# organic compounds

# 2-Fluoro-N'-(2-methoxybenzylidene)-benzohydrazide

## Cheng-Bi Xu,[a] Zong-Gui Wang,[a]* Yi Nan,[b] Ling Yuan,[c,d] Rong Wang[b] and Shu-Xiang Zhang[e]

[a]The Second Hospital of Jilin University, Changchun Jilin 130041, People's Republic of China, [b]Traditional Chinese Medicine College of Ningxia Medical University, Yinchuan Ningxia 750004, People's Republic of China, [c]Pharmacy College of Ningxia Medical University, Yinchuan Ningxia 750004, People's Republic of China, [d]Minority Traditional Medical Center of Minzu University of China, Beijing 100081, People's Republic of China, and [e]Affiliated Hospital of Ningxia Medical University, Yinchuan Ningxia 750004, People's Republic of China
Correspondence e-mail: nanyiailing10@yeah.net

The molecule of the title compound, $C_{15}H_{13}FN_2O_2$, exists in a *trans* configuration with respect to the methylidene unit. The two benzene rings form a dihedral angle of of 64.7 (2)°. In the crystal, molecules are linked through N—H···O hydrogen bonds into chains propagating along the *c* axis.

## Related literature

For the reference bond lengths, see: Allen *et al.* (1987). For structural studies of hydrazone compounds, see: Han & Zhao (2010); Zhou & Yang (2010); Huang & Wu (2010); Shalash *et al.* (2010). For a related structure, see: Xu *et al.* (2011).
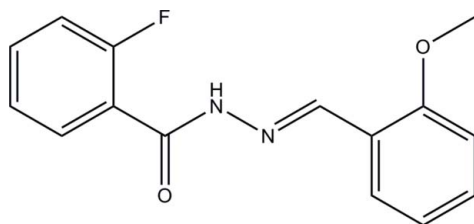


## Experimental

### Crystal data

C₁₅H₁₃FN₂O₂       $M_r$ = 272.27

Monoclinic, $P2_1/c$
$a$ = 13.612 (2) Å
$b$ = 12.278 (2) Å
$c$ = 7.925 (1) Å
$\beta$ = 92.802 (3)°
$V$ = 1322.9 (3) Å³

$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.10 mm⁻¹
$T$ = 298 K
0.17 × 0.13 × 0.12 mm

### Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min}$ = 0.983, $T_{max}$ = 0.988

10047 measured reflections
2821 independent reflections
1643 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.047

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.164
$S$ = 1.03
2821 reflections

182 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.47 e Å⁻³
$\Delta\rho_{min}$ = −0.26 e Å⁻³

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1—H1···O1$^i$ | 0.86 | 2.08 | 2.866 (2) | 152 |

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

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*.

## References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Bruker (1998). *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Han, Y.-Y. & Zhao, Q.-R. (2010). *Acta Cryst.* E**66**, o1041.
Huang, H.-T. & Wu, H.-Y. (2010). *Acta Cryst.* E**66**, o2729–o2730.
Shalash, M., Salhin, A., Adnan, R., Yeap, C. S. & Fun, H.-K. (2010). *Acta Cryst.* E**66**, o3126–o3127.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Xu, C.-B., Wang, Z.-G., Nan, Y., Yuan, L., Wang, R. & Zhang, S.-X. (2011). *Acta Cryst.* E**67**, o69.
Zhou, C.-S. & Yang, T. (2010). *Acta Cryst.* E**66**, o752.

# supporting information

## 2-Fluoro-*N*′-(2-methoxybenzylidene)benzohydrazide

Cheng-Bi Xu, Zong-Gui Wang, Yi Nan, Ling Yuan, Rong Wang and Shu-Xiang Zhang

### S1. Comment

As a contribution to a structural study of hydrazone compounds (Han & Zhao, 2010; Zhou & Yang, 2010; Huang & Wu, 2010; Shalash *et al.*, 2010), we present here the crystal structure of the title compound.

