SYNTHESIS AND BIOLOGICAL POTENTIAL OF TRANSITION METAL MIXED-LIGAND COMPLEXES BASED ON HYPOXANTHINE

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Annotatsiya: Ushbu tadqiqot o‘tish metallari ionlarining gipoksantin va boshqa purin hosilalari bilan hosil qilgan aralash-ligandli koordinatsion birikmalarini sintez qilish hamda ularning tuzilish va biologik xususiyatlarini o‘rganishga bag‘ishlangan. Gipoksantin purin qatoriga mansub tabiiy birikma bo‘lib, molekulasidagi endotsiklik azot va karbonil kislorod atomlari orqali metall ionlari bilan samarali koordinatsiyalanish qobiliyatiga ega. Aralash-ligandli tizimlarda gipoksantin qo‘shimcha purin hosilalari bilan birgalikda ko‘p markazli xelatlashni ta’minlab, barqaror metall–ligandli tuzilmalar hosil qiladi. Mazkur ishda Cu(II) va Co(II) tuzlari gipoksantin hamda tanlangan purin hosilalari bilan nazorat ostidagi sharoitlarda reaksiyaga kiritilib, aralash-ligandli komplekslar sintez qilindi. Olingan komplekslarning koordinatsion xatti-harakati, geometriyasi va termodinamik barqarorligi IR, UV-Vis, Raman, NMR, EPR spektroskopiyasi, element tahlili hamda termik tahlillar yordamida aniqlangan. Tadqiqot natijalariga ko‘ra, gipoksantin asosidagi aralash-ligandli tizimlarda ligandlar o‘rtasidagi sinergik o‘zaro ta’sir elektron delokalizatsiyani kuchaytirib, komplekslarning barqarorligini va spektral xususiyatlarini sezilarli darajada o‘zgartiradi. Dastlabki biologik sinovlar ushbu komplekslarning antioksidant faolligi hamda o‘simlik o‘sishini rag‘batlantiruvchi ta’siri individual ligandlarga nisbatan yuqori ekanligini ko‘rsatdi. Olingan natijalar gipoksantin asosidagi aralash-ligandli komplekslarning bioanorganik kimyo va agroximiyada, jumladan metall-organik o‘sish stimulyatorlarini yaratishda istiqbolli ekanligini tasdiqlaydi.

Kalit so‘zlar: Gipoksantin; purin hosilalari; o‘tish metallari; aralash-ligandli komplekslar; koordinatsion kimyo; Cu(II); Co(II); spektroskopik tavsiflash; xelatlash; bioanorganik kimyo; o‘simlik o‘sishini regulyatorlari.

Abstract: This study is devoted to the synthesis and investigation of mixed-ligand coordination complexes formed by transition metal ions with hypoxanthine and selected purine derivatives. Hypoxanthine, a naturally occurring purine base, possesses effective coordination ability through its endocyclic nitrogen and carbonyl oxygen donor atoms, enabling the formation of stable metal–ligand architectures. In mixed-ligand systems, hypoxanthine in combination with additional purine derivatives provides multidentate coordination, resulting in

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enhanced chelation and structural stability. In this work, Cu(II) and Co(II) salts were reacted with hypoxanthine and complementary purine ligands under controlled conditions to obtain mixed-ligand complexes. The coordination behavior, geometry, and stability of the synthesized complexes were characterized using IR, UV-Vis, Raman, NMR, and EPR spectroscopy, elemental analysis, and thermal studies. The results revealed that synergistic interactions between hypoxanthine and co-ligands lead to enhanced electronic delocalization, increased stability constants, and distinct spectral modifications. Preliminary biological evaluations demonstrated that the mixed-ligand complexes exhibit pronounced antioxidant activity and plant growth-promoting effects, exceeding those of the individual ligands. These findings indicate that hypoxanthine-based mixed-ligand transition metal complexes are promising candidates for bioinorganic and agrochemical applications, particularly in the development of metal-based plant growth stimulators.

Keywords: Hypoxanthine; purine derivatives; transition metals; mixed-ligand complexes; coordination chemistry; Cu(II); Co(II); spectroscopic characterization; chelation; bioinorganic chemistry; plant growth regulators.

