ACYCLOVIR–ISONIAZID MIXED-LIGAND COMPLEXES WITH Cu(II) AND Zn(II): SYNTHESIS AND PHYSICOCHEMICAL ANALYSIS

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Abstract. *In this study, mixed-ligand Cu(II) and Zn(II) coordination complexes with acyclovir ($C_8H_{11}N_5O_3$, ACV) and isoniazid ($C_6H_7N_3O$, INH) were synthesized under mild laboratory conditions in a weakly acidic medium (pH 6.5–7.0). The complexes were formulated as [$CuCl_2$(ACV)(INH)] and [$ZnCl_2$(ACV)(INH)], where both ligands coordinate to the metal ions through nitrogen and oxygen donor atoms. Fourier transform infrared spectroscopy (FTIR) confirmed the involvement of azomethine nitrogen and carbonyl oxygen atoms in the coordination process, indicating the formation of stable chelate structures. Surface morphology and elemental composition were investigated using scanning electron microscopy with energy-dispersive X-ray analysis (SEM–EDX), revealing homogeneous particle distribution and metal–ligand ratios consistent with the proposed structures. Thermogravimetric and differential thermal analyses (TGA–DTA) showed that the complexes possess higher thermal stability than the free ligands, with decomposition occurring above 250 °C. Mass spectrometric analysis supported the proposed coordination structures through characteristic fragment ions.*

Keywords: *acyclovir, coordination complexes, copper(II) chloride, isoniazid, zinc(II) chloride.*

Introduction

Coordination and bioinorganic chemistry have advanced significantly in recent decades due to the extensive investigation of transition metal complexes with diverse structural architectures and functional properties [1–5]. Among these, copper(II) and zinc(II) complexes are of particular interest because of their important biological roles, relatively low toxicity, and versatile coordination behavior [6–10]. These features make them attractive for applications in medicinal chemistry, catalysis, sensing technologies, and the development of functional materials [7–9].

Cu(II) and Zn(II) ions show a strong affinity for ligands containing nitrogen and oxygen donor atoms, particularly Schiff base and related N,O-chelating systems [10–12]. Such ligands facilitate the formation of complexes with different coordination geometries, including square planar, tetrahedral, trigonal bipyramidal, and octahedral structures, allowing fine-tuning of their physicochemical properties and reactivity [13–15]. Although numerous studies describe individual Cu(II) or Zn(II) complexes, comparative investigations employing identical ligand frameworks remain relatively limited, making it difficult to clearly establish how the metal center influences structural and functional characteristics [16–20].

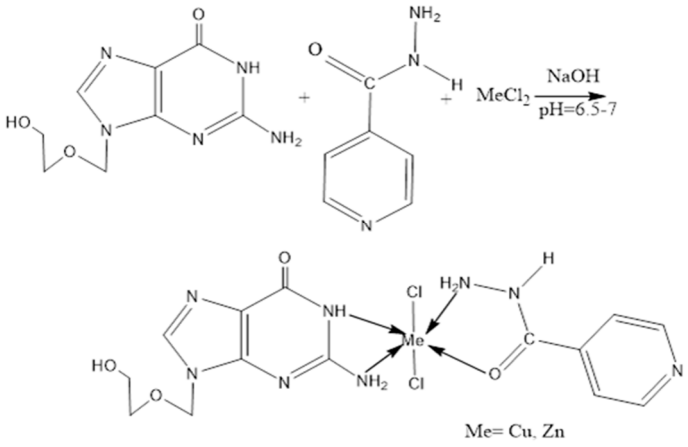
Considerable attention has also been devoted to the biological activities of these complexes, including antibacterial, antifungal, antioxidant, and anticancer effects [21–25]. The enhanced activity of Cu(II) complexes is often related to the redox properties of copper, whereas Zn(II) complexes, being redox inactive, mainly contribute to structural stabilization and enzyme modulation [26–29]. However, systematic correlations between ligand structure, metal identity, and biological performance are still not fully understood. Therefore, the present study focuses on the synthesis and comprehensive characterization of Cu(II) and Zn(II) complexes derived from a common ligand system. Using a combination of spectroscopic and analytical techniques, the work aims to clarify the

influence of the metal center on structural features and physicochemical properties, thereby contributing to the rational design of bioinorganic coordination compounds [26–48].

