



SPECTROSCOPIC, MORPHOLOGICAL, AND ELEMENTAL CHARACTERIZATION OF A BIS

(1,10-PHENANTHROLINE)MANGANESE(II) COMPLEX

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Annotation.

In this study, the bis(1,10-phenanthroline) manganese (II) complex - [Mn(Phen)2Cl2] - was synthesised, and its crystal structure and physico-chemical properties were thoroughly analysed using various analytical methods. In the FT-IR spectrum, intense peaks corresponding to C=N bonds and vibrations corresponding to the Mn-N and Mn-Cl coordination bonds were recorded. UV-Vis spectral data revealed d-d transitions and ligand-metal charge transfer (LMCT) processes. The Raman spectrum confirmed the molecular symmetry and binding energies, showing that the complex has a stable phase. Elemental analysis CHNS/O (Thermo Scientific FlashSmart) fully corresponded to the theoretical content. SEM images showed a uniformly distributed, smooth morphology of the nanocrystallites, while EDS and XRF results clearly confirmed the presence of manganese, chlorine, nitrogen, and carbon elements. The obtained results prove that the [Mn(Phen)2Cl2] complex is an ordered crystalline substance, structurally, spectrally, and thermally stable.

Key words:

Manganese(II) complex; 1,10-Phenanthroline; Spectroscopic properties.

Introduction.

MOCs received the most attention among these complexes due to their diverse morphological forms and adjustable pore sizes. These properties make MOCs preferred materials in host-guest chemistry, presenting them in various fields, including catalysis, drug delivery, and sensing, among others. Currently, extensive research is being conducted aimed at synthesising ¹MOCs, regulating molecular pore sizes, and achieving various functions. At the same

time, weak non-covalent intermolecular interactions between MOCs and the resulting supramolecular architectures remain outside systematic generalisation, although works on this topic are regularly published in the literature, which hinders the full disclosure of the potential² of MOCs in the selective adsorption of complex molecules, such as pharmaceutical residues. In the last decade, synthetic pharmaceutical drugs have been widely used to protect humans and animals from various pathogens³. However, some of these species are highly toxic, and their presence in aquatic environments (drinking water, surface waters, groundwater, and wastewater) can pose a potential risk to human health.⁴

The use of various ligands in the process of MOC synthesis is important for achieving structural diversity and improving functional properties. In this study, 1,10-phenanthroline was selected⁵ as the ligand, which is a classical bidentate chelating ligand with strong π -donor and π -acceptor properties through two nitrogen atoms. 1,10-phenanthroline has a broad spectrum of antimicrobial activity against Gram-positive and Gram-negative bacteria, as well as pathogenic fungi.⁶ At the same time, 1,10-phenanthroline and its metal complexes significantly block the replication and transcription of nucleic acids through intercalated binding with DNA. In addition, 1,10-phenanthroline is widely used in the synthesis of co-crystals and organometallic polymers. Its rigid planar structure and expanded π -conjugation system play an important role in crystal engineering through π - π stacking, hydrogen bonds, and other weak interactions.⁷ These properties allow the use of 1,10-phenanthroline-based complexes as fluorescent materials, molecular sensors, catalysts, and substances with magnetic properties. The bis(1,10-phenanthroline) manganese(II) compound, synthesised on the basis of the 1,10-phenanthroline ligand, was identified as a new complex and studied using spectroscopic methods (IR, UV-Vis, Raman).⁸

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Table 1

Element	Theoretical (%)	Practical (%)	Difference ($\Delta\%$)
C(Carbon)	65.78	65.92	-0.14
H(Hydrogen)	3.65	3.69	-0.04
N(Nitrogen)	12.73	12.85	-0.12

Comparison of theoretical and experimental elemental composition

Results and discussion.

