

SYNTHESIS AND CHARACTERIZATION OF LANTHANIDE(III) COMPLEXES WITH 3,4- METHYLENEDIOXYBENZALDEHYDE THIOSEMICARBAZONE

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Abstract—New complexes of lanthanide nitrates with 3,4-methylenedioxybenzaldehyde thiosemicarbazone having the general formula $[Ln(LH)_2(NO_3)_4]NO_3$ (where $Ln = La, Ce, Pr, Nd, Sm, Eu, Gd, Dy, Ho, Er, Yb$ and Y) have been prepared and characterized by elemental analysis, molar conductance, magnetic measurements, electronic, infrared, 1H NMR, fluorescence spectra and thermal studies. Molar conductance studies indicate 1:1 electrolytic behaviour for these complexes. The spectral studies show that two thiosemicarbazone ligands and two nitrate groups are bound to the lanthanide ion in a bidentate fashion. TG and DTA studies indicate that these complexes are stable up to 255°C and do not contain water or alcohol molecules.

Thiosemicarbazones are amongst the most widely studied nitrogen and sulphur donor ligands. They are capable of acting as neutral or charged ligands. The importance of these compounds is mainly due to their anticarcinogenic,¹ antibacterial² and antifungal activities,³ as well as their ability to form chelate complexes with transition metals.^{4,5}

In cancer treatment it has been shown that the metal chelates are more potent than chelating agents.⁶ The donor properties and biological activities of thiosemicarbazones of benzaldehyde and substituted benzaldehyde have been investigated in detail.^{7,8} A thorough survey of the literature reveals that there is no report on the lanthanide complexes of 3,4-methylenedioxybenzaldehyde thiosemicarbazone (LH). This paper, therefore, describes the synthesis and characterization of lanthanide complexes with the above ligand.

EXPERIMENTAL

All the reagents used were of analytical grade. The ligand, LH, was synthesized by refluxing 3,4-methylenedioxybenzaldehyde and thiosemicarbazide (1:1 ratio) in absolute alcohol for 2 h and

recrystallizing the product from ethanol,* m.p. 190–191°C.

Synthesis of the complexes

The following general procedure was used to isolate all the lanthanide(III) complexes. Lanthanide(III) nitrate (0.002 M) was dissolved in absolute alcohol (10 cm³). The ligand, LH (0.004 M), dissolved in the same solvent (ca 20 cm³) was added to the above solution with constant stirring. The mixture was refluxed on a water bath for ca 4 h and the pH of the mixture was then raised to ca 6.5 by the dropwise addition of alcoholic ammonia and again refluxed for 2 h. The solid complexes thus formed were filtered, washed with absolute alcohol and dried *in vacuo* at room temperature.

Physical measurements

Elemental analyses (C, H and N) were performed on an auto-elemental analyser, model 1106. The metal contents were estimated by complexometric EDTA titration using xylenol orange as an indicator¹⁰ at pH 6. Sulphur was determined by the standard method.¹¹ Conductance measurements were performed on 10⁻³ M solutions of the complexes in methanol using an Elico conductivity

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URANIUM (IV) COMPLEXES OF FUROIC ACID HYDRAZONES

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ABSTRACT New complexes of furoic acid hydrazones with Uranium (IV) acetate have been prepared and characterised by physico-chemical, ir and diffused reflectance spectral studies. The metal to ligand ratio in all the complexes was found to be 1:2. Infrared data show that the ligands are monobasic tridentate in nature, coordinating through ONO donor set. Diffused reflectance spectra suggest a cubic geometry.

INTRODUCTION

Coordination compounds of hydrazones have been widely studied due to their varied stereochemical probabilities and wide range of biological activities. In continuation of our earlier work¹ on compounds of U(IV) with schiff bases, we report in this note the synthesis and characterisation of Uranium (IV) complexes with furoic acid hydrazones.

Experimental :

The chemical used in the study are of AR grade.

Synthesis of Ligands :

The ligands were prepared by condensing furoic acid hydrazide with different aromatic aldehydes and ketones in ethanol medium².