The aim of this study was to investigate the complex formation of acyclovir (ACV) and isoniazid (INH) with Cu(II) and Zn(II) ions and to evaluate the stability and structural properties of the resulting complexes.

Experimental Part

Synthesis of Cu(II)–Acyclovir–Isoniazid Mixed-Ligand Complex (Scheme 1). In this study, a mixed-ligand complex composed of Cu(II) ions, acyclovir (ACV), and isoniazid (INH) was synthesized. All chemicals used were of analytical grade and were employed without further purification. A 1:1 (v/v) mixture of ethanol and distilled water was used as the solvent system. Initially, acyclovir (0.01 mol, approximately 2.25 g) and isoniazid (0.01 mol, approximately 1.37 g) were dissolved in 50 mL of the ethanol–water mixture under continuous stirring using a magnetic stirrer. The pH of the resulting ligand solution was adjusted to the range of 6.5–7.0 using 0.1 M NaOH solution in order to facilitate metal–ligand coordination. Separately, copper(II) chloride dihydrate ($CuCl_2 \cdot 2H_2O$, 0.01 mol, approximately 1.70 g) was dissolved in 10 mL of distilled water. The metal salt solution was then added dropwise to the ligand solution with constant stirring at 400 rpm. The reaction was carried out under ambient conditions. The reaction mixture was maintained at 60 ± 2 °C for 4 hours. To maintain the pH of the reaction medium within the range of 6.5–7, a 0.1 M NaOH solution was used. This prevented excessive acidity of the medium and enabled the coordination of the ligand under these conditions. In previous studies, a 1 M NaOH solution was also used for the preparation of [(Co, Ni or Zn)(I-hip)$_2$(ACV)$(H_2O)_3$] [49, 50]. Copper(II) chloride dihydrate was used, and a ligand-containing complex compound with $CuCl_2$ was obtained [51].



Scheme 1. Synthesis of Cu(II) and Zn(II) mixed-ligand complexes

The yield of the obtained complex was approximately 80–85%, demonstrating the efficiency of the proposed synthesis method. The procedure was reproducible and suitable for the preparation of the Cu(II)–ACV–INH mixed-ligand complex.

Synthesis of Zn(II)–Acyclovir–Isoniazid Mixed-Ligand Complex. Analytically pure reagents, namely acyclovir (ACV), isoniazid (INZ), and zinc (II) chloride ($ZnCl_2$), were used as received without further purification. A mixed solvent system of ethanol and distilled water (1:1, v/v) was employed. Acyclovir (ACV, 0.01 mol, 2.25 g) and isoniazid (INZ, 0.01 mol, 1.37 g) were dissolved in 50 mL of the ethanol–water mixture under continuous magnetic stirring until complete dissolution. The pH of the solution was adjusted to 6.5–7.0 by dropwise addition of 0.1 M sodium hydroxide (NaOH). Separately, zinc (II) chloride ($ZnCl_2$, 0.01 mol, 1.36 g) was dissolved in 10 mL of distilled water. The metal salt solution was then added dropwise to the ligand solution under continuous

stirring at 400 rpm. The reaction was carried out under ambient atmospheric conditions. The reaction mixture was heated and maintained at 60 ± 2 °C for 4 h, during which the formation of a white precipitate indicated complex formation. The precipitate was collected by vacuum filtration and washed several times with distilled water followed by ethanol. The final product was dried in a vacuum oven at 50 °C until a constant mass was obtained. The yield of the synthesized Zn(II)–ACV–INZ mixed-ligand complex was 4.12 g (≈82%). The obtained complex was subsequently used for physicochemical characterization.

