

**STUDIES ON URANYL (UO_2^{2+}) ION ACTIVITIES WITH
SCHIFF BASE DONORS IN NON-AQUEOUS
ENVIRONMENT**

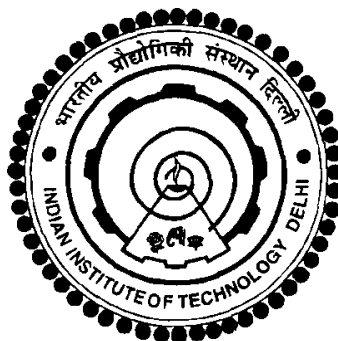
By

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Submitted

**In fulfillment for the requirements of the degree of Doctor of Philosophy
to the**



INDIAN INSTITUTE OF TECHNOLOGY DELHI

Feb 2022

Dedicated to

In Memory of My Grandfather

Sh. Suraj Bhan Singh Yadav

(Heavenly abode)

And

My beloved parents

Sh. Mahender Singh and Mrs. Krishana Yadav

Certificate

*This is to certify that the thesis entitled, “Studies on Uranyl (UO_2^{2+}) ion Activities with Schiff Base Donors in non-aqueous environment”, being submitted by Ms. Renu Kumari, to the **Indian Institute of Technology Delhi** for the award of degree of ‘**Doctor of Philosophy**’ in Chemistry is a bonafied research work carried out by her. Ms. Renu Kumari has worked under my guidance and supervision and has fulfilled the requirements for the submission of thesis, which to my knowledge has reached the requisite standard. The results contained in this thesis have not been submitted in part or in full, to any other University or Institute for award of any degree or diploma.*



Date: Feb. 10, 2022

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ACKNOWLEDGEMENTS

*I would like to thank my research supervisor **Prof. Jai Deo Singh** for his guidance, encouragement, and support throughout my research work. You have been a great mentor for me and your guidance on both research as well as on my career have been invaluable. I could not have imagined having a better mentor for my research work.*

*I am thankful to **Prof. N. D. Kurur** (Head), **A. J. Elias**, **Prof. Ravi Shankar**, and **Prof. A. K. Ganguly** (former Heads), Department of Chemistry, IIT Delhi for providing departmental facilities during the course of my research work.*

My sincere thanks also go to Mr. Alok and Mr. Keshav for helping me in NMR instrumentations, the entire instrumental lab staff, Department of Chemistry, IIT Delhi for physicochemical and spectral studies throughout the course of research work.

*I would like to express my special thanks to **Prof. Ray J. Butcher**, Dept. of Chemistry, Howard University, Washington D.C., U.S.A.; for helping me in determining crystal structures of some of the complexes during the research work.*

I am very thankful to all M.Sc. lab staff specially Mr. Bhupendra Singh for providing space as well as some common laboratory chemicals to carry out experimental work.

*I sincerely acknowledge the **University Grant Commission (UGC)**, Government of India for providing the financial assistance to carry out my research work.*

I would like to extend my gratitude to senior members of my research laboratory, Dr. Abhishek Kumar, Dr. Dolly Yadav, Dr. Mantesh Kumari Yadav, Dr. Anuj Kumar Baliyan and Dr. Jagriti Singh for their continuous support and suggestions. My special thanks to my laboratory colleagues, Deepa Bhardwaj, Jyoti Punia, Kaushal Kishore, Preeti Mishra, Dharmendra and Rahul for healthy and friendly support and encouraging scientific atmosphere in the laboratory.

I am thankful to my colleagues, Rakesh, Ankita, Mayank, Rajan, Anchala, Kunvar Om, Sanju, Ritesh, Anoop, Lahur Mani Verma, Sonali and Kaushal, Kapil, Divyansh for their friendly appearance, who joined this laboratory for their M.Sc. and M.Tech. project work.

A special thanks to my friends Dr. Pragati, Aashna and Mayuri for their support and help during the course of my research work.

