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Synthesis, Crystal Structure and Spectroscopic Study of (*E*)-2-methoxy-4-((1-phenylethylimino)methyl)phenol

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Authors' contributions

This work was carried out in collaboration among all authors. All authors read and approved the final manuscript.

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ABSTRACT

A new unsymmetrical ligand (E)-2-methoxy-4-((1-phenylethylimino)methyl)phenol $C_{16}N_{17}NO_2$ (I), was synthetized by one-step condensation reaction. The structure of this new compound was confirmed by elemental analysis, FT-IR, UV-Visible and ¹H and ¹³C NMR spectroscopy techniques. The compound (I) crystallizes in the orthorhombic space group P2₁₂₁₂₁ with the following unit cell parameters a = 11.7570 (6) Å, b = 10.0711 (6) Å, c = 23.4478(13) Å, V = 2776.4(3) Å³, Z = 4, R₁ = 0.045 and wR₂ = 0.125. The title compound crystallizes with two enantiomers (A and B) in the asymmetric unit which are both in E-stereoisomers. In the crystal structure of the racemic compound, the most noticeable difference between these two molecules is the dihedral angle values between the phenyl rings: 61.61 (1)° and 60.96(1)°.

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1. INTRODUCTION

"Over the past two decades, vanillin has been widely used as a source of Schiff bases due to its ability to react rapidly with primary aliphatic amino and amino aromatic compounds. These compounds which can present donor sites such as O or N are ligands widely used in coordination chemistry to develop complex materials with original properties" [1-3]. Depending on their topology and the nature of the substituents, they can interact with various metal ions such as bidentate [4,5], tridentate [5,6] or tetradentate [7, 8] ligands. "These ligands can also act in their neutral or deprotonated forms because of the equilibrium keto-enolic which can he established in solution" [9-11]. Several works in coordination chemistry involving ligands derived from vanillin have generated compounds with [12,13]. interesting properties Antioxidant properties acting positively on the prevention and treatment of cancer [14,15], inflammatory diseases [16], cardiovascular diseases [17], and neurodegenerative diseases [18] have been reported in the literature. In continuing our work in this area, we isolated the ligand (E)-2methoxy-4-((1-phenylethylimino)methyl)phenol from the condensation reaction between pvanillin 1-phenylethanamine. and The structure of the ligand was elucidated by FT-IR analyses, and NMR elemental spectroscopic studies and X-rav diffraction [19,20]. The ligand crystallizes with two enantiomers (A and B) in the asymmetric unit.

2. METHODOLOGY

2.1 Starting Materials and Instrumentations

4-hydroxy-3-methoxybenzadehyde and 1phenylethanamine were purchased from Aldrich and used without any further purification. The solvents and reagents were of good quality and were purified by usual methods. Elementary analyzes were carried out in a Carlo-Erba EA microanalyzer. Infrared spectra were recorded with a Perkin Elmer Spectrum Two FT-IR spectrometer in the 4000-400 cm⁻¹ region. The ¹H NMR spectrum was recorded in DMSO-d₆ on a Bruker 500 MHz spectrometer at room temperature using TMS as an internal reference. UV-Vis spectra were run on a Perkin-Elmer Lambda 365 UV/Visible spectrometer (1000-200 nm).

2.2 Synthesis of the Ligand

p-Vanillin (2 g, 13.145 mmol) was dissolved in a vial containing 20 mL of methanol. 1phenylethanamine (1.6 g, 13.145 mmol) and two drops of glacial acetic acid were added mixture successively. The reaction is brought to reflux for 4 hours. The resulting pink solution was filtered hot. After cooling, the resulting clear solution was left to slowly evaporate. Pink crystals were isolated after few days. Yield: 80%. Tf: > 260°C. Anal. Calc. for [C₁₆H₁₇NO₂] (%): C, 75.27; H, 6.71; N, 5.49. Found: C, 75.22; H, 6.70; N, 5.90. IR (v, cm⁻¹): $3200,\ 3080,\ 2980,\ 1628,\ 1577,\ 1513,\ 1396,$ 1262, 1026, 865, 826, 769, 699. ¹H NMR [DMSO-d₆; δ(ppm)]: 1.56 (d, 3H, -CH₃), 3.73 (s, 3H, -OCH₃), 4.59 (q, 1H, -CH-N), 8.11 (s, 1H, H-C=N), 6.65-7.25 (8H, H_{Ar}), 12 (s, 1H, Hphenol).

