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# 4-Hydroxy-*N*′-[1-(2-hydroxyphenyl)-ethylidene]benzohydrazide

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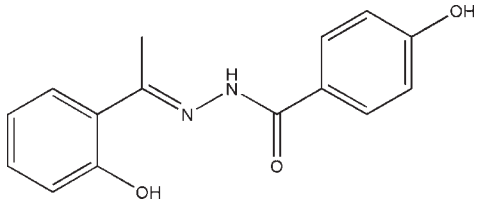
Key indicators: single-crystal X-ray study; $T$ = 298 K; mean $\sigma$(C–C) = 0.004 Å; $R$ factor = 0.069; $wR$ factor = 0.172; data-to-parameter ratio = 14.7.

In the title compound, $C_{15}H_{14}N_2O_3$, there is an intramolecular O—H···N hydrogen bond and the dihedral angle between the two aromatic rings is 13.9 (3)°. In the crystal structure, molecules are stabilized by intermolecular O—H···O and N—H···O hydrogen bonds.

## Related literature

For related structures, see: Lu *et al.* (2008*a*,*b*,*c*); Xiao & Wei (2009); He (2008); Shi *et al.* (2007). For bond-length data, see: Allen *et al.* (1987).




## Experimental

### Crystal data

$C_{15}H_{14}N_2O_3$
$M_r$ = 270.28
Monoclinic, $P2_1/c$
$a$ = 4.926 (2) Å
$b$ = 31.06 (2) Å
$c$ = 8.473 (3) Å
$\beta$ = 93.852 (3)°
$V$ = 1293.5 (11) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.10 mm$^{-1}$
$T$ = 298 K
0.21 × 0.20 × 0.17 mm

### Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004)
$T_{min}$ = 0.980, $T_{max}$ = 0.984
8965 measured reflections
2770 independent reflections
1569 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.064

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.069
$wR(F^2)$ = 0.172
$S$ = 1.05
2770 reflections
188 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.26 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.21 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O1—H1···N1 | 0.82 | 1.78 | 2.499 (3) | 146 |
| O3—H3···O1$^{i}$ | 0.82 | 1.97 | 2.786 (3) | 179 |
| N2—H2···O2$^{ii}$ | 0.90 (1) | 2.09 (2) | 2.961 (4) | 163 (3) |

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

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); 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: SU2181).

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# supporting information

*Acta Cryst.* (2010). E**66**, o1514 [https://doi.org/10.1107/S1600536810019914]

## 4-Hydroxy-*N*′-[1-(2-hydroxyphenyl)ethylidene]benzohydrazide

**Xiao-Hui Ji and Jiu-Fu Lu**

### S1. Comment

As part of our investigation of the crystal structures of Schiff bases derived from the condensation of aldehydes or ketones with benzohydrazides (Lu *et al.*, 2008*a*,b,c), we report herein on the crystal structure of the new title Schiff base compound.

In the title compound, illustrated in Fig. 1, the bond lengths have normal values (Allen *et al.*, 1987), and are comparable to those observed in similar compounds (Xiao & Wei, 2009; He, 2008; Shi *et al.*, 2007). The dihedral angle between the two aromatic rings is 13.9 (3)°, indicating that the Schiff base molecule is slightly twisted. An intramolecular O—H···N hydrogen bond is observed (Table 1).

In the crystal structure, the molecules are stabilized by intermolecular O—H···O and N—H···O hydrogen bonds (Table 1 and Fig. 2).

### S2. Experimental

The title compound was prepared by the Schiff base condensation of 1-(2-hydroxyphenyl)ethanone (0.1 mol, 13.6 g) and 4-hydroxybenzohydrazide (0.1 mmol, 15.2 g) in 95% ethanol (70 ml). The excess ethanol was removed by distillation. The colourless solid obtained was filtered and washed with ethanol. Single crystals, suitable for X-ray diffraction analysis, were obatined by slow evaporation of a 95% ethanol solution at rt.

