Crystal Structure of 4-Ethoxy-*N*-(*o*-hydroxyacetophenolidene)aniline

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The title compound, C₁₆H₁₇NO₂, was found to crystallize in the triclinic space group $P\bar{1}$. The unit cell parameters are: $a = 8.279(1)\text{Å}$, $b = 14.843(2)\text{Å}$, $c = 5.959(1)\text{Å}$, $\alpha = 97.02(1)^\circ$, $\beta = 102.75(1)^\circ$, $\gamma = 100.52(1)^\circ$; $Z = 2$. The molecule is not planar. The ethoxy group is almost coplanar with the ethoxyphenyl ring, the hydroxy group and the central moiety of the molecule are almost in the hydroxyphenyl ring plane. The dihedral angle between the two planes is $65.4(1)^\circ$. The crystal structure is stabilized by weak intermolecular interactions of the C-H...O type.

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In this paper we present the crystal and molecular structures of a Schiff's base, C₁₆H₁₇NO₂. The important place held in organic chemistry by Schiff's base ligands has been confirmed by surveys which are continuously being made. Also, since organometallic compounds exhibit conductivity and magnetic properties due to one or more metal atoms introduced into the Schiff's bases, their synthesis is becoming a object of growing interest. It is known that such substances are used in cancer research¹ and in the industry due to liquid-crystal characteristics.²

The 4-ethoxy-*N*-(*o*-hydroxyacetophenolidene)aniline was prepared by adding *p*-ethoxyaniline to the 2-hydroxyacetophenone (1:1 molar ratio) in hot dry ethanol. The reaction mixture was cooled and filtered. Upon concentration and cooling, the ligand separated out. Subsequently, the ligand was recrystallized from ethanol.

The crystal and molecular structures of C₁₆H₁₇NO₂ were determined by single-crystal X-ray diffraction. The crystal data, details of the data collection and the structure determination are given in Table 1. The structure was solved by direct methods and refined by full-matrix least squares on F^2 . The non-hydrogen atoms were refined anisotropically. The H atoms were located from difference maps and refined isotropically. The C-H bond distances are 0.90(2) to 1.04(4)Å, while U_{eq} values for the H atoms are in the range of 0.073(7) – 0.15(1)Å². The refined atomic parameters with equivalent isotropic

temperature factors for non-hydrogen atoms are given in Table 2. The bond lengths and angles are given in Table 3.

The molecular structure of the title compound is shown in Fig. 2. The N1=C9 double bond, with a length of 1.287(4)Å, is slightly longer than another similar Schiff's base³ [C₁₉H₁₈ON₂, 1.268(2)Å]. The ethoxy group is almost coplanar with the ethoxyphenyl ring, the maximum deviation from the plane being 0.034(4)Å for the C1 atom. The hydroxy group and the

Table 1 Crystal and experimental data

Formula: C ₁₆ H ₁₇ NO ₂
Formula weight: 255.31
Crystal system: triclinic
Space group: $P\bar{1}$ $Z = 2$
$T = 293(2)\text{K}$
$a = 8.279(1)\text{Å}$
$b = 14.843(2)\text{Å}$
$c = 5.959(1)\text{Å}$
$\alpha = 97.02(1)^\circ$
$\beta = 102.75(1)^\circ$
$\gamma = 100.52(1)^\circ$
$V = 692.1(2)\text{Å}^3$
$D_{\text{calc}} = 1.225\text{ g cm}^{-3}$
$\mu(\text{Mo K}\alpha) = 0.8\text{ cm}^{-1}$
Radiation: Mo K α ($= 0.71073\text{ Å}$)
Reflection collected/unique: 2829/2639
Independent reflection: $[I > 2\sigma(I)]1186$
Number of parameters: 241
$R = 0.041$
$wR = 0.12$
$(\Delta/\sigma)_{\text{max}} = 0.000$
$(\Delta\rho)_{\text{max}} = 0.12\text{ eÅ}^{-3}$
$(\Delta\rho)_{\text{min}} = -0.16\text{ eÅ}^{-3}$
Measurement: Rigaku AFC-7S
Program system: MSC/AFC Diffractometer Control Software
Structure determination: direct method with SHELXS-97
Refinement: full matrix least-squares with SHELXL-97 in WINGX ⁴

