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Article

Synthesis, Spectroscopic Characterization, And Antibacterial Activity Of Zn(Ii) Complexes Derived From A Thiazole-Containing Chalcone And Bidentate Phosphine Ligandsname

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Abstract: For this experiment, a novel thiazole-based chalcone ligand named MCT, that is, (Z)-1-(4-chlorophenyl)-3-(4-methylthiazol-5-yl)prop-2-en-1-one, was used to synthesize three complexes of Zn(II), $[Zn(MCT)_2]Cl_2$, $[Zn(MCT)(dppe)]Cl_2$, and $[Zn(MCT)(dppp)]Cl_2$. Characterization of the synthesized complexes was done via FT-IR, ^{1}H-NMR, and $^{31}P\{^1H\}$-NMR spectroscopies. According to the obtained FT-IR results, it was found that Zn(II) is coordinated to the MCT ligand by oxygen of the carbonyl group and the sulfur donor atom, while according to the $^{31}P\{^1H\}$-NMR spectra of complexes containing phosphine ligands, the phosphorus atoms are chemically equivalent in the complex structures because only one resonance for each phosphorus atom was revealed. In antibacterial screening assays against Escherichia coli and Staphylococcus aureus, different antibacterial activities dependent on the composition and concentration of complexes were detected. Thus, $[Zn(MCT)(dppe)]Cl_2$ was found to exhibit high activity against E. coli, and whereas $[Zn(MCT)(dppp)]Cl_2$ exhibited the most pronounced activity against S. aureus. These findings demonstrate that incorporation of bidentate phosphine ligands into the Zn(II) coordination sphere significantly influences the structural and antibacterial properties of the resulting complexes.

Keywords: Zinc(II) complexes; Thiazole-containing chalcone; Bidentate phosphine ligands; NMR spectroscopy; Antibacterial activity.

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1. Introduction

Chalcones represent an important class of organic compounds which are members of flavonoids possessing α,β-unsaturated carbonyl system which confer on them unique chemical and biological properties [1,2]. The structural versatility of chalcones and the ability to substitute with various functional groups and donor atoms make the derivatives of chalcones very interesting in the field of organic and coordination chemistry [3]. The particular relevance of chalcones in coordination chemistry is related to their ability to act as ligands due to the presence of donor atoms, especially the carbonyl oxygen and even nitrogen and sulfur atoms in case of heterocyclic rings in their structure; this property allows formation of stable complexes with a variety of coordination geometries with metal ions [4]. Coordination with metal ions in the complexes of chalcones leads to the significant changes in the electronic, spectroscopic and physicochemical characteristics of these complexes along with change in their biological activity in comparison with the unsubstituted ligands [5]. Biologically, chalcones and their derivatives have attracted much attention owing to their wide pharmacological activity such as antibacterial, antifungal, antiviral, antioxidant and anticancer activity [6]. RSeveral recent researches have shown that chalcone analogs could suppress the development of a number of both Gram-positive and Gram-negative bacteria, and at the same time the type and location of substitution in the chalcone skeleton are crucial for the antibacterial activity of these compounds [7,8]. Phosphines are considered to be one of the most valuable ligands in the chemistry of metal compounds, especially transition elements, as the phosphorus atom demonstrates excellent donating capability of an electron pair and formation of stable coordination bond [9].

This can be achieved by altering the electronic and sterical characteristics by changing substituent groups bound to the phosphorus atom [10]. The role of bidentate phosphines including dppe and dppp should be separately mentioned because such ligands could bind the two phosphorus donor atoms to metal centers to form stable complex due to the wide application in coordination chemistry and homogeneous catalysis [11].

2. Methodology

2.1. Materials: All chemicals used in this work were purchased from Sigma-Aldrich and BDH and used as is without any further purification, with the exception of the MCT binder [((Z)-1-(4-chlorophenyl)-3-(4-methylthiazol-5-yl)prop-2-en-1-one] which was synthesized in the laboratories of the Chemistry Department, Tikrit University

2.2. Synthesis of $[Zn(MCT)_2]Cl_2$ [12]: A solution containing (0.004 moles, 1.54 grams) of MCT ligand ((Z)-1-(4-chlorophenyl)-3-(4-methylthiazol-5-yl)prop-2-en-1-one) in 10 mL of ethanol and (0.002 moles, 0.27 grams) of zinc chloride in 10 mL of ethanol was synthesized. The solution obtained was then stirred continuously at 60 °C for 2 hours, resulting in the formation of a white solid precipitate. The solution was cooled to room temperature, and the resulting white solid was filtered off and washed several times with ethanol. Further recrystallization of the crude product from ethanol gave white crystals (1.45 grams, 80% yield).

