

PREPARATION, CHARACTERIZATION, AND STUDY OF THE BIOLOGICAL AND THERMAL EFFECTS OF SOME DIVALENT METAL COMPLEXES WITH A MIXTURE OF 4-BROMO-N-(2-OXINDOLE-3-YLIDENE) BENZO HYDRAZINE LIGANDS AND PHOSPHINES

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Abstract: A few nickels (II) complexes with the ligand Bromo-N'-(2-oxindole -3-ylidene)benzo hydrazide) (BOB) are prepared and characterized as part of this study. A dinuclear complex with a tetrahedral shape and the formula $[M_2(BOB)_2\mu-(dppm)]Cl_4$ is produced when one mole of $NiCl_2 \cdot 6H_2O$ reacts with an equivalent mole of (BOB) and an equivalent mole of dppm. In this complex, the ligand (BOB) acts as an identity through the two oxygen atoms, while the dppm acts as a bridging ligand.

The reaction between one mole of $NiCl_2 \cdot 6H_2O$ and an equivalent mole of either of the diphosphines (diphosphine = dppe, dppp, dppb) yields tetrahedral complexes with nickel with the formula $[M(BOB)(diphosphine)]Cl_2$, where the ligand (BOB) behaves as a dentate chelate through the oxygen atoms, and the diphosphines behave as identity chelates in all of their complexes. Other spectroscopic methods, including FT-IR and spectra from ^{13}C -NMR, 1H -NMR, and ^{31}P -NMR, were used to assess and describe the molar conductivity and biological activity of the produced complexes.

Keywords: Isatin, Schiff base, Biological activity.

Introduction

Isatin The development of recent and cost-effective drugs against malaria, tuberculosis, AIDS, and trypanosomiasis is urgently required as a good way to satisfy the overwhelming call for the remedy of diseases in third international countries [1]. The formation of the latest medicinal compounds primarily based on candidate molecules with recognized organic pastimes provides a possibility to lay out safe and effective drugs with minimum toxicity [2]. It is a chemical building block, capable of forming a huge number of heterocyclic molecules. Isatin has an indole ring structure (Fig. 1), that's typically used in many pharmaceutical preparations. Isatin has a wide range of biological sports [3]. Isatin derivatives exhibit diverse organic activities which include antibacterial, antifungal, antiviral, anti-HIV, antifungal, anticancer, and anti-inflammatory activities [4] along with the nature of the binding at position 2 or three of the indole nucleus of isatin significantly regulates its anti-inflammatory and antibacterial sports.]9-5[



Figure 1: Isatin compound

Isatin and its derivatives have biological importance as it has anti-HIV activity [10] and is a protective factor to prevent cancer and in chemotherapy to kill cancer cells

[11] and is used as an antibacterial [12,13]. (Atioğlu) and his group prepared the compound 2-(5-fluoro-1-methyl-2-oxoindolin-3-ylidene)-N-(3-fluorophenyl)hydrazine-1-carbothioamide and it was studied by X-ray diffraction as shown in Figure 2 where it showed anti-inflammatory and antifungal activity[14].

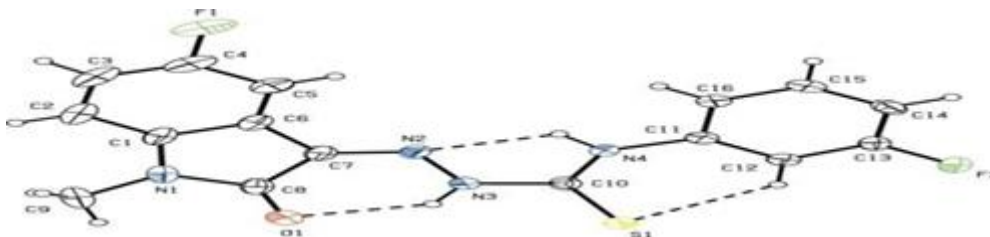


Figure 2. Crystal structure of the ligand

Schiff base The azomethine group C=N-R R₂, which is created when the nitrogen atom in the primary amine combines with the carbonyl group in different aldehydes or ketones, is known as a Schiff base. This organic compound is typically created by increasing the aldehyde or ketone with the primary amine by the following reaction scheme.



where R may be either an aryl or an alkyl group. Compared to Schiff bases with alkyl substituents, those with aryl substituents are more stable and form more quickly. Furthermore, aromatic aldehydes with good electron exchange are more stable than aliphatic aldehydes, whose bases are unstable and available for polymerization. Aldehydes or ketones can react to generate a Schiff base, which is a reversible process that often happens in the presence of heat or an acidic or basic catalyst [15].

