S1. Comment**

In recent years, hydrazone compounds have attracted much attention due to their syntheses and crystal structures (Hashemian *et al.*, 2011; Lei, 2011; Shalash *et al.*, 2010). As a continuation of our work on such compounds (Li, 2011*a,b*), the author reports herein on the crystal structure of the new title hydrazone compound.

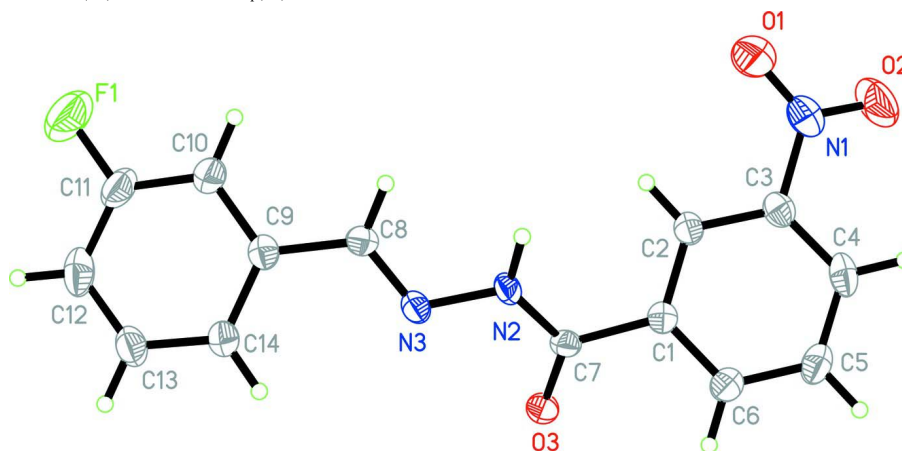
The title compound (Fig. 1) exists in a *trans* configuration with respect to the methyldene unit. The dihedral angle between the C1–C6 and C9–C14 benzene rings of the molecule is 5.1 (2)°. The N1/O1/O2 nitro group is tilted by 16.3 (3)° with respect to the C1–C6 benzene ring. In the crystal, molecules are linked through N–H···O hydrogen bonds (Table 1) to form chains along the *c* axis (Fig. 2).

**S2. Experimental**

A mixture of 3-fluorobenzaldehyde (0.124 g, 1 mmol) and 3-nitrobenzohydrazide (0.181 g, 1 mmol) in 30 ml of ethanol containing few drops of acetic acid was refluxed for about 1 h. On cooling to room temperature, a solid precipitate was formed. The solid was filtered and then recrystallized from methanol. Yellow crystals, suitable for X-ray diffraction analysis, were obtained by slow evaporation of the solvent.

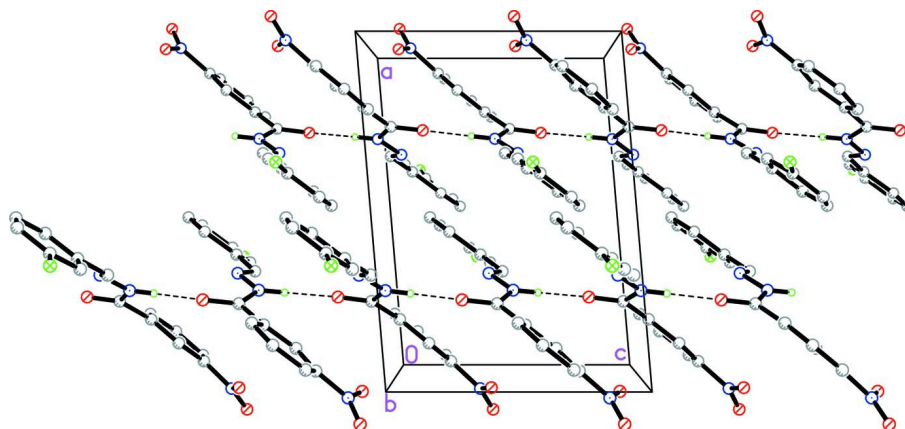
**S3. Refinement**

The amino H atom was located from a difference Fourier map and was refined isotropically with the N–H distance restrained to 0.90 (1) Å. The remaining H-atoms were positioned geometrically and refined using a riding model, with C–H = 0.93 Å, and with  $U_{\text{iso}}(\text{H})$  set to  $1.2U_{\text{eq}}(\text{C})$ .



**Figure 1**

The molecular structure of the title compound, showing displacement ellipsoids drawn at the 30% probability level.

**Figure 2**

Molecular packing diagram of the title compound, viewed along the *b* axis. Hydrogen bonds are indicated by dashed lines. The C-bound H-atoms have been omitted for clarity.

