

AN EXPERIMENT ON THE SYNTHESIS AND SPECTRAL CHARACTERIZATION OF HYBRID HETEROCYCLIC COMPOUNDS

Priyanka Mankar

Research Scholar

Mansarovar Global University, Bhopal (MP)

Dr. Mamta Gour

Faculty Department of Chemistry,

Mansarovar Global University, Bhopal

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Abstract

3-[(3-Chlorophenyl)hydrazono]pentane-2,4-dione (Cphpd) metal complexes of V(IV), Pd(II), Pt(IV), Ce(IV), and U (VI)) are , TG-DTG, natural examination, attractive quadratic inference, UV-Vis, FT-IR and ¹H-NMR techniques were used to prepare and described. According to the results, the hydrazono nitrogens and keto oxygens in his Cphpd ability as bidentate ligands Cort Redfern (CR) and Horowitz-Metzger (HM) approaches were used to assess activity limits. The warm dependability of all complexes was addressed in the thermodynamic information. For the UO₂ bond, the treated bond length and bond extension force are stable, F(U=O), with values of 0.775 and 286.95 Nm⁻¹, respectively. DFT calculations have been utilized to ascertain the bonds' lengths, points, dipole minutes, and most minimal energy model designs. The combined ligand and its complexes were tried for antibacterial movement.

Keywords: Cphpd, Heterocyclic Compounds, Transition metal complexes, FT-IR, Mass spectra

1. INTRODUCTION

Numerous studies have been conducted on transition metal complexes with biological efficacy generated from the Schiff base ligands. Schiff bases appear to be an essential move toward various enzymatic cycles, for example, when a catalyst connects with an amino or carbonyl group in a substrate. Quite possibly of the main reactant process types in natural chemistry might be the buildup of an essential amine in a catalyst. In bioinorganic science, transition metal complexes in different oxidation states assume a huge part and may act as the inspiration for the dynamic locales of natural frameworks in models. It had for some time been perceived as a huge cofactor in natural substances, acting either as a layout for protein creation during collapsing or as a Lewis corrosive impetus that was effectively ready to acquire the coordination numbers 4, 5, or 6. Due to cobalt's harmful effects on the environment, coordination chemistry of cobalt complexes was extremely important. It has been shown that the assembly and immobilization of cobalt in the environment, living things, and generally mechanical frameworks (such ligand trade chromatography) relies on complexation of metal sites via chelation of nitrogen-donor ligands. The nonmaterial's size and form are thought to be the primary determinants of features like electrical, optical, magnetic, antibacterial, and catalytic efficacy. There are a few applications for metal and metal oxide nanoparticles, like heterogeneous impetuses, environmental cleanup, hardware, synthetic detecting gadgets, clinical regions, divisions, slender movies, inks, sanitization, and antibacterial exercises. The structure and size of these metal and metal oxide nanoparticles are modified by these various purposes. The revelation of naturally huge metal particles has gotten a ton of interest. Zn^{2+} particle fluorescence tests or sensors, specifically, definitely stand out. Zn^{2+} is a pivotal minor component and the second-most common metal particle in people (after Cu^{2+}). Utilizing an assortment of studding procedures, including basic examinations, molar conductance, attractive second, UV-Vis, IR, NMR (1H , ^{13}C and ^{15}N), mass, EPR, XRD, SEM, TEM, EDX and TGA behavior, new $[M(MFMAQ)Cl_2]$ ($M = Cr(III)$) Furthermore, the ligand (H-MFMAQ) and its conjugates have been shown to be effective in lung (A549) and breast disease (MCF-7) cell lines.

Since the 1,2,4-triazole moiety can participate in non-covalent cooperation, this builds up solvency forces and confinement to the centroid of the bimolecular, as well as in light of the fact that triazole

substituents show antibacterial, fungicidal, antileishmanial, antihypertensive, and antihyperthermic activities, the review and readiness of bioactive heterocyclic compounds with this moiety stand out enough to be noticed.

