



Cobaloxime Complexes of Diphenylglyoxime with Aniline and Substituted Aniline: Synthesis and Antimicrobial Study

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ABSTRACT

Five cobaloxime complexes represented as $[\text{Co}(\text{Hdpg})_2(\text{R})(\text{SCN})]$ where H_2dpg is diphenylglyoximate, R = aniline (A), 4-bromoaniline (4BrA), 2-nitroaniline (2NA), 4-nitroaniline (4NA) or 4-anisidine (PA) have been synthesized and characterized by melting point, conductance measurement and infrared spectroscopy. The complexes were obtained in low yields (18 - 25 %) in the order $4\text{BrA} > 4\text{NA} > \text{A} > 2\text{NA}$ when the ligands interacted with cobalt(II) acetate tetrahydrate. However, $[\text{Co}(\text{Hdpg})_2(\text{A})(\text{SCN})]$ was obtained in good yield (59 %). With the exception of $[\text{Co}(\text{Hdpg})_2(\text{PA})(\text{SCN})]\text{CH}_3\text{COO}^-$ which is 1:1 electrolyte, these complexes showed low conductivity values ($35 - 54 \Omega^{-1}\text{cm}^2\text{mol}^{-1}$), hence non-electrolytic in nature. Furthermore, they are of relatively low thermostability with melting points in the range (70 - 118°C). The results from IR spectroscopy indicated coordination of the cobalt ion via the nitrogen of the ligands. An octahedral coordination in which the central cobalt ion is surrounded by four nitrogen atoms of the two diphenylglyoximate mono anions from the equatorial positions and the other two sites occupied by atoms of monodentate neutral ligands and thiocyanate anion from the axial position was proposed. The evaluation of their *in vitro* antibacterial activity of the cobaloxime complexes were carried out against different strains of bacterial isolates including *MRSA (Methicillin Resistance Staphylococcus) Staphylococcus aureus*, *Bacillus subtilis*, *Streptococcus pneumoniae*, and the Gram negative bacteria, *Salmonella typhi*, *Klebsiella pneumoniae*, *Escherichia coli*, and *Pseudomonas aeruginosa*. The results revealed that the zones of inhibition were only noticed in the case of *Staphylococcus aureus* and *Salmonella typhi*.

Keywords: Axial, Equatorial, Diphenylglyoximate, Glyoximate, Isothiocyanate, Ligand

INTRODUCTION

Oximes are becoming increasingly important as analytical, biochemical and antimicrobial reagents and, in addition, they have received much attention due to their use as liquid crystals and dyes. The oximes of various types, such as α -dioximes, α -keto oximes, amino oximes, etc. generally form coloured soluble or very slightly soluble chelates with some transition metal salts, which can be used for different analytical purposes. Dioxime ligands and complexes have been extensively studied since these structural units are thought to be involved in a variety of biochemical and industrial processes (Karipcin and Arabali, 2006). The chemistry of cobaloxime complexes has gained attention since they are considered as better model complexes of vitamin-B₁₂. The 3d-metal dioximates have been continuously studied owing to numerous possibilities of using these compounds as models of physiologically important substances, in microbiology and medicine, as catalysts, etc. (Rijab *et al.*, 2013). Moreover, Nickel(II) and

palladium(II) complexes with α -dioxime ligands (dimethylglyoxime, diphenylglyoxime, and 1,2-cyclohexanedionedioxime) have been used as efficient catalysts for the polymerization of norbornene (Berchtold *et al.*, 2022). Vicinal dioxime derivatives act as amphoteric ligands due to the presence of weak acidic -OH groups and basic -C = N groups. As such, they are capable of forming highly stable complexes with most transition metals. The exceptional stability and electronic properties displayed by these complexes are attributable to their hydrogen bond-stabilized planar structures (Nagheu *et al.*, 2016). Furthermore, their high stability has been extensively exploited for various purposes including model compounds for vitamin B12, inhibitors of enzymes and intermediates in the biosynthesis of nitrogen oxides and model compounds for trace metal analysis (Nogheu *et al.*, 2017). Most of studies reported on cobaloxime complexes have been carried out using dimethylglyoxime (Hdmg). Martin and Dayalan, (2009) described cobaloximes of the types Trans-

$\text{BH}_2[\text{Co}(\text{dmgH})_2\text{X}]$, where $\text{dmgH}^- =$ Dimethylglyoximate, $\text{X}^- = \text{Cl}^-$ or Br^- and $\text{Trans-}[\text{Co}(\text{dmgH})_2(\text{H}_2\text{O})]\text{B}$ where, $\text{B} =$ Piperazine (Pp) or 2-(N-Tert-butylamido)piperazine (t-Bu-PpAM), showing superior activity against some pathogens. Boopalan *et al.* (2022) reported series of inorganic cobaloximes of the type $[\text{Co}(\text{X})(\text{L})_2\text{B}]$, where $\text{L} =$ Dimethylglyoxime, $\text{X} = \text{Cl}^-$, Br^- , SCN^- , $\text{B} =$ p-aminophenylacetic acid or p-aminobenzoic acid

with efficient antimicrobial activities. However, the chemistry of cobaloximes with other dioximes are less explored (Sowmya *et al.*, 2023).

