

Research Article

Synthesis, Spectroscopic Studies, Thermal Stability and Crystal Structure of New Bidentate Schiff Base and Its Cadmium Complex

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Abstract

A new asymmetric bidentate Schiff base ligand was synthesized from a condensation reaction between 2,4-dichlorobenzaldehyde ($C_7H_4Cl_2O$) and 1,3-pentanediamine ($C_5H_{14}N_2$). A cadmium complex was prepared by adding Cd(II) iodide dissolved in methanol to a methanolic solution of the Schiff base ligand, in a ratio of 1:1. The ligand was characterized by elemental, spectroscopic (IR, 1H NMR), and thermogravimetric analyses. The ligand structure, thermally very stable, is revealed to be bidentate with two potential N-donor sites. The crystal structure of the Schiff base was elucidated by X-ray diffraction analysis. Compound crystallizes in a monoclinic system with a space group $P2_1/n$ and a number of units per unit cell $Z = 4$. The unit cell parameters are: $a = 7.3885(2)$, $b = 16.9332(4)$, $c = 15.5624(4)$, and $\beta = 90.4306(10)$. In the asymmetric unit of the ligand, the benzene rings C08/C10/C09/C11/C21/C17 and C20/C15/C13/C18/C22/C24 are in trans position with the aliphatic group with respect to the bonds (C07-N05) and (C19-N06), respectively. In addition, these two aromatic rings are located in two different planes, forming an angle of 3.60° between them, given that the dihedral angle between the ring C08/C10/C09/C11/C21/C17 and the aliphatic group N05-N06 is 67.49° . The spectroscopic data of the complex reveals a mononuclear complex where the Cd(II) ion is housed in a N_2I_2 coordination site with a slightly distorted tetrahedral geometry. The conductance data indicate that the complex is a neutral electrolyte.

Keywords

Schiff Base, Cadmium, Ligand, Complex, Crystal Structure

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1. Introduction

A Schiff base is defined as a product containing an imine function in which one of the constituents on the carbon or nitrogen is an aliphatic or aromatic group [1, 2]. The product results from the attack of the nucleophilic agent, which is the primary amine, on a carbonyl compound (aldehyde or ketone) having an electrophilic focus, which is the carbon atom of the carbonyl function. This step is followed by the formation of a water molecule, which must be eliminated from the reaction medium in order to be able to shift the equilibrium of the reaction towards the formation of the Schiff base (Figure 1).

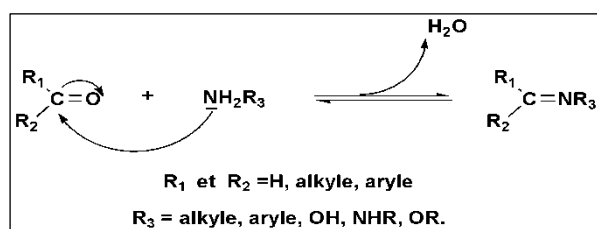


Figure 1. Schiff base formation reaction.

In recent years, researchers have given great importance to the synthesis and characterisation of Schiff base ligands and their transition metal complexes [3].

Complexes with Schiff base ligands are well known for their easy synthesis, stability, and wide application. A large number of Schiff base complexes have been reported so far.

Furthermore, Schiff base complexes have various applications as dyes, catalysts, pigments, stabilisers, and drugs [4-7].

Schiff base complexes with stable d^{10} electronic configuration have recently been extensively studied due to their diverse structures and applications [8, 9].

Cd(II) complexes are important because of the increasing recognition of their role in biological processes and their rich structural chemistry [10, 11].

There are many reports of cadmium Schiff base complexes having anticancer, antiproliferative, antioxidant, antibacterial, and antifungal activities [12, 13].

Due to interesting biological properties of cadmium complexes, in the present research, the synthesis and characterisation of a Cd(II) complex with a novel bidentate Schiff base are described.