### S3. Refinement

The N2 H-atom (H2) was located in a difference Fourier map and refined with a distance restraint: N-H = 0.90 (1) Å. The other H-atoms were positioned geometrically (C—H = 0.93–0.96 Å and O—H = 0.82 Å) and refined using a riding model, with $U_{iso}$(H) = 1.2$U_{eq}$(C) and 1.5$U_{eq}$($C_{methyl}$ and O). A rotating group model was used for the methyl and hydroxyl groups.

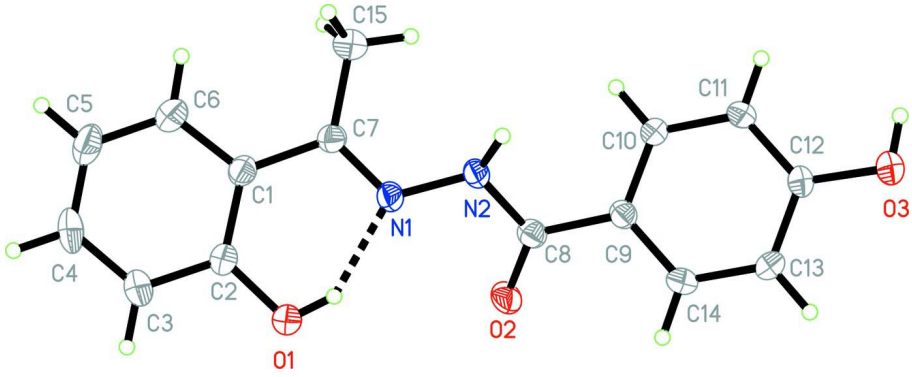


**Figure 1**

The molecular structure of the title compound, showing 30% probability displacement ellipsoids and the atom-numbering scheme. The dashed line indicates the intramolecular O-H···N hydrogen bond (see Table 1 for details.

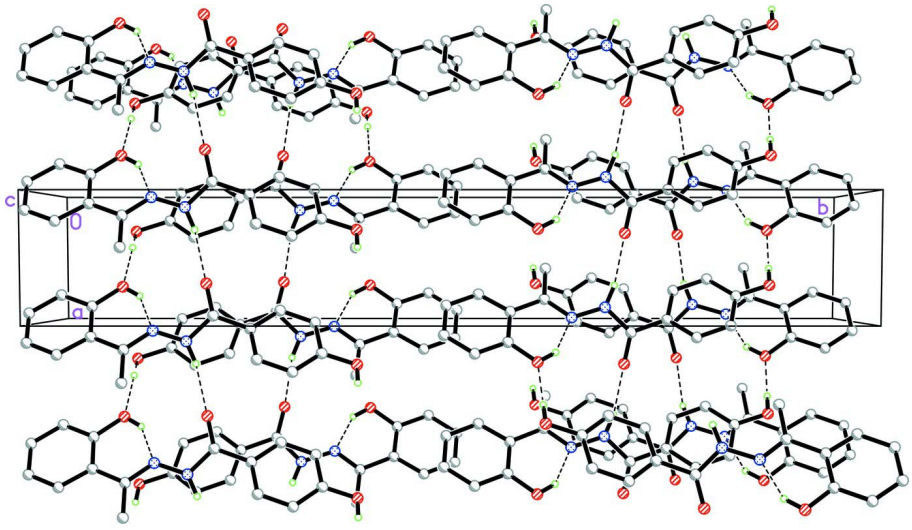


**Figure 2**

The crystal packing of the title compound, viewed along the *c* axis. H-atoms not involved in hydrogen bonding (dashed lines) have been omitted for clarity.