Herein, we present the synthesis and antimicrobial study of cobaloximes complexes of diphenylglyoxime with isothiocyanate, aniline and its derivatives as ligands (Figure 1).

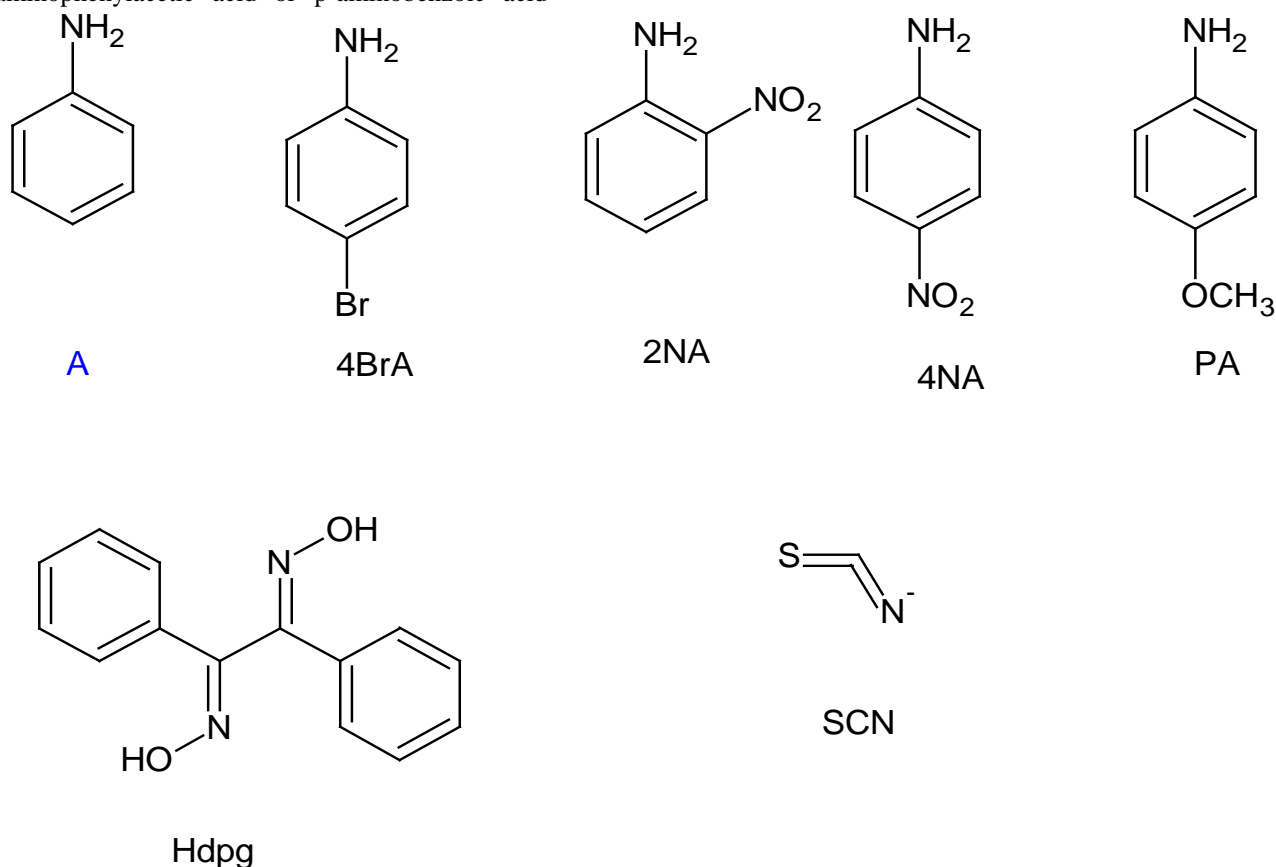


Figure 1: Structural formulae of the ligands: Aniline (A), 4-bromoaniline (4BrA), 2-nitroaniline (2NA), 4-nitroaniline (4NA), 4-anisidine (PA), diphenylglyoxime (Hdpg) and isocyanate (SCN)

MATERIALS AND METHODS

Chemicals of analytical grade were used as supplied including cobalt(II) acetate tetrahydrate, dimethylglyoxime, diphenylglyoxime, aniline, 4-bromoaniline, 2-nitroaniline, 4-nitroaniline, 4-anisidine, chloroform, benzene, ethanol, and methanol.

