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4-Hydroxy-*N*′-(2-hydroxybenzylidene)-benzohydrazide

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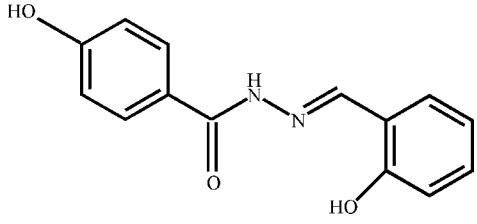
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.058; wR factor = 0.127; data-to-parameter ratio = 15.0.

In the title molecule, $C_{14}H_{12}N_2O_3$, the –N—C(=O)– and 2-(iminomethyl)phenol fragments are almost coplanar, with an r.m.s. deviation of 0.0308 Å. The dihedral angle between the mean planes of *p*-phenol and *N*′-(2-hydroxybenzylidene)formohydrazide is 20.95 (7)°. Intermolecular O—H···O hydrogen bonds link the molecules into zigzag chains running along the *b* axis. Weak intermolecular N—H···O hydrogen bonds stabilize these chains into a three-dimensional packing.

Related literature

The chemistry of aroylhydrazone compounds, their biological activities and potential chelating functions were recently discussed by Xue & Liu (2006), Yang & Pan (2004) and Qiu *et al.* (2006), respectively.



Experimental

Crystal data

$C_{14}H_{12}N_2O_3$
M_r = 256.26
Monoclinic, $P2_1/c$
a = 13.583 (8) Å
b = 8.237 (4) Å
c = 11.499 (8) Å
β = 109.53 (2)°
V = 1212.5 (13) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.10 mm^{-1}
T = 293 (2) K
0.26 × 0.10 × 0.08 mm

Data collection

Rigaku Weissenberg IP diffractometer
Absorption correction: multi-scan (*TEXRAY*; Molecular Structure Corporation, 1999)
T_{min} = 0.988, T_{max} = 0.992
11461 measured reflections
2776 independent reflections
1342 reflections with $I > 2\sigma(I)$
R_{int} = 0.096

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.058
$wR(F^2)$ = 0.127
S = 0.99
2776 reflections
185 parameters
3 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.19 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H02A···O1^{i}	0.851 (18)	1.852 (19)	2.698 (3)	172 (4)
O3—H03A···N2	0.847 (17)	1.86 (2)	2.620 (3)	149 (3)
N1—H1N···O3^{ii}	0.875 (17)	2.333 (19)	3.153 (3)	156 (2)

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

Data collection: *TEXRAY* (Molecular Structure Corporation, 1999); cell refinement: *TEXRAY*; data reduction: *TEXSAN* (Molecular Structure Corporation, 1999); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEX* (McArdle, 1995); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV2237).

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supplementary materials

Acta Cryst. (2007). E**63**, o2864 [**doi:10.1107/S1600536807021277**]

4-Hydroxy-*N*'-(2-hydroxybenzylidene)benzohydrazide

X.-C. Lin, H. Yin and Y. Lin

Comment

The chemistry of aroylhydrazone compounds have recently attracted great interest(Xue *et al.*, 2006), owing to their biological activities (Yang & Pan, 2004) and potential chelating functions (Qiu *et al.*, 2006). We report here the synthesis and crystal structure of the title compound (I), obtained by the condensation of salicylhydrazide with 4-Hydroxybenzohydrazine.

The molecule structure of (I) is shown in Fig. 1. The N1, C7, O1 fragment is almost coplanar with the 2-(iminomethyl)phenol (C8—C13, O3, C14, N2) with an r.m.s. deviation of 0.0309 Å. The dihedral angle between the phenol (C1—C6, O2) and *N*'-(2-hydroxybenzylidene) formohydrazide (C8—C13, O3, C14, N2, N1, C7, O1) is 20.95 (7)°. There are one intramolecular hydrogen bond (O3—H03A···N2) and two intermolecular hydrogen bonds (O2—H02A···O1^{i} (i: -x, y-1/2, -z+3/2) and N1—H1N···O3^{i} (i: x, -y+1/2, z-1/2))(Table 1). The intermolecular O—H···O hydrogen bonds link the molecules into zig-zag chains running along the b-axis. The weak intermolecular N—H···O hydrogen bonds stabilize the chains into a three-dimensional packing. (Fig. 2).

