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# Bimetallic dioxidovanadium(V) complex containing a malonohydrazone derivative ligand: Synthesis, characterization, and crystal structure

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## RESEARCH ARTICLE

## ABSTRACT



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Square pyramidal geometry

In this paper, we report the synthesis and characterization of the dioxidovanadium(V) complex derived from a malonohydrazone ligand ( $N^1,N^3$ -bis(2-hydroxybenzylidene) malonohydrazone). The newly synthesized complex was characterized by infrared spectroscopy (IR), nuclear magnetic resonance (NMR), and the structure of the complex was also established by a single crystal X-ray diffraction study. The bimetallic complex crystallizes in the triclinic space group  $P-1$  with the following parameters  $a = 10.8273(5)$  Å,  $b = 11.4677(6)$  Å,  $c = 15.0366(8)$  Å,  $\alpha = 81.591(4)^\circ$ ,  $\beta = 83.018(4)^\circ$ ,  $\gamma = 76.326(4)^\circ$ ,  $V = 1787.23(16)$  Å<sup>3</sup>,  $Z = 2$ ,  $T = 292.5(2)$  K,  $\mu(\text{MoK}\alpha) = 0.600$  mm<sup>-1</sup>,  $D_{\text{calc}} = 1.463$  g/cm<sup>3</sup>, 11730 reflections measured ( $6.236^\circ \leq 2\theta \leq 58.062^\circ$ ), 7981 unique ( $R_{\text{int}} = 0.0231$ ,  $R_{\text{sigma}} = 0.0506$ ) which were used in all calculations. The final  $R_1$  was 0.0496 ( $I > 2\sigma(I)$ ) and  $wR_2$  was 0.1255 (all data). The ligand was coordinated to the metal ions in a tridentate fashion through the donor O/N/O atoms. The metal ions adopted a square pyramidal geometry with slight distortion. Reaction of the complex with hydrogen peroxide was also carried out, and it was found that the complex reacts with hydrogen peroxide to form a peroxo complex.

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## 1. Introduction

Vanadium is a biologically relevant metal and is present in several naturally occurring compounds such as amavadin is found in *Amanitae mushrooms* [1], blood cells of sea-squirts (*Ascidiceae*) and farm worms [2]. The coordination chemistry of vanadium deals with a large number of oxidation states that range from -3 to +5. The +4 and +5 oxidation states are the most common oxidation states under aerobic conditions [3-8]. The large part of vanadium coordination chemistry in solution originates in +4 and +5 oxidation states from N/O donor ligand complex formation [9-11]. The coordination chemistry of oxovanadium(IV) and (V) species with polyfunctional ligands has acquired renewed interest with the discovery and characterization of vanadate dependent haloperoxidases [12-15]. The further work in vanadium coordination chemistry stems from therapeutic applications of vanadium compounds. This is in particular due to their use in the treatment of diabetes mellitus in humans [16-18]. Vanadium compounds also have the potential to inhibit phosphoryl transfer enzymes and their catalytic potential in organic transformation [19-23]. Vanadium compounds have a wide variety of applications, including as magnetic materials [24,25], as catalysts [26,27], and as cathode materials [28]. Therefore, our objective is to synthesize and characterize a new vanadium(V) complex derived from a

malonohydrazone derivative that mimics the haloperoxidases enzyme activity and helps to understand its key structural and electronic features.

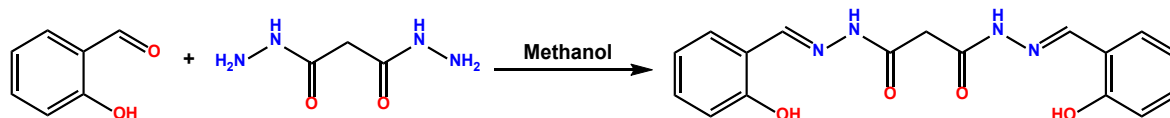
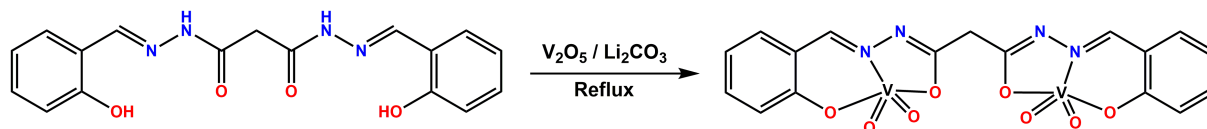
## 2. Experimental

