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Synthesis and Characterization of Oxovanadium (IV) Complexes with Tetradentate Schiff-base Ligands Having Thenil as Precursor Molecule

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ABSTRACT

The oxovanadium (IV) complexes of the type [VO(mac)] SO_4 are synthesized with tetraaza ligands derived from condensation of thenil with 1,2-diaminopropane or 1,4-diaminobutane in 1:2 molar ratio and their reaction with β-diketones such as acetylacetone, benzoylacetone, thenoyltrifluoroacetone and dibenzoylmethane are carried out to get macrocyclic products where vanadyl ion appears to act as kinetic template. Tentative structures of the complexes have been proposed on the basis of elemental analysis, infrared, e.s.r. and electronic data. All the oxovanadium (IV) complexes are five coordinate.

Key words: Macrocyclic ligands, β-diketones, diamines, oxometal ion

INTRODUCTION

The study contains several reports about oxovanadium(IV) complexes which show modulating activities of various enzymes (Rehder, 2003; Sumathi *et al.*, 2010; Yue *et al.*, 2006). The chemistry of vanadium has attracted attention due to its presence in biological system particularly its accumulation in sea squirts and in wild mushrooms like *Amanita muscaria* and others (Adetutu, 2010; Samanta *et al.*, 2003; Guohe *et al.*, 2006; Yuan *et al.*, 2009). During the past few years, a great deal of research effort has been directed to study the transition metal complexes of high denticity ligands with a view to obtain the metal complexes of unusual configuration and coordination number (Agarwal *et al.*, 2005; Sreeja and Kurup, 2005; Sutradhar *et al.*, 2006). Thenil is a versatile chelating agent having two reactive carbonyl groups capable of undergoing Schiff-base condensation with a variety of di and polyamines. This provides an opportunity to design and study the model biological systems to understand the chemical changes taking place in such cases (Maurya, 2003). However, the chemistry of transition metal complexes with macrocyclic ligands capable of imparting useful properties have received less attention (Koo *et al.*, 2003; Maurya, 2006). It is observed that intestinal absorption of vanadyl ion increases significantly when complexed with certain ligands which will allow us to explore its therapeutic applications in due course of time in further studies. With this view, some oxovanadium (IV) complexes with new series of macrocyclic ligands are obtained in-situ by the condensation of thenil with 1,2-diaminopropane or 1,4-diaminobutane in presence of vanadyl ion having cyclisation reactions with different β-diketones viz. acetylacetone, benzoylacetone, thenoyltrifluroacetone and dibenzoylmethane.

These complexes have been assigned on the basis of their elemental analysis, electrical conductance, magnetic susceptibility measurements and spectral data.

MATERIALS AND METHODS

All the chemicals and the solvents used were of the reagent grade. Oxovanadium (IV) sulfate and thenil used were Aldrich product. The β-diketones viz. acetylacetone, benzoylacetone, thenoyltrifluoroacetone and dibenzoylmethane were Sisco Research Laboratories products and the diamines used were reagent grade products. The research work was carried out between 2005/2006 in collaboration with Prof. H.S. Yadav, Department of Chemistry, NERIST, Nirjuli, Arunachal Pradesh, India.

Analytical methods and physical measurements: Vanadium was estimated gravimetrically after decomposing the complexes with concentrated nitric acid by standard method (Ferraro, 1971). Microanalysis of carbon, hydrogen and nitrogen for the complexes were done at central research facility, NERIST, nirjuli, Itanagar, Arunachal Pradesh. Sulfur was estimated as barium sulfate in the laboratory (Nakamoto, 1998). The standard technique of melting point (uncorrected) determination using sulfuric acid bath was employed. A Toshniwal conductivity bridge, Model No. CLO102 A was used for conductance measurements at room temperature. The magnetic susceptibility of the oxovanadium (IV) complexes in powder form were carried out at room temperature using Gouy's balance. The electronic spectra of the complexes were recorded on Beckmann DU-2 spectrophotometer and cΦ10 Russian spectrophotometer instrument in the ranges 2000-185 and 700-400 nm. The room temperature and liquid nitrogen temperature e.s.r. spectra were recorded at RSIC, IIT, Chennai, India. The infrared spectra of the complexes in the range 4000-200 cm^{-1} were recorded in KBr medium on Perkin-Elmer 621 and Beckmann Acculab-9 spectrophotometers.

***In-situ* preparation of oxovanadium (IV) complexes with ligands derived by condensation of thenil with 1,2-diaminopropane or 1,4-diaminobutane:** Vanadyl sulfate (2 mmol) dissolved in methanol (25 mL) was added to a refluxing solution mixture of thenil (2 mmol) and 1,2-diaminopropane (4 mmol) or 1,4-diaminobutane (4 mmol) in ethanol (25 mL). The mixture was refluxed for 6 h when the color of the solution turned green. The solvent was removed under vacuo at room temperature and the dark green color product was isolated. The complex was thoroughly washed with ethanol/methanol mixture.

