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# Synthesis, Characterization and Antimicrobial Studies of Some Metal Complexes of N-aminoquinolino-2-one and Anthranilic Acid Hydrazid

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**ABSTRACT**

A new complexes of Cr(III), Fe(III), Zn(II), Hg(II),Sn (II) and Pb (II) have been synthesized with bidentate ligands N-aminoquinolino-2- one ($L_1$) and anthranilic acid hydrazide ($L_2$)as mixed ligands .These complexes were characterized by means of microanalysis, molar conductance measurements, magnetic susceptibility measurement, infrared and electronic spectral studied. From these studies the ligands were coordinated to metal ion from nitrogen and oxygen atoms. The free ligands and its metal complexes have been tested in vitro against a number of microorganisms such as proteus mirabilis, klebsiellat pneumonia, Bacillus cereus and staph aureus in order to assess their antimicrobial properties. It has been found that the complexes showed best antimicrobial activity.

**Key word:** N-aminoquinolino-2-one**,** Anthranilic acid hydrazid, Metal complexes, Spectral studies, Antimicrobial

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## INTRODUCTION

Coumarins have stimulated extensive research in biology, organic chemistry and medicine,due to Their antibiotic[1], anti-coagulant[2]anticancer[3], anti-inflammatory[4] and antitumor[5] properties. Coumarins are now a day an important group of organic compounds that are used as additives to food and cosmetic[6].Coumarin have been synthesizedbyseveral methods including Von pechman, knovenagel, Reformatsky reactions[7,8]. It is well known that2-quinolino derivatives are found to be associated with various biological activities such as anti-malarial[9],anti-platelet[10],Anti- fungal agent[11]and antioxidant activity[12]. The derivatives of coumarin usually occur as secondary metabolites present in seeds, roots and leaves of many plant species. Their function is far from clear, through suggestions include waste products, plant growth regulators, fungi stats and bacterial stats[13]. The complexes of these compounds have more biological and chemical effect in various ways[14-15]. In this article we describe the synthesis, characterization of an aminoquinoline-2-one and anthranilic acid hydrazide and their complexes with $Cr^{+3}$, $Fe^{+3}$, $Zn^{+2}$, $Hg^{+2}$, $Sn^{+2}$ and $Pb^{+2}$ ions. Moreover, the antimicrobial activities of these complexes were determined.

**Experimental:** The ligands ($L_1$) has been synthesized according to the previous researches[16].

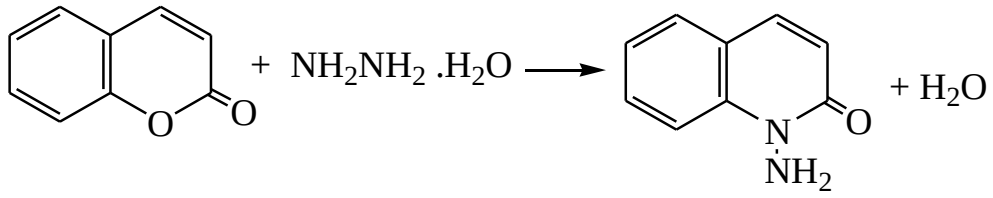
**Physical Measurement**

Infrared spectra were recorded on a FTIR Bucker Tensor 27 spectrophotometer in 400-4000 $cm^{-1}$range using KBr discs. The molar conductance of $10^{-3}$ M solution of metal complexes in dimethylformamide (DMF) was measured at 25$^{o}$C using

Jenway 4070 conductivity meter and dip-cell with platinised electrodes. The U.V/VIS spectra were recorded on Shimadzu UV-160spectrophotometer for $10^{-3}$ M solution of complexes in DMF using 1cm quartz cell. Melting points were measured using anelectro thermal digital melting point apparatus- Magnetic susceptibility was measured on the solid state by faraday's method using burker BM6 instrument .All magnetic susceptibility values were corrected for diamagnetic using Pascal's constant.

**Synthesis of the ligand**

Coumarin (1.46gm,0.01mol) with excess hydrazine hydrate (99%) (3.2 gm ,0.1 mol) in absolute ethanol (25ml) was refluxed for 12h , then cooled ,the formed solid was collected and recrystallized from chloroform (m.p 131-133 ºC).




**Preparation of the metal complexes**
**A-1 $[M(L_1)_2Cl_2]$**

A hot solution of the ligand ($L_1$)(2.68gm 0.02mol)in(20ml) ethanol was added to the solution of the corresponding metal salts (Table 1) (0.01 mol) with stirring, the mixture was refluxed for1 hr. then cooled.The complexes with different color,were filtered off, washed with cold ethanol and dried in vacuum.

