



SYNTHESIS AND CHARACTERIZATION SELECTED OF SCHIFF BASES AND THEIR COBALT (II) COMPLEXES.

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KeyWords

5-bromosalicylaldehyde, 5-bromosalicylidene-aniline, 5-bromosalicylidene-4-nitroaniline, Schiff base.

ABSTRACT

Schiff base ligand and their metal complexes have attracted the attention of researchers due to their interesting biological activity. Structural modification of Schiff base ligand and their metal complexes changes their chemical and physical properties hence affecting their biological activity. In this work, synthesis of the Schiff bases was done by mixing 5-bromosalicylaldehyde and selected anilines in ratio of 1:1 and then refluxed under ethanol solution for 4 hours. To obtain the respective complexes, hydrated cobalt (II) chloride was added to respective Schiff base ligand in ethanol solution and then heated under for 3 hours under reflux. The precipitate obtained was filtered and dried at ambient temperature. The compounds obtained melted at constant temperature, indicating that were all pure. They were later characterized with Fourier Transform-Infrared (FT-IR), Ultra Violet-Visible (UV-VIS) and Nuclear Magnetic Resonance (NMR) spectroscopy. The FT-IR spectra of the ligands ranged from 1612-1614 cm^{-1} attributed to CH=N bond stretching vibration, on complexing the ligand to Co (II), the band shifted to lower frequency. The peak due to OH appeared at range of 3472-3478 cm^{-1} which disappeared on complex formation. ^1H NMR spectra of CH=N proton appeared at 8.14ppm and 8.22ppm due to 5-bromosalicylidene-aniline (BA) and 5-bromosalicylidene-4-Nitroaniline (BN) respectively. On formation of BA and BN complexes their peaks shifted downfield. The peak at 9.28-10.19ppm is due to hydroxyl group proton of BA and BN Schiff bases respectively. The hydroxyl proton peaks disappeared on complex formation. The ^{13}C NMR spectra appeared 155-159ppm respectively due to BA and BN azomethine carbon respectively. On complex formation, the peak shifted up field to 163-165ppm respectively. The peak at 159-161ppm is due to phenolic carbon spectra of BN and BA respectively. On coordinating the ligand to Co (II), all the peaks shifted up field. The Uv-vis spectra of the ligands appeared at 269-274nm due to transition of non-bonding electrons of azomethine nitrogen of BN and BA respectively. These spectra underwent blue shift on complexes formation. These shifts in peaks indicated that the ligands coordinated to Co (II) through nitrogen and oxygen atom.

Introduction

A Schiff base is a compound formed when aldehyde and primary amine react to form azomethine nitrogen bond (Arulmugan *et al.*, 2010). Schiff base ligands and their metal complexes display many biological activity such as antibacterial activity, anti-inflammatory and anticancer. The lone pair of electron in azomethine bond has been found to be responsible for these bioactivity (Omoruyi *et al.*, 2016). They form hydrogen bond with the target receptor site in biological molecule (Basha *et al.*, 2019). Coordinating the ligand to transition metal improve the antibacterial activity by reducing the electron density in ligand chemical structure. Substituted salicylaldehyde moiety has shown a considerable activity against antibacterial agent (Meenukutty *et al.* 2022).

In this work, 5-bromosalicylidene-aniline (BA) was synthesized by refluxing 5-bromosalicylaldehyde with aniline under ethanol solution. The equation for reaction is as shown in figure 1 while 5-bromosalicylidene-4-nitroaniline (BN) was synthesized by refluxing 5-bromosalicylaldehyde and 4-nitroaniline under ethanol solution for four hours. The equation for reaction is as shown in figure 2. BA and BN Schiff base ligands was coordinated to cobalt (II) ion to form there respective complexes. The structure of BA complex and BN complex is as shown in figure 3 and 4 respectively.

Physical and spectroscopic characterization of the two ligand shows that condensation occurred successful.

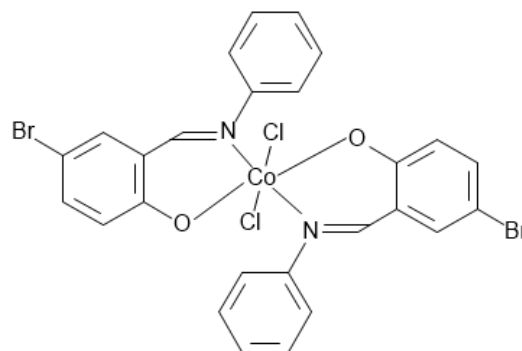


Figure 3: 5-bromosalicylidene-Aniline cobalt (II) complex synthesized.

