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4-Hydroxy-*N*′-[(*E*)-(2-hydroxyphenyl)-methylidene]benzohydrazide

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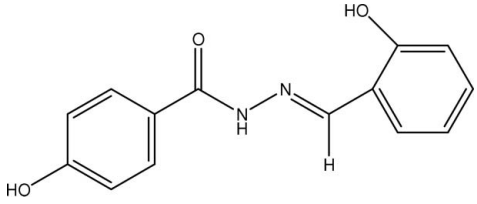
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.001 Å; R factor = 0.044; wR factor = 0.138; data-to-parameter ratio = 28.2.

In the title compound, $C_{14}H_{12}N_2O_3$, the dihedral angle between the two benzene rings is 21.70 (4)°. An intramolecular O—H···N hydrogen bond generates an S(6) ring motif. In the crystal structure, the molecules are linked by intermolecular O—H···O, N—H···O and C—H···O hydrogen bonds into a three-dimensional framework.

Related literature

For hydrogen-bond motifs, see: Bernstein *et al.* (1995). For bond-length data, see: Allen *et al.* (1987). For related structures, see: Lyubchova *et al.* (1995); Ferguson *et al.* (2005); Fun *et al.* (1996); Lever (1972); Mani Naidu *et al.* (1996); Pelizzi & Pelizzi (1980); Shan *et al.* (2003).



Experimental

Crystal data

$C_{14}H_{12}N_2O_3$
M_r = 256.26
Monoclinic, $P2_1/c$
a = 13.5978 (5) Å
b = 8.0656 (3) Å
c = 11.4128 (4) Å
β = 109.238 (2)°
V = 1181.80 (8) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 100.0 (1) K
0.41 × 0.21 × 0.09 mm

Data collection

Bruker SMART APEX2 CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.878, T_{max} = 0.991
34050 measured reflections
5185 independent reflections
4394 reflections with $I > 2\sigma(I)$
R_{int} = 0.034

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.138
S = 1.07
5185 reflections
184 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.54 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H3A···N2	0.88 (2)	1.85 (2)	2.616 (1)	146 (2)
O1—H1B···O2^{i}	0.89 (2)	1.80 (2)	2.682 (1)	171 (2)
N1—H1C···O3^{ii}	0.88 (2)	2.30 (2)	3.122 (1)	156 (1)
C12—H12A···O2^{iii}	0.93	2.45	3.176 (1)	134

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *APEX2*; data reduction: *SAINT* (Bruker, 2005); program(s) used to solve structure: *SHELXTL* (Sheldrick, 1998); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*, *PARST* (Nardelli, 1995) and *PLATON* (Spek, 2003).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI2382).

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supplementary materials

Acta Cryst. (2007). E**63**, o2880 [**doi:10.1107/S1600536807021745**]

4-Hydroxy-*N*'-[(*E*)-(2-hydroxyphenyl)methylidene]benzohydrazide

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Comment

The chemical properties of hydrazone derivatives have been intensively investigated in several research fields mainly due to their facile synthesis, tuneable electronic and steric properties, and good chelating ability (Pelizzi & Pelizzi, 1980). Some derivatives of the title compound, (I), were used for the determination of glucose (Lever *et al.*, 1972). These compounds crystallize in the E conformation (Shan *et al.*, 2003; Fun *et al.*, 1996; Naidu *et al.*, 1996), and isomeric compounds have also been prepared (Ferguson *et al.*, 2005). We report here the crystal structure of the title compound, (I).

The bond lengths and angles in (I) have normal values (Allen *et al.*, 1987) and are comparable to those in a related structure (Lyubchova *et al.*, 1995). The molecule is slightly twisted about C6—C7 bond, with diheral angle between the two benzene rings (C1—C6 and C9—C14) being 21.70 (4)°. The hydroxyl groups at C3 and C14 lie almost coplanar to the benzene rings to which they are attached, with out-of-plane distances for O1 and O3 atoms of 0.018 (1) and 0.024 (1) Å, respectively.

