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## Structure Reports

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2,4-Dihydroxy-*N'*-(2-hydroxy-4-methoxybenzylidene)benzohydrazide

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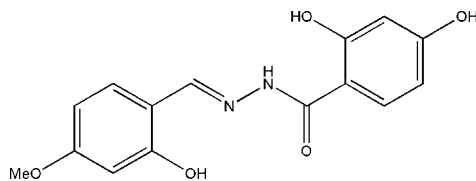
Received 30 March 2010; accepted 1 April 2010

Key indicators: single-crystal X-ray study;  $T = 298$  K; mean  $\sigma(\text{C}-\text{C}) = 0.005$  Å;  $R$  factor = 0.054;  $wR$  factor = 0.182; data-to-parameter ratio = 14.7.

In the title compound,  $\text{C}_{15}\text{H}_{14}\text{N}_2\text{O}_5$ , the dihedral angle between the two benzene rings is  $4.3(3)^\circ$  and the molecule adopts an *E* configuration with respect to the  $\text{C}=\text{N}$  bond. Intramolecular  $\text{O}-\text{H}\cdots\text{N}$  and  $\text{N}-\text{H}\cdots\text{O}$  hydrogen bonds are observed. In the crystal structure, the molecules are linked through intermolecular  $\text{N}-\text{H}\cdots\text{O}$  and  $\text{O}-\text{H}\cdots\text{O}$  hydrogen bonds to form layers parallel to the *ac* plane.

## Related literature

For the biological properties of hydrazone compounds, see: Patil *et al.* (2010); Cukurovali *et al.* (2006). For bond-length data, see: Allen *et al.* (1987). For related structures, see: Mohd Lair *et al.* (2009); Lin & Sang (2009); Suleiman Gwaram *et al.* (2010); Li & Ban (2009); Lo & Ng (2009); Ning & Xu (2009); Zhu *et al.* (2009).



## Experimental

## Crystal data

$\text{C}_{15}\text{H}_{14}\text{N}_2\text{O}_5$   
 $M_r = 302.28$   
 Monoclinic,  $P2_1/n$   
 $a = 10.560(3)$  Å  
 $b = 12.752(3)$  Å  
 $c = 11.313(2)$  Å  
 $\beta = 112.853(3)^\circ$

$V = 1403.8(6)$  Å<sup>3</sup>  
 $Z = 4$   
 Mo  $K\alpha$  radiation  
 $\mu = 0.11$  mm<sup>-1</sup>  
 $T = 298$  K  
 $0.30 \times 0.27 \times 0.25$  mm

## Data collection

Bruker SMART CCD area-detector diffractometer  
 Absorption correction: multi-scan (*SADABS*; Bruker, 2001)  
 $T_{\min} = 0.968$ ,  $T_{\max} = 0.973$   
 7972 measured reflections  
 3030 independent reflections  
 1023 reflections with  $I > 2\sigma(I)$   
 $R_{\text{int}} = 0.127$

## Refinement

$R[F^2 > 2\sigma(F^2)] = 0.054$   
 $wR(F^2) = 0.182$   
 $S = 0.74$   
 3030 reflections  
 206 parameters  
 1 restraint  
 H atoms treated by a mixture of independent and constrained refinement  
 $\Delta\rho_{\max} = 0.24$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -0.28$  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	1.92	2.635 (3)	145
$\text{N2}-\text{H2}\cdots\text{O4}$	0.91 (1)	2.03 (3)	2.670 (3)	126 (3)
$\text{N2}-\text{H2}\cdots\text{O1}^i$	0.91 (1)	2.43 (2)	3.240 (4)	149 (3)
$\text{O5}-\text{H5}\cdots\text{O2}^ii$	0.82	2.05	2.865 (4)	172
$\text{O4}-\text{H4}\cdots\text{O3}^i$	0.82	1.79	2.607 (3)	174

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

Data collection: *SMART* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

This work was supported by the Applied Chemistry Key Subject of Anhui Province (grant No. 200802187 C). The authors thank Mr Gang Wu of Chuzhou University for his help with the crystal growth.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI5074).

