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Scientific paper

Synthesis, Crystal Structures and Catalytic Oxidation Property of Two Oxidovanadium(V) Complexes with Schiff bases

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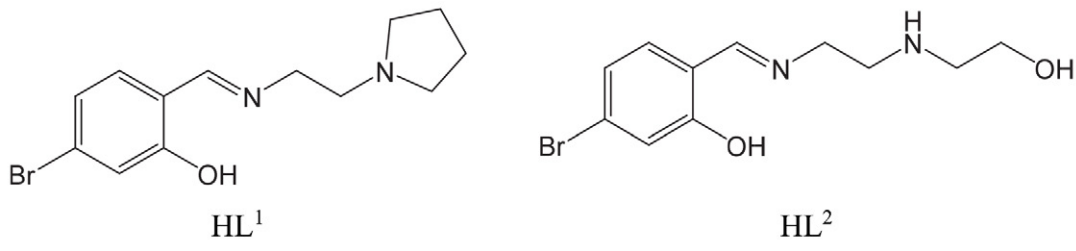
Abstract

Two new oxidovanadium(V) complexes, $[VO_2L^1]$ (**1**) and $[V_2O_2(\mu\text{-}O)_2L^2)]$ (**2**), where L^1 and L^2 are the deprotonated form of 5-bromo-2-(((2-(pyrrolidin-1-yl)ethyl)imino)methyl)phenol (HL^1) and 5-bromo-2-(((2-((2-hydroxyethyl) amino)ethyl)imino)methyl)phenol (HL^2), respectively, have been synthesized and structurally characterized by physico-chemical methods and single crystal X-ray determination. X-ray analysis indicates that the V atom in complex **1** is in square pyramidal coordination, and those in complex **2** are in octahedral coordination. Crystal structures of the complexes are stabilized by hydrogen bonds. The catalytic property for epoxidation of styrene by the complexes was evaluated.

Keywords: Vanadium complex, Schiff base, crystal structure, catalytic property

1. Introduction

Schiff bases bearing typical –CH=N– group represent one of the most attractive series of ligands in coordination chemistry.[1] The compounds can coordinate to various transition and rare earth metal atoms through the imino nitrogen, and/or other oxygen and nitrogen atoms, to form complexes with versatile structures and properties like antibacterial, enzyme inhibition, magnetism, catalytic and photoluminescence.[2] In the past years, a number of complexes with Schiff base ligands have been reported to have fascinating catalytic properties, such as oxidation of sulfides, polymerization and asymmetric epoxidation.[3] Among the complexes those with V centers are of particular interest for their catalytic applications.[4] There are a number of mononuclear and dinuclear vanadium complexes with Schiff base ligands have been reported.[5] However, there is no reasonable explanation on what kind of structures can be obtained. In pursuit of new vanadium



Scheme 1. The Schiff bases HL^1 and HL^2.

complexes with Schiff base ligands, and investigating the influence on the formation of mononuclear and dinuclear species, we report herein two new oxidovanadium(V) complexes, $[VO_2L^1]$ (**1**) and $[V_2O_2(\mu\text{-}O)_2L^2)]$ (**2**), where L^1 and L^2 are the deprotonated form of 5-bromo-2-(((2-(pyrrolidin-1-yl)ethyl)imino)methyl)phenol (HL^1) and 5-bromo-2-(((2-((2-hydroxyethyl)amino)ethyl)imino)methyl)phenol (HL^2), respectively (Scheme 1).

2. Experimental

2. 1. Materials

4-Bromosalicylaldehyde, *N*-(2-aminoethyl)pyrrolidine, *N*-hydroxyethyl-1,2-ethylenediamine and $VO(acac)_2$ were purchased from Aldrich. All other reagents with AR grade were used as received. The Schiff bases HL^1 and HL^2 were prepared by reaction of equimolar quantities of 4-bromosalicylaldehyde with *N*-(2-aminoethyl)pyrrolidine and *N*-hydroxyethyl-1,2-ethylenediamine, respectively in methanol according to the literature method.[6]

2. 2. Physical Measurements

Infrared spectra (4000–400 cm^{-1}) were recorded as KBr discs with a FTS-40 BioRad FT-IR spectrophotometer. The electronic spectra were recorded on a Lambda 35 spectrometer. Microanalyses for C, H and N of the complexes were carried out on a Carlo-Erba 1106 elemental analyzer. Solution electrical conductivity was measured at 298K using a DDS-11 conductivity meter. GC analyses were performed on a Shimadzu GC-2010 gas chromatograph.