Schiff bases are regarded as the foundation for the synthesis of many heterocyclic compounds and their coordination complexes, as well as high molecular weight polymers, making these molecules extremely significant in several domains [16]. Numerous investigations have demonstrated the diverse biological properties of Schiff bases, such as their antiviral, antifungal, and antibacterial properties [17]. In addition to their significance in the pharmaceutical and medical industries, they are also helpful in suppressing a variety of malignant tumors. It has been demonstrated that this biological activity present on Schiff bases is caused by the azomethine bond. **Experimental part**

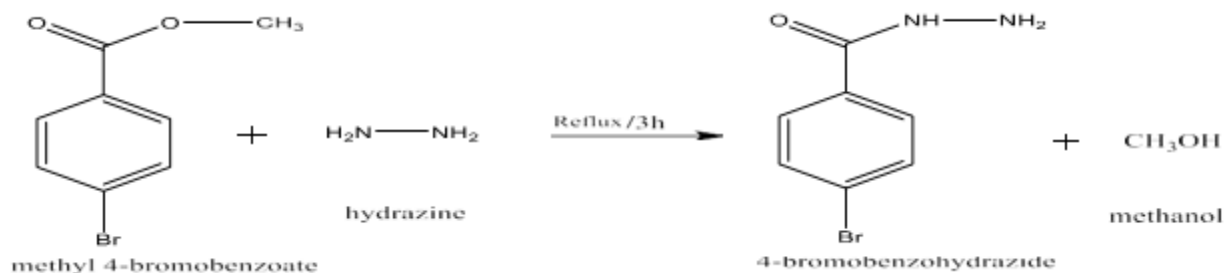
Preparation of the ligand

The ligand was prepared in two steps:

a- Preparation of benzoyl hydrazine:

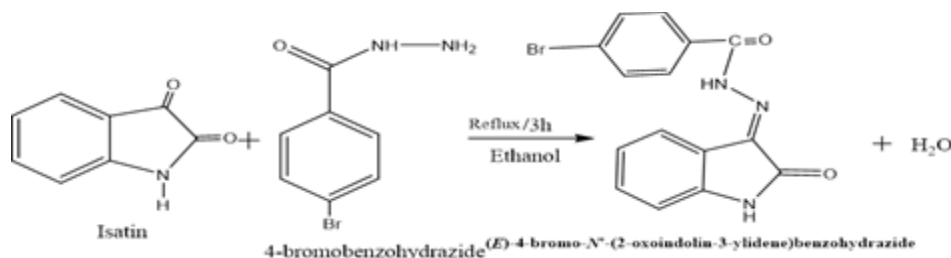
(0.010mol, 0.416g) of hydrazine was added to (0.013mol, 4.000g) of 4-methyl bromobenzoate directly, then the mixture was placed on sublimation for three hours,

then filtered and the mixture solution was left to cool, where a white precipitate (85.04%, 2.377g) was obtained.



b- Preparation of Schiff's base from isatin:-

A solution of benzoyl hydrazine (0.000393mol, 0.2026g) in (10ml) of absolute ethanol was added to a suspension of isatin (20.39mol, 0.138g) in (10ml) of absolute ethanol to which drops of glacial acetic acid were added, then the mixture was sublimated. After 15 minutes, the color of the mixture changed from orange to yellow. It was left to sublimate for three hours, then filtered and washed with cold ethanol, then dried in an oven under vacuum pressure. Melting point (287- 288) °C. (0.278g, 86.06%),



Synthesis of complexes

1. Preparation of the complex [Ni(L)dppe]Cl₂

A solution of NiCl₂.6H₂O ((0.138 gm, 0.00058 mol in (10 ml) of absolute ethanol was added to the suspension of the ligand L)) ((0.2 gm, 0.00058 mol). The mixture rose for an hour and a half, where the mixture became a yellow suspension, then (0.231 gm, 0.00058 mol) of dppe) was added to it. The final mixture rose for 3 hours, and a reddish-brown precipitate was formed. The precipitate was filtered and washed with cold ethanol, then dried under vacuum pressure (0.395 g, 78.5%). Melting point (253-255)°C.

2. Preparation of the complex [Ni(L)dppp]Cl₂

A solution of NiCl₂.6H₂O ((0.103 g, 0.00043 mol in (10 ml) of absolute ethanol was added to the suspension of the ligand L)) ((0.150 gm, 0.00043 mol). The mixture rose for an hour and a half, where the mixture became a yellow suspension. Then (0.177 gm, 0.00043 mol) of dppp) was added to it. The final mixture rose for 3 hours, and a brown precipitate was formed. The precipitate was filtered and washed with cold ethanol, then dried under vacuum pressure (0.305 gm, 80.2%) Melting point (249- 251)°C)

3. Preparation of the complex [Ni(L)(dppb)]Cl₂

The brown-colored precipitate, the weight of the product (0.511g), percentage (87.5%), melting point ((256-258)°C

4. Preparation of the complex [Ni(L)(dppm)]Cl₂

The dark brown precipitate, the weight of the product (0.311g), percentage 80.7%), melting point)259-263(°C

5. Preparation of the complex [Ni(L)dppf]Cl₂

Olive-colored precipitate, product weight (0.573g), percentage (90.6%), melting point (259-263°C)

Table 1. Magnetic measurement values of the prepared complexes measured at a temperature of (23°C)

Seq	Complexes	M.Wt (g/mol)	$\chi_g^* 10^{-6}$ (c.g.s.u)	$\chi_M^* 10^{-6}$ (c.g.s.u)	$D^* 10^{-6}$ (c.g.s.u)	$\chi_A^* 10^{-6}$ (c.g.s.u)	μ_{eff} (B.M.)
1-	[Ni(BOB)(dppe)]Cl ₂	871.02	0.91125	793.717	-)1936.64)	2730.357	2.54
2-	[Ni(BOB)(dppp)]Cl ₂	886.04	0.80977	717.4957	-(2412.02)	3129.515	2.7218
3-	[Ni(BOB)(dppb)]Cl ₂	900.08	0.80892	728.1008	-(2836.67)	3564.77	2.9049
4-	[Ni(BOB)(dppf)]Cl ₂	1017.99	3.87857	3948.345	- (574.04)	4522.385	3.272
5-	[Ni ₂ (BOB) ₂ μ -(dppm)]Cl ₄	856.99	1.218	1043.813	-)2187.06)	3230.873	2.7655