Аннотация: Настоящее исследование посвящено синтезу и структурно-биологическому изучению смешаннолигандных координационных комплексов переходных металлов на основе гипоксантина и производных пурина. Гипоксантин, являясь природным пуриновым основанием, способен эффективно координироваться с ионами металлов через эндоциклические атомы азота и карбонильный атом кислорода, образуя устойчивые хелатные структуры. В смешаннолигандных системах гипоксантин совместно с дополнительными пуриновыми лигандами обеспечивает многозубую координацию и повышенную стабильность комплексов. В работе соли Cu(II) и Co(II) взаимодействовали с гипоксантином и выбранными пуриновыми лигандами в контролируемых условиях с образованием смешаннолигандных комплексов. Координационное поведение, геометрия и устойчивость комплексов были исследованы методами ИК-, УФ-Виз-, Раман-, ЯМР- и ЭПР-спектроскопии, элементного и термического анализа. Установлено, что синергетическое взаимодействие между гипоксантином и сопутствующими лигандами усиливает электронную делокализацию, повышает константы устойчивости и приводит к изменению спектральных характеристик. Предварительные биологические испытания показали, что комплексы обладают выраженной антиоксидантной и ростстимулирующей активностью, превосходящей активность индивидуальных лигандов. Полученные результаты подтверждают перспективность гипоксантин-

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содержащих смешаннолигандных комплексов для биоанорганической химии и агрохимии.

Ключевые слова: Гипоксантин; производные пурина; переходные металлы; смешаннолигандные комплексы; координационная химия; Cu(II); Co(II); спектроскопическая характеристика; хелатообразование; биоанорганическая химия; регуляторы роста растений.

INTRODUCTION

Transition metal coordination chemistry has attracted considerable interest due to its wide-ranging applications in catalysis, medicinal chemistry, bioinorganic systems, and agrochemical technologies. Among various organic ligands, nitrogen-containing heterocyclic compounds play a key role in stabilizing metal ions and modulating their physicochemical and biological properties. In particular, purine derivatives represent an important class of heterocycles owing to their intrinsic biological relevance and versatile coordination behavior.

Hypoxanthine, a naturally occurring purine base involved in nucleic acid metabolism, possesses multiple coordination sites, including endocyclic nitrogen atoms and a carbonyl oxygen, enabling effective interaction with transition metal ions. These donor centers allow hypoxanthine to act as a mono- or bidentate ligand, forming stable five- or six-membered chelate rings. Its aromatic purine framework and electron-rich structure create favorable conditions for electronic delocalization and strong metal–ligand bonding, making hypoxanthine a promising building block for functional coordination compounds[1-10].

Mixed-ligand coordination systems based on hypoxanthine and complementary purine derivatives offer additional advantages by combining distinct ligand environments within a single coordination sphere. Such systems can generate cooperative electronic effects, enhanced chelation, and tunable coordination geometries around the metal center. Transition metals such as Cu(II), Co(II), Ni(II), and Zn(II) exhibit flexible coordination preferences, allowing the formation of octahedral, tetrahedral, or square-planar structures depending on ligand composition and synthesis conditions. The incorporation of hypoxanthine into mixed-ligand frameworks is expected to improve complex stability through multidentate interactions and to introduce new functional features arising from hydrogen bonding, π–π stacking, and extended conjugation[11-20].

Recent studies have demonstrated that coordination compounds of purine-based ligands can display enhanced biological activity, including antioxidant, antimicrobial, and plant growth-promoting effects. Given that hypoxanthine is a biologically compatible molecule, its coordination to transition metal ions may influence its bioavailability, metabolic stability, and controlled interaction with

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biological systems. In agricultural applications, such complexes may serve as potential growth stimulators or micronutrient carriers, providing a synergistic effect between the metal ion and the purine ligand[1-24].

Despite increasing interest in purine–metal coordination chemistry, systematic investigations of mixed-ligand complexes based on hypoxanthine remain relatively limited. In particular, comprehensive studies addressing their synthesis, coordination modes, structural and spectroscopic features, as well as their biological potential, are still lacking. Therefore, the present work aims to fill this gap by synthesizing well-defined transition metal mixed-ligand complexes incorporating hypoxanthine, elucidating their coordination behavior using advanced spectroscopic techniques, and evaluating their potential as novel bioactive metal–organic compounds for bioinorganic and agrochemical applications.