2. 3. X-ray Crystallography

Crystallographic data of the complexes were collected on a Bruker SMART CCD area diffractometer with graphite monochromated Mo-Kα radiation (λ = 0.71073 Å) at 298(2) K. Absorption corrections were applied by using the multi-scan program.[7] Structures of the complexes were solved by direct methods and successive Fourier difference syntheses, and anisotropic thermal parameters for all nonhydrogen atoms were refined by full-matrix least-squares procedure against F^2.[8] All non-hydrogen atoms were refined anisotropically. The amino and hydroxyl H atoms of complex **2** were located from a difference Fourier map and refined isotropically, with N–H and O–H distances restrained to 0.90(1) and 0.85(1) Å, respectively. The remaining hydrogen atoms were located at calculated positions, and refined isotropically with U_{iso}(H) values constrained to 1.2 U_{iso}(C). The C11 and C12 atoms in complex **1** are slightly disordered and were refined isotropically. The crystallographic data and experimental details for the structural analysis are summarized in Table 1.

Table 1. Crystallographic data for the single crystal of the complexes

	1	**2**
Empirical formula	$C_{13}H_{16}BrN_2O_3V$	$C_{22}H_{28}Br_2N_4O_8V_2$
Formula weight	379.13	738.18
Temperature (K)	298(2)	298(2)
Crystal system	Monoclinic	Triclinic
Space group	$P2_1/c$	P–1
a (Å)	13.2544(16)	6.8311(6)
b (Å)	10.5573(14)	11.6180(10)
c (Å)	10.7724(14)	17.2478(13)
α (°)	90	76.8550(10)
β (°)	102.5040(10)	79.1930(10)
γ (°)	90	89.7430(10)
V (Å^3)	1471.6(3)	1308.30(19)
Z	4	2
F(000)	760	736
μ, mm^{-1}	3.397	3.823
R_{int}	0.0350	0.0540
Collected data	7690	14314
Unique data	2741	4864
Observed data [$I > 2\sigma(I)$]	2045	3680
Restraints	12	4
Parameters	182	355
Goodness-of-fit on F^2	1.050	1.011
R_1, wR_2 indices [$I > 2\sigma(I)$]	0.0563, 0.1643	0.0421, 0.1145
R_1, wR_2 indices (all data)	0.0767, 0.1816	0.0627, 0.1267

2. 4. Synthesis of $[VO_2L^1]$ (1)

HL^1 (1.0 mmol, 0.30 g) and $[VO(acac)_2]$ (1.0 mmol, 0.26 g) were mixed and stirred in methanol (50 mL) for 30 min at 25 °C. The brown solution was evaporated to remove three quarters of the solvents under reduced pressure, yielding deep brown solid of the complex. Yield: 0.22 g (58%). Well-shaped single crystals suitable for X-ray diffraction were obtained by re-crystallization of the solid from methanol. Analysis calculated for $C_{13}H_{16}BrN_2O_3V$: C, 41.18; H, 4.25; N, 7.39%; found: C, 41.07; H, 4.32; N, 7.30%. IR data (KBr, cm^{-1}): 1630 (vs, $\nu_{C=N}$), 943 and 927 (m, $\nu_{V=O}$). UV-Vis data (λ_{max}, nm): 228, 366.

2. 5. Synthesis of $[V_2O_2(\mu\text{-}O)_2L^2)]$ (2)

HL^2 (1.0 mmol, 0.29 g) and $[VO(acac)_2]$ (1.0 mmol, 0.26 g) were mixed and stirred in methanol (50 mL) for 30 min at 25 °C. The brown solution was evaporated to remove three quarters of the solvents under reduced pressure, yielding deep brown solid of the complex. Yield: 0.25 g (68%). Well-shaped single crystals suitable for X-ray diffraction were obtained by re-crystallization of the solid from methanol. Analysis calculated for $C_{22}H_{28}Br_2N_4O_8V_2$:

C, 35.80; H, 3.82; N, 7.59%; found: C, 35.63; H, 3.76; N, 7.51%. IR data (KBr, cm^{-1}): 3372 (w, ν_{OH}), 3206 (w, ν_{NH}), 1632 (vs, $\nu_{C=N}$), 935 and 967 (m, $\nu_{V=O}$). UV-Vis data (λ_{max}, nm): 225, 260, 363.