An intramolecular O3—H3A···N2 interaction (Table 1 and Fig. 1) generates an S(6) ring motif (Bernstein *et al.*, 1995). In the crystal structure, the molecules are linked by intermolecular O—H···O, N—H···O and C—H···O type hydrogen bonds into a three-dimensional framework (Fig. 2).

Experimental

A solution of salicylaldehyde (122 mg, 1 mmol) in methanol (10 m) was added dropwise to a methanol solution (10 ml) of 4-hydroxybenzhydrazide (136 mg, 1 mmol) and the mixture was refluxed for 2 h. The resulting solution was evaporated on a steam bath to 5 ml and cooled to room temperature. Yellow crystals of (I) suitable for X-ray diffraction separated out and were filtered off, then washed with 5 ml of cooled methanol and dried in air.

Refinement

H atoms on N and O atoms were located in a difference map and refined isotropically [N—H = 0.88 (2) Å and O—H = 0.88 (2)-0.89 (2) Å]. The remaining H atoms were positioned geometrically and treated as riding, with C—H = 0.93 Å and $U_{iso}(H) = 1.2U_{eq}(C)$.

Figures

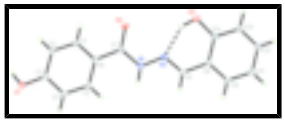

Fig. 1. The molecular structure of (I), showing 50% probability displacement ellipsoids and the atomic numbering. The dashed line indicates a hydrogen bond.

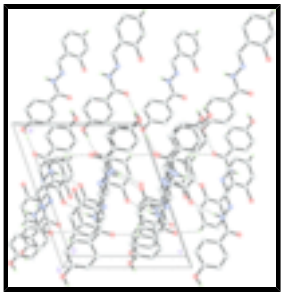

Fig. 2. The crystal packing of (I), viewed down the *b* axis. Hydrogen bonds are shown as dashed lines. H atoms not involved in hydrogen bonding have been omitted.

4-Hydroxy-*N*'-[(*E*)-(2-hydroxyphenyl)methylidene]benzohydrazide

Crystal data

$C_{14}H_{12}N_2O_3$	F_{000} = 536
M_r = 256.26	D_x = 1.440 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 5136 reflections
a = 13.5978 (5) Å	θ = 1.6–35.0º
b = 8.0656 (3) Å	μ = 0.10 mm^{-1}
c = 11.4128 (4) Å	T = 100.0 (1) K
β = 109.238 (2)º	Block, colourless
V = 1181.80 (8) Å^3	0.41 × 0.21 × 0.09 mm
Z = 4	

Data collection

Bruker SMART APEX2 CCD area-detector diffractometer	5185 independent reflections
Radiation source: fine-focus sealed tube	4394 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.034
Detector resolution: 8.33 pixels mm^{-1}	θ_{max} = 35.0º
T = 100.0(1) K	θ_{min} = 1.6º
ω scans	h = −21→21
Absorption correction: multi-scan (SADABS; Bruker, 2005)	k = −12→12
T_{min} = 0.878, T_{max} = 0.991	l = −18→18
34050 measured reflections	

Refinement

Refinement on F^2	H atoms treated by a mixture of independent and constrained refinement
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.0831P)^2 + 0.2009P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.044	$(\Delta/\sigma)_{max}$ = 0.001
$wR(F^2)$ = 0.138	$\Delta\rho_{max}$ = 0.54 e Å^{-3}

S = 1.07
5185 reflections
184 parameters
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites

$\Delta\rho_{min}$ = −0.28 e Å^{-3}
Extinction correction: none

Special details

Experimental. The data was collected with the Oxford Cyrosystem Cobra low-temperature attachment.