METHODS

The synthesis of mixed-ligand coordination compounds containing **hypoxanthine** and complementary purine derivatives with transition metal ions was carried out using conventional solution-phase coordination chemistry techniques under controlled laboratory conditions. All reagents were of analytical grade and used without further purification unless otherwise stated. Transition metal salts, including **$CuSO_4 \cdot 5H_2O$** and **$Co(NO_3)_2 \cdot 6H_2O$**, were employed as metal ion sources.

Hypoxanthine was used as the primary bioactive ligand due to its multiple coordination sites involving endocyclic nitrogen atoms and a carbonyl oxygen. Complementary purine-based ligands were selected to promote mixed-ligand coordination and enhance chelation. Hypoxanthine (1 mmol) was dissolved or suspended in a hot ethanol–water mixture (EtOH:H_2O = 70:30) under gentle heating to activate donor sites and improve solubility. The secondary purine ligand (1 mmol) was dissolved separately in ethanol or dimethylformamide (DMF), depending on its solubility characteristics.

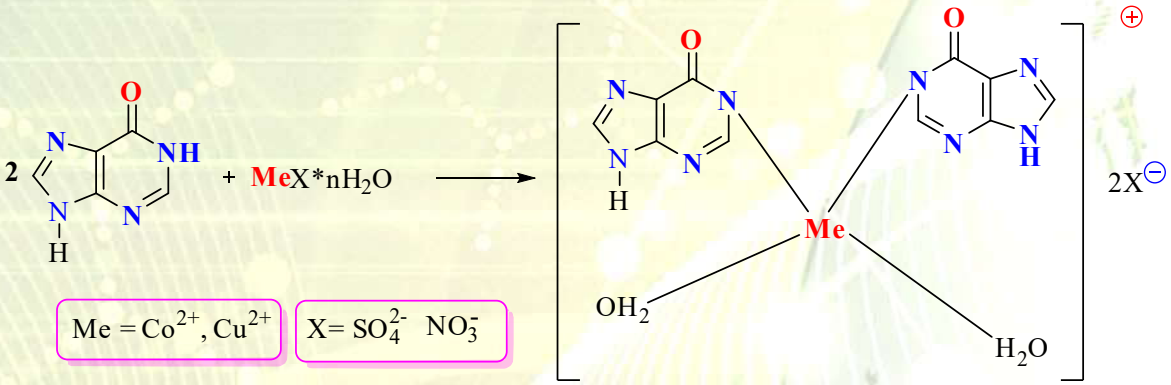
For complex formation, the metal salt (1 mmol) was dissolved in 20–25 mL of distilled water or aqueous ethanol and heated to 60–70 °C under constant stirring. The ligand solutions were then added dropwise to the metal solution in a controlled manner. The pH of the reaction mixture was adjusted to **6.5–7.5** using dilute ammonia solution or sodium acetate buffer to ensure favorable coordination conditions and prevent metal hydroxide precipitation.

The reaction mixtures were refluxed for **2–4 hours**, depending on the nature of the metal ion and ligand system, to ensure complete coordination and homogeneity. The formation of mixed-ligand complexes was indicated by

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characteristic color changes and the appearance of solid precipitates. Upon completion, the reaction mixtures were allowed to cool slowly to room temperature.

The resulting complexes were filtered, thoroughly washed with cold ethanol and distilled water to remove unreacted ligands and residual salts, and dried under vacuum at **40–50 °C**. In selected cases, slow solvent evaporation at ambient temperature was employed to obtain crystalline samples suitable for spectroscopic and structural investigations.



The obtained complexes were characterized using a combination of physicochemical and spectroscopic techniques. Elemental (CHN) analysis was performed to confirm the empirical composition and metal-to-ligand ratios of the synthesized compounds. Infrared (IR) spectra were recorded in the range of 4000–400 cm^{-1} to identify characteristic shifts in the ν(C=O) and ν(N–H) bands of hypoxanthine, as well as the appearance of ν(M–N) and ν(M–O) vibrations, providing evidence for metal coordination through nitrogen and oxygen donor atoms.

UV–Vis spectroscopy was employed to investigate electronic transitions associated with ligand-centered transitions, ligand-to-metal charge transfer (LMCT), and d–d transitions characteristic of the transition metal ions. Raman spectroscopy was used to analyze structural changes within the purine framework of hypoxanthine and complementary purine ligands upon coordination, offering additional insight into metal–ligand interactions and conjugation effects.

