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BIOACTIVITY STUDY OF NEW SYNTHESIZED METALLIC COMPLEXES OF AROYL HYDRAZONE LIGAND

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ABSTRACT

Synthesis of new Ligand has been prepared from aroyl hydrazone derivatives and characterize it spectroscopically (I.R., ^{1}HNMR.). Synthesis of the four new complexes to Nuclear Metallic Cu (II), Hg (II), Cd (II), Zn (II) and prepared ligand and studying their characteristics them spectroscopically (I.R.,^{1}H-NMR.) and the Molar Conductivity with the accurate quantitative elemental and metallic analysis of these Complexes. And studying bioactivities of the complexes which are prepared to explain the antibacterial activities (by using three genus of Pathogenic bacteria).

Key words: Ligand, Aroyl hydrazone**,** Synthesis**,** Bacteria.

INTRODUCTION

Hydrazones are azomethane compounds that contain the following effective synthetic structure as shown in groups (Aroyl Hydrazone) that connected to the in the Figure 1a Figure 1b [Belskaya et al., 2010, Salgin-Gökşen et al., 2013] Hydrazones can be distinguished from many types of azimuth compounds such as Oxime and Imine [Narang *et al.*, 2012]. successive nitrogen atoms. The difference in the compensated groups on the carbon and nitrogen peaks of different types leads to the emergence of several functional groups as shown in Figure 1b.

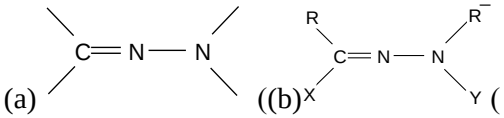


Figure 1a and b

Where :**R** and **R**$^-$= H, Alk, Ar, RCO, X, **X**= H, Alk, Ar, RCO, X. **Y**= H, Alk, Ar, OR, X, SR, CN, SO_2R, NO_2.

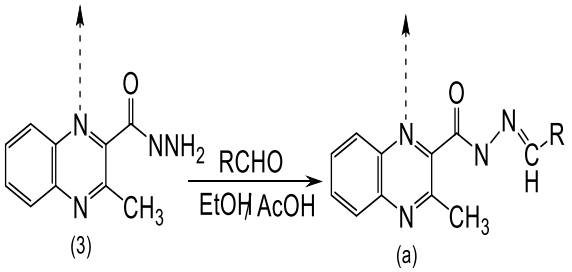
Hydrazones act as multidate legends, especially if hydrazones contain heterogeneous rings. The bond between aliquant and metal ion occurs through nitrogen atoms or by high electrolytic atoms such as sulfur and oxygen [Uppal et al., 2011].

Pathogenic bacteria: It is a microscopic microbial revival with different dimensions and shapes [Maillard et al., 2013]. There are many bacteria that cause diseases in humans such as *Staphylococcus aureus, Salmonella typhi, Proteus mirabilis*. The latter was isolated from cases of Urinary tract infections. The transmission of this bacterium is internal [Kocyigit-Kaymakcioglu et al., 2012].

The methods of work: The methods of preparation can be divided into two main parts:

First: Preparation of hydrazone ligand [Maillard, et al., 2013].

Synthesis of aliquant 3-methyl-2-((3-phenyl-hydrazine carbonyl)-quinoxaline-)4-oxide (a): The preparation process was dissolved of the compound (3)(0.01 mol, 2.18 g) in a mixture of 30 ml of 99% ethanol and 5 ml of acetic acid, then added (0.11 mol, 13.2 g) of O-Hydroxy-p- methyl -benzaldehyde, the mixture lifted for 20 minutes at room temperature with stirring until precipitation, the product was reconstituted by chloroform and petroleum ether (1: 1) as in the following equation.



Second: Preparation of metal complexes Synthesis of complex (A) using Ligand (a) and Cu (II): Add of water-soluble mineral sulfate (0.01 mol, 1.6 g) in (10 ml) water to (0.01 mol, 3.364 g) of aliquant (a) dissolved in (10 ml) of distilled water with solution color change about (85%). The following data give information on the resulting complex. there is no sense. could you clarify more washed with acetone and then distilled water. same proceture was followed in the all four complexes were prepared (Table 1).