2. Materials and Methods

2.1. Material and Physical Measurements

All chemicals used were of analytical grade, commercially available from different sources and used without further purification. Elemental analyses were performed on Perkin Elmer 2400 CHN. Infrared spectra (IR) were obtained on a

FTIR (Fourier-transform infrared) Spectrum Two of Perkin Elmer spectrophotometer in the $4000\text{--}400\text{ cm}^{-1}$ range. The ^1H NMR spectra in CDCl_3 was recorded at room temperature with a Bruker 200 MHz spectrometer. Molar conductivity of the complex was determined in DMF ($1.0 \times 10^{-3}\text{ M}$) at room temperature using "CD-2005" conductivity meter. Thermal gravimetric (TG) analysis was conducted on a TGA SHIMADZU model 50 thermal gravimetric analyzer.

2.2. X-ray Data Collection, Structure Determination and Refinement

Reflections were measured on a Bruker D8 Quest Apex3 diffractometer with graphite monochromator MoK α radiation ($\lambda = 0.71073\text{ \AA}$). Software package used for the intensity integration was SAINT (v8.40A) [14] and absorption correction was performed with SADABS [15]. The structure was solved with direct methods using SHELXT-2014/5. Least-squares refinement was performed with SHELXL-2018/3 [16] against $[F_o]^2$ of all reflections. Non-hydrogen atoms were refined freely with anisotropic displacement parameters. Hydrogen atoms were placed on calculated positions or located in difference Fourier maps. All calculated hydrogen atoms were refined with a riding model [17]. The structures obtained were drawn by the application Olex2 [18]. Crystal data and details of structure refinement for the Schiff base ligand are summarized in Table 1.

2.3. Synthesis of the Schiff Base Ligand N, N'-Bis(2,4-Dichlorobenzylidene-3-Propylammonium)Amine

After dissolving 3.500 g (20 mmol) of 2,4-dichlorobenzaldehyde ($\text{C}_7\text{H}_4\text{Cl}_2\text{O}$) in 15 mL of slightly heated methanol, add 15 mL of a methanolic solution of 1,3-pentanediamine ($\text{C}_5\text{H}_{14}\text{N}_2$) (0.881 g; 10 mmol). The reaction mixture is refluxed at 80°C for two hours, and the resultant solution is allowed to slowly evaporate at room temperature for 24 hours, during which time yellow single crystals were collected.

$[\text{C}_{19}\text{H}_{18}\text{N}_2\text{Cl}_4]$. Yield 76%. Analysis calculated, C: 54.83, H: 4.36, N: 6.45. Found, C: 54.70, H: 4.59, N: 6.71.

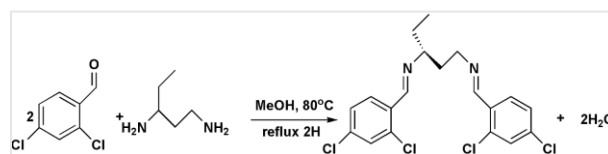


Figure 2. Synthesis of N, N'-Bis (2,4- Dichlorobenzylidene-3-Propylammonium)Amine.

2.4. Synthesis of Cadmium Complex

10 mL of a methanolic solution of the metal salt CdI_2 (0.366 g; 1 mmol) is introduced into a flask containing 10 mL of a methanolic solution of the Schiff base ligand (0.416 g; 1

mmol). The mixture is refluxed for 1 h at a temperature of 60 °C. An orange precipitate is obtained and then dried in the oven.

$[\text{C}_{19}\text{H}_{18}\text{N}_2\text{Cl}_4\text{I}_2\text{Cd}]$. Yield 62%. Analysis calculated, C: 29, 17, H: 2, 32, N: 3, 58. Found, C: 28, 51, H: 2, 52, N: 3,50.

