World Journal of
Pharmacy and Medical Science

ISSN: 3049-3501
Impact Factor: 5.157
CODEN (USA): WJPMAA

Research Article
2026 Volume 2, Issue 11
Page No.: 100-105

SYNTHESIS, CHARACTERIZATION AND BIOLOGICAL STUDIES OF COBALT METAL COMPLEXE DERIVED FROM IMINO-PYRAZOL SCHIFF BASE

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Article Received: 07 October 2026 Article Review: 28 October 2026 Article Accepted: 18 November 2026

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DOI: https://doi.org/10.5281/zenodo.24500219

How to cite this Article: Vishvas Ganpat Choudhari (2026). SYNTHESIS, CHARACTERIZATION AND BIOLOGICAL STUDIES OF COBALT METAL COMPLEXE DERIVED FROM IMINO-PYRAZOL SCHIFF BASE. World Journal of Pharmacy and Medical Science, 2(11): 100-105.

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ABSTRACT

A new Schiff base called 2-(((1H-pyrazol-3-yl) imino) methyl)-4, 6-dichlorophenol (SB) was created by refluxing 3-aminopyrazole and 3, 5-dichloro-2-hydroxybenzaldehyde in a 1:1 M composition in ethanol. Thin layer chromatography, ^{1}HNMR, and IR spectroscopy were used to examine the synthesized Schiff base. However, the cobalt, nickel, and copper complexes with SB were described utilizing thin layer chromatography, infrared spectroscopy, thermal analysis, and elemental analysis. The Schiff base is a bidentate ligand that binds to the metal ions via protonated phenolic O and imine N, according to IR spectrum research. Thermal study verifies the coordination of water molecules in complexes. The results suggest square planar geometry for copper complexes and octahedral geometry for cobalt and nickel complexes. Additionally, the antibacterial, antifungal, antioxidant, and antimalarial properties of the Schiff base SB and metal complexes were examined. When compared to the ligand, all of the produced complexes exhibit enhanced biological activity.

KEYWORDS: Schiff base complexes, antifungal, antioxidant, and 3-aminopyrazole.

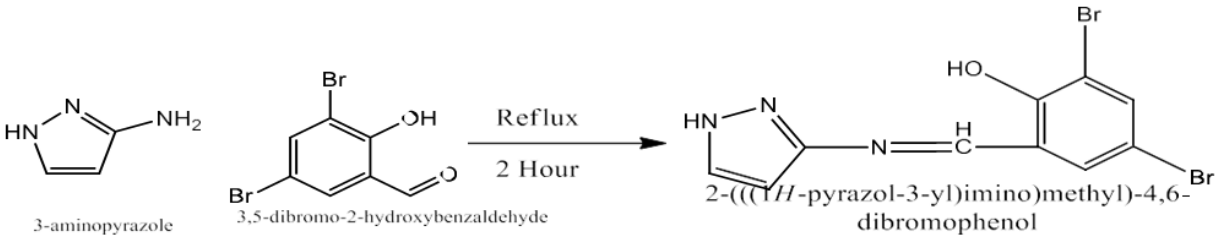
1. INTRODUCTION

In the world of medicinal chemistry, schiff bases that employ oxygen and nitrogen as donor atoms are highly intriguing and have numerous applications.[1] Schiff base ligands and their complexes have gained a well-deserved and important relevance due to recent advancements in spectroscopy as well as their remarkable antibacterial, antifungal, and anticancer capabilities.[2] Due to their structural features and obviously advantageous biologic uses, the metal complexes of pyrazole-based Schiff bases have garnered significant interest in a number of sectors. These ligands have long been important for bonding events involving transition metal ions.[3] These structures were mainly produced by the geometry surrounding the metal ion, the flexibility of the coordinating sites, and the structure of the Schiff base.[4] Pyrazole is one of the primary and most appealing azole medications against bacterial infections due to its distinctive structural features.[5] One kind of asymmetric N-containing heterocyclic ligand that has recently attracted a lot of interest in catalysis and medicinal chemistry is schiff bases based on pyrazoles.[6] A pyrazole is an organic moiety in a five-membered ring with two N on the adjacent site. Because of its many characteristics, such as antiviral, antitumoral, antibacterial, and antimalarial activity, the pyrazole moiety of metal complexes has attracted a lot of interest in the biological field.[7] The class of ligands known as pyrazoles containing aldehydes is capable of operating as good antifungal, anticancer, antibacterial, and anti-tubercular medicines. This characteristic is improved when pyrazoles are chelated with transition metals. Schiff base type ligands are structural analogs of biological molecules since they need the presence of an imine group. These ligands are synthetically flexible and easily combine with the metal ions to produce stable molecules.[8] The effectiveness of a drug is influenced by both the surrounding Schiff base group and the core metal ions. A novel drug may be produced by making a minor structural change to the Schiff base. As a result, selecting a Schiff base necessitates careful thought.