Experimental

An equimolar mixture of salicylhydrazide (15 mmol) and 4-Hydroxybenzohydrazine (15 mmol) in ethanol was refluxed in a round-bottomed flask for about 3 h. The resulting precipitate was collected by filtration and washed with methanol and diethylether. The product (0.0358 g) was dissolved in methanol (15 ml), and kept at room temperature for 8 d to obtain yellow single crystals.

Refinement

Atoms H02A, H03A and H1N were located on a difference Fourier map and refined isotropically with bond restraints O—H =0.85 (2) Å and N—H =0.86 (2) Å. All other H atoms were positioned geometrically and treated as riding [C—H =0.93Å and Uiso(H)=1.2Ueq(C)].

Figures

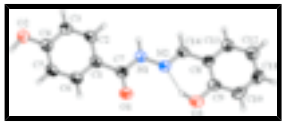

Fig. 1. The molecular structure of (I), showing the atom-numbering scheme and 50% probability displacement ellipsoids. The intramolecular hydrogen bonds are shown as dashed lines.

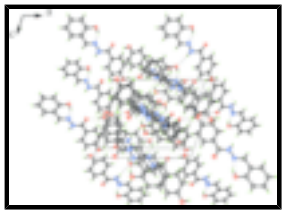

Fig. 2. Packing diagram of (I), viewed down the b axis, showing hydrogen-bonded 3D network. Hydrogen bonds are indicated by dashed lines.

N'-(2-hydroxybenzylidene)-4-hydroxybenzohydrazide

Crystal data

$C_{14}H_{12}N_2O_3$	F_{000} = 536
M_r = 256.26	D_x = 1.404 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P2ybc	Cell parameters from 6169 reflections
a = 13.583 (8) Å	θ = 3.1–27.5º
b = 8.237 (4) Å	μ = 0.10 mm^{-1}
c = 11.499 (8) Å	T = 293 (2) K
β = 109.53 (2)º	Needle, yellow
V = 1212.5 (13) Å^3	0.26 × 0.10 × 0.08 mm
Z = 4	

Data collection

Rigaku Weissenberg IP diffractometer	1342 reflections with $I > 2\sigma(I)$
Radiation source: rotor target	R_{int} = 0.096
Monochromator: graphite	θ_{max} = 27.5º
T = 293(2) K	θ_{min} = 3.1º
ω scans	h = −17→17
Absorption correction: multi-scan (TEXRAY; Molecular Structure Corporation, 1999)	k = −10→9
T_{min} = 0.988, T_{max} = 0.992	l = −14→14
11461 measured reflections	Standard reflections: none
2776 independent reflections	

Refinement

Refinement on F^2	H atoms treated by a mixture of independent and constrained refinement
Least-squares matrix: full	$w = 1/[\sigma^2(F_o^2) + (0.0481P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.058	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2)$ = 0.127	$\Delta\rho_{max}$ = 0.18 e Å^{-3}
S = 0.99	$\Delta\rho_{min}$ = −0.19 e Å^{-3}
2776 reflections	Extinction correction: SHELXL (Sheldrick, 1997), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
185 parameters	Extinction coefficient: 0.0057 (16)
3 restraints	
Primary atom site location: structure-invariant direct methods	
Secondary atom site location: difference Fourier map	