### 4-Hydroxy-*N*′-[1-(2-hydroxyphenyl)ethylidene]benzohydrazide

*Crystal data*

$C_{15}H_{14}N_2O_3$
$M_r = 270.28$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 4.926\ (2)$ Å
$b = 31.06\ (2)$ Å
$c = 8.473\ (3)$ Å
$\beta = 93.852\ (3)°$
$V = 1293.5\ (11)$ Å$^3$
$Z = 4$
$F(000) = 568$
$D_x = 1.388$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 957 reflections
$\theta = 2.5–24.5°$
$\mu = 0.10$ mm$^{-1}$
$T = 298$ K
Block, colourless
0.21 × 0.20 × 0.17 mm

*Data collection*

Bruker APEXII CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega$ scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004)
$T_{min}$ = 0.980, $T_{max}$ = 0.984
8965 measured reflections
2770 independent reflections
1569 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.064
$\theta_{max}$ = 27.0°, $\theta_{min}$ = 2.5°
$h = -6 \rightarrow 6$
$k = -39 \rightarrow 34$
$l = -10 \rightarrow 10$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.069
$wR(F^2)$ = 0.172
$S$ = 1.05
2770 reflections
188 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
$w = 1/[\sigma^2(F_o^2) + (0.0649P)^2 + 0.4306P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.26 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.21 e Å$^{-3}$
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0057 (18)

*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 (Å$^2$)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| N1 | 0.9548 (5) | 0.64659 (7) | 0.6666 (3) | 0.0384 (6) |
| N2 | 0.8786 (5) | 0.68800 (7) | 0.6186 (3) | 0.0389 (6) |
| O1 | 1.2807 (5) | 0.60332 (7) | 0.8408 (3) | 0.0581 (7) |
| H1 | 1.2112 | 0.6252 | 0.8021 | 0.087* |
| O2 | 1.3097 (4) | 0.71011 (6) | 0.6779 (3) | 0.0510 (6) |
| O3 | 0.7088 (5) | 0.89007 (6) | 0.5716 (3) | 0.0593 (7) |
| H3 | 0.5810 | 0.8920 | 0.5048 | 0.089* |
| C1 | 0.9088 (6) | 0.57209 (9) | 0.6782 (3) | 0.0377 (7) |
| C2 | 1.1338 (6) | 0.56842 (9) | 0.7889 (3) | 0.0413 (8) |
| C3 | 1.2167 (7) | 0.52855 (10) | 0.8469 (4) | 0.0530 (9) |
| H3A | 1.3668 | 0.5266 | 0.9192 | 0.064* |
| C4 | 1.0804 (8) | 0.49197 (10) | 0.7992 (4) | 0.0603 (10) |
| H4 | 1.1375 | 0.4653 | 0.8389 | 0.072* |
| C5 | 0.8616 (8) | 0.49468 (11) | 0.6938 (5) | 0.0644 (11) |
| H5 | 0.7681 | 0.4698 | 0.6618 | 0.077* |