2. EXPERIMENTAL

2.1 Synthesis of Schiff Base

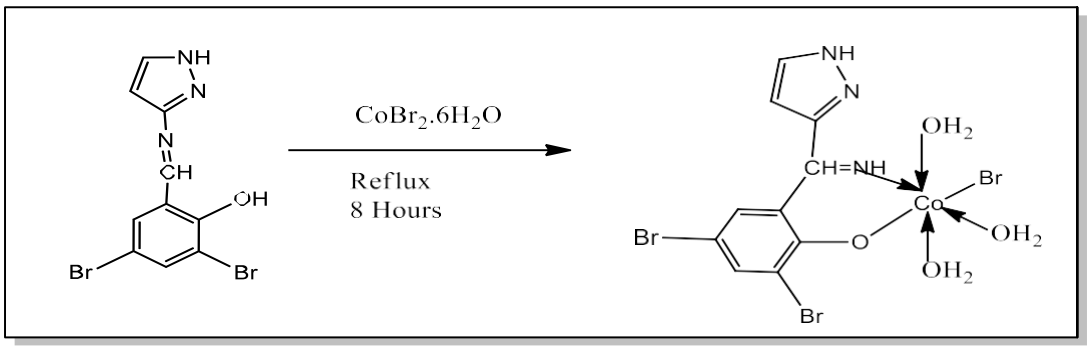
Schiffbase (SB), 2-(((1H-pyrazol-3-yl)imino)methyl)-4,6-dibromorophenol(SB) synthesized by condensing the ethanolic solutions of 3-aminolpyrazole (0.01M) and 3, 5-dibromo-2-hydroxybenzaldehyde (0.01M) by heating mental for 2.0 hours. TLC was used to verify the product's formation. After that, the yellow precipitate was filtered out and dried.



Scheme 1: Synthesis of Schiff base (SB).

2.2 Synthesis of Schiff base Co complexes

By refluxing 0.01M Cobalt bromide solution in ethanol with 0.01M Schiff base SB for 8 hours at room temperature, metal complexes were created. TLC was used to verify the reaction's completion. The resulting solid precipitate was filtered out and dried.



Scheme 2: Synthesis of Schiff base Co (II) complexes.

3. RESULTS AND DISCUSSION

IR, ^{1}HNMR, elemental analysis, and TGA, were used to characterize these compounds.

3.1 Elemental analysis

The elemental analysis of ligands and metal complexes is shown in Table No. 1.

Table No. 1: Elemental analysis of ligands and metal complexes.

Compounds	Molecular Formula	%C (% Calc.)	%H (Calc.)	% O (Calc.)	%N (Calc.)
SB	$C_{10}H_7ON_3Br_2$	34.20 (34.80)	2.1 (2.00)	4.55 (4.60)	12.20 (12.18)
Co: SB	$C_{10}H_{12}O_4N_3Br_2Co$	22.40 (22.36)	2.21 (2.23)	11.80 (11.92)	7.5 (7.82)

3.2 IR Spectra

Table No. 2 shows the bonding manner of SB with metal ions by comparing the infrared spectra of the new Schiff base with the metal complexes.. Fig. 1and 2 show IR spectra of Schiff base and its complex with Co respectively. A strong signal at 1624 cm^{-1} (Fig. 1) owing to the imine group υ (–CH = N) moiety and the absence of stretching frequencies due to the pyrazole's υ(–NH_2) and aldehyde υ(–CHO) moiety substantiated the synthesis of Schiff base.[9] A second band at 3441 cm^{-1} resulting from the aldehyde moiety's hydroxy group emerged in the Schiff base and vanished in the complexes as a result of the metal ions' interaction with the oxygen of the Schiff base.[10] The band that developed in the region 1460- 1485 cm^{-1} region as a result of the υ (C - O) further supports this bonding. The characteristic band of Schiff base for υ (–CH = N) occurs in the region of 1415-1425 cm^{-1} shifts to lower values by 10-15 cm^{-1} after complexation, signifying the binding of azomethine nitrogen to the metal ions.[11] Due to the υ(M–N) and υ(M-O) bonds, two new bands in the regions 420–430 and 470-490 cm^{-1} emerged in the metal complexes.[12] Coordination of the water molecules produced a broad signal in the spectra of metal complexes 3340–3380 cm^{-1}.[13]

Table No. 2: IR spectral data of Schiff base and its Cobalt metal complexes.