Hydrogen site location: inferred from neighbouring sites

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}
C1	0.17334 (16)	−0.0018 (3)	0.2802 (2)	0.0383 (6)
C2	0.18908 (18)	−0.0299 (3)	0.1686 (2)	0.0507 (7)
H2A	0.2472	0.0144	0.1549	0.061*
C3	0.11982 (18)	−0.1225 (3)	0.0780 (2)	0.0545 (7)
H3A	0.1313	−0.1397	0.0036	0.065*
C4	0.03341 (17)	−0.1898 (3)	0.0972 (2)	0.0434 (6)
C5	0.01702 (17)	−0.1645 (3)	0.2085 (2)	0.0451 (6)
H5A	−0.0407	−0.2101	0.2223	0.054*
C6	0.08618 (16)	−0.0720 (3)	0.2981 (2)	0.0412 (6)
H6A	0.0747	−0.0558	0.3726	0.049*
C7	0.24335 (16)	0.0966 (3)	0.3817 (2)	0.0385 (6)
C8	0.57374 (16)	0.3308 (3)	0.5743 (2)	0.0358 (6)
C9	0.54584 (18)	0.4014 (3)	0.6690 (2)	0.0397 (6)
C10	0.6200 (2)	0.4887 (3)	0.7614 (2)	0.0506 (7)
H10A	0.6017	0.5362	0.8247	0.061*
C11	0.7198 (2)	0.5046 (3)	0.7592 (3)	0.0550 (7)
H11A	0.7689	0.5620	0.8218	0.066*
C12	0.7483 (2)	0.4377 (3)	0.6669 (3)	0.0554 (8)
H12A	0.8160	0.4507	0.6658	0.067*
C13	0.67605 (17)	0.3508 (3)	0.5753 (2)	0.0472 (7)
H13A	0.6958	0.3044	0.5127	0.057*
C14	0.50090 (17)	0.2387 (3)	0.4760 (2)	0.0388 (6)
H14A	0.5217	0.1984	0.4123	0.047*
O1	0.21602 (11)	0.1535 (2)	0.46486 (16)	0.0491 (5)
O2	−0.03193 (14)	−0.2793 (2)	0.00421 (18)	0.0579 (5)
H02A	−0.087 (2)	−0.303 (4)	0.021 (3)	0.118 (14)*
O3	0.44705 (14)	0.3907 (2)	0.67470 (18)	0.0525 (5)
H03A	0.4119 (18)	0.337 (3)	0.612 (2)	0.058 (9)*
N1	0.34201 (14)	0.1221 (3)	0.3811 (2)	0.0424 (5)
H1N	0.3640 (18)	0.088 (3)	0.322 (2)	0.057 (8)*
N2	0.40823 (14)	0.2122 (2)	0.47586 (17)	0.0381 (5)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0326 (12)	0.0429 (14)	0.0403 (15)	0.0015 (11)	0.0134 (11)	0.0067 (12)
C2	0.0384 (13)	0.0720 (18)	0.0465 (17)	−0.0132 (13)	0.0206 (12)	−0.0032 (15)
C3	0.0463 (14)	0.080 (2)	0.0426 (16)	−0.0140 (14)	0.0224 (12)	−0.0061 (15)
C4	0.0363 (13)	0.0486 (15)	0.0448 (16)	−0.0020 (12)	0.0131 (11)	−0.0029 (13)
C5	0.0373 (13)	0.0503 (15)	0.0538 (18)	−0.0060 (12)	0.0233 (12)	−0.0014 (14)
C6	0.0388 (13)	0.0471 (14)	0.0412 (15)	0.0002 (11)	0.0178 (11)	0.0024 (12)
C7	0.0340 (12)	0.0462 (14)	0.0360 (15)	0.0008 (11)	0.0128 (11)	0.0070 (12)
C8	0.0359 (12)	0.0409 (13)	0.0309 (13)	0.0018 (11)	0.0115 (10)	0.0002 (11)
C9	0.0452 (13)	0.0423 (13)	0.0363 (15)	0.0036 (12)	0.0199 (11)	0.0059 (12)
C10	0.0715 (18)	0.0478 (15)	0.0346 (16)	−0.0013 (14)	0.0204 (14)	−0.0052 (13)
C11	0.0545 (16)	0.0611 (17)	0.0409 (16)	−0.0129 (14)	0.0046 (13)	−0.0038 (14)
C12	0.0444 (14)	0.0650 (18)	0.0537 (19)	−0.0068 (14)	0.0121 (14)	−0.0076 (15)
C13	0.0384 (13)	0.0575 (16)	0.0466 (16)	−0.0038 (12)	0.0155 (12)	−0.0084 (13)
C14	0.0386 (13)	0.0450 (14)	0.0366 (15)	−0.0008 (11)	0.0175 (11)	−0.0044 (11)
O1	0.0383 (9)	0.0713 (12)	0.0406 (11)	−0.0005 (8)	0.0169 (8)	−0.0042 (9)
O2	0.0492 (11)	0.0751 (13)	0.0523 (13)	−0.0176 (10)	0.0208 (10)	−0.0174 (10)
O3	0.0529 (11)	0.0667 (13)	0.0468 (12)	−0.0032 (10)	0.0287 (9)	−0.0084 (11)
N1	0.0357 (11)	0.0536 (13)	0.0392 (13)	−0.0056 (10)	0.0144 (10)	−0.0074 (11)
N2	0.0350 (10)	0.0467 (12)	0.0324 (11)	−0.0026 (9)	0.0111 (9)	−0.0012 (10)