## 2.1. Materials and instrumentation

The solvents were reagent grade and were used as received. Other chemicals were E-Merck, Himedia, or equivalent grades, and all solvents were used as received. All operations were performed under aerobic conditions. Infrared spectra in the range 4000-200 cm<sup>-1</sup> were recorded as KBr discs using a BX-III/FT-IR Perkin Elmer spectrophotometer. The <sup>1</sup>H NMR and <sup>13</sup>C NMR spectra were recorded on Bruker Avance II 400 and 100 MHz, respectively, in DMSO-*d*<sub>6</sub> solution using TMS as internal standard. Electronic spectra were recorded on a Perkin Elmer Lambda-25 spectrophotometer.

## 2.2. Single-crystal X-ray diffraction study

Single crystal X-ray diffraction data was collected using Xcalibur, EOS, Gemini diffractometer equipped with a monochromated MoK $\alpha$  radiation ( $\lambda = 0.71073$  Å).

Scheme 1. Synthesis of  $N^1,N^3$ -bis(2-hydroxybenzylidene)malonohydrazide.

Scheme 2. Synthesis of bis(dioxidovanadate(V)) complex.

The crystal structure was solved by SHELXT and refined by SHELXL-2014 [29,30]. All non-hydrogen atoms were refined anisotropically, whereas the hydrogen atoms were placed at a calculated position and refined in the final refinement.

### 2.3. Preparation of $N^1,N^3$ -bis(2-hydroxybenzylidene)malonohydrazide ligand ( $H_4SLMH$ )

To an aqueous methanol solution of malonoyldihydrazine (0.66 g, 1 mmol), 2-hydroxybenzaldehyde (1.72 g, 1 mmol) was added and the reaction mixture stirred for approximately half an hour at 40 °C (Scheme 1). The white precipitate obtained was filtered and washed with hot methanol and dried over anhydrous  $CaCl_2$ .

$N^1,N^3$ -bis(2-Hydroxybenzylidene)malonohydrazide ( $H_4SLMH$ ): Colour: White. Yield: 96%. FT-IR (KBr,  $\nu$ ,  $cm^{-1}$ ): 3279, 3188, 3063, 2967, 2905, 2870, 1667, 1610, 1569, 1487, 1391, 1359.  $^1H$  NMR (400 MHz,  $DMSO-d_6$ ,  $\delta$ , ppm): 11.85 (s, 1H, NH), 11.49 (s, 1H, NH), 11.07 (s, 1H, OH), 11.05 (s, 1H, OH), 8.41 (s, 1H,  $CH=N$ ), 8.27 (s, 1H,  $CH=N$ ), 7.65-6.70 (m, 8H, Ar-H), 3.90 (s, 1H,  $CH_2$ ), 3.60 (s, 1H,  $CH_2$ ).  $^{13}C$  NMR (100 MHz,  $DMSO-d_6$ ,  $\delta$ , ppm): 162.39 (C=O), 157.26 (Ar-C-OH), 147.10 (C=N), 131.27 (Ar-C), 129.23 (Ar-C), 119.30 (Ar-C), 118.60 (Ar-C=C), 116.28 (Ar-C), 41.60 ( $CH_2$ ).