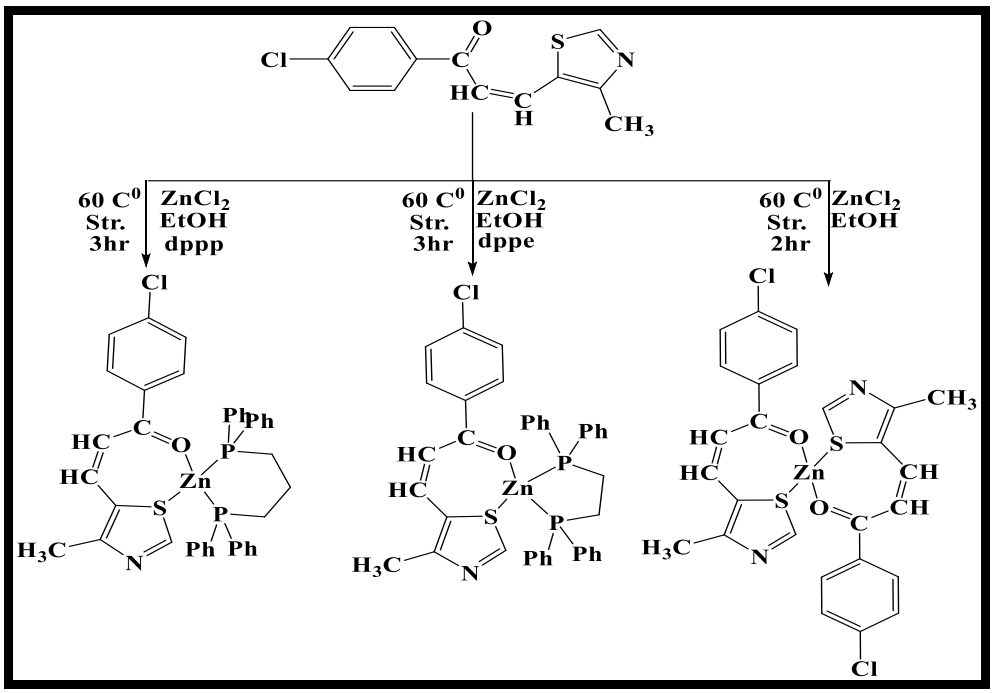
2.3. Synthesis of $[Zn(MCT)(dppe]Cl_2$ [13]: A mixture was prepared of (0.001 mol, 0.2637 g) of the MCT ligand dissolved in 10 mL of ethanol and (0.001 mol, 0.1363 g) of ZnCl2 dissolved in 10 mL of ethanol. This mixture was heated to 60 °C while stirring for 1 h. Further addition of (0.001 mol, 0.3984 g) of dppe dissolved in 10 mL of ethanol, followed by continuing the reaction in ethanol at 60 °C while stirring for an additional 2 h, where a light yellow precipitation took place. After cooling to room temperature, the precipitate was isolated by filtration, purified with ethanol, and recrystallized from ethanol to produce light-yellow crystals (0.591 g, 74% yield).

The complex $[Zn(MCT)(dppp)]Cl_2$ was prepared using the same procedure described above. It was isolated as orange crystals in 76% yield.

2.3. Evaluation of Biological Efficacy: Antibacterial efficacy of the prepared zinc(II) compounds was investigated against the Gram-negative bacterium Escherichia coli and Gram-positive bacterium Staphylococcus aureus. Mueller-Hinton agar (MHA) medium was prepared following the standard procedure; then sterilized and poured into the sterile Petri dishes [14]. Bacterial suspensions were prepared freshly, and their turbidity was adjusted to correspond to the value of approximately 1.5×10^8 CFU/mL. The solutions of tested complexes were prepared in DMSO, and its dilutions were carried out to obtain the concentrations of 0.1, 0.01, and 0.001 mg/mL [15]. The antibacterial efficacy was investigated using agar well diffusion method. Standardized bacterial suspensions were plated uniformly on the surface of MHA plates, then wells were prepared in the inoculated agar [16]. 40 μL of the prepared solution of each compound was put into wells [17]. Plated agar was incubated at 37°C for 24-48 hours. Zones of inhibitions around the wells were measured after incubation time in millimeters [18]. The diameters of inhibition zones were used as an assessment of antibacterial efficiency of the synthesized compounds. The standard antibiotic used was Amoxicillin.

1. Result And Discussion

The synthesized complexes were prepared according to the procedure illustrated in Scheme 1.



Scheme 1. All synthesized complexes.