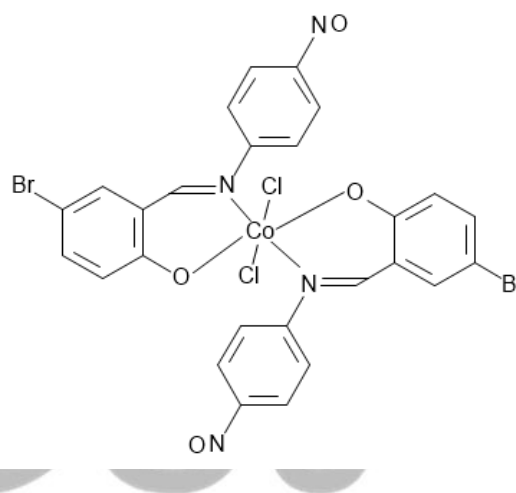


Figure 4 5-bromosalicylidene-4-Nitro-Aniline cobalt (II) complex synthesized.

Materials and Methods

Experimental site

This research was done at Chuka University located at Chuka town in Tharaka Nithi County along Meru-Nairobi highway. Synthesis of the ligands and their cobalt (II) complexes, UV-vis and FT-IR characterization was done at, chemistry laboratory. NMR characterization was done at University of Nairobi -Chiromo campus located along the Riverside drive off Chromo road.

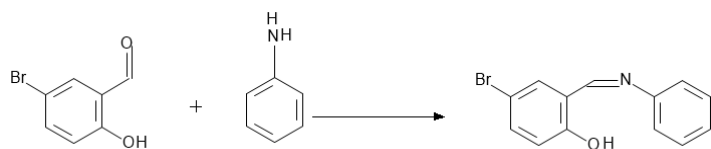


Figure 1: Synthesis of 5-bromosalicylidene-Aniline.

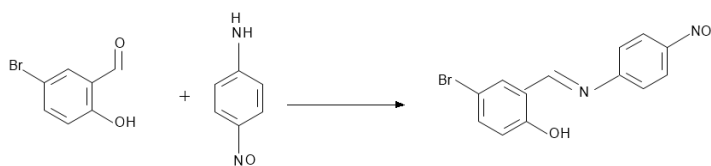


Figure 2: Synthesis of 5-bromosalicylidene-4-Nitro-Aniline.

Experimental material

All reagents, starting materials and solvents were of analytical grade. They were purchased from reputable sources: 5-bromosalicylaldehyde and 4-nitroaniline was purchased from Sigma-Eldrich while aniline, ethanol, cobalt chloride and DMSO was obtained from ASPET school supplies LTD.

Synthesis of 5-bromosalicylidene-Aniline (BA)

Here 2.01g (0.01moles) 5-bromosalicylaldehyde was dissolved in 50ml ethanol. Then 0.9cm³ (0.01moles) of aniline was added to 50ml of 5-bromosalicylaldehyde ethanol solution of in a round bottomed flask. The reaction flask was fitted with refluxing column, placed in laboratory heating mantle and then refluxed for three hours. Orange solution was obtained, sharp needle like crystals were observed on cooling. The solid crystals were obtained through vacuum filtration, washed with cold ethanol and diethyl ether to remove any unreacted aldehyde and ammine and then dried at room temperature. Some of the solids obtained were used to obtain cobalt complex while the rest were transferred in polypot container for storage awaiting characterization and antibacterial testing (Jayalakshmi *et al.*, 2017).

Synthesis of 5-bromosalicylidene-Aniline (BA)

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for storage awaiting characterization and antibacterial testing (Jayalakshmi *et al.*, 2017).

Synthesis of 5-bromosalicylidene-Aniline cobalt (II) complex (BA complex).

Here 1.0g (0.0036 moles) of BA Schiff base was carefully put into 100ml round bottomed flask and 50 ml of absolute ethanol was added. The flask was then placed in laboratory heating mantle. The mixture was heated to dissolve the Schiff base. Thereafter, 0.469g (0.0081mole) hydrated cobalt (II) chloride was carefully added into the flask containing BA Schiff base. The refluxing column was fitted over the round bottomed flask and then the solution refluxed for three hours. A brown crystal was obtained; the solids was obtained through vacuum filtration and then washed with methanol and hot ethanol to remove any unreacted Schiff base and cobalt chloride. The solid obtained was then transferred into polypot container for storage awaiting characterization and antibacterial susceptibility testing (Ommenya *et al.*, 2020). The products for the reaction was 5-bromosalicylaldehyde-Aniline cobalt (II) complex.