Synthesis of complexes :

U(IV) acetate was prepared as reported earlier³. U(IV) acetate (0.01 mol) was refluxed with (0.02 mol) of the ligands in dry benzene medium under nitrogen atmosphere for about 3 hours. The precipitated complex was filtered

Synthesis, characterization and biological studies of uranium(IV), dioxouranium(VI) and thorium(IV) complexes with coumarin bitheterocycles

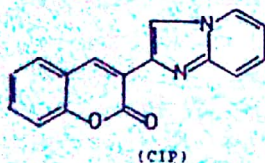
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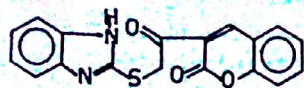
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The complexes of 2-(3-coumarinylthioacetyl) benzimidazole (CTBz) and 2-(3-coumarinyl)imidazo[1, 2-a]pyridine (CIP) with U(IV), UO₂(VI) and Th(IV) have been synthesised and characterized on the basis of their elemental analysis, conductivity, magnetic moment, thermal studies, infrared, PMR and reflectance spectral data. The metal to ligand ratio in all the complexes except U(IV) complexes is found to be 1:2. The ligands behave in a neutral bidentate fashion.

Coumarin and its derivatives have been found to exhibit antibiotic, antifungal, anticoagulating, plant growth regulating and antiinflammatory activities^{1,2} and are also extensively used as analytical reagents³ and in the complex formation⁴⁻⁶. In continuation of our work on metal complexes with coumarin bitheterocycles, we report here the synthesis and characterization of few actinide complexes with CTBz and CIP.



(CIP)



(CTBz)

Experimental

All the chemicals used were of AR grade.

Synthesis of the ligands

Equimolar quantities of 3-bromoacetyl coumarin⁷ and 2-amino pyridine in presence of K₂CO₃ were refluxed in ethanol for 2 h. The resulting solution was filtered and CIP was obtained on concentration of the filtrate. It was further recrystallised from amyl alcohol. CTBz was prepared according to the procedure followed by Kulkarni *et al.*⁸

Synthesis of the complexes

The metal complexes of UO₂ (VI) and Th(IV) with CIP were prepared by stirring the metal salt

solution (0.001 M) in ethanol (10 ml) with CIP (0.002M) in acetone (15 ml) for 3 h. The complexes separated were filtered, washed with ethanol, acetone and dried over fused calcium chloride.

The U(IV) complexes with CIP and CTBz were prepared by refluxing⁹ 0.001 M of U(CH₃COO)₄ in dry benzene with the ligand (0.002M) in dry benzene for 6h under nitrogen atmosphere. The complexes separated were filtered, washed with dry benzene, dried and stored *in vacuo*.

The dioxouranium(VI) complex with CTBz was prepared by refluxing uranyl acetate dihydrate (0.001M) with CTBz (0.002M) in ethanol for 4h. The complex thus separated was filtered, washed with boiling ethanol and dried over fused calcium chloride.

The Th(IV) complex with CTBz was prepared by refluxing thorium nitrate (0.001M) and CTBz (0.002M) in ethanol for 4 h. The resulting solution was concentrated and cooled. The solid separated was filtered, washed and dried.

The complexes were analysed for metal and sulphur by standard methods and nitrogen was estimated by Dumas method. The conductance measurements were made on an ELICO-CM-82 conductivity bridge. The magnetic measurements at room temperature were made on Gouy balance and diamagnetic corrections for the ligands were made using Pascals constants. The infrared spectra were recorded on a Perkin-Elmer 577 spectrophotometer in the region 4000-200 cm⁻¹. The PMR spectra were recorded on a Varian EM-398 spectrometer.

Results and discussion

The physical data of the complexes are summarised in Table 1. The analytical and conductance data suggest the formation of [UO₂L₂(Ac)₂], [ThL₂(NO₃)₄] and [UL(Ac)₄] type complexes. The non-electrolytic nature of the complexes is revealed by the low molar conductance values. The magnetic moment values of U(IV) complexes agree with the presence of two unpaired electrons.

The bands assigned in the reflectance spectra of U(IV) complexes are based on the earlier studies^{10,11}. The reflectance spectra of U(IV) complexes can be put into the medium intensity type class¹⁰⁻¹² and resemble with those of six coordinated U(IV) complexes. This resemblance

SYNTHETIC, SPECTRAL, THERMAL AND BIOLOGICAL STUDIES OF
LANTHANIDE(III) COMPLEXES WITH A SCHIFF BASE DERIVED FROM
3-N-METHYLPYPERIDINO-4-AMINO-5-MERCAPTO-1,2,4-TRIAZOLE

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ABSTRACT

A series of Ln(III) complexes of the type $[\text{Ln}(\text{HPSMT})_2\text{NO}_3] \cdot n\text{H}_2\text{O}$ (where Ln = La, Ce, Pr, Nd, Sm, Eu, Gd, Tb, Dy, Er, Yb, Y; HPSMT = 3-N-methylpyperidino-4-salicylideneamino-5-mercapto-1,2,4-triazole and n = 1-3) have been synthesized by the direct reaction of 3-N-methylpyperidino-4-salicylideneamino-5-mercapto-1,2,4-triazole with Ln(III) nitrate in dry alcohol. The complexes have been characterized by elemental analyses, molar conductance, magnetic measurements, electronic, infrared, and ^1H NMR studies. The elemental analyses reveal 1:2 stoichiometry with respect to metal:ligand. Molar conductance studies indicate the non-electrolytic nature of the complexes. The spectral studies show the ligand HPSMT and nitrate groups bound to the Ln(III) ion in a bidentate fashion. It is proposed that the complexes