Biological activity study

Mueller-Hinton agar medium was prepared according to the instructions of the preparation company to study its biological activity. Dissolve the medium powder (38 g) in (1) liter of distilled water in a conical flask and heat the solution until dissolved [18-20]. After dissolving the culture medium, the solution is placed in an autoclave and sterilized for (15) minutes at a temperature of 121 °C and a pressure of (1.5 bar) until the culture medium cools and then is sterilized [21-23]. Pour into a dish and place until ripe. Place the bacteria in the culture medium, then use a cork drill to make holes in these Petri dishes, sterilize the holes with alcohol, and use a fine pipette to place the prepared solution into the holes. After placing (24) hours in an incubator at a temperature of 37 °C, the diameter of the inhibition zone of the compound under study is measured using a millimeter ruler [24-28]

Results and discussion

Characterization of Schiff's base

FT-IR (3027cm⁻¹), (3155cm⁻¹), (3031cm⁻¹), (1666cm⁻¹), (1618cm⁻¹), (1589cm⁻¹), (1267cm⁻¹),

¹H-NMR (13.93ppm(1.41ppm(NH)), (7.84ppm(He)), (7.62ppm(Ha))

, (7.41ppm(Hc)), (7.13ppm(Hb)), (6.97ppm(Hd)).

¹³C-{¹H}-NMR (♣C=165.07ppm), (C=O), (♣C=163.50ppm), (C=O), (♣C=144.54, 143.02ppm) (C=N+C5), (C8, C9) (♣C=133.50ppm, ♣C=132.06ppm) (C1, C4, Ph) (♣C=123.29, 121.54, 120.20ppm) (♣C=116.04, 111.75), ppm)

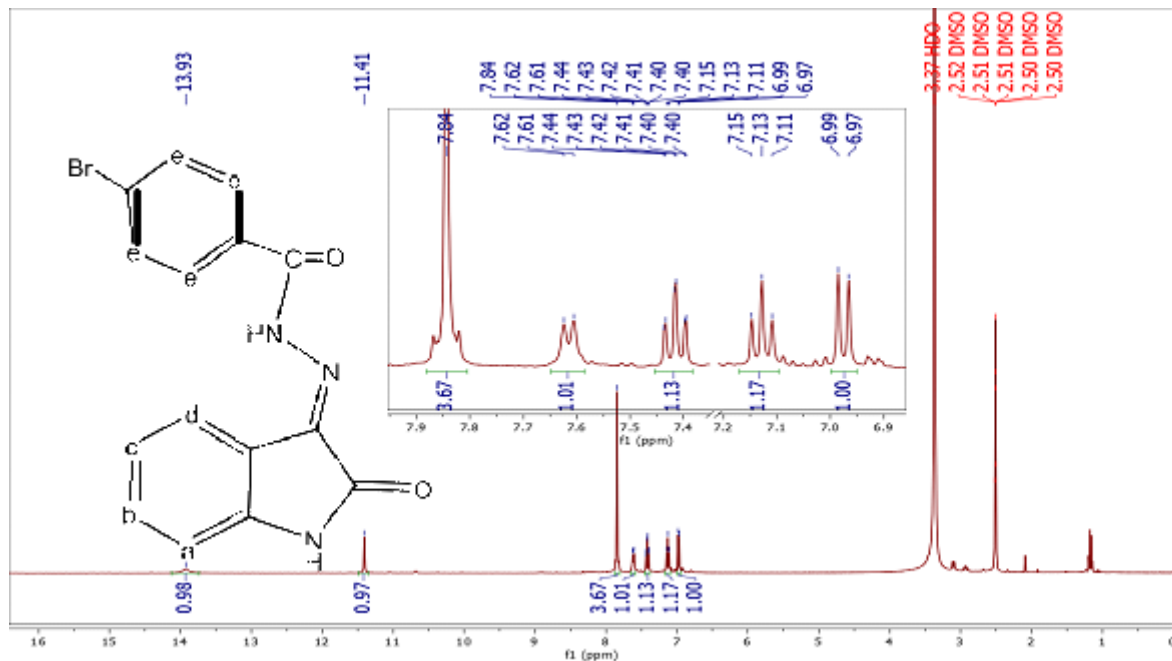


Figure 3: ¹H-NMR spectrum of the ligand [BOB]

