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# (*E*)-*N*′-[1-(2-Hydroxyphenyl)ethylidene]-3-methoxybenzohydrazide

**Cong-Ming Li[a]* and Hong-Yan Ban[b]**

[a]College of Science, Shenyang University, Shenyang 110044, People's Republic of China, and [b]School of Chemical Engineering, University of Science and Technology Liaoning, Anshan 114051, People's Republic of China
Correspondence e-mail: hongyan_ban@163.com

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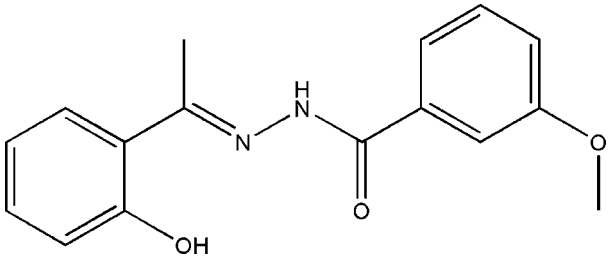
Key indicators: single-crystal X-ray study; $T$ = 298 K; mean $\sigma$(C–C) = 0.004 Å; $R$ factor = 0.057; $wR$ factor = 0.145; data-to-parameter ratio = 16.2.

In the title compound, $C_{16}H_{16}N_2O_3$, the benzohydrazide group is not planar and the molecule exists in a *trans* configuration with respect to the methylidene unit. The dihedral angle between the two substituted benzene rings is 26.9 (2)°. In the crystal structure, the molecular packing is stabilized by intramolecular O—H···N and intermolecular N—H···O hydrogen bonds. The intermolecular hydrogen bonding forms chains parallel to the *b* axis.

## Related literature

For the biological activities of hydrazones, see: Zhong *et al.* (2007); Raj *et al.* (2007); Jimenez-Pulido *et al.* (2008). For related structures, see: Ban & Li (2008*a*,*b*); Yehye *et al.* (2008); Fun *et al.* (2008*a*,*b*); Yang *et al.* (2008); Ejsmont *et al.* (2008).




## Experimental

### Crystal data

$C_{16}H_{16}N_2O_3$
$M_r$ = 284.31
Orthorhombic, *Pbca*
$a$ = 12.932 (2) Å
$b$ = 8.756 (2) Å
$c$ = 25.784 (3) Å
$V$ = 2919.7 (9) Å$^3$
$Z$ = 8
Mo $K\alpha$ radiation
$\mu$ = 0.09 mm$^{-1}$
$T$ = 298 K
0.27 × 0.23 × 0.20 mm

### Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min}$ = 0.976, $T_{max}$ = 0.982
22735 measured reflections
3180 independent reflections
2023 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.060

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.057
$wR(F^2)$ = 0.145
$S$ = 1.01
3180 reflections
196 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.17 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.13 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N2—H2···O2$^{i}$ | 0.897 (10) | 2.010 (11) | 2.894 (2) | 168 (2) |
| O1—H1···N1 | 0.82 | 1.82 | 2.534 (2) | 145 |

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

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: BX2198).

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# supplementary materials

*Acta Cryst.* (2009). E**65**, o876 [ **doi:10.1107/S1600536809010228** ]

# (*E*)-*N*'-[1-(2-Hydroxyphenyl)ethylidene]-3-methoxybenzohydrazide

**C.-M. Li and H.-Y. Ban**

## Comment

Hydrazones derived from the condensation of aldehydes with hydrazides have been demonstrated to possess excellent biological activities (Zhong *et al.*, 2007; Raj *et al.*, 2007; Jimenez-Pulido *et al.*, 2008). Due to the easy synthesis of such compounds, a great deal of hydrazones have been synthesized and structurally characterized (Yehye *et al.*, 2008; Fun *et al.*, 2008a,b; Yang *et al.*, 2008; Ejsmont *et al.*, 2008). Recently, we have reported two hydrazones (Ban & Li, 2008a,b). In this paper, we report herein the crystal structure of the title compound, (I). In the structure of (I), Fig. 1, the molecule exists in a *trans* configuration with respect to the methylidene unit. The dihedral angle between the two substituted benzene rings is 26.9 (2)°. In the 3-methoxyphenyl unit, the methoxy group is nearly coplanar with the mean plane of the C10–C15 ring, with the C16 atom deviates from the plane by 0.024 (2) Å. The torsion angle of C7-N1-N2-C9 is 8.0 (3)°. In the crystal structure the molecular packing is stabilized by intramolecular O-H···N and intermolecular N—H···O hydrogen bonds, Table 1. The intermolecular hydrogen bond form chains parallel to the *b* axis, Fig. 2.