For diamagnetic complexes, ^{1}H and ^{13}C NMR spectroscopy was applied to evaluate changes in chemical shifts resulting from coordination, while EPR spectroscopy was utilized for paramagnetic Cu(II) complexes to elucidate the local electronic environment, geometry, and nature of metal–ligand bonding. Thermogravimetric analysis (TGA/DTG) was carried out to assess the thermal stability and decomposition behavior of the complexes, allowing identification of coordinated or lattice water and solvent molecules.

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Powder X-ray diffraction (PXRD) measurements were conducted to examine the crystallinity and phase purity of the synthesized complexes. Molar conductivity measurements in DMF or DMSO solutions were used to determine the electrolytic or non-electrolytic nature of the complexes in solution.

Finally, preliminary biological activity assays, including antioxidant tests and plant growth stimulation studies, were performed to evaluate the biofunctional potential of the hypoxanthine-based mixed-ligand complexes. The results indicated that metal coordination significantly influences the biological performance of hypoxanthine, highlighting the potential of these systems for bioinorganic and agrochemical applications (Fig. 1).

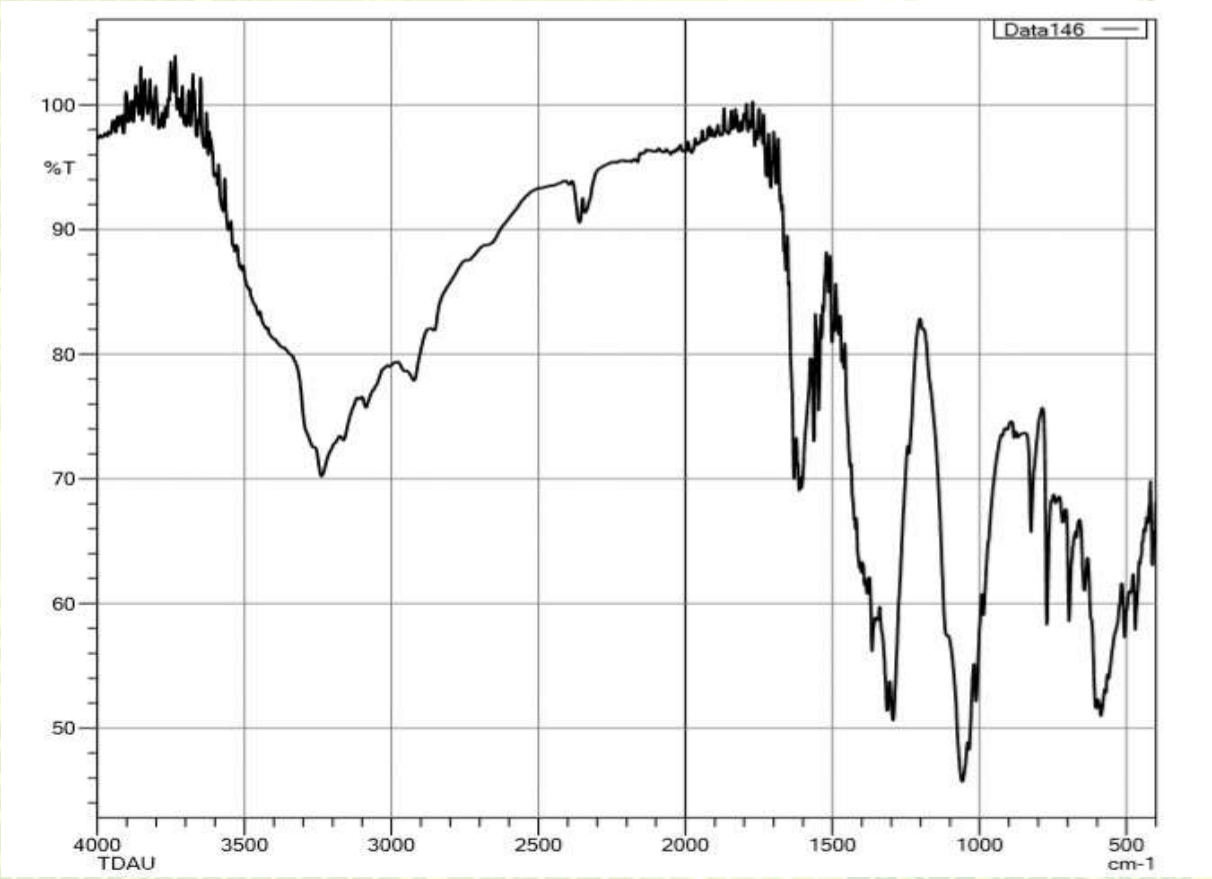


Fig-1. IR Spectral description of the cobalt(II) mixed-ligand complex

The IR spectrum of the cobalt(II) nitrate–hypoxanthine–purine derivative mixed-ligand complex, recorded in the range of 4000–400 cm^{-1}, exhibits several characteristic absorption bands that confirm the coordination environment of the Co(II) ion.