### 2.4. Synthesis of complex

$N^1,N^3$ -bis(2-Hydroxybenzylidene)malonohydrazide (1.00 g, 2.94 mmol) was dissolved in 30 mL of methanol and stirred at 70 °C. To this solution, vanadium pentoxide ( $V_2O_5$ ) (1.06 g, 5.83 mmol) in 30 mL methanol and lithium carbonate ( $Li_2CO_3$ ) (0.65 g, 8.82 mmol) in 20 mL methanol were added slowly accompanied by gentle stirring for 15 minutes (Scheme 2). The brown color solution was then refluxed for 1 h, filtered, washed three times with hot methanol (20 mL each time), and dried over anhydrous  $CaCl_2$ . Colour: Light brown. Yield: 93%. FT-IR (KBr,  $\nu$ ,  $cm^{-1}$ ): 3433, 1647, 1611, 1558, 1446, 1363, 1280, 933, 896, 753.  $^1H$  NMR (400 MHz,  $DMSO-d_6$ ,  $\delta$ , ppm): 8.78 (s, 2H,  $C(H)=N$ ), 7.93 (s, 1H, Ar-H), 7.49 (t, 2H, Ar-H), 7.30 (t, 2H, Ar-H), 6.75 (dd, 3H, Ar-H), 3.26 (s, 2H,  $CH_2$ ).  $^{13}C$  NMR (100 MHz,  $DMSO-d_6$ ,  $\delta$ , ppm): 172.12 (C=N), 164.37 (C=N), 162.36 (C=O), 154.77 (C-O), 132.91, 132.42 (Ar-C), 119.68 (Ar-C=C), 116.62 (Ar-C), 39.93, 39.09 ( $-CH_2$ ).

## 3. Results and discussion

### 3.1. Synthesis

The complex has been synthesized from the reaction of  $N^1,N^3$ -bis(2-hydroxybenzylidene)malonohydrazide with vanadium pentoxide ( $V_2O_5$ ) and  $Li_2CO_3$  in 1:1:3 molar ratio in methanol under refluxing conditions for one hour. The isolated complex has been found to have the composition  $[Li(H_2O)_4]_2[(VO_2)_2(SLMH)]$  based on data obtained from analytical and

crystallographic studies. The complex is yellow in color and air stable. The complex is insoluble in common organic solvents such as dichloromethane, chloroform, benzene, hexane, and ether; slightly soluble in water, methanol, acetonitrile and acetone but completely soluble in highly coordinating solvents such as DMSO and DMF.

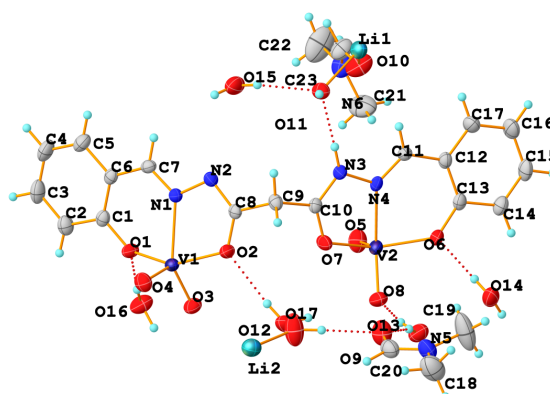
Some structurally significant IR bands for the uncoordinated ligand and vanadium(V) complex, which are useful in determining the mode of coordination of the ligand. The IR spectra of the uncoordinated ligand show very strong bands at 3279 and 3188  $cm^{-1}$ , which are attributed to the joint contributions from stretching vibrations of the secondary NH and OH groups. A couple of strong bands are observed at 1667 and 1610  $cm^{-1}$  in the uncoordinated ligands. These bands owe their origin to  $>C=O$  groups. When the IR spectra of the complex are compared with those of the uncoordinated ligand in the region below the 1000  $cm^{-1}$ , new weak to medium to strong bands have been observed in the 628-603 and 546-490  $cm^{-1}$  regions, respectively. As these bands are not observed in the IR spectra of the ligands, they are attributed to the vibrations of rocking and wagging of water molecules bonded to the metal centers, respectively [31]. In addition to the ligand bands, the complex displayed a strong band, appearing in the region of 933 and 896  $cm^{-1}$ , these bands were assigned to the  $V=O$  terminal stretching frequencies which are typical of the *cis*- $VO_2$  core, as noted previously by other workers for similar complexes [31].

The ligand  $H_4SLMH$  shows four signals at  $\delta$  11.85, 11.49, 11.07, and 11.05 ppm due to OH and NH protons. The absence of signals at  $\delta$  11.07 and  $\delta$  11.05 ppm in the complex, suggests that bonding of phenolate/naphtholate oxygen atom to metal center via deprotonation of OH group. Similarly, the non-observance of any signal at  $\delta$  11.85 and 11.49 ppm due to secondary NH protons indicates involvement of carbonyl oxygen atom in *enol* form in bonding to metal centers [32]. The aromatic protons for the ligand and complex appear in the expected region. Another important feature of  $^1H$  NMR spectra of the complex is the upfield shift shown by methylene protons which appear at  $\delta$  3.26 ppm as compared to their position at  $\delta$  3.90 and 3.60 ppm in free ligand.