The infrared (IR) spectra were recorded on FTIR-8400S model in the range $450 - 4500 \text{ cm}^{-1}$ at National Research Institute for Chemical Technology (NARICT), Federal Ministry of Science and Technology, Zaria. Conductivity was measured using DDS-307 conductivity meter at 35°C , at Soil Laboratory, Department of Soil Sciences, University of Maiduguri. Melting points were determined on a Gallenkamp G112 melting point apparatus at the Laboratory of Department of Chemistry, Kashim Ibrahim College of Education, Maiduguri, Borno State. The percentage (%) of

Cobalt content was determined by titrating a digested solution of the metal complexes against a standardized EDTA (Vogel, 2000).

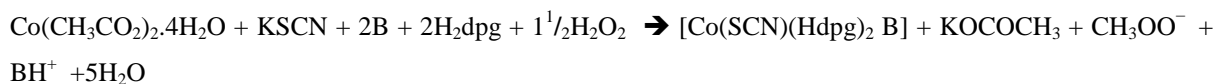
Synthesis of Diphenylglyoxime Complexes

Cobalt(III) acetate tetrahydrate (3 mmol, 0.7472 g) and KSCN (3 mmol) were dissolved in distilled water (30 mL) in a 250 mL quickfit conical flask. Diphenylglyoxime (6 mmol 0.6967 g) was dissolved in methanol (50 mL). The solution obtained from Hdpg was added to the mixture of the acetate and KSCN. The colour of the resulting mixture changed from pink to dark brown. The reaction mixture was refluxed and stirred for 3 hours after which it was cooled to room temperature. To the mixture, aniline or its derivatives (6 mmol) was added after which 30 % H_2O_2 was added and stirred at room temperature for 6 hours to effect the oxidation of Co(II) to

Co(III). The product obtained was filtered and washed with a suitable solvent and dried in a desiccator over calcium chloride (Ndahi and Kolawole, 2005; Salih and Satyanarayana, 2009).

The same procedure was applied to each Hdpg complex prepared.

A general equation for the reaction is presented as follows:



Where B = Aniline or Aniline derivatives and H₂dpg = Diphenylglyoxime

RESULTS AND DISCUSSION

Physical Properties of the Ligands and their Cobalt(III) Complexes

Table 1 shows the physical properties and analytical data of the synthesized metal complexes. The ligands on interaction with Co(II) ion formed complexes with low yields (18 – 25 %). This quite low percentage yield might be due to difficulties in optimizing all the reaction conditions such as temperature and solution concentration (Ahmed *et al.*, 2021). Nevertheless, [Co(Hdpg)₂(PA)(SCN)] was produced in good yield (59 %). All the complexes were air stable and have sharp melting

points in the range 70-118 °C. This indicates that the complexes are pure (Sunil *et al.*, 2011; Ahmed and Galtima, 2020). The measured molar conductance showed that the complexes are non-electrolytes, except [Co(Hdpg)₂(PA)(SCN)] which have 135 Ω⁻¹cm²mol⁻¹. The complexes were variously coloured, a characteristic known for transition metal complexes (Ahmed *et al.*, 2021). The solubility of the complexes tested in polar and non-polar solvents indicates that most of the complexes are either soluble or slightly soluble in all the solvents used (Table 2).

Table 1: Physical Properties of the Complexes

Compound	Molecular formul (molar mass)	Colour	Yield g(%)	Metal(%) Calc (Found)	M.P (°C)	Conductivity Ω ⁻¹ cm ² mol ⁻¹
[Co(Hdpg) ₂ (A)(SCN)]	CoC ₃₅ H ₃₁ N ₆ O ₄ S (690.7)	Brown	3.9 (21)	8.53 (8.45)	80 – 82	54
[Co(Hdpg) ₂ (4BrA)(SCN)]	CoC ₃₅ H ₃₀ N ₆ O ₄ BrS (769.5)	Brown	5.13 (25)	7.66 (7.66)	100 - 102	41
[Co(Hdpg) ₂ (2NA)(SCN)]	CoC ₃₅ H ₃₀ N ₇ O ₆ S (735.7)	Brown	3.5 (18)	8.01 (8.05)	118 - 110	21
[Co(Hdpg) ₂ (4NA)(SCN)]	CoC ₃₅ H ₃₀ N ₇ O ₆ S (735.7)	Red	4.5 (23)	8.01 (7.86)	70 – 72	36
[Co(Hdpg) ₂ (PA)(SCN)]CH ₃ COO ⁻	CoC ₃₈ H ₃₆ N ₆ O ₇ S (780.7)	Red	11.3 (59)	8.18 (8.25)	90 – 91	135