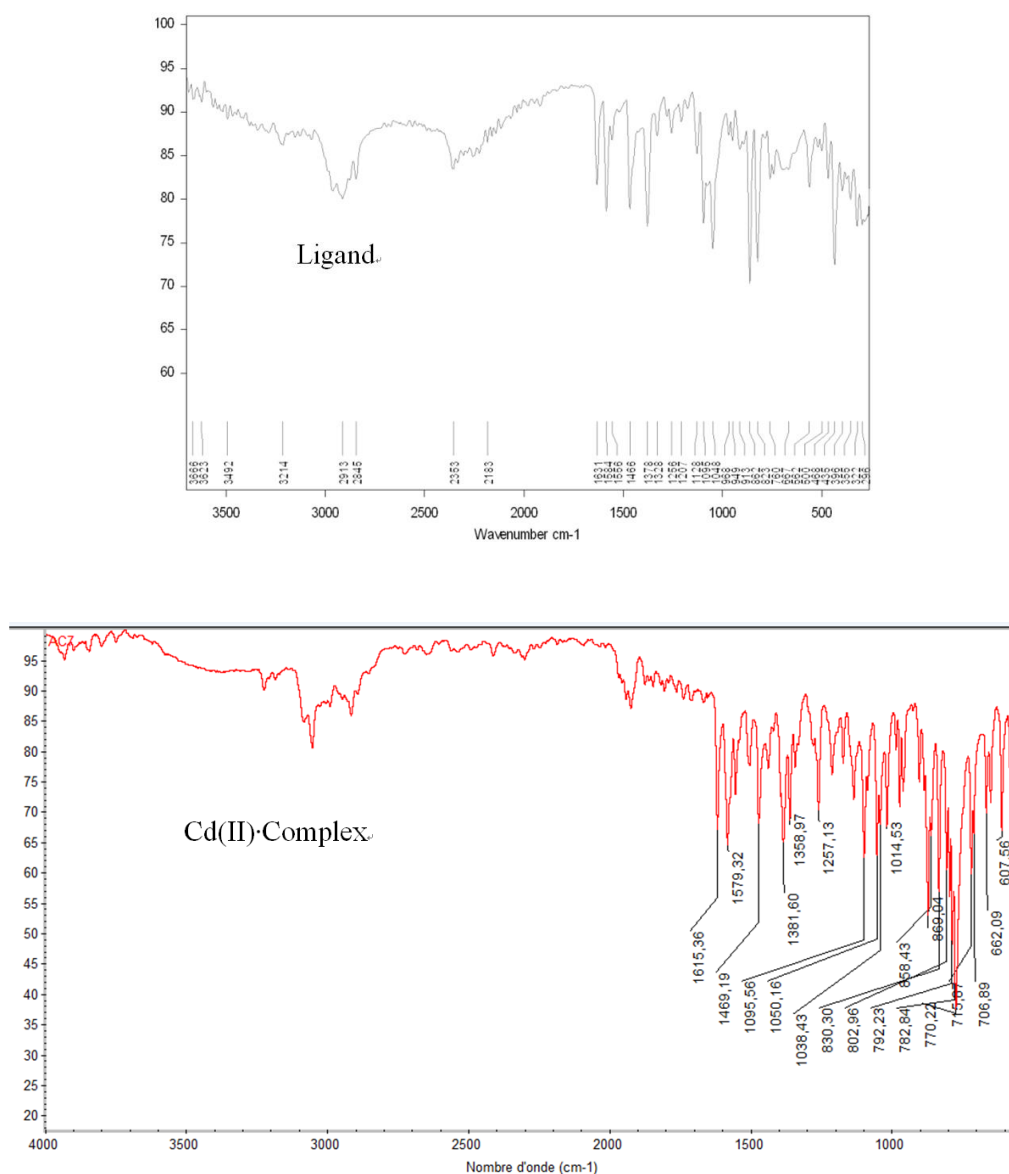


Figure 3. IR spectrum of ligand and Cd(II) complex.

3. Results and Refinement

3.1. FTIR Analysis of the Ligand and Complex

The IR spectra of the Schiff base ligand and Cd(II) complex are shown in Figure 3.

The IR spectrum of the ligand reveals an intense band at 1631 cm^{-1} attributed to the valence vibration of the imine

function $\nu(\text{C}=\text{N})$ [19]. This band is shifted to lower frequencies in the complex (1615.36 cm^{-1}), indicating that the nitrogen atoms of the azomethine groups are coordinated to the metal ion, such coordination reduces the electron density around the azomethine group which could be a reason for the observed shift towards lower frequencies [20, 21].