Compound	ν (-CH=N-) cm-1	ν (OH) cm-1	ν(H2O/OH) cm-1	ν (M-O) cm-1	ν (M-N) cm-1	ν (C-O) cm-1
SB	1624	3441				1485
Co: SB	1615		3380	489	425	1480

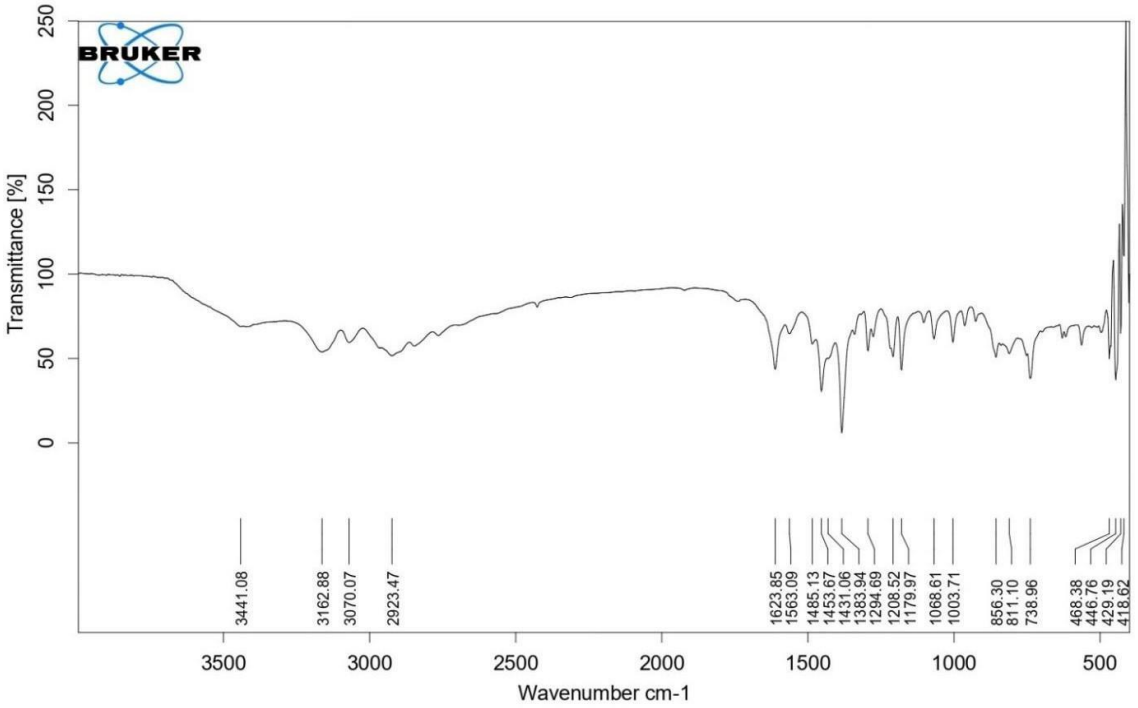


Figure 1: IR of Schiff base SB.

