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Studies on Dioxomolybdenum (VI) Complexes with Synthetic Tetraza Macrocyclic Ligands having Nitrogen and Oxygen Donor Atoms

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Abstract

The coordination compounds has traveled a long way from its starting point when ligands were synthesized first followed by its isolation, purification and characterization. In the second step characterized ligands were allowed to coordinate with the metal cations. The main step in deciding where coordination has take place or not was comparison of physical properties of ligand with the coordinated complex. In most of the cases, yield was very poor and the time required was very high.

Keywords: Dioxomolybdenum, *Synthetic Tetraza*, Macrocyclic Ligands, acetylacetonate.

Introduction

The coordination compounds has traveled a long way from its starting point when ligands were synthesized first followed by its isolation, purification and characterization. In the second step characterized ligands were allowed to coordinate with the metal cations. The main step in deciding where coordination has take place or not was comparison of physical properties of ligand with the coordinated complex. In most of the cases, yield was very poor and the time required was very high.

The 'in situ' method in which components of the ligand are howed to condense in presence of metal ion.

Such a organizational role of metal ions is guiding factor in between compound of legend is teemed as template effect.

The yield is very high and time taken is very short. In present investigation *in situ* meted or self assembly process has been utillzed for the synthesis of the complexes. The important factors in the thesis processes are.

1. Stoichiometry of reaction.
2. Order of mixing of components.
3. Selection of components.

The dioxomoly bdeum (VI) complexes were synthesized using an in-situ method by refluxing the reaction mixture of benzyl/ diacetyl, diamines and molybdenyl acetylacetonate in 1:2:1 molar ratio in aqueous ethanol.

Physical and analytical date of the molybdenum complexes.

The physical and analytical data of the complexes are give in table. The elemental analyses of the complexes agreed with the theoretical values and showed 1:1 metal to ligand stoichiometry. The molar conductivity of dioxmolybdenum (VI) complexes in dimethylformamide showed vales of ΔM between 125-140 $\text{ohm}^{-1} \text{cm}^2 \text{mol}^{-1}$, which indicate their electrolytic nature.

The above details support the tentative structures of macro cyclic complexes of type $[\text{MoO}_2(\text{mac})^{1-8}](\text{acac})_2$, as shown in following figures.

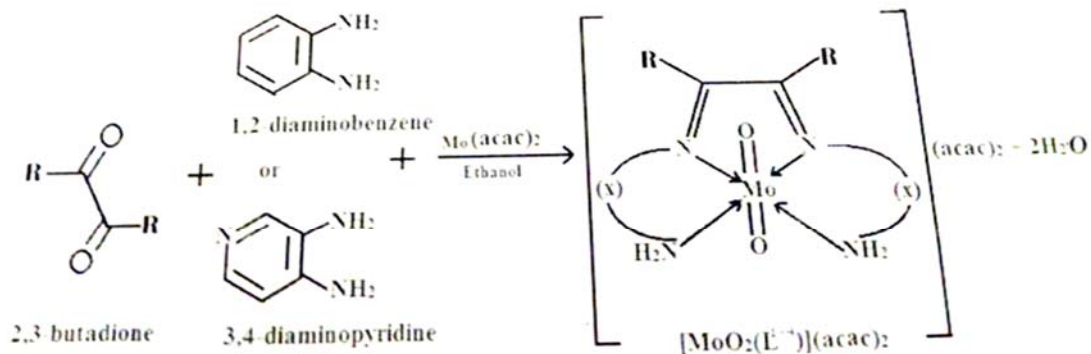
The work deals with synthesis and characterization of a series of dioxomolybdenum (VI) complexes of the type $[\text{MoO}_2(\text{mac})^{1-8}](\text{acac})_2$.

The 'in situ' method of preparation has been utilized in which methanolic solution of molybdenyl acetylacetonate has been refluxed with ethanolic solution of diketones namely benzyl of diacetyl and diketones namely 1, 2-diaminobenzen or 3, 4-diaminopyridine.