## Experimental

The compound was prepared by refluxing 1-(2-hydroxyphenyl)ethanone (1.0 mol, 0136 g ) with 3-methoxybenzohydrazide (1.0 mol), 0166 g) in methanol (100 ml). Excess methanol was removed from the mixture by distillation. The colorless solid product was filtered, and washed three times with methanol. Colorless block crystals of the title compound were obtained from a methanol solution by slow evaporation in air.

## Refinement

H2 was located in a difference Fourier map and refined isotropically, with N–H distance restrained to 0.90 (1) Å. Other H atoms were placed in calculated positions (C–H = 0.93 - 0.96 Å, O–H = 0.82 Å) and refined as riding with $U_{iso}$(H) = 1.2$U_{eq}$(C) and 1.5$U_{eq}$(O and methyl C). A rotating group model was used for the methyl groups.

## Figures

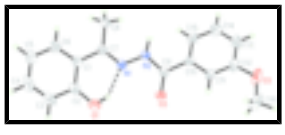

Fig. 1. The molecular structure of (I), showing 30% probability displacement ellipsoids for the non-hydrogen atoms. Intramolecular O—H···N is shown as a dashed line.

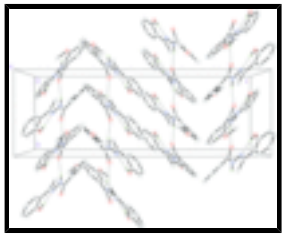

Fig. 2. The packing diagram of (I), viewed along the *a* axis. Hydrogen bonds are shown as dashed lines.

**(*E*)-*N*'-[1-(2-Hydroxyphenyl)ethylidene]-3-methoxybenzohydrazide**

*Crystal data*

| | |
|---|---|
| $C_{16}H_{16}N_2O_3$ | $F_{000}$ = 1200 |
| $M_r$ = 284.31 | $D_x$ = 1.294 Mg m$^{-3}$ |
| Orthorhombic, *Pbca* | Mo *K*α radiation<br>λ = 0.71073 Å |
| Hall symbol: -P 2ac 2ab | Cell parameters from 2030 reflections |
| *a* = 12.932 (2) Å | θ = 2.3–24.6º |
| *b* = 8.756 (2) Å | μ = 0.09 mm$^{-1}$ |
| *c* = 25.784 (3) Å | *T* = 298 K |
| *V* = 2919.7 (9) Å$^3$ | Block, colourless |
| *Z* = 8 | 0.27 × 0.23 × 0.20 mm |

*Data collection*

| | |
|---|---|
| Bruker SMART CCD area-detector diffractometer | 3180 independent reflections |
| Radiation source: fine-focus sealed tube | 2023 reflections with $I > 2\sigma(I)$ |
| Monochromator: graphite | $R_{int}$ = 0.060 |
| *T* = 298 K | $\theta_{max}$ = 27.0º |
| ω scans | $\theta_{min}$ = 1.6º |
| Absorption correction: multi-scan (SADABS; Sheldrick, 1996) | *h* = −16→16 |
| $T_{min}$ = 0.976, $T_{max}$ = 0.982 | *k* = −11→11 |
| 22735 measured reflections | *l* = −32→32 |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: inferred from neighbouring sites |
| $R[F^2 > 2\sigma(F^2)]$ = 0.057 | H atoms treated by a mixture of independent and constrained refinement |
| $wR(F^2)$ = 0.145 | $w = 1/[\sigma^2(F_o^2) + (0.0537P)^2 + 0.9645P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| *S* = 1.01 | $(\Delta/\sigma)_{max} < 0.001$ |
| 3180 reflections | $\Delta\rho_{max}$ = 0.17 e Å$^{-3}$ |
| 196 parameters | $\Delta\rho_{min}$ = −0.13 e Å$^{-3}$ |
| 1 restraint | Extinction correction: none |
| Primary atom site location: structure-invariant direct methods | |