Methods. In this study, mixed-ligand complexes were synthesized using acyclovir and isoniazid with copper(II) chloride under controlled conditions. The composition and structure of the obtained complexes were characterized by FT-IR spectroscopy and SEM–EDX analysis. Thermal properties and stability were investigated using TGA–DTA.

Results and Discussion

IR analysis. The structural features of the synthesized complexes [$CuCl_2$(ACV)(INH)] and [$ZnCl_2$(ACV)(INH)] were examined by Fourier-transform infrared (FT-IR) spectroscopy using a Shimadzu FT-IR spectrometer (Japan).

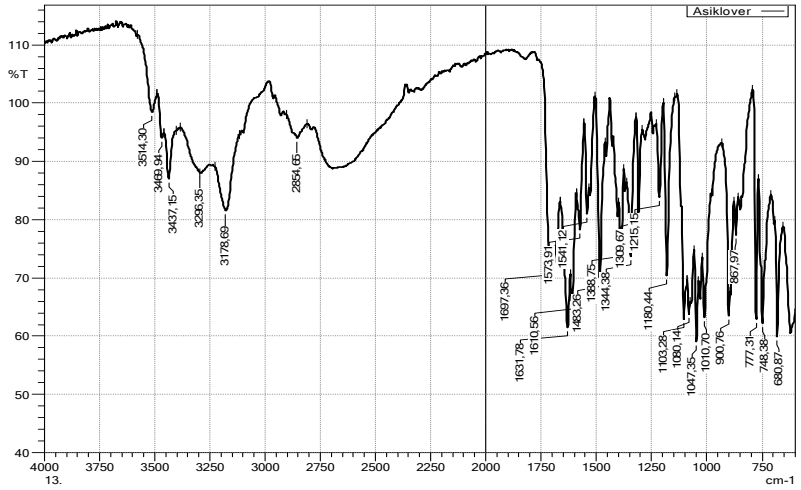


Fig. 1. FT-IR spectrum of the ACV ligand

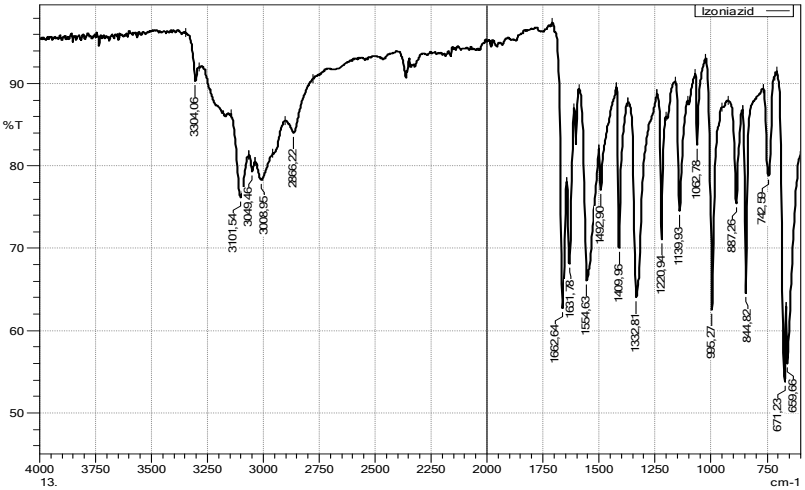


Fig. 2. FT-IR spectrum of isoniazid (INH)

According to the obtained results for ACV ligand (Fig. 1), the absorption bands observed in the region 3178.69–3296.35 cm^{-1} correspond to the O–H stretching vibrations. The bands in the range 3437.15–3514.30 cm^{-1} are attributed to the N–H stretching vibrations. A characteristic absorption

band at 1610.56 cm^{-1} is assigned to the C=O stretching vibration, while the band at 1631.78 cm^{-1} corresponds to the $–NH_2$ bending vibration.

According to the obtained results of FT-IR spectrum for isoniazid (INH), an absorption band observed at 3101.54 cm^{-1} corresponds to the N–H stretching vibration. The band at 1662.64 cm^{-1} is assigned to the C=O stretching vibration, while the band at 1631.78 cm^{-1} corresponds to the scissoring (bending) vibration of the $–NH_2$ group (Fig. 2).