2.3 Crystal Structure Determination

"Crystals suitable for X-diffraction, of the reported compound, were grown by slow evaporation of MeOH solution of the complex. Details of the X-rays crystal structure solution and refinement are given in Table 1. Diffraction data were collected using an automatic XtaLAB AFC12 (RINC): Kappa single type diffractometer with graphite monochromatized MoKa radiation $(\lambda = 0.70173 \text{ Å})$. All data were corrected for Lorentz and polarization effects. No absorption correction was applied. Complex scattering factors were taken from the program package SHELXTL" [21]. "The structures were solved by direct methods which revealed the position of all non-hydrogen atoms. All the structures were on F^2 by a full-matrix refined leastprocedure squares using anisotropic displacement parameters for all non-hydrogen atoms" [22]. "The hydrogen atoms of OH groups were located in the Fourier difference maps and refined. Others H atoms (CH and CH₃ groups) were geometrically optimized and refined as riding model by AFIX instructions. Molecular graphics were generated using ORTEP-3" [23].

Chemical formula	C ₃₂ H ₃₄ N ₂ O ₄
Mr	510.61
Crystal system, space group	Orthorhombic, P212121
Crystal size (mm)	0.20 × 0.15 × 0.02
a (Å)	11.7570 (6)
b (Å)	10.0711 (6)
c (Å)	23.4478 (13)
V (Å ³)	2776.4 (3)
Z	4
D_{calc} (g.cm ⁻³)	1.222
I (Cu Kα) (Å)	1.54187
Т(К)	296
μ (mm ⁻¹)	0.64
F(000)	1088
□ range (°)	3.770-77.246
No. of measured reflections	19470
No. of independent reflections	5862
No. of observed $[l > 2\sigma(l)]$ reflections	5001
Rint	0.030
$R[F^2 > 2\sigma(F^2)]$	0.045
$wR(F^2)$	0.125
Goodness-of-fitt (Gof) on F ²	1.03
No. of reflections	5862
No. of parameters	359
No. of restraints	0
$\Delta \rho_{max}, \Delta \rho_{min} \ (e \ Å^{-3})$	0.22, -0.19

Table 1. Crystallographic data and refinement parameter for the compound (I)

3. RESULTS AND DISCUSSION

3.1 General Study

"The ligand HL was prepared by a facile p-vanillin condensation of and 1phenylethanamine in methanol in 1/1 ratio (Scheme 1). The resulting compound is quantitatively yielded (80 %). The elemental analyses results is in accordance with the chemical formulae obtained from X-ray diffraction study. The infrared spectrum of the compound (I) reveals bands which are in agreement with those of analogous compounds reported in the literature" [24,25]. In fact, the mainly interested band pointed at ca. 1628 cm⁻¹ is characteristic of the $v_{C=N}$ absorption, thus confirming the formation of the Schiff base. The v_{O-H} (3200 cm⁻¹) absorption of the phenolic group, which is expected in the range 3500 cm⁻¹ and 3300 cm⁻¹, is shifted to low frequencies due to the intramolecular hydrogen bond established with the methoxy group situated on the ortho position of the hydroxy group [26]. Bands pointed at ca. 3080 cm⁻¹ were attributed to C_{sp2}-H stretching of the phenyl groups while bands pointed in the range 2800-2900 cm⁻¹ are due to C_{sp3}–H stretching of the aliphatic group. The band characteristic of the presence of aromatic ring are pointed in the range 1577-1450 cm⁻¹ [27]. In addition, the characteristic bands of deformation vibrations of out-of-plane aromatic C-H bonds are pointed at 865 cm⁻¹ and 769 cm⁻¹. The band which appears at 1262 cm⁻¹ was assigned to the stretching vibration v_{C-O} of the phenolic group. The ¹H spectrum of the HL ligand was recorded in DMSO (dmso-d₆). The signals at 12 ppm representing one proton is due to the -OH of the phenolic moiety. The -HC=N proton of the azomethine moiety is pointed at 8.11 ppm. Signals at 1.56 ppm, 3.73 ppm and 4.59 ppm are, respectively, assigned to the protons of the methyl groups (CH₃-C and CH₃-O) and the proton of the methylidyne group (-CH-N). The electronic spectrum of the organic molecule recorded in DMF solution shows an intense absorption at 307 nm attributed to the $\pi \rightarrow \pi^*$ transitions of the aromatic rings and/or $n \rightarrow \pi^*$ of the imine function of the ligand.

