



Characterization of Transition Metal Complexes Derived from Biologically Active Furoic Acid Hydrazone

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ABSTRACT

Biologically active schiff base ligand, N'-2-[(Z)-1-(4-hydroxy-6-methyl-2-oxo-2H-pyranyl)ethylidene]furancarbohydrazide and its M(III) complexes have been prepared and characterized by elemental analyses, molar conductance, magnetic susceptibility measurement, UV-Vis, IR and NMR spectra. Based on these studies an octahedral geometry has been proposed for these complexes. The ligand and its metal complexes were screened for their antimicrobial activities. The ligand showed higher activity than its corresponding M (III) complexes.

Key words: Furoic acid hydrazide, antimicrobial activity.

INTRODUCTION

Furoic acid hydrazones and their metal complexes show varied applications as antifungal^{1,2}, antibacterial^{3,4} antioxidative^{5,6} and cytotoxic^{7,10}. These have also proved to be potential chemotherapeutic agents^{11,12}. In addition to these, furoic acid hydrazones are potential chelating agents and exhibit excellent physiological and biological activities¹³⁻¹⁵. In view of these facts, the present research work has been undertaken.

EXPERIMENTAL

Metal salts, reagents and all chemicals used during this research work were of AR grade or equivalent grade and were procured from reputed firms. The solvents used were purified before use.

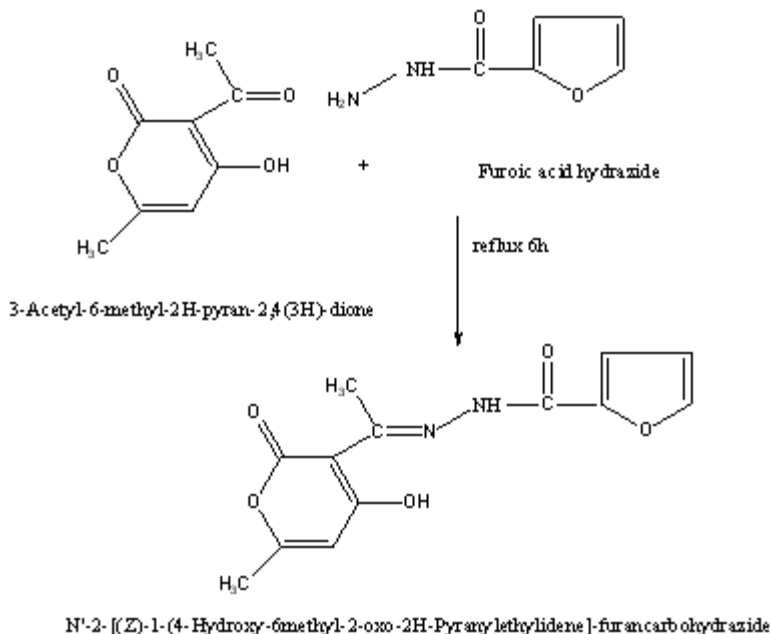
Melting points/decomposition temperatures were determined by open capillary method and are uncorrected. Electrical conductivities were determined at room temperature with freshly prepared solutions of 10⁻³ M dilution using systronics model 304 conductivity meter. Electronic spectra were recorded using Beckmann DU-spectrometer. The magnetic susceptibility was measured by Gouy's method using CuSO₄·5H₂O as calibrant. Carbon, hydrogen and nitrogen were determined by microanalysis at CDRI, Lucknow. The IR spectra were also recorded at CDRI Lucknow in KBr phase.

Preparation of ligand

It was prepared by mixing 3-acetyl-6-methyl-2H-pyran-2,4(3H) dione (0.168g, 1 m mole) in 20 ml methanol with furoic acid hydrazide (0.126

g, 1 m mole) in 20 ml methanol in 1 : 1 proportion and the mixture was refluxed and for 4 h. On cooling, a yellowish crystalline compound separated out. It

was recrystallized from methanol. The purity of the schiff base was checked by TLC.

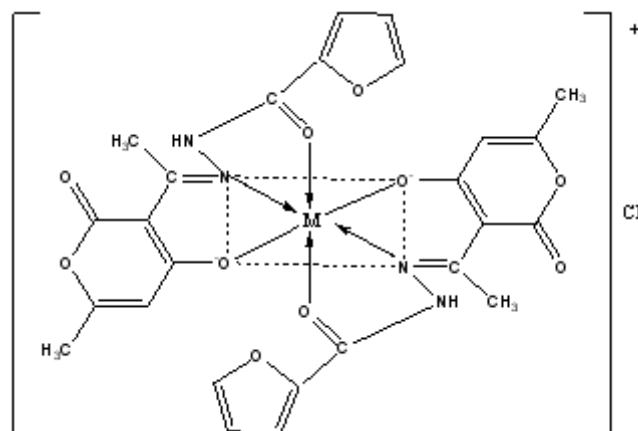


Scheme 1:

