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SYNTHESIS, SPECTRAL CHARACTERIZATION, AND ANTIBACTERIAL ACTIVITY OF A ZINC(II) COORDINATION COMPLEX BASED ON 1,10-PHENANTHROLINE

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Annotatsiya. Ushbu tadqiqotda 1,10-fenantrolin ligandi asosida rux(II) koordinatsion kompleks sintez qilindi hamda uning fizik-kimyoviy va biologik xossalari tizimli ravishda o‘rganildi. Sintez qilingan kompleksning tuzilishi infraqizil (IR) va Raman spektroskopiya usullari yordamida tavsiflandi. Spektral tahlil natijalari ligandning metall ioniga azot donor atomlari orqali koordinatsiyalanishini ko‘rsatib, kompleks hosil bo‘lganligini tasdiqladi. Bundan tashqari, kompleksning antibakterial faolligi turli mikroorganizmlarga nisbatan in vitro sharoitda baholandi. Olingan natijalar shuni ko‘rsatdiki, sintez qilingan kompleks erkin ligand va mos metall tuziga nisbatan yuqori biologik faollik namoyon etadi.

Аннотация. В данной работе был синтезирован координационный комплекс цинка(II) на основе лиганда 1,10-фенантролина, а также систематически исследованы его физико-химические и биологические свойства. Структура синтезированного комплекса была охарактеризована с использованием методов инфракрасной (ИК) и рамановской спектроскопии. Спектральный анализ показал, что лиганд координируется с ионом металла через донорные атомы азота, что подтверждает образование координационного комплекса. Кроме того, антибактериальная активность комплекса была изучена in vitro в отношении различных микроорганизмов. Полученные результаты показали, что синтезированный комплекс

проявляет более высокую биологическую активность по сравнению со свободным лигандом и соответствующей солью металла.

Abstract. In this study, a zinc(II) coordination complex based on the 1,10-phenanthroline ligand was synthesized, and its physicochemical and biological properties were investigated. The structure of the synthesized complex was characterized by infrared (IR) and Raman spectroscopy methods. Spectral analysis revealed that the ligand coordinates to the metal ion through nitrogen atoms, thereby confirming the formation of the complex. In addition, the antibacterial activity of the complex was evaluated in vitro against various microorganisms. The obtained results demonstrated that the complex exhibited higher biological activity compared to the free ligand and the metal salt.

Keywords: 1,10-phenanthroline, zinc complex, IR spectroscopy, UV–Vis spectroscopy, bioactivity, coordination compound.

Introduction. Coordination chemistry is an important scientific field that studies the interactions between metal ions and ligands. Complex compounds formed on the basis of transition metals are of particular interest due to their biological, catalytic, and pharmaceutical properties. 1,10-Phenanthroline is a nitrogen-donor bidentate ligand that forms stable complexes with transition metals. The aromatic structure of this ligand enables the formation of strong coordination bonds with metal ions. Zinc is an essential trace element for biological systems, as it is involved in the active centers of many enzymes and plays an important role in metabolic processes. For this reason, zinc-based complexes have been widely studied in the fields of bioinorganic chemistry and pharmaceuticals. In recent years, metal complexes based on 1,10-phenanthroline have been reported to possess antibacterial, antifungal, and antitumor activities. Upon complex formation between the metal ion and the ligand, the lipophilicity of the compound increases, which enhances its ability to penetrate through cell membranes. Therefore, in the present study, the synthesis, spectral characterization, and antibacterial properties of the complex obtained from 1,10-phenanthroline and zinc(II) sulfate were investigated.

Experimental Methodology

The following reagents were used in this study: 1,10-phenanthroline, zinc sulfate ($ZnSO_4 \cdot 7H_2O$), ethanol, and distilled water. All reagents were used in analytical grade purity.

