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SYNTHESIS AND CRYSTAL STRUCTURES OF OXIDOVANADIUM(V) COMPLEXES DERIVED FROM HYDRAZONES WITH THE CATALYTIC PROPERTY

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Two new structurally similar oxidovanadium(V) complexes [VOL^{1}L] (**1**) and [VOL^{2}L] (**2**) (L = acetohydroxamate) derived from N′-(5-bromo-2-hydroxybenzylidene)-2-methylbenzohydrazide (H_2L^1) and N′-(2-hydroxy-4-methoxybenzylidene)-3-methylbenzohydrazide (H_2L^2) hydrazones are prepared and characterized by elemental analyses, FT–IR, ^{1}H NMR, and single crystal X-ray diffraction (CIF files CCDC Nos. 1859442 (**1**) and 1859446 (**2**)). The V atoms in the complexes are in the octahedral coordination, with hydrazones behaving as binegative donors and acetohydroxamate acting as a mononegative ligand. The complexes function as effective olefin epoxidation catalysts.

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Keywords: hydrazone ligand, oxidovanadium complexes, catalytic property, crystal structure.

INTRODUCTION

Vanadium is a bioelement involved in various catalytic and inhibitory processes. Vanadium complexes have received considerable attention due to their biochemical significance [1–4] and industrial catalytic processes [5–8]. The complexes are widely used as efficient catalysts in the oxidation of olefins [9–11]. Recently, the increasing interest has been focused on vanadium(V) Schiff base complexes and their catalytic property [12–14]. The excellent catalytic activity of mononuclear oxidovanadium(V) complexes incorporating tridentate ligands has been already reported by H. Mimoun et al. [15]. The vanadium(V) complexes with tridentate Schiff base ligands derived from chiral and achiral aminoalcohols have been used successfully as catalysts in the enantioselective oxidation of organic sulfides, the asymmetric alkynylation of aldehydes, the epoxidation of cyclooctene, the oxidation of bromide, and the stereoselective synthesis of functionalized tetrahydrofurans [16]. Hydrazones are a kind of interesting Schiff base ligands in coordination chemistry, which can form a variety of complexes with different metal ions [17, 18]. Recently, our research group has reported the catalytic property of vanadium complexes with hydrazone ligands [19]. As a continuation of this work, two new vanadium(V) complexes [VOL^{1}L] (**1**) and [VOL^{2}L] (**2**) (L = acetohydroxamate), derived from N′-(5-bromo-2-hydroxybenzylidene)-2-methylbenzohydrazide (H_2L^1) and N′-(2-hydroxy-4-methoxybenzylidene)-3-methylbenzohydrazide (H_2L^2; Scheme 1) aroylhydrazones are presented.

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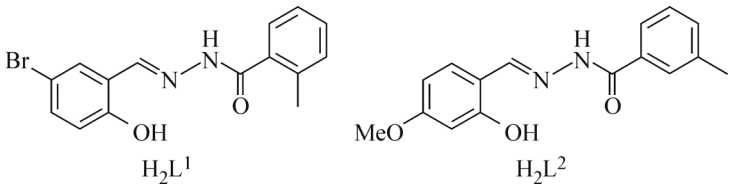


Scheme 1.

EXPERIMENTAL

Materials and methods. All chemicals and solvents were of analytical reagent grade and used as received. Microanalyses for C, H, N were carried out using a Perkin Elmer 2400 CHNS/O elemental analyzer. FT–IR spectra were recorded on FT–IR 8400-Shimadzu as KBr discs in the range 400–4000 cm^{-1}. ^1H NMR spectra were recorded at 25 °C on a Bruker AVANCE 300 MHz instrument. X-ray diffraction data were collected using a Bruker Smart APEX II diffractometer.