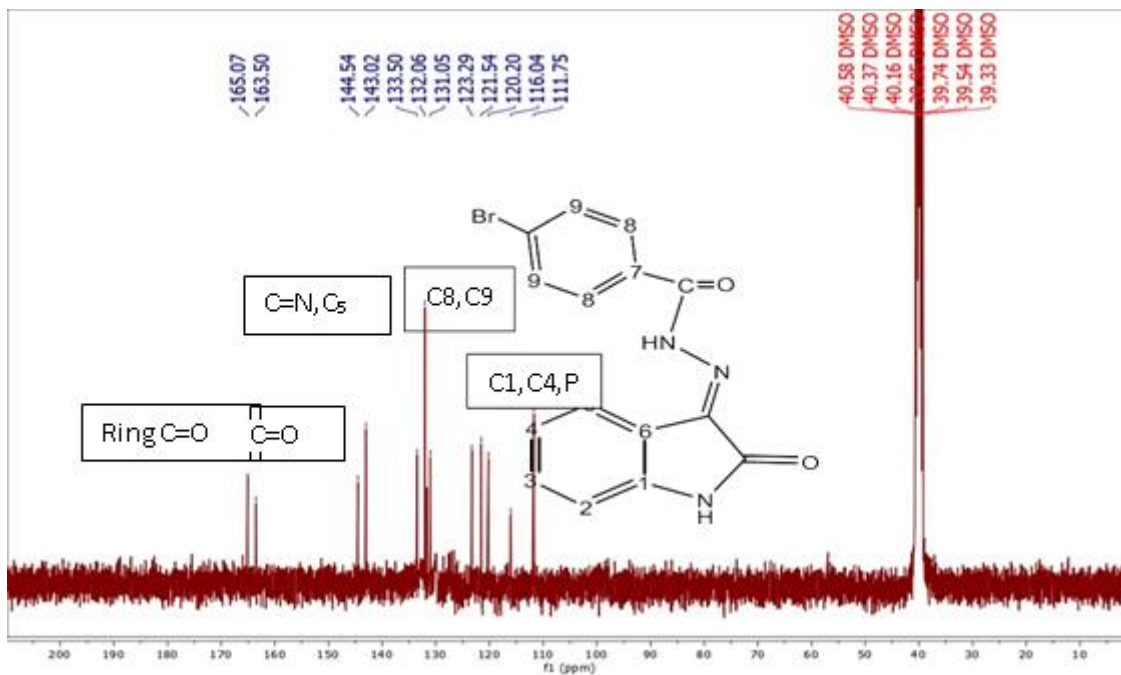


Figure 4: ¹³C-NMR spectrum of the ligand [BOB] Characterization of the ligand [Ni₂(BOB)₂μ-(dppm)₂]Cl₄

FT-IR (3309m),(3225m),(3196m),(3182m),(3024w),(3047w),(1664s),(1614s)
(1554m). CHN (C=39.10,H=3.28,Br=37.16,N=13.03,O=7.44),
¹H-NMR(9.90PPm)NH),(7.65,7.78ppm(Ha,Hb),(6.85ppm(NH₂)

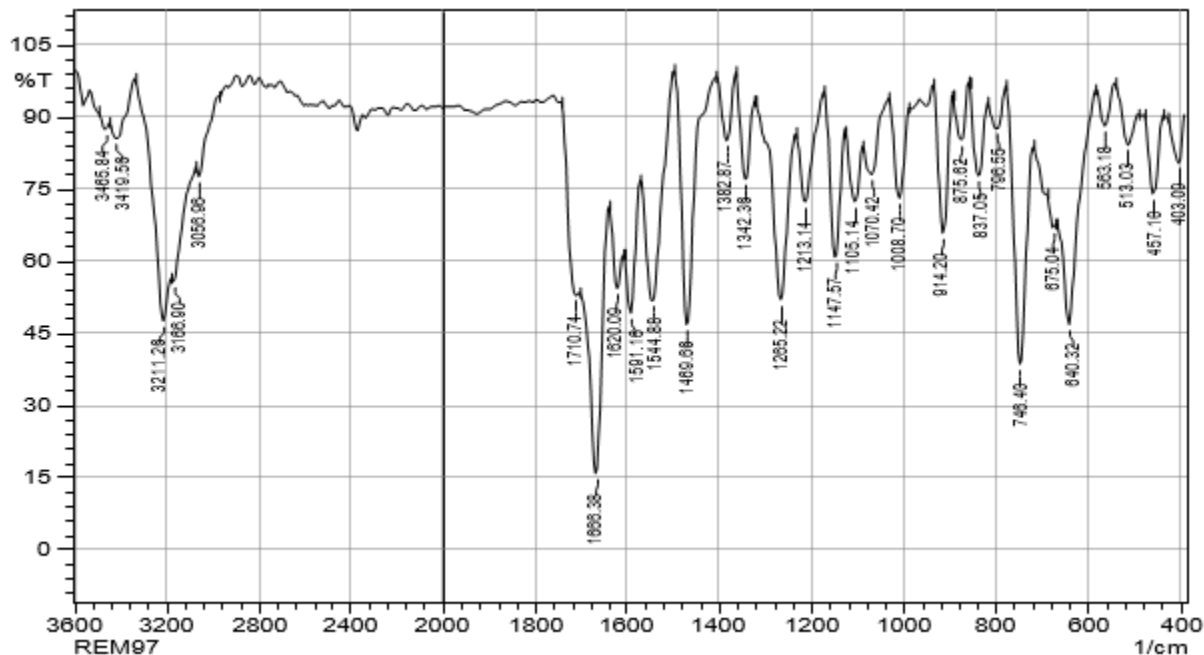


Figure 5. Infrared spectrum of the ligand $[Ni_2(BOB)_2\mu-(dppm)_2]Cl_4$

Characterization of the ligand $[Ni(BOB)(dppe)]Cl_2$

FTIR,(3174m),(3058w),(2368m)(1668s),(1546m)(1471),(1431w),(1097m),(694s),
(CHN((C=51.55,H=4.81,Br=12.70,N=6.68,Ni=9.33,o=5.09,p=9.85),magnetic
measurements(2.54B.M)

