

Synthesis, Spectroscopic Studies and Crystal Structures of N, N'-bis (4-methoxybenzylidene) Ethylenediamine and an Its New Cadmium (II) Complex

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Abstract: Schiff base (L) ligand is prepared via condensation of P-Anisaldehyde and ethylene diamine in 2:1 ratio in ethanol. A new complex is prepared by reacting this ligand with metallic salt CdI₂. The structure of the two derivatives are confirmed using various spectroscopic techniques: C, H, N analyses, IR, UV-visible and X-ray diffraction. The IR spectrums of ligand and complex show respectively intense peaks at 1629 cm⁻¹ and 1639cm⁻¹ indicating the presence of azomethine group (N=C). The crystal structure of the ligand belongs respectively to monoclinic, space group P21/n. In the crystal packing of the ligand, molecules are linked by a pair of methyl-C-H...O(methoxy) interactions, forming to an infinite one-dimensional chain. The structural characterization of the complex reveals that it crystallizes in an orthorhombic system P with as space group Pbca with a=14.8704 Å; b=12.9618 Å; c=22.4416 Å. The crystal structure of the monomer complex shows a distorted tetrahedral geometry. The Cd^{II} center is coordinated by the bidentate schiff base ligand via the two imine groups (N-donor) and the two chlorine atoms. Non-classical intermolecular H-bonds of the type benzene-C—H...O(methoxy) exist and link the monomeric complex, forming one dimensional chains.

Keywords: Crystal Structure, N, N'-bis (4-méthoxybenzylidène) Ethylenediamine, X-ray Diffraction, Schiff Base

1. Introduction

Schiff bases are important organic compounds class [1]. They were synthesized for the first time by Hugo Schiff in 1964 [2-3]. A Schiff base result from condensation of a primary amine and a carbonyl compound (ketone or aldehyde). It contains a double bond C=N with the nitrogen atom linked to an aryl or alkyl group [1, 4]. Schiff bases having multidentate coordination sites are known to form complexes with transition metal ions readily. Due to the relative ease of preparation, synthetic flexibility, and the special property of the C=N group, Schiff bases are generally excellent chelating agents, especially when a functional group such as -OH or -SH is present nearby of the azomethine group so as to form a five or six-membered ring

with the metal ion [5, 6]. Schiff bases in the broad sense have a general formula of the type R₁R₂C=NR₃, where R is an organic chain. In this definition, the Schiff base is synonymous with azomethine. Some limit the definition to secondary aldimines (azomethines or carbon is bound to only one hydrogen), and therefore have the general formula RCH=NR'. The carbon chain on the nitrogen atom makes Schiff bases (an imine) stable. The Schiff base synthesis reaction is often characterized by the release of a water molecule at the end of the reaction which could lead to a reversible reaction (hydrolysis). This reaction is usually carried out in an alcoholic medium at reflux [7, 8]. schiff bases complexes constitute one of the axes of research in

bioinorganic chemistry because of their wide applications in several fields, in medicine because of their pharmacological properties, in the catalysis of various biological systems and the preparation of enzymes, in the supramolecular chemistry as well as in industry. They also have a wide application in the field of biology with their antimicrobial, antibacterial, antifungal, [9-14], anticancer [15-18], anti-inflammatory [19] and antiviral [20-21] properties. These wide ranges of applications of these compounds have continued to attract interest among inorganic chemists.

In the present study, we report the results of our research on the synthesis of the ligand, N, N'-bis-(4-methoxybenzylidene) ethylenediamine obtained by the condensation of p-anisaldehyde and ethylene diamine, and its cadmium complex. These two synthesized compounds have been characterized by various spectroscopic studies. The monocrystalline structure of the ligand and the complex was also determined by X-ray diffraction.