**B-2 $[M(L_1)_2L_2]Cl_2$**

The same as pervious , except that the addition of( $L_2$)(1.51gm 0.01 mol) was used to obtain the mixed ligand complexes.

**Antibacterial activity:**

The antibacterial activity estimated against*staphylococcus aureus*, as *gram* positive and *E. coli* and *pseudomonasaeruginosa* as *gram* negative, and evaluated by using of agar disc diffusion method on the basis of the size of inhibition zone formed around the paper discs. For each concentration, the mean diameter (mm) of inhibition zone appeared was calculated. Different concentrations of the tested compounds were prepared in DMSO. Nutrient agar plates were poured in petri dishes, After solidification ,0.1ml of the broth culture of the tested bacteria were cultured on the surface of the Nutrient agar plates, then filter paper discs(Whatmann No.1) of 6mm diameter impregnated with different concentrations of the tested compound wereplaced on the surface of the inoculated plates, A standard antibiotic clindamycin($DA_2$)was used as a control ,the plates were incubated at 37° c for 24h.The zone of inhibition was measured in millimeters[17] .

**Note:** Weused this type of bacteria because it most common and pathogenic.

## RESULTS AND DISCUSSION

A new N- aminoquinolino-2- one as bidentate ligand ($L_1$), was synthesized from the reaction of coumarin and excess of hydrazine hydrate in absolute ethanol (Fig.1) in a high yield. Firstly complexes of the general formula $[M(L_1)_2Cl_2]$ was prepared by direct reaction of the metal chlorides (M= Cr(III),Fe(III) Zn(II), Hg(II), Sn(II) and Pb(II)) with the above ligand ($L_1$) in molar ratio (M:L)1:2.After complexation a second ligand anthranilic acid hydrazide ($L_2$)has been added to form mixed ligand complexes with general formula$[M(L_1)_2L_2]Cl_2$. The structure of the ligand and the complexes were characterized by different physicochemical methods (Table1).The analytical data are given in (Table 1). These data were found in a good agreement with the proposed formulas. All the complexes are stable in air at room temperature and they are insoluble in most common organic solvents but they are soluble in dimethylformamide (DMF) and dimethylsulfoxide (DMSO).

The electrical molar conductance of the complexes (1 and 2)were(85–78) $ohm^{-1}$ $mol^{-1}$ $cm^2$respectively,indicating 1:1 electrolytic naturewhilecomplexes .(7 and 8) gives (237-255) $ohm^{-1}$ $mol^{-1}$ $cm^2$ which are corresponding conductivity complexes indicating 1:3electrolytic nature of the complexes. Where the complexes(3,4,5,6) gives (19,21,38,24) $ohm^{-1}$ $mol^{-1}$ $cm^2$ indicating non-electrolytic nature and the complexes (9,10,11,12) were (141,155,121,159) $ohm^{-1}$ $mol^{-1}$ $cm^2$ indicating 1:2 electrolytic[18](Table 1).

The IR spectra of the ligand showed strong band at 1683 $cm^{-1}$ which belongs to υC=0 and the other bands at 3260, 1198 $cm^{-1}$ belong to $\upsilon NH_2$,$\delta NH_2$[19]. The spectra bands of complexes at (1625–1645) $cm^{-1}$ were characterized for the carbonyl group which indicates that the oxygen atom of the carbonyl group was coordinated to the metal ion and the prepared complexes exhibited $\upsilon NH_2$ in the range of (3075–3120) $cm^{-1}$ which shows a shifting to the lower frequencies, indicating the coordination($L_1$) with metal ions through the nitrogen atoms in their structures[20]. A medium band at (1223-1273) $cm^{-1}$ maybe assigned$\delta NH_2$ to stretching vibrations. The absorption band in the range (475–510) and (425–464) $cm^{-1}$ were assigned to (M–N) and (M–O)bands respectively[21](Table 3).