Table 2: Solubility of the Complexes

Compound	Distilled water		Methanol		Ethanol		Chloro-Form		Methyl acetate		Petroleum ether		Benzene	
	C	H	C	H	C	H	C	H	C	H	C	H	C	H
[Co(Hdpg) ₂ (A)(SCN)]	I	SS	S	VS	S	VS	SS	S	S	VS	SS	SS	SS	SS
[Co(Hdpg) ₂ (4BrA)(SCN)]	I	I	VS	VS	VS	VS	VS	VS	VS	VS	SS	SS	SS	SS
[Co(Hdpg) ₂ (2NA)(SCN)]	I	SS	VS	VS	VS	VS	S	VS	S	VS	I	I	SS	SS
[Co(Hdpg) ₂ (4NA)(SCN)]	I	I	VS	VS	VS	VS	S	VS	S	VS	SS	SS	S	S
[Co(Hdpg) ₂ (PA)(SCN)]CH ₃ COO ⁻	I	SS	VS	VS	VS	VS	S	VS	S	VS	SS	SS	SS	SS

I =insoluble, VS = very soluble, SS = slightly soluble, S = soluble, C= cold, H = hot

Infrared Spectra of the Metal Complexes

The selected vibrational frequencies of the ligand and complexes are presented in Table 3 and the infrared spectra shown in figures 2 - 7. The tentative assignment of the observed bands for the compounds were made by comparing the spectra of the ligands with their metal complexes and also with similar systems in the literature (Joshi *et al.*, 2006). The peak at 1647 cm⁻¹ in the diphenylglyoxime (H₂dpg) ligand is attributed to ν(C=N) (Ndahi and Kolewole, 2005). This band suffered shift to lower frequencies (1490 – 1637

cm⁻¹) indicating coordination of the ligand to the central metal ion. The peak at 980 cm⁻¹ attributed to ν(N-O) in the ligand (diphenylglyoxime) shifted to higher wave number (1197 – 1254 cm⁻¹) which was due to the increase in electron density on the Co ion supplied by the axial ligand (Rijab *et al.*, 2013; Fan *et al.*, 2015). This facilitated the back donation from Co to the nitrogen atoms of the ligands resulting in the increase in electron densities in the C=N band and have increased strength of the N-O bands (Ndahi and Kolawole, 2005). The ν(Co-N) band appeared at 421 – 578 cm⁻¹ in most of the

complexes (Rijabet *et al.*, 2013). A band at 401 - 538 cm^{-1} due to $\nu(\text{Co-S})$ was also found in the diphenylglyoxime complexes (Selviet *et al.*, 2011). The band due to $\nu(\text{NCS})$ were observed at 2114 – 2175 cm^{-1} . The peak due to $\nu(\text{OH})$ which appeared at 3308 cm^{-1} in the ligand shifted to higher and lower wavenumbers (3224 – 3435 cm^{-1}) in the complexes with probable overlap with that of $\nu(\text{N-}$

H) vibration and water of hydration which could not be accounted for as no microanalysis of CHN was conducted. However, it is evident that the dioximes are coordinated to the central atom. On the basis of the metal analysis, infrared studies and information from the literature, an octahedral geometry is proposed for the complexes (Figure 8).

Table 3: Relevant bands for the ligands and their cobalt(III) complexes (cm^{-1})

Compound	V(OH)	V(C-N)	V(C=N)	V(N-H)	V(N-O)	V(N=O)	V(Co-N)	V(NCS)
Hdpg	3308	-	1647	-	980	-	-	-
[Co(Hdpg) ₂ (A)(SCN)]	3435	990	1637	3435	1197	1424	556,421	2117
[Co(Hdpg) ₂ (4BrA)(SCN)]	3266	988	1490	3266	1209	1405	712	2114
[Co(Hdpg) ₂ (2NA)(SCN)]	3224	1074	1610	3293	1254	1498	578,492	2175
[Co(Hdpg) ₂ (4NA)(SCN)]	3266	987	1492	3266	1204	1411	713,558	2171
[Co(Hdpg) ₂ (PA)(SCN)]CH ₃ COO ⁻	3255	1031	1619	3433	1249	1497	542,426	2113