Novel transition complexes of Co(II), Ni(II), Cu(II), and Zn(II) derived from 3-[*p*-(5-substituted salicylidinehydrazinocarbonyl)]phenylsydnone

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Schiff bases derived from 5-substituted salicylidine 3-[*p*-(5-substituted salicylidinehydrazinocarbonyl)]phenylsydnones and their transition metal complexes with Co(II), Ni(II), Cu(II), and Zn(II) have been synthesized and characterized by elemental analysis, molar conductance, magnetic susceptibilities, electronic, infrared, Proton Magnetic Resonance (PMR) (¹H NMR), Fast Atomic Bombardment-(FAB)-mass spectra, Electron Spin Resonance (ESR), and thermal studies. From the above spectral studies, it is concluded that the ligands *viz.*, 3-[*p*-(salicylidinehydrazinocarbonyl)]phenylsydnone (L₁); *p*-(vanillidine hydrazinocarbonyl)]phenylsydnone (L₂); and 3-[*p*-(5'-chlorosalicylidinehydrazinocarbonyl)]phenylsydnones (L₃) act as bidentate molecules coordinating through azomethine nitrogen and phenolic oxygen. The ligands and their metal complexes have been screened *in vitro* for antibacterial, antifungal, and antitumor activities. The results indicate that the biological activity has increased on complexation. The Cu(II) complexes of the above-said ligands have proven to be antitumor agents toward the P388/s tumor cells at lower concentrations.

Keywords: sydnone; bidentate; antibacterial; antifungal; antitumor

1. Introduction

The structural features of mesoionic compounds have been of interest to medicinal chemist. A wide spectrum of biological activity has been claimed for a variety of mesoionic compounds [1]. Their potential value as biological active substances can be explained by their dipolar structure. These ring systems are composed of delocalized electrons, which have been appreciably perturbed. As a result of perturbation of electrons in mesoionic system, there is an oppositely charged dipolar segment at the extrema of a four-atom chain. The presence of oppositely charged dipolar segment in mesoionic systems is of tremendous value to the medicinal chemist [2]. Its significance perhaps lies in its ability to electrostatically interact with two complementary partially charged positions on receptor macromolecule, such as a protein helix. Another structural feature of the mesoionic system is their highly charged, yet net neutral, electrical character. Therefore, they are soluble to a much

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**Estimation of the dipole moments of the excited state of
di(2-methyl-6-chlorophenyl)carbazone and its
Co(II), Ni(II) and Zn(II) complexes from the effect
of solvent on their ultraviolet absorption spectra**

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Abstract: Di(2-methyl-6-chlorophenyl)carbazone (2M6CPC) and its Co(II), Ni(II) and Zn(II) complexes were synthesized and characterized by magnetic moment, and infrared and ¹H-NMR spectral measurements. The solvent effect in a series of polar and non-polar solvents of varying dielectric constants and refractive indices was estimated by recording the electronic spectra (S1 band) of the above compounds. The data was used to determine the magnitude and direction of the electric dipole moments in the first electronically excited state. The results indicate that the observed band systems in these compounds may be attributed to a $\pi \rightarrow \pi^*$ transition.

Keywords: carbazone; electric dipole moments; dielectric constants; $\pi \rightarrow \pi^*$ transition.

INTRODUCTION

Transition metal complexation has played a vital role in the fields of biochemistry and medicine.¹ After work on the chelation of biologically active ligands,² it was thought justifiable to study the dipole moments of some adducts. A survey of the literature revealed that in addition to investigating the problems of electronic structure, bonding stereochemistry and stability constants of metal complexes with diphenyl carbazone (DPC)³ and its derivatives, an increasing number of studies have been devoted to the dynamic and mechanism of the reaction of metal complexes and their heterocyclic nitrogen base adducts.^{4,5} Through spectrophotometry, the synthesis and characterization of Co(II), Ni(II) and Zn(II) complexes with di(2-methyl-6-chlorophenyl)carbazone (2M6CPC) and also the electric dipole moments of these compounds in the first electronically excited

