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Synthesis, characterization and structural study of new vanadium complexes with phenolic oxime ligands

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Abstract

In this work, three new vanadium complexes are reported. Their synthesis, crystallization and characterization are detailed. The complex (1) $[VO(C_7H_6NO_2)_2]$ is amononuclear and crystallizes in the monoclinic P2₁/n space group exhibiting a square pyramidal geometry around the vanadium. Dissolved in p-xylene (1-4 dimethylbenzene), this complex gives a binuclear complex (2) $[V_2O_2(C_7H_6NO_2)_2(C_7H_5NO_2)_2].C_8H_{10}$ with a triclinic P-1 space group and an octahedral geometry around the vanadium. The complex (3) $[VO_2(C_9H_9N_2O_3)]$ was obtained using the new iminoacylated oxime ligand (the result of the nucleophilic attack of 4-hydroxysalicylaldoxime with acetonitrile), it crystallizes in the triclinic P-1 space group with a trigonal bipyramidal geometry around the vanadium. All complexes were characterized by single crystal X-ray diffraction, FT-IR, NMR, UV-visible and TGA.

Keywords: vanadium complexes, salicylaldoxime, iminoacylation, spectroscopic characterization, single crystal.

1. Introduction

The salicylaldoxime or phenolic oxime and its derivatives have been the subject of extensive research in coordination chemistry, because of their highly colored complexes with a large structural diversity, formed with the majority of transition metals [1,2]. Oximes are compounds belonging to the Schiff base class. They can be synthesized by a simple reaction of aldehyde or ketones with hydroxylamine [3]. The interaction of various donor atoms in phenolic oxime ligand and its derivatives with transition metals gives interesting structural features. For example, the 3.5-dichloro-2hydroxybenzaldehye oxime allied to platinum by mono and bidentate coordination geometry [4]. Also, phenolic oxime generally forms mononuclear complexes with most of divalent and lanthanide metal ions [5compound $[Nb_2O(C_7H_5NO_2)_2(C_2H_5O)_4],$ the binuclear 9]. In each salicylaldoxime coordinates to niobium through phenolic oxygen and nitrogen of the oxime group. The oxygen atom of the oxime function bridges to the adjacent niobium [2], as found in the trinuclear compound of tin $[(C_4H_9Sn)_3O(OH)(C_7H_6NO_2)(C_7H_5NO_2)]$ [10]. The flexibility in the chelation of the oxime is responsible for the various structural packing with chrome(III) [11]. In some hexanuclear cluster of manganese, the metallic ions are linked by bridge and double coordination of the oxygen atom of oxime [12,13]. Because of the potential physical and chemical properties of polynuclear salicylaldoxime compounds, several studies were dedicated to the synthesis and structural study of those materials. Milios et al. studied the change from antiferromagnetic to ferromagnetic stems from (Mn₃O)₂ cage of a series of analogous structural compounds with phenolic oximes [14]. In addition, a review of literature shows various homometallic and heterometallic complexes of phenolic oximes with interesting structural features and various coordination behaviors[15-18]. As a polynuclear rare earth clusters with the salicylaldoxime ligands, the octanuclear lanthanide complexes of the general formula $[Ln_8(salox)_4(OH)_4(NO3)_{12}(DMF)_{12}]$ (Ln = Nd, Sm, Eu, Gd, Tb, Dy, Ho and Er), were studied revealing potential magnetic proprieties [19].

The coordination chemistry of the Schiff base vanadium has also attracted a great deal of attention because of its various field of application, such as in medicine as an insulin-mimetic compound [20], or antitumor compound [21]. In the biological activity field, it can be employed as an antimicrobial agent, an antioxidant [22] and can also find application in catalyze [23] and even in the extraction of metals [24].

The works of Zerbib et al. in 1994 [25] has drawn our attention for a number of reasons. This work discloses a salicylaldoxime in various coordinations around the vanadium metal. This diversity is caused by the recrystallization conditions and the versatile coordination chemistry of phenolic oxime which leads to the formation of different complexes confirming thereby the instability of $[VO(C_7H_6NO_2)_2]$ in organic solvent. Moreover, the nucleophilic attack of this complex and similarly structured derivatives by nitrile carbon R-C=N (R = CH₃ or C₆H₅) results in the formation of an iminoacylated oxime ligand [26,27].

In this paper, we report the structure study together with the spectroscopic and thermal characterization of a new salicylaldoxime compound and iminoacylated oxime ligand coordinated with vanadium.