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	−0.03503 (5)	1.28061 (9)	0.00201 (6)	0.01848 (13)
O2	0.21825 (4)	0.84194 (9)	0.46567 (5)	0.01660 (13)
O3	0.44856 (5)	0.60515 (9)	0.67431 (6)	0.01678 (13)
N1	0.34439 (5)	0.87903 (9)	0.38035 (6)	0.01407 (13)
N2	0.41034 (5)	0.78729 (9)	0.47462 (6)	0.01335 (13)
C1	0.08643 (6)	1.07094 (10)	0.29951 (7)	0.01423 (14)
H1A	0.0755	1.0531	0.3748	0.017*
C2	0.01575 (6)	1.16534 (11)	0.20889 (7)	0.01492 (14)
H2A	−0.0419	1.2111	0.2235	0.018*
C3	0.03152 (6)	1.19150 (11)	0.09501 (7)	0.01459 (14)
C4	0.11898 (6)	1.12422 (12)	0.07450 (7)	0.01746 (15)
H4A	0.1299	1.1425	−0.0007	0.021*
C5	0.18981 (6)	1.03015 (12)	0.16573 (7)	0.01678 (15)
H5A	0.2479	0.9857	0.1513	0.020*
C6	0.17425 (5)	1.00178 (10)	0.27965 (7)	0.01306 (13)
C7	0.24494 (5)	0.90150 (10)	0.38087 (7)	0.01272 (13)
C8	0.50372 (6)	0.76244 (10)	0.47417 (7)	0.01381 (14)
H8A	0.5245	0.8046	0.4102	0.017*
C9	0.57653 (5)	0.66847 (10)	0.57382 (6)	0.01236 (13)
C10	0.67930 (6)	0.64939 (11)	0.57431 (7)	0.01548 (14)
H10A	0.6988	0.6968	0.5110	0.019*
C11	0.75215 (6)	0.56124 (12)	0.66729 (8)	0.01823 (16)
H11A	0.8199	0.5492	0.6663	0.022*
C12	0.72291 (6)	0.49078 (12)	0.76224 (7)	0.01814 (15)

H12A	0.7717	0.4326	0.8254	0.022*
C13	0.62144 (6)	0.50647 (11)	0.76368 (7)	0.01626 (15)
H13A	0.6027	0.4582	0.8273	0.020*
C14	0.54761 (6)	0.59453 (10)	0.66996 (7)	0.01319 (13)
H1B	−0.0923 (12)	1.303 (2)	0.0209 (14)	0.029 (4)*
H1C	0.3652 (12)	0.914 (2)	0.3191 (15)	0.034 (4)*
H3A	0.4105 (13)	0.661 (2)	0.6095 (17)	0.043 (5)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0146 (2)	0.0239 (3)	0.0172 (3)	0.0055 (2)	0.00550 (19)	0.0054 (2)
O2	0.0139 (2)	0.0230 (3)	0.0136 (2)	0.0001 (2)	0.00554 (18)	0.0013 (2)
O3	0.0159 (2)	0.0211 (3)	0.0160 (2)	0.0003 (2)	0.0088 (2)	0.0024 (2)
N1	0.0115 (2)	0.0174 (3)	0.0135 (3)	0.0025 (2)	0.0044 (2)	0.0030 (2)
N2	0.0123 (2)	0.0151 (3)	0.0123 (2)	0.0016 (2)	0.0036 (2)	0.0009 (2)
C1	0.0130 (3)	0.0159 (3)	0.0149 (3)	0.0005 (2)	0.0061 (2)	−0.0003 (2)
C2	0.0127 (3)	0.0164 (3)	0.0166 (3)	0.0021 (2)	0.0061 (2)	0.0014 (3)
C3	0.0124 (3)	0.0161 (3)	0.0151 (3)	0.0006 (2)	0.0043 (2)	0.0007 (2)
C4	0.0150 (3)	0.0244 (4)	0.0143 (3)	0.0047 (3)	0.0067 (2)	0.0022 (3)
C5	0.0140 (3)	0.0226 (4)	0.0148 (3)	0.0044 (3)	0.0061 (2)	0.0012 (3)
C6	0.0113 (3)	0.0149 (3)	0.0129 (3)	0.0008 (2)	0.0039 (2)	−0.0002 (2)
C7	0.0112 (3)	0.0144 (3)	0.0123 (3)	0.0002 (2)	0.0035 (2)	−0.0018 (2)
C8	0.0132 (3)	0.0161 (3)	0.0127 (3)	0.0010 (2)	0.0049 (2)	0.0020 (2)
C9	0.0121 (3)	0.0134 (3)	0.0115 (3)	0.0003 (2)	0.0038 (2)	0.0009 (2)
C10	0.0127 (3)	0.0190 (4)	0.0153 (3)	0.0013 (3)	0.0053 (2)	0.0029 (3)
C11	0.0142 (3)	0.0215 (4)	0.0180 (3)	0.0028 (3)	0.0039 (2)	0.0033 (3)
C12	0.0192 (3)	0.0190 (4)	0.0140 (3)	0.0036 (3)	0.0025 (2)	0.0024 (3)
C13	0.0206 (3)	0.0160 (3)	0.0127 (3)	0.0020 (3)	0.0063 (2)	0.0020 (2)
C14	0.0149 (3)	0.0133 (3)	0.0124 (3)	0.0000 (2)	0.0059 (2)	−0.0007 (2)