CHNS/O analysis

The elemental composition of the synthesised complex with the formula $C_{24}H_{16}Cl_2MnN_4$ was studied using the Thermo Scientific FlashSmart CHNS/O organic element analyser. In the results of elementary analysis, the difference between the experimental and theoretical values does not exceed $\pm 0.3\%$. This confirms the complete compliance of the synthesised complex with the proposed formula and its high purity. The results reliably show the stoichiometric composition of the compound and serve as a solid basis for further spectroscopic and thermal studies (Table 1)

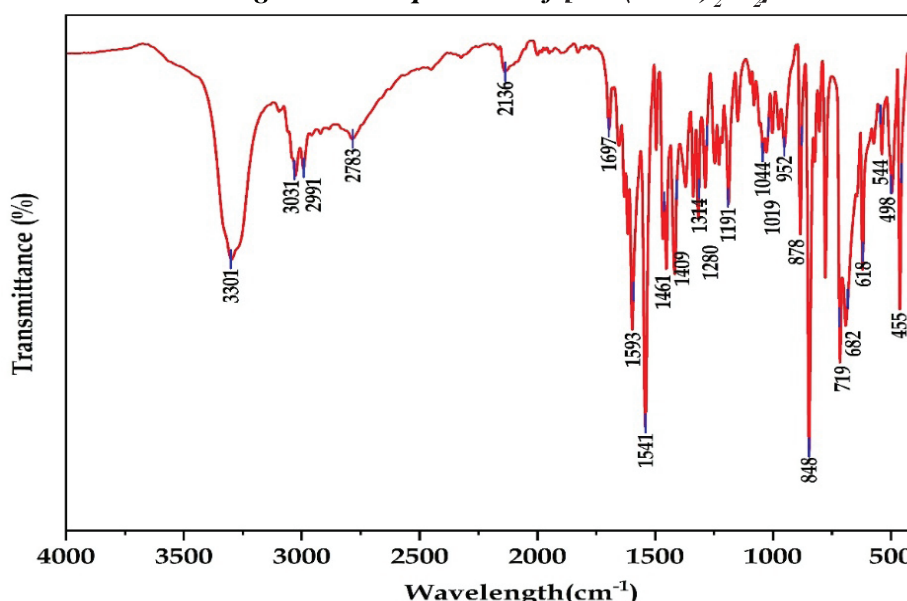
Infrared spectral analysis

The infrared spectrum of the bis(1,10-phenanthroline) manganese (II) chloride complex was analysed in the range of $400\text{--}4000\text{ cm}^{-1}$ using the Perkin-Elmer Spectrum Two FT-IR spectrophotometer. The obtained FT-IR spectrum showed a wide absorption band in the region of 3301 cm^{-1} , which confirms that the stretching vibrations correspond to the -NH group and the presence of an -NH functional group in the molecular structure. The average bands

observed in the region of 3031 and 2927 cm^{-1} correspond to aromatic and aliphatic C-H stretching vibrations, respectively. It has been established that the peak of centralised absorption at 1697 cm^{-1} is characteristic of C=N groups containing coordinated nitrogen atoms in the complex, and the sharp lines at 1593 and 1541 cm^{-1} reflect the oscillations of asymmetric stretching of C=C groups in the aromatic skeleton. The peaks at 1593 and 1541 cm^{-1} correspond to the aromatic C=C stretching vibrations of the 1,10-phenanthroline ligand. Also, the lines at 1461 , 1371 , 1280 , 1191 , and 1134 cm^{-1} can reflect, in addition to the plane, C-H and C-N contact vibrations in the aromatic rings of phenanthroline. Absorptions in the low-frequency region indicate metal-chlorine (M-Cl) and metal-nitrogen (M-N) stretching vibrations. In particular, the peaks at 878 , 848 , 719 , 682 , 616 , 545 , 498 , and 455 cm^{-1} correspond to the vibrations of Mn-Cl and Mn-N, which confirms the coordination of the nitrogen atoms of chlorine and phenanthroline to the Mn (II) centre⁹. These spectral data confirm the successful synthesis of the complex and the effective coordination [27, 28] of the 1,10-phenanthroline ligand with the Mn (II) ion (See Figure 1)