1. Experimental

2.1. Materials and measurements

All the reagents: Salicylaldoxime, VCl₃, VOSO₄.5H₂O, p-xylene and solvents were obtained from commercial sources (Sigma Aldrich and Fluka) and used without further purification. The infrared spectra were recorded on a Jasco FTIR 4100 spectrophotometer in the 4000-400 cm⁻¹ range. Ultraviolet absorption spectra were obtained from UV-Jasco V-650 spectrophotometer. UV–Vis spectra were measured from 200 nm to 900 nm at room temperature. Thermogravimetry (TGA) was carried on a Q500 TA instrument under nitrogen at a heating rate of 10 °C min⁻¹. ¹H and ¹³C NMR spectra were obtained on a Bruker 400 MHz spectrometer using DMSO-d₆ and benzene C₆D₆-d₆ as solvent. The single-crystal X-ray data were collected using Xcalibur, Atlas diffractometer and Enarf-Nonius CAD-4 diffractometer.

2.2. Synthesis of 4-hydroxysalicylaldoxime or 2,4dihydroxybenzaldoxime

The 4-hydroxysalicylaldoxime was synthesized according to the literature [3]. A boiling solution of 2,4-hydroxybenzaldehyde (10mmol, 1.38g) in 30mL of methanol, a solution of hydroxylamonium chloride (10mmol, 0.69g) and sodium hydroxide (10mmol, 0.4g) in 30mL of water were mixed. The reaction mixture was refluxed for 3h. The ligand was separated by ice, added with vigorous stirring, then filtered and washed with hot water.

Colorless, mw: 153.14, ¹H NMR (400 MHz, DMSO-d₆, δ ppm): 10.92 (s, N=OH), 10.08 and 9.70 (s, ArOH), 8.20 (s, CH=N), 7.22-6.29 (m, ArH). ¹³C NMR (101 MHz, DMSO-d6, δ ppm): 158.20 (CH=N) 102.92-160.19 (ArC).

2.3. Synthesis of complex (1): [VO(C₇H₆NO₂)₂]

Complex (1) was prepared by mixing VCl₃ (5mmol, 0.785g) dissolved in 20mL of H₂O and salicylaldoxime (10mmol, 1.37g) dissolved in 20mL of ethanol. The reaction mixture was refluxed for 2h at 60°C. The purple prism crystals suitable for X-ray analysis were obtained by slow evaporation after 6 days. Purple crystal, mw : 339.2 , FT-IR (KBr pellet, v cm⁻¹): 3310 (OH), 1601(C=N), 1283 (N-O), 987 (V=O), 592 (V-N), 487 (V-O), ¹H NMR (400 MHz, DMSO-d₆, δ ppm): 11.29 (s, N=OH), 8.33(s, CH=N), 6.87-7.48 (m, ArH). ¹³C NMR (101 MHz, DMSO-d₆, δ ppm): 148.13 (CH=N), 156.46-116.49 (ArC).

2.4. Synthesis of complex (2): [V₂O₂(C₇H₆ NO₂)₂(C₇H₅ NO₂)₂].C₈H₁₀

Complex (2) was obtained by recrystallization of complex (1) in p-xylene at 120 °C for three hours and let for slow evaporation for 4 weeks. Black crystal, mw: 729.47, FT-IR (KBr pellet, v cm-1): 3438 (OH), 1594(C=N), 1266 (N-O), 973 (V=O), 549 (V-N), 473 (V-O). ¹H NMR (400 MHz, C6D6-d6, δ ppm): 10.74 (s, N=OH), 8.91 and 7.88 (s, CH=N), 6.42- 7.75(m, ArH), 1.55 (s, CH₃ xylene), ¹³C NMR (101 MHz, C₆D₆-d₆, δ ppm) : 155.26 and 148.77 (CH=N), 115.75-166.30 (ArC), 20.64 (CH₃ xylene).