Geometric parameters (Å, °)

C1—C2	1.389 (4)	C8—C14	1.443 (3)
C1—C6	1.393 (3)	C9—O3	1.368 (3)
C1—C7	1.477 (3)	C9—C10	1.395 (3)
C2—C3	1.377 (3)	C10—C11	1.370 (4)
C2—H2A	0.9300	C10—H10A	0.9300
C3—C4	1.381 (3)	C11—C12	1.362 (4)
C3—H3A	0.9300	C11—H11A	0.9300
C4—O2	1.356 (3)	C12—C13	1.377 (3)
C4—C5	1.386 (3)	C12—H12A	0.9300
C5—C6	1.370 (3)	C13—H13A	0.9300
C5—H5A	0.9300	C14—N2	1.277 (3)
C6—H6A	0.9300	C14—H14A	0.9300
C7—O1	1.229 (3)	O2—H02A	0.851 (18)
C7—N1	1.359 (3)	O3—H03A	0.847 (17)
C8—C9	1.394 (3)	N1—N2	1.376 (3)
C8—C13	1.396 (3)	N1—H1N	0.875 (17)
C2—C1—C6	117.9 (2)	O3—C9—C8	122.5 (2)
C2—C1—C7	124.3 (2)	O3—C9—C10	117.8 (2)
C6—C1—C7	117.8 (2)	C8—C9—C10	119.6 (2)
C3—C2—C1	121.0 (2)	C11—C10—C9	120.1 (3)
C3—C2—H2A	119.5	C11—C10—H10A	119.9
C1—C2—H2A	119.5	C9—C10—H10A	119.9