2. 6. Styrene Epoxidation

The epoxidation reaction was carried out at room temperature in acetonitrile under N_2 atmosphere with constant stirring. The composition of the reaction mixture was 2.00 mmol of styrene, 2.00 mmol of chlorobenzene (internal standard), 0.10 mmol of the complexes (catalyst) and 2.00 mmol iodosylbenzene or sodium hypochlorite (oxidant) in 5.00 mL freshly distilled acetonitrile. When the oxidant was sodium hypochlorite, the solution was buffered to pH 11.2 with NaH_2PO_4 and NaOH. The composition of reaction medium was determined by GC with styrene and styrene epoxide quantified by the internal standard method (chlorobenzene). All other products detected by GC were mentioned as others. The reaction time for maximum epoxide yield was determined by withdrawing periodically 0.1 mL aliquots from the reaction mixture. This time was used to monitor the efficiency of the catalyst on performing at least two independent experiments. Blank experiments with each oxidant and using the same experimental conditions except catalyst were also performed.

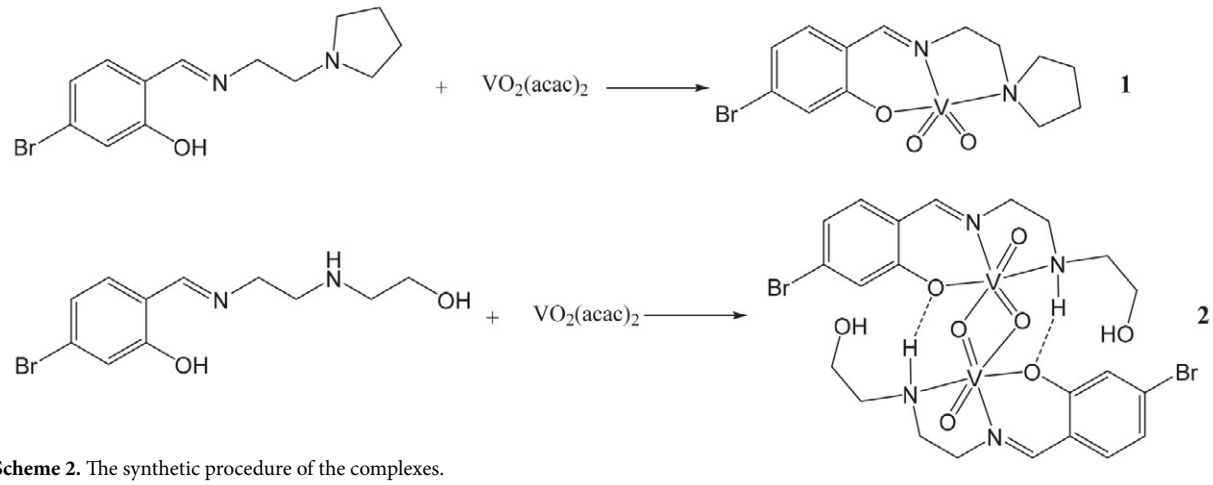
3. Results and Discussion

3. 1. Chemistry

Complexes **1** and **2** were prepared by reaction of $VO(acac)_2$ with HL^1 and HL^2, respectively in methanol (Scheme 2). Complex **1** is a mononuclear vanadium compound, while complex **2** is an oxido bridged dinuclear compound. There are two N–H···O hydrogen bonds between the two $[VO_2L^2]$ units in complex **2**, which make the two molecules close to each other. However, in the mononuclear complex **1**, there is no such hydrogen bond. Thus, the formation of mononuclear or dinuclear vanadium complexes depends on whether there are additional interactions like hydrogen bonding between the molecules. The Schiff bases were deprotonated during the coordination. The oxidation of V(IV) in $VO(acac)_2$ to V(V) in both complexes during the reaction in air is not uncommon.[9] The molar conductivities are 35 Ω^{-1} cm^2 mol^{-1} (**1**) and 30 Ω^{-1} cm^2 mol^{-1} (**2**), which are consistent with the values expected for non-electrolyte.[10]

3. 2. Crystal Structure Description of Complex 1

Selected bond lengths and angles for the complex are listed in Table 2. Single crystal X-ray analysis indicates that the complex is a mononuclear oxidovanadium(V) compound. The ORTEP plot of the complex is shown in Fig. 1. The V atom in the complex is in square pyramidal geometry, with the phenolate oxygen (O1), imino nitrogen (N1) and pyrrolidine nitrogen (N2) of the Schiff base ligand and one oxo oxygen (O2) defining the basal plane, and with the other oxo oxygen (O3) locating at the apical position. The V atom displaced towards the apical oxygen by 0.471(1) Å. The geometry can be evidenced by the τ value of 0.44, which indicates it a distorted square pyramidal coordination.[11] The distortion of the coordination can also be observed from the bond angles related to the V atom. The *cis*- and *trans*- angles at the basal plane are 76.75(16)–115.25(18)° and 132.94(19)–159.31(15)°. The deviation from the ideal square pyramidal geometry are mainly origin from the strain created by the five-membered chelate ring V1–N1–C8–C9–N2 and the repulsive force between the two oxo oxygen. The bond lengths of V1–O1 of 1.910(4) Å, V1–N1 of 2.137(4) Å and V1–N2 of 2.170(4) Å are comparable to those observed in Schiff base oxidovanadium(V) complexes.[12] The terminal V1–O2 and V1–O3 are 1.621(4) and 1.615(4) Å, which agree well with the corresponding values reported for related systems.[9]



Scheme 2. The synthetic procedure of the complexes.