2.5. Synthesis of complex 3: $[VO_2(C_9 H_9N_2 O_3)]$

Complex (3) was prepared by the reaction of VOSO₄.5H₂O (2mmol 0.506 g) dissolved in 20mL of H₂O and 4-hydroxysalicylaldoxime (4mmol 0.616g) dissolved in 20mL of ethanol. The reaction mixture formed a green precipitate immediately. This precipitate was dissolved in acetonitrile and water for one hour at 60°C and let for slow evaporation. yield yellow crystal, mw : 276.12, FT-IR (KBr pellet, v cm⁻¹): 3412 (OH), 3157 (N-H), 1604 (C=N), 1229 (N-O), 938 and 924 (O=V=O), 634 (V-N), 457 (V-O), ¹H NMR (400 MHz, DMSO-d₆, δ ppm) : 10.69 (s,NH), 9.59 (s,ArOH), 7.57(s, CH=N), 6.93-7.67(m, ArH), 2.42(s,CH3).¹³C NMR (101 MHz, DMSO-d₆,

δppm) : 173.00 (O-C=NH), 165.50 (Ar-O-V), 159.62 (Ar-OH), 137.44 (CH=N), 115.56-134.84 (Ar), 16.42(CH₃).

2.6. Data refinement

The single-crystal X-ray data of complexes (1) and (2) were obtained by an Oxford Diffraction Xcalibur four-circle diffractometer (Rigaku Oxford Diffraction, 2015) using Mo K α radiation ($\lambda = 0.71073$ Å) [28]. Data collection was done using the CrysAlis program. Empirical absorption correction was performed using spherical harmonics, implemented in the SCALE3ABSPACK scaling algorithm. The data of complex (3) were collected by Enraf-Nonius CAD-4 automatic four circle using Mo K α radiation ($\lambda = 0.71073$ Å) [29,30]. Data collection was performed using the XCAD4 [31]. The empirical absorption was corrected by psi-scan [32]. All structures were solved by direct methods using the SHELXT, and refined by full-matrix least-squares calculations on F² using SHELXL-2014 [33] integrated in WingX program [34]. The crystallographic data collection of the samples and the results of the solution structures are summarized in (**Table.1**). Diamond software was used for molecular graphics [35].

3. Results and Discussion

3.1. Crystal structures of complex (1)

The reaction between salicylaldoxime and vanadium(III) under aerobic condition, in the presence of water, ledto the oxidation of vanadium(III) into oxo-vanadium(IV) [36,37]. Complex (1) crystallizes in monoclinic space group P2₁/n. This structure is similar to the complex reported by Li et al [38], who used a different method of synthesis. The structural unit consists of a discrete neutral monomer complex with selective atom numbering scheme as shown in (**Figure.1**). The oxo-vanadium(IV) is coordinated to two independent mono-deprotonated salicylaldoximato by the phenolic oxygen and the oximato nitrogen atoms, forming a planar base with a V=O bond in an apical position.

The polyhedral environment around the vanadium ion can be described as a slightly distorted square-pyramidal geometry. The value of τ parameter is equal to 0.11, which explains the slightly distorted geometry (τ =0 for ideal square-pyramidal) [39]. The five coordinate bond lengths and their angular values are noted in **Table.2**. The vanadium atom is lifted by 0.575(6) Å from the pyramidal base.

The two phenyl rings (C1–C6) and (C8–C13) have a r-m-s deviation of the fitted atoms of 0.0037(2) Å and 0.0124(2) Å, respectively with a dihedral angle of $6.75(1)^{\circ}$. The V=O bond length is 1.5951(5) Å. Selected bond distances and angles are summarized in **Table.S1**.

The crystal packing view (**Figure.2**) shows a neighboring antiparallel stacking of the structural unit. This face-to-face arrangement is maintained by intermolecular hydrogen interactions between C-H···O and C-H··· π , which contribute to the stability and cohesion of the structure build (**Table.S2**).

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3.2. Crystal structures of complex (2)

The salicylaldoxime and its derivatives form with transition metals, such as iron and manganese, a large family of complexes[40,41]; due to the presence of several donor atoms. The work of Zerbib et al.[25] proves the instability of vanadium and salicylaldoxime complex in conventional solvents; for example, the solubilization of $[VO(C_7H_6NO_2)_2]$ in ethanol gives a trimer $[V_3O_3(C_7H_6NO_2)_2(C_2H_5O)_5]$. In this work, this instability is demonstrated by the solubilization of $[VO(C_7H_6 NO_2)_2]$ in para-xylene, which gives a dimer $[V_2O_2 (C_7H_6NO_2)_2(C_7H_5NO_2)_2].C_8H_{10}$. In parallel, we have the oxidation of vanadium(IV) to vanadium(V). Complex (2) with selective atom numbering scheme in (Figure.3), crystallizes in the triclinic space group P-1. The structural unit consists of four salicylaldoximato coordinated to two oxo-vanadium(IV) and paraxylene (1,4-dimethylbenzene). The oxo-vanadium(V) is coordinated to two deprotonated salicylaldoximato by phenolic oxygen atoms and oximatonitrogen atoms. The deprotonated oxygen atom of the oxime function bridges to the adjacent vanadium and occupies the sixth coordinate site. This double coordination between the two vanadium ions forms the dimer. The atomscoordinated to the vanadium cations form two independent octahedrawith the oxygen atom of vanadyl in a cis arrangement. The distance between the two vanadium ions is 4.0689(3) Å, the coordinate bond lengths and their angular values are summarized in Table.3.