2. Experimental Section

2.1. Materials

All reagents (P-Anisaldehyde; ethylene diamine, cadmium iodide and the solvent used is ethanol (98%)) were obtained from Sigma -Aldrich and used without any purification. The new ligand N, N'-bis (4-methoxybenzylidene) ethylene

diamine and its derivative were synthesized according to procedures described in the literature [22, 23]. Elemental analysis (C, H, N) data were obtained using an elemental analyzer. The IR spectra (KBr) were recorded on a Perkin-Elmer spectrophotometer in the range of 600–4000 cm^{-1} . A suitable crystal was selected and mounted on a cryoloop on a Bruker Smart APEX diffractometer. The crystal was kept at 100 K during data collection. Using Olex2, the structure was solved with the XT structure solution program using Intrinsic Phasing and refined with the XL refinement package using Least Squares minimization. UV spectra were obtained on a Lab-Tech UV-2100 UV Visible spectrophotometer in the range of 190–1200 nm using absolute alcohol (ethanol) as solvent and internal reference.

2.2. Synthesis

2.2.1. Synthesis of Schiff Base Ligand

The Schiff base ligand was isolated by mixing ethanoic solutions of ethylene diamine (30mmol; 1, 8g) and P-Anisaldehyde (60mmol; 7, 2 g). A colorless mixture is obtained. The latter is maintained under reflux heating for 5 hours at a temperature of 80°C. The mixture was then filtered using filter paper. After a few days of slow evaporation, crystals were obtained a mass of 6.345 g of with a yield of 80.89%. The synthesis reaction of the compound has been reported Figure 1.

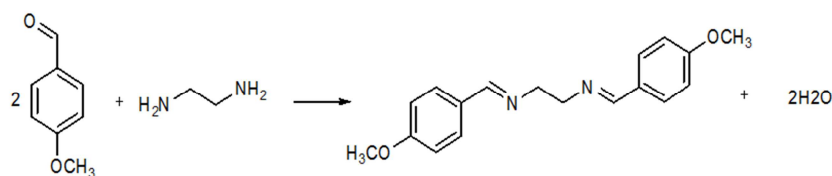


Figure 1. N, N'-bis-(4-methoxybenzylidene) ethylenediamine synthesis.

2.2.2. Synthesis of the Cadmium(II) Complex

The cadmium derivative was prepared by adding an iodide cadmium(II) solution (0.503 g; 1.37mmol) to an ethanolic solution of N, N'-bis-(4-methoxybenzylidene) ethylene diamine (0.406 g; 1.37mmol) in 1: 1 ratio. The mixture was stirred under reflux for five hours at a temperature of 80°C to give a yellow microcrystalline compound composition CdI_2L . The compound was filtered and evaporated slowly for a few days. the yield of the reaction is 44%.

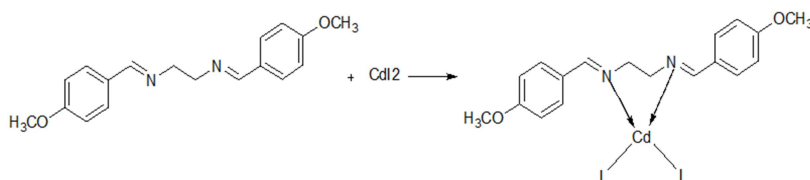


Figure 2. CdI_2L synthesis.

3. Results and Discussion

3.1. Structure Determination

X-ray data were collected using Bruker Kappa ApexII diffractometer, at $T=150$ K. Data were measured using φ and ω scans using $\text{MoK}\alpha$ radiation ($\lambda=0.71073$ Å). Data were corrected for absorption by multi-scan absorption correction

using *SADABS2012/1* [24]. The structure was solved using *SHELXT* [25] and the structure refined using least-squares minimization *SHELXL* [26]. Program used for the representation of the molecular and crystal structures: *Olex2* [27].

The two structures were determined after processing all the diffraction data for the N, N'- bis-(4-methoxybenzylidene) ethylenediamine ligand and its cadmium complex using the

Olex2 software or the model structural has been proposed by direct methods using the SHELXT program using intrinsic phasing and refined with the XL refinement package and least squares minimization. Crystallographic data are summarized in Table 1. All the hydrogen atoms were placed

by geometric calculation; atomic coordinates, anisotropic thermal agitation factors, interatomic distances and bond angles are listed respectively in the tables 2 and 3. Graphic illustrations were done using the Olex and Mercury drawing programs.