*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 $F^2 > 2\text{sigma}(F^2)$ 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* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O1 | 0.36951 (13) | −0.23812 (18) | 0.55231 (7) | 0.0649 (5) |
| H1 | 0.3402 | −0.1716 | 0.5692 | 0.097* |
| O2 | 0.16810 (12) | −0.08166 (16) | 0.63341 (7) | 0.0637 (5) |
| O3 | −0.13883 (13) | 0.2606 (3) | 0.68697 (8) | 0.0904 (7) |
| N1 | 0.35910 (13) | −0.00001 (18) | 0.60790 (6) | 0.0449 (4) |
| N2 | 0.29750 (13) | 0.09206 (19) | 0.63821 (7) | 0.0455 (4) |
| C1 | 0.51395 (16) | −0.0694 (2) | 0.56716 (8) | 0.0464 (5) |
| C2 | 0.46801 (19) | −0.1972 (2) | 0.54406 (8) | 0.0531 (6) |
| C3 | 0.5262 (2) | −0.2881 (3) | 0.51064 (10) | 0.0698 (7) |
| H3 | 0.4956 | −0.3729 | 0.4953 | 0.084* |
| C4 | 0.6267 (3) | −0.2557 (3) | 0.49994 (11) | 0.0783 (9) |
| H4 | 0.6643 | −0.3189 | 0.4779 | 0.094* |
| C5 | 0.6727 (2) | −0.1300 (3) | 0.52166 (10) | 0.0726 (8) |
| H5 | 0.7413 | −0.1067 | 0.5141 | 0.087* |
| C6 | 0.61702 (17) | −0.0388 (3) | 0.55467 (9) | 0.0605 (6) |
| H6 | 0.6489 | 0.0462 | 0.5692 | 0.073* |
| C7 | 0.45502 (16) | 0.0315 (2) | 0.60247 (8) | 0.0460 (5) |
| C8 | 0.50836 (18) | 0.1605 (3) | 0.62968 (11) | 0.0736 (8) |
| H8A | 0.5115 | 0.2475 | 0.6071 | 0.110* |
| H8B | 0.5772 | 0.1299 | 0.6389 | 0.110* |
| H8C | 0.4706 | 0.1867 | 0.6605 | 0.110* |
| C9 | 0.20029 (16) | 0.0434 (2) | 0.64786 (8) | 0.0453 (5) |
| C10 | 0.13506 (15) | 0.1510 (2) | 0.67804 (7) | 0.0416 (5) |
| C11 | 0.02940 (15) | 0.1511 (2) | 0.66810 (8) | 0.0472 (5) |
| H11 | 0.0020 | 0.0846 | 0.6436 | 0.057* |
| C12 | −0.03443 (18) | 0.2490 (3) | 0.69451 (9) | 0.0587 (6) |
| C13 | 0.0058 (2) | 0.3454 (3) | 0.73150 (10) | 0.0744 (8) |
| H13 | −0.0375 | 0.4117 | 0.7494 | 0.089* |
| C14 | 0.1094 (2) | 0.3437 (3) | 0.74187 (10) | 0.0741 (8) |
| H14 | 0.1361 | 0.4087 | 0.7671 | 0.089* |
| C15 | 0.17528 (17) | 0.2464 (3) | 0.71537 (9) | 0.0551 (6) |
| H15 | 0.2457 | 0.2454 | 0.7227 | 0.066* |
| C16 | −0.1850 (2) | 0.1657 (4) | 0.64939 (12) | 0.0932 (10) |