***In-situ* preparation of macrocyclic complexes of oxovanadium (IV):** Vanadyl sulfate (2 mmol) dissolved in methanol (25 mL) was added to a refluxing solution mixture of thenil (2 mmol) and 1,2-diaminopropane or 1,4-diaminobutane (4 mmol) in ethanol (25 mL). The mixture was refluxed for 5 h when the color of the solution intensified and turned green. To this reaction mixture, an ethanolic solution (10 mL) of acetylacetone (2 mmol) and glacial acetic acid (1 mL) were added. The reaction mixture was refluxed for about 5 h then green precipitate was obtained. The complex was purified by washing with the equimolar mixture of ethanol/methanol (10 mL), Yield 57%.

The same procedure was adopted for the synthesis of other oxovanadium (IV) macrocyclic complexes using benzoylacetone, thenoyltrifluroacetone and dibenzolylmethane as cyclising agent. The physical and analytical data of the complexes are presented in Table 1.

Table 1: Physical and analytical data of the complexes

Complex	Empirical formula	Decomp. temp. (°C)	C% calcd. (found)	H% calcd. (found)	N% calcd. (found)	V% calcd. (found)	S% calcd. (found)	$\mu_{eff.}$ BM (300°C)
[VO(L^1)] SO_4	$C_{16}H_{22}N_4VS_3O_5$	210	38.6 (38.5)	4.4 (4.3)	11.3 (11.2)	10.3 (10.2)	19.3 (19.2)	1.74
[VO(L^2)] SO_4	$C_{18}H_{26}N_4VS_3O_5$	216	41.1 (41.0)	5.0 (4.9)	10.7 (10.6)	9.7 (9.6)	18.3 (18.2)	1.73
[VO(mac^1)] SO_4	$C_{21}H_{26}N_4VS_3O_5$	218	45.0 (44.9)	4.6 (4.5)	10.0 (10.0)	9.1 (9.1)	17.0 (16.9)	1.75
[VO(mac^2)] SO_4	$C_{26}H_{28}N_4VS_3O_5$	214	50.1 (49.9)	4.5 (4.3)	9.0 (8.9)	8.2 (8.1)	15.4 (15.3)	1.74
[VO(mac^3)] SO_4	$C_{24}H_{23}N_4VS_4O_5F_3$	216	42.2 (42.1)	3.4 (3.4)	8.2 (8.1)	7.5 (7.5)	18.7 (18.6)	1.76
[VO(mac^4)] SO_4	$C_{31}H_{30}N_4VS_3O_5$	221	54.3 (54.2)	4.4 (4.2)	8.2 (8.2)	7.4 (7.3)	14.0 (14.0)	1.75
[VO(mac^5)] SO_4	$C_{23}H_{30}N_4VS_3O_5$	217	46.9 (46.8)	5.1 (5.1)	9.5 (9.4)	8.6 (8.6)	16.3 (16.2)	1.76
[VO(mac^6)] SO_4	$C_{28}H_{32}N_4VS_3O_5$	214	51.6 (51.4)	4.9 (4.8)	8.6 (8.6)	7.8 (7.7)	14.7 (14.6)	1.71
[VO(mac^7)] SO_4	$C_{26}H_{27}N_4VS_4O_5F_3$	215	43.9 (43.8)	3.8 (3.6)	7.9 (7.8)	7.2 (7.1)	18.0 (17.9)	1.72
[VO(mac^8)] SO_4	$C_{33}H_{34}N_4VS_3O_5$	220	55.5 (55.4)	4.8 (4.7)	7.9 (7.8)	7.1 (7.0)	13.5 (13.4)	1.75

Calcd: Calculated, L^1: Ligand derived by condensation of thenil with 1,2-diaminopropane (1:2), L :^{2}Ligand derived by condensation of thenil with 1,4-diaminobutane (1:2), mac^1: Macrocyclic ligand derived by condensation of L^1 with acetylacetone, mac^2: Macrocyclic ligand derived by condensation of L^1 with benzoylacetone, mac^3: Macrocyclic ligand derived by condensation of L^1 with thenoyltrifluoroacetone, mac^4: Macrocyclic ligand derived by condensation of L^1 with dibenzoylmethane, mac^5: Macrocyclic ligand derived by condensation of L^2 with acetylacetone, mac^6: Macrocyclic ligand derived by condensation of L^2 with benzoylacetone, mac :^{7} Macrocyclic ligand derived by condensation of L^2 with thenoyltrifluoroacetone, mac^8: Macrocyclic ligand derived by condensation of L^2 with dibenzoylmethane