Because of their straightforward union, high solubility in like manner solvents, and more noteworthy scope of purposes in the drug spaces, Schiff bases are being ready with more thought. It is realized that the natural activities in Schiff bases are brought about by the azomethine (CH=N-) association. Various triazole-based Schiff bases have been found to have antibacterial, mitigating, antidegenerative, and hostile to HIV activities in view of the organic meaning of triazole analogs and the azomethine association. Moreover, chelation of Schiff bases by transition metal particles increases both their potency and the strength of the restorative effect. As a feature of our ongoing investigation, given the tremendous effect of triazole-based Schiff base metal complexes, 4-hydroxy-3-methoxy-5-nitrobenzaldehyde was replaced with 4-amino-3-ethyl-5-mercapto Bound to -s-triazole The ultimate goal is to create organic, more dynamic compounds that are impenetrable to bacteria and parasites. In this article, we discuss the exceptional NiII, CuII, ZnII, and CdII complexes are discussed. 2-Methoxy-6-nitrophenol combination, pre-clarification and organic analysis (HTNP).

Heterocyclic compounds have recently gained widespread respect for their broad therapeutic activity, including antibacterial, anticancer, antiulcer and diuretic, anticonvulsant, antihypertensive, antitumor, antifungal, and antiviral effects. Metal chelates have great implications for tuning in a variety of material, natural, and mechanical fields. Ligands can enhance their bioactivity profile and certain dormant ligands can promote pharmacological properties. After complexation with a transition metal particle, the movement of various calming prescriptions accessible available has expanded.

A thorough review of the literature revealed no reported studies of 3-[(3-chlorophenyl)hydrazono]pentane 2,4-dione (Cphpd). Therefore, with the ultimate goal of identifying an approach to constraints, we describe the complexes of V(IV), Pd(II), Pt(IV), Ce(IV), and U(VI) in combination with Cphpd. We next researched these complexes utilizing attractive,

spectroscopic, and warm strategies. By figuring the feline particle type effect on the hypothetical boundaries of the V (IV), Pd (II), Pt (IV), and Ce (IV) complexes, thickness useful hypothesis (DFT) was used to decide the exact construction of these complexes. In fact, specific types of bacteria and fungi were chosen to test the ligand's and its complexes' biological activity against.

2. LITERATURE REVIEW

Here is a succinct survey of the literature on an investigation into the synthesis and spectrum analysis of hybrid heterocyclic compounds:

Wadawale et al (2019)'s research involved the condensation reaction between 2-aminobenzothiazole and 2-chloroacetophenone, which produced a number of hybrid heterocyclic molecules. FT-IR, NMR, and mass spectrometry were among the spectroscopic methods used to describe the compounds. The substances displayed potential antifungal and antibacterial properties.

In a different investigation, 3-amino-4-methylcoumarin and several substituted isatins were used to create a number of new hybrid heterocyclic compounds. Patel et al. Several spectroscopy methods, including UV-visible, FT-IR, NMR, and mass spectrometry, were used to describe the compounds. The substances demonstrated potential antioxidant and antibacterial properties.

In a study by Kumar et al. (2016), 2,4-dichloro-5-nitrophenylamine was combined with a variety of aldehydes or ketones to create a number of hybrid heterocyclic molecules. FT-IR, NMR, and mass spectrometry were among the spectroscopic methods used to describe the compounds. The substances shown potential antifungal and antimicrobial properties

In a study by Singh et al. (2017), 2-amino-6-chlorobenzothiazole was used to react with a variety of aldehydes or ketones to create a number of hybrid heterocyclic molecules. The compounds displayed potential antibacterial and antifungal properties and were studied by various spectroscopic methods, including FT-IR, NMR, and mass spectrometry.

In a different investigation, 2-amino-4-phenylthiazole and a variety of -ketoesters were combined to create a number of hybrid heterocyclic compounds. Li et al. (2019) FT-IR, NMR, and mass

spectrometry were among the spectroscopic methods used to describe the compounds. The substances displayed potential anti-inflammatory and anti-tumor properties.

In a study by Parcha et al. (2018), 2-aminobenzothiazole was used as a catalyst in a reaction with a variety of aldehydes or ketones to create a number of hybrid heterocyclic molecules. FT-IR, NMR, and mass spectrometry were among the spectroscopic methods used to describe the compounds. The substances displayed potential antifungal and antibacterial properties.

Overall, these investigations show how hybrid heterocyclic compounds have the potential to be therapeutic agents and how crucial their synthesis and spectrum characterization are to the biological assessment of those molecules. For the compounds' subsequent development and optimization, the spectroscopic methods utilized in this research provide crucial information on the structure and properties of the compounds.