3.1. FT-IR spectrum:

The IR spectrum of the $[Zn(MCT)_2]Cl_2$ complex revealed an absorption peak at 3153 cm^{-1}, which belonged to the stretching vibration of the olefinic C–H bond. The absorption peak at 3064 cm^{-1} corresponded to the stretching vibration of the aromatic C–H bond. The two peaks at 2850 and 2920 cm^{-1} corresponded to the symmetric and asymmetric stretching vibrations of the aliphatic C–H bonds, respectively. Also, the IR spectrum contained the medium intensity peak at 1644 cm^{-1} due to C=O, which experienced noticeable shift towards low-wavenumbers, indicating its participation in coordination interaction with a metal ion. Medium intensity peak at 1600 cm^{-1} corresponded to the stretching vibration of the olefinic C=C bond. Further, the two medium intensity peaks at 1460 and 1508 cm^{-1} corresponded to the symmetric and asymmetric stretching vibrations of the aromatic C=C bonds, respectively. The peak at 1253 cm^{-1} was due to C-N, while the absorption peak at 831 cm^{-1} was ascribed to C-S group [19]. The above-mentioned peaks are shown in Figure 1.

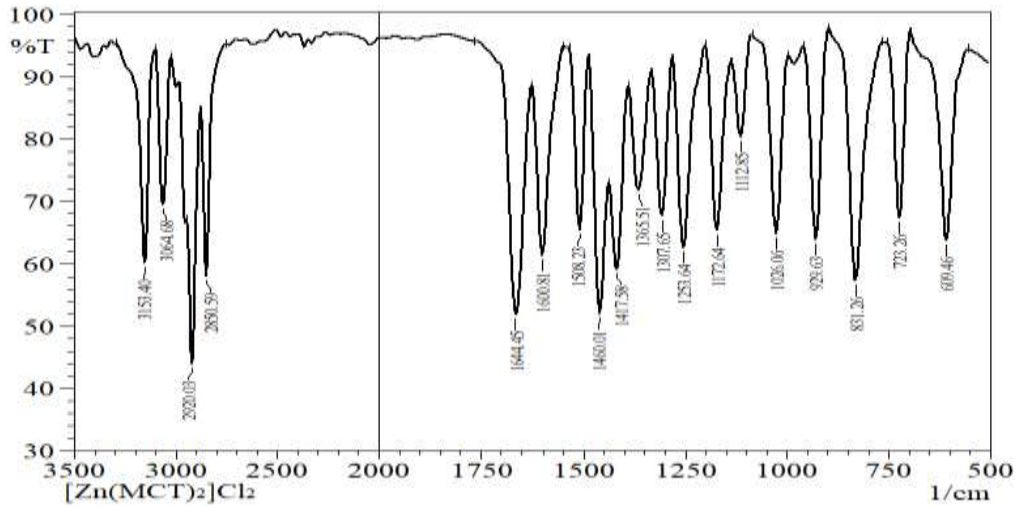


Figure 1: FT-IR from $[Zn(MCT)_2]Cl_2$

FT-IR spectra of the $[Zn(MCT)(dppe]Cl_2$ complex revealed that the spectrum has a peak at 3159 cm^{-1} for the stretching vibration of the olefinic C–H bond. Also, there is another peak located at 3072 cm^{-1} due to the stretching vibration of the aromatic C–H bond. In addition, there are two peaks at 2850 and 2920 cm^{-1} due to the symmetric and asymmetric stretching vibrations of the aliphatic C–H bonds, respectively. There is a

medium intensity peak at 1652 cm^{-1} due to C=O, where a downshift in wavenumber occurred after complex formation due to the coordination of the carbonyl oxygen with Zn(II). In addition, there is another peak at 1606 cm^{-1} due to the stretching vibration of the olefinic C=C bond. Finally, there are two peaks of medium intensity located at 1481 and 1581 cm^{-1} due to the stretching vibrations of the aromatic C=C bond. Moreover, another band at moderate intensity was identified at 1433 cm^{-1} which was assigned to be stretching vibrations for the P-Ph group. The band at 1255 cm^{-1} was found to correspond to the C-N stretching vibration. Bands were also noticed in the spectra around 1174 cm^{-1} and 795 cm^{-1} and the stretching vibrations for the P-CH were suggested for these bands. In addition, another two absorption bands were found at 657 and 586 cm^{-1} which were considered to be the stretching vibrations for Zn-O and Zn-S vibrations respectively. As show in Figure 2.