The magnetic moments measurements of Cr (III)complexes(1and7) were in the rang(3.77-3.92) B.M well inaccordance with those having octahedral structure with the three unpaired electronic[22].
While the electronic spectra of these two complexes (1and7)wereexhibited bands at (23534-31422) $cm^{-1}$ due to charge transfer. The magnetic moments values of the Fe(III)complexes (2, 8) were(5.99-6.48) B.M.These values were in accord to the literature[23].On other hand the electronic spectra showed bands at (9690-12870)$cm^{-1}$which were attributed to the $^5T_2g \rightarrow {}^5Eg$ transition and could be assigned to a distorted octahedral structure. The electronic spectra of Zn(II), Hg(II), Sn(II) and pb(II) in the range (25000-31460) $cm^{-1}$were assigned to charge transfer bands and the magnetic susceptibility showed that all these complexes were diamagnetic[24](Table 2).

**Antibacterial activity**

The ligand N-aminoquinolino-2-one was found to be biologically active. It is known that chelation tends to make ligands act as more powerful and potent bactericidal agent. The value indicate that the metal complexes had a higher antibacterial activity than the free ligand. Such increased activity of the metal complexes can be explained on the bases of the overtone concept and chelation theory[25].According to the overtone concept of the cell permeability, the lipid membrane that surround favors the passage of only lipid soluble materials, due to which lip solubility is an important factor controlling the antimicrobial activity on chelation the polarity of the metal ion is reduced to a great extent due to the overlap of the ligand orbital and the partial sharing of the positive charge of the metal ion with donor groups. Furthermore, it increases the delocalization of electrons over whole chelate ring and hances the lipophilicity of the complex.This increased lipophilicity enhances the penetration of the complex into the lipid membrane and blocks the metalbinding sites on the enzymes of the microorganism . In (Table4) these complexes showed inhibitory effect for twotypes of bacteria G+ and G-,but its effect on G+ was more that on G-, when comparing these results with clindamycin($DA_2$)as antibacterial it was regarded as a good result, and Fe(III) complex showed higher activity than the other complexes in porteus Mirabilis, Kiebsiella Pneumonia, Staphylococcus Aureus. while in (Table 5) showed the minimum inhibitory concentration (MLC) for each complex on four types of bacteria, the results showed that (MIC) for these complex on G+had more activity than the ligands, and Hg(II)complex hadhigher activity against tested bacteria.

## CONCLUSION

The ligands N-amino quinolino-2-one($L_1$)and anthranilic acid hydrazide ($L_2$) act as bidentate ligand in all complexes. Infrared spectral study showed that bonding happened among nitrogen of amine group and oxygen of carbonyl group with metal ions. Molar conductance, magnetic susceptibility, electronic spectral studies showed that all complexes were octahedral geometry, and they were active against bacteria more than the free ligand.

**Table 1: Physical properties of ligands and their complexes**

| No. | Complex | Color | M.P | Yield | Elemental analysis | Molar conductivity, $cm^{-1}$, $mol^{-1}$ |
|---|---|---|---|---|---|---|
| | $L_1$ | Orange | 131-133 | 91% | | |
| 1. | $[Cr(L_1)_2Cl_2]Cl$ | Light blue | 246 | 80% | 10.94 (18.92) | 85 |
| 2. | $[Fe(L_1)_2Cl_2]Cl$ | Brown | 270 | 81% | 11.69 (22.26) | 78 |
| 3. | $[Zn(L_1)_2Cl_2]$ | Yellow | 198 | 73% | 14.34 (20.99) | 19 |
| 4. | $[Hg(L_1)_2Cl_2]$ | Light yellow | 218 | 60% | 34.12 (49.14) | 21 |
| 5. | $[Sn(L_1)_2Cl_2]$ | Grey | 153 | 86% | 23.47 (12.50) | 38 |
| 6. | $[Pb(L_1)_2Cl_2]$ | Brown | 182 | 84% | 34.78 (14.33) | 24 |
| 7. | $[Cr(L_1)_2L_2]Cl_3$ | Dark yellow | 144 | 67% | 8.33 (13.69) | 237 |

| | | | | | | |
|---|---|---|---|---|---|---|
| 8. | $[Fe(L_1)_2L_2]Cl_3$ | Dark Brown | 139 | 69% | 8.91 (35.10) | 255 |
| 9. | $[Hg(L_1)_2L_2]Cl_2$ | Grey | 118 | 70% | 27.19 (9.99) | 141 |
| 10. | $[Zn(L_1)_2L_2]Cl_2$ | Dark green | 143 | 64% | 10.79 (10.02) | 155 |
| 11. | $[Sn(L_1)_2L_2]Cl_2$ | Light yellow | 124 | 72% | 18.14 (11.20) | 121 |
| 12. | $[Pb(L_1)_2L_2]Cl_2$ | Light orange | 133 | 57% | 27.82 29.15 | 159 |