Fig. 1 Chemical diagram of the title compound

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Table 2 Final atomic coordinates and equivalent isotropic temperature factors ($\text{\AA}^2$) for the non-H atoms

Atom	x	y	z	U_{eq}
O1	0.0589(3)	0.4017(1)	0.2073(3)	0.0981(9)
O2	0.6035(2)	0.0880(1)	0.8431(3)	0.0761(6)
N1	0.1323(3)	0.2936(1)	0.4971(3)	0.0719(7)
C1	0.8190(5)	0.0433(2)	1.1086(7)	0.0933(12)
C2	0.7002(4)	0.1073(2)	1.0802(5)	0.0779(10)
C3	0.4904(3)	0.1425(2)	0.7726(4)	0.0666(8)
C4	0.4007(3)	0.1201(2)	0.5403(4)	0.0755(10)
C5	0.2850(4)	0.1705(2)	0.4529(5)	0.0752(9)
C6	0.2520(3)	0.2425(2)	0.5946(4)	0.0672(8)
C7	0.3440(3)	0.2651(2)	0.8250(4)	0.0749(10)
C8	0.4636(3)	0.2162(2)	0.9143(5)	0.0732(9)
C9	-0.0087(3)	0.2910(1)	0.5562(3)	0.0634(8)
C10	-0.1219(3)	0.3489(2)	0.4514(3)	0.0654(8)
C11	-0.0837(4)	0.4011(2)	0.2814(4)	0.0771(10)
C12	-0.1957(6)	0.4550(2)	0.1845(5)	0.0980(13)
C13	-0.3400(6)	0.4569(2)	0.2568(6)	0.0989(13)
C14	-0.3814(5)	0.4069(2)	0.4225(5)	0.0892(10)
C15	-0.2730(4)	0.3538(2)	0.5170(5)	0.0760(10)
C16	-0.0651(5)	0.2297(2)	0.7202(6)	0.0773(11)

$$U_{eq} = (1/3) \sum_i \sum_j U_{ij} a_i^* a_j^* (\mathbf{a}_i \cdot \mathbf{a}_j)$$

Table 3 Selected bond lengths ($\text{\AA}$) and bond angles ($^\circ$) for $\text{C}_{16}\text{H}_{17}\text{NO}_2$

O1 - C11	1.348(4)	C2 - O2 - C3	118.6(2)
O2 - C2	1.427(3)	C11 - O1 - H1	109.6(17)
O2 - C3	1.376(3)	C6 - N1 - C9	122.0(2)
O1 - H1	0.93(3)	O2 - C2 - C1	108.2(3)
N1 - C6	1.423(3)	O2 - C3 - C4	115.6(2)
N1 - C9	1.287(4)	O2 - C3 - C8	124.8(2)
C1 - C2	1.485(5)	N1 - C6 - C7	122.3(2)
C9 - C10	1.474(3)	N1 - C9 - C16	123.2(2)
C9 - C16	1.505(4)	C10 - C9 - C16	118.9(2)
		N1 - C9 - C10	118.0(2)
		O1 - C11 - C12	118.3(3)

Fig. 2 Molecular structure of the title compound along with the labeling scheme for non-H atoms. Displacement ellipsoids are shown at the 50% probability levels; H atoms are drawn as small circles of arbitrary radii. The intramolecular hydrogen bond is shown as a dashed line.

central moiety of the molecule are almost in the hydroxyphenyl ring plane, the maximum deviation from the plane being $-0.139(3)\text{\AA}$ for the C16 atom. The dihedral angle between the two planes is $65.4(1)^\circ$. The crystal structure is stabilized by one intramolecular hydrogen bond between O1 and N1 [$\text{O1}\cdots\text{N1}$ $2.549(3)$, O1-H1 $0.93(3)\text{\AA}$, and $\text{O1-H1}\cdots\text{N1}$ $145(2)^\circ$] and one weak intermolecular interaction between C16 and O1^i [$\text{C16}\cdots\text{O1}^i$ $3.452(4)$, C16-H $0.97(3)\text{\AA}$ and $\text{C-H}\cdots\text{O1}^i$ $156(3)^\circ$; symmetry code: (i) $x, y, 1 + z$].

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