Synthesis of 5-bromosalicylidene-4-Nitroaniline cobalt (II) complex (BN complex)

Whereby 1.0g (0.0031 moles) of BN Schiff base was carefully put into 100 ml round bottomed flask. 50 ml of ethanol solvent was then added into the flask containing the Schiff base. The flask was put in laboratory heating mantle and then heated to dissolve the Schiff base. Thereafter, 0.369g (0.00156mole) hydrated cobalt (II) chloride was then added carefully into the round botted flask containing BN Schiff base solution. The refluxing column was fitted over the round bottomed flask and then refluxed for three hours. Green crystals were observed on cooling the solution. The solid crystals were obtained through vacuum filtration; purified by washing with methanol and hot ethanol to remove any unreacted ligand and cobalt chloride. The solids obtained was then dried at room temperature. It was transferred into polypot container

for storage awaiting characterization and antibacterial susceptibility testing (Ommenya *et al.*, 2020). The products for the reaction expected was 5-bromosalicylidene-4-Hydroxo-Aniline cobalt (II) complex.

Characterization of the Obtained Schiff bases and their cobalt (II) Complexes

After synthesis, the ligands and their complexes formed were characterized to obtain data to clearly establish their purity and elucidate their chemical structure. The following are characterization techniques used to determine purity and the elucidate the chemical structure of the synthesized ligands and complexes.

Determination of Melting Point

The Schiff bases melting point and their corresponding cobalt (II) complexes was determined by an open capillary method. One of the open end of Capillary tube was heated non-luminous flame for 2-3 minutes to close it. The Solid substances whose melting point was to be determined was placed on a tile and then crushed into a fine powder. The capillary tube with closed end was held firmly between the fingers and thumb. The open end of the capillary tube was dipped into finely powdered solid sample to pick them. It was then gently tapped to fill the compound to about a length of 1-2 cm.

With the help of a thread, the capillary tube was attached to the thermometer and then dipped into a beaker containing liquid paraffin such that bulbs of thermometer and the closed end capillary tube are on the same label and they were both dipped into a liquid paraffin up to length of around 2cm. The thermometer was then held using stand and clamps. The liquid paraffin was heated using non luminous flames of Bunsen burner as shown in the plate 8 (Basu & Date, 1998).

The temperature of the liquid was observed as it rises continuously and the temperature at which the substance started to melt was noted immediately and recorded as T_1 . The temperature at which the compound had melted completely was noted as T_2 . T_1 and T_2 values were averaged to determine correct melting point of the substances. i.e

$$\text{Average temperature} = (T_1 + T_2) / 2$$

Average temperature was presumed to be the melting point of substances.

UV-VIS Spectra

The UV-VIS spectra of synthesized Schiff bases and their corresponding complexes was determined according to (Bahreldin *et al.*, 2020) using UV-VIS Spectrophotometer Shimadzu –UV-1800. 0.01g of the sample was dissolved in 5 ml of DMSO. 1 ml of the solution placed in cell of the Spectrophotometer and then the absorbance of each sample was scanned from range of 190 nm -1000 nm and recorded. The solution was diluted up to order of 10^{-3} where an absorbance value of less than 10 was obtained (Jackson & Mantsch, 1995).

FT-IR Spectra

FT-IR spectra of the synthesized ligands and their complexes was determined using FT-IR Spectrophotometer Shimadzu – IR affinity according to (Bahreldin *et al.*, 2020). The 0.1g of sample was mixed with 0.001g of KBr and pressed under pressure. The resulting melt matrix of KBr melts was placed in sample holder and then inserted into FT-IR Spectrophotometer Shimadzu –IR affinity and then scanned in wave number region 4000cm^{-1} - 200cm^{-1}

NMR

The determination the ^1H and ^{13}C NMR spectra of the ligands and cobalt complexes was determined according to (Bhatt *et al.*, 2019). For ^1H NMR analysis 6mg of each solid synthesized sample material was measured and DMSO- D_6 used as a deuterated solvent. 0.7ml of deuterated solvent was added to the measured NMR sample and shook to dissolve completely. The soluble NMR sample was then transferred into clean unscratched NMR tube by glass Pasteur pipette and then closed by clean cap. TMS was used as internal standard for chemical shift calibration. One drop of TMS was used for each NMR tube used. The NMR tube was then labelled using permanent marker to avoid confusion. The NMR tube was then inserted

in NMR spectrometer and then scanned at 100 megahertz for ¹H resonance frequency. The same procedure was used to carry out ¹³C NMR analysis however 60 mg of solid NMR sample was measured and NMR spectrometer was scanned at 400 Megahertz

RESULTS AND DISCUSSION

Synthesis of selected Schiff base.

BA and BN Schiff base ligands was synthesized successfully. There was a change of colours of the final products as compared to the starting materials as shown by Table 2 This agrees with other previously reported publications (Omoruyi *et al.*, 2016 & Ommenya *et al.*, 2020), who reported that change of colour of products to a different from the starting material suggest the formation of new product. This was further confirmed through spectral analysis as shown in section 4.2

The percentage yield of both Schiff base ligands was 89% and 95% for BN and BA respectively. BA and BN Schiff bases ligands were insoluble in cold ethanol but soluble in hot ethanol and DMSO.