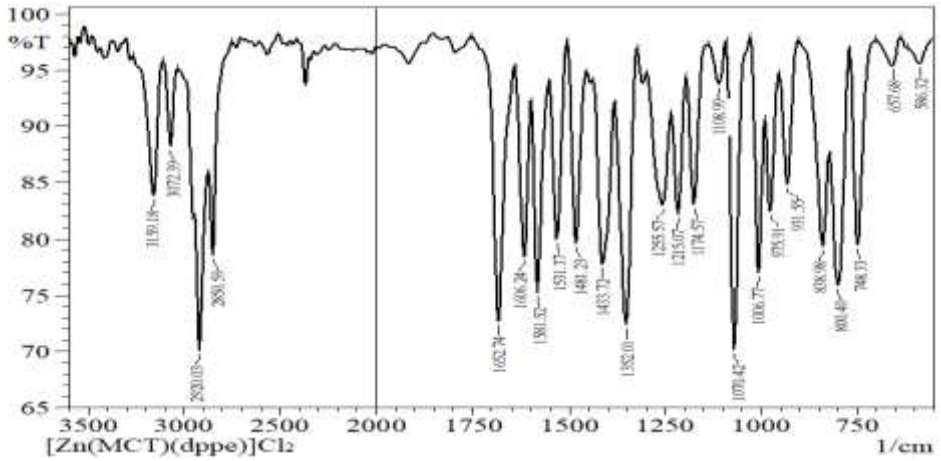


Figure 2: FT-IR from [Zn(MCT))(dppe)]Cl_2

The FT-IR spectra of the synthesized [Zn(MCT)(dppp)]Cl_2 complex showed absorption in the region of 3188 cm^{-1} corresponding to the stretching vibration of the olefinic C-H bond. At 3080 cm^{-1}, there was a band for the stretching vibration of the aromatic C-H bond. Bands for the stretching vibrations of the symmetric and asymmetric aliphatic C-H were detected at 2875 and 2947 cm^{-1}, respectively. The medium-intensity band in the range of 1647 cm^{-1} is for the C=O group; the band shows a shift to lower frequency due to complexation, implying that the carbonyl oxygen participates in coordination with Zn(II). The medium-intensity band in the range of 1604 cm^{-1} is for the stretching vibration of the olefinic C=C bond. The medium-intensity bands for the stretching vibrations of the aromatic C=C bonds were at 1490 and 1548 cm^{-1}.A medium intensity band appeared at 1428 cm-1, which was due to the stretching vibration of the P-Ph bond. The absorption band seen at 1242 cm-1 was attributed to the C-N stretching vibration. In addition to that, absorption bands were seen at 1178 and 825 cm-1, which corresponded to P-CH stretching vibration. Moreover, absorption bands seen at 609 and 503 cm-1 were attributed to the stretching vibrations of the Zn-O and Zn-S bonds, respectively, indicating that the coordination of the ligand was through oxygen and nitrogen donors with zinc(II) [21]. As shown in Figure 3.

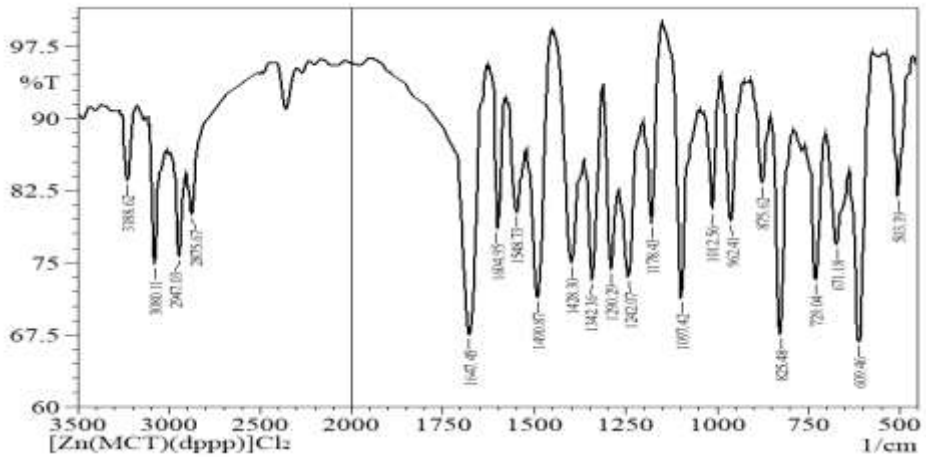


Figure 3: FT-IR from [Zn(MCT))(dppp)]Cl_2

3.2. ^{1}H-NMR spectrum:

The ^{1}H-NMR spectrum of $[Zn(MCT)_2]Cl_2$, where DMSO-d^6 was used as the solvent, had a singlet resonance at δ = 8.15 ppm and it belonged to the proton of the N=CH group in the thiazole ring. Moreover, the spectrum had a multiplet between δ = 7.27 – 7.71 ppm which represented the aromatic ring protons. In addition, the doublets observed at δ = 6.92 and 6.89 ppm belong to the (=HC) proton beside the thiazole ring while other doublets at δ = 6.79 and 6.77 ppm were the proton of O=C–CH beside the carbonyl group. The spectrum had also a singlet at δ = 3.52 ppm which is attributed to the proton of the CH_3 group while the signal at δ = 2.48 ppm was attributed to the residual protons of the DMSO-d^6 solvent [22]. These results are presented in Figure 4 below.