Table 1. Crystallographic data of the ligand and its complex.

Compounds	Ligand	Complex
Empirical formula	C ₁₈ H ₂₀ N ₂ O ₂	C ₁₈ H ₂₀ CdI ₂ N ₂ O ₂
Formula weight	296.36	662.56
Temperature/K	100	295
Crystal system	Monoclinic	Orthorhombique
Space group	P21/n	Pbca
a/Å	9.9522 (5)	14.8704 (4)
b/Å	7.8338 (4)	12.9618 (3)
c/Å	10.6286 (5)	22.4416 (4)
α/°	90	90
β/°	110.972 (2)	90
γ/°	90	90
Volume/Å ³	773.75 (7)	4325.55 (17)
Z	2	8
ρ _{calc} /cm ³	1.272	2.035
μ/mm ⁻¹	0.668	3.88
F (000)	316.0	2496
Crystal size/mm ³	0.28 × 0.15 × 0.09	0.16 × 0.08 × 0.08 mm
Radiation	CuKα (λ=1.54178)	Mo Kα (λ=0.71073 Å)
2θ range for data collection/°	10.456 to 143.43	2.7- 27.5
Index ranges	-12 ≤ h ≤ 12, -9 ≤ k ≤ 9, -13 ≤ l ≤ 13	-19 ≤ h ≤ 13, -16 ≤ k ≤ 16, -29 ≤ l ≤ 27
Reflections collected	13	
Independent reflections	14622	24974
Data/restraints/parameters	1511 [Rint=0.0298, Rsigma=0.0151]	4920 [R _{int} =0.047, R _{sigma} =0.0]
Goodness-of-fit on F ²		4920/0.228
Final R indexes [I ≥ 2σ (I)] Final R indexes [all data] Largest diff.	1511/0/101 1.070	R1=0.0291; wR2=0.0674
peak/hole / e Å ⁻³	R1=0.0425, wR2=0.1156 R1=0.0448, wR2=0.1186 0.27/-0.21	0.6 / -0.68

Table 2. Length and angle of connection in the ligand.

Ligand			
Length (Å)	Angle (°)		
O ₁ - C ₆	1.3649 (14)	C ₆ - O ₁ - C ₉	117.54 (9)
O ₁ - C ₉	1.4351 (14)	C ₂ - N ₁ - C ₁	116.86 (10)
N ₁ - C ₁	1.4616 (15)	N ₁ - C ₂ - C ₃	123.71 (11)
N ₁ - C ₂	1.2674 (16)	C ₄ - C ₃ - C ₂	122.45 (11)
C ₁ - C ₁ ¹	1.518 (3)	C ₄ - C ₃ - C ₈	118.32 (11)
C ₂ - C ₃	1.4699 (16)	C ₈ - C ₃ - C ₂	119.23 (11)
C ₃ - C ₄	1.3941 (17)	C ₅ - C ₄ - C ₃	121.25 (11)
C ₃ - C ₈	1.3986 (18)	C ₄ - C ₅ - C ₆	119.44 (11)
C ₄ - C ₅	1.3907 (16)	O ₁ - C ₆ - C ₅	124.23 (11)
C ₅ - C ₆	1.3946 (18)	O ₁ - C ₆ - C ₇	115.81 (10)
C ₆ - C ₇	1.3937 (17)	C ₇ - C ₆ - C ₅	119.96 (11)
C ₇ - C ₈	1.3823 (17)	C ₈ - C ₇ - C ₆	119.91 (11)
		C ₇ - C ₈ - C ₃	121.10 (11)

Table 3. The bond lengths and angles selected of the complex between the atoms of the crystal.