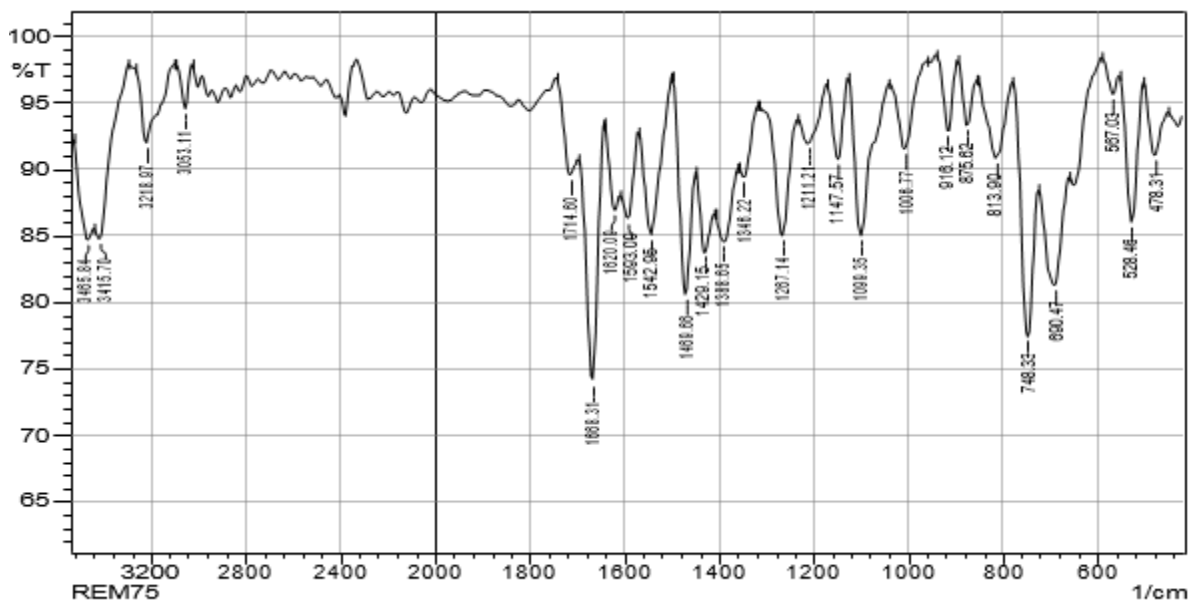


Figure 6. Infrared spectrum of the ligand $[Ni(BOB)(dppe)]Cl_2$

Characterization of the ligand $[Ni(BOB)(dppp)]Cl_2$

FT-IR (3215m),(3159m),(3053w),(1668s),(1616m),(1595m),(1413s), (1101m),(690s)

.CHN(C=80.50, H=8.78, O=10.72, Ni=7.37, Br=100.00, P=10.86)

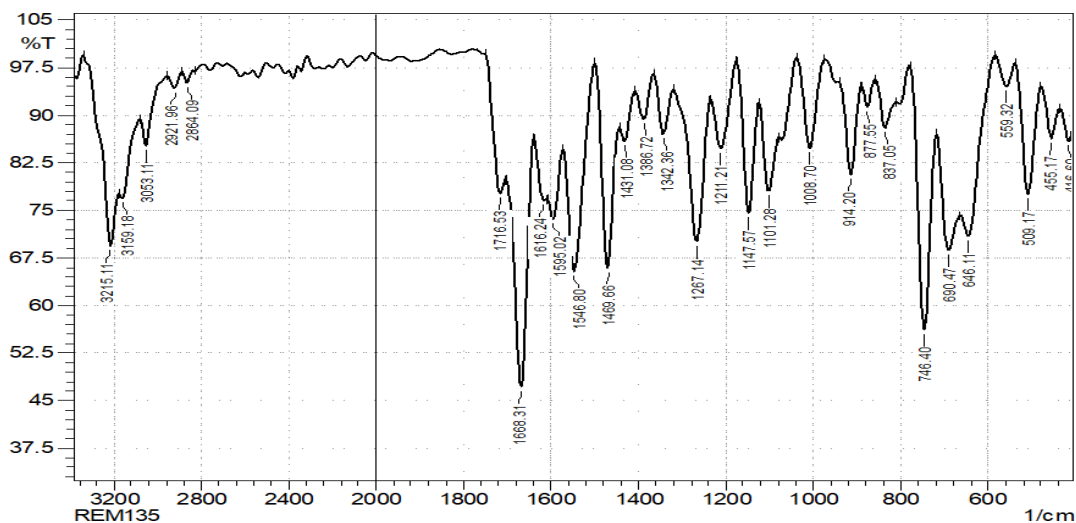


Figure 7. Infrared spectrum of the ligand [Ni(BOB)(dppp)]Cl₂ Characterization of the ligand [Ni(BOB)(dppe)]Cl₂

FT-IR(3211m),(3060m),(1666s),(1620w),(1593m),(1433m),(1103m), (690s).