The IR spectrum of the metal complex would indicate new peaks in the $475\text{--}545\text{ cm}^{-1}$ and $430\text{--}480\text{ cm}^{-1}$ regions because of the constitution of the Cd-N and Cd-I peaks, respectively due respectively to the elongation vibrations $\nu(\text{Cd-N})$ and

$\nu(\text{Cd-I})$ [20-22].

3.2. ^1H -NMR Spectrum of the Ligand and Complexes

The ^1H NMR spectra of the Schiff base ligand and Cd(II) complex are shown in Figure 4, along with the carbon atom numbering of the ligand.

The ^1H NMR spectrum of the ligand shows the presence of two singlets at 8.61 ppm and 8.59 ppm attributed to the imine protons [23], while in the spectrum of the complex they appear as two singlets at 8.65 ppm and 8.60 ppm. A slight variation in the chemical shifts of the azomethine protons of the complex is noted compared to the free ligand, suggesting an involvement of the nitrogens of the imine groups in the coordination with the metal ion [23].

The aromatic ring protons are observed in the [7.25-8.12] ppm range as expected [23].

In the spectrum of the Schiff base ligand, the multiplet

observed in the region [2.90-3.78] ppm is attributed to the $\text{HC}_{(8)}$ and $\text{HC}_{(12)}$ protons (3H, m, $\text{H}_{(8,12)}$).

The triplet observed at 0.79 ppm is due to the protons of the methyl group (3H, t, CH_3) while the signals observed at 1.62 ppm and 1.96 ppm are attributable to the $\text{CH}_{2(9)}$ (2H, m, CH_2) and $\text{CH}_{2(11)}$ (2H, t, CH_2) protons, respectively [24].

The aliphatic and aromatic protons exit in the appropriate region in the spectrum of the complex.

3.3. Crystal Structure Analysis of Free Ligand

The bidentate Schiff base ligand crystallizes in a monoclinic system with a space group P21/n and a number of units per unit cell $Z=4$. The unit cell parameters are: $a=7.3885(2)$, $b=16.9332(4)$, $c=15.5624(4)$, and $\beta=90.4306(10)$ (Table 1).

The asymmetric unit view and the atom numeration are given in Figure 5.