Complex			
Length (Å)	Angle (°)		
Cd1-I1	2.7098 (4)	I2-Cd1-I1	112.750 (13)
Cd1-I2	2.7015 (4)	N1-Cd1-I1	123.07 (7)
Cd1-N	12.308 (3)	N1-Cd1-I2	106.38 (7)
Cd1-N2	2.308 (3)	N1-Cd1-N2	78.33 (10)

Complex			
Length (Å)	Angle (°)		
C1-C6	1.464 (4)	N2-Cd1-I1	117.82 (7)
O1-C9	1.444 (4)	N2-Cd1-I2	114.15 (7)
O1-C17	1.357 (4)	N1-C6-C1	128.2 (3)
O2-C15	1.365 (5)	C7-N2-Cd1	107.3 (2)
C4-C1	11.453 (5)	C11-N2-Cd1	135.1 (3)
N1-C6	1.273 (4)	C11-N2-C7	116.9 (3)
N1-C8	1.477 (4)	C6-N1-Cd1	139.8 (3)
N2-C7	1.474 (4)	N1-C8-C7	110.5 (3)
N2-C11	1.272 (4)	C6-N1-C8	116.8 (3)
C7-C8	1.497 (5)	C8-N1-Cd1	103.3 (2)
O2-C18	1.431 (5)	C17-O1-C9	118.0 (3)
		C15-O2-C18	118.0 (3)
		O1-C17-C10	114.7 (4)
		O1-C17-C16	125.0 (3)
		O2-C15-C14	115.9 (4)
		O2-C15-C12	124.8 (3)

3.2. The Elementary Analysis of C₁₈H₂₀CdI₂N₂O₂

The cadmium complex is subjected to elemental analyses. Elemental analyses data (C, H and N) reported in Table 4 are in good agreement with those calculated for the suggested formula.

Table 4. Elementary analysis of cadmium complex.

Elements	% Found	% calculated
Carbone	31.28	32.60
Hydrogen	3.29	3.02
Nitrogen	4.28	4.23

3.3. Infrared (IR) Spectroscopy

Comparative study of IR spectral data of reported complex with those of free ligand gives sufficient evidence towards the better understanding of the coordinating behavior of the ligand.

Table 5. Spectral data IR of compounds

Compounds	ν (C=N)	ν (C=C) _{arom}	ν (C-O) _{ether}	ν (Cd-N)
Ligand	1639 cm ⁻¹	(1603; 1505; 1461; 1448) cm ⁻¹	1019 cm ⁻¹	
Complex	1629 cm ⁻¹	(1600; 1575; 1514) cm ⁻¹	1018 cm ⁻¹	767 cm ⁻¹

3.4. The Electronic Spectrum

The electronic transition study of the ligand and its complex were carried out in ethanol. For the ligand we observe one band at 317 nm, this absorption band corresponds to the $n-\pi^*$ transitions of the azomethine group ($-C=N$). while for the complex there is a slight decrease in the absorption band at 306 nm which explains the formation of the cadmium complex.

3.5. X-ray Crystallography

The ligand crystallizes in a monoclinic system with a space group P21/n with the lattice parameters given in Table 3. The overall structure of the ligand is an infinite chain. The monomeric are linked together by two bridges of weak intermolecular hydrogen bonds between oxygen O1 and carbon C9 (vice versa). These are only visible through Van der Waal links in the Mercury program. These bonds have an interatomic distance of 0.98.

