

## *N'*-(2-Hydroxy-4-methoxybenzylidene)-4-methoxybenzohydrazide

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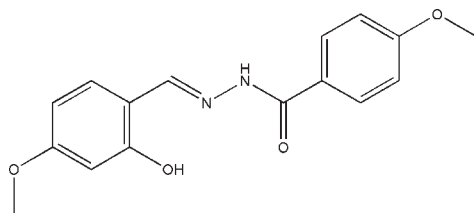
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Key indicators: single-crystal X-ray study;  $T = 298$  K; mean  $\sigma(\text{C}-\text{C}) = 0.003$  Å;  $R$  factor = 0.046;  $wR$  factor = 0.129; data-to-parameter ratio = 15.0.

In the title compound,  $\text{C}_{16}\text{H}_{16}\text{N}_2\text{O}_4$ , the dihedral angle between the two benzene rings is  $8.7(2)^\circ$ . The molecule adopts an *E* configuration about the  $\text{C}=\text{N}$  bond, with an intramolecular  $\text{O}-\text{H}\cdots\text{N}$  hydrogen bond involving the hydroxy substituent and the hydrazide N atom. In the crystal structure, adjacent molecules are linked through intermolecular  $\text{N}-\text{H}\cdots\text{O}$  hydrogen bonds, forming chains propagating in the *b*-axis direction.

### Related literature

For related structures, see: Alhadi *et al.* (2008); Küçükgülzel *et al.* (2003); Mohd Lair *et al.* (2009*a,b*); Li *et al.* (2009); Zhang *et al.* (2009). For a similar hydrazone compound, see: Zhang (2009). For reference structural data, see: Allen *et al.* (1987).



### Experimental

#### Crystal data

$\text{C}_{16}\text{H}_{16}\text{N}_2\text{O}_4$   
 $M_r = 300.31$   
 Monoclinic,  $P2_1/c$   
 $a = 17.692(2)$  Å

$b = 5.4131(7)$  Å  
 $c = 14.933(2)$  Å  
 $\beta = 97.431(7)^\circ$   
 $V = 1418.1(3)$  Å<sup>3</sup>

$Z = 4$   
 Mo  $K\alpha$  radiation  
 $\mu = 0.10$  mm<sup>-1</sup>

$T = 298$  K  
 $0.23 \times 0.20 \times 0.20$  mm

#### Data collection

Bruker SMART CCD area-detector diffractometer  
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)  
 $T_{\text{min}} = 0.977$ ,  $T_{\text{max}} = 0.980$

8164 measured reflections  
 3069 independent reflections  
 2001 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.033$

#### Refinement

$R[F^2 > 2\sigma(F^2)] = 0.046$   
 $wR(F^2) = 0.129$   
 $S = 1.03$   
 3069 reflections  
 205 parameters  
 1 restraint

H atoms treated by a mixture of independent and constrained refinement  
 $\Delta\rho_{\text{max}} = 0.16$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.16$  e Å<sup>-3</sup>

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{O1}-\text{H1}\cdots\text{N1}$	0.82	1.86	2.577 (2)	146
$\text{N2}-\text{H2}\cdots\text{O2}^i$	0.90 (1)	2.393 (11)	3.281 (2)	168 (2)

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

Data collection: SMART (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: SHELXTL (Sheldrick, 2008); software used to prepare material for publication: SHELXL97.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SU2137).

### References

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**supplementary materials**

*Acta Cryst.* (2009). E65, o2200 [ doi:10.1107/S1600536809032449 ]

## *N'*-(2-Hydroxy-4-methoxybenzylidene)-4-methoxybenzohydrazide

X. Zhang

### Comment

Hydrazone compounds are readily synthesized by the reaction of aldehydes with hydrazides. A large number of hydrazone compounds have been reported (Alhadi *et al.*, 2008; Küçükgülzel *et al.*, 2003; Li *et al.*, 2009; Zhang *et al.*, 2009). Recently, the author reported on the crystal structure of a hydrazone compound derived from the reaction of 2-methoxybenzaldehyde with 4-methoxybenzohydrazide (Zhang, 2009). Herein, the crystal structure of the new title hydrazone, prepared from the reaction of 2-hydroxy-4-methoxybenzaldehyde with 4-methoxybenzohydrazide, is reported on.

