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Short communication

Preparation and crystal structures of new schiff bases derived from 3, 3'-dihydroxy-4, 4'-(propane-1, 2-dieloxy)-dibenzaldehyde

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ABSTRACT

In this research study, two Schiff base compounds AP-DHPDB (1) synthesized from the reaction of 2-aminophenol (AP) with 3, 3'-dihydroxy-4, 4'-(propane-1, 2-dieloxy)-dibenzaldehyde (DHPDB) and FA-DHEDB (2) synthesized from the reaction of furfuryl amine (FA) with 3, 3'-dihydroxy-4, 4'-(ethane-1, 2-dieloxy)-dibenzaldehyde (DHEDB) by reflux in methanol as solvent for 2 h. Suitable crystals of 1 and 2 get by slow evaporation of solvent after few days and characterized by elemental analysis. Crystal structures of the title compounds 1 and 2 were determined using the single crystal X-ray diffraction analysis. The title compounds 1 and 2 were found to be in monoclinic and orthorhombic with the space group of C2/c and *Pnca*, respectively. The unit cell parameter of 1 were a=29/8994(10), b=4.86618(7), c=21.7214(4) Å, β=124.4901(17)° and V=2604.85(12) Å^3, and the unit cell parameter of 2 were a=22/3513(7), b=26.0250(7), c=4.6681(9) Å and V=2715(12) Å^3. The C-N bond distances around the iminic nitrogen of compound 1 are 1.418 and 1.268 Å, and in compound 2 are 1.467 and 1.269 Å, indicated the single and double bonds, respectively. Also, the bond angles around the iminic nitrogen are distorted from 120° corresponding to sp^2 hybrid (N1-C7-C8 = 122.5° and C6-N1-C7 = 119.3° for 1 and N7-C8-C9 = 123.9° and C6-N7-C8 = 116.12° for 2).