I owe my deepest gratitude towards my family members for their continuous support and encouragement especially to my parents Mr. Mahender Singh and Mrs. Krishana Yadav for their patience and sacrifice and without this I would not have been able to become visible in research world. I am also thankful to Mr. Jaspal Singh, Mrs. Sunita Yadav, Mr. Ombir Singh, Mrs. Beena Yadav. I would also like to thank my siblings Vijit, Neha, Amit, Sumit, Ritu, Nikhil, shikha, Nikita, Aaradhya, Ankush and Sujata for their infallible love and support. The list is incomplete without acknowledging my cute little nephew 'Avyaan' and my cute princess 'Avisha' whose small activities always made me smile in challenging periods.



RenuKumari

ABSTRACT

Aiming the critical aspects as indicated in the introductory remark (Chapter I) and in knowing the behavior of UO_2^{2+} ion in nonaqueous medium, the current thesis research work entitled “*Studies on Uranyl (UO_2^{2+}) Ion Activities with Schiff Base Donors in Non-aqueous Environment*” was undertaken with a view to examine the behavior of UO_2^{2+} ion to further exploit the different level of covalency and subsidiary interactive behavior of uranyl ion in anhydrous medium. The thesis is divided into six chapters including introduction (Chapter I) and materials and methods (Chapter II) and the findings of the present thesis research work in four Chapters III-VI

Chapter III describes the synthesis of mononuclear uranyl (VI) Schiff base complexes of general formula, $[(\text{UO}_2)(\text{saldien})(\text{pyridine})]$ (C_1) (saldien^{2-} = disalicylidenediethylenetriaminate) and $[(\text{UO}_2)(\text{MeO-saldien})(\text{CH}_3\text{CN}/\text{H}_2\text{O})]$ (C_2) (MeO-saldien^{2-} = di-*o*-methoxysalicylidenediethylenetriaminate) and their characterization by IR, ^1H NMR and X-ray crystallography. In complex species (C_1) instead acting as donor, pyridine was found as solvent molecule embedded in the crystal structure of the complex species with weaker interactions. While, the isolated crystals of complex species (C_2) from acetonitrile consisted of two molecules $[(\text{UO}_2)(\text{MeO-saldien})(\text{CH}_3\text{CN}$ or $\text{H}_2\text{O})]$ in asymmetric unit exhibiting a competitive ($\text{CH}_3\text{CN}/\text{H}_2\text{O}$) scenario without any direct coordination to uranyl ion. This forms an alternative basis in recognizing the reaction pathways for uranyl ion (hydrolysis v/s. coordination) if both water and another solvent are present at the same time in reactions.

Chapter IV describes the synthesis of donor bases (H_3L) (N_2O_3 -type donor) derived from salicylaldehyde or substituent-salicylaldehydes and 1,3-diamino-2-propanol. The

trianionic N_2O_3 donor sites of these bases (H_3L) are capable of displaying an equatorial coordination plane to UO_2^{2+} ions which results the formation dinuclear uranyl complexes. The remaining coordination site of each uranyl ion is further completed by water and solvent molecules resulting in a pentagonal bipyramidal geometry for uranyl cations. These complexes have been studied in the solution state for their behavior and finally solid-state structures are established by single-crystal X-ray studies. The structural trends in uranyl complexation in different solvents such as CH_3OH , C_2H_5OH , $n-C_4H_9OH$, $tert-C_4H_9OH$ and DMSO are noticeable with the view of a competitive behavior of solvent/water allowing to see the influential role of water in coordination scenario.

Chapter V discusses the synthesis and characterization of potentially heptadentate (N_4O_3) tripodal-type Schiff bases of enough hydrolytic stability and their responsive behavior towards UO_2^{2+} in non-aqueous medium. The interactive behavior of a uranyl ion with the tripodal-type Schiff bases in non-aqueous medium revealed a striking behavior. The structural investigation on the interactive behavior leads to the isolation of species which seems to be formed as a result of hydrolytic activity in reaction medium. Although the uranyl capturing by Schiff bases was progressively noticeable, even then an actual reaction scenario in solution also needs to be addressed before claiming them as an extractant.

Chapter VI describes the synthesis of 2,6-bis (benzothiazol-2-yl)-4-(alkyl) phenol derivatives (L_1 and L_2) from 2-aminothiophenol and 4-alkyl-2,6-diformylphenol and their $U^{VI}O_2^{2+}$ complexes. The characterization of donor species and their complexes has been made by physicochemical and spectroscopic methods including single crystal X-ray. A comparative solution and solid-state study of uranyl complexes was carried out and the findings are presented herein.