Metal ion catalyzed Schiff base condensation leads to the complexes $[\text{MoO}_2(\text{L1-4})](\text{acac})_2$ according following scheme:-

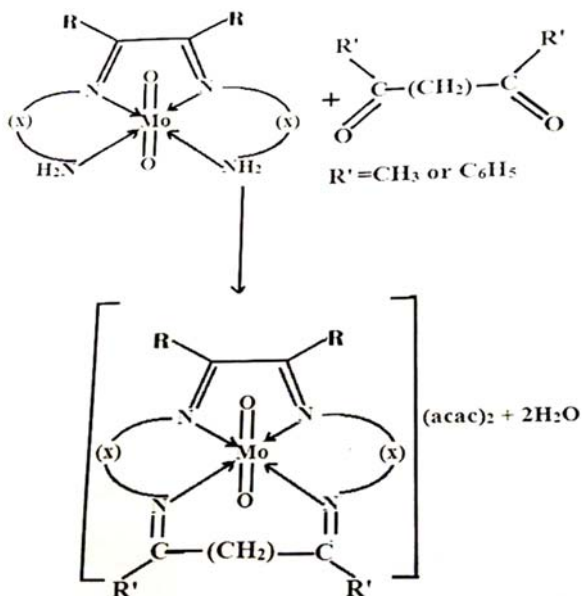
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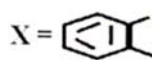


X=1, 2-diaminobenzene or 3, 4-diaminopyridine.

The ethanolic solution of yellow complexes isolated in the 1st step were refluxed with diketones namely acetyl acetone or benzoylacetone to give macro cyclic complexes of the type $[\text{MoO}_2(\text{mac})^{1-8}](\text{acac})_2$ as per following scheme:-



Mac¹ R=CH₃, R'= CH₃



Mac² R=C₆H₅, R'= CH₃



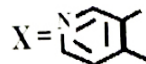
Mac³ R = CH₃, R' = C₆H₅



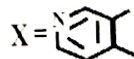
Mac⁴ R = C₆H₅, R' = C₆H₅



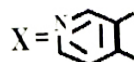
Mac⁵ R = CH₃, R' = CH₃



Mac⁶ R = C₆H₅, R' = CH₃



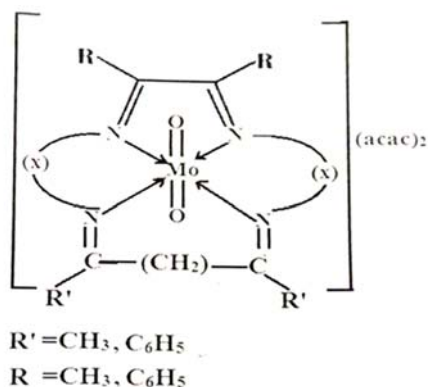
Mac⁷ R = CH₃, R' = C₆H₅



Mac⁸ R = C₆H₅, R' = C₆H₅

The complexes has been characterized by molar conductance, elemental analysis, infrared and electronic spectral data.

In on the complexes molybdenum (VI) has been found to be in an octahedral stereochemistry with four nitrogen donor atoms of the macro cyclic lagan surrounded by Mo(VI) in a square planar manner and two oxygen donor atoms in the axial position as shown in the following figures.



Complexes	Mo% Found (Ca.)	N% Found (Ca.)	C% Found (Ca.)	H% Found (Ca.)
$[\text{MoO}_2\text{mac}^1](\text{acac})_2$	14.32 (14.63)	8.49 (8.53)	56.11 (56.70)	5.47 (5.48)
$[\text{MoO}_2\text{mac}^2](\text{acac})_2$	12.25 (12.13)	7.06 (7.17)	62.91 (63.07)	5.08 (5.13)
$[\text{MoO}_2\text{mac}^3](\text{acac})_2$	14.41 (14.54)	8.32 (8.48)	56.08 (56.36)	5.99 (5.06)
$[\text{MoO}_2\text{mac}^4](\text{acac})_2$	10.50 (10.61)	6.03 (6.19)	67.12 (67.69)	4.84 (4.86)
$[\text{MoO}_2\text{mac}^5](\text{acac})_2$	10.51 (10.61)	10.53 (10.65)	54.45 (54.79)	5.30 (5.32)
$[\text{MoO}_2\text{mac}^6](\text{acac})_2$	12.12 (12.29)	8.88 (8.96)	61.12 (61.46)	4.83 (4.99)
$[\text{MoO}_2\text{mac}^7](\text{acac})_2$	14.46 (14.52)	10.38 (10.59)	53.98 (54.46)	5.83 (5.90)
$[\text{MoO}_2\text{mac}^8](\text{acac})_2$	10.50 (10.60)	7.68 (7.73)	66.15 (66.30)	4.70 (4.75)

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