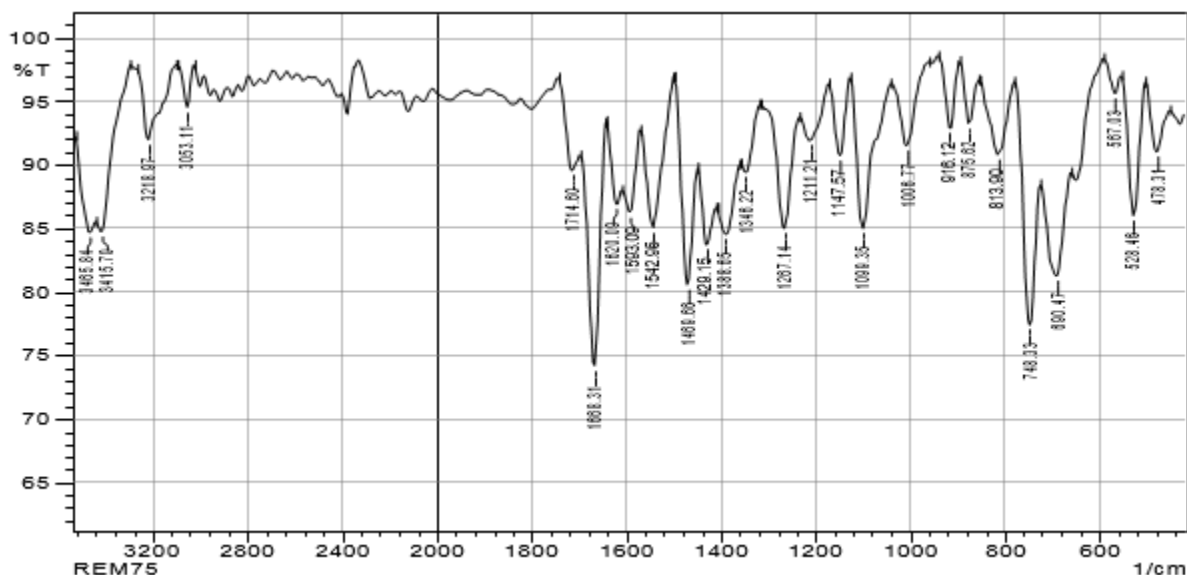


Figure 8. Infrared spectrum of the ligand [Ni(BOB)(dppe)]Cl₂ Characterization of the ligand [Ni(BOB)(dppb)]Cl₂

FT-IR (3215m),(3060m),(1664s),(1600m),(1546m),(1265m), (1103m)

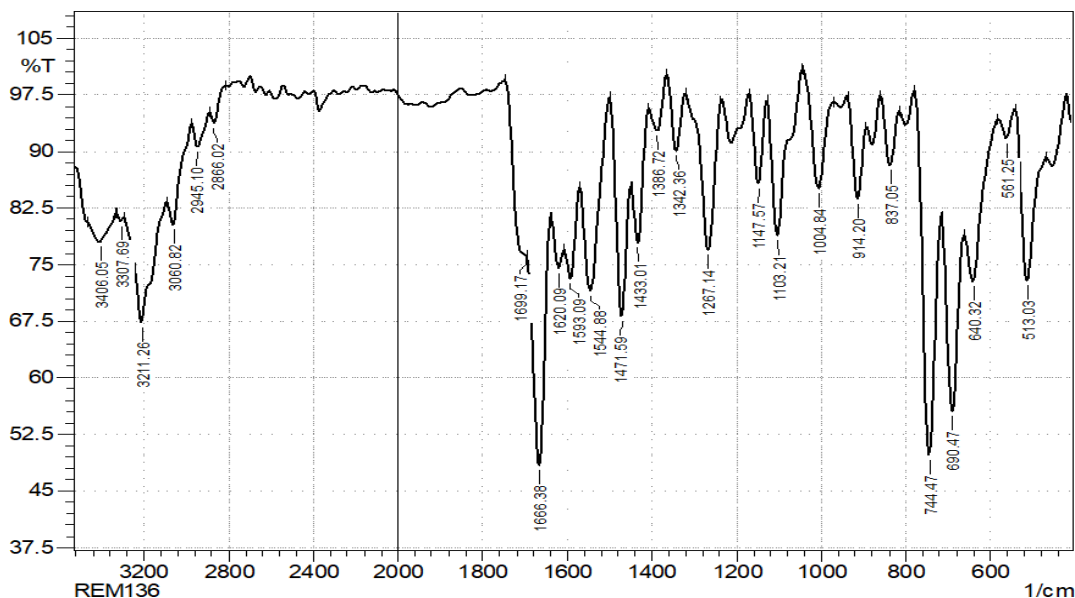


Figure 9. Infrared spectrum of the ligand $[Ni(BOB)(dppb)]Cl_2$ Characterization of the ligand $[Ni(L)dppf]Cl_2$

FT-IR (3213m),(3058m),(1668s),(1910w),(1429m),(1265m),(1091m),690m)