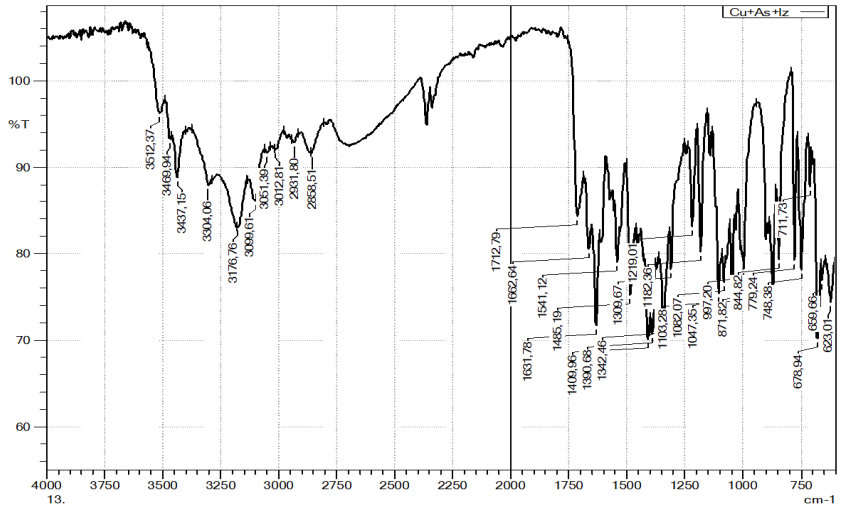


Fig. 3. FT-IR spectrum of the $[CuCl_2(ACV)(INH)]$ complex

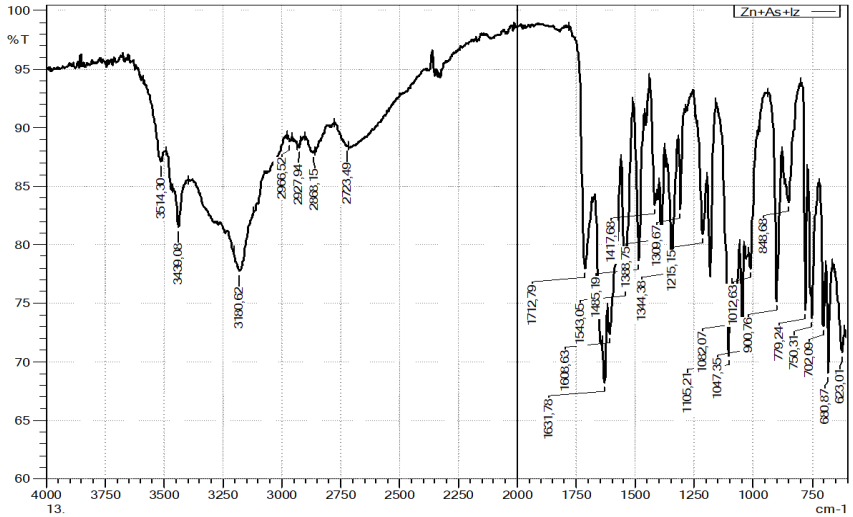


Fig. 4. FT-IR spectrum of the $[ZnCl_2(ACV)(INH)]$ complex.

According to the FT-IR analysis, the absorption bands observed at 3176.76 cm^{-1} and 3180.62 cm^{-1} for the Cu(II) and Zn(II) complexes, respectively, are attributed to the O–H stretching vibrations. The bands at 3437.15 cm^{-1} and 3439.08 cm^{-1} correspond to the N–H stretching vibrations. The characteristic bands at 1631.78 cm^{-1} are assigned to the C=O stretching vibration, while the bands at 1541.12 cm^{-1} and 1543.95 cm^{-1} correspond to the scissoring (bending) vibrations of the $–NH_2$ group. In addition, the bands observed at 659.66 cm^{-1} and 623.01 cm^{-1} for the first complex are attributed to M–O and M–N vibrations, respectively. For the second complex, the corresponding bands appear at 680.87 cm^{-1} and 623.01 cm^{-1}, indicating the presence of metal–nitrogen coordination bonds (Fig.s 3 and 4, Table 1).