RESULTS AND DISCUSSION

The oxovanadium (IV) complexes were synthesized using in-situ method by refluxing the reaction mixture of thenil, diamines and vanadylsulfate in 1:2:1 molar ratio in aqueous ethanol. The reaction appears to proceed according to the following equation:

$$\begin{gathered}\textbf{Thenil + 1, 2-diaminopropane + VOSO}_4\textbf{. 3H}_2\textbf{O}\\ \textbf{or}\\ \textbf{1, 4-diaminobutane}\\ \Big\downarrow \textbf{Ethanol}_{(aq)}\\ \textbf{[VO(L)] SO}_4 + \textbf{5H}_2\textbf{O}\end{gathered} \tag{1}$$

Where:
L^1 = Thenil + 1,2-diaminopropane
L^2 = Thenil + 1,4-diaminobutane

The parent complexes [VO (L)] SO_4 react with β-diketones to yield [VO (mac)] SO_4 as given below:

$$[VO(L)]\ SO_4 + \text{ß-Diketones} \longrightarrow [VO(mac)]\ SO_4 + 2H_2O \tag{2}$$

where, mac = tetraazamacrocyclic ligands derived from condensation of L^1 or L^2 with β-diketones in presence of oxovanadium(IV) cation.

The elemental analysis (Table 1) of complexes show 1:1 metal to ligand stoichiometry. The molar conductivity of oxovanadium (IV) complexes in dimethylformamide showed values of Λ_M between 118-135 ohm^{-1} cm^2 mol^{-1} which indicate their electrolytic nature.

Table 2: Infrared spectral bands of complexes

	Bands (cm^{-1})							
Complex	(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)
$[VO(L^1)]\ SO_4$	1616	304	980	1134	955	600	3350	3180
$[VO(L^2)]\ SO_4$	1622	301	981	1132	958	602	3352	3182
$[VO\ (mac^1)]\ SO_4$	1620	303	982	1132	956	608		
$[VO\ (mac^2)]\ SO_4$	1624	301	981	1135	960	604		
$[VO\ (mac^3)]\ SO_4$	1624	304	982	1130	956	608		
$[VO\ (mac^4)]\ SO_4$	1618	302	980	1132	968	602		
$[VO\ (mac^5)]\ SO_4$	1620	304	982	1132	960	602		
$[VO\ (mac^6)]\ SO_4$	1620	303	981	1134	958	604		
$[VO\ (mac^7)]\ SO_4$	1624	302	980	1133	958	606		
$[VO\ (mac^8)]\ SO_4$	1622	300	982	1135	960	602		

L^1: Ligand derived by condensation of thenil with 1,2-diaminopropane (1:2) ,L^2: Ligand derived by condensation of thenil with 1,4-diaminobutane (1:2), mac^1: Macrocyclic ligand derived by condensation of L^1 with acetylacetone, mac^2: Macrocyclic ligand derived by condensation of L^1 with benzoylacetone, mac^3: Macrocyclic ligand derived by condensation of L^1 with thenoyltrifluoroacetone, mac^4: Macrocyclic ligand derived by condensation of L^1 with dibenzoylmethane, mac^5: Macrocyclic ligand derived by condensation of L^2 with acetylacetone, mac^6: Macrocyclic ligand derived by condensation of L^2 with benzoylacetone, mac^7: Macrocyclic ligand derived by condensation of L^2 with thenoyltrifluoroacetone, mac^8: Macrocyclic ligand derived by condensation of L^2 with dibenzoylmethane where, 1: ν (>C = N) 2:ν (V-N) 3:ν (V = 0) 4: ν_3 of SO_4-5: ν_1 of SO^{--}_4 6: ν_4 of $SO_4^{..}$ 7: ν_{as} (N-H) 8: ν_s (N-H)

Infrared spectra: The important bands of the infrared spectra for the complexes are listed in Table 2. The parent complexes (Type I) of oxovanadium (IV) exhibit >C = N absorption around 1624-1616 cm^{-1} which normally appears at 1665 cm^{-1} in free ligands (Meisch *et al.*, 1979; Weeks and Leicester, 1968). The lowering of this band in the complexes (Type I) is due to the coordination of nitrogen atoms of the azomethine groups to the vanadium (Ando *et al.*, 2004; Shashidhara and Goudar, 2000). The presence of a band at around 300 cm^{-1} may be assigned to **ν** (V-N) vibration (Maurya *et al.*, 2006a). The appearance of >C = N band and the absence of the >C=O band around 1700 cm^{-1} is a conclusive evidence for condensation of the diamines with the two keto group of precursor molecule thenil (Maurya, 2006). The bands appearing at 3350 and 3180 cm^{-1} may be assigned to asymmetrical and symmetrical N-H stretching modes of the coordinated terminal amino group (Nakamoto, 1998). The oxovanadium (IV) complexes show a band at around 980 cm^{-1} which is assigned to **ν** (V = O) vibration (Sakata *et al.*, 1989). The presence of an ionic sulfate group in the complexes is confirmed by the appearance of three bands at ca. 1130-1135 cm^{-1} (ν_3), 955-960 cm^{-1} (ν_1) and 600-610 cm^{-1} (ν_4). The absence of a ν_2 band and non-splitting band of ν_3 band indicate that Td symmetry is retained (Nonoyama *et al.*, 1975). The infrared spectra of macrocyclic complexes of type-II show the same pattern of bands but the asymmetrical and symmetrical N-H stretching modes of terminal amino groups disappear due to condensation of these amino groups with carbonyl group of β-diketones in cyclization reactions.

