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Synthesis and Crystal Structure of 1-(4-{[(*E*)-4-methoxy-2-hydroxybenzylidene]amino}phenyl)ethanoe Oxime

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The compound 1-(4-{[(*E*)-4-methoxy-2-hydroxybenzylidene]amino}phenyl)ethanoe oxime with the molecular formula $C_{16}H_{16}N_2O_3$, has been synthesized and characterized structurally. The two benzene rings in the molecule form a dihedral angle of 3.05(2)º. There is one strong intramolecular O-H···N hydrogen bond between the hydroxyl (O2-H2) groups and the Schiff base nitrogen (N2) atom, with the distance of *d*(N2-O2) = 2.593(4) Å. In the crystal structure, each molecule links other three molecules into an infinite 2D supramolecular structure by three pairs of intermolecular O1-H1···N1, C1-H1A···O1 and C16-H16B···O3 hydrogen bonds.

Key Words: Mono-oxime compounds, Synthesis, Crystal structure.

INTRODUCTION

Oxime-type compounds are a significant class of organic compounds because of their applications in many fields of organic[1], biological, analytical and coordination chemistry[2-8]. The complexes with oxime-type compounds are also important in biomaterial applications[9], often exhibit satisfactory insecticidal, fungicidal or herbicidal activity with low-grade toxicity and residue. Most of them are stable in solution and solid state and are stable to hydrolysis compared with the corresponding imines[10]. Thus, new materials can be produced by using these compounds, which seem to be suitable candidates for further chemical modifications[11]. Herein, we report the synthesis and crystal structure of 1-(4-{[(E)-4-methoxy-2-hydroxybenzylidene]amino}phenyl)ethanoe oxime.

EXPERIMENTAL

4-Methoxy salicylaldehyde and 4-amino phenylethanone were purchased from Alfa Aesar and used without further purification. 4-Aminophenylethanone oxime was synthesized according to an analogous method reported earlier[12]. The other reagents and solvents were analytical grade reagents from Tianjin Chemical Reagent Factory. C, H and N analyses were carried out with a GmbH VariuoEL V3.00 automatic elemental analyzer. IR spectra in the range 4000-400 cm^{-1} were recorded on a VERTEX70 FT-IR spectrophotometer using KBr pellets. ^1H NMR spectra were recorded on a Mercury-400BB spectrometer at room temperature using $CDCl_3$ as solvent. X-ray single crystal structure was determined on a Bruker Smart 1000 CCD area detector. Melting points were measured by the use of a microscopic melting point apparatus made in Beijing Taike Instrument Limited Company and the thermometer was uncorrected.

1-(4-{[(E)-4-methoxy-2-hydroxybenzylidene]amino}-phenyl)ethanoe oxime was synthesized according to reported method[3]. A solution of 4-aminophenylethanone oxime (75.1 mg, 0.5 mmol) in ethanol (3 mL) was added to a solution of 4-methoxy salicylaldehyde (76.2 mg, 0.5 mmol) in ethanol (3 mL) and the mixture was heated at 328 K for 12 h. After cooling to room temperature, yellow precipitate was collected on a suction filter, washed with ethanol and ethanol/hexane (1:4) respectively. The isolated compound was dried under reduced pressure and purified with recrystallization from ethanol to yield 112.9 mg of crystalline solid. Yield 79.4 %. m.p. 474-475 K. Anal. calcd. for: $C_{16}H_{16}N_2O_3$: C, 67.59; H, 5.67; N, 9.85. Found: C, 67.72; H, 5.48; N, 6.71.

Yellow needle-like single crystals suitable for X-ray diffraction studies were obtained after about one month by slow evaporation from a ethanol/chloroform(1:1) solution (5 mL) of the title compound.

X-Ray structure determination: The single crystal of the title compound, with approximate dimensions of 0.38 mm × 0.11 mm × 0.07 mm was placed on a Bruker Smart 1000 diffractmeter equipped with Apex CCD area detector. The diffraction data were collected using a graphite monochromated MoK_α radition (λ = 0.71073 Å) at 298(2) K. The structure was solved by using the program SHELXS-97 and Fourier difference techniques and refined by full-matrix least-squares

method on F^2 using SHELXL-97. Details of the data collection and refinements of title compound are given in Table-1. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were added theoretically. CCDC: 757720.

TABLE-1
CRYSTAL DATA AND STRUCTURE REFINEMENT FOR THE TITLE COMPOUND

Empirical formula	$C_{16}H_{16}N_2O_3$
Formula weight	284.31
Temperature	298(2) K
Wavelength	0.71073 Å
Crystal system	Triclinic
Space group	*P*-1
Cell dimensions	a = 4.8140(5) Å, b = 7.0401(8) Å, c = 20.700(2) Å; α = 90.090(1)º, β = 96.310(2)º, γ = 94.110(2)º
Volume	695.48(13) $Å^3$
Z	2
Density (calculated)	1.358 mg/m^3
Absorption coefficient	0.095 mm^{-1}
F(000)	300
Index ranges	$-5 \le h \le 5$, $-6 \le k \le 8$, $-24 \le l \le 22$
Reflections collected/unique	3568/2421 [*R*(int) = 0.0350]
Independent reflections	515
Data/restraints/parameters	2421/0/192
Goodness of fit indicator	1.033
R [I > 2σ(I)]	$R_1 = 0.0625$, $wR_2 = 0.1078$
Largest diff. peak and hole	0.183 and -0.207 e·$Å^{-3}$