Table 1. FT-IR spectral analysis of the ligands and the synthesized complexes

Vibrational frequencies in the FT-IR spectra (cm^{-1})				Bond assignment
ACV	INH	$[CuCl_2(ACV)(INH)]$	$[ZnCl_2(ACV)(INH)]$	

3178.69-3296.35	-	3176.76	3180.62	OH
3437.15-3514.30	3101.54	3437.15	3439.08	NH
1610.56	1662.64	1631.78	1631.78	>C=O
1631,78	1631.78	1541.12	1543.95	NH_2
-	-	659.66	680.87	Me-O
-	-	623.01	623.01	Me-N

The thermogravimetric analysis (TGA). The thermogravimetric analysis (TGA) of the synthesized complex crystal indicates that the TG curve exhibits three main stages of mass loss in Fig. 5.

Fig. 5. Thermal analysis of the Cu(II) complex

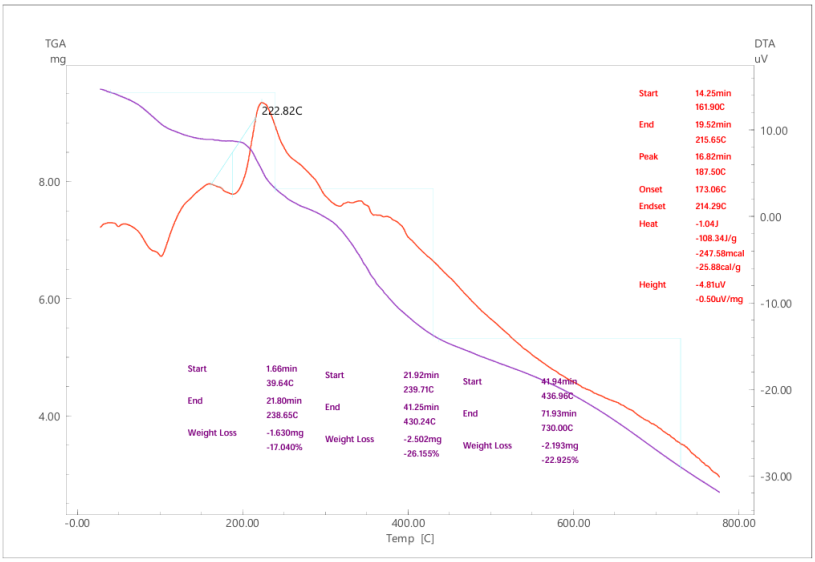


Fig. 6. Thermal analysis of the Zn(II) complex

The first mass-loss stage occurs in the temperature range of 37.23–296.60 °C, the second stage in the range of 296.60–494.65 °C, and the third stage in the range of 497.47–744.51 °C. The first mass loss amounts to 3.256 mg (47.64%), which can be attributed to the removal of moisture and the initial decomposition of the complex structure. The second mass loss of 1.079 mg (15.79%) corresponds to the release of the isoniazid (INH) ligand from the complex. The third mass loss of

1.385 mg is associated with the decomposition and removal of the acyclovir (ACV) ligand. At the final stage of the thermal decomposition process, $CuCl_2$ is assumed to remain as the residual product in Fig. 6.

The thermogravimetric analysis (TGA) of the synthesised complex crystal indicates that the TG curve exhibits three main stages of mass loss. The first mass-loss stage occurs in the temperature range of 39.64–238.65 °C, the second in 239.71–430.24 °C, and the third in 436.96–730.00 °C. The first mass loss of 1.630 mg (17.04%) is attributed to the removal of moisture and the initial decomposition of the complex structure. The second mass loss of 2.502 mg (26.16%) corresponds to the release of the isoniazid (INH) ligand from the complex. The third mass loss of 2.193 mg is associated with the decomposition and removal of the acyclovir (ACV) ligand. At the final stage of the thermal decomposition process, $ZnCl_2$ is assumed to remain as the residual product. In addition, an exothermic effect was observed at 222.8. A similar mass reduction occurred in the complex compounds of the acyclovir ligand [52].

