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Research Article

Synthesis and Characterization of Some Novel Schiff Base Complexes of Oxovanadium(IV) Cation

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A series of oxovanadium(IV) complexes of the type [VO(mac)]SO₄ (where mac = tetraazamacrocyclic ligands derived from condensation of 4,4,4-trifluro-1-(2-furyl)-1,3-butanedione or 4,4,4-trifluro-1-(2-thenyl)-1,3-butanedione with p-phenylenediamine and their reaction with β -diketones) have been prepared using oxometal ion of vanadium as kinetic template. These complexes have been ascertained by electrical conductance, magnetic moment, elemental analyses, infrared, e.s.r. and electronic spectral data. All the oxovanadium(IV) complexes are five-coordinate ones.

1. Introduction

Vanadium is found naturally in soil and water as a trace metal. Vanadium compounds with oxidation state IV and V exist in the environment and in biological systems. The literature contains several reports about oxovanadium(IV) complexes which show modulating activities of various enzymes [1]. 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 [2-4]. The oral administration of vanadate has been proved to reduce hyperglycemia in diabetic rats. However, in most cases, the template effect of metal ions of the first transition series has been studied but chemistry of vanadium complexes with macrocyclic ligands incorporating four nitrogen donor atoms has received less attention [5-7]. The pphenylenediamine can form chelate ring using insitu method of synthesis where diketone, p-phenylenediamine were condensed in presence of metal cation due to kinetic template effect [8-10]. Considering these facts a new series of oxovanadium(IV) complexes have been prepared by condensation of 4,4,4-trifluro-1-(2-furyl)-1,3-butanedione or 4,4,4-trifluro-1-(2-thenyl)-1,3-butanedione with p-phenylenediamine in 1:2 molar ratio in the presence of VO^{2+} cation. The reactions of oxovanadium(IV) complexes with β -diketones, namely acetylacetone, benzoylacetone, thenoyltrifluroacetone, and dibenzoylmethane have been carried out which result macrocyclic complexes which are characterized and their tentative structures have been ascertained.

2. Experimental

2.1. Materials and Methods. Oxovanadium(IV) sulfate was procured from Aldrich. The β -diketones namely acetylacetone, benzoylacetone, thenoyltrifluoroacetone and dibenzoylmethane were SRL products and the diamines used were reagent grade products. 4,4,4-Trifluro-1-(2-furyl)-1,3-butanedione or 4,4,4-trifluro-1-(2-thenyl)-1,3-butanedione and p-phenylenediamine used were Aldrich product.

2.2. Analytical Methods and Physical Measurements. Vanadium was estimated gravimetrically as its vanadate after

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decomposing the complex with concentrated nitric acid [11]. Sulfur was estimated as barium sulfate using standard procedure [12]. The standard technique of melting point (uncorrected) determination using sulfuric acid bath was employed. Toshniwal conductivity bridge, Model no. CLO102A was used for conductance measurements at room temperature. The magnetic susceptibility of the complexes in powder form was carried out at room temperature using Gouy's balance. Mercury tetrathiocyanatocobaltate(II), Hg $[Co(CNS)_4]$, $(\chi_a = 16.44 \times 10^{-6} \text{ c.g.s. unit at } 20^{\circ}\text{C})$, was used as calibrant. The electronic spectra of the complexes were recorded on c Φ10 Russian spectrophotometer instrument in the ranges 700–400 nm. The room temperature and liquid nitrogen temperature ESR. 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 on Perkin-Elmer 621 spectrophotometers.