Synthesis of H_2L^1. A methanolic solution (20 mL) containing 5-bromo-2-hydroxybenzaldehyde (1.0 mmol, 0.201 g) was added dropwise to a methanolic solution of 2-methylbenzohydrazide (1.0 mmol, 0.148 g) with constant stirring. The mixture was refluxed for 30 min, and the resulting precipitate was filtered off, washed with cold methanol, and dried in a desiccator over silica gel.

Yield: 0.26 g, 78%. FT–IR (KBr), cm^{-1}: ν(O*H*) 3447, ν(N*H*) 3231, ν(C*H*) 2810–3130, ν(C=O) 1647, ν(C=N) 1607, ν(C–O) 1252. ^1H NMR (300 MHz, DMSO-d^6, ppm): δ = 12.15 (s, 1H, O*H*), 11.21 (s, 1H, N*H*), 8.59 (s, 1H, C*H*=N), 7.92 (d, 1H, Ar*H*), 7.78 (s, 1H, Ar*H*), 7.61 (t, 1H, Ar*H*), 7.43 (d, 1H, Ar*H*), 7.31 (d, 1H, Ar*H*), 7.21 (t, 1H, Ar*H*), 6.90 (d, 1H, Ar*H*), 2.45 (s, 3H, CH_3). Anal. calcd. (%) for $C_{15}H_{13}BrN_2O_2$: C 54.07, H 3.93, N 8.41. Found (%): C 53.85, H 4.02, N 8.33.

Synthesis of H_2L^2. A methanolic solution (20 mL) containing 2-hydroxy-4-methoxybenzaldehyde (1.0 mmol, 0.152 g) was added dropwise to a methanolic solution of 3-methylbenzohydrazide (1.0 mmol, 0.148 g) with constant stirring. The mixture was refluxed for 30 min, and the resulting precipitate was filtered off, washed with cold methanol, and dried in a desiccator over silica gel.

Yield: 0.23 g, 81%. FT–IR (KBr), cm^{-1}: ν(O*H*) 3439, ν(N*H*) 3218, ν(C*H*) 2810–3130, ν(C=O) 1645, ν(C=N) 1605, ν(C–O) 1255. ^1H NMR (300 MHz, DMSO-d^6, ppm): δ = 12.27 (s, 1H, O*H*), 11.10 (s, 1H, N*H*), 8.62 (s, 1H, C*H*=N), 7.87 (d, 1H, Ar*H*), 7.71 (s, 1H, Ar*H*), 7.68 (d, 1H, Ar*H*), 7.45 (d, 1H, Ar*H*), 7.40 (t, 1H, Ar*H*), 6.56 (d, 1H, Ar*H*), 6.45 (s, 1H, Ar*H*), 3.87 (s, 3H, OCH_3), 2.32 (s, 3H, CH_3). Anal. calcd. (%) for $C_{16}H_{16}N_2O_3$: C 67.59, H 5.67, N 9.85. Found (%): C 67.72, H 5.75, N 9.80.

Synthesis of [VOL^{1}L] (1). A methanolic solution (10 mL) of $VO(acac)_2$ (0.1 mmol, 0.026 g) was added to a solution of H_2L^1 (0.1 mmol, 0.033 g) and acetohydroxamic acid (0.1 mmol, 0.0075 g) in methanol (10 mL) and the resulting orange mixture was refluxed for 30 min. After cooling, the solution was filtered and left to stand overnight. Orange single crystals suitable for crystallography were separated after a week and dried in a vacuum desiccator over silica gel.

Yield: 0.023 g, 49%. IR (KBr), cm^{-1}: ν(N*H*) 3252, ν(C*H*) 2810–3150, ν(C=N) 1600, ν(C–O) 1165, ν(VO) 973. ^1H NMR (300 MHz, DMSO-d^6, ppm): δ = 14.01 (s, 1H, N*H*), 8.96 (s, 1H, C*H*=N), 7.93 (d, 1H, Ar*H*), 7.78 (s, 1H, Ar*H*), 7.62 (m, 1H, Ar*H*), 7.38 (d, 1H, Ar*H*), 7.27 (d, 1H, Ar*H*), 7.17 (q, 1H, Ar*H*), 6.85 (d, 1H, Ar*H*), 2.51 (s, 3H, PhCH_3), 2.09 (s, 3H, CH_3). Anal. calcd. (%) for $C_{17}H_{15}BrN_3O_5V$: C 43.24, H 3.20, N 8.90. Found (%): C 43.11, H 3.32, N 8.82.