**Table 2: Magnetic moment and electronic spectra data of the complexes**

| **No.** | **Complexes** | **μeff** | **λ max $cm^{-1}$** |
|---|---|---|---|
| 1. | $[Cr(L_1)_2Cl_2]Cl$ | 3.92 | 23534, 30300, 31422 |
| 2. | $[Fe(L_1)_2Cl_2]Cl$ | 5.99 | 9890, 30303, 31112 |
| 3. | $[Zn(L_1)_2Cl_2]$ | ____ | 27933, 30120, 31457 |
| 4. | $[Hg(L_1)_2Cl_2]$ | ____ | 28970, 31419 |
| 5. | $[Sn(L_1)_2Cl_2]$ | ____ | 25400, 30300, 31350 |
| 6. | $[Pb(L_1)_2Cl_2]$ | ____ | 29410, 31300 |
| 7. | $[Cr(L_1)_2L_2]Cl_3$ | 3.77 | 25090, 32330 |
| 8. | $[Fe(L_1)_2L_2]Cl_3$ | 6.48 | 12870, 31460 |
| 9. | $[Zn(L_1)_2L_2]Cl_2$ | ____ | 26040, 31211 |
| 10. | $[Hg(L_1)_2L_2]Cl_2$ | ____ | 29200, 33000 |
| 11. | $[Sn(L_1)_2L_2]Cl_2$ | ____ | 26450, 31460 |
| 12. | $[Pb(L_1)_2L_2]Cl_2$ | ____ | 25000, 30300, 31000 |

**Table3: Infrared spectral data of ligand and this complexes**

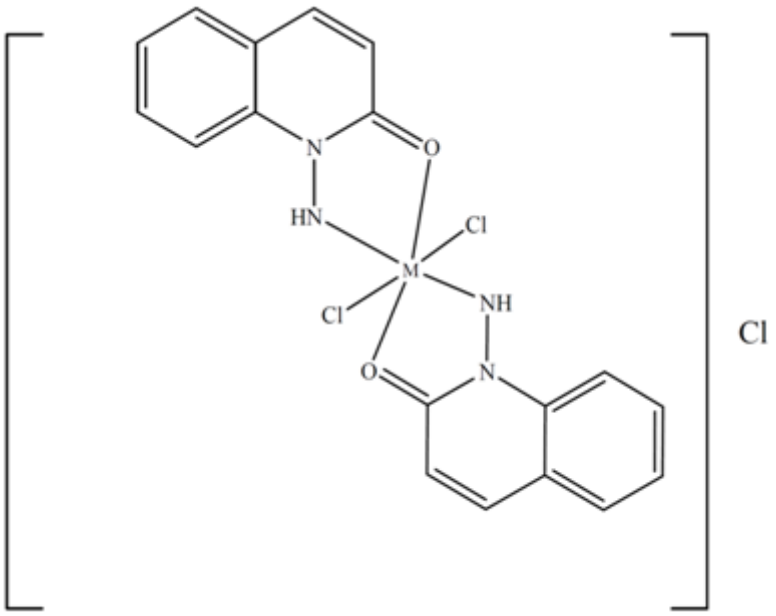
| **No.** | **Complexes** | **δN-H** | **υC=O** | **υN-H** | **υM-N** | **υM-O** |
|---|---|---|---|---|---|---|
| | L | 1298 | 1683 | 3260 | | |
| 1. | $[Cr(L_1)_2Cl_2]Cl$ | 1260 | 1640 | 3115 | 557 | 431 |
| 2. | $[Fe(L_1)_2Cl_2]Cl$ | 1257 | 1645 | 3116 | 484 | 442 |
| 3. | $[Zn(L_1)_2Cl_2]$ | 1225 | 1625 | 3114 | 495 | 441 |
| 4. | $[Hg(L_1)_2Cl_2]$ | 1263 | 1641 | 3110 | 503 | 465 |
| 5. | $[Sn(L_1)_2Cl_2]$ | 1252 | 1634 | 3115 | 475 | 432 |
| 6. | $[Pb(L_1)_2Cl_2]$ | 1273 | 1636 | 3115 | 510 | 462 |
| 7. | $[Cr(L_1)_2L_2]Cl_3$ | 1261 | 1628 | 3111 | 532 | 432 |
| 8. | $[Fe(L_1)_2L_2]Cl_3$ | 1261 | 1640 | 3115 | 517 | 476 |
| 9. | $[Zn(L_1)_2L_2]Cl_2$ | 1238 | 1630 | 3120 | 512 | 427 |
| 10. | $[Hg(L_1)_2L_2]Cl_2$ | 1230 | 1641 | 3115 | 527 | 427 |
| 11. | $[Sn(L_1)_2L_2]Cl_2$ | 1223 | 1625 | 3120 | 525 | 466 |
| 12. | $[Pb(L_1)_2L_2]Cl_2$ | 1247 | 1639 | 3075 | 505 | 448 |