- 2.3. InSitu Preparation of Oxovanadium(IV) Complexes with Ligands Derived by Condensation of 4,4,4-Trifluro-1-(2-furyl)-1,3-butanedione or 4,4,4-Trifluro-1-(2-thenyl)-1,3-butanedione with p-Phenylenediamine. Vanadyl sulfate (2 mmol) dissolved in methanol (25 mL) was added to a solution of 4,4,4-trifluro-1-(2-furyl)-1,3-butanedione or 4,4,4-trifluro-1-(2-thenyl) and p-phenylenediamine (4 mmol) in ethanol (25 mL). The mixture was refluxed for 6 hours, till 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 complexes were thoroughly washed with methanol/ethanol mixture. Yield was 65%.
- 2.4. InSitu Preparation of Macrocyclic Complexes of Oxovanadium(IV). Vanadyl sulfate (2 mmol) dissolved in methanol (25 mL) was added to a solution of 4,4,4trifluro-1-(2-furyl)-1,3-butanedione or 4,4,4-trifluro-1-(2thenyl)-1,3-butanedione(2 mmol) and p-phenylenediamine (4 mmol) in ethanol (25 mL). The mixture was refluxed for 5 hours, till 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 (5 mL) was added. The reaction mixture was refluxed for about 5 hours then green precipitate was obtained. The complex was purified by washing with the mixture (10 mL) of methanol/ethanol (1:1). Yield was 60%. The same procedure was adopted for the synthesis of other oxovanadium(IV) macrocyclic complexes using benzoylacetone, thenoyltrifluroacetone, and dibenzolylmethane. The physical and analytical data of the complexes are presented in Table 1.

3. Results and Discussion

The oxovanadium(IV) complexes were synthesized using *insitu* method by refluxing the reaction mixture of 4,4,4-trifluro-1-(2-furyl)-1,3-butanedione or 4,4,4-trifluro-1-(2-thenyl)-1,3-butanedione and p-phenylenediamine and vanadylsulfate in 1:2:1 molar ratio in aqueous ethanol. The

macrocyclic complexes were obtained by reacting the parent complexes with respective β -diketones and the reactions appear to proceed according to Scheme 1, where The values of R and R' represent the β -diketones and L¹ = 4,4,4-trifluro-1-(2-furyl)-1,3-butanedione + p-phenylenediamine; L² = 4,4,4-trifluro-1-(2-thenyl)-1,3-butanedione + p-phenylenediamine; mac = tetraazamacrocyclic ligands derived from condensation of L¹ and L² with β -diketones in presence of oxovanadium(IV) cation, respectively.

- 3.1. Infrared Spectra. The important bands of the infrared spectra for the complexes are listed in Table 2. The macrocyclic complexes of oxovanadium(IV) exhibit >C=N absorption around 1624 -1616 cm⁻¹, which normally appears at 1660 cm⁻¹ in free ligands [13-15]. The lowering of this band in the complexes (typeI) indicates the coordination of nitrogen atoms of the azomethine groups to the VO²⁺ [15-17]. The presence of a band at around 300 cm⁻¹ may be assigned to v(V-N) vibration [18]. The appearance of >C=N band and the absence of the >C=O band around 1710 cm⁻¹ are a conclusive evidence for condensation of the p-phenylenediamine with the two keto group of 4,4,4-trifluro-1-(2-furyl)-1,3-butanedioneor 4,4,4-trifluro-1-(2-thenyl)-1,3-butanedione furil [17]. 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 (Nonoyama et al.) [19]. The oxovanadium(IV) complexes show a band at around 980 cm⁻¹, which is assigned to v(V=O) vibration [20]. The presence of an ionic sulfate group in the complexes is confirmed by the appearance of three bands at ca. $1130-1135 \,\mathrm{cm}^{-1}$ (v_3), $955-960 \text{ cm}^{-1}$ (v_1), and $602-608 \text{ cm}^{-1}$ (v_4). The absence of a v_2 band and nonsplitting band of v_3 band confirms that tetrahedral symmetry is retained [21]. Infrared spectra of macrocyclic complexes of typeII 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.
- 3.2. Electronic Spectra. The electronic spectra show bands in the regions 11,045–11,985 cm⁻¹, 15,030–15,900 cm⁻¹, and 21,090–22,390 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 Tsuchimoto et al. for distorted five coordinate square pyramidal oxovanadium(IV) complexes [22]. Accordingly, the observed bands can be assigned to ${}^2B_2 \rightarrow {}^2E$, ${}^2B_2 \rightarrow {}^2B_1$, and ${}^2B_2 \rightarrow {}^2A_2$ transitions, respectively. One more band is observed in the region 35,160–35,660 cm⁻¹, which may be due to transition of the azomethine linkages [23].
- 3.2.1. Molar Conductance Measurements. The molar conductivity (Λ_M) values of all the oxovanadium(IV) complexes were measured in dimethylformamide and the obtained