The molecule structure of the title compound is illustrated in Fig. 1. The molecule adopts an *E* configuration about the C=N bond. The dihedral angle involving the two benzene rings is 8.7 (2)°. There is an intramolecular O1—H1...N1 hydrogen bond, involving the hydroxyl substituent and the hydrazide N-atom (Table 1). All the bond lengths are within normal values (Allen *et al.*, 1987) and are comparable with those observed in similar compounds (Mohd Lair *et al.*, 2009a,b; Zhang, 2009).

In the crystal structure of the title compound adjacent molecules are linked through intermolecular N—H...O hydrogen bonds, forming chains propagating in *b* direction (Table 1 and Fig. 2).

### Experimental

2-Hydroxy-4-methoxybenzaldehyde (1.0 mmol, 152.2 mg) and 4-methoxybenzohydrazide (1.0 mmol, 166.2 mg) were mixed in a methanol solution, and the mixture was refluxed for 1 h. Colorless block-shaped crystals of the title compound were formed by slow evaporation of the solution in air.

### Refinement

Atom H2 attached to N2 was located from a difference electron-density map and freely refined with  $U_{\text{iso}}(\text{H})$  restrained to 0.08 (2) Å<sup>2</sup>. The other H-atoms were included in calculated positions and refined as riding atoms:  $d(\text{C—H}) = 0.93\text{--}0.96$  Å,  $d(\text{O—H}) = 0.82$  Å, with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$  and  $1.5U_{\text{eq}}(\text{C}_{\text{methyl}} \text{ and } \text{O})$ .

### Figures

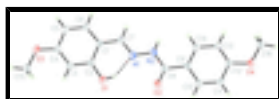


Fig. 1. A view of the molecular structure of the title compound, showing 30% displacement ellipsoids. The intramolecular N—H...O hydrogen bond is shown as a dashed line.

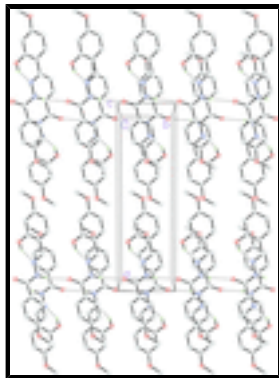


Fig. 2. A perspective view, along the *c*-axis, illustrating the infinite chain structure of the title compound. O-H $\cdots$ N and N-H $\cdots$ O hydrogen bonds are shown as dashed lines [see Table 1 for details; H atoms not involved in hydrogen bonding have been omitted for clarity].

***N'*-(2-Hydroxy-4-methoxybenzylidene)-4-methoxybenzohydrazide**

*Crystal data*

$C_{16}H_{16}N_2O_4$

$M_r = 300.31$

Monoclinic,  $P2_1/c$

Hall symbol: -P 2ybc

$a = 17.692 (2) \text{ \AA}$

$b = 5.4131 (7) \text{ \AA}$

$c = 14.933 (2) \text{ \AA}$

$\beta = 97.431 (7)^\circ$

$V = 1418.1 (3) \text{ \AA}^3$

$Z = 4$

$F_{000} = 632$

$D_x = 1.407 \text{ Mg m}^{-3}$

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

Cell parameters from 1647 reflections

$\theta = 2.3\text{--}24.5^\circ$

$\mu = 0.10 \text{ mm}^{-1}$

$T = 298 \text{ K}$

Block, colorless

$0.23 \times 0.20 \times 0.20 \text{ mm}$

*Data collection*

Bruker SMART CCD area-detector diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 298 \text{ K}$

