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

Zhi-Gang Luo

College of Chemistry and Chemical Engineering, JiangXi Province Key Laboratory of Coordination Chemistry, JingGangShan University, Ji'an, JiangXi 343009, People's Republic of China
Correspondence e-mail: lsj_578@126.com

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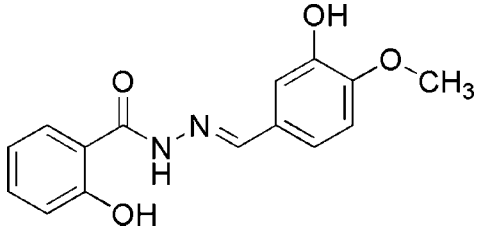
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.002 Å; R factor = 0.049; wR factor = 0.166; data-to-parameter ratio = 16.8.

The molecule of the title compound, $C_{15}H_{14}N_2O_4$, is roughly planar, except for the methyl H atoms, and displays a *trans* configuration with respect to the C=N double bond. The dihedral angle between the two rings is 3.7 (2)°. The crystal structure is stabilized by intermolecular O—H···O and N—H···O hydrogen bonds.

Related literature

For related literature, see: Allen *et al.* (1987); Qiu, Fang, Liu & Zhu (2006); Qiu, Luo, Yang & Liu (2006).



Experimental

Crystal data

$C_{15}H_{14}N_2O_4$
M_r = 286.28
Monoclinic, $P2_1/c$
a = 12.436 (3) Å
b = 7.1581 (14) Å
c = 15.048 (3) Å
β = 100.95 (3)°
V = 1315.2 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 298 (2) K
0.34 × 0.15 × 0.06 mm

Data collection

Bruker SMART APEX area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.96, T_{max} = 0.99
11560 measured reflections
3288 independent reflections
2008 reflections with $I > 2\sigma(I)$
R_{int} = 0.030

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.166
S = 1.10
3288 reflections
196 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O3^{i}	0.86	2.26	2.9616 (16)	138
O1—H2···O2	1.00 (2)	1.65 (2)	2.5424 (19)	146.3 (19)
O3—H11···O2^{ii}	0.82	2.17	2.8451 (16)	140
O3—H11···O4	0.82	2.23	2.6781 (17)	115

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

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

The author acknowledges the support of JingGangShan University.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WN2180).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Bruker (1998). *SMART* (Version 5.628) and *SAINT* (Version 6.02). Bruker AXS Inc., Madison, Wisconsin, USA.
Qiu, X.-Y., Fang, X.-N., Liu, W.-S. & Zhu, H.-L. (2006). *Acta Cryst.* E**62**, o2685–o2686.
Qiu, X.-Y., Luo, Z.-G., Yang, S.-L. & Liu, W.-S. (2006). *Acta Cryst.* E**62**, o3531–o3532.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997*a*). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Sheldrick, G. M. (1997*b*). *SHELXTL*. Version 5.10. Bruker AXS Inc., Madison, Wisconsin, USA.

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supplementary materials

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

Z.-G. Luo

Comment

Recently, we have reported a few Schiff base complexes (Qiu, Luo *et al.*, 2006; Qiu, Fang *et al.*, 2006), As an extension of our work on the structural characterization of Schiff base compounds, the crystal structure of the title compound is reported here.

In the title compound, all bond lengths are within normal ranges (Allen *et al.*, 1987) (Fig. 1). The C8═N2 bond length of 1.275 (2) Å conforms to the value for a double bond. The bond length of 1.337 (2) Å between N1 and C7 is greater than the value for a double bond, and less than the value for a single bond, because of conjugation in the molecule. The dihedral angle between the two rings is 3.7 (2)°.

The crystal structure is stabilized by intermolecular O—H···O and N—H···O hydrogen bonds. (Table 1 and Fig. 2)

Experimental

The reagents were commercial products and were used without further purification. 3-Hydroxy-4-methoxybenzaldehyde (0.1 mmol, 15.2 mg) and 2-hydroxybenzhydrazide (0.1 mmol, 15.2 mg) were dissolved in ethanol (15 ml). The reaction mixture was stirred for 30 minutes to give a clear solution. After allowing the resulting clear solution to stand at room temperature in air for 9 d, large colourless crystals were formed at the bottom of the vessel on slow evaporation of the solvent. The crystals were isolated, washed three times with ethanol and dried in a vacuum desiccator using anhydrous $CaCl_2$ (yield 66%).