The complex has fairly highly solubility in DMF solution, hence their electronic spectrum was recorded in this solvent. The ligand shows absorption bands at 296 and 328 nm, whereas the complex shows absorption bands at 305 nm (30240  $dm^3/mol.cm$ ), 424 (20400  $dm^3/mol.cm$ ) nm. Such a large shift of ligand bands on complexation indicates a strong bonding between phenolate oxygen atoms and vanadium metal centers. This may be attributed to the complexation effect of the ligand do not show any band in the visible region of electronic spectra as the metal center being present in +5 oxidation state does not contain any electron in its 3d orbital [32].

**Table 1.** Crystal data and structure refinement for bis(dioxidovanadate(V)) complex.

Empirical formula	C <sub>23</sub> H <sub>39</sub> Li <sub>2</sub> N <sub>6</sub> O <sub>17</sub> V <sub>2</sub>
Formula weight	787.36
Temperature (K)	292.5(2)
Crystal system	Triclinic
Space group	<i>P</i> -1
<i>a</i> , (Å)	10.8273(5)
<i>b</i> , (Å)	11.4677(6)
<i>c</i> , (Å)	15.0366(8)
$\alpha$ (°)	81.591(4)
$\beta$ (°)	83.018(4)
$\gamma$ (°)	76.326(4)
Volume (Å <sup>3</sup> )	1787.23(16)
<i>Z</i>	2
$\rho_{\text{calc}}$ (g/cm <sup>3</sup> )	1.463
$\mu$ (mm <sup>-1</sup> )	0.600
<i>F</i> (000)	814.0
Crystal size (mm <sup>3</sup> )	0.19 × 0.16 × 0.08
Radiation	MoK $\alpha$ ( $\lambda$ = 0.71073)
2 $\theta$ range for data collection (°)	6.236 to 58.062
Index ranges	-13 ≤ <i>h</i> ≤ 12, -15 ≤ <i>k</i> ≤ 9, -18 ≤ <i>l</i> ≤ 20
Reflections collected	11730
Independent reflections	7981 [ <i>R</i> <sub>int</sub> = 0.0231, <i>R</i> <sub>sigma</sub> = 0.0506]
Data/restraints/parameters	7981/0/481
Goodness-of-fit on <i>F</i> <sup>2</sup>	1.038
Final <i>R</i> indexes [ <i>I</i> ≥ 2 $\sigma$ ( <i>I</i> )]	<i>R</i> <sub>1</sub> = 0.0496, <i>wR</i> <sub>2</sub> = 0.1151
Final <i>R</i> indexes [all data]	<i>R</i> <sub>1</sub> = 0.0682, <i>wR</i> <sub>2</sub> = 0.1255
Largest diff. peak/hole (e.Å <sup>-3</sup> )	0.63/-0.51

**Figure 1.** Molecular structure of bis(dioxidovanadate(V)) complex.

### 3.2. Molecular structure of the complex

The ORTEP plot of the crystal of the complex is shown in Figure 1 and the crystal packing diagram is given in Figure 2. The complex crystallizes in triclinic form with the space group *P*-1. Crystal and structure refinement data are given in Table 1. The bond lengths and bond angles are given in Tables 2 and 3. The complex is composed of one ligand, two vanadium atoms, together with lithium atom bonded to different water molecules, and DMF molecules in the crystal lattice. The ligand bonded to two vanadium atoms in a tridentate fashion; as a result, the protonation likely occurred at the oxygen atoms of the ligand.