$\omega$  scans

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

$T_{\min} = 0.977$ ,  $T_{\max} = 0.980$

8164 measured reflections

3069 independent reflections

2001 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.033$

$\theta_{\max} = 27.0^\circ$

$\theta_{\min} = 2.3^\circ$

$h = -21 \rightarrow 22$

$k = -6 \rightarrow 6$

$l = -17 \rightarrow 19$

*Refinement*

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.046$

$wR(F^2) = 0.129$

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

$w = 1/[\sigma^2(F_o^2) + (0.0559P)^2 + 0.2712P]$

$S = 1.03$	where $P = (F_o^2 + 2F_c^2)/3$
3069 reflections	$(\Delta/\sigma)_{\max} < 0.001$
205 parameters	$\Delta\rho_{\max} = 0.16 \text{ e } \text{\AA}^{-3}$
1 restraint	$\Delta\rho_{\min} = -0.16 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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

	$x$	$y$	$z$	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.25296 (7)	0.8356 (2)	0.42022 (10)	0.0497 (4)
H1	0.2079	0.7986	0.4079	0.075*
O2	0.03919 (7)	0.9056 (3)	0.38193 (10)	0.0550 (4)
O3	0.49908 (7)	0.5665 (3)	0.36425 (10)	0.0584 (4)
O4	-0.29179 (7)	0.4958 (3)	0.40281 (9)	0.0479 (4)
N1	0.13948 (8)	0.5576 (3)	0.35985 (10)	0.0426 (4)
N2	0.06360 (9)	0.5020 (3)	0.35871 (12)	0.0449 (4)
C1	0.26537 (10)	0.4485 (3)	0.34327 (12)	0.0375 (4)
C2	0.29666 (10)	0.6628 (3)	0.38635 (12)	0.0381 (4)
C3	0.37456 (10)	0.7056 (3)	0.39548 (13)	0.0419 (5)
H3	0.3949	0.8454	0.4258	0.050*
C4	0.42197 (10)	0.5403 (4)	0.35937 (13)	0.0425 (5)
C5	0.39204 (11)	0.3296 (4)	0.31433 (13)	0.0464 (5)
H5	0.4237	0.2202	0.2889	0.056*
C6	0.31544 (11)	0.2860 (4)	0.30805 (13)	0.0437 (5)
H6	0.2959	0.1430	0.2793	0.052*
C7	0.18490 (10)	0.3929 (4)	0.33728 (12)	0.0422 (5)
H7	0.1665	0.2389	0.3172	0.051*
C8	0.01619 (10)	0.6911 (4)	0.37443 (12)	0.0407 (4)
C9	-0.06413 (10)	0.6228 (3)	0.38074 (12)	0.0375 (4)
C10	-0.10700 (10)	0.7816 (4)	0.42781 (13)	0.0429 (5)
H10	-0.0843	0.9226	0.4548	0.052*
C11	-0.18213 (11)	0.7328 (4)	0.43479 (13)	0.0435 (5)
H11	-0.2100	0.8399	0.4666	0.052*
C12	-0.21659 (10)	0.5231 (3)	0.39429 (12)	0.0374 (4)

## supplementary materials

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C13	-0.17495 (10)	0.3632 (4)	0.34784 (13)	0.0437 (5)
H13	-0.1979	0.2228	0.3206	0.052*
C14	-0.09899 (10)	0.4126 (4)	0.34193 (13)	0.0436 (5)
H14	-0.0709	0.3030	0.3114	0.052*
C15	0.53412 (11)	0.7685 (4)	0.41459 (18)	0.0660 (7)
H15A	0.5154	0.9209	0.3873	0.099*
H15B	0.5884	0.7599	0.4150	0.099*
H15C	0.5222	0.7609	0.4754	0.099*
C16	-0.32941 (11)	0.2764 (4)	0.36696 (15)	0.0531 (5)
H16A	-0.3256	0.2651	0.3035	0.080*
H16B	-0.3821	0.2823	0.3759	0.080*
H16C	-0.3057	0.1346	0.3974	0.080*
H2	0.0500 (13)	0.341 (2)	0.3594 (16)	0.080*

