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4-Hydroxy-*N*′-(2-hydroxy-3-methoxybenzylidene)benzohydrazide monohydrate

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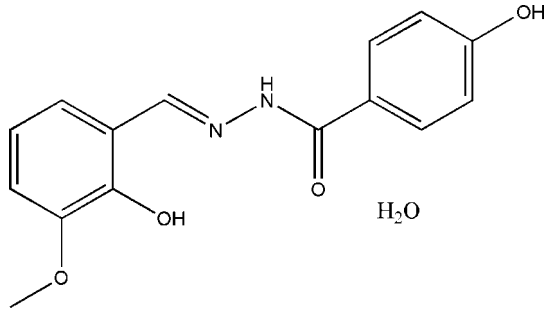
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.008 Å; R factor = 0.051; wR factor = 0.119; data-to-parameter ratio = 7.5.

In the title compound, $C_{15}H_{14}N_2O_4\cdot H_2O$, the dihedral angle between the two aromatic rings is 33.3 (5)°. The methoxy group is twisted slightly away from the attached benzene ring [C—O—C—C = 13.8 (9)°]. An intramolecular O—H···N hydrogen bond is observed. In the crystal structure, the molecules are linked into a two-dimensional network parallel to the (010) plane by intermolecular N—H···O, O—H···O and C—H···O hydrogen bonds

Related literature

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



Experimental

Crystal data

$C_{15}H_{14}N_2O_4\cdot H_2O$
M_r = 304.30
Monoclinic, *Pn*
a = 4.891 (2) Å
b = 12.171 (5) Å
c = 12.371 (5) Å
β = 95.724 (7)°
V = 732.8 (5) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 298 (2) K
0.08 × 0.07 × 0.07 mm

Data collection

Bruker APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004)
T_{min} = 0.992, T_{max} = 0.993
5972 measured reflections
1582 independent reflections
791 reflections with $I > 2\sigma(I)$
R_{int} = 0.086

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.051
$wR(F^2)$ = 0.119
S = 0.92
1582 reflections
211 parameters
6 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.14 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 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	1.99	2.687 (5)	142
N2—H2···O5^{i}	0.90 (3)	1.99 (3)	2.820 (5)	154 (5)
O4—H4···O2^{ii}	0.82	2.15	2.872 (7)	147
O5—H5*A*···O3^{iii}	0.86 (3)	1.97 (3)	2.769 (6)	156 (6)
O5—H5*B*···O3^{iv}	0.84 (4)	2.01 (3)	2.769 (6)	148 (5)
C7—H7···O5^{i}	0.93	2.48	3.229 (7)	138
C14—H14···O5^{i}	0.93	2.34	3.218 (7)	158

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

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: CI2678).

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supplementary materials

Acta Cryst. (2008). E**64**, o2032 [**doi:10.1107/S1600536808030894**]

4-Hydroxy-*N*'-(2-hydroxy-3-methoxybenzylidene)benzohydrazide monohydrate

J.-F. Lu

Comment

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

The asymmetric unit of the title compound (Fig. 1), consists of a Schiff base molecule and a water molecule of crystallization. The bond lengths have normal values (Allen *et al.*, 1987), and are comparable to those observed in similar compounds (Nie, 2008; He, 2008; Shi *et al.*, 2007). The dihedral angle between the two aromatic rings is 33.3 (5)°, indicating that the Schiff base molecule is twisted. An intramolecular O—H···N hydrogen bond is observed.

In the crystal structure, the molecules are linked into a two-dimensional network parallel to the (010) by intermolecular N—H···O, O—H···O and C—H···O hydrogen bonds (Table 1).

Experimental

The title compound was prepared by the Schiff base condensation of 2-hydroxy-3-methoxybenzaldehyde (0.1 mol) and 4-hydroxybenzohydrazide (0.1 mmol) in 95% ethanol (50 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 were obatined by slow evaporation of a 95% ethanol solution at room temperature.