The coordination modes of the ligand to metal ions are shown in Figure 1. The basal position around V1 is composed of O1, N1, and O2 whereas the terminal position is composed of O3 and O4 and the basal position around V2 is composed of O5, O6 and N4 whereas the terminal position is composed of O7 and O8, respectively. The vanadium-nitrogen bond distances in the complex are 2.154(2) Å for V1-N1 and 2.132(2) Å for V2-N4, whereas the vanadium-oxygen bond distances around V1 are 1.9042(18) Å (V1-O1), 1.9757(18) Å (V1-O2), 1.6306(18) Å (V1-O3) and 1.6125(18) Å for V1-O4. The angles around V1 are 148.87(8)° for O1-V1-O2 and 142.18(9)° for O3-V1-N1. The distance of the vanadium-oxygen bond around V2 is 1.9154(18) Å (V2-O6), 1.9761(18) Å (V2-O5), 1.599(2) Å (V2-O7), and 1.642(2) Å (V2-O8), with their angles 145.19(8)° for O6-V2-O5

and 144.92(10)° for O8-V2-N4. The geometrical index around V1 and V2 are  $\tau_1 = 0.11$ ,  $\tau_2 = 0$ , respectively. Hence, the geometry around V1 can be described as a square pyramidal geometry with slight distortion whereas around V2 a perfectly square pyramidal geometry with no distortion was observed.

### 3.3. Reaction of the complex with hydrogen peroxide

When an aqueous solution of 30% H<sub>2</sub>O<sub>2</sub> is added to a methanolic solution of the bimetallic bis(dioxidovanadate(V)) complex, the formation of the bimetallic bis(monooxidomono peroxidovanadate(V)) complex occurs [33]. We could isolate the bis(monooxoperoxidovanadate(V)) complex by performing the reaction between the prepared complex and 30% H<sub>2</sub>O<sub>2</sub> in a 1:5 molar ratio in methanol at 0 °C (Scheme 3). We have been able to establish its formation by iodometric titration. The experimentally determined value of peroxide was 5.0 and 4.5% in peroxo compound of complex and calculated on the basis of formation bis(monooxidomonoperoxidovanadate(V)) complex. This indicated that the instability in the solid state [33]. However, the complex might be stable in the solution state. Furthermore, the formation of the complex was established by absorption spectroscopy (Figure 3). The IR spectra of the complex show very strong bands at 933 and 866 cm<sup>-1</sup>. These bands most probably masks band due to *cis*-VoO<sub>2</sub><sup>2+</sup> group which also appears almost at the same position [28].

**Table 2.** Bond lengths for bis(dioxidovanadate(V)) complex.

Atom	Atom	Length (Å)	Atom	Atom	Length (Å)
V1	O1	1.9042(18)	O9	C20	1.234(4)
V1	O2	1.9757(18)	C8	C9	1.503(3)
V1	O3	1.6306(18)	C12	C11	1.438(3)
V1	O4	1.6125(18)	C12	C13	1.404(4)
V1	N1	2.154(2)	C12	C17	1.400(4)
V2	O6	1.9154(18)	C6	C7	1.424(4)
V2	O5	1.9761(18)	C6	C1	1.410(4)
V2	N4	2.132(2)	C6	C5	1.406(4)
V2	O7	1.599(2)	O12	Li2	1.928(6)
V2	O8	1.642(2)	N5	C20	1.301(4)
O1	C1	1.328(3)	N5	C18	1.451(4)
O2	C8	1.295(3)	N5	C19	1.447(4)
O11	Li1	1.929(5)	C13	C14	1.396(4)
O6	C13	1.336(3)	N6	C23	1.313(4)
O5	C10	1.289(3)	N6	C21	1.439(5)
N4	N3	1.395(3)	N6	C22	1.432(5)
N4	C11	1.284(3)	C1	C2	1.394(4)
O16	Li2 <sup>1</sup>	1.925(5)	C14	C15	1.369(4)
N2	N1	1.402(3)	C5	C4	1.366(4)
N2	C8	1.294(3)	C17	C16	1.376(4)
N3	C10	1.286(3)	C4	C3	1.376(5)
N1	C7	1.293(3)	C2	C3	1.381(4)
O13	Li1 <sup>2</sup>	1.917(5)	C15	C16	1.382(5)
O14	Li1 <sup>3</sup>	1.934(5)	Li1	O13 <sup>6</sup>	1.917(5)
O17	Li2 <sup>4</sup>	1.971(6)	Li1	O14 <sup>3</sup>	1.934(5)
O15	Li2 <sup>5</sup>	1.929(6)	Li2	O16 <sup>7</sup>	1.925(5)
O10	C23	1.210(4)	Li2	O17 <sup>4</sup>	1.971(6)
O10	Li1	1.923(5)	Li2	O15 <sup>5</sup>	1.929(6)
C10	C9	1.501(3)			