## References

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**supplementary materials**

*Acta Cryst.* (2010). E66, o1041 [ doi:10.1107/S1600536810012420 ]

## 2,4-Dihydroxy-*N'*-(2-hydroxy-4-methoxybenzylidene)benzohydrazide

Y.-Y. Han and Q.-R. Zhao

### Comment

Hydrazone compounds have been widely investigated for their biological properties (Patil *et al.*, 2010; Cukurovali *et al.*, 2006). Furthermore, the crystal structures of hydrazone compounds have also attracted much attention in recent years (Mohd Lair *et al.*, 2009; Lin & Sang, 2009; Suleiman Gwaram *et al.*, 2010). In the present work, the title new hydrazone compound is reported.

In the molecule of the title compound (Fig. 1), the dihedral angle between the two benzene rings is  $4.3(3)^\circ$ . The molecule adopts an *E* configuration with respect to the C=N bond. There are intramolecular O—H $\cdots$ N and N—H $\cdots$ O hydrogen bonds (Table 1) in the molecule. All the bond lengths are within normal ranges (Allen *et al.*, 1987) and are comparable with those observed in related structures (Li & Ban, 2009; Lo & Ng, 2009; Ning & Xu, 2009; Zhu *et al.*, 2009).

In the crystal structure, molecules are linked through intermolecular N—H $\cdots$ O and O—H $\cdots$ O hydrogen bonds (Table 1) to form layers parallel to the *ac* plane (Fig. 2).

### Experimental

A mixture of 2-hydroxy-4-methoxybenzaldehyde (0.152 g, 1 mmol) and 2,4-dihydroxybenzohydrazide (0.168 g, 1 mmol) in methanol (50 ml) was stirred at room temperature for 1 h. The mixture was filtered to remove impurities, and then left at room temperature. After a few days, single crystals of the title compound, suitable for X-ray diffraction, were formed.

### Refinement

Atom H2 was located in a difference Fourier map and refined isotropically, with the N—H distance restrained to 0.90 (1) Å. Other H atoms were positioned geometrically and refined using the riding-model approximation, with C—H = 0.93 or 0.96 Å, O—H = 0.82 Å and  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$  or  $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{methyl C and O})$ . The ratio of observed to unique reflections is low (34%), and the value of  $R_{\text{int}}$  is greater (0.127) probably due to the poor diffraction quality of the crystal.

### Figures

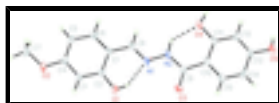


Fig. 1. The molecular structure of the title compound with atom labels and 30% probability displacement ellipsoids for non-H atoms. Intramolecular hydrogen bonds are shown as dashed lines.

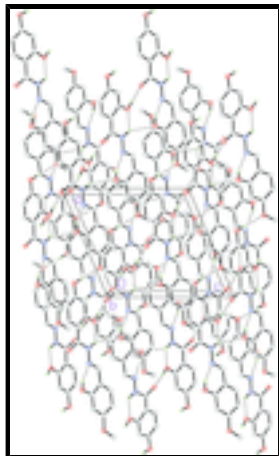


Fig. 2. The molecular packing of the title compound, viewed along the *b* axis. Hydrogen bonds are shown as dashed lines.

**2,4-Dihydroxy-*N'*-(2-hydroxy-4-methoxybenzylidene)benzohydrazide**

*Crystal data*

$C_{15}H_{14}N_2O_5$

$M_r = 302.28$

Monoclinic,  $P2_1/n$

Hall symbol:  $-P\ 2_1n$

$a = 10.560\ (3)\ \text{\AA}$

$b = 12.752\ (3)\ \text{\AA}$

$c = 11.313\ (2)\ \text{\AA}$

$\beta = 112.853\ (3)^\circ$

$V = 1403.8\ (6)\ \text{\AA}^3$

$Z = 4$

$F(000) = 632$

$D_x = 1.430\ \text{Mg m}^{-3}$

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

Cell parameters from 691 reflections

$\theta = 2.5\text{--}24.5^\circ$

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

$T = 298\ \text{K}$

Block, colourless

$0.30 \times 0.27 \times 0.25\ \text{mm}$

*Data collection*

Bruker SMART CCD area-detector diffractometer

Radiation source: fine-focus sealed tube graphite

$\omega$  scans

Absorption correction: multi-scan (SADABS; Bruker, 2001)