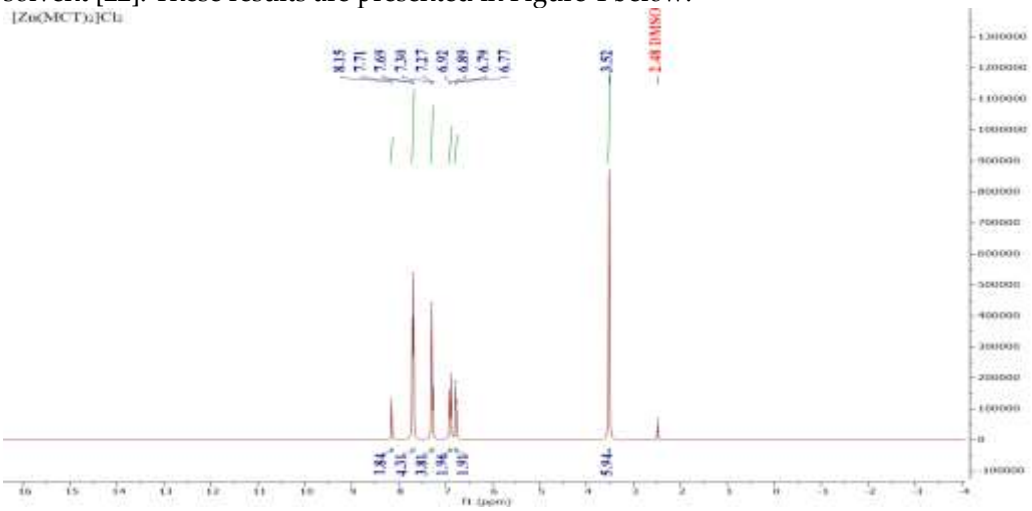
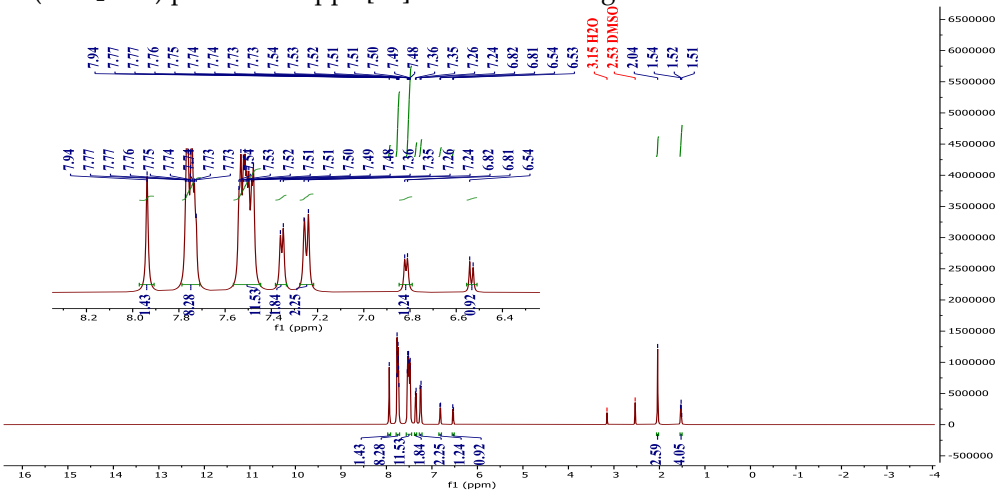


Figure 4: ^{1}H-NMR spectra of $[Zn(MCT)_2]Cl_2$

Spectrum for $[Zn(MCT)(dppe)]Cl_2$ revealed singlets of an integration of one proton with a chemical shift value of 7.94 ppm, indicating a presence of N=CH protons from the thiazole group. Singlet proton peak, having three protons in its integration, was seen at 2.04 ppm, which corresponded to the presence of protons from CH_3 group. Doublet peaks with an integration of one proton were identified at the chemical shift values of (6.53, 6.54) and (6.81, 6.82) ppm and corresponded to the presence of protons of olefinic group, namely =CH. Multiplets were found in a range of 7.48-7.77 ppm for 20 protons and corresponded to phenyl groups' protons within the phosphine group, dppe. Doublet peaks were found at chemical shifts of (7.24, 7.26) and (7.35, 7.36) ppm; two protons in each of these doublets indicated that they correspond to aromatic group's C-H protons. Triplet peak was seen at 1.51-1.54 ppm for an integration of four protons and was related to ($2CH_2$-CH) protons in dppe [23]. As shown in Figure 5.