In the crystal structure of the complex, the molecules are linked by C13–H13B···O3 hydrogen bonds (Table 3), to form one-dimensional chains running along the *c* axis. The chains are further linked through C3–H3···O2 hydrogen bonds at the *b* axis, generate two-dimensional network (Fig. 2).

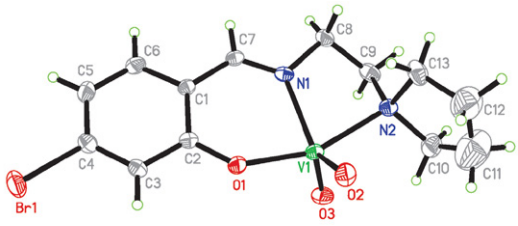


Fig. 1. ORTEP diagram of complex **1** with 30% thermal ellipsoid.

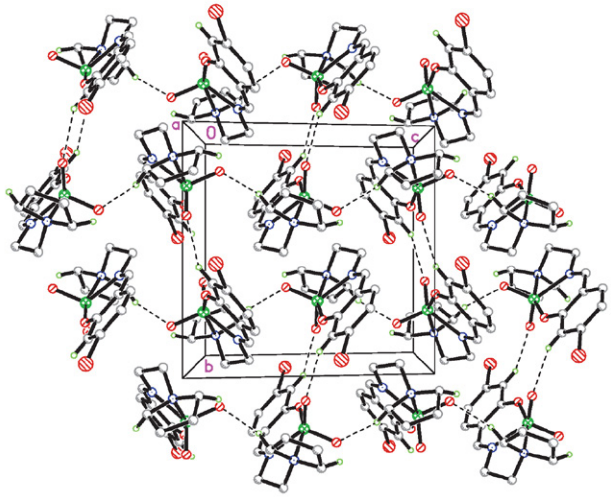


Fig. 2. Molecular packing structure of complex **1** linked by hydrogen bonds (dashed lines).

3. 3. Crystal Structure Description of Complex 2

Selected bond lengths and angles for the complex are listed in Table 2. Single crystal X-ray analysis indicates that the complex is an oxido bridged dinuclear oxidovanadium(V) compound. The molecule possesses crystallographic inversion center symmetry. The ORTEP plot of the complex is shown in Fig. 3. The V atoms in the complex are in octahedral geometry, with the phenolate oxygen (O1), imino nitrogen (N1) and pyrrolidine nitrogen (N2) of the Schiff base ligand and one bridging oxo oxygen (O2) defining the equatorial plane, and with one terminal oxo oxygen (O3) and the other bridging oxo oxygen (O2A) locating at the axial positions. The V atom displaced towards the axial oxygen (O3) by 0.312(1) Å. The distortion of the coordination can be observed from the bond angles related to the V atom. The *cis*- and *trans*- angles at the equatorial plane are 75.86(13)–106.68(14)° and 155.19(13)–173.35(12)°, respectively. The deviation from the ideal octahedral geometry is mainly caused by the five-membered chelate ring V1-N1-C8-C9-N2 and the repulsive force between the two oxo oxygen. The bond lengths of V1–O1 of 1.918(3) Å, V1–N1 of 2.149(3) Å and V1–N2 of 2.182(3) Å are similar to complex **1**, and comparable to those observed in Schiff base oxidovanadium(V) complexes.[13] The bond lengths agree well with the corresponding values reported for related systems.[12,13]

In the crystal structure of the complex, the molecules are linked by N2–H2···O1, O4–H4···O8, N4–H4A···O5, O8–H8···O2, C19–H19A···O6 and C20–H20B···O4 hydrogen bonds, to form two-dimensional network (Fig. 4).