Both Schiff bases BA and BN melted at constant temperatures of 266^oc and 268^oc respectively, indicating that they were all pure. This agrees with other previously reported publications (Ommenya *et al.*, 2020), who reported that pure substance melt at constant temperature while impure substances melt at range of temperatures.

Synthesis of cobalt (II) complexes from synthesized selected Schiff bases

BA complexes and BN complexes were synthesized successful as suggested by the change of colour of the final products which is different from the colour of their respective Schiff bases ligands as shown in Table 1 and plate 9. The percentage yield of all the complexes was high. BA complex had percentage yield of 92% while BN complex had percentage yield of 97%. Both complexes were insoluble in both hot and cold eth-

anol. It was only soluble in DMSO solvent. The two complexes had higher melting point than their respective Schiff base ligand as shown in Table 2. This shows that Co (II) complexes of the Schiff base ligand synthesized were more stable than their respective ligand. This is in agreement with previously reported publication (Goodwin, 2002), who stated that coordinating the N and O Schiff base to metal ions increases the stability of the complexes hence this make metal complexes to have melting point higher than their corresponding ligands.

Table 1 : Physical properties of the ligand and their complexes.

Name	Appearance	Colour	Percentage yield	Melting point (°c)
BA	Needle-like crystal	Orange	95%	266
BA complexes	Irregular shaped crystal	Brown	92%	280
BN	plate-like crystals	Green-orange	89%	268
BN complexes	Irregularly shaped crystal	Green	97%	278

Spectroscopic characterization of the selected Schiff base ligand and their Co (II) complexes

The spectral obtained were analyzed keenly to find out if bonds expected was formed and bond expected to disappear were absent. The subsection below is detailed analysis of each spectroscopic spectrum obtained.

FT-IR spectra

The absence of weak band at 3280 cm⁻¹ and 1664 cm⁻¹ for NH₂ and C=O function group indicate the formation of the Schiff base through azomethine nitrogen (HC=N). This agrees with

publication reported by (Johnson, 2021), who reported that NH₂ and C=O function group displayed weak band at 3280 cm⁻¹ and 1664 cm⁻¹ respectively. This was confirmed by the presence of band 1612 cm⁻¹ and 1614 cm⁻¹ due to formation of HC=N bond stretching vibration for BA and BN respectively as reported by (Jayalakshmi *et al.*, 2017). On complexing cobalt(II) ion to the Schiff base the band shift to lower frequency of range 1172-1173cm⁻¹ this show the possibility of Schiff base coordinating through azomethine nitrogen (Om-menya *et al.*, 2019).

The presence of the band at 3472-3478cm⁻¹ assignable to –OH (intermolecular hydrogen bonding) stretching bond and its absence on the complexes indicate the possibility of Schiff base coordinating through phenolic oxygen to the cobalt(II) ion (Kuddushi *et al.*, 2018). The peaks at 2858cm⁻¹ and 2866 cm⁻¹ are assignable to C-H vibrational mode (Johnson, 2021).

BA ligand FT-IR spectra

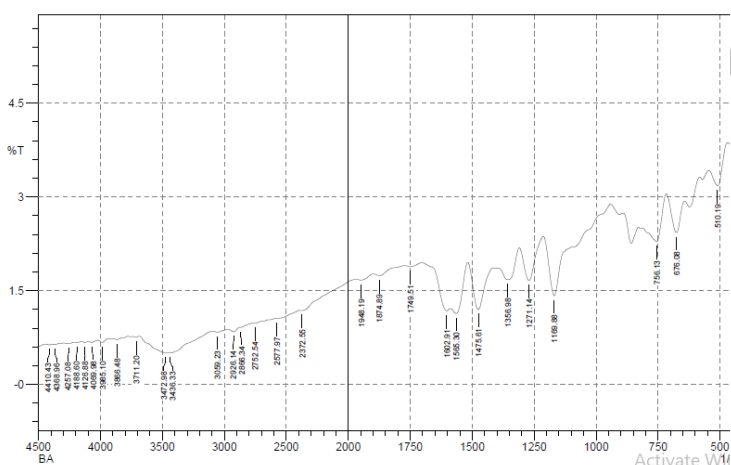


Figure 5 : BA Schiff base FT-IR spectra.

BA complex FT-IR spectra

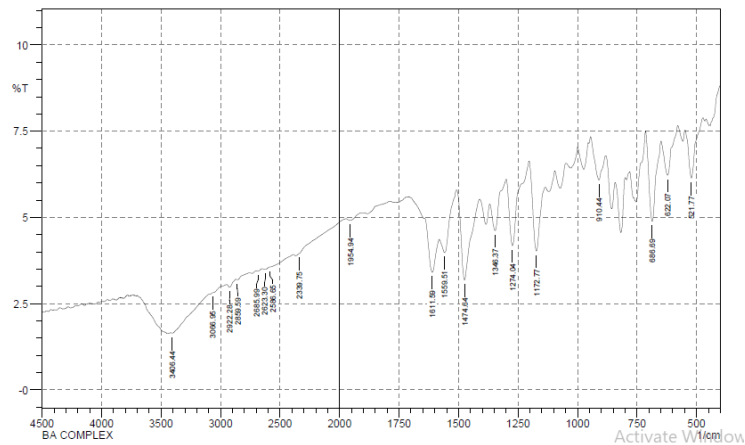


Figure 6 : BA complex FT-IR spectra.