The two phenyl rings (C1–C6) and (C8–C13) have an r-m-s deviation of the fitted atoms of 0.0044(4) Å and 0.0101(4) Å, respectively with a dihedral angle of 62.60(2)°. The r-m-s deviations of the fitted atoms of (C15-C20) and (C22-C27) are 0.0093(4) Å and 0.0017(4) Å, respectively, with a dihedral angle between the two rings of 86.24(2)°. In the crystal packing illustrated in (**Figure.4**), the network structure is maintained through intermolecular C—H···O and C—H··· π hydrogen interactions (**Table.S4**). Moreover, the presence of π – π interactions in the structure between Cg3 [C22-C23/C27] and Cg3^V (Symmetry code 1-x, -y, 2-z), which are 4.068(3) Å apart, contribute to the cohesion and stability of the structure build.

3.3. Crystal structures of complex (3)

Complex (3) was synthesized using a similar procedure to that reported by Grigg et al [26]. The X-ray diffraction study reveals an iminoacylation reaction of 4-hydroxysalicylaldoxime by acetonitrile, leading to formation of a new iminoacylated ligand. The complex is obtained by the nucleophilic attack of oxygen of the oxime function on the unsaturated carbon atom of the nitrile group of the MeCN solvent, resulting in the formation of iminoacylated ligand [42]. In parallel, during the reaction, vanadium undergoes oxidation (V^{IV}O to V^VO₂)due to the presence of water.

Complex (3) crystallizes in triclinic space group P-1. The structural unit consists of a discrete monomer complex with selective atom numbering scheme in (Figure.5). The structural unit consists of tridentate iminoacylated ligand monodepronated, coordinated to dioxo-vanadium(V). The bond lengths of V=O4 and V=O5 are 1.6300(2) Å and 1.6169(3) Å, respectively. These are, according to the literature, typical distances of V=O double bonds with an angle of $108.0715(1)^{\circ}$ [43]; the coordination bond lengths and their angular values are shown in Table.4. The vanadium ion adopts a trigonal bipyramidal environment with five atoms (N_2O_3) . The trigonal base is formed by two oxygen atoms of dioxo-vanadium and one nitrogen atom from oxime. The r-m-s deviation of the fitted atoms(V1, N1, O4, O5), which isequal to 0.0465(7) Å, reveals acoplanar vanadium with a trigonal base. The iminoacyled ligand is planar with anRMS deviation of the fitted atoms equal to 0.0517(2) Å. The crystal structure can be described as a packing of parallel layers of a planar complex along the *a*axis with a linear coordination of the structural units, and that are arranged parallel to each other at a distance of 3.867(2) Å (Figure.6). The cohesion of the structure and stability are ensured by intermolecular N-H···O and C-H···O hydrogen bonds (Table.S6).

3.4. UV–Vis spectral study

The UV–Vis absorption spectra study of complexes 1–3 and ligands, recorded in the 200-900 nm region in DMSO solution at room temperature, are presented in (**Figure.7**). In the ultraviolet region, the spectrum displays a strong band around 260 nm and 310 nm for complexes (1) and (2) as well as for the salicylaldoxime ligand. These bands are attributed to $\pi \rightarrow \pi^*$ and $n \rightarrow \pi^*$ intramolecular transitions of the ligand. For complexes (3) and 4-hydroxysalicylaldoxime, the spectrum presents transitions at 275 nm and 310 nm, respectively due to the intramolecular $\pi \rightarrow \pi^*$ and $n \rightarrow \pi^*$ transitions [6]. In the visible region, one can observe very weak intensity band around 565nm for complex (1) [VO(HL)₂] attributed to the d-d transition of vanadium(IV) [44]. For complex (2) [VO(HL)₂(L)₂].C₈H₁₀ and complex (3) [VO₂(L')], no d-d transition absorption appears in the spectrum. This absence is due to the d⁰ of the electronic configuration of vanadium(V) [45].