Synthesis of [VOL^{2}L] (2). A methanolic solution (10 mL) of $VO(acac)_2$ (0.1 mmol, 0.026 g) was added to a solution of H_2L^2 (0.1 mmol, 0.028 g) and acetohydroxamic acid (0.1 mmol, 0.0075 g) in methanol (10 mL) and the resulting orange mixture was refluxed for 30 min. After cooling, the solution was filtered and left to stand overnight. Orange single crystals suitable for crystallography were separated after a week and dried in a vacuum desiccator over silica gel.

Yield: 0.026 g, 62%. IR (KBr), cm^{-1}: ν(N*H*) 3245, ν(C*H*) 2810–3150, ν(C=N) 1595, ν(C–O) 1162, ν(VO) 971. ^1H NMR (300 MHz, DMSO-d^6, ppm): δ = 13.97 (s, 1H, N*H*), 8.97 (s, 1H, C*H*=N), 7.70–7.64 (m, 3H, Ar*H*), 7.35 (d, 2H,

Ar*H*), 6.62 (q, 1H, Ar*H*), 6.47 (d, 1H, Ar*H*), 3.83 (s, 3H, OCH_3), 2.37 (s, 3H, PhCH_3), 2.09 (s, 3H, CH_3). Anal. calcd. (%) for $C_{18}H_{18}N_3O_6V$: C 51.07, H 4.29, N 9.93. Found (%): C 51.23, H 4.18, N 10.05.

X-ray structure determination. The crystal structures of the complexes were measured on a Bruker SMART Apex II CCD diffractometer using MoK_α radiation ($\lambda = 0.71073$ Å) and a graphite monochromator at 25 °C. Unit cell and reflection data were obtained by standard methods [20] and are summarized in Table 1. The structures were solved, refined, and prepared for publication using the SHEXTL package (structure solution refinements and molecular graphics) [21], and using full matrix least squares techniques against F^2 with anisotropic displacement factors for all non-hydrogen atoms. The amino H atoms were located from difference Fourier maps and refined isotropically, with N–H distances restrained to 0.90(1) Å. Positions of the remaining hydrogen atoms were calculated from the geometry of the molecular skeleton and their thermal displacement parameters were refined isotropically on a groupwise basis. Selected bond lengths and angles are reported in Table 2. H-bonding distances and angles are shown in Table 3.

Catalytic epoxidation of olefins. To a solution of olefins (0.28 mmol), $NaHCO_3$ (0.11 mmol), and a catalyst (9.4×10^{-4} mmol) in MeCN (0.5 mL) H_2O_2 (1.1 mmol, 30% H_2O_2 in water) as an oxidant was added. After the reaction was over, for the analysis of the products, the solution was subjected to multiple ether extraction, and the extract was also concentrated down to 0.5 mL by distillation in a rotary evaporator at room temperature. Then a sample (2 μL) was taken from the solution and analyzed by GC. The retention times of the peaks were compared with those of commercial standards, and chlorobenzene was used as the internal standard for the GC yield calculation.