SEM-EDT Analysis. The morphological structure and elemental composition of the synthesised [$CuCl_2$(ACV)(INH)] and [$ZnCl_2$(ACV)(INH)] complex were investigated using scanning electron microscopy (SEM) and energy-dispersive X-ray (EDX) analysis. The analysis was performed using a JEOL JSM-IT200 scanning electron microscope. The samples were prepared under a vacuum of 10^{-4} Pa, and electrical conductivity was ensured by coating the sample surface with a thin layer of gold (Au).

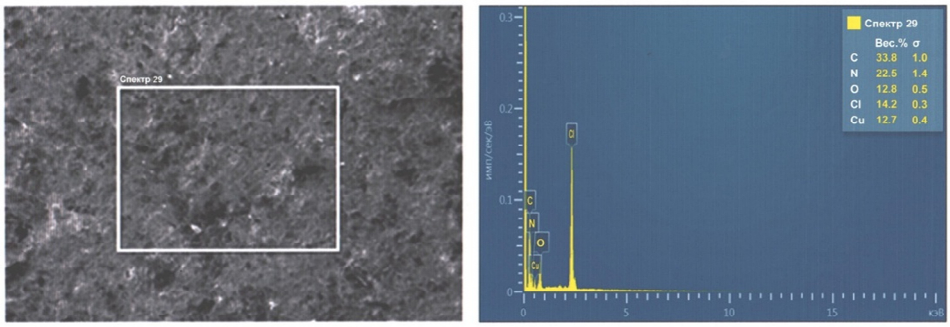


Fig. 7. SEM image (a) and EDX elemental analysis (b) of the [$CuCl_2$(ACV)(INH)] complex

The obtained SEM micrograph (Fig. 7a) revealed that the complex consists of aggregated particles with an inorganic–organic hybrid nature. The particle sizes were mainly in the range of 0.5–2.0 μm, and the surface exhibited a heterogeneous and microporous structure. Such a microporous morphology may be attributed to coordination interactions between the metal center and the ligand molecules during the crystallization process. The elemental composition of the complex was determined by EDX analysis (Fig. 7b).

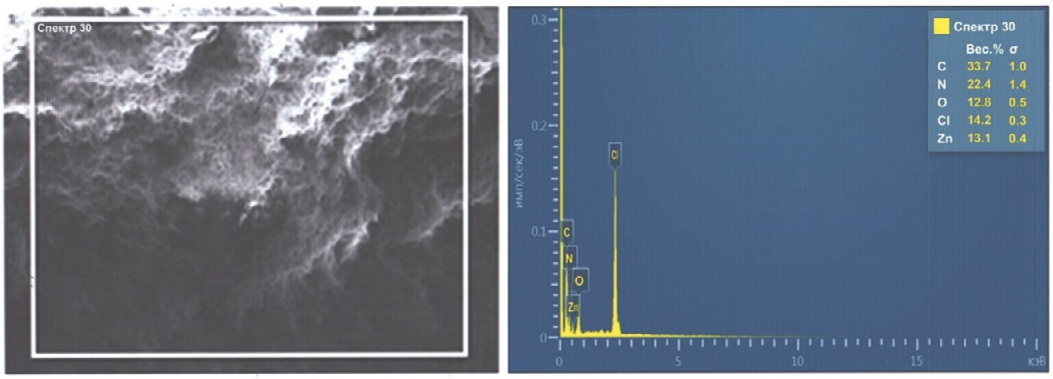


Fig. 8. SEM image (a) and EDX spectrum (b) of the [$ZnCl_2$(ACV)(INH)] complex

The spectrum showed characteristic peaks corresponding to C, N, O, Cl, and Cu, confirming that the elemental composition is consistent with the proposed molecular formula of the complex. The elemental contents were determined as follows: C (33.8%), N (22.5%), O (12.8%), Cl (14.2%), and Cu (12.7%). The obtained values are in good agreement with the calculated theoretical values, indicating the presence of two organic ligand molecules and two chloride ions in the coordination structure. Based on these results, the empirical formula of the synthesized complex was proposed as $[CuCl_2(C_{14}H_{18}N_8O_4)]$. The obtained SEM–EDX results are in good agreement with previously reported data for Cu(II) coordination complexes in the literature.