The molecule of the title compound exists in a *trans* configuration with respect to the methylidene unit (Fig. 1). The molecule is twisted, with the dihedral angle between the two benzene rings of 64.7 (2)°. The torsion angle C7—N1—N2—C8 is 8.6 (2)°. The bond lengths are within normal ranges (Allen *et al.*, 1987) and are comparable with those observed in the similar compound (Xu *et al.*, 2011).

In the crystal structure, molecules are linked through intermolecular N—H⋯O hydrogen bonds (Table 1), to form chains down the *c* axis (Fig. 2).

### S2. Experimental

2-Methoxybenzaldehyde (0.1 mmol, 13.6 mg) and 2-fluorobenzohydrazide (0.1 mmol, 15.4 mg) were mixed in ethanol (20 ml). The mixture was stirred at room temperature to give a clear colorless solution. Colourless well shaped crystals of the title compound were formed by gradual evaporation of the solvent over a period of five days at room temperature.

### S3. Refinement

All H atoms were placed in geometrically idealized positions, with C—H = 0.93-0.96 Å, N—H = 0.86 Å, and refined as riding, with $U_{iso}(H) = 1.2U_{eq}(C,N)$ and $1.5U_{eq}(C15)$.
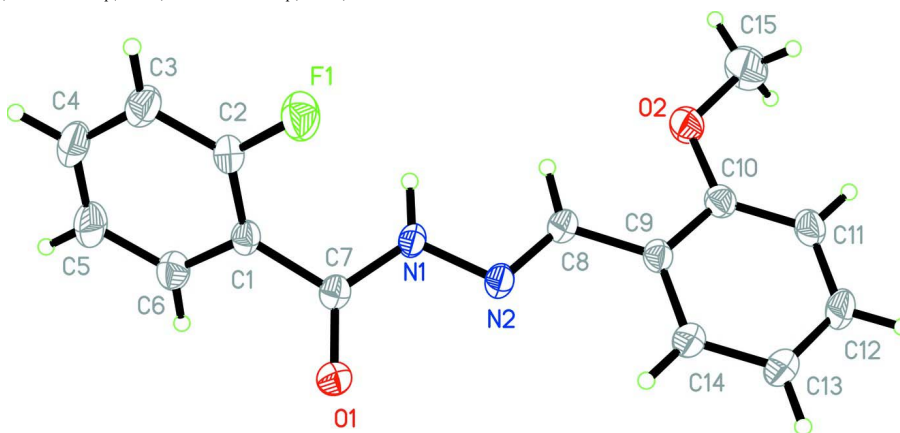


**Figure 1**

The molecular structure of the title compound, with displacement ellipsoids drawn at the 30% probability level.
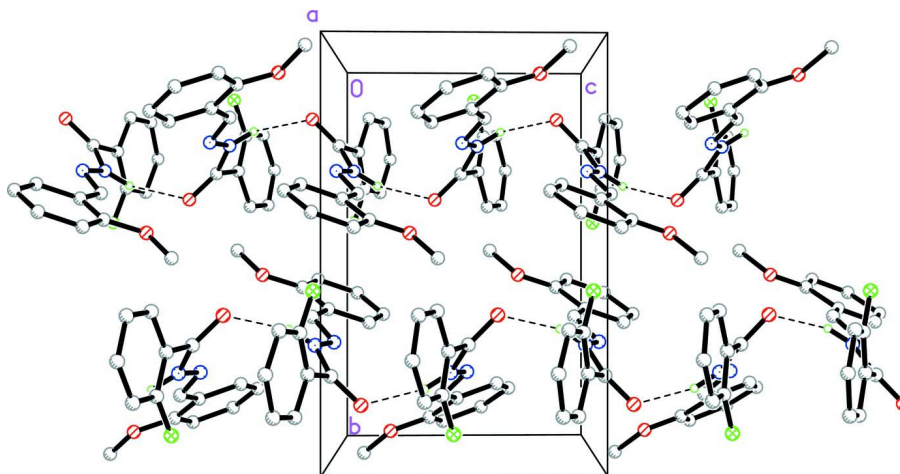
**Figure 2**

A portion of the crystal packing showing the hydrogen-bonded (dashed lines) chains of the molecules. H atoms omitted for clarity/