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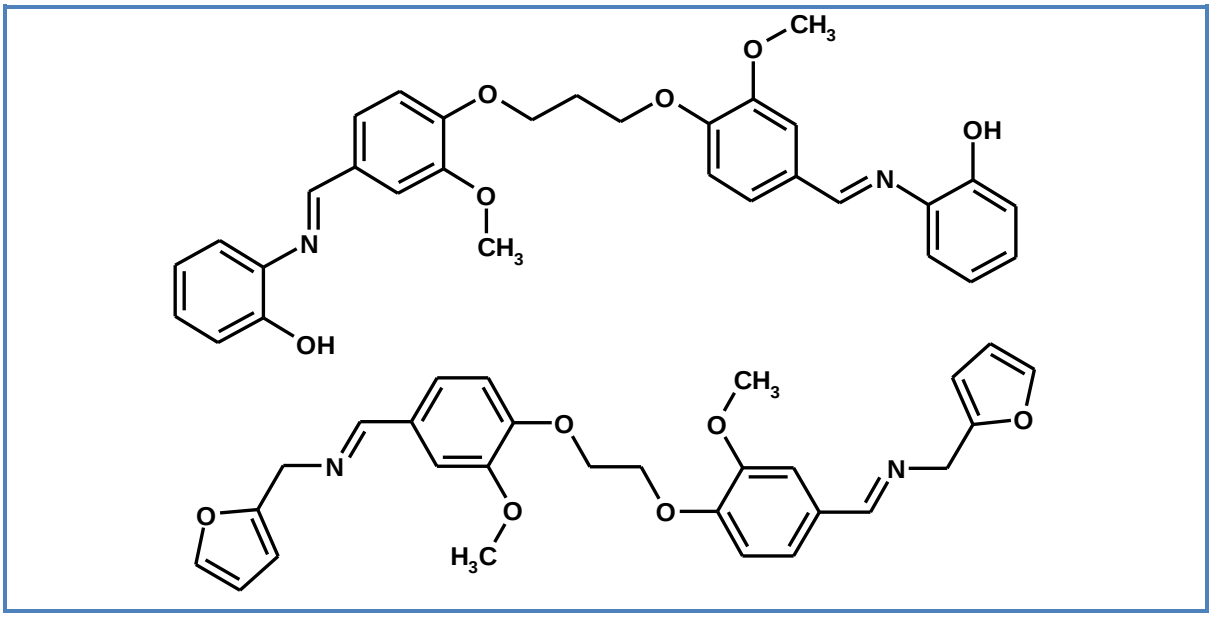
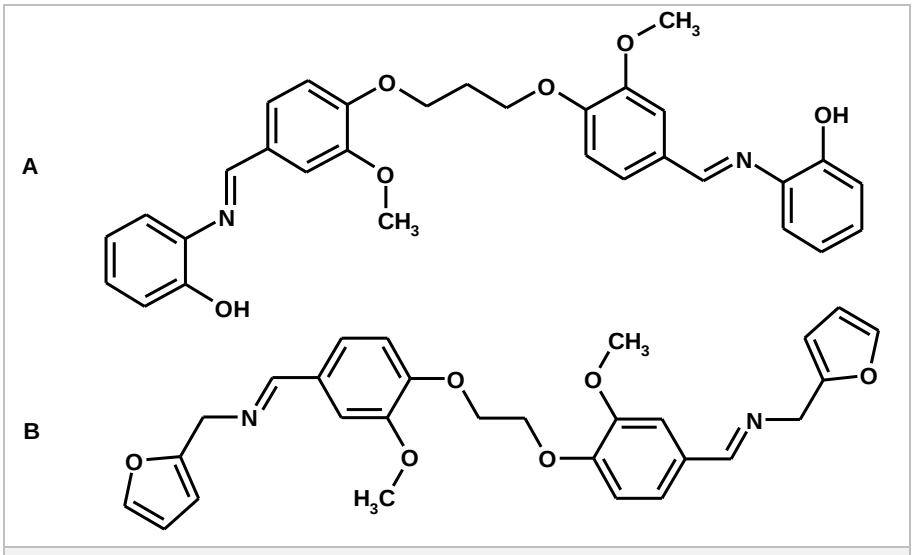
Graphical Abstract



Introduction

The Schiff bases are one of the most important compounds that are not only used as ligand for preparation various coordination compounds with transition metal ions such as copper(I) [1-3], copper(II) [4, 5], Ag(I) [1], Zn(II) [6-8], Hg(II) [9] and V(IV) [10], but also for various application and properties such as fluorescent pH prob [11], photoluminscent [12], detection of F^- and AcO^- [13], corrosion inhibition [14] and optical properties [15]. This is due to the fact that, Schiff base compounds are a) easily obtained by one-step procedure [16-19] b) the existence of tautomerization between phenol-imine and keto-amine form in Schiff bases derived from salicylaldehyde derivatives having 2-hydroxy group [20, 21] and c) are prepared stable complexes [1-10]. Reimann et al [22] prepare water-soluble sulfonate Schiff base ligands and reported the fluorescent detectors of various for metal ions. Novel tridentate NO_2 Schiff bases as Al^{3+} sensors have been prepared by Berrones-Reyes et al [23]. Zhu et al [24], prepared new Schiff base ligand from the reaction of 4-(diethylamino)benzaldehyde And 2-aminobenzoic acid in an ethanol and acetic acid medium solution and used it for the recognition of Fe(III), Fe(II) and Cu(II) ions. In 2019 [25], Kumar et al prepared turn-on multidentate Schiff bases as fluorescent sensors for selective detection of Al^{3+} and Ga^{3+} and pyrophosphate ion.

In continuation of our research on synthesis and characterization of Schiff base compounds [26-29], in this paper, we report on synthesis and crystal structures of two bis-iminic Schiff base compounds AP-DHPDB (**1**) and AP-DHEDB (**2**) (Scheme 1).



Scheme 1. Chemical structures of Schiff bases **1** (A) and **2** (B).