Synthesis of the Coordination Complex

1,10-Phenanthroline was dissolved in ethanol. In a separate vessel, zinc sulfate was dissolved in water. The resulting solutions were then mixed in a 1:2 molar ratio. The mixture was stirred for 1 hour at 50–60 °C using a magnetic stirrer. During the reaction, a homogeneous transparent solution was formed. The resulting solution was then placed in a thermostat at 40 °C and kept for 20–25 days. During this period, a gradual crystallization process occurred from the solution. The formed crystalline product was isolated by filtration, washed with distilled water, and dried. As a result, a coordination complex of the [$Zn(phen)SO_4$] type was obtained.

Results

IR (FTIR) Spectroscopic Analysis

After the synthesis of the zinc(II) complex based on 1,10-phenanthroline, IR (FTIR) spectroscopy was employed to determine its structure. The obtained spectral results clearly confirmed the formation of the complex and the coordination of the ligand to the metal center. In the high-frequency region of the spectrum, a broad intense band was observed around 3444 cm^{-1}, which corresponds to O–H stretching vibrations and indicates the presence of adsorbed or coordinated water molecules in the complex structure.

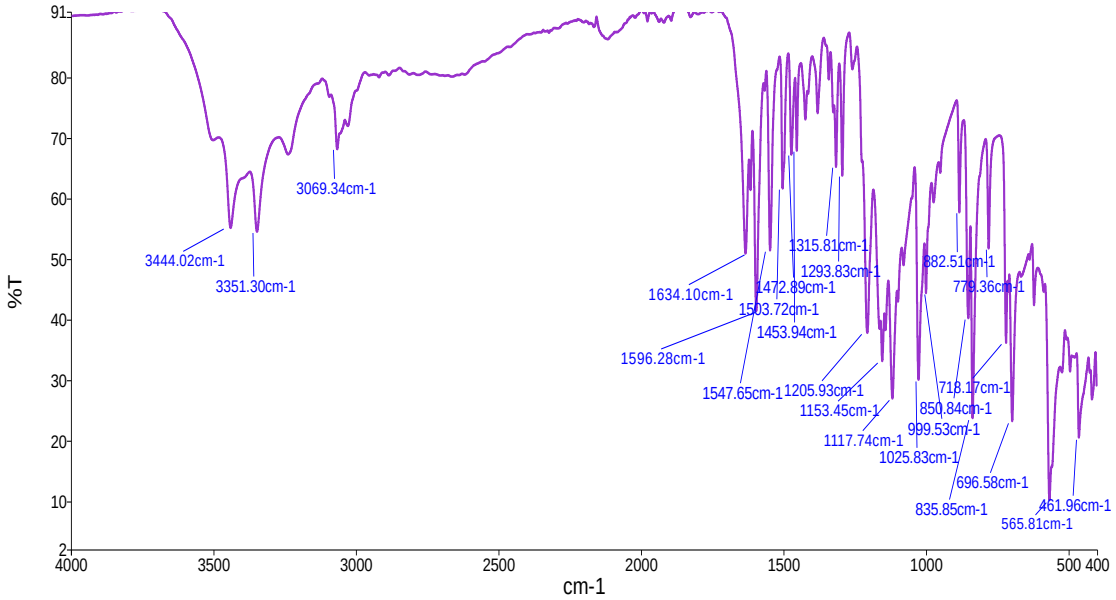


Figure 1. FT–IR spectrum of the synthesized Zn(II) coordination complex based on 1,10-phenanthroline.

The band observed at 3351 cm^{-1} may be attributed to O–H stretching vibrations associated with hydrogen bonding or to weak N–H vibrations. The band at 3069 cm^{-1} corresponds to the aromatic C–H stretching vibration, confirming the presence of the 1,10-phenanthroline ligand. The most important region of the spectrum is located within the 1500–1650 cm^{-1} range, where the vibrations of the azomethine (C=N) group and the aromatic ring are observed. In particular, the band at 1634 cm^{-1} is assigned to the C=N bond. In free 1,10-phenanthroline, this vibration is usually observed in the 1610–1620 cm^{-1} region. The shift of this peak toward a higher frequency indicates the coordination of the ligand to the zinc(II) ion through the nitrogen atoms. In addition, the bands at 1596, 1547, and 1513 cm^{-1} are attributed to aromatic C=C and C=N vibrations, indicating that the aromatic system of the ligand remains preserved after complex formation. The bands observed at 1315 and 1293 cm^{-1} in the 1200–1400 cm^{-1} region are assigned to C–N vibrations, while the intense bands at 1205, 1153, and 1117 cm^{-1} correspond to the S=O stretching vibrations of the sulfate ion (SO_4^{2-}). This confirms the presence of sulfate ions in the complex structure. Furthermore, the bands at 1025 and 999 cm^{-1} are attributed to the symmetric vibrations of the sulfate ion. In the low-frequency region (850–700 cm^{-1}), the bands at 835, 779, and 718 cm^{-1} are assigned to the out-of-plane deformation vibrations of the aromatic ring, confirming