Hdpg = Diphenylglyoxime, 4NA = 4-Nitroaniline, PA = 4- nitroaniline

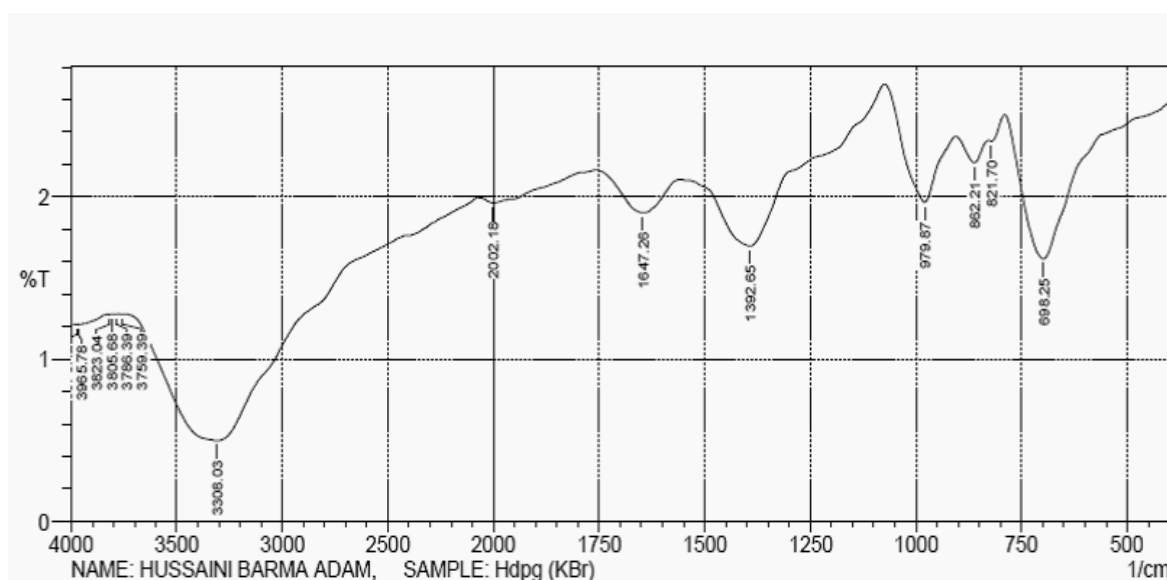


Figure 2: FT-IR spectrum of Diphenylglyoxime

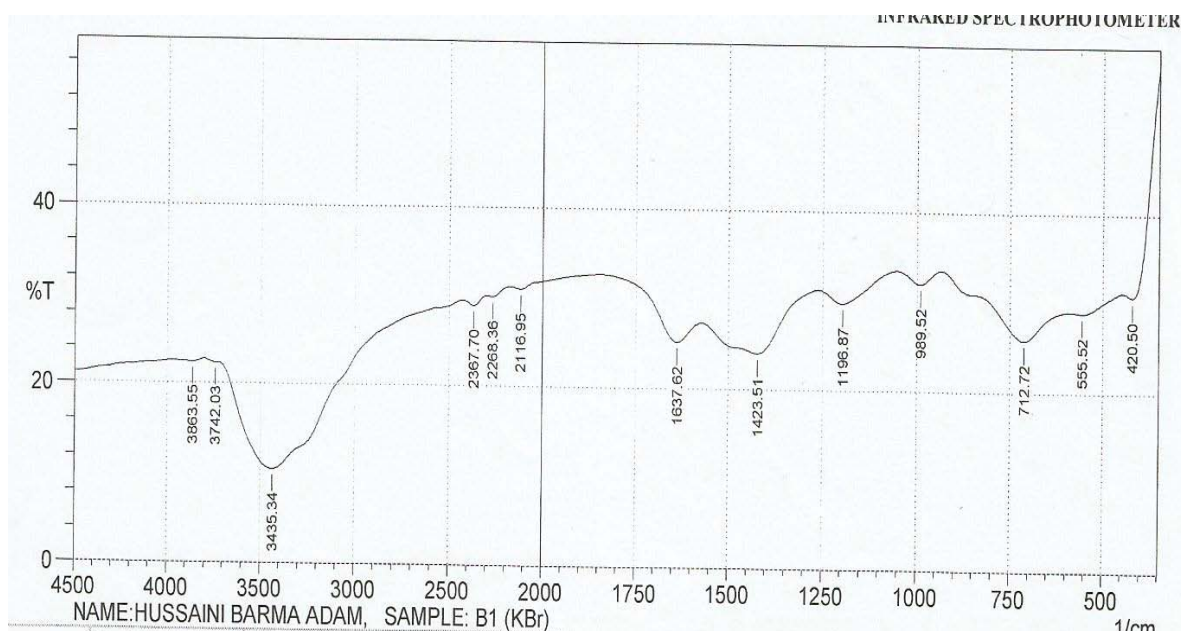


Figure 3: FT-IR spectrum of [Co(Hdpg)₂(A)(SCN)]