Table 1: Physical data of prepared complexes using ligand (a)

Complex number	Duration of stirring hour	Complex color after purification	Melting point (Percent)	Percentage of output	Salt used ($M X_2$)	Oxidation of the metal M	Solvent used for salt and ligand
A	1	Red deposit	207-208	85	$CuSO_4$	Cu (ll)	Distilled water
B	Directly	Brown deposit	203-204	77.5	$HgCl_2$	Hg (ll)	Distilled water
C	1.30	Brown deposit	221-220	76.3	$Cd(Ac)_2$	Cd (ll)	Distilled water
D	1.5	Dark brown deposit	215-214	94.1	$Zn(Ac)_2$	Zn (ll)	Distilled water

Biological Study: The biological efficacy of all prepared complexes was tested with different concentrations (0.1, 1, 10, 30 and 50) / ml and the method of diffusion of the modified disks was used by the global additive 6 & 8. This method is one of the common methods used in laboratory antibiotic testing on four different strains of pathogenic bacteria, including positive for chromosomal species such as Staphylococcus areas and other chromosomal species such as sex Salmonella typhi, Proteus mirabilis.

Preparation Discussion of Ligand 2-(4-like-2- -hydroxy)-hydroxide carbonyl- 3 -methyl-quinoxa- line- 4-oxide (a) : The preparation of Ligand (a) was done by adding 2-hydroxy 4-methyl benzaldehyde to the compound (2-Amino-3-methyl-quinoxaline-4-Oxide) and with pure ethanol as the medium of the reaction, and the interaction is completed at room temperature with continuous stirring for twenty minutes where we have a yellow deposit. The lycand record has the solubility of ethanol as well as water and its melting point ranges from 188-187 °C.

Infrared Spectrum I.R.: The infrared spectrum (IR Spectra) shows a diagnosis of the major groups present in the Ligand, thus confirming the validity of the expected chemical composition of Ligand. The diagnostic process was performed using the KBr-disc.

The isomethane group, which appeared as a weak ride package in the region 1565-1580 cm^{-1}, is due to the frequency ((C=N) [Rajitha et al., 2011]. The emergence of a strong ride package in the area 1600-1640 cm^{-1} is due to the frequency ((C=O). Associated with the bradine ring, there was a weak ride package in the region at 3010-3080 cm^{-1}due to the frequency of ν (C-H). In addition, a medium ride package was found in the region at 1430-1600 cm^{-1}, due to the frequency of ν (C=N), and the other beams, appeared in the region 760 cm^{-1}, were a medium ride package. There was also a weak package in the region 2950 cm^{-1}, dating to the group of methyl(C-H). On the other hand, there was a package of hydroxyl groups in the region 3373 cm^{-1} due to the frequency of the association ν (OH) [Mashayekhi, et al., 2013].

The prepared metal complexes D, C, B, A, respectively: A group of metallic complexes of Cu (II), Zn (II), Hg (II) and Cd (II) were present with the Ligand (a) reaction with the salts of these metals. A group of complexities with different shapes and colors 2). These complexities were characterized by their low solubility in known organic solvents where the prepared D and C complex did not dissolve in any of the known solvents. Complexes (B) and (A) dissolved in DMF and polymethyl sulfoxide (DMSO) but not dissolved in any of the other solvents, these complexities are highly stable in weather conditions of light, heat, humidity and atmospheric oxygen as they are not affected by these factors. Complexities were diagnosed by spectral methods such as IR, ^{1}HNMR. Molar Conductivity was also measured by quantitative analysis of Elemental Analysis [Mashayekhi et al., 2013].

Molar Conductivity: Complex solutions did not show any value indicating their ability to conduct electrical conduction, making them non-conductive to the electrode, as the constants (100^{-3} molar in DMSO) (B), (A) showed very weak values, respectively (2.0) and (0) (C) and (D). Molecular conductivity was not measured because it was not fully dissolved in knownsavants.

Infrared spectra of prepared complexes (D, C, B, A): The diagnosis was performed using the KBr-disc, where the major groups of these complexities [Mashayekhi et al., 2013] were identified, which showed a set of similar and interrelated bundles as shown in Table 2, Figure 4 and 5.

Table 2: The spectrum data (I.R.) of prepared complexes.