TABLE 1. Crystal Data and Structure Refinement for the Complexes

Parameters	**1**	**2**
Molecular formula	$C_{17}H_{15}BrN_3O_5V$	$C_{18}H_{18}N_3O_6V$
Formula weight	472.17	423.29
Temperature, K	298(2)	298(2)
Wavelength, Å	0.71073	0.71073
Crystal size, mm	0.18×0.15×0.15	0.30×0.27×0.27
Crystal system	Orthorhombic	Monoclinic
Space group	*Pccn*	$P2_1/n$
a, *b*, *c*, Å	31.1131(11), 7.6141(3), 15.7710(6)	7.3220(7), 18.7057(13), 13.6304(12)
β, deg.	–	92.867(1)
V, Å^3	3736.1(2)	1864.5(3)
Z	8	4
D_{calc}, g/cm^3	1.679	1.508
Absorption coefficient, mm^{-1}	2.704	0.573
F(000)	1888	872
θ range for data collection, deg.	2.58–25.50	2.64–25.39
Index ranges	$-36 \le h \le 37$, $-9 \le k \le 8$, $-19 \le l \le 19$	$-8 \le h \le 8$, $-22 \le k \le 21$, $-16 \le l \le 16$
Reflections collected	30969	16658
Independent reflection (R_{int})	3484 (0.0467)	3380 (0.0586)
Reflections observed ($I > 2\sigma(I)$)	2625	2353
Data completeness, %	100	98.7
Maximum and minimum transmission	0.6872 and 0.6418	0.8606 and 0.8468
Data / restraints / parameters	3484 / 1 / 249	3380 / 1 / 260
GOOF on F^2	1.085	1.027
Final *R* indices ($I > 2\sigma(I)$)	0.0403, 0.1009	0.0458, 0.0917
R indices (all data)	0.0636, 0.1124	0.0840, 0.1061
Highest peak and deepest hole, e/Å^3	1.654 and –0.314	0.354 and –0.216

TABLE 2. Selected Bond Lengths (Å) and Angles (deg.) for the Complexes

		1	
V(1)–O(1)	1.860(2)	V(1)–O(2)	1.943(2)
V(1)–O(4)	1.856(2)	V(1)–O(5)	1.589(3)
V(1)–O(3)	2.202(2)	V(1)–N(1)	2.084(2)
O(5)–V(1)–O(4)	96.50(11)	O(5)–V(1)–O(1)	99.32(13)
O(4)–V(1)–O(1)	103.22(10)	O(5)–V(1)–O(2)	99.83(12)
O(4)–V(1)–O(2)	92.99(9)	O(1)–V(1)–O(2)	153.24(10)
O(5)–V(1)–N(1)	98.17(11)	O(4)–V(1)–N(1)	162.09(10)
O(1)–V(1)–N(1)	84.49(10)	O(2)–V(1)–N(1)	74.43(9)
O(5)–V(1)–O(3)	173.01(11)	O(4)–V(1)–O(3)	76.54(9)
O(1)–V(1)–O(3)	83.06(10)	O(2)–V(1)–O(3)	80.17(10)
N(1)–V(1)–O(3)	88.58(9)		
		2	
V(1)–O(1)	1.8556(19)	V(1)–O(2)	1.9502(18)
V(1)–O(5)	1.8559(19)	V(1)–O(6)	1.590(2)
V(1)–O(4)	2.219(2)	V(1)–N(1)	2.072(2)
O(6)–V(1)–O(1)	99.01(10)	O(6)–V(1)–O(5)	96.14(10)
O(1)–V(1)–O(5)	105.66(8)	O(6)–V(1)–O(2)	99.91(9)
O(1)–V(1)–O(2)	154.02(9)	O(5)–V(1)–O(2)	89.83(8)
O(6)–V(1)–N(1)	99.70(10)	O(1)–V(1)–N(1)	84.80(8)
O(5)–V(1)–N(1)	159.41(9)	O(2)–V(1)–N(1)	74.60(8)
O(6)–V(1)–O(4)	172.37(10)	O(1)–V(1)–O(4)	82.82(8)
O(5)–V(1)–O(4)	76.24(8)	O(2)–V(1)–O(4)	80.78(8)
N(1)–V(1)–O(4)	87.83(8)		

TABLE 3. Hydrogen Bonding Interactions (Å, deg.)