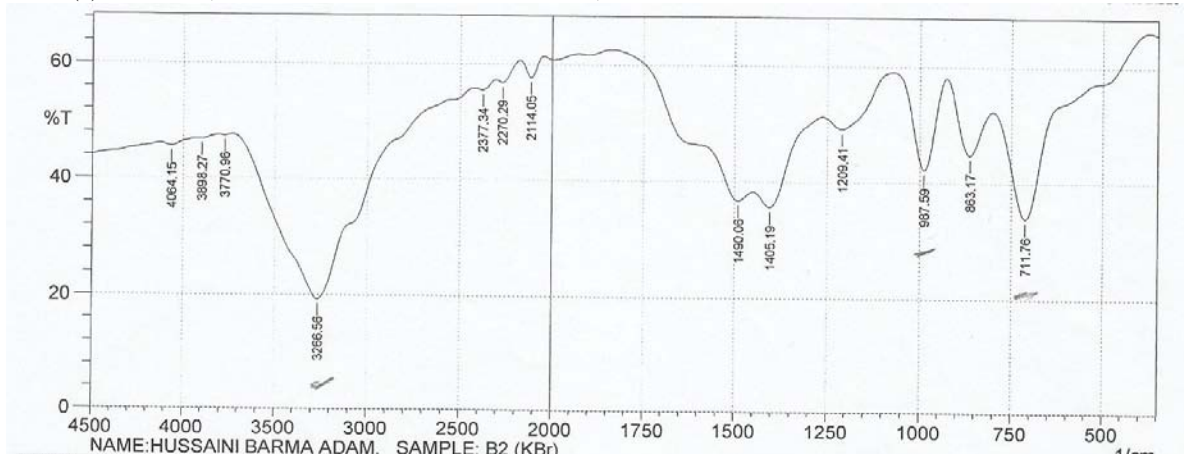


Figure 4: FT-IR spectrum of [Co(Hdpg)₂(4BrA)(SCN)]

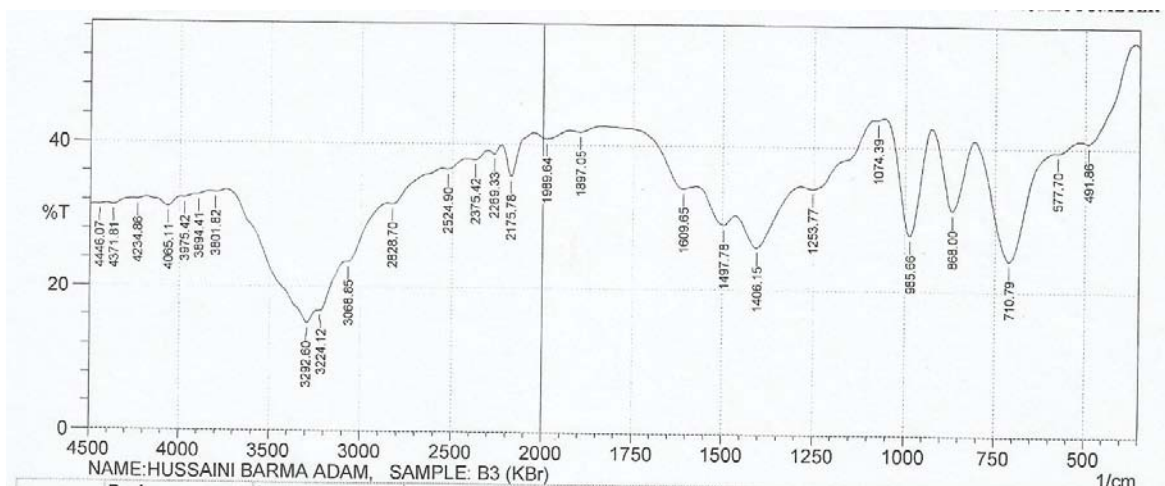


Figure 5: FT-IR spectrum of [Co(Hdpg)₂(2NA)(SCN)]

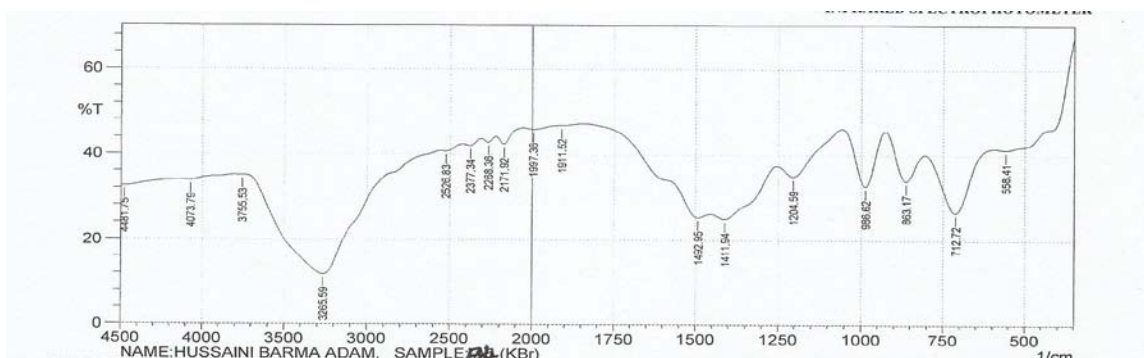


Figure 6: FT-IR spectrum of [Co(Hdpg)₂(4NA)(SCN)]

