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2-Hydroxy-5-(2-hydroxy-3-methoxybenzylideneamino)benzoic acid

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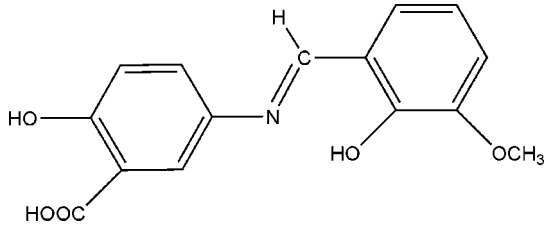
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.006 Å; R factor = 0.056; wR factor = 0.170; data-to-parameter ratio = 12.3.

The title compound, $C_{15}H_{13}NO_5$, was obtained by the condensation of *o*-vanillin with 5-aminosalicylic acid. The molecule is nonplanar with a dihedral angle of 13.2 (2)° between the two aromatic rings. The carboxyl and methoxy groups are almost coplanar with the attached rings. The molecular structure is stabilized by O—H···N and O—H···O hydrogen bonds. Intermolecular O—H···O hydrogen bonds link the molecules into a chain along the *c* axis, and adjacent chains are crosslinked by C—H···O hydrogen bonds and π–π stacking interactions involving the two benzene rings [centroid–centroid distance = 3.685 (3) Å].

Related literature

For general background, see: Yamada (1999); Yang *et al.* (2000). For a related structure, see: Bourque *et al.* (2005).



Experimental

Crystal data

$C_{15}H_{13}NO_5$
M_r = 287.26
Monoclinic, $P2_1/c$
a = 8.1049 (9) Å
b = 14.084 (2) Å
c = 12.1998 (17) Å
β = 108.264 (2)°
V = 1322.5 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 298 (2) K
0.49 × 0.45 × 0.12 mm

Data collection

Bruker SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.948, T_{max} = 0.987
6151 measured reflections
2332 independent reflections
1041 reflections with $I > 2\sigma(I)$
R_{int} = 0.069

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.170
S = 1.00
2332 reflections
190 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.32 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
O1—H1···N1	0.82	1.81	2.555 (4)	150
O5—H5···O3	0.82	1.81	2.533 (4)	147
O4—H4···O1^{i}	0.82	1.71	2.520 (4)	168
C12—H12···O5^{ii}	0.93	2.50	3.358 (5)	153

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

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); 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*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI2431).

References

Bourque, T. A., Nelles, M. E., Gullon, T. J., Garon, C. N., Ringer, M. K., Leger, L. J., Mason, J. W., Wheaton, S. L., Baerlocher, F. J., Vogels, C. M., Decken, A. & Westcott, S. A. (2005). *Can. J. Chem.* **83**, 1063–1070.
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 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.
Siemens (1996). *SMART* and *SAINT*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Yamada, S. (1999). *Coord. Chem. Rev.* **192**, 537–555.
Yang, Z. Y., Yang, R. D., Li, F. S. & Yu, K. B. (2000). *Polyhedron*, **19**, 2599–2604.

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supplementary materials

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2-Hydroxy-5-(2-hydroxy-3-methoxybenzylideneamino)benzoic acid

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Comment

Schiff bases have been intensively investigated recently owing to their strong coordination capability (Yamada, 1999) and diverse biological activities, such as antibacterial, antitumor activities *etc* (Yang *et al.*, 2000).

The title compound has a non-planar molecular structure (Fig. 1). The dihedral angle between the two aromatic rings is 13.2 (2)°. The O3—C9—C10—C11 [−5.3 (7)°], O4—C9—C10—C15 [−3.6 (6)°] and C8—O2—C4—C5 [12.8 (6)°] torsion angles indicate that the carboxyl and methoxy groups are almost coplanar with the attached rings. Intramolecular O—H···N1 and O—H···O hydrogen bonds are observed in the molecular structure, similar to those reported in a related structure (Bourque *et al.*, 2005).

In the crystal structure, intermolecular O4—H4···O1(x,3/2 − y,1/2 + z) hydrogen bonds link the molecules into a chain along the c axis (Fig. 2). The adjacent chains are cross-linked by C—H···O hydrogen bonds (Table 1) and π-π stacking interactions involving the two benzene rings, with a centroid···centroid distance of 3.685 (3) Å.

Experimental

To an ethanol (10 ml) solution of 5-aminosalicylic acid (1.5305 g, 10 mmol) was added an ethanol (5 ml) solution of *o*-vanillin (1.5212 g, 10 mmol). The mixture was heated under reflux for 2 h to ensure completion, at which point a yellow precipitate was collected by suction filtration and washed with ethanol and Et_2O. Crystals of the title compound suitable for X-ray analysis were grown from an ethanol solution after about two weeks.