### (*E*)-*N'*-(3-Fluorobenzylidene)-3-nitrobenzohydrazide

#### Crystal data

$C_{14}H_{10}FN_3O_3$

$M_r = 287.25$

Monoclinic,  $P2_1/c$

Hall symbol:  $-P\ 2ybc$

$a = 11.823\ (2)\ \text{\AA}$

$b = 12.813\ (3)\ \text{\AA}$

$c = 8.7020\ (17)\ \text{\AA}$

$\beta = 94.855\ (2)^\circ$

$V = 1313.5\ (5)\ \text{\AA}^3$

$Z = 4$

$F(000) = 592$

$D_x = 1.453\ \text{Mg m}^{-3}$

Mo  $K\alpha$  radiation,  $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 690 reflections

$\theta = 2.4\text{--}26.2^\circ$

$\mu = 0.11\ \text{mm}^{-1}$

$T = 298\ \text{K}$

Block, yellow

$0.17 \times 0.17 \times 0.15\ \text{mm}$

#### Data collection

Bruker SMART CCD area-detector  
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

$\omega$  scans

Absorption correction: multi-scan  
(*SADABS*; Sheldrick, 1996)

$T_{\min} = 0.981$ ,  $T_{\max} = 0.983$

9452 measured reflections

2429 independent reflections

1438 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.075$

$\theta_{\max} = 25.5^\circ$ ,  $\theta_{\min} = 1.7^\circ$

$h = -14 \rightarrow 14$

$k = -15 \rightarrow 15$

$l = -10 \rightarrow 10$

#### Refinement

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.080$

$wR(F^2) = 0.218$

$S = 1.02$

2429 reflections

193 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

$w = 1/[\sigma^2(F_o^2) + (0.0985P)^2 + 0.4222P]$

where  $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{\max} = 0.59\ \text{e \AA}^{-3}$

$\Delta\rho_{\min} = -0.22\ \text{e \AA}^{-3}$

*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 ( $\text{\AA}^2$ )*

|     | <i>x</i>   | <i>y</i>     | <i>z</i>   | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|------------|--------------|------------|----------------------------------|
| F1  | 0.6421 (4) | -0.2974 (2)  | 1.1301 (4) | 0.1160 (14)                      |
| N1  | 1.0252 (3) | 0.4021 (4)   | 0.6545 (5) | 0.0679 (12)                      |
| N2  | 0.7281 (3) | 0.2345 (2)   | 0.9891 (3) | 0.0395 (8)                       |
| N3  | 0.6810 (3) | 0.1600 (2)   | 1.0796 (3) | 0.0378 (8)                       |
| O1  | 1.0149 (4) | 0.3120 (3)   | 0.6079 (6) | 0.1118 (16)                      |
| O2  | 1.0930 (3) | 0.4628 (3)   | 0.6109 (4) | 0.0929 (13)                      |
| O3  | 0.7513 (2) | 0.34723 (19) | 1.1894 (3) | 0.0479 (8)                       |
| C1  | 0.8197 (3) | 0.3996 (3)   | 0.9543 (4) | 0.0342 (9)                       |
| C2  | 0.8882 (3) | 0.3651 (3)   | 0.8433 (4) | 0.0375 (9)                       |
| H2A | 0.8947     | 0.2943       | 0.8222     | 0.045*                           |
| C3  | 0.9466 (3) | 0.4386 (3)   | 0.7646 (4) | 0.0427 (10)                      |
| C4  | 0.9357 (4) | 0.5431 (3)   | 0.7881 (5) | 0.0527 (11)                      |
| H4  | 0.9744     | 0.5910       | 0.7318     | 0.063*                           |
| C5  | 0.8655 (4) | 0.5763 (3)   | 0.8979 (5) | 0.0538 (11)                      |
| H5  | 0.8568     | 0.6473       | 0.9156     | 0.065*                           |
| C6  | 0.8089 (3) | 0.5051 (3)   | 0.9806 (5) | 0.0445 (10)                      |
| H6  | 0.7628     | 0.5282       | 1.0551     | 0.053*                           |
| C7  | 0.7631 (3) | 0.3250 (3)   | 1.0550 (4) | 0.0354 (9)                       |
| C8  | 0.6781 (3) | 0.0680 (3)   | 1.0258 (4) | 0.0367 (9)                       |
| H8  | 0.7070     | 0.0547       | 0.9316     | 0.044*                           |
| C9  | 0.6300 (3) | -0.0173 (3)  | 1.1103 (4) | 0.0385 (9)                       |
| C10 | 0.6562 (4) | -0.1197 (3)  | 1.0745 (5) | 0.0529 (11)                      |
| H10 | 0.7025     | -0.1344      | 0.9961     | 0.064*                           |
| C11 | 0.6121 (4) | -0.1984 (3)  | 1.1575 (5) | 0.0619 (13)                      |
| C12 | 0.5428 (4) | -0.1806 (4)  | 1.2734 (5) | 0.0634 (13)                      |
| H12 | 0.5138     | -0.2358      | 1.3273     | 0.076*                           |
| C13 | 0.5176 (4) | -0.0806 (4)  | 1.3071 (5) | 0.0546 (12)                      |
| H13 | 0.4702     | -0.0673      | 1.3848     | 0.066*                           |
| C14 | 0.5605 (3) | 0.0019 (3)   | 1.2291 (4) | 0.0447 (10)                      |
| H14 | 0.5433     | 0.0701       | 1.2555     | 0.054*                           |
| H2  | 0.736 (4)  | 0.215 (4)    | 0.891 (2)  | 0.080*                           |