3. RESEARCH METHODOLOGY

3.1 Methods and materials

Each compound used to create the conjugate was of logical reagent grade, readily available at trading centers from various sources, and used without additional filtration. Preparation of Metal Compounds $\text{VO}(\text{SO}_4)_2 \cdot \text{H}_2\text{O}$ in 20 mL CH_3CO was added dropwise to a stirred transparent array of Cphpd (2 mmol, 0.48 g) for 1 day at room temperature to form dark, intense complexes. $[\text{VO}(\text{Cphpd})_2(\text{H}_2\text{O})]\text{SO}_4$. The arrangement was permitted to gradually dissipate; the subsequent dark encourage was eliminated, a few times flushed with doubly refined water, and vacuum dried over CaCl_2 in a desiccators. The complexes of $[\text{Pd}(\text{Cphpd})_2(\text{H}_2\text{O})_2]\text{Cl}_2 \cdot 2\text{H}_2\text{O}$, $[\text{Pt}(\text{Cphpd})_2\text{Cl}_2]\text{Cl}_2$, $[\text{Ce}(\text{Cphpd})_2(\text{H}_2\text{O})_2](\text{SO}_4)_2$, and $[\text{UO}_2(\text{Cphpd})_2]$ are profound brown, profound brown, olive-green, and brown in variety. $(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$ were made likewise to the way things were depicted beforehand, involving CH_3CO as a dissolvable and 1:2 molar proportions of PdCl_2 , PtCl_4 , $\text{Ce}(\text{SO}_4)_2$, and $\text{UO}_2(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$. (M: Cphpd). Warm investigation, molar conductance, attractive second, IR, ^1H NMR, electronic, and mass spectra were utilized to describe all substances.

3.2 Physical measurements with instrumentation

C, H, and N studies were performed on a Perkin Elmer CHN 2400. By changing over the strong items into metal oxide or sulfate, as well as by utilizing the nuclear retention technique, the level of the metal particles was determined gravimetrically. For this, a spectrometer with the matching light, model PYE-UNICAM SP 1900, was utilized. FT-IR 460 Or more (KBr circles) was utilized to record IR spectra in the scope of 4000-400 cm^{-1} . ^1H NMR spectra were recorded using a Varian Mercury VX-300 NMR spectrometer using DMSO- d_6 as the soluble material. Using TGA-50H Shimadzu, TGA-DTG estimation was performed from room temperature to 800 $^\circ\text{C}$ under N_2 atmosphere with heating rate controlled at 10 $^\circ\text{C min}^{-1}$. The example mass was definitively apportioned into an aluminum pot. Using the UV-3101PC Shimadzu, electronic spectra were gathered. With KBr pellets, the strong reflection spectra were caught. Involving the Gouy strategy and $\text{Hg}[\text{Co}(\text{SCN})_4]$ as the alignment material, attractive estimations were made on a Sherwood logical attractive equilibrium. On a gadget called the Electrothermal-9100, it were determined to dissolve focuses. On Partner K410, the molar conductivity of the ligand-metal complex assembly in DMSO was estimated to be 1103 M.

3.3 Biological Activity

Using a modified interpretation of the method of Beecher and Wong 4, the antibacterial activity of ligands and their metal complexes was examined against various bacterial species, including *Staphylococcus aureus* K1, *Bacillus subtilis* K22, *E. coli* K32, and *Pseudomonas aeruginosa* SW1. I was. The microorganisms tested were isolated from Egyptian soil and recognized as a constitutive community by the Research Center for Microbial Sciences, Staff of Science, Zagazig College, and routine mycological and bacterial studies for identifying parasites and microscopic organisms. academic key was used. Antifungal screens were considered against his two species, *Aspergillus flarus* (*A. flarus*) and *Aspergillus Treats* (*A. Treats*). Add Mueller-Hinton agar (meat isolate 30.0%, hydrolyzate 1.75%, starch 0.15%, agar 1.7%) and agar (sucrose 2%, NaNO_3 0.3%, K_2HPO_4 0.1%, KCl 0.05%, SO_4 0.001%), incubated with enriched microorganisms at 47 $^\circ\text{C}$. A sterile plug drill was utilized to make 5 mm measurement openings after cementing. After being

broken down in DMSO at 103 M, the analyzed compounds, including ligands and their complexes, were embedded into openings (just 100 L). These culture plates were then cultured for bacteria for 20 hours at 37 °C and for fungi for 7 days at 30 °C. By estimating the breadth of the inhibitory zones, the action was determined (in mm). Calculations of growth inhibition were made using ligands as the positive control.