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Synthesis, characterization and biological studies of Co(II), Ni(II), Cu(II) and Zn(II) complexes of Schiff bases derived from 3-formyl-2-mercaptoquinolines

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ABSTRACT

Co(II), Ni(II), Cu(II) and Zn(II) complexes have been synthesized by the Schiff bases derived from 3-formyl-2-mercaptoquinoline with 4-nitro-ortho-phenylenediamine. The synthesized Schiff bases and chelates were characterized by elemental analysis, molar conductance, magnetic susceptibilities, UV-Fluorescence, IR, ¹H-NMR, Fabmass, ESR and thermal studies. The results indicate that, the ligand acts as a octahedral geometry. The ligands and their metal complexes have been screened in vitro for antibacterial and antifungal studies. The results showed that the biological activity of the ligands get increased on complexation. The Cu(II) and Ni(II) complexes are found to be potent in DNA cleavage studies.

Keywords: Quinoline; coordination; metal complexes; NMR; antibacterial, antifungal.

INTRODUCTION

Quinolines are a class of nitrogen heterocycles, which are an integral part of a large number of natural and synthetic compounds which play important roles in many biological systems [1,2]. As a structural subunit in many natural products, the quinoline ring system is one of the most commonly encountered heterocycles in medicinal chemistry. A literature survey revealed that substituted quinolines possess diverse chemotherapeutic activities including antibacterial [3, 4], antifungal [5, 6], anti-amoebic [7,8], antileishmanial [9,10], antimalarial [11,12], and antitumor [13-15], immunosuppressive [16], analgesic, vasorelaxing [17], antiplasmodial [18], anticonvulsant, and antihypertensive [19].



Synthesis, spectral characterization, antimicrobial and cytotoxicity studies of some lanthanide(III) complexes of quinoline derivatives

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ABSTRACT

The new lanthanide(III) complexes with quinoline derivative (MQAP) obtained by the reaction between 3-formyl-2-mercapto quinoline with 4-amino pyridine have been synthesized and characterized by elemental analysis, molar conductance, magnetic moment measurements, FT-IR, electronic spectra, ¹H-NMR spectra, FAB mass spectra, and thermal analysis studies. The molar conductance studies indicate non-electrolytic behavior of the complexes. The IR and ¹H-NMR studies indicate that MQAP acts as mono basic bidentate ligand of potential donor site of azo methine nitrogen and thiolate sulphur. TG and DTA studies of La(III) and Sm(III) complexes indicate the presence of two coordinated water molecules along with two nitrate and two MQAP moieties. Based on these studies the complexes have been formulated as [Ln(MQAP)₂(H₂O)₂](where Ln = La, Pr, Nd, Sm, Eu, Gd, Yb) with coordination number 8. The cytotoxicity of the ligand and Ln(III) complexes have been screened against cancer cells line HeLa (Cervical cancer cells, Human), which reveals that Ln(III) complexes shows moderate cytotoxicity against tested cell lines.

Key words: Lanthanum(III), coordination, cytotoxicity.

INTRODUCTION

Coordination chemistry of lanthanides is one of the active research fields in inorganic chemistry [1-7]. It seems from the published work that lanthanides are capable of forming stable complexes with quinoline and its derivatives. The work on quinoline Schiff's base complexes have been reported from our laboratory with transition elements [8-9]. As a part of a programme for the synthesis and characterization of solid complexes of lanthanides, presently, complexes of seven lanthanide(III) ions, viz. La(III), Pr(III), Nd(III), Pm(III), Sm(III), Eu(III), Gd(III) and Yb(III) with 3-formyl-2-mercaptoquinoline synthesized. These complexes were characterized by elemental analysis, molar conductance measurements, magnetic moment, infrared, electronic and proton nmr spectra. The TG studies of the lanthanide(III) complexes were carried out in dynamic air, with a heating rate of 10 °C/min.

EXPERIMENTAL SECTION

All the chemicals used were of analytical grade purchased from Aldrich, Merck, S.D. Fine, or BDH and the solvents used were purified by standard methods [10]. The complexes were analyzed for their metal content by standard methods [11]. Elemental analyses (C, H and N) were performed on a Perkin- Elmer 2400 CHN elemental Analyzer Model 1106, Carloerba Strumentazione. The IR spectra of the Schiff bases and their La(III) complexes were recorded on a HITACHI-270 IR spectrophotometer in the 4000-250 cm⁻¹ region using KBr disks. Molar conductivity measurements were recorded on an ELICO-CM-82 T conductivity bridge with a cell having cell constant 0.51. The electronic spectra of the complexes were recorded in DMF on a VARIAN CARY 50-BIO UV-spectrophotometer in the region of 200-1100 nm. The ¹H-NMR spectra of Schiff bases and complexes were