$T_{\min} = 0.968$ ,  $T_{\max} = 0.973$

7972 measured reflections

3030 independent reflections

1023 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.127$

$\theta_{\max} = 27.0^\circ$ ,  $\theta_{\min} = 2.2^\circ$

$h = -11 \rightarrow 13$

$k = -16 \rightarrow 16$

$l = -14 \rightarrow 9$

*Refinement*

Refinement on  $F^2$

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.054$

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

$wR(F^2) = 0.182$

$S = 0.74$

3030 reflections

206 parameters

1 restraint

H atoms treated by a mixture of independent and constrained refinement

$$w = 1/[\sigma^2(F_o^2) + (0.0795P)^2]$$

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

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

$$\Delta\rho_{\max} = 0.24 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.28 \text{ e } \text{\AA}^{-3}$$

### 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  $F^2 > \sigma(F^2)$  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>	$U_{\text{iso}}^*/U_{\text{eq}}$
N1	0.6400 (2)	0.2494 (2)	0.3907 (3)	0.0457 (8)
N2	0.4991 (3)	0.2654 (2)	0.3472 (3)	0.0472 (8)
O1	0.8985 (2)	0.2824 (2)	0.5431 (3)	0.0609 (8)
H1	0.8162	0.2961	0.5104	0.091*
O2	1.2425 (2)	0.08925 (18)	0.4832 (2)	0.0563 (7)
O3	0.5297 (2)	0.36628 (17)	0.5199 (2)	0.0504 (7)
O4	0.23759 (19)	0.21715 (19)	0.2060 (2)	0.0505 (7)
H4	0.1705	0.1952	0.1455	0.076*
O5	-0.1093 (2)	0.4346 (2)	0.2541 (3)	0.0650 (8)
H5	-0.1518	0.4218	0.1778	0.097*
C1	0.8268 (3)	0.1629 (3)	0.3636 (3)	0.0391 (9)
C2	0.9293 (3)	0.2101 (3)	0.4705 (3)	0.0407 (9)
C3	1.0660 (3)	0.1834 (3)	0.5062 (3)	0.0446 (9)
H3	1.1327	0.2147	0.5775	0.054*
C4	1.1035 (3)	0.1099 (3)	0.4356 (3)	0.0432 (9)
C5	1.0068 (3)	0.0628 (3)	0.3298 (4)	0.0478 (10)
H5A	1.0325	0.0140	0.2822	0.057*
C6	0.8696 (3)	0.0899 (3)	0.2955 (4)	0.0492 (10)
H6	0.8038	0.0580	0.2242	0.059*
C7	1.2890 (3)	0.0103 (3)	0.4192 (4)	0.0709 (13)
H7A	1.2419	-0.0543	0.4179	0.106*
H7B	1.3861	0.0002	0.4639	0.106*
H7C	1.2700	0.0322	0.3328	0.106*
C8	0.6814 (3)	0.1853 (3)	0.3265 (3)	0.0435 (9)
H8	0.6174	0.1524	0.2548	0.052*

## supplementary materials

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C9	0.4506 (3)	0.3273 (3)	0.4183 (4)	0.0415 (9)
C10	0.3002 (3)	0.3483 (2)	0.3673 (3)	0.0374 (9)
C11	0.1983 (3)	0.2962 (3)	0.2647 (3)	0.0370 (8)
C12	0.0617 (3)	0.3252 (2)	0.2260 (3)	0.0419 (9)
H12	-0.0052	0.2916	0.1569	0.050*
C13	0.0249 (3)	0.4037 (3)	0.2898 (4)	0.0437 (9)
C14	0.1218 (3)	0.4546 (3)	0.3917 (4)	0.0519 (10)
H14	0.0958	0.5076	0.4343	0.062*
C15	0.2581 (3)	0.4264 (2)	0.4305 (4)	0.0457 (9)
H15	0.3235	0.4603	0.5004	0.055*
H2	0.443 (3)	0.241 (3)	0.2685 (18)	0.080*