that the 1,10-phenanthroline structure remains intact. The peak at 696 cm^{-1} is also considered characteristic of the aromatic system. One of the most important pieces of evidence is the appearance of new bands in the low-frequency region below 600 cm^{-1}. In particular, the bands at 568 and 461 cm^{-1} are attributed to the stretching vibrations of Zn–N bonds, which clearly demonstrate the formation of a coordination bond between the metal center and the ligand. Overall, the IR spectral results indicate that 1,10-phenanthroline acts as a bidentate ligand coordinated to the zinc(II) ion through its nitrogen atoms, while the sulfate ions are also present within the complex structure. The shift of the C=N vibration together with the appearance of new Zn–N bands confirms the successful synthesis of the coordination complex.

Raman Analysis

To determine the structure of the synthesized zinc(II)–1,10-phenanthroline coordination complex, Raman spectroscopy was employed. A number of characteristic vibrational bands were observed in the recorded spectrum, and these confirm the presence of coordination bonding between the ligand and the metal center. In the low-frequency region of the spectrum (266–432 cm^{-1}), the observed peaks are attributed to metal–ligand vibrations, indicating the presence of Zn–N coordination bonds. In particular, the intense peak around 266 cm^{-1} is considered an important spectral feature confirming the coordination of the zinc ion with the nitrogen atoms of the 1,10-phenanthroline molecule. The peak observed at 573 cm^{-1} corresponds to the deformation vibrations of the aromatic ring skeleton. The bands located within the 700–872 cm^{-1} range are assigned to the out-of-plane C–H vibrations and aromatic ring deformation modes of the 1,10-phenanthroline ligand.