C2—C3—C4	120.2 (3)	C12—C11—C10	121.1 (2)
C2—C3—H3A	119.9	C12—C11—H11A	119.4
C4—C3—H3A	119.9	C10—C11—H11A	119.4
O2—C4—C3	117.3 (2)	C11—C12—C13	119.4 (3)
O2—C4—C5	123.0 (2)	C11—C12—H12A	120.3
C3—C4—C5	119.7 (2)	C13—C12—H12A	120.3
C6—C5—C4	119.8 (2)	C12—C13—C8	121.4 (3)
C6—C5—H5A	120.1	C12—C13—H13A	119.3
C4—C5—H5A	120.1	C8—C13—H13A	119.3
C5—C6—C1	121.5 (2)	N2—C14—C8	120.6 (2)
C5—C6—H6A	119.3	N2—C14—H14A	119.7
C1—C6—H6A	119.3	C8—C14—H14A	119.7
O1—C7—N1	120.0 (2)	C4—O2—H02A	110 (3)
O1—C7—C1	122.8 (2)	C9—O3—H03A	105.8 (19)
N1—C7—C1	117.1 (2)	C7—N1—N2	117.6 (2)
C9—C8—C13	118.3 (2)	C7—N1—H1N	123.3 (16)
C9—C8—C14	122.4 (2)	N2—N1—H1N	119.0 (16)
C13—C8—C14	119.2 (2)	C14—N2—N1	118.5 (2)
C6—C1—C2—C3	0.9 (4)	C13—C8—C9—C10	−0.2 (3)
C7—C1—C2—C3	−179.6 (2)	C14—C8—C9—C10	179.9 (2)
C1—C2—C3—C4	−0.3 (4)	O3—C9—C10—C11	−179.2 (2)
C2—C3—C4—O2	179.8 (2)	C8—C9—C10—C11	−0.2 (4)
C2—C3—C4—C5	−0.3 (4)	C9—C10—C11—C12	0.7 (4)
O2—C4—C5—C6	−179.7 (2)	C10—C11—C12—C13	−0.9 (4)
C3—C4—C5—C6	0.5 (4)	C11—C12—C13—C8	0.6 (4)
C4—C5—C6—C1	0.1 (4)	C9—C8—C13—C12	0.0 (4)
C2—C1—C6—C5	−0.8 (3)	C14—C8—C13—C12	179.9 (2)
C7—C1—C6—C5	179.7 (2)	C9—C8—C14—N2	−3.3 (4)
C2—C1—C7—O1	161.2 (2)	C13—C8—C14—N2	176.8 (2)
C6—C1—C7—O1	−19.3 (3)	O1—C7—N1—N2	0.1 (3)
C2—C1—C7—N1	−19.4 (3)	C1—C7—N1—N2	−179.32 (19)
C6—C1—C7—N1	160.1 (2)	C8—C14—N2—N1	−179.2 (2)
C13—C8—C9—O3	178.8 (2)	C7—N1—N2—C14	−179.3 (2)
C14—C8—C9—O3	−1.1 (4)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H02A···O1^{i}	0.851 (18)	1.852 (19)	2.698 (3)	172 (4)
O3—H03A···N2	0.847 (17)	1.86 (2)	2.620 (3)	149 (3)
N1—H1N···O3^{ii}	0.875 (17)	2.333 (19)	3.153 (3)	156 (2)

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

Fig. 1

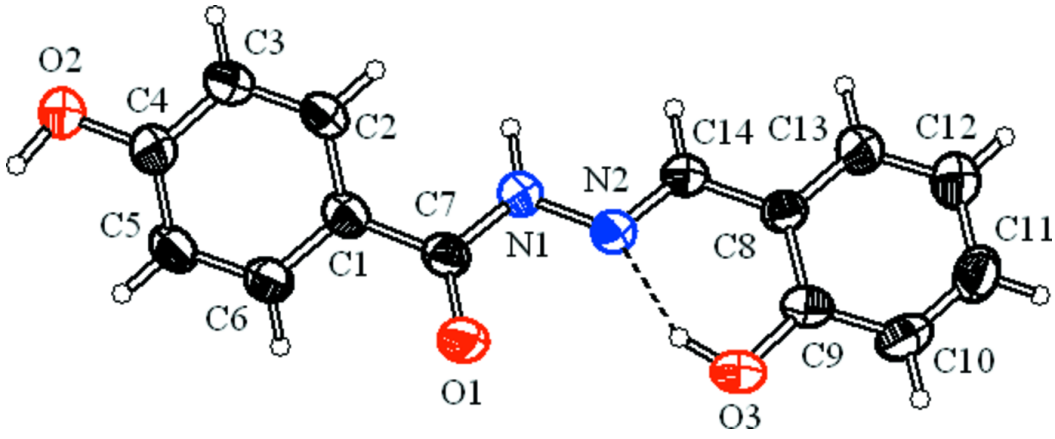

Fig. 2