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

	$U^{11}$	$U^{22}$	$U^{33}$	$U^{12}$	$U^{13}$	$U^{23}$
N1	0.0189 (15)	0.0627 (19)	0.050 (2)	0.0027 (12)	0.0073 (13)	-0.0013 (16)
N2	0.0205 (15)	0.068 (2)	0.048 (2)	0.0037 (13)	0.0070 (14)	-0.0070 (17)
O1	0.0297 (13)	0.0741 (17)	0.0720 (19)	0.0085 (13)	0.0121 (13)	-0.0242 (16)
O2	0.0291 (13)	0.0700 (17)	0.0685 (19)	0.0128 (11)	0.0176 (12)	-0.0086 (15)
O3	0.0308 (12)	0.0562 (15)	0.0506 (17)	0.0006 (11)	0.0010 (12)	-0.0063 (14)
O4	0.0261 (12)	0.0640 (16)	0.0546 (18)	0.0019 (12)	0.0083 (12)	-0.0129 (14)
O5	0.0297 (13)	0.0760 (18)	0.082 (2)	0.0135 (13)	0.0131 (13)	-0.0088 (17)
C1	0.0244 (17)	0.049 (2)	0.042 (2)	-0.0017 (15)	0.0103 (16)	0.0014 (19)
C2	0.0298 (18)	0.045 (2)	0.049 (2)	0.0039 (16)	0.0172 (17)	-0.0038 (19)
C3	0.0260 (18)	0.057 (2)	0.047 (2)	0.0004 (16)	0.0100 (16)	-0.008 (2)
C4	0.0302 (18)	0.051 (2)	0.054 (2)	0.0027 (16)	0.0217 (18)	0.003 (2)
C5	0.042 (2)	0.051 (2)	0.054 (3)	0.0009 (17)	0.0217 (19)	-0.010 (2)
C6	0.033 (2)	0.058 (2)	0.054 (2)	-0.0082 (17)	0.0142 (18)	-0.010 (2)
C7	0.050 (3)	0.082 (3)	0.085 (3)	0.024 (2)	0.031 (2)	-0.002 (3)
C8	0.0288 (19)	0.053 (2)	0.043 (2)	-0.0066 (15)	0.0084 (17)	0.0018 (19)
C9	0.0279 (18)	0.045 (2)	0.045 (2)	0.0013 (15)	0.0074 (18)	0.0103 (19)
C10	0.0237 (17)	0.044 (2)	0.043 (2)	0.0015 (15)	0.0110 (16)	0.0066 (18)
C11	0.0264 (17)	0.0429 (19)	0.041 (2)	-0.0007 (15)	0.0123 (16)	0.0046 (18)
C12	0.0237 (17)	0.047 (2)	0.049 (2)	-0.0006 (15)	0.0072 (16)	0.0019 (19)
C13	0.0239 (17)	0.048 (2)	0.056 (2)	0.0062 (15)	0.0119 (17)	0.006 (2)
C14	0.040 (2)	0.049 (2)	0.067 (3)	0.0041 (17)	0.021 (2)	-0.008 (2)
C15	0.0331 (19)	0.045 (2)	0.053 (2)	-0.0009 (16)	0.0104 (17)	-0.004 (2)

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

N1—C8	1.278 (4)	C4—C5	1.374 (4)
N1—N2	1.389 (3)	C5—C6	1.390 (4)
N2—C9	1.362 (4)	C5—H5A	0.93
N2—H2	0.911 (10)	C6—H6	0.93
O1—C2	1.355 (4)	C7—H7A	0.96
O1—H1	0.82	C7—H7B	0.96
O2—C4	1.378 (3)	C7—H7C	0.96
O2—C7	1.434 (4)	C8—H8	0.93
O3—C9	1.232 (4)	C9—C10	1.488 (4)