D–H···*A*	*d*(*D*–H)	*d*(H···*A*)	*d*(*D*···*A*)	Angle(*D*–H···*A*)
		1		
N(3)–H(3)··· N(2)^{i}	0.90(1)	2.017(14)	2.909(3)	170(4)
		2		
N(3)–H(3A)···N(2)^{ii}	0.90(1)	1.962(11)	2.862(3)	179(4)

Symmetry codes: ^{i} x, 1/2–y, –1/2+z; ^{ii} 1/2+x, 1/2–y, 1/2+z.

RESULTS AND DISCUSSION

Chemistry. The reaction of $VO(acac)_2$ and acetohydroxamic acid with the tridentate hydrazone ligands H_2L^1 and H_2L^2 in methanol led to the formation of the complexes. Crystals of the complexes are stable at room temperature and soluble in DMSO, DMF, methanol, ethanol, acetonitrile and less soluble in other common solvents such as dichloromethane, chloroform, and insoluble in benzene, *n*-hexane, and CCl_4.

Spectral analysis. ^{1}H NMR data on the hydrazone ligands when compared with the complexes reveal that the ligands serve as a tridentate binegative ONO donor. The azomethine C–H signals in the complexes shifted up-field from their original positions in the free ligands upon coordination of the –CH=N– groups due to the reduction of the electron density at azomethine C–H. The aromatic protons also show some deviation in the complexes as compared to the free ligands since in the complexes they are in direct conjugation to the coordinated O and N atoms of the hydrazone ligands.

IR spectra of the free hydrazone ligands show bands at 3218–3231 cm^{-1} for ν(N–*H*), 1645–1647 cm^{-1} for ν(C=O), and 3439–3447 cm^{-1} for ν(O–*H*) [22]. The ν(C=O) bands are absent in the spectra of the complexes as the ligands bind in the binegative mode losing protons from the carbohydrazide groups. The strong peaks at 1605–1607 cm^{-1} for hydrazones and 1599–1600 cm^{-1} for the complexes can be assigned to ν(C=N) [6]. The complexes exhibit characteristic bands at 973 cm^{-1} for **1** and 971 cm^{-1} for **2** for the stretching of V=O groups [23].

Crystal structure description of the complexes. The perspective views of complexes **1** and **2** together with the atom numbering schemes are shown in Figs. 1 and 2, respectively. The coordination geometry around each V atom reveals a distorted octahedral environment with an NO_5 chromophore. The ligand molecule behaves as binegative tridentate one binding through the phenolate oxygen, the enolate oxygen, and the imine nitrogen atoms and occupies three positions in the basal plane. The fourth donor of the basal plane is furnished by the hydroxyl O atom of the acetohydroxamate ligand. The oxo group and the carbonyl O atom of the acetohydroxamate ligand are located at the axial positions. The V atoms are found to deviate from the corresponding mean basal planes by 0.279(2) Å for **1** and 0.284(2) Å for **2**. The C(8)–O(2) bond lengths are closer to a single bond length rather than the C–O double bond length. However, the shorter length compared to the C–O single bond may be attributed to extended electron delocalization in the ligand. Similarly, the shortening of C(8)–N(2) bond lengths together with the elongation of N(1)–N(2) lengths also supports the electron cloud delocalization in the ligand system. The ligand molecules form five- and six-membered chelate rings with the V centers. All coordinate bond lengths are comparable to those observed in vanadium complexes with hydrazone ligands [24, 25]. The crystal structures of the complexes are stabilized by hydrogen bonds (Figs. 3 and 4).

Catalytic property. The complexes show the effective catalytic property in the oxidation of various olefins to their corresponding epoxides. The details of the catalytic properties with respect to epoxidation of olefins with the complexes as catalysts are given in Table 4. Excellent epoxide yields and selectivity were observed for all aliphatic and aromatic substrates.

