

Synthesis, Spectral And Antimicrobial Studies Of Some Thorium (IV) And Dioxouranium (VI) Complexes With Nitrogen Donor Ligand

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Abstract

We report here series of new the Th(IV) and UO₂ (VI) complexes with Schiff base having general composition Th X₄.nL (X=NO₃, n=2) and UO₂X₂.nl (X=CH₃ COO, n=2), Where L = Schiff base. The complexes were characterized on the basis of analytical conductance, molecular weight and spectral studies. The Schiff base behaves as neutral monodentate ligand which coordinate to the central metal atom through azomethine nitrogen.

Key words: Schiff base ligand, Th(IV) and UO₂ (VI).

1- INTRODUCTION

A number of complexes with linear UO₂ (VI) ion in 6- or 8- coordinator number and with th(IV) in 6- , 8- or 10 coordination number known ref (1-6). In the present work, we wish to report the synthesis and characterization of series of complexes of these metal ions with Schiff base ligand which is derived from the condensation of p-toluidine and 4-NN-bis -2'-cyanoethylaminobenzaldehyde.

Preparation of 4-(NN bis - 2'-Cyanoethyl) amino benzaldehyde:-

It was moduled on the procedure give in the literature ref. J.T. Brain Holtz F.g. Mann I. chem.. Soc. 1817 (1953)

Ref. V.S. Jolly and P.I. Ittyrah J. Indian Chem. Soc. 46, 997 (1969)

Preparation of Schiff base ligand:-

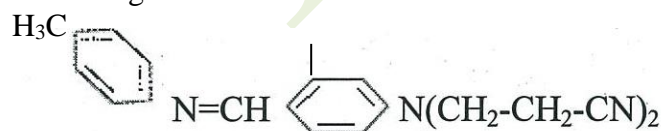
A mixture of the aldehyde (1 mmol) and the P-toluidine (1 m.mol) in absolute ethanol in taken in a round bottom flask and two drops of piperdine were added. The mixture was refluxed for 4-5 hrs. On cooling dark coloured solid separated which was filtered under suction and recrystallized, from ethanol as yellow solids.

M.P. -146°C

Yield -82%

Colour -Yellow

Stru.of Ligand

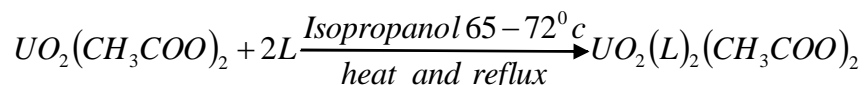
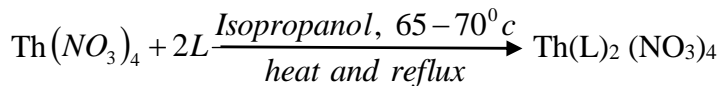


4-NN-bis-2'- Cyanoethyl amino benzyldine-p-toluidine (4CABPT)

Synthesis of complexes:-

The respective metal salt solutions were treated with ligand solution in the required molar concentrations. In some of the cases complexes were isolated immediately in cold while in some cases in not solutions. In other cases the resulting solutions were refluxed for 2-3 hrs at Ca

65-70⁰c. The solvents uses were ethanol, Isopropanol or acetone. The complexes were collected washed with the solvents and finally with ether and dried in vacuo over anlyd. CaCl₂.



The analytical data Table – 1 indicate that the complex are non-ionic in nature the complexes are fairly stable at room temperature except. The Iodo complexes which convert in to stickly mass after some time (7-12)

Table – 1

Complexes	Colour	M.P.	Yield	M.W. found (calcd.)	Analysis			\wedge (Ohm ⁻¹ cm ² mole ⁻¹)
					Found	(Calcd.) Analysis		
					C	H	N	
Th(NO ₃) ₄ ·2(4CABPT)	yellow	176	81	1012 (1014)	47.18 (44.11)	4.28 (4.70)	15.24 (15.44)	3.1
UO ₂ (CH ₃ COO) ₂ ·2(4CABPT)	Light Yellow	168	74	980 (1022)	48.10 (51.10)	4.20 (4.50)	10.70 (10.80)	4.1
ThI ₄ ·4(4CABPT)	Light yellow	146	781	1214 (2008)	50.10 (47.90)	4.20 (3.90)	11.80 (11.17)	53.2
UO ₂ (NO ₃) ₄ ·2(4CABPT)	Orange	145	86	1010 (1028)	44.15 (47.10)	4.10 (3.80)	13.12 (13.90)	3.6

Electronic Spectral Studies:-

The electronic spectral studies of these complexes are of loss interest since metal ion does not contain any unpaired electrons in its outer most shell. All the complexes which are studies on the bases of electronic spectra exhibit $n \rightarrow \pi^*$ bands which are around 240-220 nm and bands at 330-250 nm which corresponds to $\pi - \pi^*$ transition (13-16).

Suggested structures of the complexes:-

The preferred coordination number of Th(IV) metal atom is 6 or 10 but higher coordination numbers have also been observed (15). It has been observed by conductance and molecular weight value. The nitrate group are linked to through two oxygen atoms, each nitrate group functioning as a bidentate ligand (16). In the nitrate complex of this ligand the thorium metal is 10 coordinated as it is surrounded by eight coordinated oxygen atoms and two azomethine nitrogen atoms.