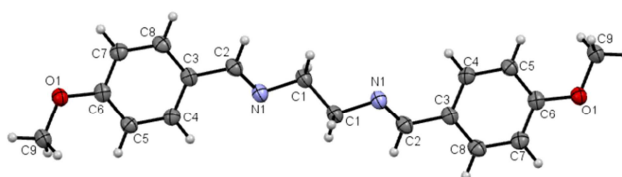
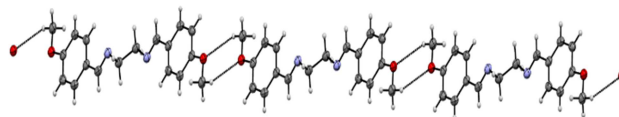
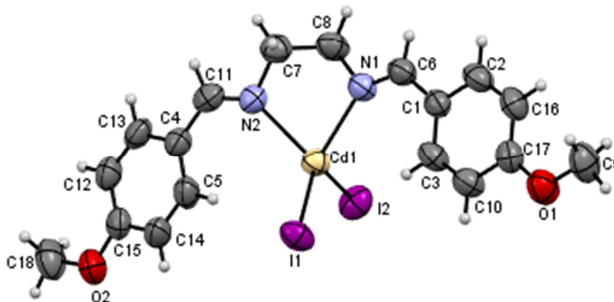
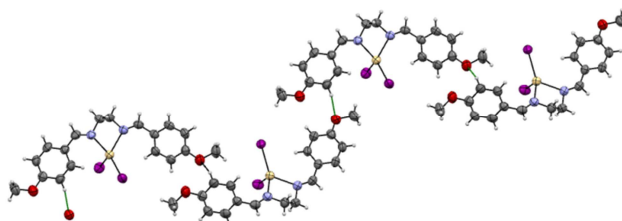
\tilde{A} along 1-X; 2.62 \tilde{A} along 1-Y and 3.4150 \tilde{A} along -Z with a D-H-A angle of 138.3° (figure 4). The elementary cell contains 2 molecules of the ligand.

The structural characterization of the complex reveals that it crystallizes in an orthorhombic system P with as space group Pbca with as mesh parameters are: $a=14.8704 \tilde{A}$; $b=12.9618 \tilde{A}$; $c=22.4416 \tilde{A}$; $\alpha=\beta=\gamma=90^\circ$ (table 1).

The complex structure shows that cadmium atom is tetra coordinated with N atoms as donor group, imine group contained in the ligand L2 and two iodides of CdI₂. Cd(II) are in tetrahedral environment. Bond lengths of the two azometine groups (N1-C6 equal to 1.272 \tilde{A} and N2-C11 equal to 1.273 \tilde{A}) in the cadmium complex are longer than those found in the ligand (N1-C2 equal to 1.2674 \tilde{A}). This extension confirms that azometine nitrogen atom participates at the coordination of the cadmium complex [32]. The molecules appear to be stacked and this hydrogen bond, when viewed along the axis, links the molecules forming infinite chains (Figure 5). The asymmetric unit consists of eight (08) molecules of the complex.

The azomethine band C=N appears around 1629 cm⁻¹ in CdI₂. L complex spectrum and around 1639 cm⁻¹ in the ligand one. This shift clearly shows coordination of this groupmate with Cadmium [28- 30].

Absorption band in 767 cm⁻¹, assigned to Cd-N bonds indicates the formation of the complex [31]. The C-O ether-oxide elongation vibration band located at 1019 cm⁻¹ in the free ligand spectrum, appearing at lower frequency 1018 cm⁻¹ in the complex one confirms interaction via hydrogen bonds of this group in the complex.

**Figure 3.** Monomeric structure of the ligand.**Figure 4.** Infinite chain of the ligand view. The C-H...O interactions are shown as dashed cyan lines (see Table 1 for details; H atoms not involved in these interactions have been omitted for clarity).**Figure 5.** Molecular structures of the ligand and its complex molecules.**Figure 6.** Crystal structure in infinite chain of the complex with hydrogen bonds (C-O...H) in green.

4. Conclusion

Schiff base N, N'-bis-(4-methoxybenzylidene) ethylenediamine and its cadmium complex have been synthesized P-Anisaldehyde and ethylenediamine in 2:1 ratio in ethanol solvent with good yield. They are characterized by FTIR, elemental analysis, electron spectra and X-ray diffraction. The crystallographic study confirms the spectroscopic conclusions. The complex X-ray data shows an infinite chain monomeric cadmium complex. The bidentate ligand is coordinated to the central cadmium (IV) atoms via the two imine-N. The Cd^{II} centre adopts a distorted tetrahedral coordination geometry. In the crystal structures, the monomeric are connected by a non-classical intermolecular H-bonds of the type C—H...O(methoxy). Furthermore, the biological activity of these two derivatives remains to be determined in order to propose drug candidates.

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