Refinement

Atom H2, attached to O1, was located in a difference Fourier map and refined with an O—H distance restraint of 1.00 (2) Å. The other H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms with C—H, N—H and O—H, distances of 0.93–0.96, 0.86 and 0.82 Å, respectively. and with U_{iso}(H) = 1.2U_{eq}(Csp^2, N) or 1.5U_{eq}(methyl C, O).

Figures

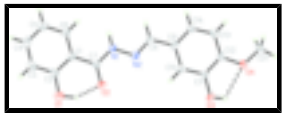

Fig. 1. The molecular structure of the title compound, showing 30% probability displacement ellipsoids and the atom-numbering scheme. Dashed lines indicate intramolecular hydrogen bonds.

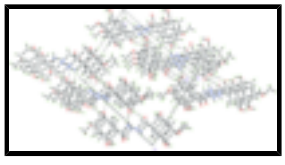

Fig. 2. The crystal packing of the title compound, viewed along the *a* axis. Dashed lines indicate intermolecular hydrogen bonds.

(*E*)—*N*'-(3-Hydroxy-4-methoxybenzylidene)-2-hydroxybenzohydrazide

Crystal data

$C_{15}H_{14}N_2O_4$	$F_{000} = 600$
$M_r = 286.28$	$D_x = 1.446$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: -P 2ybc	Cell parameters from 2865 reflections
$a = 12.436$ (3) Å	$\theta = 4.2$–26º
$b = 7.1581$ (14) Å	$\mu = 0.11$ mm^{-1}
$c = 15.048$ (3) Å	$T = 298$ (2) K
$\beta = 100.95$ (3)º	Block, colourless
$V = 1315.2$ (5) Å^3	0.34 × 0.15 × 0.06 mm
$Z = 4$	

Data collection

Bruker SMART APEX area-detector diffractometer	3288 independent reflections
Radiation source: fine-focus sealed tube	2008 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.030$
$T = 298$(2) K	$\theta_{max} = 28.5$º
ω scans	$\theta_{min} = 2.8$º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -16\rightarrow16$
$T_{min} = 0.96$, $T_{max} = 0.99$	$k = -9\rightarrow9$
11560 measured reflections	$l = -19\rightarrow20$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.049$	$w = 1/[\sigma^2(F_o^2) + (0.0815P)^2 + 0.0205P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.166$	$(\Delta/\sigma)_{max} < 0.001$
$S = 1.10$	$\Delta\rho_{max} = 0.28$ e Å^{-3}
3288 reflections	$\Delta\rho_{min} = -0.26$ e Å^{-3}
196 parameters	Extinction correction: SHELXL97 (Sheldrick, 1997a), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.009 (3)
Secondary atom site location: difference Fourier map	