Geometric parameters (Å, °)

O1—C3	1.3524 (10)	C4—H4A	0.93
O1—H1B	0.892 (15)	C5—C6	1.4031 (11)
O2—C7	1.2372 (9)	C5—H5A	0.93
O3—C14	1.3666 (9)	C6—C7	1.4768 (10)
O3—H3A	0.877 (19)	C8—C9	1.4513 (10)
N1—C7	1.3663 (9)	C8—H8A	0.93
N1—N2	1.3687 (9)	C9—C10	1.4040 (10)
N1—H1C	0.884 (16)	C9—C14	1.4137 (10)
N2—C8	1.2872 (9)	C10—C11	1.3857 (11)
C1—C2	1.3849 (11)	C10—H10A	0.93
C1—C6	1.4025 (10)	C11—C12	1.3925 (12)
C1—H1A	0.93	C11—H11A	0.93
C2—C3	1.4012 (11)	C12—C13	1.3911 (11)
C2—H2A	0.93	C12—H12A	0.93
C3—C4	1.3953 (11)	C13—C14	1.3959 (11)
C4—C5	1.3880 (11)	C13—H13A	0.93

C3—O1—H1B	109.3 (10)	O2—C7—N1	119.86 (7)
C14—O3—H3A	108.5 (11)	O2—C7—C6	122.78 (6)
C7—N1—N2	117.47 (6)	N1—C7—C6	117.34 (6)
C7—N1—H1C	122.4 (10)	N2—C8—C9	119.83 (7)
N2—N1—H1C	119.9 (10)	N2—C8—H8A	120.1
C8—N2—N1	118.21 (6)	C9—C8—H8A	120.1
C2—C1—C6	121.08 (7)	C10—C9—C14	118.80 (7)
C2—C1—H1A	119.5	C10—C9—C8	118.78 (7)
C6—C1—H1A	119.5	C14—C9—C8	122.42 (6)
C1—C2—C3	119.66 (7)	C11—C10—C9	121.25 (7)
C1—C2—H2A	120.2	C11—C10—H10A	119.4
C3—C2—H2A	120.2	C9—C10—H10A	119.4
O1—C3—C4	117.51 (7)	C10—C11—C12	119.34 (7)
O1—C3—C2	122.73 (7)	C10—C11—H11A	120.3
C4—C3—C2	119.76 (7)	C12—C11—H11A	120.3
C5—C4—C3	120.39 (7)	C13—C12—C11	120.71 (7)
C5—C4—H4A	119.8	C13—C12—H12A	119.6
C3—C4—H4A	119.8	C11—C12—H12A	119.6
C4—C5—C6	120.33 (7)	C12—C13—C14	120.20 (7)