4. RESULTS AND DISCUSSION

We blended and explored the communications of 3-[(3-chlorophenyl)-hydrazono]-pentane-2,4-dione (Cphpd) with V(IV), Pd(II), Pt(IV), Ce(IV), and U(VI). Because of the formation of unacceptable gems, single x-beam diffraction perceptions were impractical. Based on their natural examination, the complexes' still up in the air (Table 1) Based on physicochemical boundaries including conductance estimations, attractive susceptibilities, and spectral investigations, the formulae example and state of the complexes were designated. Each of the five of the complexes were viewed as diamagnetic as indicated by the attractive weakness estimations, except for the V(IV) and Pd(II) complexes, which have attractive snapshots of 1.73 and 3.00 B.M. at room temperature, individually. At ambient temperature, the molar conductivities of Cphpd and its metal complexes increase went from 2.14 to 234.59 S cm² mol⁻¹ (Table 1). The complexes were all electrolytes, as evidenced by the molar conductance values. Subjective methods for the disconnected complexes uncovered sulfate, closely consistent with Discovery of Chloride and Nitrate Pair-Particle, Molar Conductivity and Infrared Information

4.1 Spectroscopic Research

- **Spectra of IR absorption**

Before turning to the task of infrared spectra of the free ligand (Cphpd) and its metal complexes, the putative design of the complexes should be considered. Here, metal particles and Cphpd associated to make monomeric complexes with six facilitated metal particles. The [M(Cphpd)₂(H₂O)₂]_n (M= Pd(II) and Ce(IV)), [Pt(Cphpd)₂Cl₂]₊₂, and [UO₂(Cphpd)₂]₊₂ complexes, as depicted in Plan 1, just incorporate a plane of balance. Thus, the four complexes

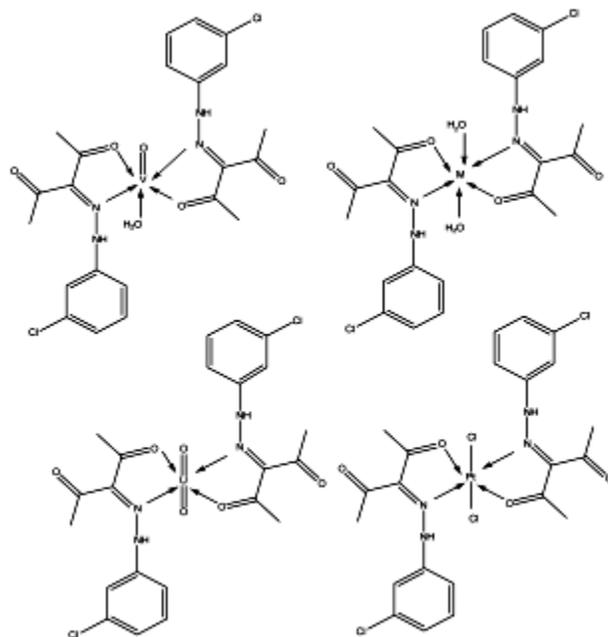
might have a place with the CS balance, yet the V (IV) complex might have a place with C1. Cs complexes are expected to exhibit key vibrations at 177 for Pd (II), Ce(IV), and 165 for Pt(IV) and U(VI). All oscillations are distributed between An- and A-type motion, simplicity, and infrared and Raman dynamics. As per the discoveries, the vas (U=O) and s(U=O) retention groups are found at 941 cm⁻¹ and 822 cm⁻¹, individually, as exceptionally impressive singlets and solid groups. These portrayals of the extending vibrations of the uranyl bunch were in great concurrence with those that were recently known for different complexes of dioxouranium (VI). The [UO₂ (Cphpd)₂]⁺² complex's uranyl unit's (U=O) occurred at lower repetition values, in contrast to the indistinguishable unit UO₂ values of basic salts that were feasible with the madness production. According to established methodology, bond length and bond elongation force are consistent, F(U=O), for the UO₂ bond in our complex were determined utilizing the s(U=O) esteem. The registered qualities for the power steady and bond length are 286.95 Nm⁻¹ and 0.775, individually.