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 $F^2 > 2\text{sigma}(F^2)$ 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	1.19143 (13)	0.11923 (19)	0.43668 (9)	0.0383 (3)
C2	1.30560 (13)	0.1048 (2)	0.46186 (11)	0.0455 (4)
C3	1.35507 (15)	0.1122 (2)	0.55284 (12)	0.0585 (5)
H3	1.4308	0.1022	0.5695	0.070*
C4	1.29249 (16)	0.1342 (2)	0.61828 (12)	0.0569 (5)
H4	1.3266	0.1396	0.6788	0.068*
C5	1.18013 (14)	0.1484 (2)	0.59570 (10)	0.0482 (4)
H5	1.1384	0.1631	0.6404	0.058*
C6	1.13091 (13)	0.1405 (2)	0.50587 (10)	0.0426 (4)
H6	1.0551	0.1496	0.4904	0.051*
C7	1.13998 (13)	0.10751 (19)	0.34001 (9)	0.0396 (4)
C8	0.87839 (12)	0.1348 (2)	0.20923 (9)	0.0408 (4)
H8	0.8400	0.1461	0.2563	0.049*
C9	0.81933 (12)	0.12816 (19)	0.11589 (9)	0.0384 (3)
C10	0.87688 (12)	0.11754 (19)	0.04428 (9)	0.0382 (3)
H10	0.9530	0.1131	0.0567	0.046*
C11	0.82212 (12)	0.11374 (19)	−0.04359 (9)	0.0364 (3)
C12	0.70753 (13)	0.1193 (2)	−0.06330 (10)	0.0423 (4)
C13	0.64983 (13)	0.1286 (2)	0.00710 (11)	0.0511 (4)
H13	0.5737	0.1317	−0.0054	0.061*
C14	0.70586 (13)	0.1333 (2)	0.09602 (11)	0.0470 (4)
H14	0.6668	0.1400	0.1429	0.056*
C21	0.54812 (16)	0.1023 (3)	−0.17878 (14)	0.0823 (7)
H21A	0.5231	−0.0063	−0.1512	0.123*
H21B	0.5280	0.0921	−0.2434	0.123*
H21C	0.5150	0.2119	−0.1589	0.123*
H2	1.3208 (19)	0.093 (3)	0.3393 (15)	0.084 (7)*
N1	1.03213 (10)	0.13674 (18)	0.31683 (8)	0.0444 (3)
H1	0.9941	0.1624	0.3574	0.053*
N2	0.98266 (11)	0.12502 (17)	0.22650 (8)	0.0448 (4)
O1	1.37067 (10)	0.08214 (18)	0.39977 (9)	0.0628 (4)
O2	1.19532 (9)	0.06595 (17)	0.28146 (7)	0.0521 (3)
O3	0.88194 (8)	0.10779 (14)	−0.11127 (7)	0.0461 (3)

H11	0.8403	0.1059	−0.1605	0.069*
O4	0.66287 (9)	0.11568 (16)	−0.15360 (7)	0.0558 (4)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0423 (8)	0.0413 (8)	0.0314 (7)	0.0009 (6)	0.0073 (6)	0.0002 (6)
C2	0.0432 (9)	0.0495 (9)	0.0437 (9)	−0.0010 (7)	0.0083 (7)	0.0026 (6)
C3	0.0445 (10)	0.0752 (12)	0.0513 (10)	−0.0006 (8)	−0.0020 (8)	0.0011 (8)
C4	0.0639 (12)	0.0638 (11)	0.0377 (9)	−0.0005 (8)	−0.0035 (8)	0.0007 (7)
C5	0.0609 (10)	0.0502 (9)	0.0336 (8)	0.0018 (7)	0.0096 (7)	−0.0002 (6)
C6	0.0441 (8)	0.0483 (9)	0.0357 (8)	0.0017 (6)	0.0080 (6)	−0.0003 (6)
C7	0.0433 (8)	0.0446 (8)	0.0323 (8)	0.0014 (6)	0.0107 (6)	0.0015 (6)
C8	0.0440 (9)	0.0489 (9)	0.0311 (7)	−0.0006 (6)	0.0112 (6)	−0.0023 (6)
C9	0.0423 (8)	0.0425 (8)	0.0315 (7)	−0.0020 (6)	0.0094 (6)	−0.0020 (6)
C10	0.0352 (8)	0.0453 (8)	0.0343 (8)	−0.0006 (6)	0.0072 (6)	0.0011 (6)
C11	0.0375 (8)	0.0395 (8)	0.0337 (7)	−0.0003 (6)	0.0103 (6)	0.0016 (5)
C12	0.0401 (8)	0.0514 (9)	0.0337 (8)	0.0025 (6)	0.0024 (6)	−0.0034 (6)
C13	0.0347 (8)	0.0733 (11)	0.0461 (9)	−0.0006 (7)	0.0100 (7)	−0.0076 (8)
C14	0.0410 (8)	0.0640 (10)	0.0385 (8)	−0.0023 (7)	0.0143 (7)	−0.0058 (7)
C21	0.0435 (11)	0.143 (2)	0.0542 (12)	0.0125 (11)	−0.0075 (9)	−0.0168 (11)
N1	0.0432 (7)	0.0644 (9)	0.0259 (6)	0.0056 (6)	0.0076 (5)	−0.0030 (5)
N2	0.0479 (8)	0.0582 (9)	0.0281 (7)	0.0047 (6)	0.0068 (5)	−0.0014 (5)
O1	0.0413 (7)	0.0958 (10)	0.0534 (8)	0.0035 (6)	0.0142 (6)	0.0052 (6)
O2	0.0480 (7)	0.0753 (8)	0.0357 (6)	0.0072 (5)	0.0147 (5)	−0.0020 (5)
O3	0.0422 (6)	0.0685 (7)	0.0285 (5)	−0.0020 (5)	0.0093 (4)	0.0001 (4)
O4	0.0415 (7)	0.0885 (9)	0.0347 (6)	0.0068 (5)	0.0001 (5)	−0.0039 (5)