Synthesis, spectral characterization of Co(II), Ni(II), Cu(II) and Zn(II) complexes of Schiff bases derived from 3-formyl quinoline and 2,6-diaminopyridine and their biological studies

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Abstract. A series of Schiff bases derived from 2-mercapto-3-formyl quinoline/2-hydroxy-3-formyl quinoline with 2,6-diaminopyridine (DAP) and their corresponding transition metal complexes were synthesized, characterized by elemental analysis, spectral (IR, ¹H-NMR, Mass, UV-Vis, Fluorescence, ESR, TGA) and magnetic studies. The molar conductances in DMF solution indicated that the complexes were non-electrolytic. The Schiff bases and their metal complexes were screened for analgesic, anti-inflammatory and antipyretic activities. Among all the compounds, the copper complexes had the greatest pharmacological activities.

Keywords: Quinoline, diaminopyridine, coordination, antipyretic, analgesic, anti-inflammatory

1. Introduction

Nitrogen heterocyclic compounds have been used widely in the pharmaceutical industry because of their useful biological activities [1–5]. Some derivatives of 2-hydroxy quinoline have also been synthesized and investigated, as they exhibit some preferable biological activities such as antioxidant, anti-proliferative, anti-inflammatory and anticancer activities [6–9]. Also, some Schiff-bases and their metal complexes

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ANTIMICROBIAL STUDIES OF Co (II), Ni (II), Cu (II) AND Zn (II) COMPLEXES DERIVED FROM SCHIFF BASES OF 3-FORMYL QUINOLINE AND 3-HYDRAZINOQUINOXALIN-2(1H) ONE

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ABSTRACT

Cobalt(II), nickel(II), copper(II) and zinc(II) complexes of two new multi-dentate ligands, 3-[(2E)-2-[(2-hydroxyquinolin-3-yl)methylidene]hydrazinyl]quinoxalin-2-ol(QZOH) and 3-[(2E)-2-[(2-sulfanyl quinolin-3-yl)methylidene]hydrazinyl]quinoxalin-2-ol(QZSH) were prepared and characterized by elemental analysis, conductance, thermal, spectral and magnetic data. The ligand QZOH de-protonates to give a monobasic ONO donor and binds to the divalent metal ions in bis-tridentate fashion, using two ONO donor sets. The other ligand QZSH also de-protonates to give a monobasic SNO donor and binds to the divalent metal ions in bis-tridentate fashion, using two SNO donor sets. Octahedral geometries are proposed for all these complexes, and preliminary studies show that they possess potential antimicrobial activity.

Keywords: Quinoline, Quinoxaline, monobasic tri-dentate, antimicrobial.

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INTRODUCTION

Quinoxaline derivatives exhibit a wide variety of biological activities. It has been reported that some quinoxalines demonstrated antibacterial, antifungal, antiviral, antineoplastic, antidepressant, hypoglycemic, anti-inflammatory, excitatory amino acid antagonistic, antiglaucoma, antiparasite, antituberculosis, anticancer, and anti HIV-1 activities¹⁻¹³.

However literature survey revealed that Schiff base containing Quinoxaline and Quinoline has not been prepared and studied so far. Thus the present study deals with synthesis and characterization metal complexes derived from Schiff bases of 2-substituted-3-formyl Quinoline and 3-hydrazinoquinoxalin-2(1H) one.

EXPERIMENTAL

All the reagents are of reagent grade were used as received. All the chemicals and solvents used were dried and purified by standard methods, and moisture was excluded from the glass apparatus using CaCl₂ drying tubes.

Synthesis of 1,4-Dihydrquinoxaline -2,3- dione

A solution of oxalic acid dihydrate (0.238mole, 30g) in H₂O (100ml) was heated to 100°C and conc.HCl(4.5ml) was added, followed by Ophenyldiamine (0.204 mole, 22g) with stirring, temperature was maintained at 100°C for 20 min. the mixture cool by addition of ice. The precipitate was formed and washed with water. Crystallized from 5% NaOH /dil.HCl, yield : 98%. m.p. = >300°C, color: white.

Synthesis of 3-hydrazinoquinoxalin-2(1H) one (a)

A mixture of the 1,4-Dihydrquinoxaline-2,3- dione (0.062mole, 10.04g), hydrazine hydrate 99.9% (1mole, 50ml) and water (50ml) was heated under reflux for 2h, then cooled to room temperature, the precipitate was filtered, washed with water and crystallized from 2-butanol. m.p: >300°C, yield: 84%, color: yellow needles.