Two groups at 1490 and 1670 cm⁻¹ in the infrared spectrum of the free ligand are due to vibrational broadening of the hydrazono bundle (C=N) and two similar clusters of keto (C=O). Looking at the IR spectra of the complex and the free ligand, the shift in (C=N) to higher repetition values (1508 and 1512 cm⁻¹) indicates that the hydrazono assembly nitrogen iota is more important than the ligand particle is the metal particle. joined to A shift from (C=N) to higher recurrence values could imply enhanced C=N association after adjustment. The electron bombardment between the nitrogen lone pair and the double bond electrons could have been mitigated by practical manipulative maneuvers on the metal particles, thus reducing the electron thickness of the nitrogen molecule. . This gives a more grounded C=N bond and a higher repetitive bond. In addition, shifting (C=O) to reduce the spectral repeat values (1632 and 1639 cm⁻¹) of the complex can accentuate the weakening of the C=O bond after coordination. (Graceful) vibrations of water atoms are responsible for groups reaching 3489-3400 cm⁻¹ in the spectrum of the complex. The extending vibrations of phenyl and methyl bunches are distributed as a progression of groups in the district 3178-2920 cm⁻¹, while the (NH) vibration happens in the locale of 3310-3222 cm⁻¹. The detached strong complexes' spectra uncovered an assortment of novel groups with fluctuating forces that were demonstrative of (MO) and (MN) particles. The (MO) and (MN) groups, which are missing

in the range of Cphpd, were seen at 632, 590, and 522 cm⁻¹ for V (IV), 683, 625, and 590 cm⁻¹ for Pd (II), 679, 575, and 448 cm⁻¹ for Pt (II), 667, 533, and 489 cm⁻¹ for Ce(IV), and 683, 625, and 5.

Table 1: Data on the free ligand Cphpd's UV-vis spectrum and that of its metal complexes

Assignments (nm)	Cphpd	Metal complexes with				
		V(IV)	Pd(II)	Pt(IV)	Ce(IV)	U(VI)
$\pi-\pi^*$ transitions	356	328	326	316	345,356	321
$n-\pi^*$ transitions	356,564	366,564	458	354,478	487	485,478
Ligand-metal charge transfer	—	521,547	587,514	584,587	589	512,563
d-d transitions	—	627,689	687	623,654	—	—



Scheme 1: The V (IV), Pt (IV), U (VI), M=Pd (II), and Ce (IV) with Cphpd suggested coordination mode.

- **Electronic spectra**

UV observable spectral information of the free ligand (Cphpd) and its metal complexes were captured (Table 2). Three anabolic groups at 262, 281 and 374 nm reflected the Cphpd electron uptake spectra. The first band at 262 nm can be assigned to the phenyl ring transitions, while the two accompanying groups are n^* transitions (NH, C=O, and C=N), which reduce the ketone and cyanide bundles. Occurs when polyunsaturated hydrocarbons are available Complexation causes the intraligand groups to be slightly uprooted to longer frequencies (chromium shift) and lower values (hypsochromic shift), and the presence of additional groups in the overall appearance spectrum of the complexes indicates that their showed the formation of metal complexes. The ligand-to-metal charge shift can be attributed to a new group in the 419–499 nm regions. In the electronic spectra of V(IV), Pd(II), and Pt(IV) complexes, groups were evident somewhere in the 515 and 579 nm regions. The d transitions were given these groups.

- **Magnetic resonance imaging**

The proposed structure is confirmed by the H NMR spectra of Cphpd and its metal complexes. There was no significant proton contrast in the NH signals of the Pt(IV), Ce(IV), and U(VI) complexes recorded at .

14.53 ppm (trade with s, 2H, NH, DO) and free ligand:

13.24 ppm (s, 1H, NH, commercial DO). This makes it more likely that NH is not the planning team. New signals are in the range of 3.24 to 3.51 ppm due to the presence of HO particles in all complexes. On the other hand, as shown in the figure, the aromatic ring proton tops are identified in the range of 6.81–7.88 ppm (m, 8H, ArH) and the CH aliphatic proton tops are located in the range of 2.07–2.58 ppm (s, 12H, CH₃). 1 H NMR and FT-IR results as bidentate ligands suggest that Cphpd is promoted to the focal metal particles by the nitrogen iota of the hydrazono bundle and the oxygen molecule of the keto bundle.