Complex	Empirical formula	Decomp. temp.	C% found (Calcd)	H% found (Calcd)	N% found (Calcd)	V% found (Calcd)	S% found (Calcd)	μ _{eff} BM (300°C)
[VO(L ¹)]SO ₄	$C_{20}H_{17}N_4VSO_6F_3$	212	43.6 (43.7)	3.0 (3.1)	10.1 (10.2)	9.2 (9.3)	5.7 (5.8)	1.73
$[VO(L^2)]SO_4$	$C_{20}H_{17}N_4VS_2O_5F_3$	213	42.4 (42.5)	2.9 (3.0)	9.9 (9.9)	9.0 (9.0)	11.2 (11.3)	1.74
[VO(mac ^{1A})]SO ₄	$\mathrm{C}_{25}\mathrm{H}_{21}\mathrm{N}_{4}\mathrm{VSO}_{6}\mathrm{F}_{3}$	215	48.9 (48.9)	3.4 (3.5)	9.0 (9.1)	8.2 (8.3)	5.1 (5.2)	1.72
[VO(mac ^{1B})]SO ₄	$\mathrm{C_{30}H_{23}N_{4}VSO_{6}F_{3}}$	217	53.2 (53.3)	3.3 (3.4)	8.3 (8.3)	7.5 (7.5)	4.5 (4.6)	1.75
[VO(mac ^{1C})]SO ₄	$C_{28}H_{18}N_4VS_2O_6F_6$	219	45.6 (45.7)	2.4 (2.5)	7.5 (7.6)	6.8 (6.9)	8.6 (8.7)	1.74
[VO(mac ^{1D})]SO ₄	$\mathrm{C_{35}H_{25}N_4VSO_6F_3}$	227	57.0 (57.0)	3.3 (3.4)	7.5 (7.6)	6.9 (6.9)	4.3 (4.4)	1.73
[VO(mac ^{2A})]SO ₄	$C_{25}H_{21}N_4VS_2O_5F_3$	220	47.6 (47.7)	3.3 (3.4)	8.8 (8.9)	8.0 (8.1)	10.1 (10.2)	1.72
[VO(mac ^{2B})]SO ₄	$C_{30}H_{23}N_4VS_2O_5F_3$	218	52.0 (52.1)	3.3 (3.4)	8.1 (8.1)	7.4 (7.4)	9.2 (9.3)	1.76
[VO(mac ^{2C})]SO ₄	$C_{28}H_{18}N_4VS_3O_5F_6$	221	44.7 (44.8)	2.3 (2.4)	7.4 (7.5)	6.7 (6.8)	12.7 (12.8)	1.75
[VO(mac ^{2D})]SO ₄	$C_{35}H_{25}N_4VS_2O_5F_3$	218	55.7 (55.8)	3.2 (3.3)	7.4 (7.4)	6.8 (6.8)	8.4 (8.5)	1.74

 L^1 : ligand derived by condensation of 4,4,4-trifluro-1-(2-furyl)-1,3-butanedione and p-phenylenediamine (1:2); L^2 : ligand derived by condensation of 4,4,4-trifluro-1-(2-thenyl)-1,3-butanedione and p-phenylenediamine (1:2); Mac^{1A} : macrocyclic ligand derived by condensation of L^1 with acetylacetone; Mac^{1B} : macrocyclic ligand derived by condensation of L^1 with thenoyltrifluoroacetone; Mac^{1D} : macrocyclic ligand derived by condensation of L^2 with dibenzoylmethane; Mac^{2A} : macrocyclic ligand derived by condensation of L^2 with acetylacetone; Mac^{2B} : macrocyclic ligand derived by condensation of L^2 with benzoylacetone; Mac^{2C} :macrocyclic ligand derived by condensation of L^2 with thenoyltrifluoroacetone; Mac^{2D} : macrocyclic ligand derived by condensation of L^2 with dibenzoylmethane.

TABLE 2: Infrared spectral bands of complexes.