शोध प्रबंध का सार

परिचयात्मक टिप्पणी (अध्याय I) में बताए गए महत्वपूर्ण पहलुओं को लक्षित करते हुए और गैर-जलीय माध्यम में UO_2^{2+} आयन के व्यवहार को जानने में, वर्तमान थीसिस शोध कार्य "यूरेनिल पर अध्ययन (UO_2^{2+}) आयन गतिविधियां गैर-जलीय में शिफ बेस दाताओं के साथ एनहाइड्रस माध्यम में यूरेनिल आयन के सहसंयोजकता के विभिन्न स्तर और सहायक अंतःक्रियात्मक व्यवहार का और अधिक दोहन करने के लिए UO_2^{2+} आयन के व्यवहार की जांच करने के उद्देश्य से पर्यावरण" शुरू किया गया था। थीसिस को छह अध्यायों में विभाजित किया गया है जिसमें परिचय और सामग्री और वर्तमान शोध कार्य के तरीकों और निष्कर्षों की चर्चा चार अध्यायों (III-VI) में की गई है। थीसिस को छह अध्यायों में विभाजित किया गया है जिसमें परिचय और सामग्री और वर्तमान के तरीके और निष्कर्ष शामिल हैं। थीसिस शोध कार्य की चर्चा शेष अध्यायों (III-VI) में की गई है।

अध्याय III सामान्य सूत्र के मोनोन्यूक्लियर यूरेनिल (VI) शिफ बेस कॉम्प्लेक्स के संश्लेषण, $[(\text{UO}_2)(\text{saldien})(\text{pyridine})]$ (C_1) ($\text{saldien}2\text{-}=\text{disalicylidenediethylenetrimate}$) और $[(\text{UO}_2)(\text{MeO-saldien}) (\text{CH}_3\text{CN}/\text{H}_2\text{O})]$ (C_2) ($\text{MeO-saldien}2\text{-}=\text{di-o-methoxysalicylidenediethylenediethylenetrimate}$) और IR, ^1H NMR और एक्स-रे

क्रिस्टलोग्राफी द्वारा लक्षण वर्णन का वर्णन करता है। जटिल प्रजातियों (C_1) में दाता के रूप में कार्य करने के बजाय, पाइरीडीन को कमजोर अंतःक्रियाओं के साथ जटिल प्रजातियों के क्रिस्टल संरचना में एम्बेडेड विलायक अणु के रूप में पाया गया। जबकि, एसिटोनिट्राइल से जटिल प्रजातियों (C_2) के पृथक क्रिस्टल में दो अणु [(UO₂) (MeO-saldien) (CH₃CN या H₂O)] शामिल होते हैं, जो यूरेनिल से किसी प्रत्यक्ष समन्वय के बिना एक प्रतिस्पर्धी (CH₃CN/H₂O) परिदृश्य प्रदर्शित करते हैं। आयन यह यूरेनिल आयन (हाइड्रोक्सिलिस बनाम समन्वय) के लिए प्रतिक्रिया पथ को पहचानने में एक वैकल्पिक आधार बनाता है यदि प्रतिक्रियाओं में एक ही समय में पानी और एक अन्य विलायक दोनों मौजूद हैं।

अध्याय IV सैलिसिलिडहाइड या सबस्टीटेंट-सैलिसिलिडहाइड और 1,3-डायमिनो-2-प्रोपेनॉल से प्राप्त डोनर बेस (H₃L) (N₂O₃-टाइप डोनर) के संश्लेषण का वर्णन करता है। इन आधारों (H₃L) के त्रिकोणीय N₂O₃ दाता स्थल UO₂²⁺ आयनों के लिए भूमध्यरेखीय समन्वय विमान प्रदर्शित करने में सक्षम हैं, जिसके परिणामस्वरूप डाइन्यूक्लियर यूरेनिल कॉम्प्लेक्स बनते हैं। एक परमाणु प्रजाति के प्रत्येक यूरेनिल आयन के शेष समन्वय स्थल को पानी और विलायक अणुओं द्वारा पूरा किया जाता है जिसके परिणामस्वरूप यूरेनिल केशन के लिए पंचकोणीय द्विपिरामिड ज्यामिति होती है। इन परिसरों का उनके व्यवहार के लिए