For dioxouranium (VI) acetato complex I.R. data reveal that the anions are bidentately covalently bonded to the metal atom there by generating an 8-coordination number on the central Uranium atom.

Anti-microbial Studies

Anti-microbial namely antifungal, antibacterial studies of complexes as well as that of organic compounds were the field the interest of various workers [17,-19]. Antifungal/Antibacterial activities against various pathogens using Thorium (VI) and dioxouranium (IV) compounds have been reported in the past [20-27].

2- MATERIAL AND METHODS

Antimicrobial activity was determined by disc diffusion method. Filter paper disc measuring 6.0 mm diameter were cut with a punch from Whatman filter paper No.1 sterilized at 160°C in hot air oven for one hour, antimicrobial solutions of desired concentration were dropped on discs and were dried in incubator in 37 °C and stored in freezer. Nutrient agar was poured into plates,keeping depth of the medium 4.0 mm. After the medium solidified,the plates were kept for 30 minutes in an incubator (35 to 55 °C) to remove excess of moisture,4-5 colonies of pure culture on Nutrient agar /Sabouroud's Dextrose agar were transferred into a culture tube containing peptone water with the help of wire loop.The culture was incubated by lawn culture method. A loop full of 2 mm diameter was used to streak the plates with the test organisms and kept it for 5-10 minutes at room temperature.

The discs were removed with the help of flamed forceps from their respective vials and placed in the plate 15 mm away from the edge,at equal distance and sufficiently separated from each other to avoid overlapping of zone of inhibition, finally pressed them lightly with forceps to make complete contact with surface of medium

.The plates were incubated at 35-38°C for 24-30 hours.

3- CULTURE MEDIA USED

Nutrient agar

Agar 2% was added to prepare nutrient agar for antimicrobial sensitivity test. The pH was maintained between 7.4-7.6.

Compositio

Peptone	=	10g
Sodium Chloride	=	05g
Beef extract	=	10g
Distilled water	=	1000ml.

After addition of 2% agar , media was autoclaved at 15-25 minutes and poured in sterilized plates.

Sabouraod's Dextrose agar

Composition

Peptone	=	10.0g
Dextrose	=	40.0g
Agar	=	20.0g
Distilled water	=	1000ml

In the present study new synthesized I to XII were first dissolved in dimethylsulphoxide or acetone to prepare the drug impregnated disc used in anti-bacterial and anti-fungal by disc diffusion technique. Organism tested were E.coli Pseudomonas, Aspergillus niger, and Candida albicans Diameter of zone of inhibition was measured in millimeter and reported as (+) for intermediate zone (partially sensitive), (++) for sensitive zone, (+++) for resistant zone,(+++++) for significant resistant zone and (-) for no inhibition zone.

Table-2

Anti-bacterial activity of some new Complexes

Solubility data - All Solutions were prepared in DMSO

Concentration data	-
Medium	-	Nutrient agar
pH range	-	7.4 to 7.6
Period of growth	-	24 to 48 hrs.

S.No.	Name of Complexes	E. coli	Pseudomonas species
I	Th(NO ₃) ₄ .2(4CABPT)	-	+
II	ThI ₄ .4(4CABPT)	+	-
III	UO ₂ (NO ₃) ₂ .2(4CABPT)	++	-
IV) UO ₂ (CH ₃ COO) ₂ .2(4CABPT	-	+++

Table-3

Anti-fungal activity of some new complexes

Solubility data – All solutions were prepared in DMSO

Concentration tested -

Medium - Sabouraud's Dextrose agar

pH range - 7.5

Period of growth - 4 days

S.No.	Name of Complexes	Aspergillus niger	Candida Species
I	Th(NO ₃) ₄ .2(4CABPT)	+	-
II	ThI ₄ .4(4CABPT)	++	++
III	UO ₂ (NO ₃) ₂ .2(4CABPT)	-	++
IV	UO ₂ (CH ₃ COO) ₂ .2(4CABPT)	-	++++

Zone of inhibition

(+) 0-4 mm (++) 4-8 mm (+++) 8-12 mm (++++) 12-16 mm (-) No inhibition

4- RESULTS AND DISCUSSION

The analytical data Table – 1 temperature indicate that the complex are non-ionic in nature the complexes are fairly stable at room except. The Iodo complexes which convert in to stickly mass after some time.Four new synthesized I to IV Complexes were screened for their anti bacterial activity against several species of E. coli, and Pseudomonas species using agar plate diffusion technique. The testing were carried out in dimethylsulphoxide solution at a concentration of 30 gm/ml. Ofloxacin and tetracycline were used as the standard drugs.Results are assembled in Table 6.1.

Anti-fungal activity

Four new complexes (I to IV) were screened for antifungal activity against Aspergillus niger and Candida species at concentration of 30gm/ml Using Sabouraud's Dextrose agar media disc diffusion technique.The testing was carried out in dimethylsulphoxide solution.Amphotericin B discs were used as the standard drugs. Results are assembled in Table 6.2.

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