RESULTS AND DISCUSSION

X-ray crystallographic analysis revealed the crystal structure of the title compound. The structure is shown in Fig. 1 and packing arrangement of the unit cell is shown in Fig. 2. Selected bond distances and angles are listed in Table-2. Hydrogen bonds for the title compound is listed in Table-3. The crystal structure is only built up by the $C_{16}H_{16}N_2O_3$ molecules, in which all bond lengths are in normal ranges. The two benzene rings in the molecule form a dihedral angle of 3.05(2)º. There is one strong intramolecular O-H···N hydrogen bond between the hydroxyl (O2-H2) groups and the Schiff base nitrogen (N2) atom, with the distance of *d*(N2-O2) = 2.593(4) Å. In the crystal structure, each molecule links other three molecules into an infinite 2D supramolecular structure[13,14] by three pairs of intermolecular O1-H1···N1, C1-H1A···O1 and C16-H16B···O3 hydrogen bonds.

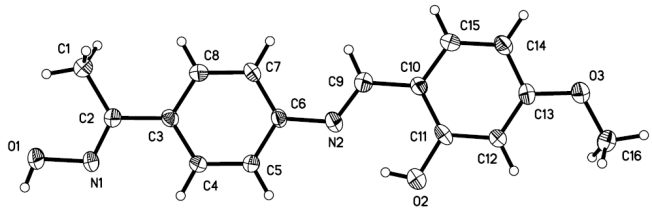

Fig. 1. Molecule structure of the title compound with atom numbering scheme. Displacement ellipsoids for non-hydrogen atoms are drawn at the 30 % probability level

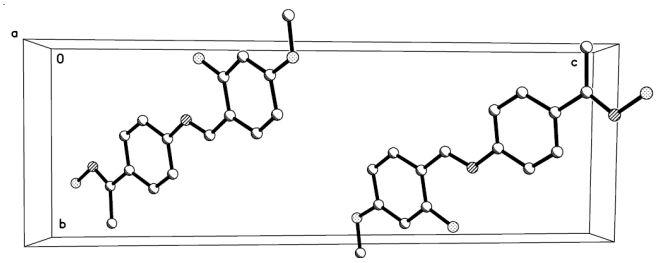

Fig. 2. Packing arrangement of the unit cell of the title compound. H atoms are omitted for clarity

TABLE-3
HYDROGEN BONDS [Å, º] FOR THE TITLE COMPOUND

D-H···A	*d*(D-H)	*d*(H···A)	∠DHA	*d*(D···A)
O2-H2···N2	0.82	1.86	148	2.593(4)
O1-H1···N1	0.82	2.10	149	2.828(4)
C1-H1A···O1	0.96	2.56	145	3.396(4)
C16-H16B···O3	0.96	2.57	153	3.458(4)

TABLE-2
SELECTED BOND DISTANCES (Å) AND ANGLES (º) FOR THE TITLE COMPOUND

Bond	Lengths	Bond	Lengths	Bond	Lengths
N(1)-C(2)	1.275(4)	C(2)-C(3)	1.478(4)	C(10)-C(15)	1.391(4)
N(1)-O(1)	1.410(3)	C(3)-C(8)	1.388(4)	C(10)-C(11)	1.403(4)
N(2)-C(9)	1.279(4)	C(3)-C(4)	1.390(4)	C(11)-C(12)	1.397(5)
N(2)-C(6)	1.427(4)	C(4)-C(5)	1.368(5)	C(12)-C(13)	1.366(4)
O(2)-C(11)	1.343(4)	C(5)-C(6)	1.373(4)	C(13)-C(14)	1.397(4)
O(3)-C(13)	1.375(4)	C(6)-C(7)	1.377(4)	C(14)-C(15)	1.370(5)
O(3)-C(16)	1.418(4)	C(7)-C(8)	1.384(5)		
C(1)-C(2)	1.491(4)	C(9)-C(10)	1.434(5)		
Bond	Angles	Bond	Angles	Bond	Angles
C(2)-N(1)-O(1)	113.8(3)	C(4)-C(5)-C(6)	121.8(3)	O(2)-C(11)-C(12)	117.6(4)
C(9)-N(2)-O(6)	123.1(3)	C(5)-C(6)-C(7)	118.1(3)	O(2)-C(11)-C(10)	121.5(3)
C(13)-O(3)-C(16)	117.3(3)	C(5)-C(6)-N(2)	117.6(3)	C(12)-C(11)-C(10)	120.9(4)
N(1)-C(2)-C(3)	116.7(3)	C(7)-C(6)-N(2)	124.3(3)	C(13)-C(12)-C(11)	119.6(4)
N(1)-C(2)-C(1)	123.5(3)	C(6)-C(7)-C(8)	119.9(4)	C(12)-C(13)-O(3)	124.9(3)
C(3)-C(2)-C(1)	119.8(3)	C(7)-C(8)-C(3)	122.7(3)	C(12)-C(13)-C(14)	120.9(4)
C(8)-C(3)-C(4)	116.0(3)	N(2)-C(9)-C(10)	123.0(3)	O(3)-C(13)-C(14)	114.2(3)
C(8)-C(3)-C(2)	121.8(3)	C(15)-C(10)-C(11)	117.2(3)	C(15)-C(14)-C(13)	118.8(4)
C(4)-C(3)-C(2)	122.2(3)	C(15)-C(10)-C(9)	121.9(4)	C(14)-C(15)-C(10)	122.6(4)
C(5)-C(4)-C(3)	121.5(3)	C(11)-C(10)-C(9)	120.9(4)		

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