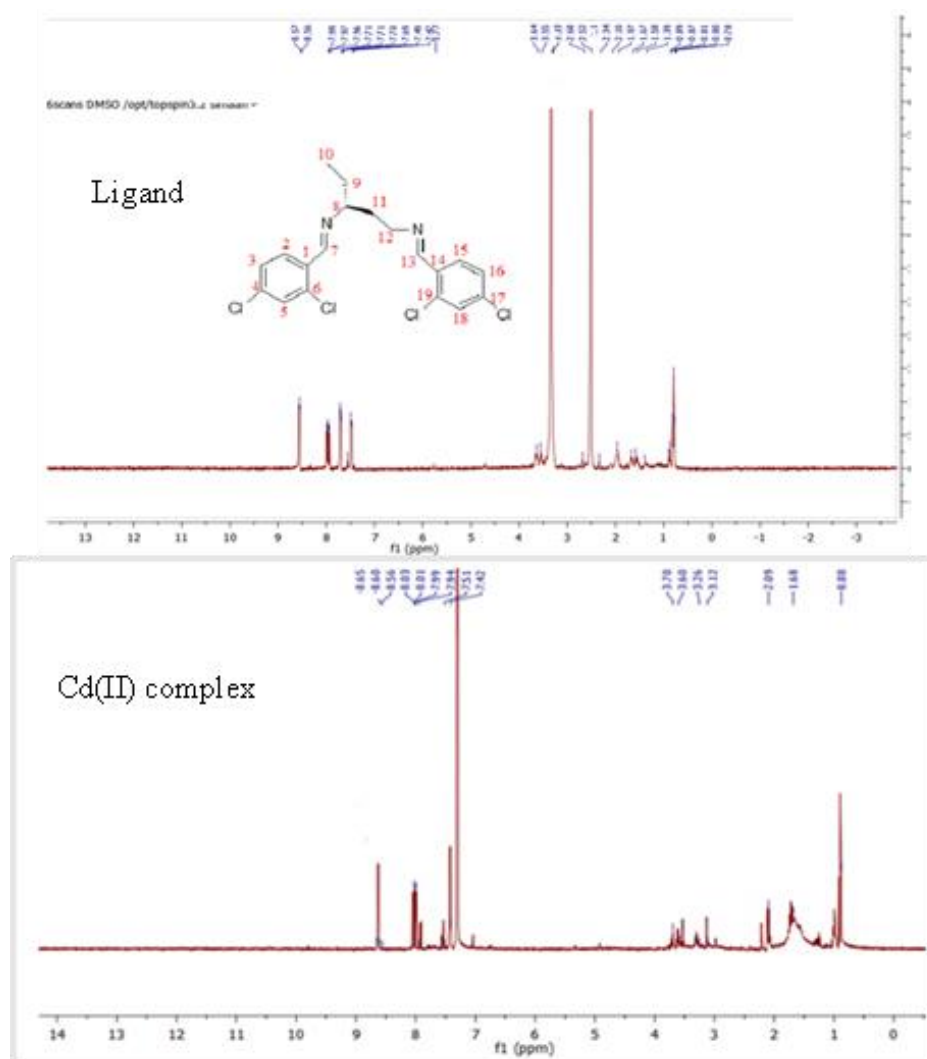


Figure 4. ^1H NMR spectrum of Schiff Base ligand and Cd(II) complex.

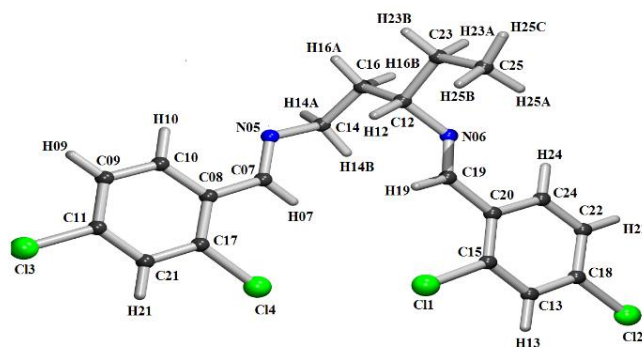


Figure 5. Asymmetric unit and atom numbering for the Schiff base ligand.

The benzene rings C08/C10/C09/C11/C21/C17 and C20/C15/C13/C18/C22/C24 are in the Trans position with the aliphatic group relative to the (C07-N05) and (C19-N06) bonds, respectively. The length of the C07-N05 bond is 1.2633 Å and that of C19-N06 is 1.2622 Å. They correspond to an iminic bond $>C=N$ [25, 26].

The C07 and C16 atoms are in trans position with respect to the C14-N05 bond, as are the C12 and C20 atoms with respect to the C19-N06 bond.

Moreover, these two aromatic rings are located in two different planes forming an angle of 3.60° between them, knowing that the dihedral angle between the C08/C10/C09/C11/C21/C17 ring and the aliphatic group N05-N06 is 67.49° (Figure 6).

In the crystal structure of the ligand, the molecules are grouped in parallel layers along the \vec{b} axis and there are no significant interactions between the present layers (Figure 7).

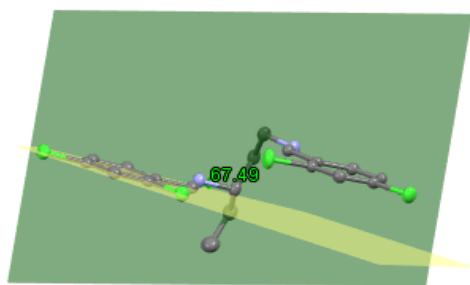


Figure 6. View of the dihedral angle between the planes of the ring C08/C10/C09/C11/C21/C17 and the aliphatic group N05-N06. In addition, the crystal structure reveals that the internal cavity of the ligand is not planar and that the aromatic rings are not coplanar.

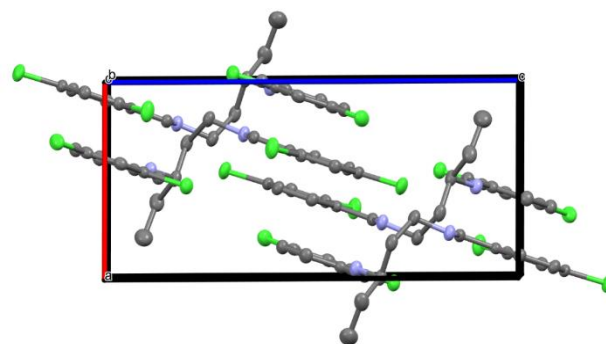


Figure 7. Crystal packing along the \vec{b} axis.