**2-Fluoro-*N′*-(2-methoxybenzylidene)benzohydrazide**

*Crystal data*

$C_{15}H_{13}FN_2O_2$
$M_r = 272.27$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 13.612 (2)$ Å
$b = 12.278 (2)$ Å
$c = 7.925 (1)$ Å
$\beta = 92.802 (3)°$
$V = 1322.9 (3)$ Å$^3$
$Z = 4$

$F(000) = 568$
$D_x = 1.367$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 1625 reflections
$\theta = 2.2–26.4°$
$\mu = 0.10$ mm$^{-1}$
$T = 298$ K
Block, colorless
$0.17 \times 0.13 \times 0.12$ 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)
$T_{min} = 0.983$, $T_{max} = 0.988$

10047 measured reflections
2821 independent reflections
1643 reflections with $I > 2\sigma(I)$
$R_{int} = 0.047$
$\theta_{max} = 27.0°$, $\theta_{min} = 1.5°$
$h = -17\rightarrow16$
$k = -15\rightarrow15$
$l = -10\rightarrow10$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.058$
$wR(F^2) = 0.164$
$S = 1.03$
2821 reflections
182 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
$w = 1/[\sigma^2(F_o^2) + (0.0839P)^2]$
    where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.47$ e Å$^{-3}$
$\Delta\rho_{min} = -0.26$ e Å$^{-3}$

*Special details*

**Geometry**. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 $F^2 > 2\mathrm{sigma}(F^2)$ 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 | 0.89639 (10) | 0.58503 (11) | 0.9614 (2) | 0.0657 (5) |
| N1 | 0.72769 (12) | 0.71726 (15) | 0.9490 (2) | 0.0409 (5) |
| H1 | 0.7427 | 0.6742 | 0.8687 | 0.049* |
| N2 | 0.63391 (12) | 0.71379 (15) | 1.0089 (2) | 0.0392 (5) |
| O1 | 0.77942 (11) | 0.85629 (12) | 1.1218 (2) | 0.0442 (4) |
| O2 | 0.43863 (11) | 0.55230 (14) | 0.7104 (2) | 0.0561 (5) |
| C1 | 0.89489 (15) | 0.77601 (18) | 0.9435 (3) | 0.0369 (5) |
| C2 | 0.94195 (16) | 0.67840 (18) | 0.9204 (3) | 0.0414 (6) |
| C3 | 1.03448 (17) | 0.6706 (2) | 0.8625 (3) | 0.0546 (7) |
| H3 | 1.0642 | 0.6030 | 0.8504 | 0.065* |
| C4 | 1.08295 (18) | 0.7651 (3) | 0.8224 (4) | 0.0603 (8) |
| H4 | 1.1458 | 0.7614 | 0.7817 | 0.072* |
| C5 | 1.03917 (19) | 0.8636 (2) | 0.8423 (4) | 0.0615 (8) |
| H5 | 1.0721 | 0.9269 | 0.8143 | 0.074* |
| C6 | 0.94636 (17) | 0.8701 (2) | 0.9034 (3) | 0.0508 (7) |
| H6 | 0.9177 | 0.9379 | 0.9182 | 0.061* |
| C7 | 0.79557 (15) | 0.78675 (18) | 1.0143 (3) | 0.0367 (6) |
| C8 | 0.57425 (15) | 0.65389 (18) | 0.9208 (3) | 0.0384 (6) |
| H8 | 0.5954 | 0.6201 | 0.8240 | 0.046* |
| C9 | 0.47321 (15) | 0.63784 (17) | 0.9704 (3) | 0.0376 (6) |
| C10 | 0.40509 (16) | 0.58424 (17) | 0.8620 (3) | 0.0418 (6) |
| C11 | 0.31043 (16) | 0.56584 (19) | 0.9121 (4) | 0.0504 (7) |
| H11 | 0.2652 | 0.5297 | 0.8403 | 0.061* |
| C12 | 0.28342 (17) | 0.6008 (2) | 1.0670 (4) | 0.0591 (8) |
| H12 | 0.2197 | 0.5881 | 1.0996 | 0.071* |
| C13 | 0.34873 (18) | 0.6544 (2) | 1.1753 (4) | 0.0607 (8) |
| H13 | 0.3295 | 0.6781 | 1.2801 | 0.073* |
| C14 | 0.44315 (17) | 0.6725 (2) | 1.1266 (3) | 0.0500 (7) |
| H14 | 0.4876 | 0.7086 | 1.1998 | 0.060* |
| C15 | 0.3743 (2) | 0.4906 (3) | 0.6007 (4) | 0.0838 (10) |
| H15A | 0.3546 | 0.4257 | 0.6575 | 0.126* |
| H15B | 0.4076 | 0.4711 | 0.5010 | 0.126* |
| H15C | 0.3172 | 0.5333 | 0.5694 | 0.126* |