O4—C11	1.357 (4)	C10—C15	1.397 (4)
O4—H4	0.82	C10—C11	1.406 (4)
O5—C13	1.372 (3)	C11—C12	1.386 (4)
O5—H5	0.82	C12—C13	1.375 (4)
C1—C6	1.390 (4)	C12—H12	0.93
C1—C2	1.407 (4)	C13—C14	1.372 (4)
C1—C8	1.455 (4)	C14—C15	1.380 (4)
C2—C3	1.382 (4)	C14—H14	0.93
C3—C4	1.385 (4)	C15—H15	0.93
C3—H3	0.93		
C8—N1—N2	116.6 (3)	H7A—C7—H7B	109.5
C9—N2—N1	118.2 (3)	O2—C7—H7C	109.5
C9—N2—H2	122 (2)	H7A—C7—H7C	109.5
N1—N2—H2	120 (2)	H7B—C7—H7C	109.5
C2—O1—H1	109.5	N1—C8—C1	121.1 (3)
C4—O2—C7	117.3 (3)	N1—C8—H8	119.4
C11—O4—H4	109.5	C1—C8—H8	119.4
C13—O5—H5	109.5	O3—C9—N2	120.7 (3)
C6—C1—C2	117.1 (3)	O3—C9—C10	121.6 (3)
C6—C1—C8	120.3 (3)	N2—C9—C10	117.7 (3)
C2—C1—C8	122.5 (3)	C15—C10—C11	117.8 (3)
O1—C2—C3	117.4 (3)	C15—C10—C9	115.7 (3)
O1—C2—C1	121.7 (3)	C11—C10—C9	126.5 (3)
C3—C2—C1	120.9 (3)	O4—C11—C12	121.5 (3)
C2—C3—C4	119.9 (3)	O4—C11—C10	118.3 (3)
C2—C3—H3	120.0	C12—C11—C10	120.2 (3)
C4—C3—H3	120.0	C13—C12—C11	120.0 (3)
C5—C4—O2	125.1 (3)	C13—C12—H12	120.0
C5—C4—C3	121.0 (3)	C11—C12—H12	120.0
O2—C4—C3	113.8 (3)	O5—C13—C14	117.4 (3)
C4—C5—C6	118.4 (3)	O5—C13—C12	121.6 (3)
C4—C5—H5A	120.8	C14—C13—C12	121.0 (3)
C6—C5—H5A	120.8	C13—C14—C15	119.3 (3)
C5—C6—C1	122.7 (3)	C13—C14—H14	120.4
C5—C6—H6	118.7	C15—C14—H14	120.4
C1—C6—H6	118.7	C14—C15—C10	121.6 (3)
O2—C7—H7A	109.5	C14—C15—H15	119.2
O2—C7—H7B	109.5	C10—C15—H15	119.2

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	1.92	2.635 (3)	145
N2—H2 $\cdots$ O4	0.91 (1)	2.03 (3)	2.670 (3)	126 (3)
N2—H2 $\cdots$ O1 <sup>i</sup>	0.91 (1)	2.43 (2)	3.240 (4)	149 (3)
O5—H5 $\cdots$ O2 <sup>ii</sup>	0.82	2.05	2.865 (4)	172
O4—H4 $\cdots$ O3 <sup>i</sup>	0.82	1.79	2.607 (3)	174

Symmetry codes: (i)  $x-1/2, -y+1/2, z-1/2$ ; (ii)  $x-3/2, -y+1/2, z-1/2$ .

Fig. 1

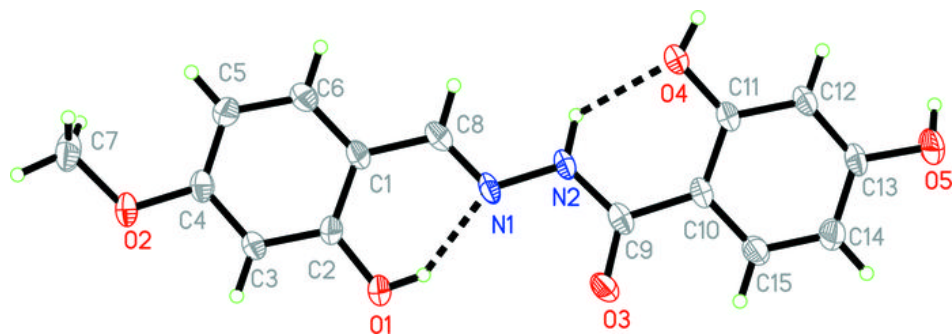




Fig. 2