Table 1. Crystal data and structure refinement for Schiff base Ligand.

Formula	$C_{19}H_{18}Cl_4N_2$
Mr	416,15
Temperature (K)	150
Crystal system, Space group	Monoclinic, $P 2_1/n$
a, b, c (Å)	7.3885(2), 16.9332(4), 15.5625(4)
Crystal size (mm)	0.42 x 0.18 x 0.17
$\alpha = \gamma (^\circ), \beta (^\circ)$	90, 90.4306(10)
$V(\text{\AA}^3)$	1946.98 (9)

Formula	$C_{19}H_{18}Cl_4N_2$
Z	4
Crystal size (mm)	0.43 x 0.33 x 0.10
Dcalc. (mg/m ³)	1.420
Absorption correction μ/mm^{-1}	0.61
F(000)	856
Θ full (°)	2.7 – 28.3
Index ranges h, k, l	$-9 \leq h \leq 9, -22 \leq k \leq 22, -20 \leq l \leq 20$
Reflections collected	23967
Independent reflections R(int)	4837 [R _{int} = 0.022]
Parameters	227
Indices finaux R [$I \geq 2\sigma(I)$]	R ₁ = 0.031 W _{r2} = 0.081
$\Delta\rho_{\text{max}}, \Delta\rho_{\text{min}} / e \text{ \AA}^{-3}$	0.52/ -0.36

3.4. Thermal Analysis of Schiff Base Ligand

TG curve of the Schiff base ligand (Figure 8) was obtained at a heating rate of 5 °C min over a temperature range of 20-800 °C.

The TG curve of the Schiff base ligand shows that it is thermally stable up to approximately 105 °C. The compound decomposed in two steps during the conversion. The first step was observed in the range 105 °C - 290 °C with a weight loss of 17,5% (calculated 16.93%) which assigned to the loss of part of the ligand (C₅H₁₀) followed by a second step of the decomposition which is completed at 760 °C, implying a mass loss of 82,5 % (calculated 83.07 %) corresponded to the loss of the two remaining identical fragments (2C₇H₄NCl₂). The residue is zero, which confirms the total loss of the Schiff base [27].

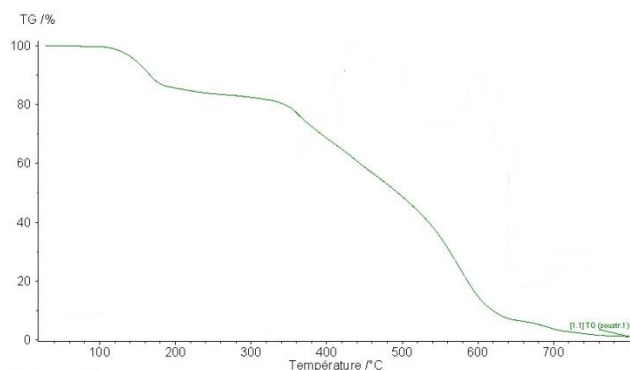


Figure 8. TG curve of the Schiff base.

3.5. UV-Visible Spectrophotometry of Cd(II) Complex

The electronic spectrum of the cadmium (II) complex (Figure 9) was recorded after its dissolution in methanol in the wavelength range from 200 to 800 nm at room temperature.

In the electronic spectrum of the complex, there are two absorption bands at 271 nm and 291 nm, attributed to the $\pi \rightarrow \pi^*$ transitions of the aromatic rings. The broad absorption band observed around 350 nm is attributed to the $n \rightarrow \pi^*$ transitions due to the azomethine groups. The spectrum does not show any d-d electronic transition because of the d¹⁰ electronic configuration of the Cd²⁺ ion [27].