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
O1	0.0404 (8)	0.0411 (8)	0.0694 (9)	0.0031 (6)	0.0135 (7)	-0.0102 (7)
O2	0.0463 (8)	0.0446 (9)	0.0753 (10)	-0.0066 (7)	0.0117 (7)	-0.0086 (8)
O3	0.0352 (8)	0.0575 (10)	0.0830 (11)	0.0003 (6)	0.0101 (7)	-0.0097 (8)
O4	0.0360 (7)	0.0486 (8)	0.0610 (9)	0.0001 (6)	0.0130 (6)	-0.0039 (7)
N1	0.0330 (8)	0.0451 (10)	0.0502 (9)	0.0006 (7)	0.0069 (7)	0.0031 (8)
N2	0.0322 (8)	0.0430 (10)	0.0598 (10)	-0.0019 (7)	0.0074 (7)	0.0010 (9)
C1	0.0369 (10)	0.0346 (10)	0.0410 (10)	0.0016 (8)	0.0054 (8)	0.0025 (8)
C2	0.0395 (10)	0.0329 (10)	0.0429 (10)	0.0051 (8)	0.0088 (8)	0.0012 (8)
C3	0.0398 (10)	0.0364 (11)	0.0494 (11)	-0.0002 (8)	0.0048 (8)	-0.0029 (9)
C4	0.0333 (10)	0.0451 (12)	0.0496 (11)	0.0048 (8)	0.0069 (8)	0.0050 (9)
C5	0.0439 (11)	0.0431 (12)	0.0531 (12)	0.0092 (9)	0.0095 (9)	-0.0035 (10)
C6	0.0442 (11)	0.0358 (11)	0.0508 (11)	0.0026 (8)	0.0052 (9)	-0.0037 (9)
C7	0.0409 (11)	0.0375 (11)	0.0476 (11)	-0.0024 (8)	0.0030 (8)	-0.0010 (9)
C8	0.0372 (10)	0.0443 (12)	0.0407 (10)	-0.0016 (9)	0.0051 (8)	-0.0024 (9)
C9	0.0349 (9)	0.0374 (10)	0.0401 (10)	0.0016 (8)	0.0043 (8)	0.0009 (8)
C10	0.0426 (11)	0.0377 (11)	0.0482 (11)	0.0003 (8)	0.0046 (9)	-0.0071 (9)
C11	0.0453 (11)	0.0403 (11)	0.0459 (11)	0.0075 (9)	0.0093 (9)	-0.0057 (9)
C12	0.0341 (9)	0.0388 (11)	0.0398 (10)	0.0034 (8)	0.0061 (7)	0.0028 (8)
C13	0.0399 (11)	0.0380 (11)	0.0534 (12)	-0.0016 (8)	0.0064 (9)	-0.0093 (9)
C14	0.0390 (10)	0.0405 (11)	0.0521 (11)	0.0032 (8)	0.0096 (9)	-0.0096 (9)
C15	0.0375 (12)	0.0611 (15)	0.0982 (19)	-0.0027 (10)	0.0041 (11)	-0.0059 (14)
C16	0.0390 (11)	0.0527 (13)	0.0678 (14)	-0.0066 (9)	0.0072 (10)	-0.0012 (11)

### Geometric parameters ( $\text{\AA}$ , $^\circ$ )

O1—C2	1.353 (2)	C6—H6	0.9300
O1—H1	0.8200	C7—H7	0.9300
O2—C8	1.231 (2)	C8—C9	1.483 (2)
O3—C4	1.364 (2)	C9—C14	1.385 (3)
O3—C15	1.423 (3)	C9—C10	1.395 (2)
O4—C12	1.361 (2)	C10—C11	1.372 (2)
O4—C16	1.431 (2)	C10—H10	0.9300