<sup>1</sup>+x, 1+y, +z; <sup>2</sup>-1+x, +y,+z; <sup>3</sup>1-x, 1-y, -z; <sup>4</sup>-x, 1-y, 1-z; <sup>5</sup>1-x, 1-y, 1-z; <sup>6</sup>1+x, +y,+z; <sup>7</sup>+x, -1+y, +z.

**Table 3.** Bond angles for bis(dioxidovanadate(V)) complex.

Atom	Atom	Atom	Angle (°)	Atom	Atom	Atom	Angle (°)
O1	V1	O2	148.87(8)	C17	C12	C13	119.5(2)
O1	V1	N1	82.01(8)	C1	C6	C7	122.1(2)
O2	V1	N1	73.14(7)	C5	C6	C7	118.8(3)
O3	V1	O1	95.72(9)	C5	C6	C1	119.0(2)
O3	V1	O2	92.74(9)	N4	C11	C12	123.7(2)
O3	V1	N1	142.18(9)	C20	N5	C18	120.8(3)
O4	V1	O1	103.77(9)	C20	N5	C19	120.8(3)
O4	V1	O2	101.49(9)	C19	N5	C18	118.5(3)
O4	V1	O3	109.44(11)	O6	C13	C12	122.3(2)
O4	V1	N1	107.73(9)	O6	C13	C14	119.3(2)
O6	V2	O5	145.19(8)	C14	C13	C12	118.3(2)
O6	V2	N4	82.41(8)	C23	N6	C21	122.0(3)
O5	V2	N4	73.29(8)	C23	N6	C22	120.7(3)
O7	V2	O6	105.43(10)	C22	N6	C21	117.3(3)
O7	V2	O5	104.49(10)	N1	C7	C6	123.7(2)
O7	V2	N4	104.31(10)	O1	C1	C6	121.9(2)
O7	V2	O8	109.50(12)	O1	C1	C2	119.1(2)
O8	V2	O6	97.14(9)	C2	C1	C6	118.9(2)
O8	V2	O5	89.25(9)	C10	C9	C8	112.2(2)
O8	V2	N4	144.92(10)	C15	C14	C13	120.8(3)
C1	O1	V1	134.79(16)	C4	C5	C6	121.1(3)
C8	O2	V1	118.68(16)	C16	C17	C12	121.2(3)
C13	O6	V2	131.93(16)	C5	C4	C3	119.6(3)
C10	O5	V2	118.64(16)	C3	C2	C1	120.2(3)
N3	N4	V2	115.46(15)	C14	C15	C16	121.5(3)
C11	N4	V2	128.39(17)	C17	C16	C15	118.6(3)
C11	N4	N3	116.0(2)	O9	C20	N5	126.0(3)
C8	N2	N1	108.1(2)	C4	C3	C2	121.2(3)
C10	N3	N4	109.2(2)	O10	C23	N6	125.4(3)
N2	N1	V1	115.79(15)	O11	Li1	O14 <sup>1</sup>	117.6(3)
C7	N1	V1	128.93(17)	O13 <sup>2</sup>	Li1	O11	109.9(2)
C7	N1	N2	115.2(2)	O13 <sup>2</sup>	Li1	O14 <sup>1</sup>	106.0(3)
C23	O10	Li1	132.3(3)	O13 <sup>2</sup>	Li1	O10	112.9(3)
O5	C10	C9	118.0(2)	O10	Li1	O11	105.6(3)
N3	C10	O5	122.9(2)	O10	Li1	O14 <sup>1</sup>	105.0(2)
N3	C10	C9	119.1(2)	O16 <sup>3</sup>	Li2	O17 <sup>4</sup>	103.2(3)
O2	C8	C9	118.1(2)	O16 <sup>3</sup>	Li2	O15 <sup>5</sup>	119.4(3)
N2	C8	O2	123.8(2)	O16 <sup>3</sup>	Li2	O12	101.2(3)
N2	C8	C9	118.2(2)	O15 <sup>5</sup>	Li2	O17 <sup>4</sup>	115.1(3)
C13	C12	C11	121.7(2)	O12	Li2	O17 <sup>4</sup>	114.1(3)
C17	C12	C11	118.7(2)	O12	Li2	O15 <sup>5</sup>	103.3(3)