*Atomic displacement parameters ( $\text{\AA}^2$ )*

|    | $U^{11}$  | $U^{22}$    | $U^{33}$  | $U^{12}$   | $U^{13}$  | $U^{23}$     |
|----|-----------|-------------|-----------|------------|-----------|--------------|
| F1 | 0.192 (4) | 0.0477 (18) | 0.111 (3) | -0.004 (2) | 0.029 (3) | -0.0008 (17) |

|     |             |             |             |              |              |              |
|-----|-------------|-------------|-------------|--------------|--------------|--------------|
| N1  | 0.065 (3)   | 0.073 (3)   | 0.069 (3)   | -0.009 (2)   | 0.028 (2)    | 0.006 (2)    |
| N2  | 0.054 (2)   | 0.0383 (18) | 0.0278 (16) | -0.0094 (16) | 0.0141 (15)  | 0.0014 (14)  |
| N3  | 0.0390 (18) | 0.0411 (19) | 0.0342 (17) | -0.0043 (15) | 0.0084 (13)  | 0.0036 (14)  |
| O1  | 0.133 (4)   | 0.077 (3)   | 0.139 (4)   | -0.010 (3)   | 0.088 (3)    | -0.010 (3)   |
| O2  | 0.087 (3)   | 0.109 (3)   | 0.089 (3)   | -0.039 (2)   | 0.042 (2)    | 0.002 (2)    |
| O3  | 0.0715 (19) | 0.0427 (16) | 0.0312 (14) | -0.0021 (14) | 0.0154 (13)  | -0.0040 (12) |
| C1  | 0.036 (2)   | 0.036 (2)   | 0.0298 (18) | -0.0029 (17) | -0.0025 (16) | -0.0001 (16) |
| C2  | 0.041 (2)   | 0.039 (2)   | 0.033 (2)   | -0.0069 (18) | 0.0019 (17)  | 0.0001 (16)  |
| C3  | 0.039 (2)   | 0.049 (3)   | 0.041 (2)   | -0.0064 (18) | 0.0036 (17)  | 0.0022 (18)  |
| C4  | 0.058 (3)   | 0.053 (3)   | 0.048 (2)   | -0.021 (2)   | 0.004 (2)    | 0.013 (2)    |
| C5  | 0.067 (3)   | 0.034 (2)   | 0.059 (3)   | -0.007 (2)   | 0.000 (2)    | 0.003 (2)    |
| C6  | 0.048 (2)   | 0.040 (2)   | 0.044 (2)   | 0.0012 (19)  | 0.0018 (19)  | -0.0007 (18) |
| C7  | 0.040 (2)   | 0.037 (2)   | 0.031 (2)   | 0.0026 (17)  | 0.0073 (16)  | -0.0030 (16) |
| C8  | 0.037 (2)   | 0.039 (2)   | 0.035 (2)   | 0.0036 (17)  | 0.0050 (16)  | -0.0006 (17) |
| C9  | 0.039 (2)   | 0.041 (2)   | 0.035 (2)   | -0.0062 (18) | -0.0013 (17) | 0.0014 (17)  |
| C10 | 0.069 (3)   | 0.039 (2)   | 0.052 (3)   | -0.003 (2)   | 0.014 (2)    | 0.001 (2)    |
| C11 | 0.092 (4)   | 0.031 (2)   | 0.063 (3)   | -0.001 (2)   | 0.008 (3)    | 0.001 (2)    |
| C12 | 0.080 (3)   | 0.055 (3)   | 0.055 (3)   | -0.025 (3)   | 0.000 (2)    | 0.015 (2)    |
| C13 | 0.058 (3)   | 0.057 (3)   | 0.051 (3)   | -0.014 (2)   | 0.015 (2)    | 0.006 (2)    |
| C14 | 0.049 (2)   | 0.041 (2)   | 0.045 (2)   | -0.0040 (19) | 0.0083 (19)  | 0.0042 (18)  |