Refinement

The imino and water H atoms were located in a difference map and refined with N-H, O-H and H···H distances restrained to 0.90 (1), 0.85 (1), and 1.37 (2) Å, respectively. 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) = 1.2U_{eq}(C) and 1.5U_{eq}(C_{methyl} and O). A rotating group model was used for methyl and hydroxyl groups. In the absence of significant anomalous scattering effects, Friedel pairs were averaged.

Figures

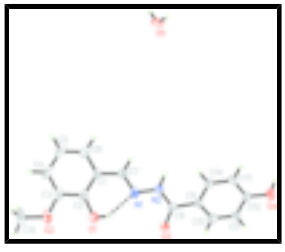

Fig. 1. The molecular structure of the title compound, showing 30% probability displacement ellipsoids and the atom-numbering scheme. The dashed line indicates a hydrogen bond.

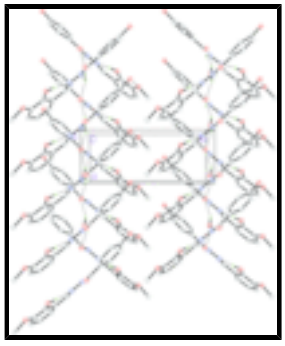

Fig. 2. The crystal packing of the title compound, viewed along the *c* axis. H atoms not involved in hydrogen bonding (dashed lines) have been omitted for clarity.

4-Hydroxy-*N*'-(2-hydroxy-3-methoxybenzylidene)benzohydrazide monohydrate

Crystal data

$C_{15}H_{14}N_2O_4 \cdot H_2O$	$F_{000} = 320$
$M_r = 304.30$	$D_x = 1.379$ Mg m^{-3}
Monoclinic, *Pn*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: P -2yac	Cell parameters from 623 reflections
a = 4.891 (2) Å	θ = 2.3–24.0º
b = 12.171 (5) Å	μ = 0.11 mm^{-1}
c = 12.371 (5) Å	*T* = 298 (2) K
β = 95.724 (7)º	Block, colourless
V = 732.8 (5) Å^3	0.08 × 0.07 × 0.07 mm
Z = 2	

Data collection

Bruker APEXII CCD area-detector diffractometer	1582 independent reflections
Radiation source: fine-focus sealed tube	791 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.086$
T = 298(2) K	$\theta_{max} = 27.0$º
ω scans	$\theta_{min} = 1.7$º
Absorption correction: multi-scan (SADABS; Sheldrick, 2004)	*h* = −6→6
$T_{min} = 0.992$, $T_{max} = 0.993$	*k* = −15→15
5972 measured reflections	*l* = −15→15

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.051$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.119$	$w = 1/[\sigma^2(F_o^2) + (0.0473P)^2]$

	where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.92$	$(\Delta/\sigma)_{max} = 0.001$
1582 reflections	$\Delta\rho_{max} = 0.14$ e Å^{-3}
211 parameters	$\Delta\rho_{min} = -0.15$ e Å^{-3}
6 restraints	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	0.2886 (8)	0.7446 (3)	0.9901 (3)	0.0588 (11)
H1	0.2167	0.8048	0.9782	0.088*
O2	0.6029 (9)	0.5752 (3)	1.0195 (4)	0.0747 (13)
O3	−0.1590 (7)	1.0380 (3)	0.9939 (3)	0.0564 (10)
O4	−0.9973 (10)	1.3626 (4)	0.7359 (3)	0.0823 (15)
H4	−1.0197	1.3619	0.6694	0.123*
O5	0.3665 (9)	1.0067 (4)	0.0948 (3)	0.0717 (13)
N1	0.1007 (8)	0.9097 (3)	0.8590 (3)	0.0460 (11)
N2	−0.0723 (9)	0.9954 (4)	0.8239 (3)	0.0454 (11)
C1	0.4174 (11)	0.7745 (4)	0.8075 (5)	0.0494 (14)
C2	0.4327 (11)	0.7168 (4)	0.9064 (5)	0.0463 (14)
C3	0.6063 (12)	0.6245 (5)	0.9194 (5)	0.0579 (17)
C4	0.7584 (13)	0.5927 (5)	0.8385 (6)	0.072 (2)
H4A	0.8732	0.5319	0.8484	0.087*
C5	0.7435 (13)	0.6500 (6)	0.7418 (6)	0.073 (2)
H5	0.8484	0.6279	0.6870	0.088*
C6	0.5746 (12)	0.7393 (5)	0.7267 (5)	0.0594 (16)
H6	0.5647	0.7771	0.6611	0.071*
C7	0.2392 (11)	0.8680 (4)	0.7868 (4)	0.0485 (14)
H7	0.2243	0.8993	0.7180	0.058*
C8	−0.2022 (10)	1.0526 (4)	0.8945 (5)	0.0421 (13)
C9	−0.4033 (10)	1.1358 (4)	0.8497 (4)	0.0410 (13)
C10	−0.5053 (12)	1.2129 (5)	0.9167 (5)	0.0589 (16)
H10	−0.4389	1.2138	0.9898	0.071*
C11	−0.6996 (13)	1.2877 (5)	0.8800 (5)	0.0694 (19)