Refinement

All H atoms were placed in geometrically idealized positions (O—H = 0.82 Å and C—H = 0.93–0.96 Å) and treated as riding on their parent atoms, with U_{iso}(H) = 1.5U_{eq}(C_{methyl}, O) or 1.2U_{eq}(C).

Figures

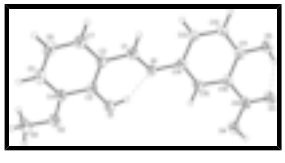

Fig. 1. The molecular structure of the title compound, showing 30% probability displacement ellipsoids and the atom-numbering scheme. Hydrogen bonds are shown as dashed lines.

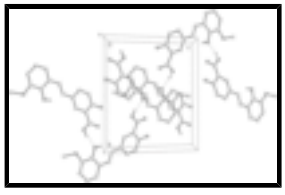

Fig. 2. View of O—H···O hydrogen-bonded (dashed lines) chains in the title compound.

2-Hydroxy-5-(2-hydroxy-3-methoxybenzylideneamino)benzoic acid

Crystal data

$C_{15}H_{13}NO_5$	$F_{000} = 600$
$M_r = 287.26$	$D_x = 1.443$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: -P 2ybc	Cell parameters from 741 reflections
$a = 8.1049$ (9) Å	$\theta = 2.6$–20.0º
$b = 14.084$ (2) Å	$\mu = 0.11$ mm^{-1}
$c = 12.1998$ (17) Å	$T = 298$ (2) K
$\beta = 108.264$ (2)º	Block, red
$V = 1322.5$ (3) Å^3	0.49 × 0.45 × 0.12 mm
$Z = 4$	

Data collection

Bruker SMART CCD area-detector diffractometer	2332 independent reflections
Radiation source: fine-focus sealed tube	1041 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.069$
$T = 298$(2) K	$\theta_{max} = 25.0$º
φ and ω scans	$\theta_{min} = 2.3$º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -9 \rightarrow 9$
$T_{min} = 0.948$, $T_{max} = 0.987$	$k = -13 \rightarrow 16$
6151 measured reflections	$l = -11 \rightarrow 14$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.056$	H-atom parameters constrained
$wR(F^2) = 0.170$	$w = 1/[\sigma^2(F_o^2) + (0.0562P)^2 + 0.7223P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.00$	$(\Delta/\sigma)_{max} = 0.001$
2332 reflections	$\Delta\rho_{max} = 0.32$ e Å^{-3}
190 parameters	$\Delta\rho_{min} = -0.26$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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}
N1	0.7167 (4)	0.4976 (2)	0.5754 (2)	0.0374 (9)
O1	0.8177 (4)	0.54458 (19)	0.4044 (2)	0.0471 (8)
H1	0.7885	0.5505	0.4626	0.071*
O2	0.8971 (4)	0.5021 (2)	0.2205 (2)	0.0581 (9)
O3	0.6394 (4)	0.78059 (19)	0.9247 (3)	0.0633 (10)
O4	0.7650 (4)	0.80386 (19)	0.7888 (2)	0.0608 (9)
H4	0.7795	0.8569	0.8178	0.091*
O5	0.5298 (4)	0.6170 (2)	0.9536 (2)	0.0583 (9)
H5	0.5517	0.6735	0.9667	0.087*
C1	0.7309 (5)	0.4100 (3)	0.5451 (3)	0.0436 (11)
H1A	0.7054	0.3618	0.5893	0.052*
C2	0.7826 (5)	0.3842 (3)	0.4492 (3)	0.0393 (10)
C3	0.8223 (5)	0.4552 (3)	0.3802 (3)	0.0365 (10)
C4	0.8684 (5)	0.4269 (3)	0.2827 (3)	0.0424 (11)
C5	0.8794 (6)	0.3335 (3)	0.2583 (4)	0.0525 (12)
H5A	0.9107	0.3159	0.1941	0.063*
C6	0.8440 (6)	0.2633 (3)	0.3295 (4)	0.0594 (14)
H6	0.8547	0.1994	0.3133	0.071*
C7	0.7944 (6)	0.2882 (3)	0.4215 (4)	0.0545 (13)
H7	0.7677	0.2412	0.4669	0.065*
C8	0.9075 (6)	0.4835 (3)	0.1078 (3)	0.0697 (15)
H8A	0.9274	0.5420	0.0734	0.105*
H8B	0.8005	0.4558	0.0609	0.105*
H8C	1.0015	0.4405	0.1133	0.105*
C9	0.6839 (6)	0.7504 (3)	0.8431 (4)	0.0448 (11)
C10	0.6492 (5)	0.6533 (3)	0.8013 (3)	0.0352 (10)
C11	0.5761 (5)	0.5903 (3)	0.8611 (3)	0.0403 (11)
C12	0.5489 (5)	0.4966 (3)	0.8259 (3)	0.0467 (11)
H12	0.4997	0.4547	0.8658	0.056*
C13	0.5934 (5)	0.4648 (3)	0.7332 (3)	0.0412 (11)
H13	0.5760	0.4015	0.7111	0.049*
C14	0.6654 (5)	0.5275 (3)	0.6713 (3)	0.0342 (10)
C15	0.6903 (5)	0.6201 (3)	0.7050 (3)	0.0376 (10)