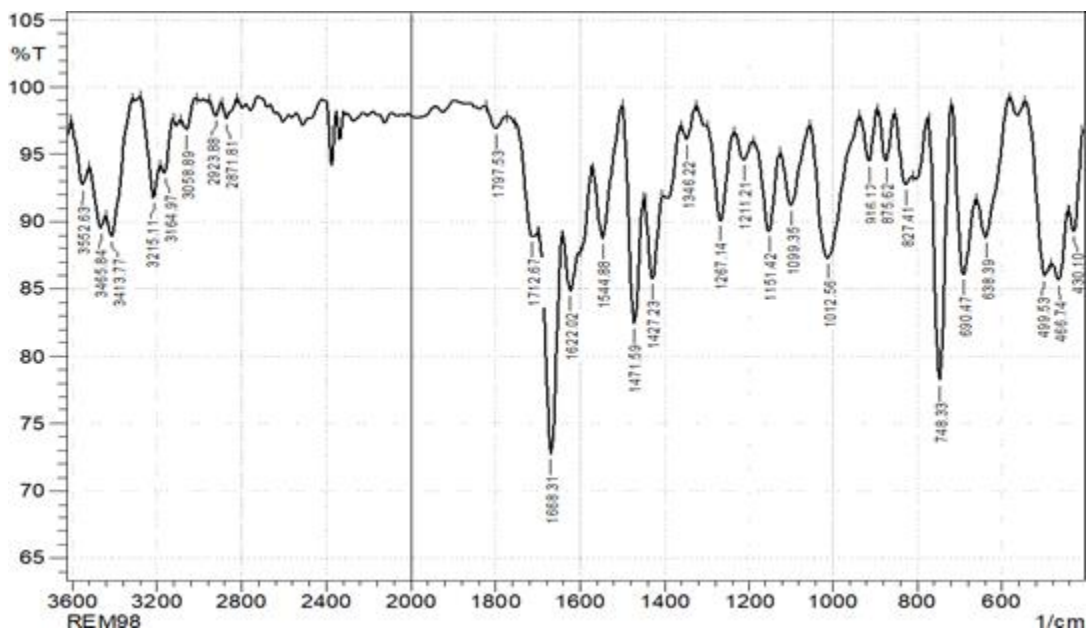
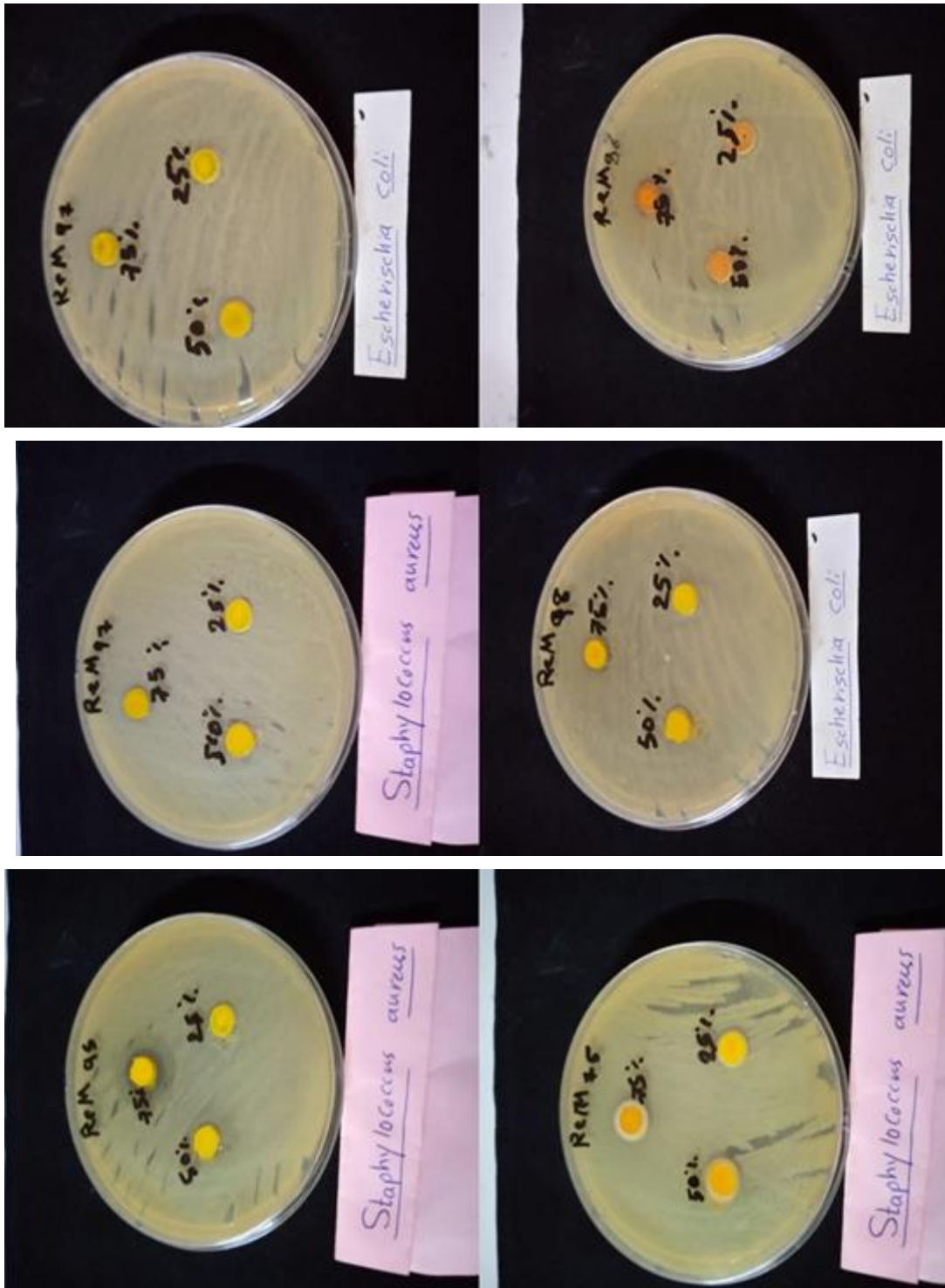