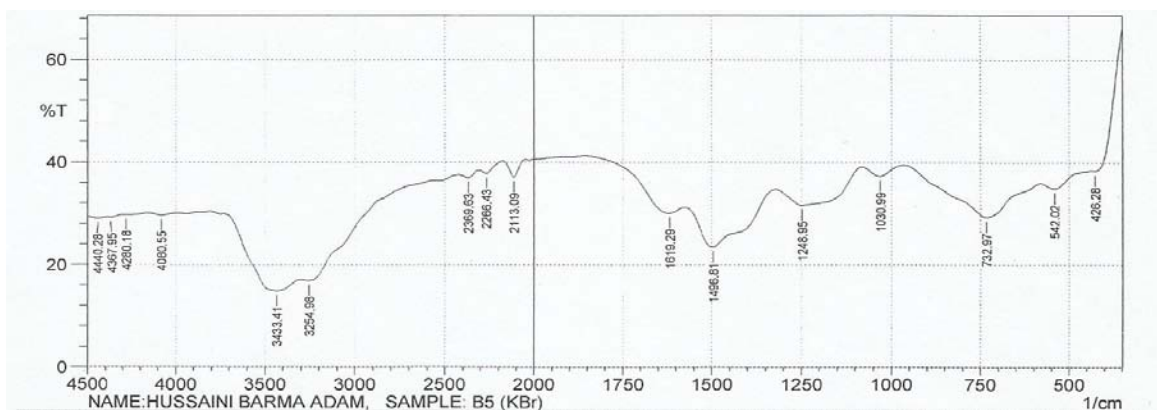


Figure 7: FT-IR spectrum of [Co(Hdpg)₂(PA)(SCN)]CH₃COO

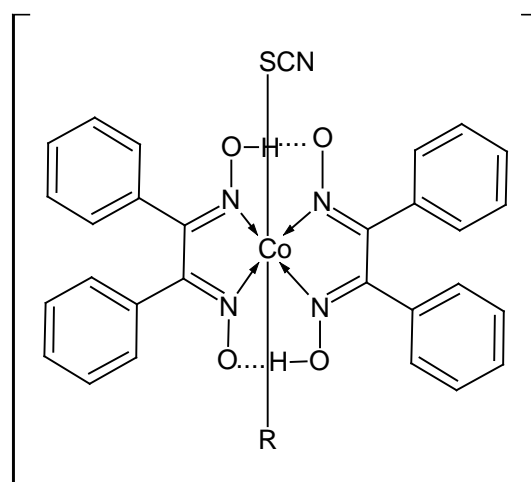
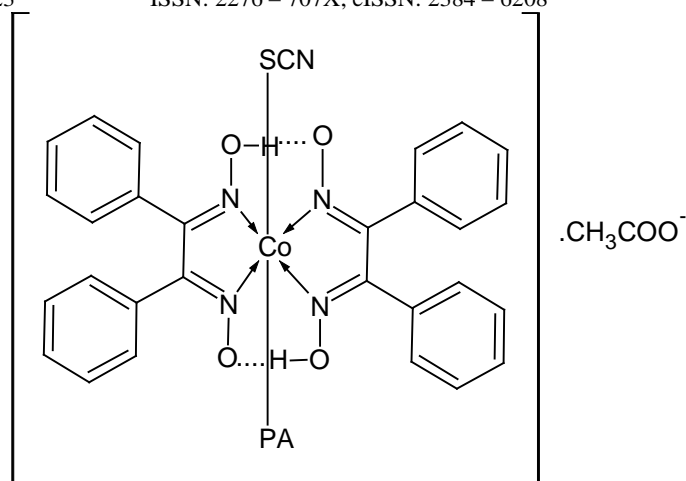


Figure 8: Proposed structure of (a) $[\text{Co}(\text{Hdpg})_2(\text{PA})(\text{SCN})]$, PA= 4-anisidine (b) $[\text{Co}(\text{Hdpg})_2(\text{R})(\text{SCN})]$, R= aniline, 4-bromoaniline, 2-nitroaniline and 4-nitroaniline

Antimicrobial Studies

The metal complexes show no activity against all tested microbe except in case of *Staphylococcus aureus* and *Salmonella typhi* whose inhibition zone ranges from 7 – 10mm. the results are presented in Table 4.