H11	−0.7650	1.3381	0.9277	0.083*
C12	−0.7996 (11)	1.2885 (5)	0.7710 (5)	0.0558 (16)
C13	−0.7035 (13)	1.2128 (5)	0.7021 (5)	0.0655 (18)
H13	−0.7698	1.2125	0.6289	0.079*
C14	−0.5079 (11)	1.1370 (5)	0.7412 (4)	0.0549 (16)
H14	−0.4450	1.0857	0.6938	0.066*
C15	0.8230 (15)	0.4970 (7)	1.0500 (8)	0.107 (3)
H15A	0.7983	0.4329	1.0048	0.160*
H15B	0.8185	0.4763	1.1246	0.160*
H15C	0.9970	0.5302	1.0405	0.160*
H2	−0.076 (13)	1.017 (5)	0.7543 (17)	0.080*
H5A	0.211 (5)	0.997 (5)	0.058 (4)	0.080*
H5B	0.490 (7)	0.992 (5)	0.054 (4)	0.080*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.063 (3)	0.055 (2)	0.057 (2)	0.014 (2)	0.001 (2)	0.002 (2)
O2	0.071 (3)	0.059 (2)	0.090 (3)	0.024 (2)	−0.013 (3)	0.007 (2)
O3	0.056 (3)	0.081 (3)	0.032 (2)	0.017 (2)	0.0050 (18)	0.007 (2)
O4	0.090 (4)	0.068 (3)	0.086 (3)	0.044 (3)	−0.002 (3)	−0.005 (2)
O5	0.054 (3)	0.122 (4)	0.038 (2)	−0.011 (3)	0.003 (2)	0.005 (2)
N1	0.042 (3)	0.043 (3)	0.051 (3)	0.004 (2)	−0.004 (2)	−0.003 (2)
N2	0.048 (3)	0.050 (3)	0.039 (3)	0.009 (2)	0.007 (2)	0.002 (2)
C1	0.043 (3)	0.046 (3)	0.057 (4)	0.004 (3)	−0.004 (3)	−0.022 (3)
C2	0.041 (4)	0.046 (3)	0.050 (3)	0.001 (3)	−0.001 (3)	−0.014 (3)
C3	0.047 (4)	0.047 (3)	0.076 (5)	0.010 (3)	−0.012 (4)	−0.010 (3)
C4	0.056 (5)	0.063 (4)	0.097 (6)	0.025 (4)	−0.001 (4)	−0.027 (4)
C5	0.052 (4)	0.076 (5)	0.091 (6)	0.011 (4)	0.011 (4)	−0.035 (4)
C6	0.048 (3)	0.065 (4)	0.064 (4)	0.001 (3)	0.000 (3)	−0.017 (3)
C7	0.050 (4)	0.055 (4)	0.041 (3)	0.003 (3)	0.001 (3)	−0.009 (3)
C8	0.034 (3)	0.042 (3)	0.051 (4)	−0.001 (3)	0.003 (3)	−0.005 (3)
C9	0.041 (3)	0.040 (3)	0.042 (3)	0.003 (3)	0.002 (3)	−0.002 (2)
C10	0.065 (4)	0.068 (4)	0.043 (3)	0.009 (4)	−0.001 (3)	−0.007 (3)
C11	0.078 (5)	0.070 (4)	0.059 (5)	0.037 (4)	0.008 (4)	−0.014 (3)
C12	0.052 (4)	0.049 (3)	0.064 (4)	0.017 (3)	−0.004 (3)	−0.001 (3)
C13	0.076 (5)	0.072 (4)	0.047 (4)	0.023 (4)	−0.003 (3)	0.004 (3)
C14	0.057 (4)	0.063 (4)	0.044 (3)	0.025 (3)	0.001 (3)	−0.007 (3)
C15	0.066 (5)	0.088 (5)	0.160 (8)	0.039 (4)	−0.015 (5)	0.028 (5)