In the ^{1}H-NMR spectrum of $[Zn(MCT)(dppp)]Cl_2$ complex, there was a singlet at 7.71 ppm with integration of one proton due to the proton at N=CH in the thiazole ring. There were also multiplets with integration of 20 protons between chemical shifts 7.77-7.20 ppm that were associated with the phenyl ring protons in the phosphine (dppp) ligand. Two doublets with integration of two protons each appeared at chemical shifts 7.42-7.40 ppm and 7.29-7.27 ppm; these were associated with the C-H protons of the 4-chlorophenyl ring. Two doublets with integration of one proton each appeared at

chemical shifts 6.96-6.92 ppm and 6.66-6.62 ppm, and were associated with (=CH) protons of the unsaturated system of the ligand. Singlet with integration of three protons was found at chemical shift 2.03 ppm which were associated with the CH_3 protons of the thiazole ring. Other signals found between chemical shifts 2.31-2.02 ppm with integration of six protons were associated with the CH_2 protons of the dppp ligand. Also a singlet at 2.49 ppm was assigned to the remaining proton of the DMSO-d^6 solvent [24], as shown in Figure 6.

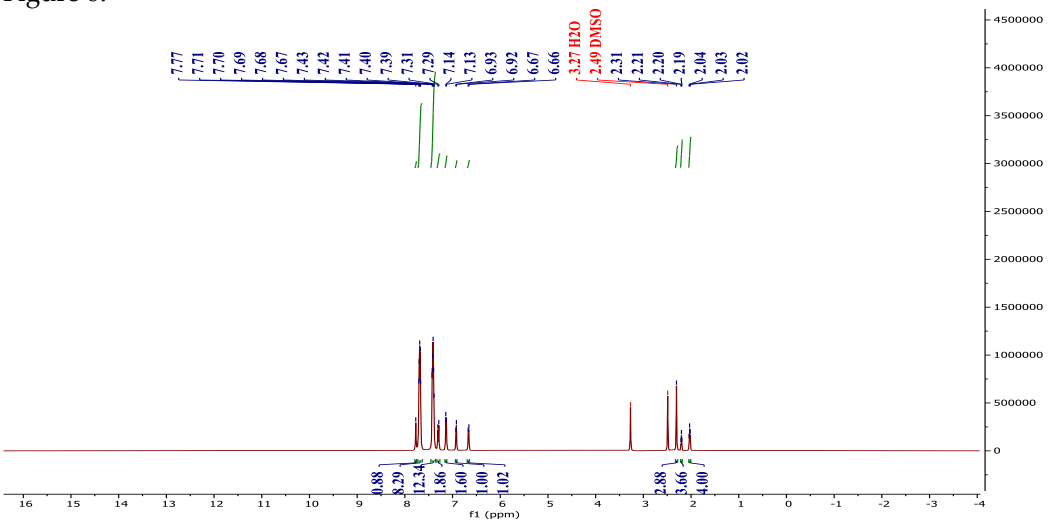


Figure 6: ^{1}H-NMR spectra of [Zn(MCT)(dppp)]Cl_2

3.3. ^{31}P-{^{1}H}-NMR

In the case of the [Zn(MCT)(dppe)]Cl_2 complex, the ^{31}P{^{1}H}-NMR spectrum provided a single peak at δ = 44.79 ppm due to chemical equivalence of both phosphorus atoms in the studied complex [25]. Single phosphorus peak is an indication of chemical equivalence of both phosphorus atoms and confirms the existence of only one isomer of the complex [25]. The spectrum is presented in Figure 7.