3.2 Structure Description of the Compound (I)

The title compound crystallizes in the orthorhombic system with space group $P2_{1}2_{1}2_{1}$. The asymmetric unit of the title compound (I),

C₁₆H₁₇NO₂, contains two neutral molecules which are enantiomers (Fig. 1). The selected bond lengths and angles are collected in Table 2. The predominant structural feature is the formation of the C=N bonds between the carbonyl carbon atom of the p-vanillin molecules C9/C26 with the amino nitrogen atom of the 1-phenylethanamine molecules N1/N2 [C9-N1 = 1.269(4) Å and C26-N2 = 1.268(4) Å]. These two distances are found to be quite equal and are comparable to those found for similar Schiff bases [28,29]. All other bond lengths and angles are of expected values with C7-N1 [1.480(4) Å], C13-O2 [1.350(3) Å], C14-O1[1.368(3) Å], C23-N2 [1.484(4) Å], C30-O4 [1.348(3) Å], C31-O3[1.361(3) Å]. In the isomer A, the methyl group on the asymmetric carbon atom is disordered with two sites having occupancies of 0.7(1) and 0.3(1) for C24 and C25 respectively. The title compound crystallizes

with two enantiomers (A and B) in the asymmetric unit which are both *E*-stereoisomers. In the crystal structure of the racemic compound, the most noticeable difference in the structures of two enantiomers is the dihedral angle values between the phenyl rings : 61.61(1)° for A and 60.96(1)° for B. The 1-Hydroxy-2-methoxy units of the enantiomers A and B are planar (rms = 0.0139 (A), rms = 0.0070 (B)) and are guite parallel with a dihedral angle value of 1.091(2)°. The two phenyl rings of the two units are slightly twisted each other with a dihedral angle value of 4.851(3)°. The crystal packing of compound (I) (Fig. 2) is stabilized by intermolecular hydrogen bonds N(imino)-H···O(phenol). Intermolecular hydrogen bonds, N2(imino)-H2···O2ⁱ(phenol) (i: 1/2-x, 1-y, -1/2+z), N1(imino)-H4...O4ⁱ(phenol), (ii: 3/2-x, 1-y, 1/2+z), to the formation of layers parallel to b axis (Fig. 2, Table 3).



Scheme 1. Synthetic scheme for the compound (I)



Fig. 1. Molecular structures of both enantiomers (A) and (B) of the title compound with displacement ellipsoids drawn at the 30% probability

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Fig. 2. View along the a axis of the crystal packing of molecule of compound and hydrogen bond



Fig. 3. View along the a axis of the crystal packing of molecule of compound without hydrogen atom

Table 2. Selected bond distances	۲Å	l and and	ales	[dea] fo	r the	compou	und (I)
	<i>L'</i> 'Y	ana ang	9.00	[acg] .c		00111000	

O2-C13	1.350 (3)	O1—C16	1.413 (4)
O4—C30	1.348 (3)	N2—C26	1.268 (4)
O3—C31	1.361 (3)	N2—C23	1.484 (4)
O3—C33	1.419 (4)	N1—C9	1.269 (4)
O1—C14	1.368 (3)	N1—C7	1.480 (4)
N1—C7—C6	109.7 (3)	C24—C23—N2	117.9 (4)
N1—C7—C8	108.4 (3)	C25—C23—C22	125.8 (6)
C6—C7—C8	113.9 (3)	C24—C23—C22	115.6 (4)
N1—C7—H7	108.2	N2-C23-C22	109.5 (3)
C6—C7—H7	108.2	C24—C23—H23	104.0
C8—C7—H7	108.2	N2—C23—H23	104.0
C25—C23—N2	116.7 (6)	C22—C23—H23	104.0

<i>D</i> —H… <i>A</i>	<i>D</i> —Н	H <i>A</i>	D…A	<i>D</i> —H… <i>A</i>	
O2—H2…N2 ⁱ	0.82	1.88	2.689(3)	168.3	
O4—H4…N1 [⊪]	0.82	1.93	2.738(3)	166.2	

Table 3.	Hydrogen-bond	geometry (Å, °).
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4. CONCLUSION

In this present work we reported the synthesize of an unsymmetrical (*E*)-2-methoxy-4-((1phenylethylimino)methyl)phenol $C_{16}N_{17}NO_2$ (I). The structure of the new Schiff base was discussed by elemental analysis and spectroscopic techniques (FT-IR, UV-visible, ¹H NMR). X-rays diffraction technique was used to determine the molecular structure of (I).

SUPPLEMENTARY MATERIALS

CCDC-2310613 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge via https://www.ccdc.cam.ac.uk/structures/ or by contacting The Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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