N1—C7	1.274 (2)	C11—C12	1.389 (3)
N1—N2	1.374 (2)	C11—H11	0.9300
N2—C8	1.363 (2)	C12—C13	1.380 (2)
N2—H2	0.903 (10)	C13—C14	1.384 (2)
C1—C6	1.398 (2)	C13—H13	0.9300
C1—C2	1.405 (3)	C14—H14	0.9300
C1—C7	1.447 (2)	C15—H15A	0.9600
C2—C3	1.387 (2)	C15—H15B	0.9600
C3—C4	1.383 (2)	C15—H15C	0.9600
C3—H3	0.9300	C16—H16A	0.9600
C4—C5	1.393 (3)	C16—H16B	0.9600
C5—C6	1.367 (3)	C16—H16C	0.9600
C5—H5	0.9300		
C2—O1—H1	109.5	C14—C9—C10	118.38 (17)
C4—O3—C15	118.20 (15)	C14—C9—C8	123.83 (17)
C12—O4—C16	117.91 (14)	C10—C9—C8	117.79 (17)
C7—N1—N2	119.46 (17)	C11—C10—C9	120.98 (18)
C8—N2—N1	117.10 (16)	C11—C10—H10	119.5
C8—N2—H2	123.5 (15)	C9—C10—H10	119.5
N1—N2—H2	118.2 (15)	C10—C11—C12	119.92 (17)
C6—C1—C2	117.41 (16)	C10—C11—H11	120.0
C6—C1—C7	120.80 (17)	C12—C11—H11	120.0
C2—C1—C7	121.77 (16)	O4—C12—C13	124.80 (17)
O1—C2—C3	117.15 (17)	O4—C12—C11	115.29 (16)
O1—C2—C1	122.13 (16)	C13—C12—C11	119.90 (17)
C3—C2—C1	120.71 (16)	C12—C13—C14	119.78 (18)
C4—C3—C2	119.92 (18)	C12—C13—H13	120.1
C4—C3—H3	120.0	C14—C13—H13	120.1
C2—C3—H3	120.0	C13—C14—C9	121.03 (17)
O3—C4—C3	124.39 (18)	C13—C14—H14	119.5
O3—C4—C5	115.21 (16)	C9—C14—H14	119.5
C3—C4—C5	120.40 (17)	O3—C15—H15A	109.5
C6—C5—C4	119.08 (17)	O3—C15—H15B	109.5
C6—C5—H5	120.5	H15A—C15—H15B	109.5
C4—C5—H5	120.5	O3—C15—H15C	109.5
C5—C6—C1	122.43 (18)	H15A—C15—H15C	109.5
C5—C6—H6	118.8	H15B—C15—H15C	109.5
C1—C6—H6	118.8	O4—C16—H16A	109.5
N1—C7—C1	119.14 (18)	O4—C16—H16B	109.5
N1—C7—H7	120.4	H16A—C16—H16B	109.5
C1—C7—H7	120.4	O4—C16—H16C	109.5
O2—C8—N2	121.38 (17)	H16A—C16—H16C	109.5
O2—C8—C9	122.52 (17)	H16B—C16—H16C	109.5
N2—C8—C9	116.10 (17)		

Hydrogen-bond geometry ( $\text{\AA}$ ,  $^\circ$ )

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1—H1 $\cdots$ N1	0.82	1.86	2.577 (2)	146

## supplementary materials

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N2—H2 $\cdots$ O2<sup>i</sup>                      0.90 (1)                      2.393 (11)                      3.281 (2)                      168 (2)  
Symmetry codes: (i)  $x, y-1, z$ .

Fig. 1

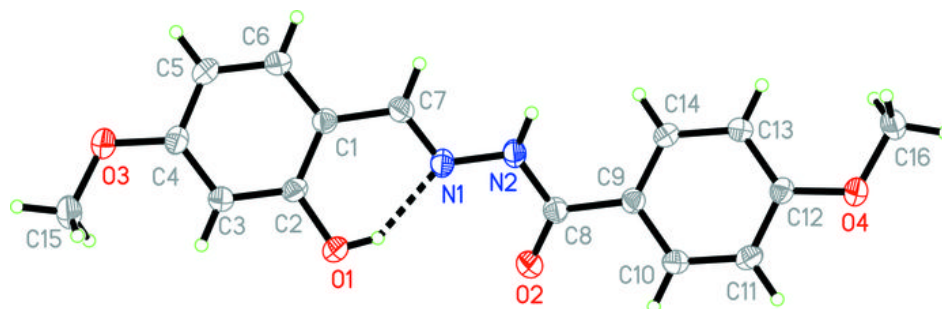




Fig. 2