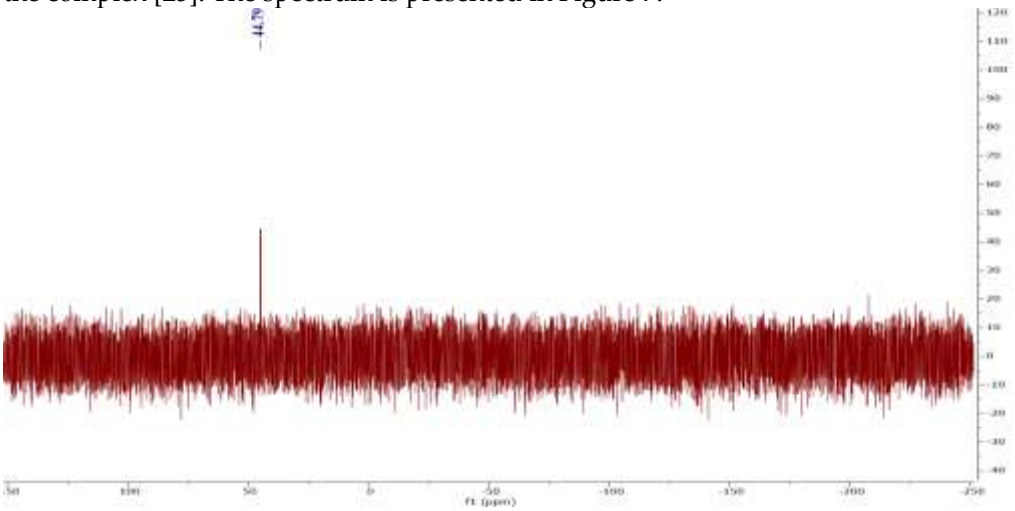


Figure 7: ^{31}P{^{1}H}-NMR spectra of [Zn(MCT)(dppp)]Cl_2

The ^{31}P{^{1}H}-NMR spectrum of [Zn(MCT)(dppp)]Cl_2 showed only one peak at δ = 51.14 ppm because of the equivalence of the two phosphorous atoms in the compound. This means that the chemical environment of the two phosphorous atoms is similar and there is only one type of isomer that can be formed. The ^{31}P{^{1}H}-NMR spectrum is shown in Figure 8.

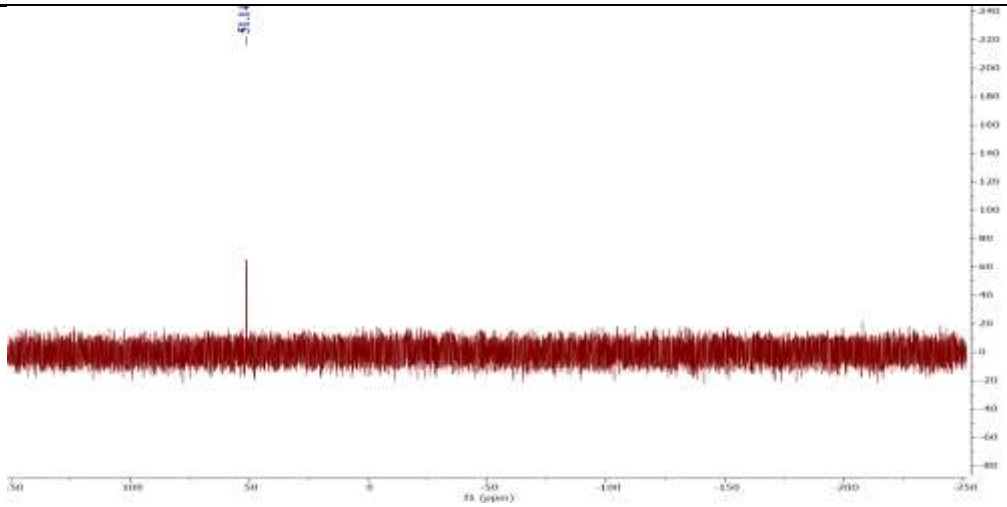


Figure 8: $^{31}P\{^{1}H\}$-NMR spectra of $[Zn(MCT)(dppp)]Cl_2$

3.4. Biological Activity Discussion[27-30]:

The antibacterial activity results, as presented in Table 1, showed that the synthesized zinc(II) complexes exhibited variable inhibitory effects against both Escherichia coli and Staphylococcus aureus. The $[Zn(MCT)_2]Cl_2$ complex showed moderate activity, with inhibition zones ranging from 1.8–2.2 cm against E. coli and 1.0–2.0 cm against S. aureus. In contrast, incorporation of the dppe ligand resulted in a clear enhancement of antibacterial activity, particularly against E. coli, where $[Zn(MCT)(dppe)]Cl_2$ produced inhibition zones ranging from 3.5–3.9 cm, as shown in Table 1 and Figure 9.

The $[Zn(MCT)(dppp)]Cl_2$ complex exhibited pronounced activity against both bacterial species, producing the highest inhibition zone against E. coli (4.0 cm) at a concentration of 0.1 mg/Ml, while inhibition zones against S. aureus were 3.5, 2.9, and 2.9 cm at concentrations of 0.1, 0.01, and 0.001 mg/Ml, respectively, as shown in Table 1 and Figure 10. The enhanced activity of the phosphine-containing complexes may be associated with changes in the electronic and lipophilic properties of the Zn(II) coordination environment, which may facilitate interactions between the complexes and bacterial cell membranes and biological targets.

Generally, $[Zn(MCT)(dppe)]Cl_2$ had good activity on E. coli, while $[Zn(MCT)(dppp)]Cl_2$ was highly active on S. aureus. These results suggest that the presence of phosphine ligands in the coordination environment of the Zn(II) ion can greatly affect their antibacterial activities.

Table 1. Inhibition zones (cm) of complexes.