Table 4: Antimicrobial activities of the ligands and their Complexes

Compounds	Zone of inhibition (mm)								
	Conc. (µg/ml)	MRSA	<i>S. aureus</i>	<i>S. pneumonia</i>	<i>B. subtilis</i>	<i>S. typhi</i>	<i>E. coli</i>	<i>K. pneumonia</i>	<i>P. aeruginosa</i>
A	30	15.33±0.58	14.00±0.00	15.00±0.00	12.00±0.00	13.67±0.58	11.67±0.58	10.00±0.00	11.67±0.58
	20	12.33±0.58	11.00±0.00	11.33±0.58	9.00±0.00	10.67±0.58	8.67±0.58	8.67±0.58	9.00±0.00
	10	8.00±0.00	8.00±0.00	8.33±0.00	7.00±0.00	8.33±0.58	7.00±0.00	NZI	7.00±0.00
PA	30	10.33±0.58	12.00±0.00	15.00±0.00	11.67±0.58	13.67±0.58	12.00±0.00	10.00±0.00	11.67±0.58
	20	7.33±0.58	9.00±0.00	11.67±0.58	9.00±0.00	11.00±0.00	9.00±0.00	7.67±0.58	9.00±0.00
	10	NZI	7.00±0.00	9.00±0.00	7.00±0.00	8.33±0.58	7.33±0.00	NZI	7.00±0.00
Hdpg	30	NZI	NZI	NZI	NZI	NZI	NZI	NZI	NZI
	20	NZI	NZI	NZI	NZI	NZI	NZI	NZI	NZI
	10	NZI	NZI	NZI	NZI	NZI	NZI	NZI	NZI
[Co(Hdmg) ₂ (A)(SCN)]	30	NZI	NZI	NZI	NZI	12.00±0.00	NZI	NZI	9.67±0.58
	20	NZI	NZI	NZI	NZI	8.67±0.058	NZI	NZI	7.00±0.00
	10	NZI	NZI	NZI	NZI	NZI	NZI	NZI	NZI
[Co(Hdmg) ₂ (4BrA)(SCN)]	30	NZI	NZI	NZI	NZI	NZI	11.67±0.58	NZI	9.67±0.58
	20	NZI	NZI	NZI	NZI	NZI	8.33±0.58	NZI	7.00±0.00
	10	NZI	NZI	NZI	NZI	NZI	NZI	NZI	NZI
[Co(Hdmg) ₂ (2NA)(SCN)]	30	NZI	10.67±0.58	NZI	11.00±0.00	13.00±0.00	11.33±0.58	NZI	NZI
	20	NZI	7.67±0.58	NZI	8.00±0.00	9.00±0.00	8.00±0.00	NZI	NZI
	10	NZI	NZI	NZI	NZI	7.00±0.00	NZI	NZI	NZI
[Co(Hdmg) ₂ (4NA)(SCN)]	30	NZI	10.67±0.58	NZI	11.00±0.00	13.00±0.00	NZI	NZI	NZI
	20	NZI	7.67±0.58	NZI	8.00±0.00	9.00±0.00	NZI	NZI	NZI
	10	NZI	NZI	NZI	NZI	7.00±0.00	NZI	NZI	NZI
[Co(Hdmg) ₂ (PA)(SCN)]	30	NZI	10.00±0.58	10.00±0.00	NZI	10.00±0.00	NZI	NZI	NZI
	20	NZI	7.00±0.00	7.00±0.00	NZI	7.33±0.58	NZI	NZI	NZI
	10	NZI	NZI	NZI	NZI	NZI	NZI	NZI	NZI
Gentamycine	30	15.00±0.00	15.00±0.00	15.00±0.00	15.00±0.00	15.00±0.00	15.00±0.00	15.00±0.00	15.00±0.00

Hdpg = diphenylglyoxime, A= aniline, PA= 4-nitroaniline, NZI= No zone of inhibition, *S. aureus* = *Staphylococcus aureus*, *S. pneumonia* = *Streptococcus pneumonia*, *B. subtilis* = *Bacillus subtilis*, *S. typhi*= *Salmonella typhi*, *E. coli* = *Escherichia coli*, *K. pneumonia* = *Klebsiella pneumonia* *P. aeruginosa* = *Pseudomonas aeruginosa*

CONCLUSION

The synthesis of the glyoximate complexes using aniline and its derivatives have been described. The complexes were characterized using melting point, conductivity and infrared spectroscopy. The complexes gave sharp melting points with low molar conductivity value in most instances hence they are non-electrolytes except $[\text{Co}(\text{Hdpg})_2(\text{PA})(\text{SCN})]\text{CH}_3\text{COO}^-$. Infrared studies indicate that the ligands coordinated through the nitrogen atom of the glyoxime group to the metal ions. The results of the antimicrobial study showed that the complexes were potent as the ligands against the microorganisms used.

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