H15	0.7355	0.6622	0.6630	0.045*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.039 (2)	0.039 (2)	0.0338 (18)	0.0012 (17)	0.0120 (16)	−0.0096 (16)
O1	0.064 (2)	0.0417 (19)	0.0417 (17)	0.0020 (15)	0.0258 (15)	−0.0024 (14)
O2	0.075 (2)	0.063 (2)	0.0466 (18)	0.0013 (17)	0.0340 (17)	−0.0001 (16)
O3	0.095 (3)	0.047 (2)	0.065 (2)	0.0026 (17)	0.050 (2)	−0.0042 (16)
O4	0.093 (3)	0.0392 (18)	0.070 (2)	−0.0052 (17)	0.0539 (19)	−0.0074 (16)
O5	0.078 (2)	0.058 (2)	0.0528 (19)	−0.0047 (17)	0.0402 (18)	−0.0057 (15)
C1	0.052 (3)	0.042 (3)	0.039 (2)	−0.002 (2)	0.017 (2)	0.002 (2)
C2	0.047 (3)	0.038 (3)	0.035 (2)	0.003 (2)	0.015 (2)	−0.003 (2)
C3	0.031 (3)	0.038 (3)	0.038 (2)	0.006 (2)	0.0071 (19)	−0.005 (2)
C4	0.040 (3)	0.045 (3)	0.042 (2)	0.003 (2)	0.013 (2)	−0.005 (2)
C5	0.054 (3)	0.061 (3)	0.043 (3)	0.009 (3)	0.016 (2)	−0.012 (2)
C6	0.074 (4)	0.044 (3)	0.058 (3)	0.009 (3)	0.018 (3)	−0.012 (3)
C7	0.070 (4)	0.044 (3)	0.050 (3)	−0.002 (2)	0.020 (3)	−0.003 (2)
C8	0.076 (4)	0.099 (4)	0.041 (3)	0.014 (3)	0.029 (3)	0.009 (3)
C9	0.057 (3)	0.041 (3)	0.044 (3)	0.008 (2)	0.027 (2)	0.003 (2)
C10	0.038 (3)	0.038 (2)	0.032 (2)	0.003 (2)	0.0139 (19)	0.0016 (19)
C11	0.044 (3)	0.047 (3)	0.034 (2)	0.006 (2)	0.018 (2)	0.004 (2)
C12	0.051 (3)	0.049 (3)	0.045 (3)	−0.008 (2)	0.023 (2)	0.005 (2)
C13	0.046 (3)	0.041 (3)	0.039 (2)	−0.008 (2)	0.017 (2)	−0.002 (2)
C14	0.033 (3)	0.038 (3)	0.033 (2)	0.0008 (19)	0.0118 (19)	0.0009 (19)
C15	0.035 (3)	0.041 (3)	0.038 (2)	0.003 (2)	0.0116 (19)	0.009 (2)

Geometric parameters (Å, °)

N1—C1	1.304 (4)	C5—H5A	0.93
N1—C14	1.423 (4)	C6—C7	1.351 (5)
O1—C3	1.297 (4)	C6—H6	0.93
O1—H1	0.82	C7—H7	0.93
O2—C4	1.364 (5)	C8—H8A	0.96
O2—C8	1.428 (4)	C8—H8B	0.96
O3—C9	1.236 (4)	C8—H8C	0.96
O4—C9	1.308 (5)	C9—C10	1.455 (5)
O4—H4	0.82	C10—C11	1.394 (5)
O5—C11	1.349 (4)	C10—C15	1.398 (5)
O5—H5	0.82	C11—C12	1.384 (5)
C1—C2	1.408 (5)	C12—C13	1.365 (5)
C1—H1A	0.93	C12—H12	0.93
C2—C7	1.405 (5)	C13—C14	1.402 (5)
C2—C3	1.408 (5)	C13—H13	0.93
C3—C4	1.411 (5)	C14—C15	1.363 (5)
C4—C5	1.358 (5)	C15—H15	0.93
C5—C6	1.404 (6)		
C1—N1—C14	126.0 (3)	O2—C8—H8B	109.5