BN Schiff base FT-IR spectra

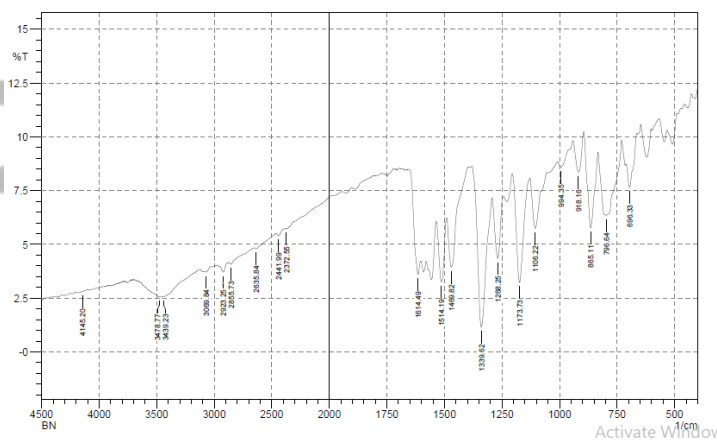


Figure 7: BN Schiff base FT-IR spectra.

BN complex FT-IR spectra

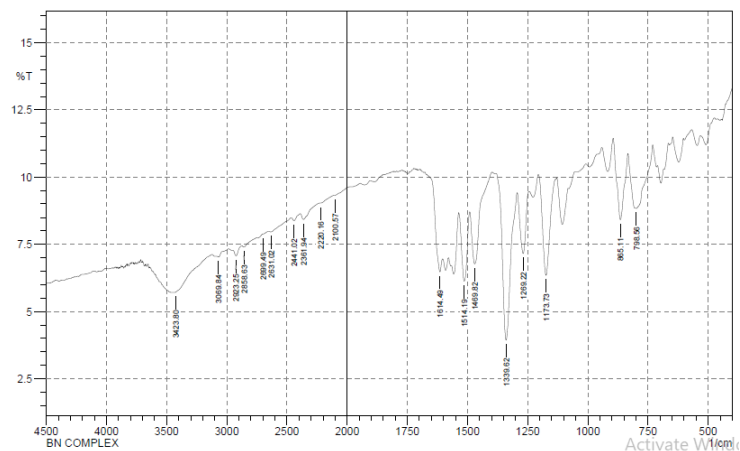


Figure 8: BN complex FT-IR spectra.

BN ligand ¹H NMR spectra

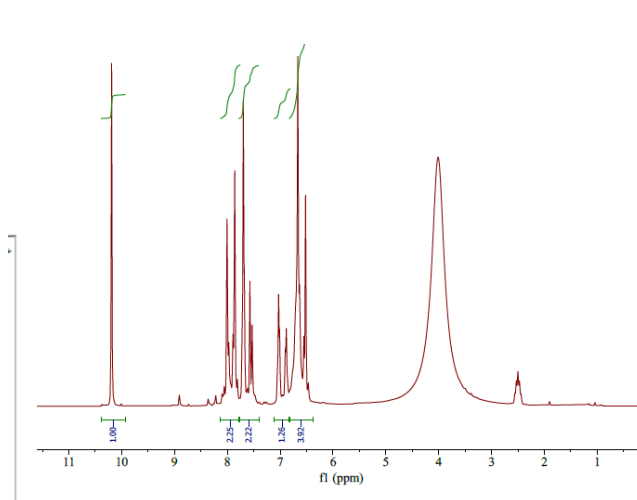


Figure 9 : BN¹H NMR spectra.

¹H-NMR spectra

The ¹H-NMR spectra of (HC=N) proton (Figure 20) in BN Schiff base was observed at 8.22 ppm and shift downfield to 8.14 ppm on complexing to cobalt (II) ion (Figure 21 (Omoruyi *et al.*, 2016) while the BA Schiff base was observed at 8.31 ppm (Figure 22) and also shift downfield to 8.27 ppm on complexing (Yousif *et al.*, 2017). This is due to deshielding of the imine proton as the nitrogen atom donate their lone pair to the Co (II) ion (Gichumbi *et al.*, 2017). This evidence supports the complexing of the Schiff base to cobalt (II) ion through azomethine nitrogen (Figure 23) (Nworie *et al.*, 2016).