Experimental

Materials and measurements

All the chemicals (1,2-dibromoethane, 1,3-dibromopropane, 2-amino phenol, furfuryl amine, and ethanol) were purchased from the Merck and Aldrich chemical companies in high purity and used as received without any further purification. The bis-aldehyde compounds DHPDB and DHPDB were freshly prepared in accordance with our previous report [30]. Elemental analysis was carried out using a Heraeus CHN-O-Rapid analyzer.

Synthesis of AP-DHPDB (1)

The ethanolic solution of ortho amino phenol (2 mmol) was added to an ethanolic solution of DHPDB (1 mmol) and stirred for 0.5 h until the colourless clear solution was obtained. The mixture was cooled down at room temperature. After keeping the solution in air for several days by very slow evaporation of the solvent, the suitable crystals formed at the bottom of the vessel. The resulting crystals were filtered and washed twice with ethanol, and dried at room temperature. *Anal*. calcd. for $C_{31}H_{28}N_2O_6$: C, 70.45; H, 5.34; N, 5.34;. Found: C, 70.51; H, 5.38; N, 5.32%.

Synthesis of FA-DHEDB (2)

The ethanolic solution of furfuryl amine (2 mmol) was added to an ethanolic solution of DHEDB (1 mmol) and stirred and heated for 0.5 h until the colourless clear solution was obtained. The mixture was cooled down at the ambient temperature. After keeping the solution in air for several days by very slow evaporation of the solvent, the suitable crystals formed at the bottom of the vessel. The resulting crystals were filtered and washed twice with ethanol, and dried at room temperature. *Anal*. calcd. for $C_{28}H_{28}N_2O_8$: C, 64.61; H, 5.38; N, 5.38%. Found: C, 64.67; H, 5.41; N, 5.43%.

X-ray Crystallography

Suitable single crystals of **1** and **2** were chosen for X-ray diffraction study. Crystallographic measurements were done with four circle CCD diffractometer Gemini of Oxford diffraction, Ltd., with mirrors-collimated Cu-*K*α radiation. Crystal structure were solved by charge flipping with program

SUPERFLIP [31] and refined with the Jana2006 program package [32] by full-matrix least-squares technique on F^2. The molecular structure plots were prepared by Diamond 4.0 [33]. All hydrogen atoms were discernible in difference Fourier maps and could be refined to reasonable geometry. According to common practice H atoms bonded to C were kept in ideal positions with C–H = 0.96 Å, U_{iso}(H) was set to 1.2U_{eq}(C). All non-hydrogen atoms were refined using harmonic refinement. For disordered lattice water molecules hydrogen atoms could not be determined. Crystallographic data, details of the data collection, structure solution and refinements are listed in Table 1.

Results and discussion

The results revealed that, there is a good agreement between the experimental and theoretical results. The colourless crystals of the Schiff bases **1** and **2** were very stable at the solid state. The chemical structure of the title compounds is shown in Scheme 1. Figure 1 depicts the molecular structures of **1** and **2** with the numbering scheme. Selected bond distances and angles of **1** and **2** were summarized in Table2. The bond distances N1-C7 (1.27 Å) and N1-C8 (1.415 Å) in **1** and N7-C6 (1.471 Å) and N7-C8 (1.272 Å) in **2** are consistent with the distances of the C-N double and single bonds and are larger than those distances in similar Schiff base compounds [26]. The bond angles around the C and N atoms with sp^2 hybrid character were found to be at the range of 122º and 119º in **1** and 123º and 116º in **2**.