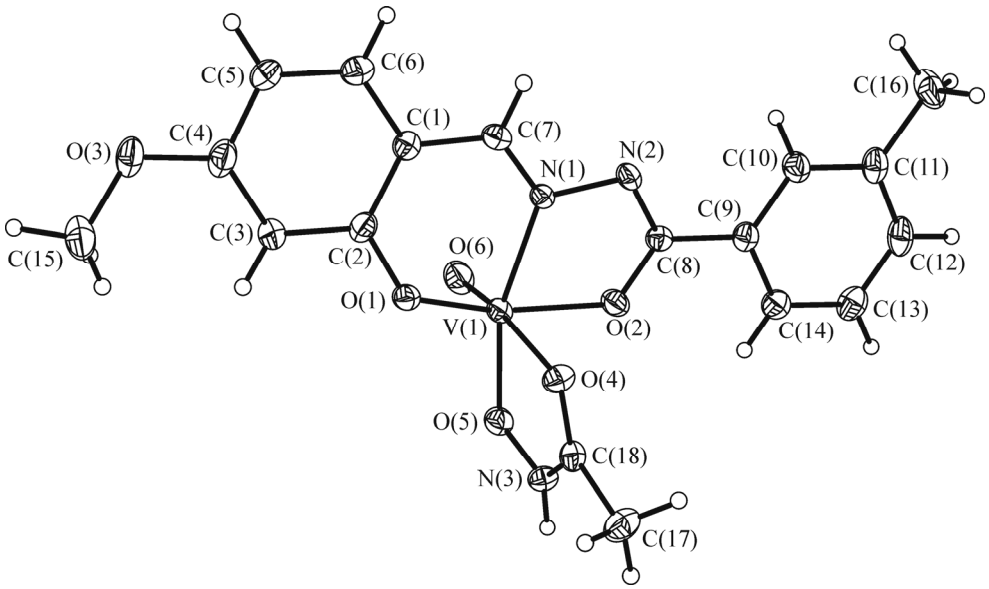


Fig. 1. ORTEP diagram of complex **1** with the atom labeling scheme and 30% probability thermal ellipsoids for all non-hydrogen atoms.

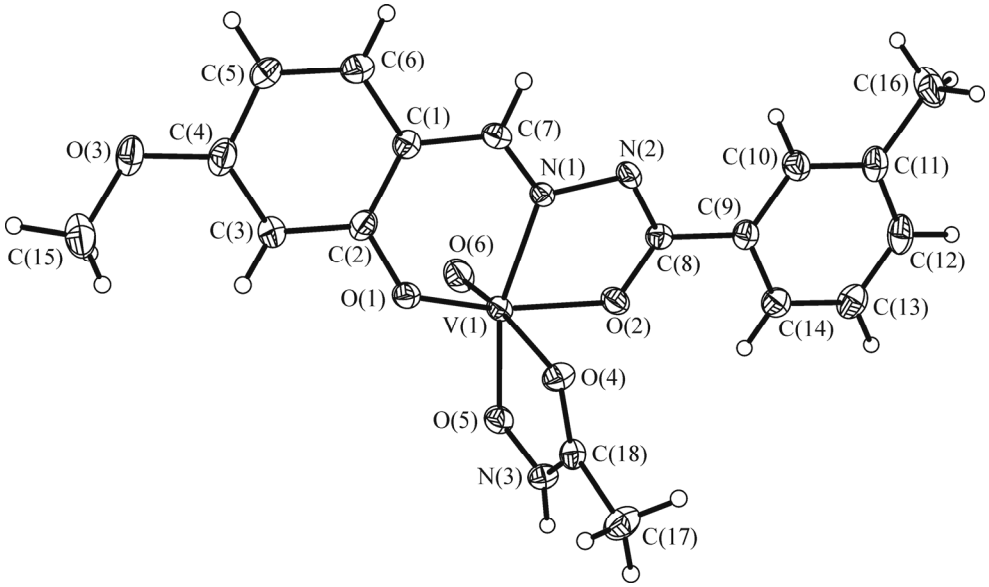


Fig. 2. ORTEP diagram of complex **2** with the atom labeling scheme and 30% probability thermal ellipsoids for all non-hydrogen atoms.