Complex number	CH_3 cm^{-1}	C-O phenyl ringcm^{-1}	C-H phenyl ringcm^{-1}	C=C phenyl ringcm^{-1}	C-O Hydrazonecm^{-1}	C-H **Out ring** cm^{-1}	C=N cm^{-1}	Other	
								group	cm^{-1}
A	2943	1263	770	1640	1230	850	1612	——	——
B	2939	1242	750	1650	1106	790	1627	——	——
C	2910	1240	800	1650	1200	800	1680	C=O, C-O	1870,1220
D	2950	1242	790	1590	1240	950	1620	C=O, C-O	1690,1200

Table 3: Quantitative analysis data for prepared complexes

Complex number	Molecular formula (molecular weight)	Quantitative Analysis of Elements: Actual Calculated (Calculated in Practice)						
		%C	%H	%N	%Cl	%S	Element	Analysis
A	$C_{18}H_{14}O_7N_4CuS$ (493.96)	43.74 (43.08)	2.86 (2.08)	11.35 (10.97)	—	6.49 (5.86)	Cu	12.87 (11.23)
B	$C_{18}H_{14}O_3N_4HgCl_2$ (605.95)	35.68 (34.98)	2.33 (1.96)	9.25 (8.94)	11.72 (10.87)	—	Hg	33.10 (32.66)
C	$C_{22}H_{20}O_7N_4Cd$ (564.86)	46.78 (45.09)	3.58 (3.77)	9.92 (9.43)	—	—	Cd	19.85 (18.84)
D	$C_{22}H_{20}O_7N_4Zn$ (517.83)	51.02 (49.99)	3.90 (3.78)	10.82 (9.67)	—	—	Zn	12.63 (11.79)

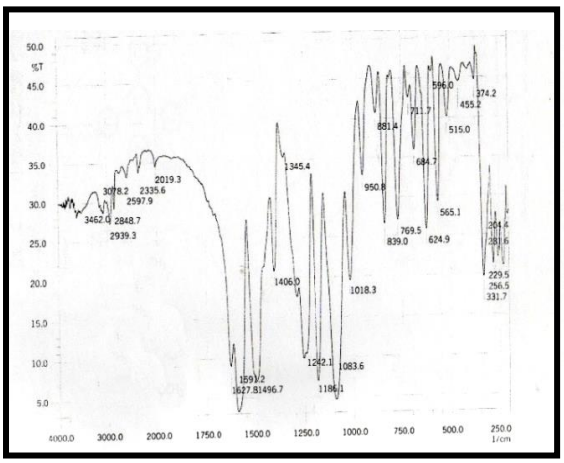


Figure 4: I.R spectrum of composite A

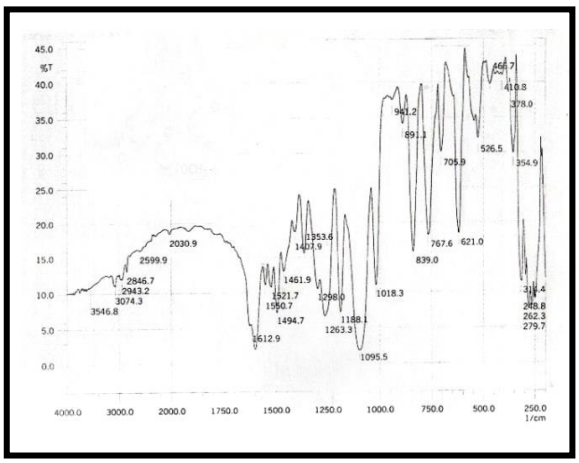


Figure 5: I.R spectrum of composite B,

NMR spectrum of ^{1}HNMR. of the Ligand (a): The N.M.R. spectrum of ^{1}HNM.R gave clear evidence of the validity of the expected chemical structure of this Ligand, by the chemical displacements shown by this Ligand, Table 4 and Figure 6 show the values of chemical shifts by Ligand (a).

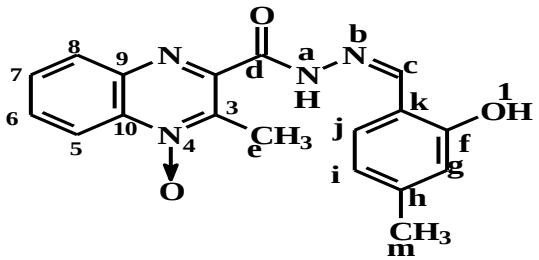
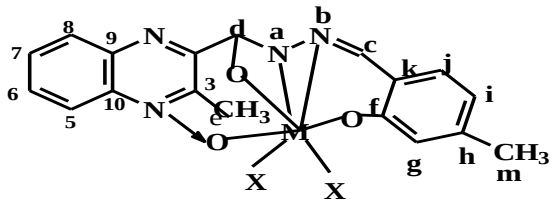


Table 4: shows the values of chemical displacements of different protons in ligand (a)

A	H(5-8) (4H,m)		H-a (1H,s	H-c (1H,s)	H(f-j) (4H,m)	H-l (1H,s)	H-m (3H-s)	H-m (3H-s)
	3.1		7.7-7.5	11.1	8.6	88.2-8.	11.4	2.35

^{1}HNMRproton magnetic resonance spectrum (A): The spectrum gave clear evidence of the health of the prepared complexes through the chemical displacements shown by these complexs.