The peak at 9.28 ppm in BA was due to phenolic proton while the peak at 10.19 ppm is due to BN phenolic proton (Ommenya *et al.*, 2020). Both peaks disappear in the two complexes. This means bidentate coordination of the Schiff base to cobalt (II) ion through azomethine nitrogen and phenolic oxygen took place.

BN complex¹H NMR spectra

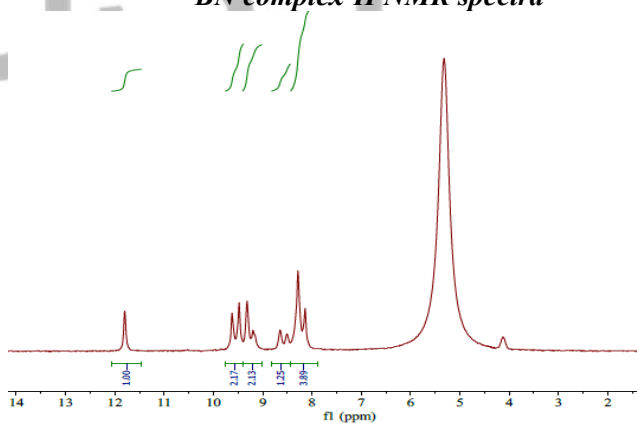


Figure 10 : BN complex ¹H NMR spectra.

BA ligand ¹H NMR spectra

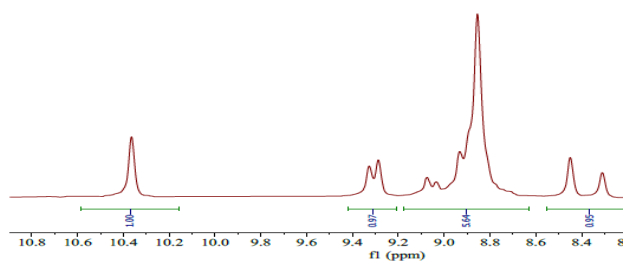


Figure 11 : ¹H NMR spectra for BA.

BA complex ¹H NMR spectra

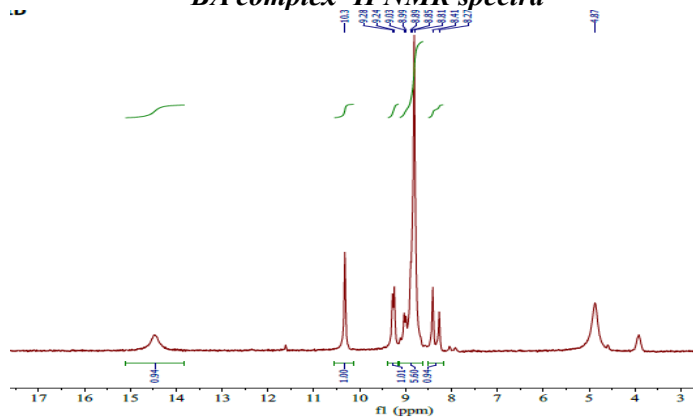


Figure 12 : BA complex ¹H NMR spectra.

shifted up field indicating the use of phenolic oxygen in coordination as reported by (Bahreldin *et al.*, 2020). He reported that phenolic carbon atom display peak from range of 157-163ppm which shift up field upon complexing of the ligand to metal ion (Figure 24-27).

BA ¹³Carbon-NMR spectra

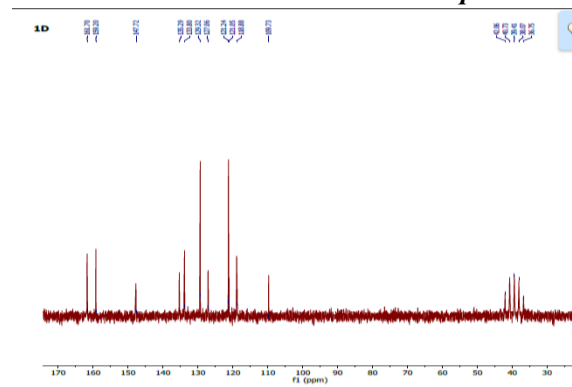


Figure 13 : BA ¹³Carbon-NMR spectra.

BA complex ¹³Carbon NMR Spectra

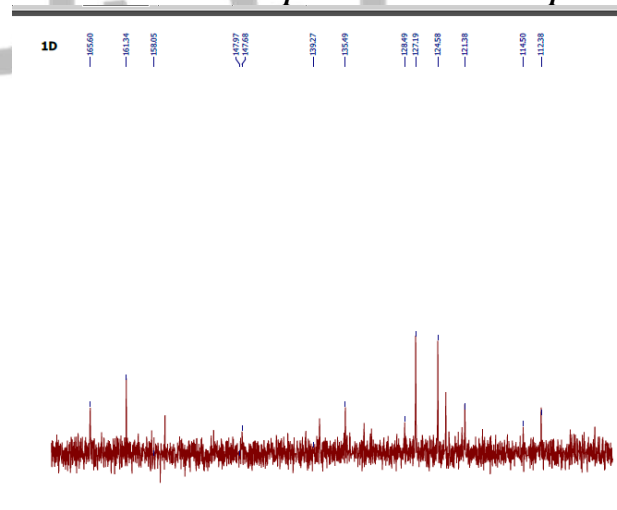


Figure 14: BA complex ¹³Carbon NMR Spectra.