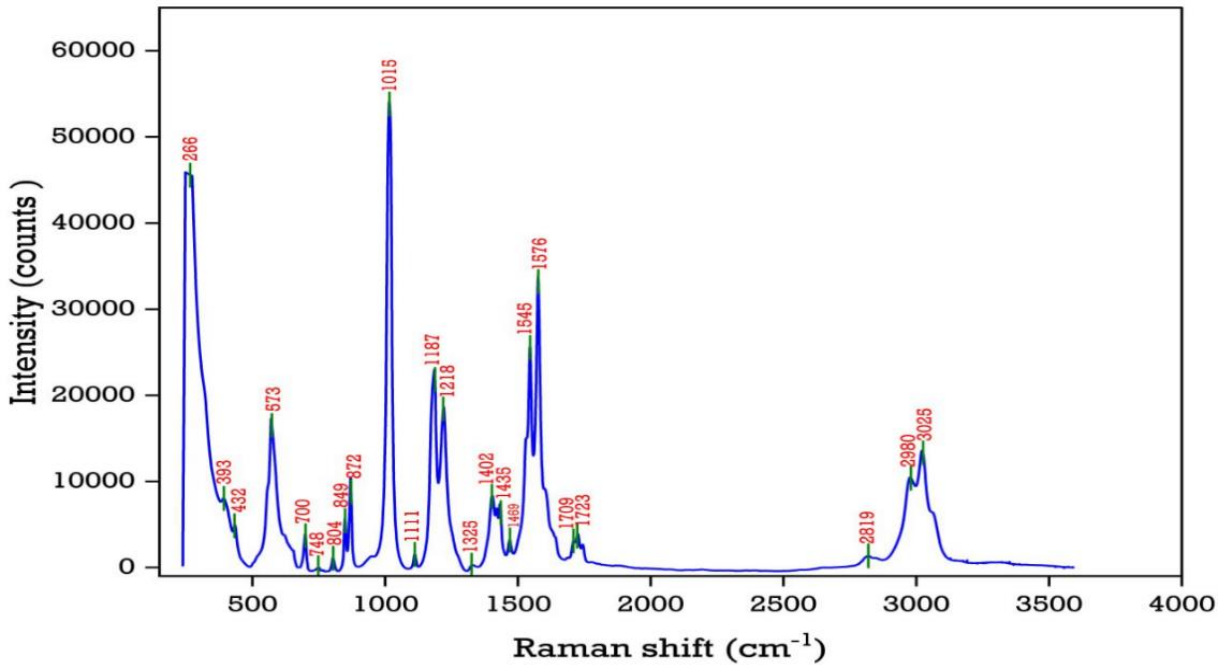


Figure 2. Raman spectrum of the Zn(II)–1,10-phenanthroline coordination complex, displaying characteristic vibrational modes associated with ligand framework and metal–ligand interactions.

A strong peak observed at 1015 cm^{-1} is attributed to the vibrational mode of the aromatic ring. This peak is characteristic of the phenanthroline ligand, and its increased intensity upon complex formation is commonly observed. The bands detected at 1111, 1187, and 1218 cm^{-1} correspond to C–H in-plane bending and C–N stretching vibrations. The presence of these peaks indicates that the molecular skeleton of the ligand remains preserved in the complex.

The peaks located in the 1325–1469 cm^{-1} region are assigned to the vibrations of aromatic C=C and C=N bonds. The intensity and slight shift of these bands indicate that the ligand has undergone coordination with the metal center. The intense peaks observed at 1545 and 1576 cm^{-1} in the spectrum correspond to aromatic C=N and C=C stretching vibrations. These vibrations are sensitive to coordination, and their presence confirms the binding of the 1,10-phenanthroline ligand to the Zn(II) ion. In the high-frequency region, the peaks observed at 2819, 2980, and 3025 cm^{-1} are attributed to aromatic C–H stretching vibrations, indicating that the aromatic framework of the ligand remains intact. Overall, the Raman spectroscopic results confirm that the synthesized substance is a coordination complex formed as a result of the coordination of zinc(II) ions with 1,10-phenanthroline ligands. The presence of low-frequency Zn–N vibrations, together with the characteristic aromatic skeletal vibrations, clearly supports the formation of the complex.

Bioactivity Analysis

The diameter of the growth inhibition zone was measured in millimeters (mm), and the results were presented according to the name of each test culture species. This method allows the sensitivity of microorganisms to a chemical substance to be evaluated in an accurate and reproducible manner, and it is widely used in pharmaceutical, agricultural, and microbiological research.

