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Metal Complexes of acetohydrazide Derivative: Characterization, and **Antimicrobial Activity Study**

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Abstract

A novel derivative of 2,3-disubstituted quinazolin4(3H)-ones has been produced as of p-methoxybenzoylchloride and anthranilic acid, this prepared compound was used to produce a stable complexes. The ligand and complexes were isolated from the reaction in the solid form and characterized by FT-IR, UV-Visible, TLC, ¹H NMR data and some physical measurements. Magnetic susceptibility data coupled with electronic spectra suggested octahedral structure of the complexes. The ligand and complexes were also employed with various bacterial. The products exhibited that the metal complexes have larger antimicrobial activities than ligand.

Keywords: Quinazolinone, Carbohydrazide, complexes and antimicrobial

1. Introduction

The quinazolinone skeleton has been attracted much more attention of medicinal and organic chemists due to their biological activities, most of them are

extensively used as antimicrobial[1],antioxidant[2],anti-tubercular[3], anticancer [4], anti-HIV [5], anti-inflammator 6 ituted thiosemicarbazide, and some heterocyclic compounds like thiadiazoles, triazoles, pyrazoles, and oxadiazoles[7,8].

2. Methodology

2.1 MATERIALS AND METHODS

All of the substances are commercially obtainable in the (Aldrich Co.). infra-red spectra measurements have been recorded through the model Shimadzu FT-IR- 8400S. The UV-Visible spectra were measured in ethanol using Shimadzu UV- Vis. 160 a spectrophotometer. The 1HNMR spectra were recorded on a BRUKER spectrometer model ultra-shield 300 MHz, using DMSO-d6, as solvents and tetramethylsilane (TMS) as an internal reference. The magnetic susceptibility of the solid compounds was acquired at room temperature using Magnetic Susceptibility Balance Johnson Matthey.

2.2. Synthesis of compounds

2.2.1 Synthesis of 2-mercapto-3-phenylquinazolin-4(3H)-one (1)

Phenyl isothiocyanate (0.01 mole,1.31mL) was added to anthranilic (0.01mol) in absolute ethanol(25 mL). The mixture refluxed for 8 hours then decant into ice- water. The precipitate was filtered, washed with water then recrystallized from ethanol. Yield: 78%. M.p.285-287 °C, infra-red (v⁻¹): 3253 v(N-H) , 1680v(C=O) 1350v(C=) ¹ H-N R (δ , ppm): 9.23(s, 1H, NH), 8.46-6,5 (m, 8H,Ar-H).

2.2.2 Synthesis of ethyl2-((4-oxo-3-phenyl-3,4-dihydroquinazolin- 2-yl)thio)acetate (2)

(0.01 mole,0.9g) Ethyl chloroacetate was added to a stirred solution of compound (1) (0.01 mole,2.4g) and anhydrous sodium acetate (0.01mole) in (20ml) absolute ethanol. The mixture refluxed for 6hours. The precipitate was filtered and recrystallized from the solvent. Yield: 78%. M.p.180-182°C, FT-IR (v cm-1): 3367 v(N-H) 1685v(C=O) 1350v(C=)¹ H-N R (δ): 1.19-1.24(t, 3H,CH3),3.97(s, 2H,CH2), 4.17(q, 2H,CH2O), 8.90-7.49(m, 8H,Ar- H).

2.2.3 Synthesis of2-((4-oxo-3-phenyl-3,4dihydroquinazolin yl)thio)acetohydrazide (3)

A mixture of hydrazine hydrate (99%) (10 mL) in absolute ethanol (25 mL) and

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2-mercapto-3-phenylquinazolin-4(3H)-one (1) (0.01mol) was refluxed for 7 h. The excess of solvent was distilled off then the product was left to cool, filtered thereafter recrystallized from solvent(Scheme 1). M.p.: 150-151 °C, Yield: 88%,

FT-IR (ν -1): 3338-3265 υ(N-H₂, NH) , 1664υ(C=O) ¹ H-NMR (δ): 9,85(1H, s, NH) 8.46.8 (4H, m, ArH), 5.6(2H, s, NH₂).



Scheme 1. Synthesis of ligand

2.2.4 Preparation of Metal Complexes

A solution of cobalt(II) chloride hexahydrate (1.70g, 5mmol), nickel(II) chloride hexahydrate (1.8gm, 5mmol) and copper(II) chloride hexahydrate (0.73gm, 5mmol) in ethanol was mixed with the solution of 2-(4-oxo-3-phenyl-3,4-dihydro quinazoline-2-yl) acetohydrazide (4.10gm, 10mmol) in the same volume of ethanol and refluxed for two hours. After distillation, the contents were cooled at room temperature. The precipitates were first filtered, then washed with ethanol and dried in air. The analytical, elemental information of the metal complexes was summarized in Table 1.

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Molecular formula	Color, Yield%	0	M %
			Calc. (found)
C ₁₅ H ₁₄ N ₄ O ₂	Yellowish, 88	150- 151	
$[Co(C_{15}H_{14}N_4O_2)_2Cl_2]$	Red	247-249	8.10(8.61)
$[Ni(C_{15}H_{14}N_4O_2)_2Cl_2]$	Greenish yellow, 72	232-234	8.46(9.83)
$[Cu(C_{15}H_{14}N_4O_2Cl_2]$,78green	300-302	9.08 (8.28)

Table1: showed some physical properties of the prepared ligand and its metal complexes.