C3—O1—H1	109.5	H8A—C8—H8B	109.5
C4—O2—C8	117.8 (3)	O2—C8—H8C	109.5
C9—O4—H4	109.5	H8A—C8—H8C	109.5
C11—O5—H5	109.5	H8B—C8—H8C	109.5
N1—C1—C2	123.8 (4)	O3—C9—O4	122.0 (4)
N1—C1—H1A	118.1	O3—C9—C10	122.2 (4)
C2—C1—H1A	118.1	O4—C9—C10	115.9 (4)
C7—C2—C3	119.6 (4)	C11—C10—C15	118.6 (4)
C7—C2—C1	120.6 (4)	C11—C10—C9	118.8 (4)
C3—C2—C1	119.8 (4)	C15—C10—C9	122.5 (4)
O1—C3—C2	121.7 (3)	O5—C11—C12	117.8 (4)
O1—C3—C4	120.0 (4)	O5—C11—C10	122.4 (4)
C2—C3—C4	118.3 (4)	C12—C11—C10	119.8 (4)
C5—C4—O2	126.6 (4)	C13—C12—C11	120.8 (4)
C5—C4—C3	120.7 (4)	C13—C12—H12	119.6
O2—C4—C3	112.7 (4)	C11—C12—H12	119.6
C4—C5—C6	120.5 (4)	C12—C13—C14	120.1 (4)
C4—C5—H5A	119.8	C12—C13—H13	119.9
C6—C5—H5A	119.8	C14—C13—H13	119.9
C7—C6—C5	120.1 (4)	C15—C14—C13	119.2 (4)
C7—C6—H6	119.9	C15—C14—N1	118.5 (3)
C5—C6—H6	119.9	C13—C14—N1	122.3 (4)
C6—C7—C2	120.7 (4)	C14—C15—C10	121.4 (4)
C6—C7—H7	119.7	C14—C15—H15	119.3
C2—C7—H7	119.7	C10—C15—H15	119.3
O2—C8—H8A	109.5		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···N1	0.82	1.81	2.555 (4)	150
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C12—H12···O5^{ii}	0.93	2.50	3.358 (5)	153

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

Fig. 1

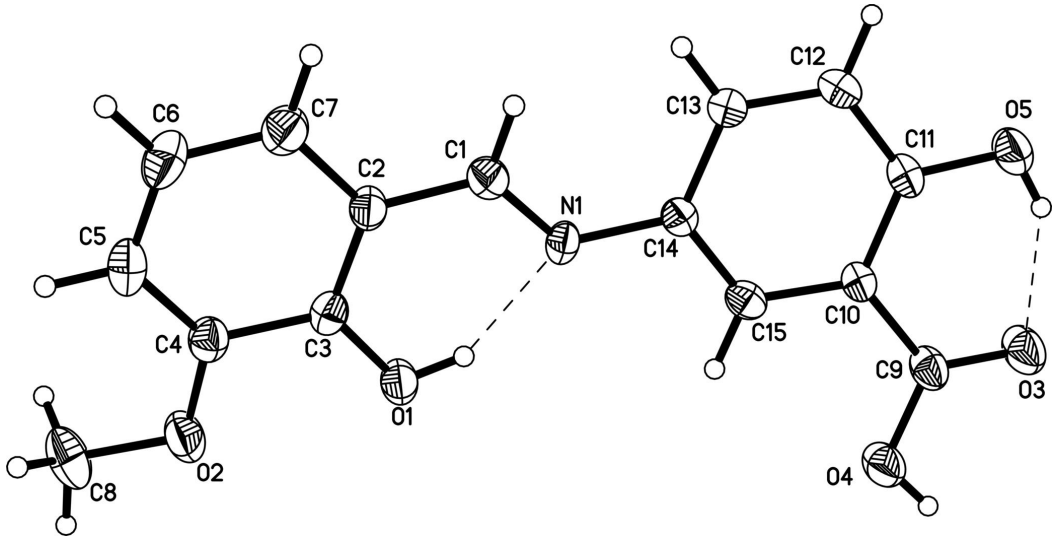

Fig. 2