The obtained SEM micrograph (Figure 8a) shows that the synthesised complex consists of aggregated particles with an inorganic–organic hybrid nature. The particle sizes are mainly in the range of 0.5–2.0 μm, and the surface exhibits a heterogeneous and microporous morphology. Such a structure may result from coordination interactions between the ligand molecules and the metal centre during the crystallisation process. The elemental composition was determined by EDX analysis (Figure 8b). The spectrum shows characteristic peaks corresponding to C, N, O, Cl, and Zn, confirming that the elemental composition is consistent with the proposed formula. The elemental contents were determined as C (33.7%), N (22.4%), O (12.8%), Cl (14.2%), and Zn (13.1%). These values are in good agreement with the calculated theoretical data, indicating the presence of two organic ligand molecules and two chloride ions in the complex structure. Accordingly, the empirical formula of the complex was proposed as $[ZnCl_2(C_{14}H_{18}N_8O_4)]$.

Chromatomass-Spectrum Analysis. The molecular composition and purity of the synthesized mixed-ligand complex $[CuCl_2(ACV)(INH)]$ were investigated using liquid chromatography–mass spectrometry (LC–MS), which allows reliable confirmation of molecular weight and compound homogeneity. LC–MS analysis was performed using a 6420 Triple Quadrupole mass spectrometer. The powdered complex was dissolved in dimethyl sulfoxide (DMSO) and subjected to chromatographic separation before mass spectrometric detection.

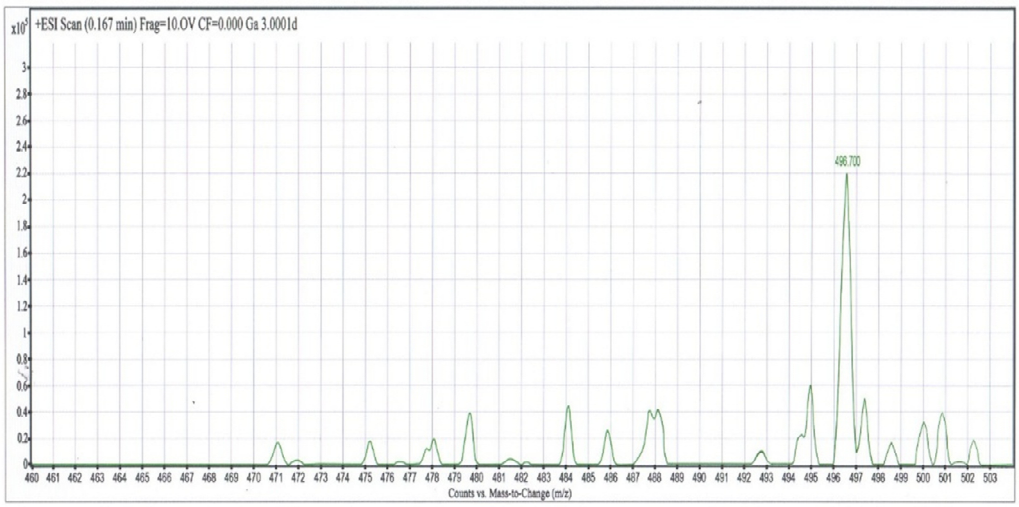


Fig. 9. LC–MS chromatogram and mass spectrum of the $[CuCl_2(ACV)(INH)]$ complex

The LC–MS chromatogram of the $[CuCl_2(ACV)(INH)]$ complex exhibited a single, sharp, and symmetrical peak, indicating the absence of free ligands, metal salts, or by-products and confirming the chemical homogeneity of the compound. The mass spectrum showed a dominant molecular ion peak at m/z = 496.79, in excellent agreement with the calculated molecular weight of the proposed complex. No significant fragment or impurity-related ions were observed, suggesting that the coordination framework remains intact under the applied ionization conditions. These results, consistent with FT–IR and SEM–EDX analyses, confirm the successful synthesis, high purity, and structural integrity of the $[CuCl_2(ACV)(INH)]$ complex.