3. Results and discussions

Molar conductance's of the metal complexes

Conductivity amounts of all the mononuclear complexes were agreed out in the DMSO medium. The Am values in the range from (11 to 18) (ohm-1 cm2 mole-1), The smaller value of molar conductance indicate that the prepared metal complexes are non-electrolytic in nature Table 2 Table 2: Molar conductance's of the metal complexes

Table 2: Molar conductance's of the metal complexes

Compound	$(ohm^{-1} cm^2 mole^{-1})$
$[Co(C_{15}H_{14}N_4O_2)_2Cl_2]$	18
$[Ni(C_{15}H_{14}N_4O_2)_2Cl_2]$	11
$[Cu(C_{15}H_{14}N_4O_2)_2Cl_2]$	15

The FT-IR spectroscopy

All the complexes stable in room temperature, insoluble in water but soluble in methanol. The FT-IR spectrum for the ligand and complexes show significant vibrational band, these bands were summarized in Table 3. The ligand spectrum exhibit bands at (3338-3265)cm⁻¹attributed to NH₂ stretching vibrations. These bands were moved to lesser vibration with a difference in shape and the spectra of the metal complexes advising the coordination of nitrogen of (NH₂) to the metal ion.

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The band localized in range (458-464) cm⁻¹ outcome of coordinate (M-N) frequency [9-11]. A band at (1763)cm⁻¹ which is attributed to [C=O] stretching vibrations This frequency shifted to lesser vibration in the all complexes as compared to ligand, representing coordination of carbonyl group with metal ions through the oxygen atom. The spectra of all complexes extant feeble bands between 407-415 cm⁻¹ which can be assigned to v(M-Cl)[12].

 Table 3: FT-IR spectra of ligand and their complexes
vNH₂ (sy.aym) Compound vC=O **vМ-О** vM-N vM-Cl C15H14N4O2 3338,3265 1763 _ _ _ 510 $[Co(C_{15}H_{14}N_4O_2)_2Cl_2]$ 2853, 3178 1708 412 464 $[Ni(C_{15}H_{14}N_4O_2)_2Cl_2]$ 3097, 3020 1725 525 458 407 [Cu(C15H14N4O2)Cl₂] 3040, 3157 535 422 415



Fig. 1: FT-IR spectra of [C₁₅H₁₄N₄O₂]

Fig. 2: FT-IR spectra of [Co(C₁₅H₁₄N₄O₂)₂Cl₂]



Fig. 3: FT-IR spectra of [Ni (C₁₅H₁₄N₄O₂)₂Cl₂]

Fig. 4: FT-IR spectra of [Cu (C₁₅H₁₄N₄O₂)₂Cl₂]

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Uv-vis. Spectral Properties

The ligand solution in ethanol displays two peaks at 213 and 260nm (Table 4). The $\mu(B)$ 62B The obvious electronic spectral peaks at 590, 632 and 644nm attributed to $4T_1 \rightarrow 4T_1g$ (p), $4T_1 \rightarrow 4A_2g$ (F) and $4T_1 \rightarrow 4_2g$ (F) transferences respectively [13-16] these spectra additional support to the octahedral structure of the Cobalt complex. The nickel complex spectrum was individual of an octahedral structure with outstanding peaks at 395,440 and 585nm assigned to $3A_2$ (F) $\rightarrow 3T_1g(P),3A_2$ (F) $\rightarrow 3_1g(F)$ and $3A_2$ (F) $\rightarrow 3_2g(F)$ transferences[17]. The magnetic sensitivity of 2.35 B.M. also projected an octahedral structure. The Copper complex exhibited absorption bandat 679nm which may be assigned to the $2E \rightarrow 2_2g$ on the basis of electronic spectra octahedral geometry about

the Cu+2 ion was proposed [18], the magnetic sensitivity value (1.87 B.M.).

Table 4:Electronic transitions and magnetic moments of the preparedcompounds.



Fig 5: Electronic Spectral data of ligand and $Ni(C_{15}H_{14}N_4O_2)_2Cl_2$ complex.

Biological Activity

The 2-(4-oxo-3-phenyl-3,4-dihydro quinazoline-2-yl) acetohydrazideand the corresponding metal compounds under study were tested against five antimicrobial activity Staphylococcus aureus, S.epidermidis, E.coli, Klebsiella spp, and candid by plate well into agar nutrient method. Antimicrobial activity was expressed in millimeters (mm) by measuring the diameters of the inhibitory region and contrasting with the DMSO control and the values were explained by Table5.

Bacterial isolates	DMSO	Ligand/ DMSO	Ni- ligand/ DMSO	Co-ligand/ DMSO	Cu-ligand/ DMSO
Staphylococcus aureus	-	-	13	20	11
S.epidermidis	-	15	18	22	17
E. coli	-	-	10	-	14
Klebeilla spp	-	12	-	26	29
candidx	-	11	14	-	-

Table 5: Antimicrobial properties of ligand and its complexes.

Suggested chemistry structure of metal complexes

From the result of the elemental and spectral analysis in addition to magnetic moment and conductivity measurements, the structure of the complexes can be clarified as following, Fig 4



Fig.6: Suggested chemistry structure of metal complexes

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