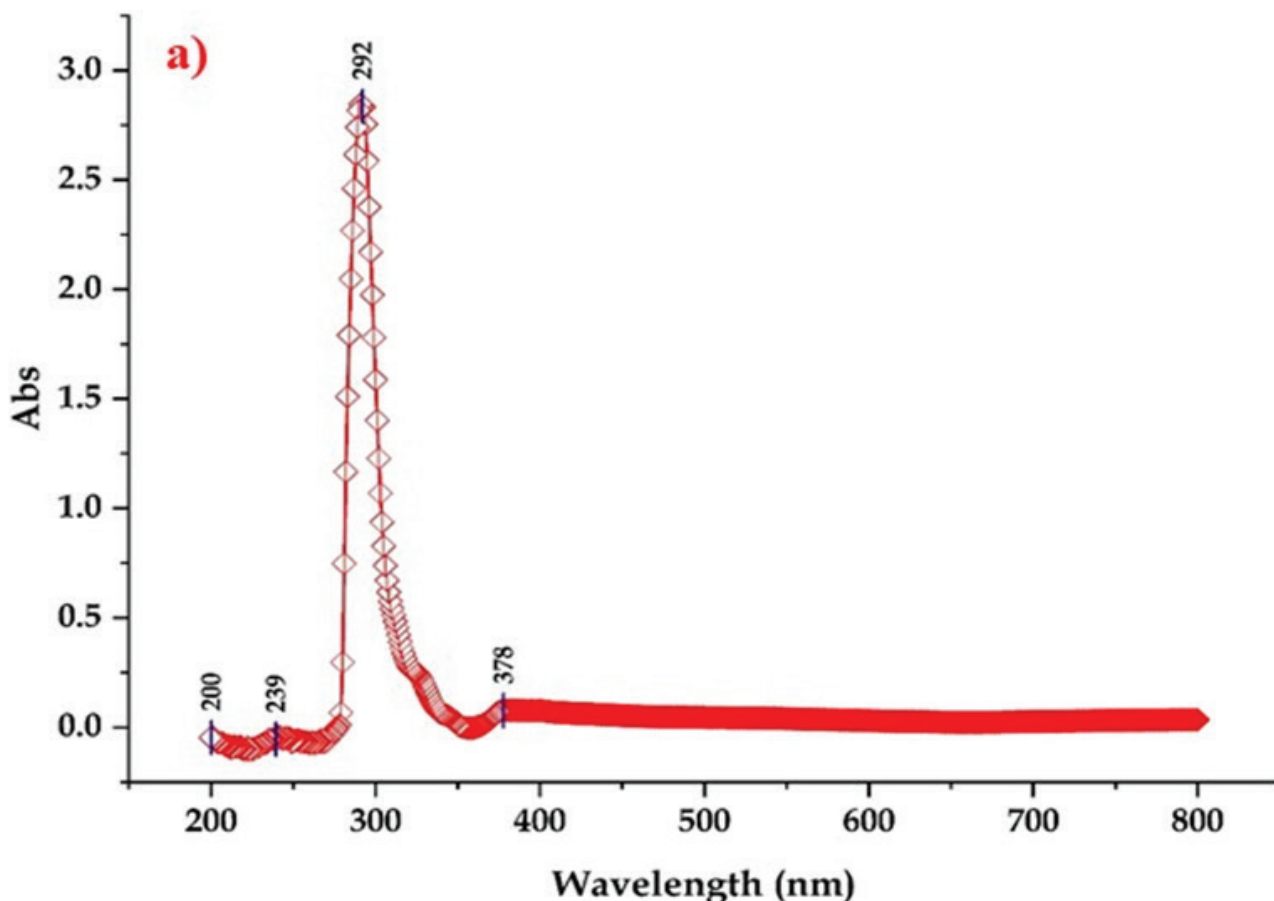
Fig. 1

Fig. 1 FT-IR spectrum of $[Mn(Phen)_2Cl_2]$



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Fig. 2.
UV-Vis spectra of $[Mn(Phen)_2Cl_2]$