Table 1. Crystallographic data and structural refinement details

	1	**2**
Chemical formula	$C_{31}H_{28}N_2O_6$	$C_{28}H_{28}N_2O_8$
Formula weight	524.6	520.6
Crystal system	Monoclinic	Orthorhombic
Space group	*C*2/*c*	*Pnca*
a, Å	29.8994 (10)	22.3513 (7)
b, Å	4.86618 (7)	26.0250 (7)
c, Å	21.7214 (4)	4.66811 (9)
β, deg	124.4901 (17)	90
V, Å^3	2604.85 (12)	2715.41 (12)
Z, S	4, 2.57	8, 3.38
Parameter*s*	99	93
Restraints	49	45
$2\theta_{min}$, deg	4.01	4.02
$2\theta_{max}$, deg	100.00	79.99
R_p, R_{wp}, R_{exp}	0.023, 0.031, 0.012	0.023, 0.031, 0.009

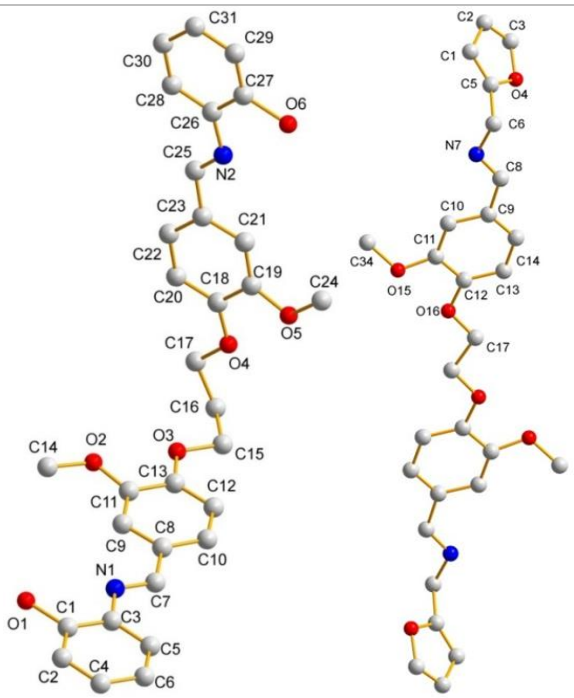


Figure 1. An ORTEP view of Schiff base compound **1** (left) and **2** (right).

Table 2. Selected bond distances (in Å) and angles (in deg) of **1** and **2**.

C1-O1	1.375	C6-C5	1.489
C12-O2	1.355	O4-C3	1.372
C19-O3	1.423	N7-C6	1.471
N1-C6	1.415	C8-N7	1.272
C7-N1	1.27	O4-C5	1.377
C8-C7	1.48	O15-C11	1.366
C16-O2	1.439	O16-C12	1.368
O3-C11	1.371	C17-O16	1.428
		C34-O15	1.427
C2-C1-O1	122.217	O4-C5-C1	109.64
C6-C1-O1	116.242	O15-C11-C10	125.79
C13-C12-O2	124.483	O4-C3-C2	110.24
C10-C11-O3	124.494	C3-O4-C5	106.57
N1-C6-C1	118.692	C6-C5-O4	115.35
N1-C6-C5	123.623	O16-C12-C11	114.34
C7-N1-C6	119.37	O16-C12-C13	125.46
C8-C7-N1	122.52	C17-C17-O16	108.06
C19-O3-C11	116.591	N7-C6-C5	110.17
C11-C12-O2	115.796	C8-N7-C6	116.1
C17-C16-O2	106.22	C9-C8-N7	123.87
		O15-C11-C12	114.96
		C34-O15-C11	116.53
		C17-O16-C12	117.39

Conclusions

In this work, two new bis-iminic Schiff base compounds were synthesized in an ethanolic solution and characterized using the elemental analysis (CHN) and single-crystal X-ray diffraction. Elemental analyses confirm the chemical composition of the as-prepared Schiff base compouns. Single crystal structure determination showed that the title compounds **1** and **2** have C_2 symmetry. Also, the compound **1** has a centre of symmetry with one half-molecule on the asymmetric unit. In addition, X-ray results revealed that the compounds 1 and 2 are not planar and have *E*-configuration with respect to the imine C=N bonds. These compounds are capable to coordinate as multidentate ligands and prepared various transition metal complexes.

Acknowledgments

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Conflict of interest

We have no conflicts of interest to disclose.

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