Comp. No.	Conc. mg/mL	*E. coli*			*S. aureus*		
		0.1	0.01	0.001	0.1	0.01	0.001
$[Zn(MCT)_2]Cl_2$		2.0	2.2	1.8	2.0	2.0	1.0
$[Zn(MCT)(dppe)]Cl_2$		3.5	3.9	3.8	2.1	2.3	0.9
$[Zn(MCT)(dppp)]Cl_2$		4.0	2.0	2.0	3.5	2.9	2.9
Amoxicillin		3.6	2.5	1.4	3.0	2.1	1.4

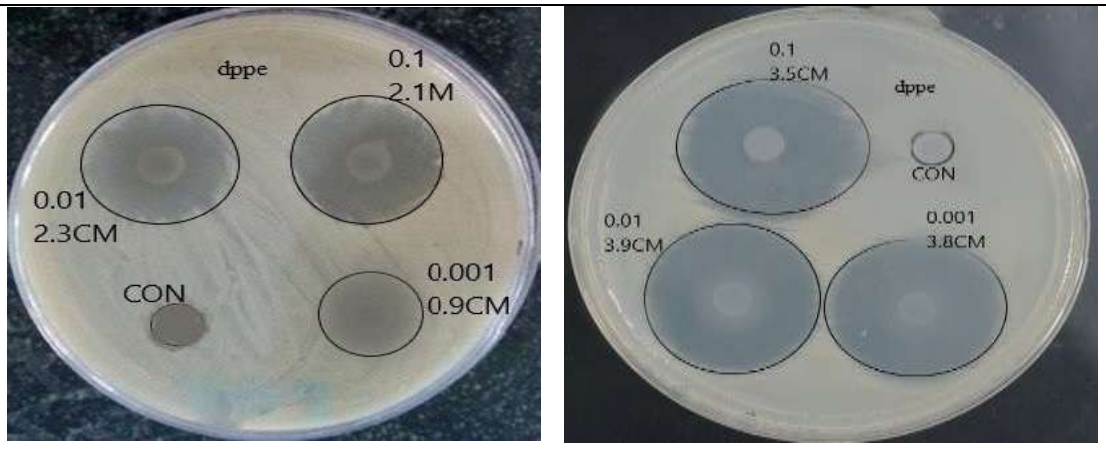


Figure 9. Inhibitory activity of the $[Zn(MCT)(dppe)]Cl_2$ complex against bacteria.

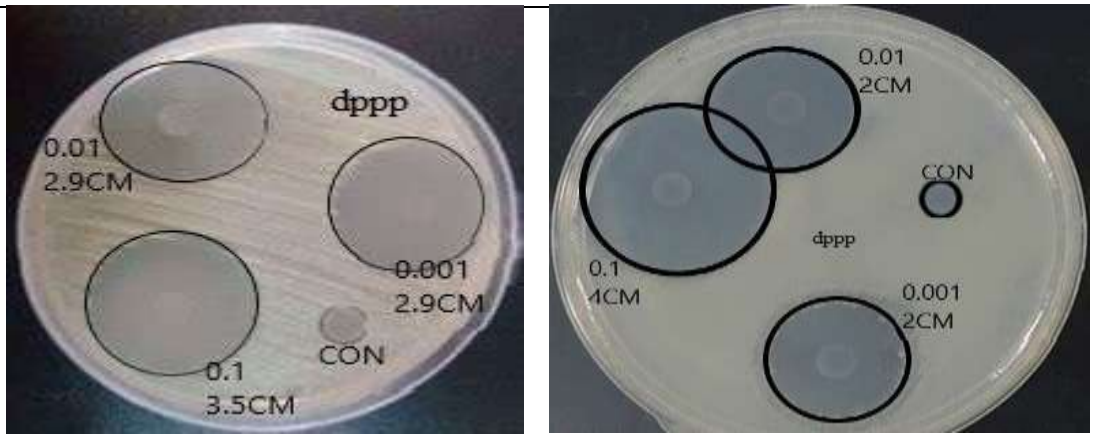


Figure 10. Inhibitory activity of the [Zn(MCT)(dppp)]Cl_2 complex against bacteria.

4. Conclusions

In this paper, successful preparation of three complexes of Zn(II), [Zn(MCT)?]Cl?, [Zn(MCT)(dppe)]Cl?, and [Zn(MCT)(dppp)]Cl?, where the ligand was MCT, was achieved. Spectroscopic analysis by means of FT-IR, ^{1}H-NMR, and ^{31}P{^{1}H}-NMR confirmed the expected structures of the complexes and proved coordination of the ligand through its carbonyl oxygen and sulfur donor atoms. Single ^{31}P{^{1}H}-NMR resonance in complexes containing phosphine proved equivalent chemical environment of phosphorus. Antimicrobial evaluation of the new compounds revealed that all the prepared compounds showed antibacterial activity towards both Escherichia coli and Staphylococcus aureus, depending on the structure and concentration of the complexes. Significant change in antibacterial action in response to introduction of dppe and dppp into the compound was noticed. In particular, the complex [Zn(MCT)(dppe)]Cl? demonstrated very strong antibacterial action against E. coli, and [Zn(MCT)(dppp)]Cl? was the most active complex towards S. aureus.

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