UV spectral analysis

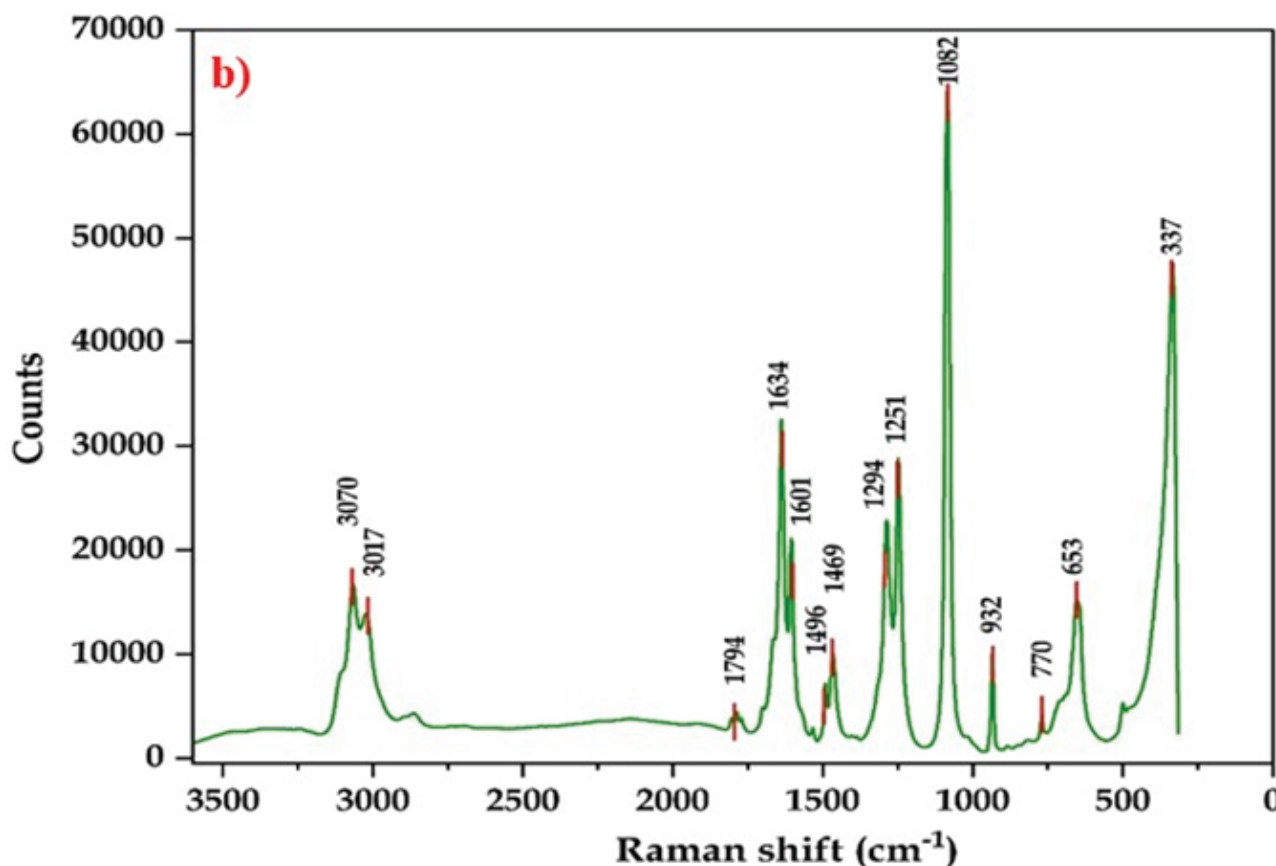
A 0.05 M solution of the $[Mn(Phen)_2Cl_2]$ complex was prepared in 96 % ethanol and studied using a Cary 5000 UV-Vis-NIR spectrophotometer in the range of 200-800 nm (See Figure 2). The obtained spectrum makes it possible to determine the optical properties of the complex and ligand-metal interactions. The clear absorption bands observed in the spectrum at 200, 239, 292, and 378 nm reflect the characteristics of electron transition processes and ligand-metal interactions in the complex. The strong absorption peak, observed at 292 nm, is associated with $\pi \rightarrow \pi^*$ electron transitions in the aromatic rings of the 1,10-phenanthroline ligand and $n \rightarrow \pi^*$ transitions, representing the transition of free electron pairs from nitrogen atoms to the π -system. The absorption bands observed at 200 and 239 nm belong to the $n \rightarrow \pi^*$ transitions, representing the transition of free electron pairs from nitrogen or oxygen atoms in the ligand to the π -system. The relatively weak absorption band, observed