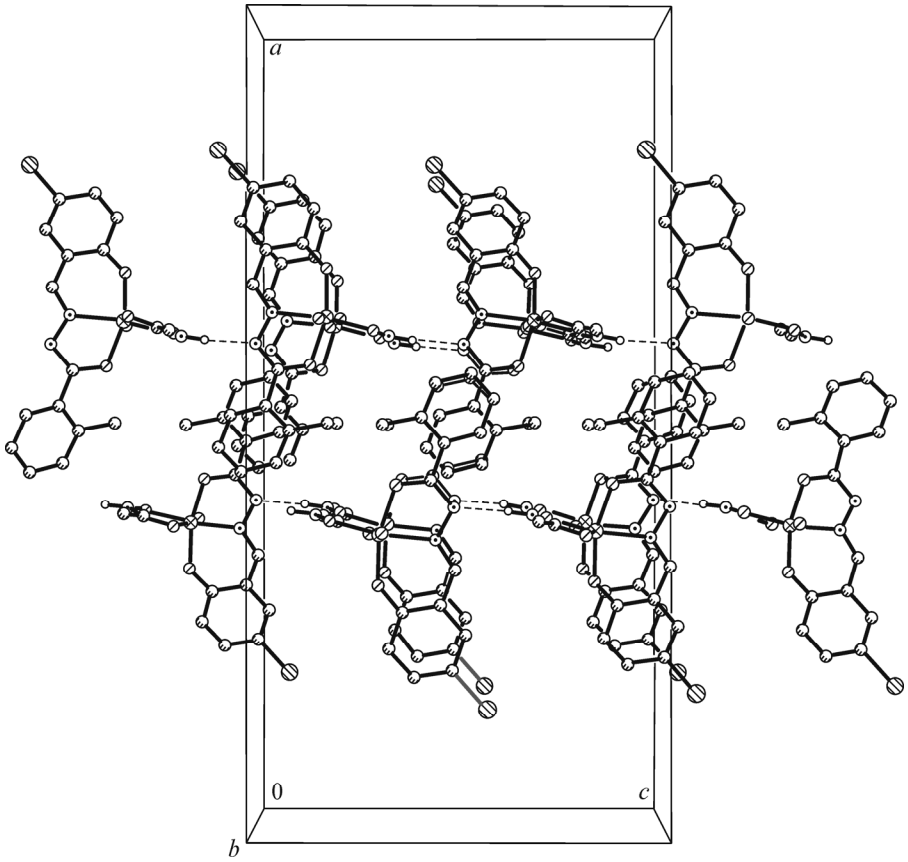


Fig. 3. Hydrogen bonded structure of complex **1**.