*Geometric parameters (Å, °)*

|          |            |            |           |
|----------|------------|------------|-----------|
| F1—C11   | 1.344 (5)  | C4—H4      | 0.9300    |
| N1—O2    | 1.201 (5)  | C5—C6      | 1.372 (6) |
| N1—O1    | 1.225 (5)  | C5—H5      | 0.9300    |
| N1—C3    | 1.467 (5)  | C6—H6      | 0.9300    |
| N2—C7    | 1.343 (5)  | C8—C9      | 1.459 (5) |
| N2—N3    | 1.383 (4)  | C8—H8      | 0.9300    |
| N2—H2    | 0.897 (10) | C9—C10     | 1.390 (6) |
| N3—C8    | 1.268 (4)  | C9—C14     | 1.396 (5) |
| O3—C7    | 1.223 (4)  | C10—C11    | 1.369 (6) |
| C1—C6    | 1.379 (5)  | C10—H10    | 0.9300    |
| C1—C2    | 1.384 (5)  | C11—C12    | 1.371 (7) |
| C1—C7    | 1.493 (5)  | C12—C13    | 1.353 (6) |
| C2—C3    | 1.384 (5)  | C12—H12    | 0.9300    |
| C2—H2A   | 0.9300     | C13—C14    | 1.376 (5) |
| C3—C4    | 1.362 (6)  | C13—H13    | 0.9300    |
| C4—C5    | 1.384 (6)  | C14—H14    | 0.9300    |
| O2—N1—O1 | 123.8 (4)  | O3—C7—N2   | 123.6 (3) |
| O2—N1—C3 | 118.4 (4)  | O3—C7—C1   | 120.3 (3) |
| O1—N1—C3 | 117.8 (4)  | N2—C7—C1   | 116.0 (3) |
| C7—N2—N3 | 118.5 (3)  | N3—C8—C9   | 120.6 (3) |
| C7—N2—H2 | 126 (3)    | N3—C8—H8   | 119.7     |
| N3—N2—H2 | 115 (3)    | C9—C8—H8   | 119.7     |
| C8—N3—N2 | 115.5 (3)  | C10—C9—C14 | 119.2 (4) |
| C6—C1—C2 | 119.8 (3)  | C10—C9—C8  | 119.4 (3) |

|           |           |             |           |
|-----------|-----------|-------------|-----------|
| C6—C1—C7  | 118.6 (3) | C14—C9—C8   | 121.4 (3) |
| C2—C1—C7  | 121.5 (3) | C11—C10—C9  | 118.4 (4) |
| C3—C2—C1  | 118.3 (4) | C11—C10—H10 | 120.8     |
| C3—C2—H2A | 120.9     | C9—C10—H10  | 120.8     |
| C1—C2—H2A | 120.9     | F1—C11—C10  | 118.9 (5) |
| C4—C3—C2  | 122.5 (4) | F1—C11—C12  | 118.1 (4) |
| C4—C3—N1  | 119.0 (4) | C10—C11—C12 | 122.9 (4) |
| C2—C3—N1  | 118.5 (4) | C13—C12—C11 | 118.2 (4) |
| C3—C4—C5  | 118.4 (4) | C13—C12—H12 | 120.9     |
| C3—C4—H4  | 120.8     | C11—C12—H12 | 120.9     |
| C5—C4—H4  | 120.8     | C12—C13—C14 | 121.6 (4) |
| C6—C5—C4  | 120.4 (4) | C12—C13—H13 | 119.2     |
| C6—C5—H5  | 119.8     | C14—C13—H13 | 119.2     |
| C4—C5—H5  | 119.8     | C13—C14—C9  | 119.7 (4) |
| C5—C6—C1  | 120.6 (4) | C13—C14—H14 | 120.2     |
| C5—C6—H6  | 119.7     | C9—C14—H14  | 120.2     |
| C1—C6—H6  | 119.7     |             |           |

*Hydrogen-bond geometry (Å, °)*

| <i>D</i> —H... <i>A</i> | <i>D</i> —H | H... <i>A</i> | <i>D</i> ... <i>A</i> | <i>D</i> —H... <i>A</i> |
|-------------------------|-------------|---------------|-----------------------|-------------------------|
| N2—H2...O3 <sup>i</sup> | 0.90 (1)    | 1.96 (1)      | 2.846 (4)             | 172 (5)                 |

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