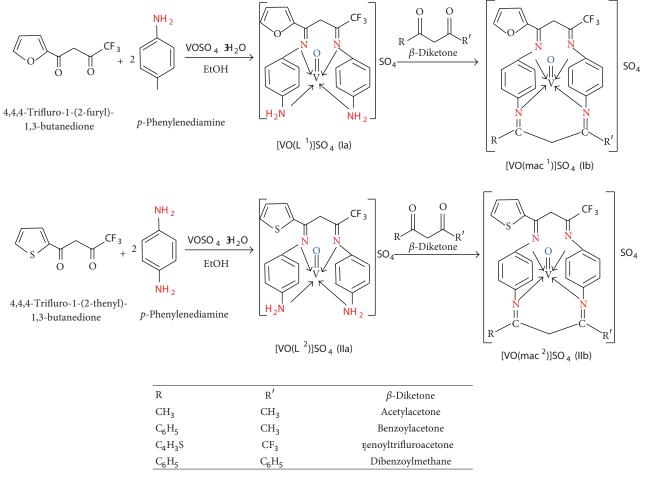
Complex	Bands (cm ⁻¹)									
	ν (>C=N)	ν (V–N)	v(V = O)	v_3 of $SO_4^{}$	v_1 of $SO_4^{}$	v_4 of ${ m SO_4}^{}$	$v_{ m asym}$ (N–H)	v_{sym} (N–H)		
[VO(L ¹)]SO ₄	1616	304	980	1133	955	604	3348	3181		
$[VO(L^2)]SO_4$	1622	301	981	1132	958	603	3350	3180		
[VO(mac ^{1A})]SO ₄	1620	304	980	1134	960	606				
[VO(mac ^{1B})]SO ₄	1624	303	982	1133	958	608				
[VO(mac ^{1C})]SO ₄	1622	302	981	1132	958	606				
[VO(mac ^{1D})]SO ₄	1624	304	982	1133	956	603				
[VO(mac ^{2A})]SO ₄	1622	302	981	1132	956	604				
[VO(mac ^{2B})]SO ₄	1624	303	980	1134	958	606				
[VO(mac ^{2C})]SO ₄	1622	304	980	1133	960	604				
[VO(mac ^{2D})]SO ₄	1620	301	981	1135	958	602				

values lie between 98–105 ohm⁻¹ cm² mol⁻¹ indicating their 1:1 electrolytic nature.

to a single electron of the 3d¹ system of square-pyramidal oxovanadium(IV) [22].

3.2.2. Magnetic Moment Measurements. Magnetic moments of oxovanadium(IV) complexes were measured at room temperature and effective magnetic moment ($\mu_{\rm eff}$) values are given in table1. The magnetic moment values of the vanadyl complexes range from 1.70 to 1.76 B.M which correspond

3.3. ESR Spectra. The X-band ESR spectra of an oxovanadium(IV) complex was recorded in DMSO at room temperature and at nitrogen temperature (177 K). ESR spectra of the complexes were analyzed by the method of Mishra, Sand and Ando et al. [23–25]. The room temperature ESR



SCHEME 1: Insitu preparation of macrocyclic complexes of oxovanadium(IV).

spectra show eight lines, which are due to hyperfine splitting arising from the interaction of the unpaired electron with a ⁵¹V 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 which are in good agreement for a square pyramidal vanadyl complexe [26–28]. The $g_{\parallel}, g_{\perp}, A_{\parallel}$ and A_{\perp} values are measured from the spectra, which are in good agreement for a squarepyramidal structure. The $g_{\rm iso}$ value from mobile solution at room temperature and $g_{\rm av}$ 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).

4. Conclusion

The spectral data show that the 4,4,4-trifluro-1-(2-furyl)-1,3-butanedione and 4,4,4-trifluro-1-(2-thenyl)-1,3-butanedione are good chelating agents having two reactive carbonyl groups capable of undergoing Schiff base condensation with a variety of diamine. Schiff bases behave as tetradentate ligands by bonding to the metal ion through the azomethine nitrogen and amino group. The analytical data show the presence of one metal ion per ligand molecule and suggest a mononuclear structure for the VO²⁺ complexes. The electrical conductance, magnetic moment values, infrared, *ESR*. and electronic data are in the favour of square pyramidal, structure for VO(IV) complexes.

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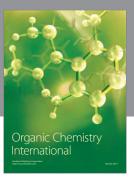
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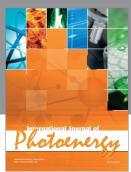
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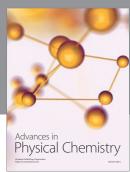
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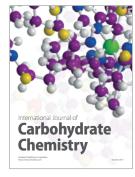
















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