around 378 nm, corresponds to d-d electron transitions in the Mn (II) centre, which indicates the coordination of the metal ion with 1,10-phenanthroline ligands. The obtained spectroscopic results clearly confirm the successful synthesis of the complex and the effective coordination of the 1,10-phenanthroline ligand with the Mn (II) ion.

Raman spectral analysis

The FT-Raman spectrum of the $[Mn(Phen)_2Cl_2]$ complex, with characteristic vibrational lines confirming its coordination medium and structural integrity, is presented (See Figure 3). Wide bands around 3070, 3017, and 2872 cm^{-1} are characteristic of aromatic ν (C-H) vibrations of 1,10-phenanthroline and are observed as a result of the coordination effect. The bands in the range of 1634-1469 cm^{-1} are characteristic of the C=C and C-N stretching vibrations within the aromatic rings of phenanthroline.

Fig. 3.
FT-Raman spectrum of $[\text{Mn}(\text{Phen})_2\text{Cl}_2]$.



The shift of C=N oscillations to a lower frequency indicates that the ligand is coordinated to the Mn (II) centre through nitrogen atoms. Strong peaks at 1082 cm^{-1} and 932 and 770 cm^{-1} correspond to the circular expansion-constriction vibration of phenanthroline. The peak at the lowest frequency of 337 cm^{-1} indicates stretching vibrations of Mn-Cl and confirms the presence of chloride ligands in the complex. Raman analysis confirms that in the $[\text{Mn}(\text{Phen})_2\text{Cl}_2]$ complex, phenanthroline ligands are coordinated to the Mn (II) centre through N-donor atoms and bonded to Cl⁻ anions.

SEM-EDS

The $[\text{Mn}(\text{Phen})_2\text{Cl}_2]$ complex's microstructure and morphology were investigated using a JEOL JSM-IT210 scanning electron microscope. Observations were performed at an accelerating voltage of 3-10 kV with a magnification range of 5-100 μm , enabling both overall structural examination and analysis of nanoscale features. To reduce surface charging and enhance image quality, the sample was coated with a thin platinum layer. These settings produced high-resolution images that clearly illustrated the

complex's morphological and microstructural traits. The elemental composition and distribution were analyzed via energy dispersive spectroscopy (EDS) with an Oxford Instruments InTouchScope™ system, using a 20 kV accelerating voltage and a 50-second acquisition time to achieve optimal spectral resolution. Elemental mapping and point analyses were conducted to identify and quantify both major and minor elements within the sample. The SEM images (See Figure 4) of the $[\text{Mn}(\text{Phen})_2\text{Cl}_2]$ complex reveal a heterogeneous, layered, and partially porous surface. At high magnification, the material exhibits irregular, uneven structural elements, fragmented particles, and microscopic pits and holes, indicating microscale porosity as well as a layered morphology resulting from the coordination between the ligand and the metal ions. At lower magnification, the overall macroscopic structure of the complex becomes apparent, showing elongated, cylindrical, or lenticular aggregates, with interconnected particles and surface unevenness, reflecting heterogeneity in crystal growth.