Figure 10. Infrared spectrum of the ligand $[Ni(L)dppf]Cl_2$

Biological activity evaluation

The complexes showed ineffectiveness towards the selected bacterial species at the lowest concentration (10-5g/mol), while the complex $[Pd_2(ITC)_2\mu-(dppm)]Cl_4$ showed the highest inhibition percentage of (32mm) at a concentration of (10-4g/mol), while the complex $[Ni(BOB)(dpe)]Cl_2$ showed the highest inhibition percentage of (36mm) at a concentration of (10-3g/mol) for Gram-negative bacteria[30-34]. The complexes showed ineffectiveness towards the selected bacterial species at the lowest concentration (10-5g/mol), while the complex $[Ni(BOB)(dppf)]Cl_2$ showed the highest inhibition rate of (25mm) at a

concentration of (10-4g/mol), while the complex [Pd(ITC)(Phen)]Cl₂ showed the highest inhibition rate of (36mm) at a concentration of (10-3g/mol) for Gram-positive bacteria[35-40].



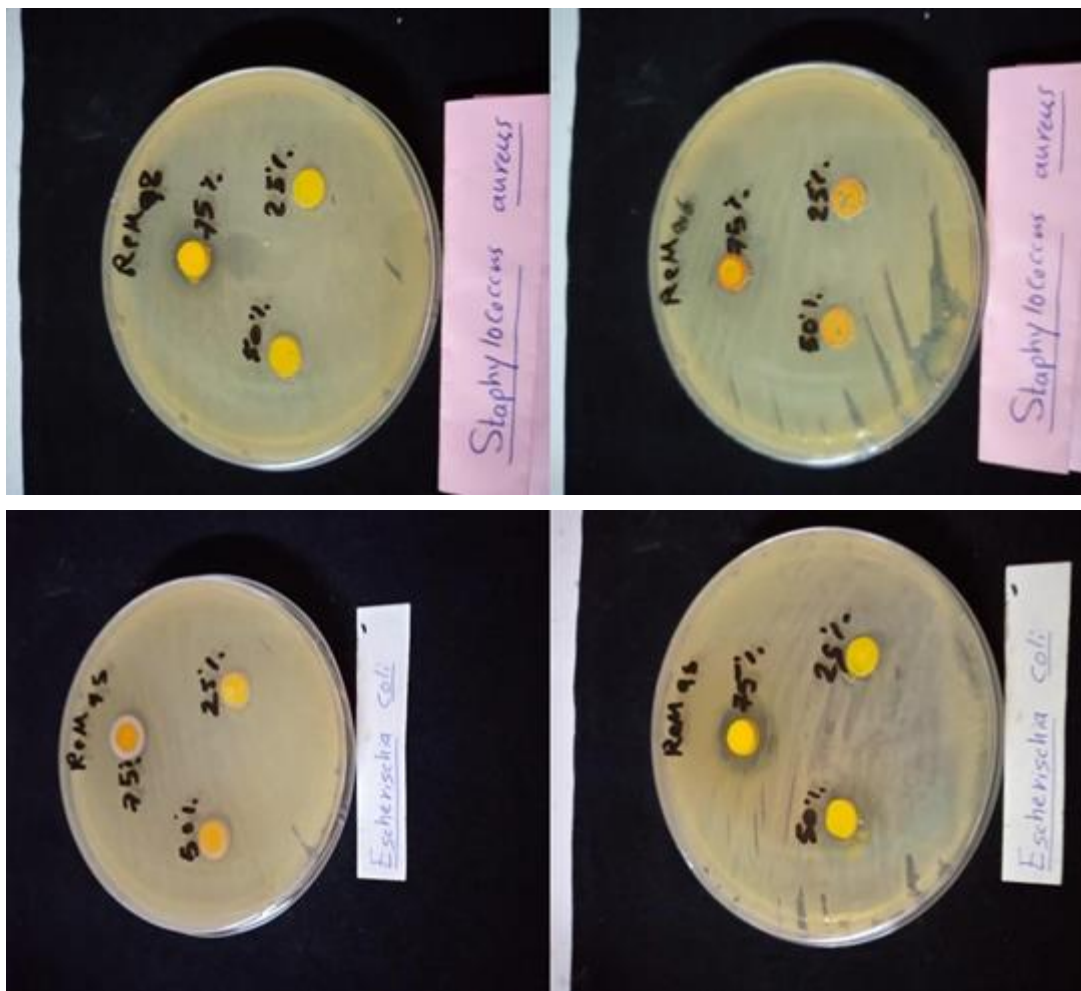


Figure 11: Biological activity images of the prepared complexes.

Conclusions:

The reaction of Schiff bases with certain chlorides or phosphines always gives complexes. The correctness of the ligand and complex structures was proven using spectroscopic measurements such as infrared spectroscopy. The sensitivity of the complexes was also measured and their biological activity against two types of Gram- positive and Gram-negative bacteria was proven.

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