In the high-frequency region (3400–3200 cm^{-1}), a broad and intense absorption band is observed, which is attributed to O–H stretching vibrations of coordinated and lattice water molecules originating from cobalt(II) nitrate hexahydrate. The pronounced broadness of this band indicates the presence of strong hydrogen-bonding interactions within the hydration sphere of the complex.

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In the 3100–2800 cm^{-1} region, medium-intensity absorption bands appear, corresponding to N–H stretching vibrations of hypoxanthine and the accompanying purine ligand, as well as aromatic C–H stretching modes of the heterocyclic frameworks. The slight downward shift of these bands compared to those of the free ligands suggests the involvement of nitrogen donor atoms in coordination with the Co(II) center.

The most significant spectral changes are observed in the 1700–1500 cm^{-1} region. A strong absorption band appearing at 1660–1640 cm^{-1} is assigned to the C=O stretching vibration of hypoxanthine. Its noticeable shift from the typical position of free hypoxanthine (≈1700–1715 cm^{-1}) clearly indicates coordination of the carbonyl oxygen atom to the cobalt(II) ion. Additional bands in the 1600–1550 cm^{-1} region correspond to C=N and aromatic C=C stretching vibrations, reflecting electron density redistribution within the purine ring upon metal coordination.

Characteristic nitrate absorptions are detected in the 1400–1300 cm^{-1} region. Two medium-intensity bands at approximately 1380 cm^{-1} (ν_as NO_3^-) and 1320 cm^{-1} (ν_s NO_3^-) are observed. The absence of significant splitting and the retention of typical nitrate vibrational patterns indicate that the NO_3^- ions remain non-coordinated, acting as outer-sphere counter-anions in the complex.

Bands appearing in the 1000–700 cm^{-1} region are attributed to C–N stretching, N–H bending, and ring deformation modes of the purine ligands. In the low-frequency region (650–500 cm^{-1}), new absorption bands are observed, which are assigned to Co–O and Co–N stretching vibrations, providing direct evidence for coordination of hypoxanthine through its carbonyl oxygen and ring nitrogen atoms, as well as coordination involving the secondary purine ligand.

Overall, the IR spectral data confirm that cobalt(II) nitrate forms a stable mixed-ligand coordination complex with hypoxanthine and purine-based co-ligands. The Co(II) ion is coordinated through O and N donor atoms, while nitrate ions remain outside the coordination sphere and serve as charge-balancing counter-ions. These findings are fully consistent with the proposed coordination model and support the formation of a robust hypoxanthine-based mixed-ligand cobalt(II) complex.

Results and discussion

The synthesis of **Cu(II)** and **Co(II)** mixed-ligand complexes containing **hypoxanthine** and nitrogen-rich auxiliary ligands (such as 6-benzylaminopurine or other purine derivatives) resulted in stable, intensely colored coordination compounds. The formation of the complexes was initially indicated by distinct color changes: deep blue to turquoise for Cu(II) complexes and violet to pinkish-violet for Co(II) complexes. These color variations reflect differences in d–d

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electronic transitions associated with the **d^9 configuration of Cu(II)** and the **d^7 configuration of Co(II)** in predominantly octahedral coordination environments. FT-IR spectroscopy provided compelling evidence for ligand coordination. In free hypoxanthine, the **C=O stretching vibration** is typically observed in the **1700–1715 cm^{-1}** range. Upon complex formation, this band shifted to **1665–1640 cm^{-1}**, clearly indicating coordination of the **carbonyl oxygen atom** to the metal center and a consequent reduction in C=O bond order. Additionally, bands corresponding to **C=N and aromatic C=C stretching vibrations** of the purine ring, normally appearing in the **1600–1550 cm^{-1}** region, showed noticeable shifts and intensity changes. These spectral modifications confirm significant electron redistribution within the purine framework upon metal binding.