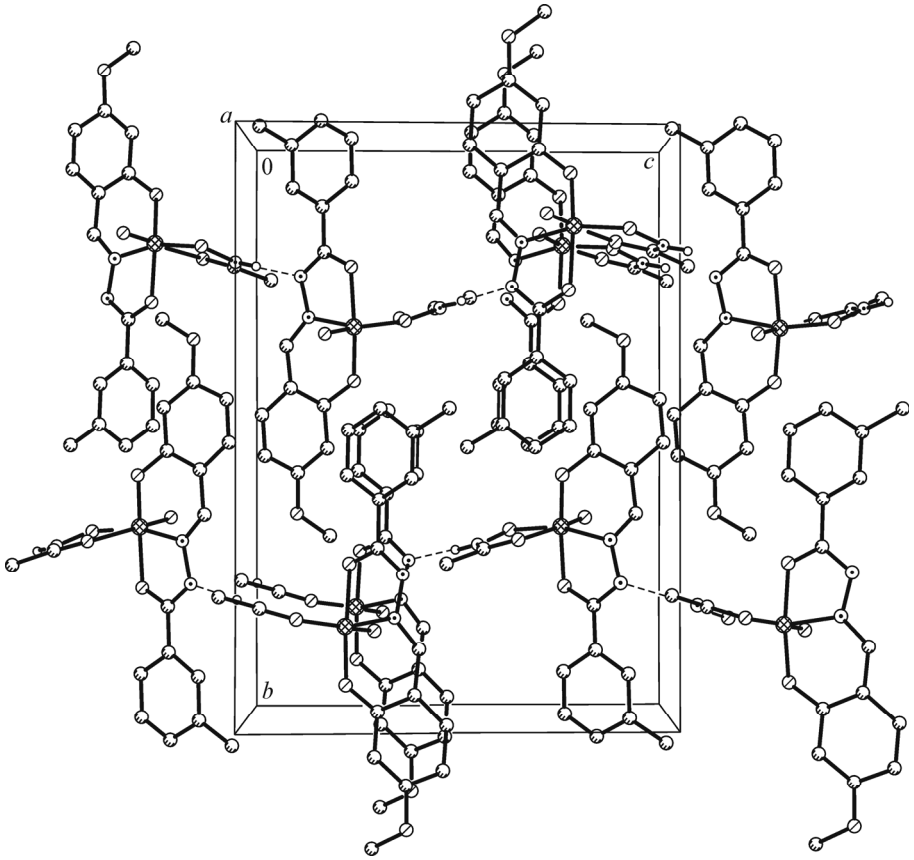
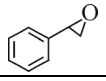
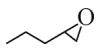


Fig. 4. Hydrogen bonded structure of complex **2**.

TABLE 4. Catalytic Oxidation Results [a]

Substrate	Product	Complex	Conversion (%)[b] (TON)[c]
		**1** **2**	100 (352) 100 (341)
		**1** **2**	100 (330) 100 (325)
		**1** **2**	97 (302) 93 (311)
		**1** **2**	95 (287) 97 (291)

[a] The molar ratios for catalyst : substrate : $NaHCO_3$: H_2O_2 are 1:298:117:1170. The reactions were performed in a (70:30) mixture of CH_3OH/CH_2Cl_2 (1.2 mL) in air at room temperature. The pH of the medium is 8.4.
[b] The GC conversion (%) is measured relative to starting olefin after 75 min.
[c] TON = (mmol of product)/mmol of catalyst.

The results of the catalytic studies using the catalysts reveal that the catalyst efficiency toward all the substrates is similar with the maximum conversion, TON, and selectivity. When H_2O_2 was used as a sole oxidant the catalytic efficiency is not high, but when $NaHCO_3$ was added as a co-catalyst the efficiency of the system increased many times. The key aspect of such a reaction is that H_2O_2 and hydrogen carbonate react in an equilibrium process to produce peroxymonocarbonate, HCO_4^-, which is a more reactive nucleophile than H_2O_2 and speeds up the epoxidation reaction.

CONCLUSIONS

Two new oxidovanadium(V) complexes with hydrazone ligands have been prepared and structurally characterized using the X-ray structure analysis, FT–IR and ^{1}H NMR spectra. The complexes have the octahedral geometry with positions around the central atom being occupied with donor atoms of the hydrazone ligand, the acetohydroxamate ligand, and one oxo group. The complexes show the effective catalytic property in the oxidation of various olefins to their corresponding epoxides.

ADDITIONAL INFORMATION

Supplementary material. CCDC reference numbers 1859442 and 1859446 contain the supplementary crystallographic data for this article. These data can be obtained free of charge at http://www.ccdc.cam.ac.uk, or from Cambridge Crystallographic Data Center, 12 Union Road, Cambridge CB2 1EZ, UK; Fax: +44 1223 336 033; Email: deposit@ccdc.cam.ac.uk.

CONFLICT OF INTERESTS

The authors declare that they have no conflict of interests.

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