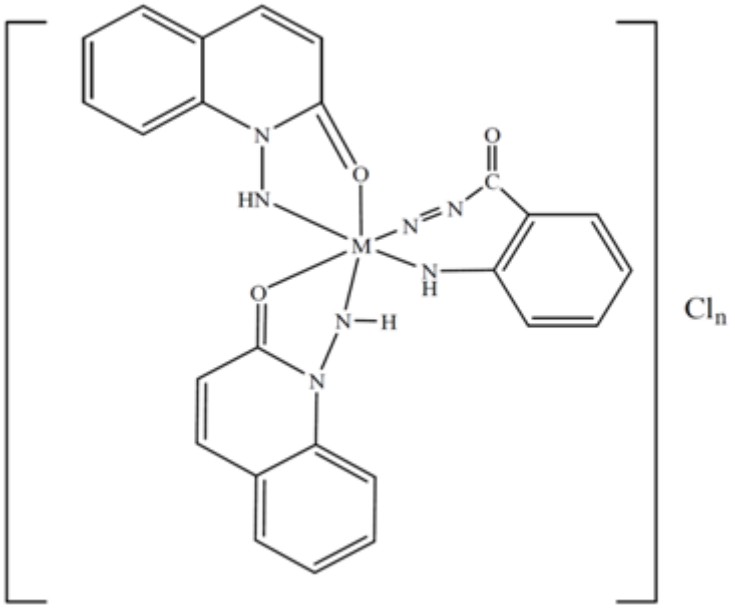
**Table 4: The antibacterial activity of ligands and metal complexes against the tested bacteria. The antibacterial activity of ligands and metal complexes the zone of inhibition was measured in mm(concentration in ppm).**

| No. | Complexes | *Proteus Mirabilis* | *Klebsiella Pneumonia* | *Bacillus Cereus* | *Staphylococcus Aureus* |
|---|---|---|---|---|---|
| | $L_1$ | 18 | 15 | 27 | 30 |
| 1 | $[Cr(L_1)_2Cl_2]Cl$ | 18 | 30 | ____ | 27 |
| 2 | $[Fe(L_1)_2Cl_2]Cl$ | 6 | 12 | ____ | 10 |
| 3 | $[Zn(L_1)_2Cl_2]$ | 12 | 27 | 20 | 27 |
| 4 | $[Hg(L_1)_2Cl_2]$ | 17 | 20 | 25 | 33 |
| 5 | $[Sn(L_1)_2Cl_2]$ | 11 | 20 | ____ | 27 |
| 6 | $[Pb(L_1)_2Cl_2]$ | 13 | 12 | ____ | 14 |
| 7 | Clindamycin ($DA_2$) | 25 | ____ | 24 | 24 |

**Table 5: The minimum inhibitory concentration (MIC) for the compounds against tested bacteria**

| No. | Complexes | *Proteus Mirabilis* | *Klebsiella Pneumonia* | *Bacillus Cereus* | *Staphylococcus Aureus* |
|---|---|---|---|---|---|
| | $L_1$ | 6.26 | 12.5 | 1.5 | 3.125 |
| 1 | $[Cr(L_1)_2Cl_2]Cl$ | 50 | 6.25 | ____ | 50 |
| 2 | $[Fe(L_1)_2Cl_2]Cl$ | 100 | 50 | ____ | 100 |
| 3 | $[Zn(L_1)_2Cl_2]$ | 50 | 1.5 | 1.5 | 1.5 |
| 4 | $[Hg(L_1)_2Cl_2]$ | 1.5 | 1.5 | 1.5 | 1.5 |
| 5 | $[Sn(L_1)_2Cl_2]$ | 50 | 6.25 | ____ | 6.25 |
| 6 | $[Pb(L_1)_2Cl_2]$ | 25 | 6.25 | ____ | 3.125 |






**Suggested structure of complexes, where M = $Cr^{+3}$, $Fe^{+3}$, $Zn^{+2}$, $Hg^{+2}$, $Sn^{+2}$ and $Pb^{+2}$**

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