*Atomic displacement parameters (Å²)*

|  | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| F1 | 0.0498 (9) | 0.0509 (9) | 0.0966 (13) | −0.0034 (7) | 0.0045 (8) | 0.0114 (9) |
| N1 | 0.0280 (10) | 0.0526 (12) | 0.0425 (12) | −0.0043 (8) | 0.0072 (9) | −0.0084 (9) |
| N2 | 0.0299 (10) | 0.0461 (11) | 0.0420 (12) | −0.0021 (8) | 0.0045 (9) | 0.0006 (9) |
| O1 | 0.0435 (9) | 0.0482 (9) | 0.0412 (10) | −0.0005 (7) | 0.0046 (8) | −0.0040 (8) |
| O2 | 0.0504 (10) | 0.0698 (12) | 0.0480 (11) | −0.0142 (9) | 0.0016 (9) | −0.0147 (9) |
| C1 | 0.0306 (11) | 0.0478 (13) | 0.0323 (13) | −0.0031 (10) | 0.0004 (10) | 0.0015 (11) |
| C2 | 0.0331 (13) | 0.0460 (14) | 0.0449 (15) | −0.0051 (11) | −0.0007 (11) | 0.0034 (12) |
| C3 | 0.0377 (14) | 0.0665 (17) | 0.0593 (18) | 0.0072 (12) | 0.0019 (13) | −0.0005 (14) |
| C4 | 0.0358 (14) | 0.083 (2) | 0.0631 (19) | −0.0034 (14) | 0.0111 (13) | 0.0023 (16) |
| C5 | 0.0482 (16) | 0.0693 (19) | 0.068 (2) | −0.0163 (14) | 0.0163 (14) | 0.0086 (16) |
| C6 | 0.0441 (14) | 0.0538 (15) | 0.0550 (17) | −0.0073 (12) | 0.0078 (13) | 0.0024 (13) |
| C7 | 0.0349 (12) | 0.0434 (13) | 0.0319 (13) | −0.0011 (10) | 0.0016 (10) | 0.0050 (11) |
| C8 | 0.0322 (12) | 0.0444 (13) | 0.0387 (14) | 0.0013 (10) | 0.0029 (10) | 0.0020 (11) |
| C9 | 0.0313 (12) | 0.0347 (12) | 0.0470 (15) | 0.0000 (9) | 0.0021 (11) | 0.0021 (11) |
| C10 | 0.0393 (13) | 0.0362 (13) | 0.0497 (16) | −0.0007 (10) | 0.0009 (12) | −0.0001 (11) |
| C11 | 0.0333 (13) | 0.0487 (15) | 0.069 (2) | −0.0054 (11) | −0.0012 (13) | −0.0069 (13) |
| C12 | 0.0325 (13) | 0.0613 (16) | 0.085 (2) | −0.0047 (12) | 0.0172 (14) | −0.0060 (16) |
| C13 | 0.0435 (15) | 0.0712 (18) | 0.069 (2) | −0.0037 (13) | 0.0179 (14) | −0.0168 (16) |
| C14 | 0.0390 (14) | 0.0585 (16) | 0.0531 (17) | −0.0051 (11) | 0.0087 (12) | −0.0129 (13) |
| C15 | 0.080 (2) | 0.112 (3) | 0.059 (2) | −0.0263 (19) | −0.0072 (17) | −0.0299 (19) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| F1—C2 | 1.351 (2) | C5—H5 | 0.9300 |
| N1—C7 | 1.343 (3) | C6—H6 | 0.9300 |
| N1—N2 | 1.384 (2) | C8—C9 | 1.462 (3) |
| N1—H1 | 0.8600 | C8—H8 | 0.9300 |
| N2—C8 | 1.278 (3) | C9—C14 | 1.390 (3) |
| O1—C7 | 1.234 (3) | C9—C10 | 1.397 (3) |
| O2—C10 | 1.364 (3) | C10—C11 | 1.385 (3) |
| O2—C15 | 1.422 (3) | C11—C12 | 1.368 (4) |
| C1—C2 | 1.375 (3) | C11—H11 | 0.9300 |
| C1—C6 | 1.395 (3) | C12—C13 | 1.373 (4) |
| C1—C7 | 1.495 (3) | C12—H12 | 0.9300 |
| C2—C3 | 1.365 (3) | C13—C14 | 1.378 (3) |
| C3—C4 | 1.379 (4) | C13—H13 | 0.9300 |
| C3—H3 | 0.9300 | C14—H14 | 0.9300 |
| C4—C5 | 1.361 (4) | C15—H15A | 0.9600 |
| C4—H4 | 0.9300 | C15—H15B | 0.9600 |
| C5—C6 | 1.377 (3) | C15—H15C | 0.9600 |
| | | | |
| C7—N1—N2 | 121.09 (19) | N2—C8—H8 | 119.6 |
| C7—N1—H1 | 119.5 | C9—C8—H8 | 119.6 |
| N2—N1—H1 | 119.5 | C14—C9—C10 | 118.4 (2) |
| C8—N2—N1 | 113.74 (19) | C14—C9—C8 | 121.3 (2) |