¹³C-NMR spectra

The ¹³C-NMR peak of the BA and BN Schiff base was observed at 155 ppm and 159 ppm respectively corresponding to azomethine carbon. On complexing Schiff base to cobalt (II) ion, these peaks were shifted up field to 163 ppm and 165 ppm respectively. This shows participation of nitrogen atom of azomethine group in coordination. This in an agreement with publication reported by (Kratky *et al.*, 2017), who reported that peak of azomethine carbon is expected from range of 150-159ppm and these peaks shift up field upon coordinating the ligands to the metal ion.

The peaks at 161 ppm in BA and 159 ppm in BN Schiff base is due to phenolic carbon at the respective Schiff bases. On complexing the respective Schiff bases each carbon peak is

BN ¹³Carbon-NMR spectra

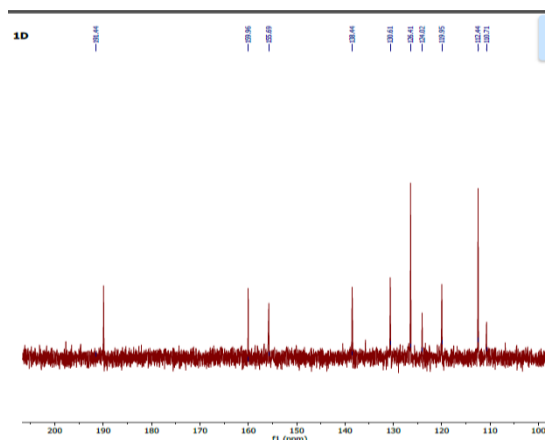


Figure 15: BN ¹³Carbon-NMR spectra.

BN complex ¹³Carbon-NMR spectra

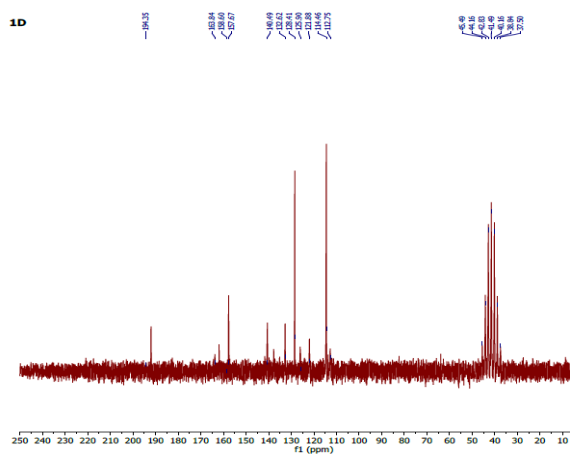


Figure 16 : BN complex ¹³Carbon-NMR spectra.

Uv-vis spectra

The Uv-vis spectra of BN Schiff base showed two peaks at 269 nm ($37,174 \text{ cm}^{-1}$) and 367 nm ($27,247 \text{ cm}^{-1}$). The peak at 269 nm ($36,496 \text{ cm}^{-1}$) is attributed to $n=\pi^*$ transition of non-bonding electron presents in azomethine nitrogen ($\text{HC}=\text{N}$) and the peak 367 nm ($27,247 \text{ cm}^{-1}$) is attributed to $n=\pi^*$ transition of phenolic group (OH) (Ommenya *et al.*, 2019).

The Uv- vis spectra of the Schiff base cobalt complexes showed similar absorption spectra as the free Schiff base ligand which had undergone a blue shift. In addition, the band in electronic spectra of BN complex due to $\text{CH}=\text{N}$ transition shifted toward shorter wavelength of 266nm ($37,593 \text{ cm}^{-1}$)

and the band attributed to phenolic group (OH) shifted toward shorter wavelength of 365 nm ($27,397 \text{ cm}^{-1}$). BA Schiff base showed four peaks. The peak at 274nm ($37,174 \text{ cm}^{-1}$) is due to $n=\pi^*$ transition of non-bonding electron of azomethine nitrogen ($\text{HC}=\text{N}$) and the peak at 348nm ($25,735 \text{ cm}^{-1}$) is attributed to $n=\pi^*$ transition of phenolic group (OH). on coordinating BN Schiff base to Co (III) the peak due to $n=\pi^*$ transition of non-bonding electron presents in azomethine nitrogen ($\text{HC}=\text{N}$) shifted toward shorter wavelength of 272nm ($36,769 \text{ cm}^{-1}$) and the peak due to phenolic oxygen shifted toward longer wavelength of 349nm ($28,653 \text{ cm}^{-1}$). The result was in agreement with other previously reported publication (Johnson, 2021), who reported that peak for $n=\pi^*$ transition for non-bonding electron in phenolic group is observed at range of $25,000\text{-}27,500 \text{ cm}^{-1}$ wavenumber and peaks for $n=\pi^*$ transition of non-bonding electron of azomethine nitrogen is observed at $36,000\text{-}37500 \text{ cm}^{-1}$. Both blue shift and red shift indicate involvement of oxygen atom and nitrogen atom in coordination.