Geometric parameters (Å, °)

C1—C2	1.402 (2)	C9—C10	1.404 (2)
C1—C6	1.404 (2)	C10—C11	1.3678 (19)
C1—C7	1.476 (2)	C10—H10	0.9300
C2—O1	1.357 (2)	C11—O3	1.3712 (16)
C2—C3	1.391 (2)	C11—C12	1.400 (2)
C3—C4	1.375 (2)	C12—O4	1.3671 (18)
C3—H3	0.9300	C12—C13	1.389 (2)
C4—C5	1.378 (3)	C13—C14	1.387 (2)
C4—H4	0.9300	C13—H13	0.9300
C5—C6	1.375 (2)	C14—H14	0.9300
C5—H5	0.9300	C21—O4	1.409 (2)
C6—H6	0.9300	C21—H21A	0.9600
C7—O2	1.2524 (16)	C21—H21B	0.9600
C7—N1	1.337 (2)	C21—H21C	0.9600
C8—N2	1.275 (2)	N1—N2	1.3840 (17)
C8—C9	1.457 (2)	N1—H1	0.8600
C8—H8	0.9300	O1—H2	1.00 (2)
C9—C14	1.386 (2)	O3—H11	0.8200

C2—C1—C6	117.74 (14)	C11—C10—H10	119.7
C2—C1—C7	119.30 (14)	C9—C10—H10	119.7
C6—C1—C7	122.95 (14)	C10—C11—O3	118.55 (12)
O1—C2—C3	118.17 (15)	C10—C11—C12	120.27 (13)
O1—C2—C1	121.90 (14)	O3—C11—C12	121.17 (13)
C3—C2—C1	119.93 (15)	O4—C12—C13	125.99 (14)
C4—C3—C2	120.29 (16)	O4—C12—C11	114.52 (13)
C4—C3—H3	119.9	C13—C12—C11	119.49 (14)
C2—C3—H3	119.9	C14—C13—C12	119.95 (15)
C3—C4—C5	121.15 (16)	C14—C13—H13	120.0
C3—C4—H4	119.4	C12—C13—H13	120.0
C5—C4—H4	119.4	C9—C14—C13	120.79 (14)
C6—C5—C4	118.73 (15)	C9—C14—H14	119.6
C6—C5—H5	120.6	C13—C14—H14	119.6
C4—C5—H5	120.6	O4—C21—H21A	109.5
C5—C6—C1	122.15 (15)	O4—C21—H21B	109.5
C5—C6—H6	118.9	H21A—C21—H21B	109.5
C1—C6—H6	118.9	O4—C21—H21C	109.5
O2—C7—N1	120.78 (13)	H21A—C21—H21C	109.5
O2—C7—C1	120.90 (14)	H21B—C21—H21C	109.5
N1—C7—C1	118.29 (13)	C7—N1—N2	118.93 (12)