Table 5 and Figure 7 showing the chemical shift values in Complex (A).



Where M= Cu (II), Hg (II), Cd (II), Zn (II)

Table 5: shows the values of the chemical displacements of the different protons of the complex (A).

	H(6-7) (4H,s)	H-l (3H-s)	H-e (3H,s)	H(5-8) (4H,m)	H-g (1H,s)	H-c (1H,s)	H(j) (1H,s)	H-l (1H,s)
A	8.7	2.35	2.35	8.1	7.45	3.84	7.77	7.5

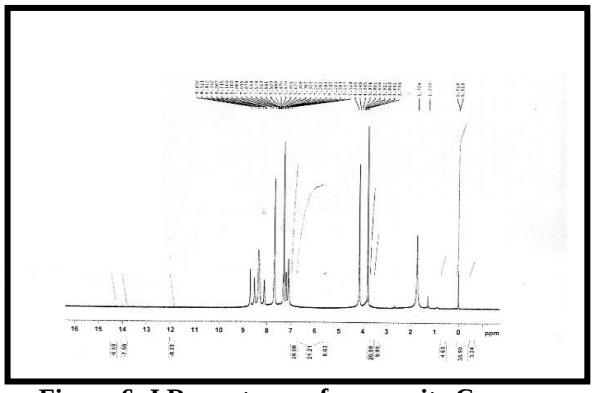


Figure 6: I.R spectrum of composite C

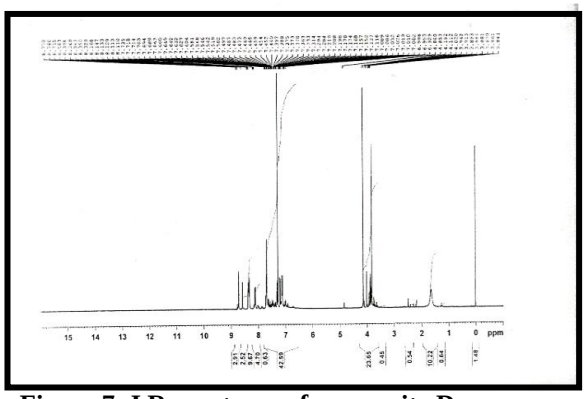


Figure 7: I.R spectrum of composite D,

Bioactivity: By observing the biological efficacy of the compounds prepared on three strains of bacteria, different values were given. The Staphylococcusaureus inhibition diameter (6.4-10.2) in addition to some complexities did not produce any effect against the bacteria. The reason may be that Staphylococcus aureus produces mucus (D) has the highest excitation diameter (17.4 mm) in the concentration (30ml/m) amalgam). Proteus Mirabilis and Salmonella typhi. The values of relatively high inhibition were compared with the previous six (16.4-30.1) and (18.0-30.4), respectively. Table 6 shows the biological efficacy data of prepared and measured complexes in millimeters (Table 6).

Table 6: Data on the biological effectiveness of prepared complexes.

Compound number	Conc. mg/ml	Bacterial used		
		Salmonella typhi (mm)	Staphylococcus aureus (mm)	Proteus mirabillis (mm)
A	0.1	22.4	----	19.0
	1	18.0	10.0	18.4
	10	21.4	9.0	17.4
	30	30.1	8.0	22.1
	50	20.1	9.0	18.0
B	0.1	18.2	----	19.4
	1	19.0	11.0	24.0
	10	18.4	7.5	16.8
	30	19.2	4.4	25.2
	50	18.5	10.2	22.2
C	0.1	24.2	10.0	29.0
	1	22.4	15.2	15.2
	10	30.4	10.2	22.0
	30	19.2	8.0	30.1
	50	20.8	----	15.4
D	0.1	19.4	6.4	17.0
	1	20.0	----	14.2
	10	25.0	9.0	16.4
	30	19.2	17.4	19.6
	50	23.2	----	15.6

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