Geometric parameters (Å, °)

O1—C2	1.352 (6)	C4—H4A	0.93
O1—H1	0.82	C5—C6	1.367 (8)
O2—C3	1.377 (7)	C5—H5	0.93
O2—C15	1.459 (7)	C6—H6	0.93
O3—C8	1.240 (6)	C7—H7	0.93
O4—C12	1.361 (6)	C8—C9	1.480 (7)
O4—H4	0.82	C9—C10	1.378 (7)

O5—H5A	0.86 (3)	C9—C14	1.388 (6)
O5—H5B	0.84 (4)	C10—C11	1.361 (7)
N1—C7	1.279 (6)	C10—H10	0.93
N1—N2	1.385 (6)	C11—C12	1.388 (8)
N2—C8	1.328 (6)	C11—H11	0.93
N2—H2	0.90 (3)	C12—C13	1.369 (7)
C1—C6	1.388 (7)	C13—C14	1.381 (7)
C1—C2	1.407 (7)	C13—H13	0.93
C1—C7	1.440 (7)	C14—H14	0.93
C2—C3	1.407 (7)	C15—H15A	0.96
C3—C4	1.362 (8)	C15—H15B	0.96
C4—C5	1.379 (8)	C15—H15C	0.96
C2—O1—H1	109.5	C1—C7—H7	118.5
C3—O2—C15	116.3 (5)	O3—C8—N2	122.1 (5)
C12—O4—H4	109.5	O3—C8—C9	120.7 (5)
H5A—O5—H5B	108 (3)	N2—C8—C9	117.1 (5)
C7—N1—N2	115.5 (4)	C10—C9—C14	117.0 (5)
C8—N2—N1	120.4 (4)	C10—C9—C8	120.5 (5)
C8—N2—H2	121 (4)	C14—C9—C8	122.5 (5)
N1—N2—H2	118 (4)	C11—C10—C9	122.5 (5)
C6—C1—C2	119.2 (5)	C11—C10—H10	118.8
C6—C1—C7	119.0 (6)	C9—C10—H10	118.8
C2—C1—C7	121.8 (5)	C10—C11—C12	119.7 (5)
O1—C2—C3	117.8 (5)	C10—C11—H11	120.2
O1—C2—C1	123.7 (5)	C12—C11—H11	120.2
C3—C2—C1	118.5 (5)	O4—C12—C13	121.6 (5)
C4—C3—O2	126.4 (6)	O4—C12—C11	119.0 (5)
C4—C3—C2	120.7 (6)	C13—C12—C11	119.4 (5)
O2—C3—C2	112.9 (5)	C12—C13—C14	120.0 (6)
C3—C4—C5	120.5 (6)	C12—C13—H13	120.0
C3—C4—H4A	119.7	C14—C13—H13	120.0
C5—C4—H4A	119.7	C13—C14—C9	121.5 (5)
C6—C5—C4	120.0 (6)	C13—C14—H14	119.3
C6—C5—H5	120.0	C9—C14—H14	119.3
C4—C5—H5	120.0	O2—C15—H15A	109.5
C5—C6—C1	121.1 (6)	O2—C15—H15B	109.5
C5—C6—H6	119.4	H15A—C15—H15B	109.5
C1—C6—H6	119.4	O2—C15—H15C	109.5
N1—C7—C1	122.9 (5)	H15A—C15—H15C	109.5
N1—C7—H7	118.5	H15B—C15—H15C	109.5