| | | | | |
|---|---|---|---|---|
| H16A | −0.1543 | 0.1853 | 0.6162 | 0.140* |
| H16B | −0.2578 | 0.1865 | 0.6478 | 0.140* |
| H16C | −0.1743 | 0.0607 | 0.6586 | 0.140* |
| H2 | 0.3118 (19) | 0.1919 (13) | 0.6414 (10) | 0.080* |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0719 (12) | 0.0497 (10) | 0.0731 (12) | −0.0087 (9) | −0.0043 (9) | −0.0107 (8) |
| O2 | 0.0519 (9) | 0.0352 (8) | 0.1039 (13) | −0.0034 (7) | −0.0095 (9) | −0.0069 (8) |
| O3 | 0.0454 (10) | 0.1393 (19) | 0.0865 (14) | 0.0128 (11) | 0.0099 (10) | −0.0058 (13) |
| N1 | 0.0432 (10) | 0.0389 (9) | 0.0524 (10) | 0.0035 (8) | −0.0032 (8) | −0.0017 (8) |
| N2 | 0.0426 (10) | 0.0339 (9) | 0.0602 (11) | 0.0017 (8) | 0.0002 (8) | −0.0049 (9) |
| C1 | 0.0512 (13) | 0.0411 (12) | 0.0468 (12) | 0.0051 (10) | −0.0031 (10) | 0.0027 (9) |
| C2 | 0.0687 (16) | 0.0415 (13) | 0.0490 (13) | 0.0059 (11) | −0.0025 (11) | 0.0042 (10) |
| C3 | 0.104 (2) | 0.0451 (15) | 0.0607 (16) | 0.0093 (14) | 0.0061 (15) | −0.0058 (12) |
| C4 | 0.101 (2) | 0.0675 (18) | 0.0664 (17) | 0.0287 (17) | 0.0235 (16) | 0.0042 (14) |
| C5 | 0.0695 (17) | 0.0764 (19) | 0.0720 (17) | 0.0163 (15) | 0.0202 (14) | 0.0064 (15) |
| C6 | 0.0561 (15) | 0.0587 (15) | 0.0666 (15) | 0.0054 (12) | 0.0031 (12) | 0.0024 (12) |
| C7 | 0.0440 (12) | 0.0407 (11) | 0.0534 (13) | 0.0070 (9) | −0.0078 (10) | −0.0012 (10) |
| C8 | 0.0466 (14) | 0.0697 (17) | 0.105 (2) | 0.0040 (12) | −0.0096 (14) | −0.0346 (16) |
| C9 | 0.0429 (12) | 0.0363 (11) | 0.0567 (13) | 0.0008 (9) | −0.0096 (10) | 0.0052 (10) |
| C10 | 0.0436 (11) | 0.0387 (11) | 0.0427 (11) | −0.0043 (9) | −0.0015 (9) | 0.0073 (9) |
| C11 | 0.0445 (12) | 0.0505 (13) | 0.0466 (12) | −0.0060 (10) | 0.0016 (10) | 0.0068 (10) |
| C12 | 0.0467 (14) | 0.0763 (17) | 0.0531 (14) | 0.0014 (12) | 0.0097 (11) | 0.0084 (13) |
| C13 | 0.0687 (18) | 0.094 (2) | 0.0608 (16) | 0.0095 (15) | 0.0184 (14) | −0.0177 (15) |
| C14 | 0.0771 (19) | 0.092 (2) | 0.0529 (15) | −0.0094 (16) | 0.0051 (13) | −0.0249 (14) |
| C15 | 0.0464 (12) | 0.0690 (15) | 0.0498 (13) | −0.0057 (11) | −0.0005 (10) | −0.0029 (12) |
| C16 | 0.0430 (15) | 0.140 (3) | 0.097 (2) | −0.0071 (16) | −0.0068 (15) | 0.012 (2) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| O1—C2 | 1.340 (3) | C6—H6 | 0.9300 |
| O1—H1 | 0.8200 | C7—C8 | 1.498 (3) |
| O2—C9 | 1.229 (2) | C8—H8A | 0.9600 |
| O3—C12 | 1.368 (3) | C8—H8B | 0.9600 |
| O3—C16 | 1.409 (4) | C8—H8C | 0.9600 |
| N1—C7 | 1.278 (2) | C9—C10 | 1.485 (3) |
| N1—N2 | 1.377 (2) | C10—C15 | 1.376 (3) |
| N2—C9 | 1.351 (3) | C10—C11 | 1.390 (3) |
| N2—H2 | 0.897 (10) | C11—C12 | 1.371 (3) |
| C1—C6 | 1.397 (3) | C11—H11 | 0.9300 |
| C1—C2 | 1.401 (3) | C12—C13 | 1.376 (3) |
| C1—C7 | 1.480 (3) | C13—C14 | 1.367 (4) |
| C2—C3 | 1.394 (3) | C13—H13 | 0.9300 |
| C3—C4 | 1.358 (4) | C14—C15 | 1.385 (3) |
| C3—H3 | 0.9300 | C14—H14 | 0.9300 |
| C4—C5 | 1.371 (4) | C15—H15 | 0.9300 |
| C4—H4 | 0.9300 | C16—H16A | 0.9600 |