Preparation of complexes

The ligand and the metal salts were mixed in the 2 : 1 molar ratio and kept overnight, when

metal chelates separated out. The solid was filtered, washed with aqueous methanol and pet ether and dried in vacuum



Scheme 2:

RESULTS AND DISCUSSION

The analytical data of the metal chelates suggested 1:2 (M:L) stoichiometry for all the complexes and is in good agreement with the proposed molecular formulae and proposed structures of the complexes. All the metal complexes are colored and non hygroscopic. The Ti (III), & V (III) complexes are sensitive to air but all other complexes are stable in air. The complexes are insoluble in water and common organic solvents but soluble in DMF, DMSO & acetonitrile. The molar conductance values in DMF suggested 1 : 1 electrolytic nature for all the complexes.

The ir spectrum of the ligand exhibits bands at 3500, 1720, 1670, 1620 and 1310 cm^{-1} assignable to ν OH (hydrogen bonded), ν C=O (azide), ν C=N (azomethine) and ν C=O (phenolic) respectively. The ν C–O–C of furan ring appeared at 1080 cm^{-1} . In the spectra of the complexes ν C–O lactone has undergone upward shift 25–30 cm^{-1} ¹⁶. The upward shift of ν C=O (phenolic) in the spectra of the complex indicated the participated of phenolic oxygen in coordination¹⁷. The azomethine frequency has undergone 20–25 cm^{-1} in the complexes suggesting coordination through N-atom of the azomethine group thus the ligand has behaved in mono basic tridentate manner. These coordination site as further supported by the presence of two non ligand bands in the ir spectra of the complex in the region of 540–560 cm^{-1} and 450–470 cm^{-1} assignable ν M–O, ν M–N frequencies respectively.

Electronic Spectra

The magnetic susceptibility of Ti (III) complex was determined by Gouy's method at room temperature which gave a value of 1.96 B.M. for magnetic moment. This value indicated presence of one unpaired electron. The higher value may be due to orbital contribution. The electronic spectrum of the complex shows a single broadband at 19430 cm^{-1} assignable to ${}^2t_{2g} \rightarrow {}^2E_g$ transition¹⁹ which is characteristics of octahedral geometry.

The magnetic moment for Mn (III) complex is 4.91 m_B revealing the high spin nature of the complex, corresponding to four unpaired electrons. Electronic spectrum shows a strong band at 20000 cm^{-1} which can be assigned to ligand to metal

charge transfer and a shoulder at 18310 cm^{-1} which may be assigned to the ${}^5E_{2g} \rightarrow {}^5T_{2g}$ transition¹⁹, characteristic of octahedral geometry.

The Co (III) complex is diamagnetic (at 27°C) as expected for a low spin d^6 ion. The electronic spectrum of cobalt (III) complex displayed bands at 15110, 15450, 21095–21640 and 23370–23860 cm^{-1} . These are similar to these reported for other six coordinated cobalt (III) complexes and may be assigned to ${}^1A_{1g} \rightarrow {}^3T_{2g}$, ${}^1A_{1g} \rightarrow {}^1T_{1g}$ and ${}^1A_{1g} \rightarrow {}^1T_{2g}$ transitions, respectively²⁰.

The Fe (III) complex shows magnetic movement 5.20 m_B which indicates high spin octahedral geometry corresponding to five unpaired electrons. The complex shows bands at 17666, 23825, 29880 cm^{-1} corresponding to ${}^6A_{1g} \rightarrow {}^4T_{1g}$, ${}^6A_{1g} \rightarrow {}^4T_{2g}$ and ${}^6A_{1g} \rightarrow {}^4E_g$ transition respectively. All these bands are characteristics of octahedral geometry.

The electronic spectrum of the Ru(III) complex displayed band at 18710, 22620 and 27630 cm^{-1} which has been assigned to ${}^2T_{2g} \rightarrow {}^4T_{1g}$, ${}^2T_{2g} \rightarrow {}^4T_{2g}$ and ${}^2T_{2g} \rightarrow {}^2A_{2g}$, ${}^2T_{1g}$ transitions respectively. There assignment are comparable to these reported for other Ru(III) derivatives involving nitrogen oxygen donor molecules.

The magnetic moment of Cr (III) complex is 3.80 B.M., which suggests the presence of three unpaired electrons. The electronic spectrum showed three bands at 17750, 26200 and 37410 cm^{-1} which are assigned to ${}^4A_{2g} \rightarrow {}^4T_{2g}$ (F), ${}^4A_{2g} \rightarrow {}^4T_{1g}$ (F) and ${}^4A_{2g} \rightarrow {}^4T_{1g}$ (P) transitions respectively for octahedral stereo chemistry.

CONCLUSION

On the basis of study performed and discussed Octahedral geometry has been proposed for all the synthesized M(III) complexes

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