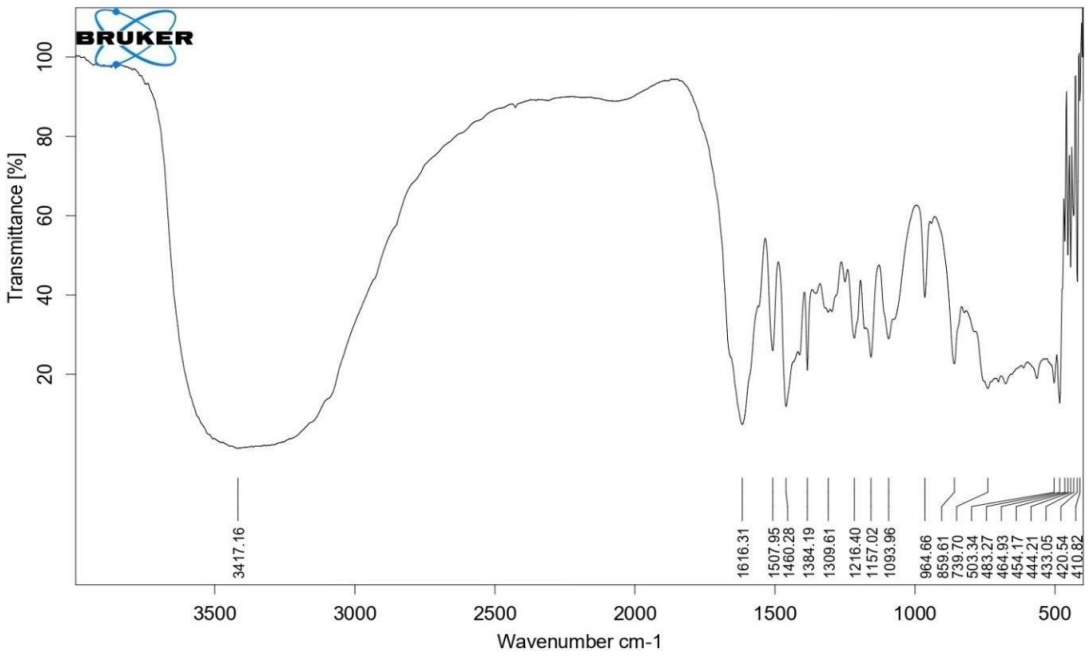


Figure 2: IR of Co: SB complex.

^{1}H NMR spectra:

Using TMS as the standard, the ^{1}HNMR spectra of the SB in CDCl3 is shown in Fig.

3. The appearance of Schiff base SB was confirmed by the development of two singlets at δ 13.12 and δ 8.97 ppm, which were induced by the hydroxy proton and proton of the imine's -C=NH- group. A singlet was observed by the SB at δ 6.16 ppm due to the pyrazole's – CH proton. The proton of pyrazole's -NH caused a singlet to form at δ12.57 ppm. The aromatic protons shows a signal between δ 6.87 and δ 7.79 ppm.