3.5. Thermal analysis

Thermogravimetric analysis (TGA) was employed to study the thermal proprieties of the complexes. Itwas carried out at a heating rate of 10 C/min under nitrogen. All the complexes exhibit stabilityat room temperature but decompose differently according to the nature and number of ligands (**Figure.8**). Complex (1) gradually decomposes in two steps, the melting point is just before the first partial weight loss of 32% at 165°C and the second weight loss of 43% at 470°C, due to the major decomposition. Complex (2) degrades in three steps. The first loss of 21% is between 152°C-210°C and the second of 13% at 286°C while the third and the major one is observed at 392°C with 39%. Complex (3) begins to degrade at 197°C with 16% of weight loss,followed by asmaller weight loss of 9.5% at 250°C, to end up with the last and largest weight loss of 41% at 350°C.

4. Conclusion

In summary, we studied three complexes of vanadium with oxime ligands, twomonomer and a binuclear. All complexes structures were elucidated by single crystal x-ray diffraction and characterized by spectroscopic methods and thermal analysis. Complex (1) exhibits a classical square pyramidal Complex (2)dimeric coordination. has a new vanadium $[VO(HL)_2(L)_2].C_8H_{10}$ structure, the bridge connecting two doubly deprotonated salicylaldoxime ligand. Complex (3) was synthesized by iminoaclylation reaction between 4-hydroxysalicylaldoxime vanadium complex and acetonitrile, which produces the tridentate iminoacylated ligand dioxovanavium. The packing of all the complexes is ensured by various intermolecular interactions; H-bonds and π - π serve the stabilization and cohesion of the structure build. The UV-visible spectroscopy study and thermal analysis showed a good agreement with the structures of the complexes.

Further applications are under way, including the biological application such as antimicrobial, antioxidant and antitumor activity. Further research is plannedfor the synthesis of new complexes in this category.

5. Supplementary Material

CCDC 1851810, CCDC 1857553 and CCDC 1938771contain complete supplementary crystallographic data for complexes (1), (2) and (3), respectively. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif

Supplementary information (SI): Tables.

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	complex (1)	complex (2)	complex (3)
crystal data			
Chemical formula	C14H12N2O5V	C32H27N4O10V2	C9H9N2O5V
Mr	339.20	729.45	276.12
Crystal system, space	Monoclinic	Triclinic	Triclinic
group	P21 /n	P-1	P-1
Temperature (K)	293	100	293
a (Å)	14.718 (4)	10.196 (6)	7.9703 (3)
b (Å)	7.182 (3)	11.895(7)	8.251 (2)
c (Å)	14.781 (5)	14.201 (9)	9.105 (2)
α (°)	90	83.614 (5)	82.285 (10)
β (°)	113.120 (5)	69.880 (6)	82.015 (10)
γ (°)	90	72.708 (6)	65.797
V (Å3)	1437.1 (9)	1544.0 (16)	538.87 (19)
Z	4	2	2
Color	purple	black	yellow
Crystal size (mm)	$0.46 \times 0.31 \times 0.19$ Xcalibur, Atlas	$0.32 \times 0.28 \times 0.17$ Xcalibur, Atlas	$\begin{array}{c} 0.41 \times 0.14 \times 0.10 \\ \text{Enraf-Nonius} \end{array} \text{CAD-4} \end{array}$
Diffractometer	diffractometer	diffractometer	diffractometer
<mark>R1, wR2</mark>	<mark>0.048, 0.128</mark>	<mark>0.074, 0.198</mark>	<mark>0.032, 0.092</mark>
GOF	1.100	1.095	1.075
$(\sin \theta / \lambda) \max(\text{\AA} - 1)$	0.695	0.650	0.660
No. of reflections	3710	5971	2573
(Δ/σ) max	0.001	< 0.001	3.241
Δρmax, Δρmin (eÅ–3)	0.59, -0.50	1.14, -0.65	0.32, -0.21

Table.1: Crystal data and refinement results for complexes (1), (2) and (3).

Bond distances (Å)			
V—005	1.5951 (17)	V—N2	2.060 (2)
V—O3	1.9174 (16)	V—N1	2.060 (2)
V—01	1.9216 (17)		
Bond angles (°)			
005—V—O3	108.55 (8)	O1—V—N2	83.14 (8)
005—V—01	108.95 (9)	O05—V—N1	104.46 (9)
03—V—01	142.50 (8)	O3—V—N1	84.82 (7)
005—V—N2	106.00 (9)	O1—V—N1	86.70 (7)
O3—V—N2	85.97 (7)	N2—V—N1	149.54 (8)

Table.2: Bond distances and bond angles for complex (1).