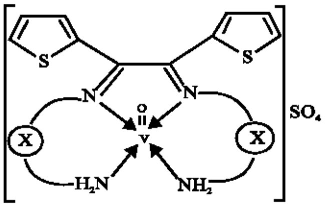
Magnetic moments and electronic spectra: Oxovanadium (IV) complexes show magnetic moment values of the order of 1.76 B.M. at room temperature which are in the range reported for oxovanadium (IV) complexes with one unpaired electron (Rao *et al.*, 1997). The electronic spectra show bands in the regions 11,040-11,980 and 21,080-22,380 cm^{-1}. These spectra are similar to other five coordinate oxovanadium (IV) complexes involving nitrogen donor atoms. These spectral bands are interpreted according to an energy level scheme reported by (Sutradhar *et al.*, 2007) for distorted, five coordinate square pyramidal oxovanadium (IV) complexes (Maurya *et al.*, 2006b).

Accordingly, the observed bands can be assigned to $^2B_2 \rightarrow {}^2E_2$, $^2B_2 \rightarrow {}^2B_1$ and $^2B_2 \rightarrow {}^2A_2$ transitions, respectively. One more band is observed in the region 35,260-35,760 cm^{-1} which may be due to transition of the azomethine linkages (Mitchell and Scott, 1990).

ESR spectra: The X-band ESR spectra of an oxovanadium (IV) complex was recorded in DMSO at room temperature and at liquid nitrogen temperature (177 K). ESR spectra of the complexes were analyzed by the method of (Narang *et al.*, 1994; Sands, 1955; Yadav, 1993). The room temperature ESR spectra show eight lines which are due to hyperfine splitting arising from the interaction of the unpaired electron with a ^{51}V nucleus having the nuclear spin I = 7/2. This confirms the presence of a single oxovanadium (IV) cation as the metallic centre in the complex. The anisotropy is not observed due to rapid tumbling of molecules in solution at room temperature and only g-average values are obtained. The anisotropy is clearly visible in the spectra at liquid nitrogen temperature and eight bands each due to $g_{||}$ and $g_{\perp}$ are observed separately. The $g_{||}$, $g_{\perp}$, $A_{||}$ and $A_{\perp}$- values (RT/LNT) are measured from the spectra which are in good agreement for a square-pyramidal structure (Boucher and Yen, 1969; Costes and Fernandez-Garcia, 1988; Dodwad *et al.*, 1989). The g_{iso} value from mobile solution at room temperature and g_{av} from frozen solution at liquid nitrogen temperature do not agree very closely since the g and A tensors are corrected for second-order. Further, g values are all very close to the spin-only value (free electron value) of 2.0023, suggesting little spin-orbit coupling.

On the basis of the above studies, the following tentative structures may be proposed for these oxovanadium (IV) complexes of the type (I) and (II).

Tentative structures may be proposed for oxovanadium (IV) complexes of the type (I):



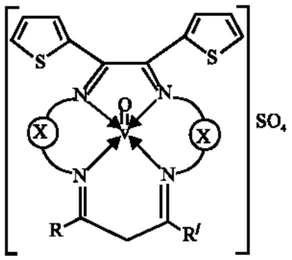
Where,

$X = -CH_2-\overset{|}{C}H-CH_3$

or

$-(CH_2)_4-$

The tentative structures for macrocyclic complexes of the type (II) are shown below:



Where:
R = CH_3, C_6H_5, C_4H_3S, C_6H_5
R$'$ = CH_3, CH_3, CF_3, C_6H_5
β-Diketone = Acetylacetone, Benzoylacetone, Thenoyltrifluroacetone, Dibenzoylmethane

CONCLUSION

The infra-red spectral data show that the thenil is a good precursor molecule having two reactive carbonyl groups capable of undergoing Schiff base condensation with a variety of diamines leading to formation of tetraaza ligands. Schiff bases behave as tetradentate ligands by bonding to the metal ion through the azomethine nitrogen and amine nitrogen as donor atoms. The analytical data show the presence of one metal ion per molecule and suggest a mononuclear structure for these vanadyl complexes. The magnetic moment values and electronic data are in the favour of square pyramidal structure for VO (IV) complexes.

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