All the three spectroscopic technique confirms that the Schiff bases ligand synthesized formed bidentate Co (II) complexes involving of the oxygen atom and nitrogen.

BA Schiff base UV VIS spectra

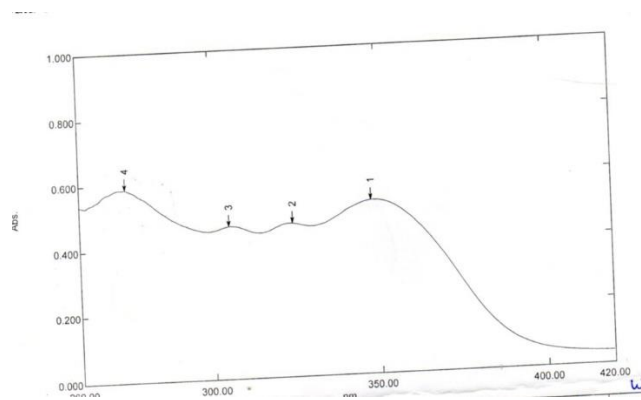


Figure 17: BA Schiff base UV VIS spectra.

BA complex UV VIS spectra

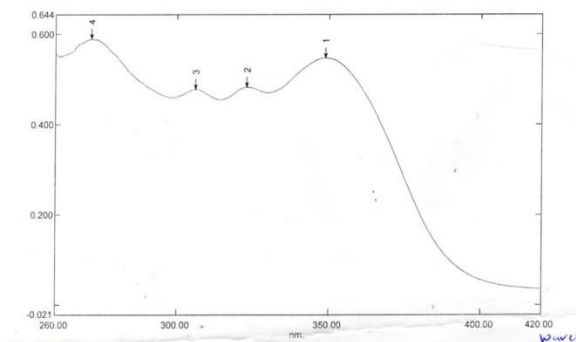


Figure 18: BA complex UV VIS spectra.

BN Schiff base UV VIS spectra

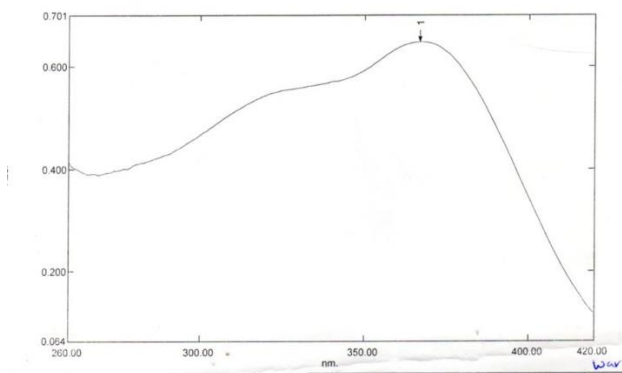


Figure 19: BN Schiff base UV VIS spectra.

BN complex UV Vis spectra

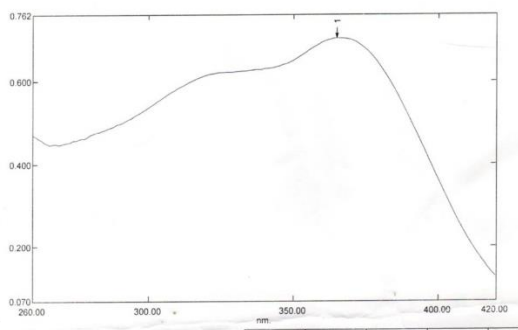


Figure 20: BN complex UV VIS spectra.

Conclusion

Condensation of 5-bromosalicylaldehyde to aniline and 4-nitroaniline separately occurred separately as expected, leading to the the formation of 5-bromosalicylidene-aniline and 5-bromosalicylidene-4-nitroaniline schiff bases respectively. These Schiff bases 5-bromosalicylidene-aniline and 5-bromosalicylidene-4-nitroaniline coordinated to cobalt (II) ion successfully. The new Schiff bases BA, BN and their Co (II) complexes were synthesized successfully and structurally characterized. The NMR, FT-IR and UV-VIS spectra of BA, BN and their Co (II) complexes had shifted there peaks when compared to those of starting materials.

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