Table.3: Bond distances and bond angles for complex (2).

Bond distances (Å)			
V01—O05	1.591 (4)	V02—O10	1.600 (4)
V01—O01	1.823 (4)	V02—O06	1.833 (4)
V01—O03	1.864 (4)	V02—O08	1.875 (4)
V01—O09	1.952 (4)	V02—O04	1.939 (4)
V01—N02	2.111 (5)	V02—N04	2.121 (5)
V01—N01	2.318 (5)	V02—N03	2.313 (5)
Bond angles (°)			
O05—V01—O01	102.6 (2)	O10—V02—O06	103.9 (2)
O05—V01—O03	100.25 (19)	O10—V02—O08	100.78 (18)
O01—V01—O03	95.76 (18)	O06—V02—O08	91.75 (18)
O05—V01—O09	95.13 (19)	O10—V02—O04	93.28 (19)
O01—V01—O09	96.31 (17)	O06—V02—O04	97.97 (17)
O03—V01—O09	157.86 (17)	O08—V02—O04	160.50 (16)
O05—V01—N02	92.6 (2)	O10—V02—N04	94.7 (2)
O01—V01—N02	164.80 (17)	O06—V02—N04	161.22 (18)
O03—V01—N02	80.97 (18)	O08—V02—N04	81.84 (18)
O09—V01—N02	82.52 (18)	O04—V02—N04	83.62 (17)
O05—V01—N01	170.6 (2)	O10-V02-N03	171.1 (2)
O01—V01—N01	81.44 (18)	O06—V02—N03	81.26 (19)
O03—V01—N01	87.69 (17)	O08—V02—N03	86.16 (17)
O09—V01—N01	75.86 (17)	O04—V02—N03	78.71 (17)
N02—V01—N01	83.58 (18)	N04—V02—N03	80.72 (19)

Bond distances (Å)			
V001—O5	1.6170 (15)		
V001—O4	1.6300 (17)		
V001—O1	1.8972 (15)		
V001—N2	2.0546 (19)		
V001—N1	2.1185 (17)		
Bond angles (°)			
O5—V001—O4	108.07 (9)	O1—V001—N2	151.83 (7)
O5—V001—O1	100.21 (7)	O5—V001—N1	130.73 (8)
O4—V001—O1	101.69 (8)	O4—V001—N1	120.08 (8)
O5—V001—N2	94.98 (8)	O1-V001-N1	80.66 (7)
O4—V001—N2	95.87 (8)	N2-V001-N1	71.47 (7)

Table.4: Bond distances and bond angles for complex (3).

1.69 (8) ..98 (8) O1-5.87 (8) N2--V001-



Figure.1: The structural unit of complex (1), showing the atomic numbering scheme. Displacement ellipsoids are shown at 50% probability level; H atoms have been omitted for clarity.



Figure.2: Crystal packing of complex (1) viewed along the [100] direction.



Figure.3: The structural unit of complex (2), showing the atomic numbering scheme. Displacement ellipsoids are shown at 50% probability level, H atoms have been omitted for clarity.



Figure.4: Crystal packing of complex (2) viewed along the [100] direction.



Figure.5: The structural unit of complex (3), showing the atomic numbering scheme. Displacement ellipsoids are shown at 50% probability level, H atoms have been omitted for clarity.



Figure.6: Crystal packing of complex (3) viewed along the [010] direction.



Figure.7: UV-Visible absorption spectra in DMSO, comparison between ligand and their complexes.



Figure.8: TGA-Curves of vanadium complexes.

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Highlights

- Synthesis of new vanadium complexes with phenolic oxime.

- The complexes were determined by X-ray diffraction and characterized by thermal and spectroscopic analysis.

- The instability of the complex in organics solvent p-xylene.

- The iminoacylation reaction by acetonitril result a new compound.

Ilyas Belkhettab Study, conception and design.

Sultana Boutamine Supervision, original draft preparation.

Hennia Slaouti Interpretation of results and Contribution to the interpretation of the results.

Med Faouzi Zid Data collection of single crystal and structural resolution.

Habib Boughzala Refinement structures and representation of crystallographic results.

Zakia Hank Involvement in the drafting of the manuscript - Reviewing and Submission procedure.

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Declaration of interests

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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