समाधान अवस्था में अध्ययन किया गया है और अंत में एकल-क्रिस्टल एक्स-रे विवर्तन अध्ययन द्वारा ठोस अवस्था संरचनाएं स्थापित की गई हैं। CH_3OH , $\text{C}_2\text{H}_5\text{OH}$, $n\text{-C}_4\text{H}_9\text{OH}$, $\text{tert-C}_4\text{H}_9\text{OH}$ और डाइमिथाइलसल्फॉक्साइड (DMSO) जैसे विभिन्न सॉल्वेंट्स में यूरेनिल कॉम्प्लेक्सेशन में संरचनात्मक रुझान विलायक / पानी के प्रतिस्पर्धी व्यवहार के दृष्टिकोण से ध्यान देने योग्य हैं जो पानी की प्रभावशाली भूमिका को देखने की अनुमति देते हैं। समन्वय परिदृश्य। ये निष्कर्ष हमेशा बेहतर पृथक्करण की खोज और गैर-मीडिया में यूरेनिल आयन प्रजाति के अध्ययन के लिए उपयोगी हो सकते हैं, जहां पानी एक अन्य विलायक के साथ समवर्ती रूप से प्रतिस्पर्धी है।

अध्याय V में पर्याप्त हाइड्रोलाइटिक स्थिरता के संभावित हेप्टाडेंटेट (N_4O_3) ट्राइपोडल-टाइप शिफ बेस के संश्लेषण और लक्षण वर्णन और गैर-जलीय माध्यम में UO_2^{2+} के प्रति उनके उत्तरदायी व्यवहार पर चर्चा की गई है। हालांकि, गैर-जलीय माध्यम में ट्राइपोडल-टाइप शिफ बेस के साथ यूरेनिल आयन के इंटरएक्टिव व्यवहार ने विचित्र व्यवहार का खुलासा किया। UO_2^{2+} आयन और दाता प्रजातियों के अंतःक्रियात्मक व्यवहार पर संरचनात्मक जांच से प्रजातियों का अलगाव होता है, जो प्रतिक्रिया माध्यम में हाइड्रोलाइटिक गतिविधि के परिणामस्वरूप बनते हैं। हालांकि, शिफ ठिकानों द्वारा यूरेनिल कैपचरिंग उत्तरोत्तर

ध्यान देने योग्य थी, फिर भी समाधान में एक वास्तविक प्रतिक्रिया परिदृश्य को भी एक एक्सट्रैक्ट के रूप में दावा करने से पहले संबोधित करने की आवश्यकता होती है।

अध्याय VI 2-एमिनोथियोफेनॉल और 4-एल्काइल-2,6-डिफॉर्मिलफेनोल और उनके $U^{VI}O_2^{2+}$ परिसरों से संश्लेषण 2,6-बीआईएस (बेंजोथियाज़ोल-2-वाईएल) -4- (अल्काइल) फिनोल डेरिवेटिव (L_1 और L_2) का वर्णन करता है। दाता प्रजातियों और उनके परिसरों का लक्षण वर्णन एकल क्रिस्टल एक्स-रे सहित भौतिक रासायनिक और स्पेक्ट्रोस्कोपिक विधियों द्वारा किया गया है। यूरेनिल परिसरों का एक तुलनात्मक समाधान और ठोस-अवस्था का अध्ययन किया गया और निष्कर्ष यहां प्रस्तुत किए गए हैं।

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Abbreviations used

THF	Tetrahydrofuran
TFA	Trifluoroacetic acid
HBr	Hydrobromic acid
DMF	Dimethylformamide
IR	Infrared
DIPEA	N,N-diisopropylethylamine
Hz	Hertz
MHz	Mega Hertz
h	Hour
g	Gram
M	Molar
mp	Melting point
equiv.	Equivalent
mmol	Millimole
Anal.calcd.	Analytically calculated
NMR	Nuclear magnetic resonance
mL	Milliliter
s	singlet
d	doublet
dd	double doublet
t	triplet
m	multiplet
br	broad
ppm	parts per million