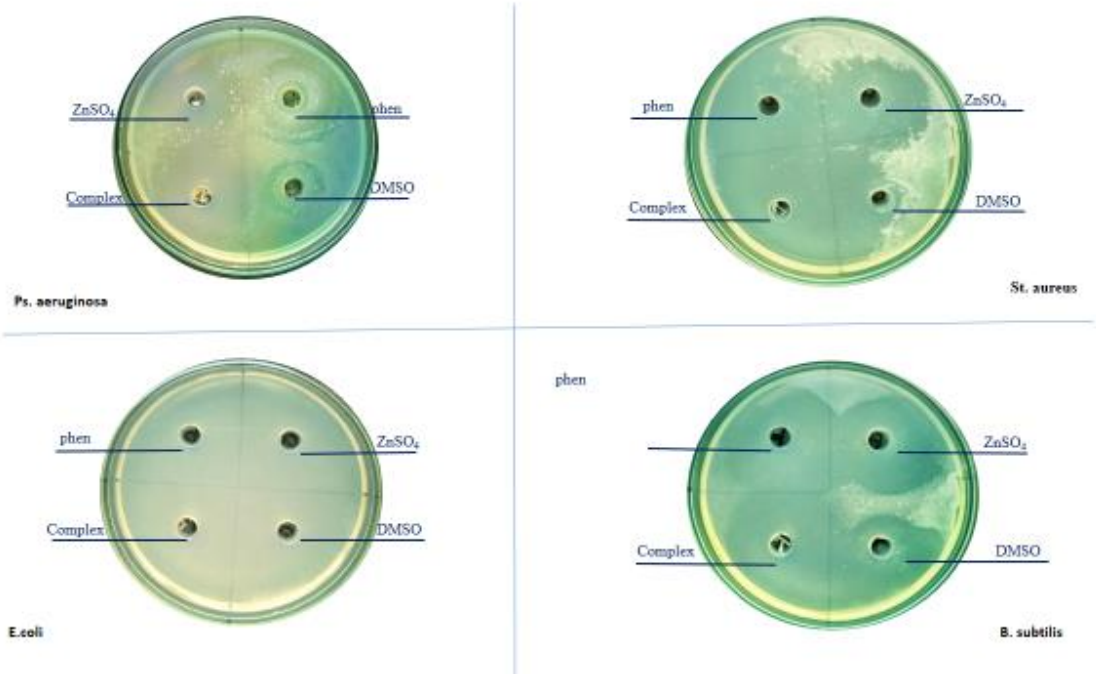


Figure 3. In vitro antibacterial activity of the synthesized Zn(II) coordination complex based on 1,10-phenanthroline against *Pseudomonas aeruginosa*, *Staphylococcus aureus*, *Escherichia coli*, and *Bacillus subtilis* using the agar well diffusion method.

The antibacterial activity of the synthesized zinc sulfate-based complex was evaluated against Gram-positive (B. subtilis, S. aureus) and Gram-negative (E. coli, P. aeruginosa) bacterial strains in comparison with the control (DMSO), zinc sulfate salt, and the ligand (1,10-phenanthroline). The inhibition zones were expressed in millimeters (mm).

Test bacteria	Control	$ZnSO_4$	Ligand	Complex
B. subtilis	–	30	34	38
P. aeruginosa	–	15	29	32
E. coli	–	38	40	42
S. aureus	–	36	42	42

Table 1. In vitro antibacterial activity of the synthesized Zn(II) coordination complex, free ligand (1,10-phenanthroline), and $ZnSO_4$ against selected bacterial strains (inhibition zone diameters, mm).

According to the results, the control (DMSO) did not exhibit any antibacterial activity, confirming that the observed biological effect was associated with the synthesized substances. Among the Gram-positive bacteria, the highest activity against B. subtilis was recorded for the complex (38 mm), followed by the ligand (34 mm) and zinc sulfate salt (30 mm). This result indicates that complex formation enhanced the antibacterial effectiveness. Against S. aureus, both the ligand and the complex showed the same high activity (42 mm),

while the zinc sulfate salt exhibited slightly lower activity (36 mm). For the Gram-negative bacteria, the complex produced the largest inhibition zone against E. coli (42 mm), while the ligand (40 mm) and zinc sulfate (38 mm) also demonstrated significant activity. In the case of P. aeruginosa, the complex (32 mm) was the most effective, followed by the ligand (29 mm) and the metal salt (15 mm). The relatively weak activity of the metal salt against this strain indicates that complex formation significantly enhanced the biological effect. Overall, the results show that although the ligand itself exhibited considerable antibacterial activity, the formation of the complex further improved the effect against certain bacteria, particularly B. subtilis, P. aeruginosa, and E. coli. This behavior may be explained by the increase in lipophilicity upon coordination of the metal ion with the ligand, the resulting improvement in membrane permeability, and the emergence of a synergistic effect. It was therefore established that the biological activity of the complex is superior to or more balanced than that of its individual components.

Conclusion

The obtained results indicate that the 1,10-phenanthroline ligand is coordinated to the zinc ion. The shift of the C=N vibration in the IR spectrum, together with the appearance of new bands assigned to Zn–N bonds, confirms the formation of the complex. The UV–Vis spectrum reflects the electronic transitions characteristic of the complex and provides information about its electronic structure. The bioactivity studies further demonstrated that the synthesized coordination compound possesses antibacterial properties.

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