C4—C5—H5A	119.8	C12—C13—H13A	119.9
C6—C5—H5A	119.8	C14—C13—H13A	119.9
C1—C6—C5	118.77 (7)	O3—C14—C13	118.20 (7)
C1—C6—C7	117.44 (6)	O3—C14—C9	122.09 (7)
C5—C6—C7	123.79 (7)	C13—C14—C9	119.71 (7)
C7—N1—N2—C8	178.74 (7)	C5—C6—C7—N1	20.95 (12)
C6—C1—C2—C3	0.48 (12)	N1—N2—C8—C9	178.82 (7)
C1—C2—C3—O1	179.17 (8)	N2—C8—C9—C10	−177.14 (8)
C1—C2—C3—C4	−0.89 (12)	N2—C8—C9—C14	3.29 (12)
O1—C3—C4—C5	−179.38 (8)	C14—C9—C10—C11	−0.55 (12)
C2—C3—C4—C5	0.67 (13)	C8—C9—C10—C11	179.87 (8)
C3—C4—C5—C6	−0.04 (13)	C9—C10—C11—C12	−0.23 (14)
C2—C1—C6—C5	0.15 (12)	C10—C11—C12—C13	0.75 (14)
C2—C1—C6—C7	−179.41 (7)	C11—C12—C13—C14	−0.48 (13)
C4—C5—C6—C1	−0.37 (13)	C12—C13—C14—O3	179.26 (8)
C4—C5—C6—C7	179.17 (8)	C12—C13—C14—C9	−0.31 (12)
N2—N1—C7—O2	1.13 (11)	C10—C9—C14—O3	−178.74 (7)
N2—N1—C7—C6	179.47 (7)	C8—C9—C14—O3	0.83 (12)
C1—C6—C7—O2	18.77 (12)	C10—C9—C14—C13	0.81 (12)
C5—C6—C7—O2	−160.76 (8)	C8—C9—C14—C13	−179.62 (7)
C1—C6—C7—N1	−159.51 (7)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H3A···N2	0.88 (2)	1.85 (2)	2.616 (1)	146 (2)
O1—H1B···O2^{i}	0.89 (2)	1.80 (2)	2.682 (1)	171 (2)
N1—H1C···O3^{ii}	0.88 (2)	2.30 (2)	3.122 (1)	156 (1)
C12—H12A···O2^{iii}	0.93	2.45	3.176 (1)	134

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

Fig. 1

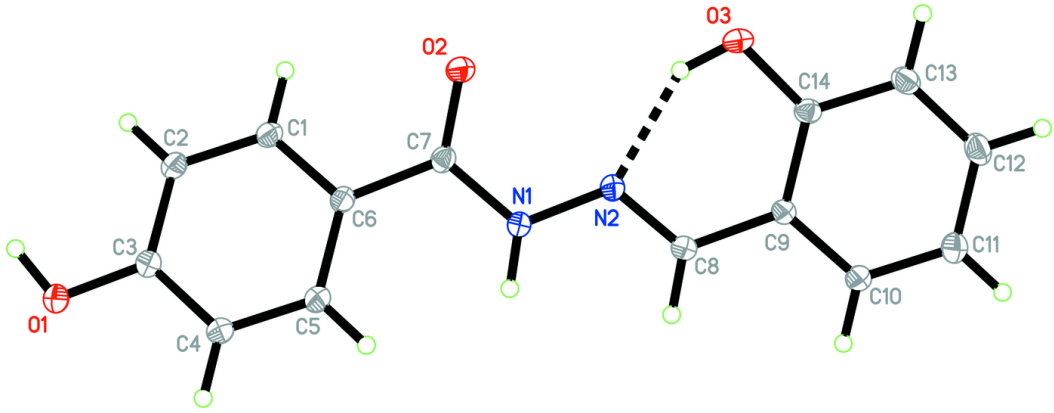

Fig. 2