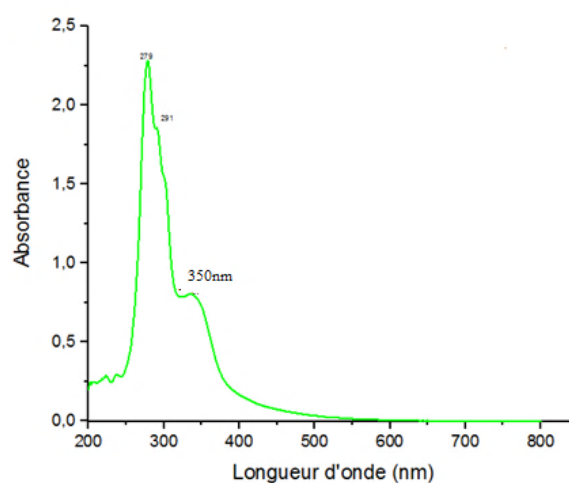


Figure 9. UV-Visible Spectrum of the Cd(II) complex.

3.6. Conductance Measurement of Cd(II) Complex

The Cd(II) complex was dissolved in DMF and the molar conductivity of 10^{-3} M of its solution at 25 °C was measured. Two measurements were taken within a 15 days interval to determine the stability of the complex in DMF.

The molar conductivity value obtained ($20.90 \text{ S.cm}^2.\text{mol}^{-1}$) indicates that the complex is a neutral electrolyte [28], revealing that the iodide ions are coordinated in the complex.

After 15 days, the molar conductivity of the complex ($29.20 \text{ S.cm}^2.\text{mol}^{-1}$) did not change significantly, indicating that the complex is stable in DMF solution.

3.7. Proposed Structure of the Cd(II) Complex

Based on the spectral and conductometric data discussed above, a distorted tetrahedral geometry is suggested for the studied complex. The structure of the complex is presented in Figure 10.

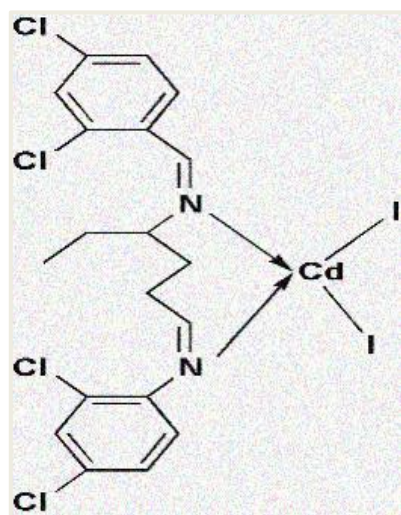


Figure 10. Structure of the cadmium complex.

4. Conclusion

In conclusion, a new Schiff base ligand and its cadmium complex have been synthesized and characterized using microanalysis (CHN), IR and ^1H NMR spectroscopies. The structure of the Schiff base ligand is confirmed by XRD, which is revealed to be bidentate with two N-donor sites and asymmetric with very good thermal stability (105 °C). The results of elemental analysis of the cadmium complex confirmed a ligand/metal molar ratio of 1:1. In addition, based on spectroscopic data and conductimetric measurements, the mononuclear cadmium complex adopts a slightly distorted tetrahedral geometry around the metal.

Abbreviations

IR	Infrared Spectra (IR)
FTIR	Fourier-Transform Infrared
TG	Thermal Gravimetric
UV	Ultraviolet
XRD	X Ray Diffraction
IMM	Institute for Molecules and Materials

Supplementary Material

(Please see the link <http://www.ccdc.cam.ac.uk/services/structures?access=refere&pid=ccdc:2389093&author=Tinnemans>)

CCDC-2389093 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 e-mailing data_request@ccdc.cam.ac.uk, or by contacting The Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44(0)1223-336033.

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Author Contributions

Aboubacar Diop: Writing-original draft, Writing-review & editing

Tidiane Diop: Supervision, Visualization, Validation

Daouda Ndoye: Supervision, Visualization, Validation

Dame Seye: Supervision, Visualization, Validation

Paul Tinnemans: Data curation, Formal Analysis, Resources, Software, Validation

Cheikh Abdoul Khadir Diop: Supervision, Validation, Visualization

Conflicts of Interest

The authors declare no conflicts of interest.

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