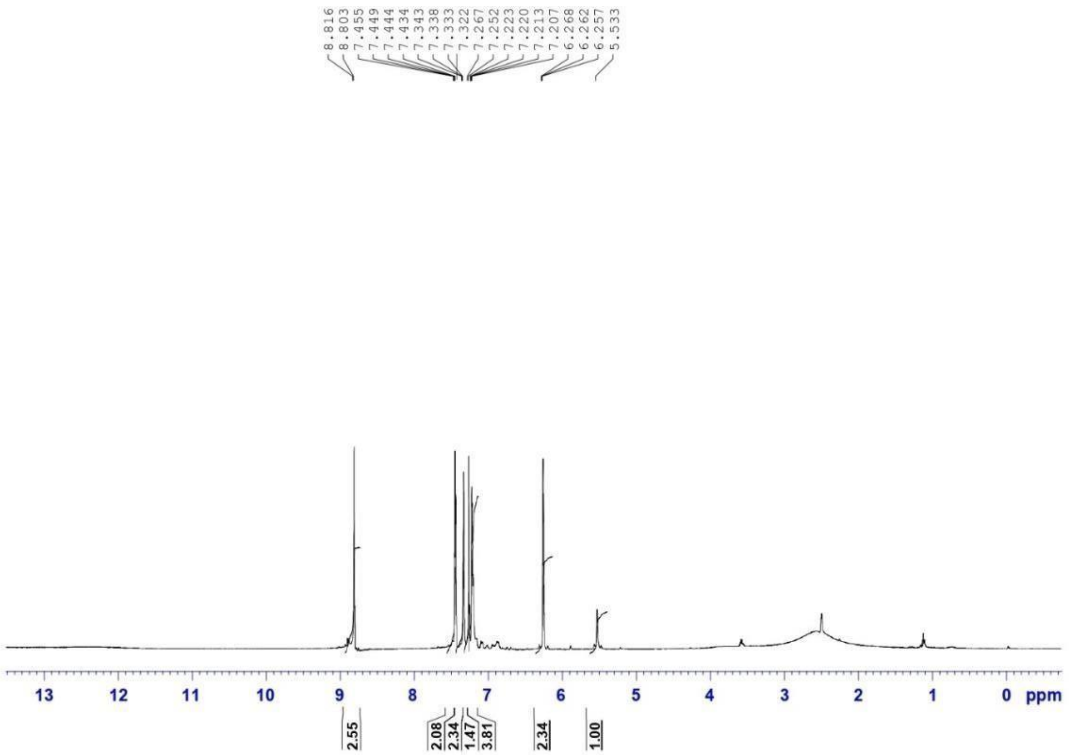


Figure 3: ^{1}HNMR spectrum of 2-(((1H-pyrazol-3-yl) imino) methyl)-4, 6-dichlorophenol (SB).

3.1. Thermal analysis

Figures 4 shows the thermal analysis cobalt complexe. The thermal deterioration of Co: SB (1:1) occurs in three stages (Fig. 6). The first process involves the breakdown of three coordinated H2O molecules between 70 and 290°C, with a mass loss percentage of 12.9% (cal. 13.43%). The pyrazole moiety breaks down in the next stage at temperatures between 290 and 450 °C, resulting in a mass loss of 10.5% (cal. 9.7%). The organic moiety breaks down in the last stage at a temperature between 450 and 550 °C, resulting in a mass loss of 53.5% (or 55.2%). The residue that remains is cobalt oxide, which has a mass of 20.2% (or 18.62%).

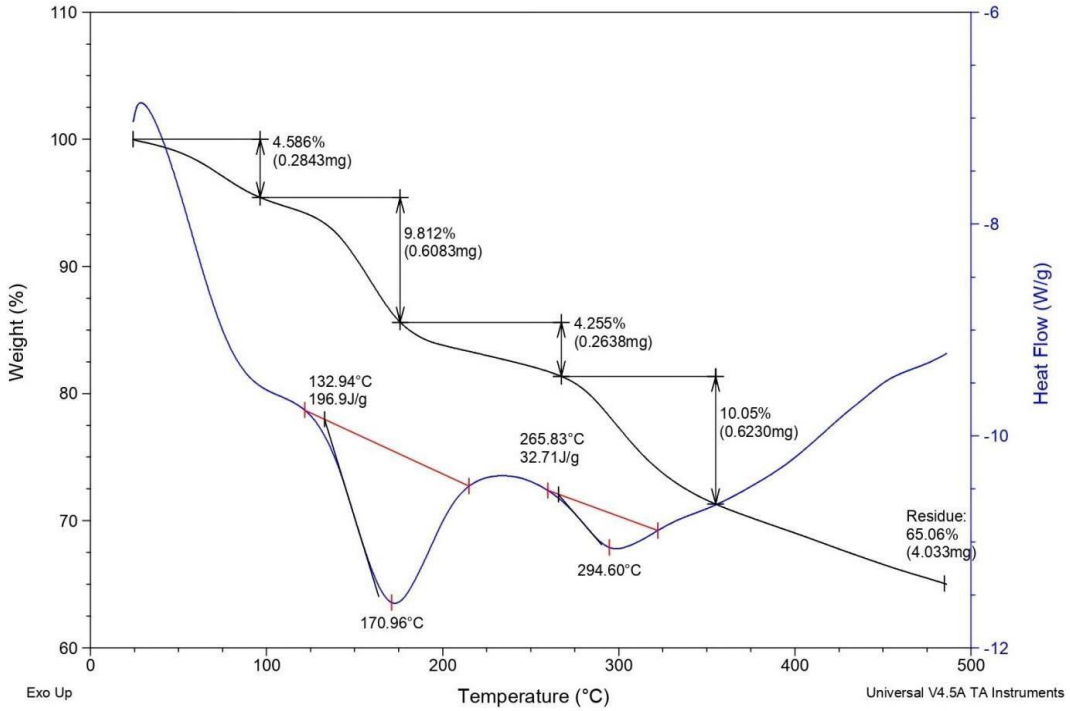


Figure 4: TGA-DSC spectra of Co: SB complex.

3.2. Biological activities

As indicated in tables 2, 3, and 4, every molecule was examined for biological properties such antibiotic, antifungal, and antioxidant. The antibacterial activity of all these combinations range from moderate to high. Excellent antibiotic and antifungal action is demonstrated by Co: PBSB at 1000 µg/ml. Likewise, Schiff base metal complex of cobalt have significant antioxidant and antimalarial activity. Co: SB shows better.