| | | | | |
|---|---|---|---|---|
| C6 | 0.7776 (7) | 0.53373 (10) | 0.6342 (4) | 0.0544 (9) |
| H6 | 0.6274 | 0.5348 | 0.5617 | 0.065* |
| C7 | 0.8165 (6) | 0.61399 (9) | 0.6136 (3) | 0.0366 (7) |
| C8 | 1.0701 (6) | 0.71922 (9) | 0.6405 (3) | 0.0374 (7) |
| C9 | 0.9732 (6) | 0.76376 (9) | 0.6167 (3) | 0.0346 (7) |
| C10 | 0.7529 (6) | 0.77407 (9) | 0.5133 (3) | 0.0394 (7) |
| H10 | 0.6639 | 0.7523 | 0.4547 | 0.047* |
| C11 | 0.6634 (6) | 0.81585 (9) | 0.4958 (3) | 0.0411 (8) |
| H11 | 0.5158 | 0.8222 | 0.4254 | 0.049* |
| C12 | 0.7927 (6) | 0.84846 (9) | 0.5827 (3) | 0.0392 (7) |
| C13 | 1.0155 (6) | 0.83870 (10) | 0.6841 (4) | 0.0477 (8) |
| H13 | 1.1056 | 0.8605 | 0.7415 | 0.057* |
| C14 | 1.1051 (6) | 0.79695 (10) | 0.7005 (4) | 0.0446 (8) |
| H14 | 1.2560 | 0.7908 | 0.7687 | 0.054* |
| C15 | 0.5813 (7) | 0.61695 (11) | 0.4922 (4) | 0.0541 (9) |
| H15A | 0.4183 | 0.6075 | 0.5378 | 0.081* |
| H15B | 0.6154 | 0.5990 | 0.4035 | 0.081* |
| H15C | 0.5595 | 0.6463 | 0.4575 | 0.081* |
| H2 | 0.710 (3) | 0.6991 (11) | 0.622 (4) | 0.080* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| N1 | 0.0324 (14) | 0.0301 (14) | 0.0522 (15) | 0.0026 (11) | 0.0000 (11) | 0.0024 (11) |
| N2 | 0.0325 (14) | 0.0302 (14) | 0.0531 (15) | 0.0022 (11) | −0.0030 (12) | 0.0062 (11) |
| O1 | 0.0596 (15) | 0.0364 (13) | 0.0744 (16) | −0.0035 (11) | −0.0252 (12) | 0.0046 (11) |
| O2 | 0.0294 (12) | 0.0443 (13) | 0.0784 (16) | 0.0045 (10) | −0.0033 (11) | 0.0088 (11) |
| O3 | 0.0686 (18) | 0.0336 (13) | 0.0721 (17) | 0.0086 (11) | −0.0224 (13) | −0.0024 (10) |
| C1 | 0.0364 (17) | 0.0327 (16) | 0.0439 (16) | −0.0017 (13) | 0.0030 (13) | −0.0025 (13) |
| C2 | 0.0458 (19) | 0.0313 (17) | 0.0464 (17) | −0.0017 (14) | 0.0001 (14) | −0.0005 (13) |
| C3 | 0.062 (2) | 0.0367 (19) | 0.060 (2) | 0.0050 (16) | −0.0040 (17) | 0.0084 (15) |
| C4 | 0.077 (3) | 0.0292 (19) | 0.076 (2) | 0.0094 (17) | 0.013 (2) | 0.0120 (16) |
| C5 | 0.072 (3) | 0.0326 (19) | 0.088 (3) | −0.0091 (18) | 0.000 (2) | −0.0022 (17) |
| C6 | 0.055 (2) | 0.041 (2) | 0.066 (2) | −0.0074 (16) | −0.0070 (17) | −0.0049 (16) |
| C7 | 0.0340 (17) | 0.0351 (16) | 0.0405 (16) | 0.0006 (13) | −0.0001 (13) | −0.0023 (12) |
| C8 | 0.0338 (17) | 0.0386 (17) | 0.0397 (16) | 0.0010 (14) | 0.0017 (13) | 0.0043 (13) |
| C9 | 0.0314 (16) | 0.0334 (16) | 0.0388 (15) | 0.0013 (13) | 0.0017 (12) | 0.0036 (12) |
| C10 | 0.0415 (18) | 0.0313 (16) | 0.0438 (17) | −0.0016 (13) | −0.0080 (13) | −0.0003 (13) |
| C11 | 0.0426 (18) | 0.0367 (17) | 0.0419 (17) | 0.0008 (14) | −0.0125 (14) | 0.0048 (13) |
| C12 | 0.0450 (18) | 0.0277 (16) | 0.0445 (17) | 0.0026 (13) | −0.0013 (14) | 0.0032 (13) |
| C13 | 0.046 (2) | 0.0353 (18) | 0.0588 (19) | −0.0043 (14) | −0.0146 (16) | −0.0076 (15) |
| C14 | 0.0380 (18) | 0.0428 (18) | 0.0509 (19) | 0.0019 (15) | −0.0127 (14) | 0.0019 (14) |
| C15 | 0.049 (2) | 0.053 (2) | 0.058 (2) | −0.0024 (17) | −0.0111 (16) | 0.0030 (16) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| N1—C7 | 1.284 (3) | C5—H5 | 0.9300 |
| N1—N2 | 1.393 (3) | C6—H6 | 0.9300 |