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N1	0.82	1.99	2.687 (5)	142
N2—H2···O5^{i}	0.90 (3)	1.99 (3)	2.820 (5)	154 (5)
O4—H4···O2^{ii}	0.82	2.15	2.872 (7)	147
O5—H5A···O3^{iii}	0.86 (3)	1.97 (3)	2.769 (6)	156 (6)

O5—H5B···O3^{iv}	0.84 (4)	2.01 (3)	2.769 (6)	148 (5)
C7—H7···O5^{i}	0.93	2.48	3.229 (7)	138
C14—H14···O5^{i}	0.93	2.34	3.218 (7)	158

Symmetry codes: (i) *x*−1/2, −*y*+2, *z*+1/2; (ii) *x*−3/2, −*y*+2, *z*−1/2; (iii) *x*, *y*, *z*−1; (iv) *x*+1, *y*, *z*−1.

Fig. 1

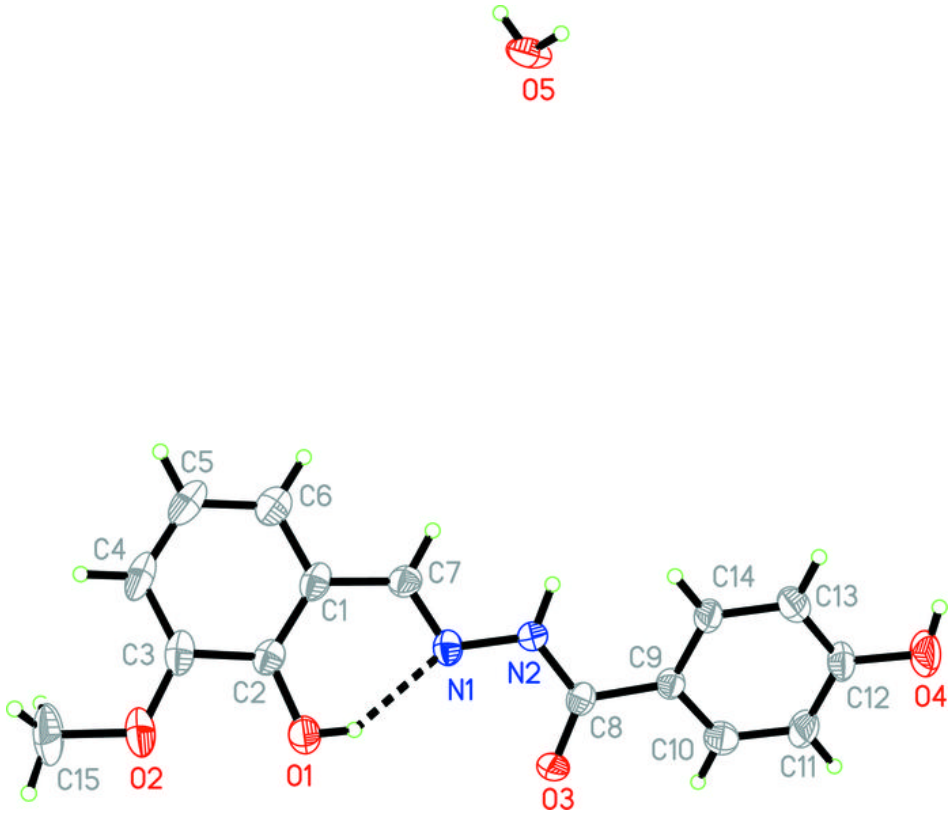
O5
C5
C6
C4
C7
C1
C3
C2
N1
N2
C9
C14
C13
C8
C12
O4
C10
C11
C15
O2
O1
O3

Fig. 2