| | | | |
|---|---|---|---|
| C10—O2—C15 | 117.99 (19) | C10—C9—C8 | 120.2 (2) |
| C2—C1—C6 | 116.6 (2) | O2—C10—C11 | 124.2 (2) |
| C2—C1—C7 | 124.2 (2) | O2—C10—C9 | 115.86 (19) |
| C6—C1—C7 | 119.1 (2) | C11—C10—C9 | 120.0 (2) |
| F1—C2—C3 | 117.6 (2) | C12—C11—C10 | 120.0 (2) |
| F1—C2—C1 | 119.0 (2) | C12—C11—H11 | 120.0 |
| C3—C2—C1 | 123.3 (2) | C10—C11—H11 | 120.0 |
| C2—C3—C4 | 118.6 (2) | C11—C12—C13 | 121.1 (2) |
| C2—C3—H3 | 120.7 | C11—C12—H12 | 119.4 |
| C4—C3—H3 | 120.7 | C13—C12—H12 | 119.4 |
| C5—C4—C3 | 120.3 (2) | C12—C13—C14 | 119.1 (3) |
| C5—C4—H4 | 119.9 | C12—C13—H13 | 120.4 |
| C3—C4—H4 | 119.9 | C14—C13—H13 | 120.4 |
| C4—C5—C6 | 120.4 (2) | C13—C14—C9 | 121.3 (2) |
| C4—C5—H5 | 119.8 | C13—C14—H14 | 119.4 |
| C6—C5—H5 | 119.8 | C9—C14—H14 | 119.4 |
| C5—C6—C1 | 120.8 (2) | O2—C15—H15A | 109.5 |
| C5—C6—H6 | 119.6 | O2—C15—H15B | 109.5 |
| C1—C6—H6 | 119.6 | H15A—C15—H15B | 109.5 |
| O1—C7—N1 | 124.29 (19) | O2—C15—H15C | 109.5 |
| O1—C7—C1 | 121.0 (2) | H15A—C15—H15C | 109.5 |
| N1—C7—C1 | 114.7 (2) | H15B—C15—H15C | 109.5 |
| N2—C8—C9 | 120.8 (2) | | |

### Hydrogen-bond geometry (Å, º)

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1—H1···O1[i] | 0.86 | 2.08 | 2.866 (2) | 152 |

Symmetry code: (i) $x$, −$y$+3/2, $z$−1/2.