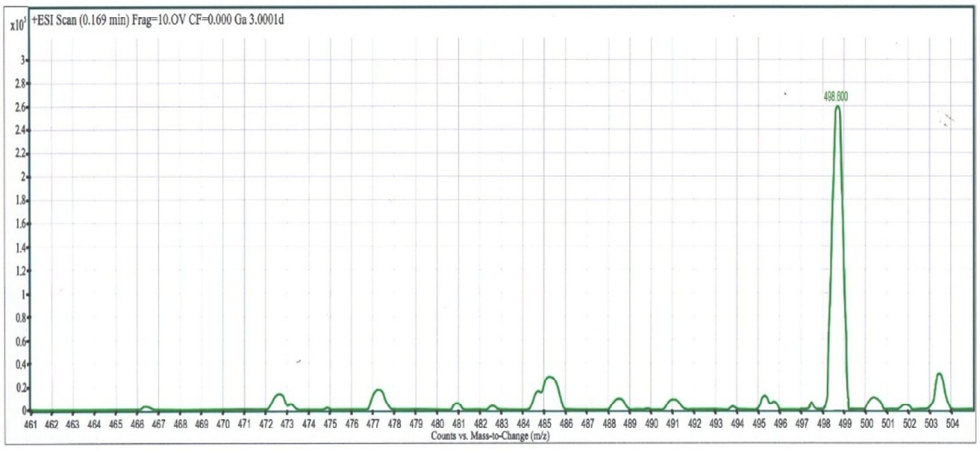


Fig. 10. LC–MS spectrum of the [$ZnCl_2$(ACV)(INH)] complex

The molecular composition and purity of the synthesized mixed-ligand complex [$ZnCl_2(C_{14}H_{18}N_8O_4)$] were investigated using liquid chromatography–mass spectrometry (LC–MS), which allows reliable confirmation of molecular weight and homogeneity. LC–MS analysis was performed using a 6420 Triple Quadrupole mass spectrometer. The powdered complex was dissolved in dimethyl sulfoxide (DMSO) and subjected to chromatographic separation before mass spectrometric detection, ensuring effective separation and minimal interference from impurities. The chromatogram exhibited a single, sharp, symmetrical peak, indicating chemical homogeneity. The mass spectrum showed a dominant molecular ion peak at m/z = 498.63, consistent with the calculated molecular weight. No significant fragment or impurity-related ions were observed, suggesting that the coordination framework remains intact during ionization.

Conclusion

In this study, mixed-ligand Cu(II) and Zn(II) complexes with acyclovir (ACV) and isoniazid (INH) were successfully synthesized under mild conditions, yielding [$CuCl_2$(ACV)(INH)] and [$ZnCl_2$(ACV)(INH)] in good yields. Spectroscopic analyses (FT-IR) confirmed that coordination occurs via carbonyl oxygen and azomethine nitrogen atoms, forming stable chelate structures. Thermogravimetric and differential thermal analyses (TGA–DTA) demonstrated that the complexes possess significant thermal stability, with multistep decomposition above 250 °C. SEM–EDX investigations revealed that both complexes consist of aggregated microparticles with an inorganic–organic hybrid morphology, and the elemental composition closely matches the proposed molecular formulas. LC–MS analysis further confirmed the molecular weights, chemical homogeneity, and structural integrity of the complexes, with no detectable free ligands or by-products. These findings indicate that acyclovir and isoniazid can effectively form stable mixed-ligand coordination complexes with transition metal ions.

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