| | | | |
|---|---|---|---|
| C5—C6 | 1.372 (3) | C16—H16B | 0.9600 |
| C5—H5 | 0.9300 | C16—H16C | 0.9600 |
| C2—O1—H1 | 109.5 | C7—C8—H8C | 109.5 |
| C12—O3—C16 | 118.2 (2) | H8A—C8—H8C | 109.5 |
| C7—N1—N2 | 119.81 (17) | H8B—C8—H8C | 109.5 |
| C9—N2—N1 | 117.28 (17) | O2—C9—N2 | 122.7 (2) |
| C9—N2—H2 | 118.8 (16) | O2—C9—C10 | 122.1 (2) |
| N1—N2—H2 | 120.2 (17) | N2—C9—C10 | 115.14 (18) |
| C6—C1—C2 | 117.4 (2) | C15—C10—C11 | 120.0 (2) |
| C6—C1—C7 | 121.3 (2) | C15—C10—C9 | 122.46 (19) |
| C2—C1—C7 | 121.4 (2) | C11—C10—C9 | 117.50 (18) |
| O1—C2—C3 | 117.3 (2) | C12—C11—C10 | 120.0 (2) |
| O1—C2—C1 | 123.3 (2) | C12—C11—H11 | 120.0 |
| C3—C2—C1 | 119.3 (2) | C10—C11—H11 | 120.0 |
| C4—C3—C2 | 121.6 (3) | O3—C12—C11 | 124.7 (2) |
| C4—C3—H3 | 119.2 | O3—C12—C13 | 115.2 (2) |
| C2—C3—H3 | 119.2 | C11—C12—C13 | 120.0 (2) |
| C3—C4—C5 | 120.0 (3) | C14—C13—C12 | 120.0 (2) |
| C3—C4—H4 | 120.0 | C14—C13—H13 | 120.0 |
| C5—C4—H4 | 120.0 | C12—C13—H13 | 120.0 |
| C4—C5—C6 | 119.5 (3) | C13—C14—C15 | 120.9 (2) |
| C4—C5—H5 | 120.2 | C13—C14—H14 | 119.6 |
| C6—C5—H5 | 120.2 | C15—C14—H14 | 119.6 |
| C5—C6—C1 | 122.2 (2) | C10—C15—C14 | 119.1 (2) |
| C5—C6—H6 | 118.9 | C10—C15—H15 | 120.5 |
| C1—C6—H6 | 118.9 | C14—C15—H15 | 120.5 |
| N1—C7—C1 | 115.99 (19) | O3—C16—H16A | 109.5 |
| N1—C7—C8 | 123.94 (19) | O3—C16—H16B | 109.5 |
| C1—C7—C8 | 120.07 (19) | H16A—C16—H16B | 109.5 |
| C7—C8—H8A | 109.5 | O3—C16—H16C | 109.5 |
| C7—C8—H8B | 109.5 | H16A—C16—H16C | 109.5 |
| H8A—C8—H8B | 109.5 | H16B—C16—H16C | 109.5 |

*Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N2—H2···O2$^{i}$ | 0.897 (10) | 2.010 (11) | 2.894 (2) | 168 (2) |
| O1—H1···N1 | 0.82 | 1.82 | 2.534 (2) | 145 |

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

**Fig. 1**

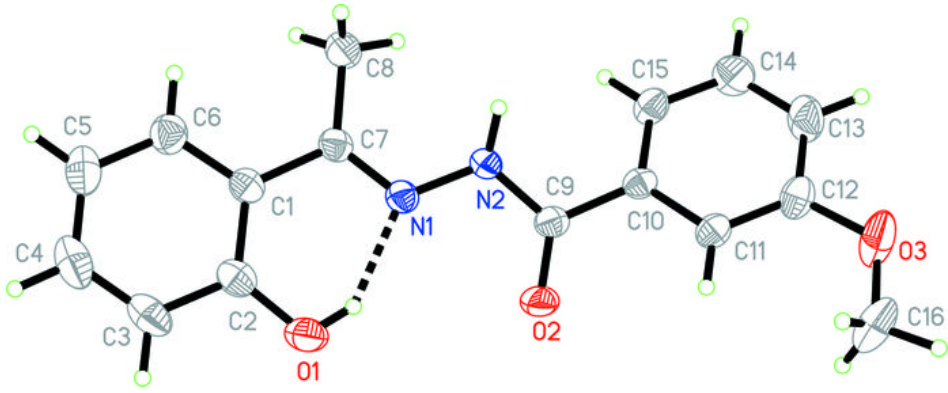

**Fig. 2**