N2—C8—C9	120.12 (13)	C7—N1—H1	120.5
N2—C8—H8	119.9	N2—N1—H1	120.5
C9—C8—H8	119.9	C8—N2—N1	116.24 (13)
C14—C9—C10	118.82 (14)	C2—O1—H2	105.6 (12)
C14—C9—C8	120.88 (14)	C11—O3—H11	109.5
C10—C9—C8	120.30 (14)	C12—O4—C21	117.93 (14)
C11—C10—C9	120.68 (14)		
C6—C1—C2—O1	179.36 (13)	C8—C9—C10—C11	−179.14 (13)
C7—C1—C2—O1	0.7 (2)	C9—C10—C11—O3	178.47 (11)
C6—C1—C2—C3	−0.1 (2)	C9—C10—C11—C12	−0.4 (2)
C7—C1—C2—C3	−178.83 (13)	C10—C11—C12—O4	179.66 (12)
O1—C2—C3—C4	−179.71 (15)	O3—C11—C12—O4	0.8 (2)
C1—C2—C3—C4	−0.2 (2)	C10—C11—C12—C13	0.0 (2)
C2—C3—C4—C5	0.3 (3)	O3—C11—C12—C13	−178.86 (13)
C3—C4—C5—C6	−0.1 (2)	O4—C12—C13—C14	−179.32 (14)
C4—C5—C6—C1	−0.2 (2)	C11—C12—C13—C14	0.3 (2)
C2—C1—C6—C5	0.4 (2)	C10—C9—C14—C13	−0.2 (2)
C7—C1—C6—C5	179.01 (13)	C8—C9—C14—C13	179.45 (13)
C2—C1—C7—O2	7.4 (2)	C12—C13—C14—C9	−0.2 (2)
C6—C1—C7—O2	−171.25 (13)	O2—C7—N1—N2	−1.6 (2)
C2—C1—C7—N1	−174.64 (12)	C1—C7—N1—N2	−179.59 (11)
C6—C1—C7—N1	6.7 (2)	C9—C8—N2—N1	178.54 (12)
N2—C8—C9—C14	178.18 (13)	C7—N1—N2—C8	172.94 (13)
N2—C8—C9—C10	−2.2 (2)	C13—C12—O4—C21	−6.4 (2)
C14—C9—C10—C11	0.5 (2)	C11—C12—O4—C21	173.87 (14)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1···O3^{i}	0.86	2.26	2.9616 (16)	138
O1—H2···O2	1.00 (2)	1.65 (2)	2.5424 (19)	146.3 (19)
O3—H11···O2^{ii}	0.82	2.17	2.8451 (16)	140
O3—H11···O4	0.82	2.23	2.6781 (17)	115

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

Fig. 1

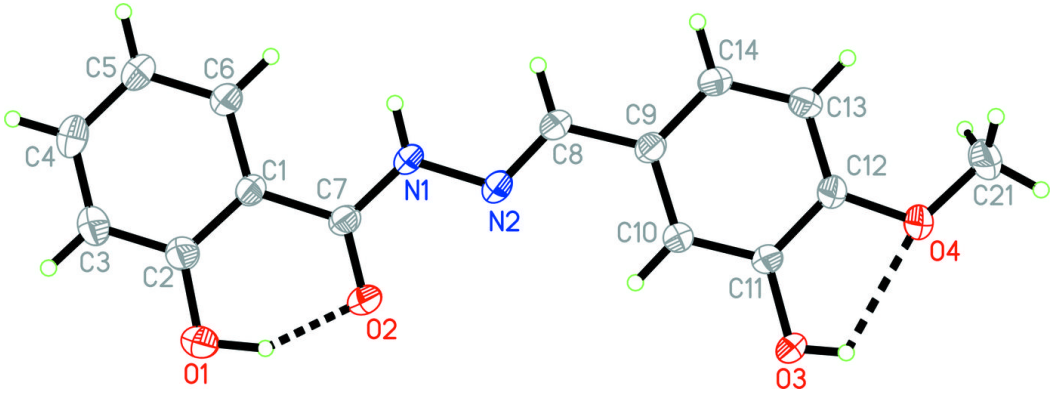

Fig. 2