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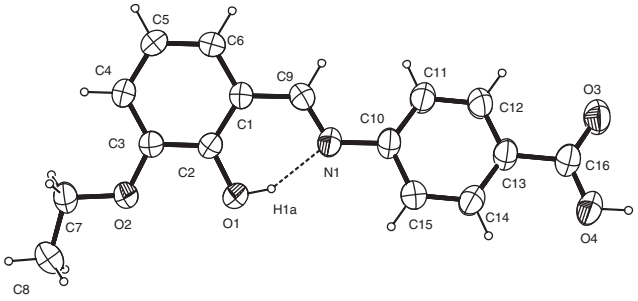
The crystal structure of (*E*)-4-(3-ethoxy-2-hydroxy-benzylideneamino)benzoic acid, $C_{16}H_{15}NO_4$



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Abstract

$C_{16}H_{15}NO_4$, triclinic, $P\bar{1}$ (no. 2), $a = 4.9960(4)$ Å, $b = 6.9139(5)$ Å, $c = 20.7650(15)$ Å, $\alpha = 83.718(6)°$, $\beta = 84.805(6)°$, $\gamma = 78.648(6)°$, $V = 697.25(9)$ Å^3, $Z = 2$, $R_{gt}(F) = 0.0480$, $wR_{ref}(F^2) = 0.0988$, $T = 296$ K.

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The asymmetric unit of the title crystal structure is shown in the figure. Tables 1 and 2 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Brown needle
Size:	0.76 × 0.30 × 0.03 mm
Wavelength:	Mo *Kα* radiation (0.71073 Å)
μ:	1.0 cm^{-1}
Diffractometer, scan mode:	STOE IPDS 2, ω scans
$2\theta_{max}$, completeness:	52°, 98.8%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	7346, 2710, 0.141
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1509
$N(param)_{refined}$:	198
Programs:	Stoe programs [1], SHELX [2], ORTEP-3 [3], WinGX [4]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
C1	−0.0486(4)	0.5847(3)	0.28544(10)	0.0414(5)
C2	−0.1998(4)	0.4428(3)	0.31452(10)	0.0416(5)
C3	−0.4125(4)	0.4952(3)	0.36158(10)	0.0437(5)
C4	−0.4763(5)	0.6873(3)	0.37826(11)	0.0488(6)
H4	−0.6198	0.7229	0.4090	0.059*
C5	−0.3276(5)	0.8288(3)	0.34940(11)	0.0528(6)
H5	−0.3722	0.9581	0.3609	0.063*
C6	−0.1167(5)	0.7773(3)	0.30435(10)	0.0500(6)
H6	−0.0164	0.8719	0.2859	0.060*
C7	−0.7522(5)	0.3840(3)	0.43814(11)	0.0516(6)
H7A	−0.8995	0.4881	0.4231	0.062*
H7B	−0.6788	0.4261	0.4748	0.062*
C8	−0.8560(5)	0.1955(4)	0.45762(12)	0.0664(7)
H8A	−0.7084	0.0936	0.4723	0.080*
H8B	−0.9290	0.1558	0.4210	0.080*
H8C	−0.9972	0.2162	0.4921	0.080*
C9	0.1799(5)	0.5306(3)	0.23893(10)	0.0466(6)
H9	0.2836	0.6252	0.2225	0.056*
C10	0.4725(4)	0.3035(3)	0.17547(10)	0.0458(6)
C11	0.5881(5)	0.4351(4)	0.13141(11)	0.0609(7)
H11	0.5124	0.5695	0.1289	0.073*
C12	0.8139(5)	0.3671(3)	0.09157(11)	0.0599(7)
H12	0.8913	0.4564	0.0625	0.072*
C13	0.9279(5)	0.1666(3)	0.09416(10)	0.0488(6)
C14	0.8084(5)	0.0359(3)	0.13666(11)	0.0537(6)
H14	0.8811	−0.0990	0.1382	0.064*
C15	0.5821(5)	0.1029(3)	0.17687(11)	0.0515(6)
H15	0.5024	0.0129	0.2051	0.062*
C16	1.1746(5)	0.0938(4)	0.05226(10)	0.0500(6)
N1	0.2443(4)	0.3570(3)	0.21973(8)	0.0471(5)
O1	−0.1432(4)	0.2540(2)	0.29939(8)	0.0572(5)
O2	−0.5430(3)	0.3452(2)	0.38719(7)	0.0551(5)
O3	1.2797(4)	0.2190(3)	0.01362(8)	0.0686(5)
O4	1.2701(4)	−0.0897(3)	0.05632(9)	0.0663(5)
H1A	−0.013(5)	0.249(4)	0.2681(11)	0.089(10)*
H4A	1.423(6)	−0.129(6)	0.0302(18)	0.19(2)*

Source of materials

The title compound was prepared by refluxing a mixture of a solution containing 3-ethoxy-2-hydroxybenzaldehyde (0.5 g; 3.01 mmol) in 20 mL ethanol and a solution containing 4-aminobenzoic acid (0.41 g; 3.01 mmol) in 20 mL ethanol. The reaction mixture was stirred for 1 h under reflux. The crystals

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for the crystal structure determination were obtained from acetone by slow evaporation (yield 72%, m.p. 488–490 K).

Experimental details

All H atoms except for H1A and H4A were refined using riding models, with C—H distances of 0.96 Å for CH_3 group, 0.97 Å for CH_2 group and 0.93 Å for aromatic groups. The displacement parameters of these H atoms were fixed at $1.2U_{eq}$ of their parent carbon atom for aromatic groups and methylene, $1.5U_{eq}$ of their parent atoms for methyl group.

Comment

The reaction of an aldehyde and a primary amine forms the Schiff base compounds having a −C=N− double bond. These compounds have a wide range of application in the fields of coordination chemistry, biochemistry, pharmacy, nanotechnology, optical devices and textile industries [5–7]. Particularly the *o*-hydroxy Schiff base class is more attractive for physicists and chemists because of its interesting photo-, solvato- and thermochromic features which are caused by an intramolecular proton transfer from the hydroxyl O atom to the imine N atom with a change in the π-electronic system in the solid state. *o*-Hydroxy Schiff base compounds adopt OH (enol-imine/benzenoid) [8] or NH (keto-amine/quinoid) [9] tautomeric forms with reference to the location of the transferred proton.

The title compound adopts enol-imine tautomeric form and has *E* configuration around the C=N double bond. The bond lengths and angles are in the expected ranges and comparable with those of similar compounds in literature [10, 11]. The compound displays a strong intramolecular hydrogen bond including the atoms O1 and N1 (O···N = 2.588(2) Å) as a common feature of *o*-hydroxysalicylidene systems. As can be seen from the figure, this strong hydrogen bond constitutes a S(6) ring. In three dimensional network, the crystal structure is stabilized by an intermolecular O—H···O hydrogen bond and weak van der Waals interactions. The donor-acceptor distance of this intermolecular O4—H4A···O3′ hydrogen bond is 2.621(2) Å [symmetry code (′) = 3−x, −y, −z].

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