The EDS analysis of the complex provides comprehensive insight into its elemental composition, surface distribution, and structural features. The spectrum displays distinct peaks corresponding to Mn, Cl, C, and N,

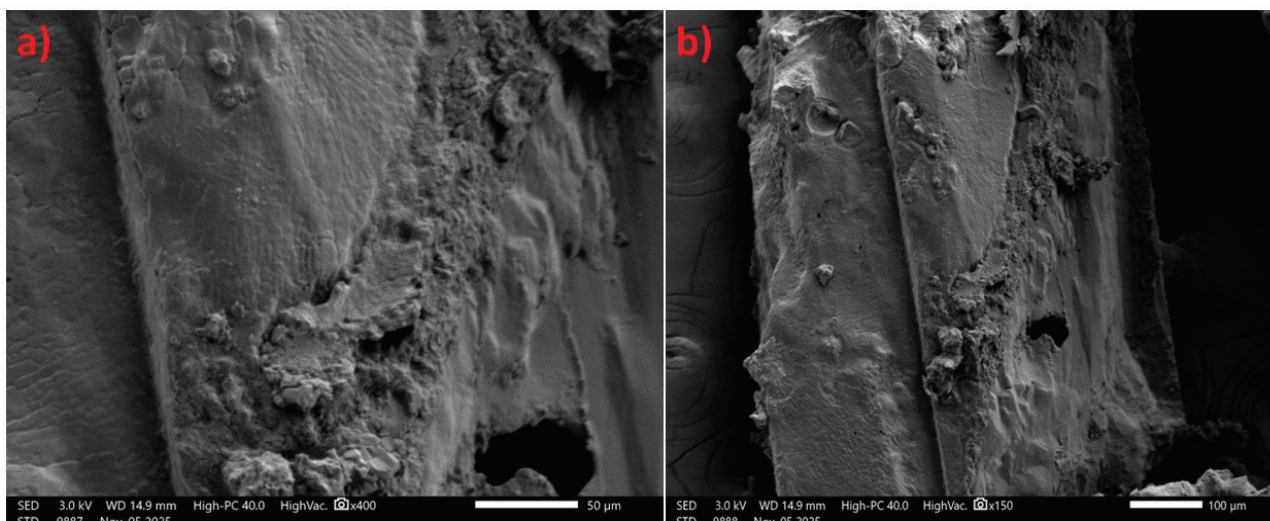
Table 2.

Element	Line	Mass%	Atom%
C	K	56.84	57.77
N	K	14.84	15.17
Cl	K	13.67	16.01
Mn	K	14.65	11.05
Total		100	100

Elemental composition of the sample based on EDS analysis, expressed in weight and atomic percentages.

Fig.4.

SEM images of the $[Mn(Phen)_2Cl_2]$ complex at magnifications of (a) 400x and (b) 150x



confirming the presence of the main components of the complex. Carbon and nitrogen, accounting for approximately 57.77 at.% and 15.17 at.% (56.84 wt.% and 14.84 wt.%), correspond to the phenanthroline ligand, while chlorine, at around 16.01 at.% (13.67 wt.%), represents the chloride ions within the complex. Manganese constitutes about 11.05 at.% (14.65 wt.%), confirming the central Mn^{2+} ion and suggesting its relatively uniform surface distribution. (Table 2.) The relative intensities and distribution of these elements are consistent with the heterogeneous, layered surface morphology observed by SEM, further supporting the structural integrity of the material and its associated high adsorption capacity and potential catalytic activity.¹

Conclusions:

A new manganese(II) coordination complex, $[Mn(Phen)_2Cl_2]$, based on the 1,10-phenanthroline ligand was successfully synthesised and comprehensively characterised using complementary physicochemical techniques. FT-IR, Raman, and UV-Vis spectroscopic analyses confirmed the coordination of the 1,10-phenanthroline ligand to the Mn(II) centre and verified the characteristic structural features of the complex. SEM-EDS analysis revealed a well-defined rod-like crystalline morphology together with a homogeneous distribution of the constituent elements, confirming the compositional uniformity of the synthesised material. The combined experimental results demonstrate the successful formation of the title complex and provide a reliable basis for understanding its structural and physicochemical characteristics.

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