Table No. 3: % Zone of inhibition (Antibiotic) of Schiff base (SB) and its metal complexes.

Sr. No	Sample	Concentration	Zone of Inhibition (mm) E. coli	Zone of Inhibition (mm) S. aureus
1.	Control		-	-
2.	Standard Streptomycin	1000µg/ml	31	38
3	Co: SB	500µg	19	25
		1000µg	29	32
4	SB	500µg	Nil	04
		1000µg	01	06

Table No. 4: Zone of inhibition (Antifungal) of Schiff base (SB) and its metal complexes.

Sr. No.	Sample	Concentration	Zone of Inhibition (mm) C.albicans	Zone of Inhibition (mm) A. niger
1.	Control		-	-
2.	Standard Fluconazole	1000µg/ml	26	22
3.	Sample (Co: SB)	500µg	18	16
		1000µg	29	23
4.	SB	500µg	02	Nil
		1000µg	08	Nil

Table No. 5 : % zone of inhibition (Antioxidant) of Schiff base (SB) and its metal complexes.

	Antioxidant activity by DPPH (96 well method)					
Sample code	**Concentration**	**Absorbance**			**Mean**	**% inhibition**
Control	-	1.356	1.355	1.356	1.355	-
STD (Ascorbic Acid)	100µg/ml	0.356	0.350	0.352	0.352	73.98
	200µg/ml	0.342	0.340	0.344	0.342	74.77
	400 µg/ml	0.288	0.28	0.285	0.284	79.02
	600µg/ml	0.274	0.270	0.269	0.271	80.00
	800 µg/ml	0.256	0.255	0.243	0.251	81.46
	1000µg/ml	0.235	0.230	0.212	0.225	83.35
SB	100µg/ml	0.956	0.952	0.954	0.954	29.62
	200µg/ml	0.945	0.935	0.940	0.940	30.66
	400 µg/ml	0.935	0.932	0.931	0.932	31.20
	600µg/ml	0.925	0.921	0.923	0.923	31.91
	800 µg/ml	0.886	0.895	0.896	0.892	34.17
	1000 µg/ml	0.856	0.855	0.845	0.852	37.15
Co: SB	100µg/ml	0.742	0.741	0.744	0.742	45.24
	200µg/ml	0.732	0.730	0.725	0.729	46.22
	400 µg/ml	0.712	0.715	0.710	0.712	47.45
	600µg/ml	0.685	0.688	0.682	0.685	49.47
	800 µg/ml	0.675	0.665	0.664	0.668	50.72
	1000 µg/ml	0.615	0.611	0.602	0.609	55.05

Table No 6: % zone of inhibition (Antimalarial) of Schiff base and its complexes.

Compound	Concentration (µg/mL)	Absorbance			Mean	% Inhibition
Control	-	0.808	0.926	0.932	0.888	
Standard (chloroquine diphosphate)	250	0.281	0.272	0.241	0.264	70.2175
	500	0.156	0.163	0.166	0.161	81.8079
	1000	0.112	0.101	0.103	0.105	88.1470

SB	250	0.763	0.763	0.763	0.763	14.1410
	500	0.675	0.637	0.675	0.662	25.4688
	1000	0.586	0.588	0.473	0.549	38.2220
Co: SB	250	0.658	0.556	0.645	0.619	30.2700
	500	0.565	0.568	0.565	0.566	36.3090
	1000	0.478	0.489	0.467	0.478	46.2115

4. CONCLUSION

By condensing 3-aminopyrazole with 3, 5-dichloro-2-benzaldehyde to form metal complexes, a novel Schiff base was produced. IR spectroscopy, thin layer chromatography, and elemental analysis were used to confirm the production of metal complexes. Using infrared spectroscopy, the mechanism of ligand coordination with the metal ions was clarified. Specifically, the nitrogen (N) of the metal ions' imine moiety and the oxygen (O) of the hydroxyl group form a bidentate bond with the Schiff base. Coordinated water was found either inside or outside the coordination sphere, according to thermogravimetric measurements. It is proposed that the cobalt and nickel metal complexes have a square planar to copper complex and an octahedral geometry. The results demonstrated that the biological characteristics of the metal complexes were better than those of their equivalent ligands. Co: SB is the synthesized complex with the highest above activities.

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