<sup>1</sup>1-x, 1-y, -z; <sup>2</sup>1+x, +y, +z; <sup>3</sup>+x, -1+y, +z; <sup>4</sup>-x, 1-y, 1-z; <sup>5</sup>1-x, 1-y, 1-z.

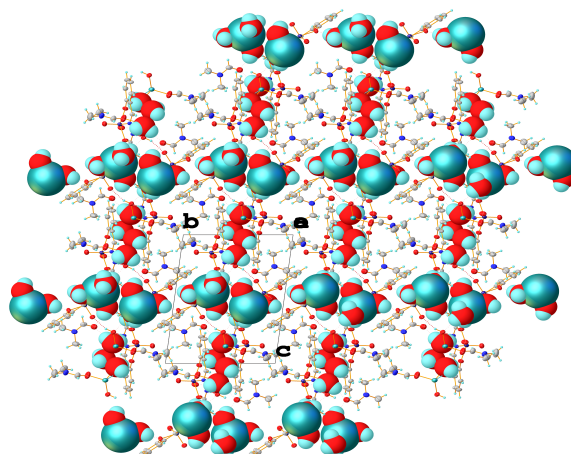


Figure 2. Packing diagram of the bis(dioxidovanadate(V)) complex along the crystallographic *a* axis.

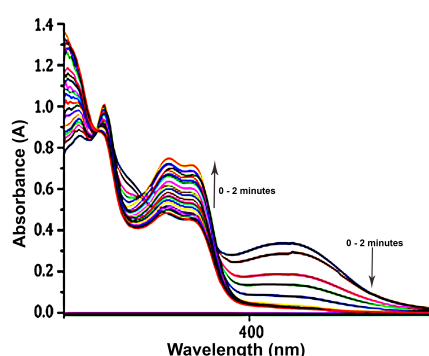
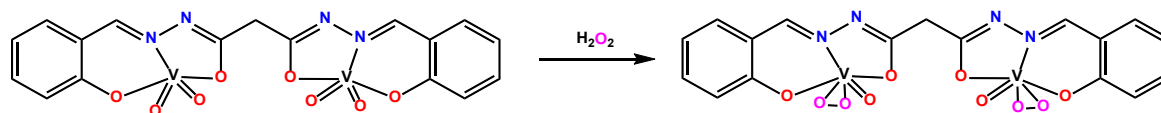


Figure 3. Electronic spectrum of complex titration with 30% H<sub>2</sub>O<sub>2</sub> (0-2 minutes).



Scheme 3. Schematic diagram showing the reaction of complex with hydrogen peroxide.

#### 4. Conclusion

We have successfully synthesized and characterized bimetallic dioxidovanadium(V) complex derived from from *N*'<sup>1</sup>,*N*'<sup>3</sup>-bis(2-hydroxybenzylidene)malonohydrazide. The ligand coordinated to the vanadium ions in tridentates fashion through -ONO- donor's atoms. The ligand is present in enol form in the complex and therefore the ligand coordinates with the vanadium center through phenolate oxygen, enolate oxygen, and azomethine nitrogen atoms. The metal ions adopted a square pyramidal geometry with slight distortion.

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#### Supporting information

CCDC-2189533 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge via [www.ccdc.cam.ac.uk/data\\_request/cif](http://www.ccdc.cam.ac.uk/data_request/cif), or by e-mailing [data\\_request@ccdc.cam.ac.uk](mailto:data_request@ccdc.cam.ac.uk), or by contacting The Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44(0)1223-336033.

#### Disclosure statement


Conflict of interest: The authors declare that they have no conflict of interest.

Ethical approval: All ethical guidelines have been adhered.

Sample availability: Sample of the compound is available from the author.

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