| | | | |
|---|---|---|---|
| N2—C8 | 1.357 (4) | C7—C15 | 1.500 (4) |
| N2—H2 | 0.900 (10) | C8—C9 | 1.473 (4) |
| O1—C2 | 1.360 (3) | C9—C10 | 1.385 (4) |
| O1—H1 | 0.8200 | C9—C14 | 1.388 (4) |
| O2—C8 | 1.234 (3) | C10—C11 | 1.375 (4) |
| O3—C12 | 1.358 (3) | C10—H10 | 0.9300 |
| O3—H3 | 0.8200 | C11—C12 | 1.382 (4) |
| C1—C6 | 1.394 (4) | C11—H11 | 0.9300 |
| C1—C2 | 1.407 (4) | C12—C13 | 1.381 (4) |
| C1—C7 | 1.472 (4) | C13—C14 | 1.374 (4) |
| C2—C3 | 1.384 (4) | C13—H13 | 0.9300 |
| C3—C4 | 1.367 (4) | C14—H14 | 0.9300 |
| C3—H3A | 0.9300 | C15—H15A | 0.9600 |
| C4—C5 | 1.356 (5) | C15—H15B | 0.9600 |
| C4—H4 | 0.9300 | C15—H15C | 0.9600 |
| C5—C6 | 1.367 (5) | | |

| | | | |
|---|---|---|---|
| C7—N1—N2 | 120.0 (2) | O2—C8—N2 | 121.0 (3) |
| C8—N2—N1 | 116.7 (2) | O2—C8—C9 | 123.1 (3) |
| C8—N2—H2 | 111 (2) | N2—C8—C9 | 115.9 (2) |
| N1—N2—H2 | 125 (2) | C10—C9—C14 | 118.1 (3) |
| C2—O1—H1 | 109.5 | C10—C9—C8 | 122.5 (3) |
| C12—O3—H3 | 109.5 | C14—C9—C8 | 119.4 (2) |
| C6—C1—C2 | 116.1 (3) | C11—C10—C9 | 121.2 (3) |
| C6—C1—C7 | 122.0 (3) | C11—C10—H10 | 119.4 |
| C2—C1—C7 | 121.9 (2) | C9—C10—H10 | 119.4 |
| O1—C2—C3 | 117.4 (3) | C10—C11—C12 | 120.1 (3) |
| O1—C2—C1 | 122.0 (2) | C10—C11—H11 | 120.0 |
| C3—C2—C1 | 120.6 (3) | C12—C11—H11 | 120.0 |
| C4—C3—C2 | 120.7 (3) | O3—C12—C13 | 118.5 (3) |
| C4—C3—H3A | 119.6 | O3—C12—C11 | 122.2 (3) |
| C2—C3—H3A | 119.6 | C13—C12—C11 | 119.3 (3) |
| C5—C4—C3 | 119.8 (3) | C14—C13—C12 | 120.4 (3) |
| C5—C4—H4 | 120.1 | C14—C13—H13 | 119.8 |
| C3—C4—H4 | 120.1 | C12—C13—H13 | 119.8 |
| C4—C5—C6 | 120.4 (3) | C13—C14—C9 | 120.9 (3) |
| C4—C5—H5 | 119.8 | C13—C14—H14 | 119.6 |
| C6—C5—H5 | 119.8 | C9—C14—H14 | 119.6 |
| C5—C6—C1 | 122.4 (3) | C7—C15—H15A | 109.5 |
| C5—C6—H6 | 118.8 | C7—C15—H15B | 109.5 |
| C1—C6—H6 | 118.8 | H15A—C15—H15B | 109.5 |
| N1—C7—C1 | 115.0 (2) | C7—C15—H15C | 109.5 |
| N1—C7—C15 | 124.0 (3) | H15A—C15—H15C | 109.5 |
| C1—C7—C15 | 121.0 (3) | H15B—C15—H15C | 109.5 |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| O1—H1···N1 | 0.82 | 1.78 | 2.499 (3) | 146 |
| O3—H3···O1$^{i}$ | 0.82 | 1.97 | 2.786 (3) | 179 |
| N2—H2···O2$^{ii}$ | 0.90 (1) | 2.09 (2) | 2.961 (4) | 163 (3) |

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