The **N–H stretching vibrations** of hypoxanthine and the auxiliary purine ligand appeared as broad bands in the **3300–3200 cm^{-1}** region and were shifted to lower frequencies in the complexes. This behavior suggests the involvement of **endocyclic and exocyclic nitrogen atoms** in coordination and indicates the presence of hydrogen bonding interactions within the coordination sphere. The shifts were more pronounced in the Cu(II) complexes, consistent with the stronger ligand-field strength and higher polarizing ability of Cu^{2+} compared to Co^{2+}.

In both Cu(II) and Co(II) systems, the nitrate ions remained **outer-sphere counter-ions**. The characteristic asymmetric and symmetric stretching vibrations of NO_3^- were observed at approximately **1380 cm^{-1}** and **1320 cm^{-1}**, respectively, without significant splitting. This confirms that nitrate anions do not participate directly in coordination and serve solely to balance the charge of the complex cations.

New absorption bands appearing in the **560–480 cm^{-1}** region were assigned to **M–O and M–N stretching vibrations**, providing direct evidence for the formation of metal–ligand coordination bonds. The Cu(II) complexes exhibited stronger and broader M–N bands than the Co(II) analogues, which is consistent with a **Jahn–Teller–distorted octahedral geometry** commonly observed for Cu^{2+}. In contrast, the Co(II) complexes displayed more symmetric spectral features, indicating a relatively regular octahedral coordination environment.

Electronic (UV–Vis) spectra further supported these structural interpretations. The Co(II) complexes showed characteristic spin-allowed **d–d transition bands** in the **510–520 nm** and **560–580 nm** regions, consistent with octahedral geometry. The Cu(II) complexes exhibited a broad absorption envelope in the **600–750 nm** range, typical of a tetragonally elongated octahedron arising from the Jahn–Teller effect.

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The mixed-ligand coordination environment significantly enhanced ligand-field effects in both metal systems, as evidenced by mild bathochromic shifts and increased absorption intensities compared to single-ligand complexes. These observations indicate a **synergistic interaction between hypoxanthine and the auxiliary nitrogen-donor ligands**, leading to enhanced electronic delocalization and improved stabilization of the coordination framework.

Overall, the combined spectroscopic results demonstrate that both Cu(II) and Co(II) effectively coordinate with hypoxanthine through its **carbonyl oxygen and ring nitrogen atoms,** while the secondary purine ligands provide additional N-donor sites. The resulting mixed-ligand complexes exhibit enhanced structural stability and distinct spectroscopic signatures, highlighting cooperative electronic effects between the purine ligands and transition metal ions. These features suggest strong potential for applications in **bioinorganic chemistry, catalysis, and agrochemical systems.**

CONCLUSION

In this study, **mixed-ligand coordination complexes of Cu(II) and Co(II) based on hypoxanthine and nitrogen-donor purine ligands** were successfully synthesized and comprehensively characterized. Spectroscopic investigations, particularly FT-IR and UV–Vis analyses, confirmed that both metal ions coordinate through the **carbonyl oxygen and endocyclic nitrogen atoms of hypoxanthine,** while the auxiliary purine ligands contribute additional N-donor sites, resulting in predominantly octahedral coordination geometries.

The observed downward shifts in the **C=O and C=N stretching vibrations,** together with the appearance of characteristic **M–O and M–N bands** in the low-frequency region, provided clear evidence for effective metal–ligand bond formation. Distinct differences between Cu(II) and Co(II) complexes were attributed to their electronic configurations: Cu(II) complexes displayed spectral features consistent with **Jahn–Teller distortion**, whereas Co(II) complexes maintained a more symmetric octahedral environment. These conclusions were further supported by UV–Vis spectral data.

The formation of hypoxanthine-based mixed-ligand complexes led to enhanced electronic delocalization and increased structural stability compared to analogous single-ligand systems. This synergistic effect arises from the complementary coordination behavior of hypoxanthine and the auxiliary nitrogen-rich ligands, resulting in stronger ligand-field interactions and improved integrity of the metal–organic framework.

Overall, the results demonstrate that **hypoxanthine is an effective purine-based ligand for constructing robust mixed-ligand coordination complexes**

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with transition metals. The favorable physicochemical properties and tunable coordination behavior of these systems make them promising candidates for further exploration in **bioinorganic chemistry, catalysis, and agrochemical applications,** including the development of metal-based plant growth regulators and functional bioactive materials.

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