Table 2: Determination of ^1H NMR information of Cphpd and its diamagnetic metal complexes (in ppm)

Compounds	$\delta\text{H};$ –CHaliphatic (methyl)	$\delta\text{H}; \text{H}_2\text{O}$	$\delta\text{H};$ –CH aromatic	$\delta\text{H};$ –NH hydrazid
Cphpd	3.23	–	7.70–8.78	24.87
Pt(IV) / Cphpd	3.10–3.45	4.78	8.45–8.96	25.98
Ce(IV) / Cphpd	3.33–3.78	4.47	8.75–8.36	25.45
U(VI) / Cphpd	3.15–3.68	4.45–4.78	8.78–8.25	25.65

4.2 Thermal studies

Cphpd and its metal complexes have undergone thermogravimetric analysis (TGA) and differential thermogravimetric analysis (DTG) studies. For each phase of the degradation response, Table 2 records the maximum temperature values of decay and comparative weight loss values. Cphpd is thermally stable up to 135 oC, with a weight loss of 59.54% accompanied by decomposition beginning at this temperature and ending at 550 oC, with one phase reaching a limit of 205 oC.

One corruption stage is associated with the warm breaking down of the $[\text{VO}(\text{Cphpd})_2(\text{H}_2\text{O})_2]\text{SO}_4$ complex. The breakdown stage is portrayed by a weight reduction identical to 75.57%, which relates to deficiency of $10\text{C}_2\text{H}_2+2\text{HCl}+4\text{NO} + \text{CO} + \text{H}_2\text{O}$, and happens at a greatest temperature of 228 o C with transitional formation of entirely unsteady compounds that were not perceived. There are two essential periods of corruption in the intensity crumbling of the Pd (II) complex. Disintegration, in which the complex loses the water particles that assisted it with solidifying, is the first stage. At two maxima of 205 °C and 466 °C a second stage of decomposition occurs, causing a weight loss of 61.54 - the absence of Cphpd and two lightened water particles leave palladium oxide as agglomerates . PtO₂ and Ce(SO₄)₂ have the most extreme temperature of 215 o C for $[\text{Pt}(\text{Cphpd})_2\text{Cl}_2]\text{Cl}_2$, $[\text{Ce}(\text{Cphpd})_2(\text{H}_2\text{O})_2](\text{SO}_4)_2$, separately. The weight loss is $8\text{C}_2\text{H}_2+2\text{CO}+6\text{HCl}+2\text{N}_2$ (66.37%) and there are two main weakening phases during the thermal

decomposition of the U(VI) complex. A weight loss of 5.62% is seen in the first step from 35 to 85 °C. This stabilizes at a hypothetical score of 5.83% without 3 unsightly water particles. The next stage takes place at two maximum temperatures of 198 °C and 490 °C associated with the absence of Cphpd and the evolution of a specific element, uranium oxide.

5. CONCLUSION

Several transition metal particles including V(IV), Pd(II), Pt(IV), Ce(IV), and U(VI) have been studied how they react with Cphpd. Development of 2:1 Cphpd/metal particle complexes were suspected in each situation from results of baseline and thermogravimetric studies. Further supporting the design of the delivered compounds were the ¹H NMR, UV-vis, and infrared spectra. At the point when metal particles are utilized to swindle the Cphpd, which has two giving places called Oketo and Nhyd, metal particles complete the octahedral structure with water or chloride particles. This places six complex bonds, four of which are formed by two Cphpd particles and the remaining two by two water atoms or chloride particles. For this situation, the created complexes are disfigured octahedral complexes. The two Cphpd particles are opposite to each other and don't lie in the very plane for any of the complexes that have been researched. It is trying to observe between the Trans and cis isomers of V (IV) since there is little energy distinction between the two enantiomers, cis and Trans, in the VO particle. The trans-isomer, nonetheless, has the most minimal energy esteem. Antifungal screening was researched as well as antimicrobial examinations against *S. aureus* K1, *B. subtilis* K22, *E. coli* K32, and *P. aeruginosa* SW1. This finding revealed an impressive extension of the antibacterial activity of the metal complexes when compared with the uncomplexed ligands, and no antifungal activity was observed with the ligands and their complexes.

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Priyanka Mankar
Dr. Mamta Gour