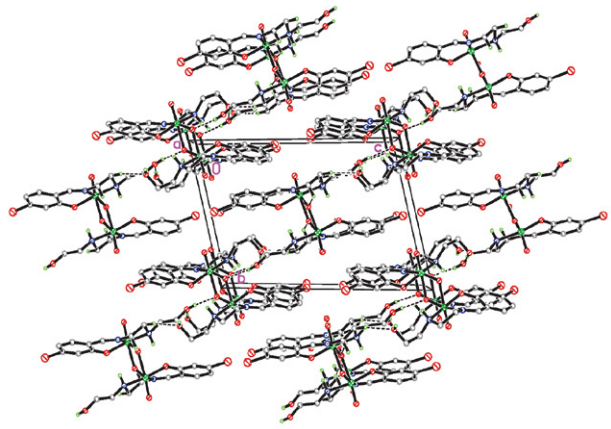


Fig. 4. Molecular packing structure of complex **2** linked by hydrogen bonds (dashed lines).

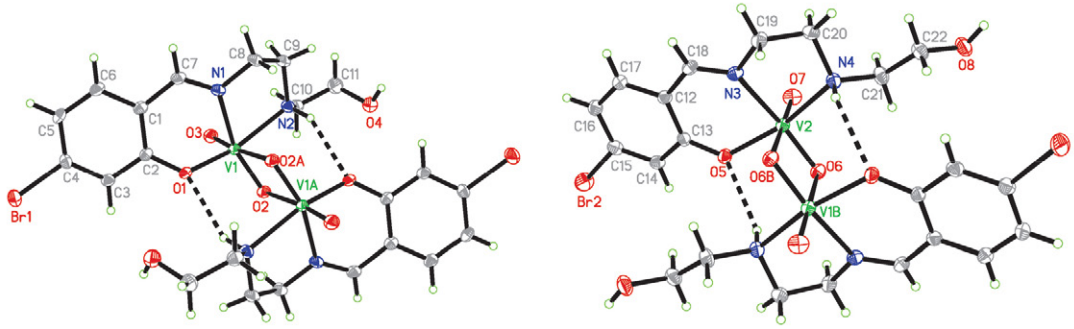


Fig. 3. ORTEP diagram of complex **2** with 30% thermal ellipsoid. Atoms labeled with the suffix A and B are related to the symmetry operations −*x*, −*y*, −*z*, and −*x*, 1 − *y*, 1 − *z*.

Table 2. Selected bond distances (Å) and bond angles (°) for the complexes

1			
V1–O1	1.910(4)	V1–O2	1.621(4)
V1–O3	1.614(4)	V1–N1	2.137(4)
V1–N2	2.169(4)		
O3–V1–O2	110.5(2)	O3–V1–O1	101.28(19)
O2–V1–O1	98.20(18)	O3–V1–N1	115.25(18)
O2–V1–N1	132.95(19)	O1–V1–N1	83.41(15)
O3–V1–N2	92.52(18)	O2–V1–N2	91.16(19)
O1–V1–N2	159.32(15)	N1–V1–N2	76.75(16)
2			
V1–O1	1.918(3)	V1–O2	1.692(3)
V1–O3	1.600(3)	V1–N1	2.149(3)
V1–N2	2.182(3)	V1–O2A	2.328(3)
V2–O5	1.925(3)	V2–O6	1.660(3)
V2–O7	1.615(3)	V2–N3	2.161(3)
V2–N4	2.163(3)	V2–O6A	2.365(3)
O3–V1–O2	106.68(14)	O3–V1–O1	98.29(14)
O2–V1–O1	102.97(13)	O3–V1–N1	95.99(14)
O2–V1–N1	155.19(13)	O1–V1–N1	83.14(12)
O3–V1–N2	94.18(14)	O2–V1–N2	92.26(13)
O1–V1–N2	156.56(13)	N1–V1–N2	75.86(13)
O3–V1–O2A	173.35(12)	O2–V1–O2A	79.22(12)
O1–V1–O2A	83.09(11)	N1–V1–O2A	77.68(11)
N2–V1–O2A	82.38(12)	O7–V2–O6	107.54(15)
O7–V2–O5	101.54(14)	O6–V2–O5	98.45(13)
O7–V2–N3	98.71(15)	O6–V2–N3	152.54(14)
O5–V2–N3	83.76(13)	O7–V2–N4	92.25(15)
O6–V2–N4	93.52(13)	O5–V2–N4	158.02(13)
N3–V2–N4	77.23(13)	O7–V2–O6B	170.70(13)
O6–V2–O6B	77.58(13)	O5–V2–O6B	85.08(12)
N3–V2–O6B	75.34(11)	N4–V2–O6B	79.55(12)

Symmetry operations: A: –*x*, –*y*, –*z*; B: –*x*, 1 – *y*, 1 – *z*.

Table 3. Hydrogen bond distances (Å) and bond angles (°) for the complexes

D*–H···*A **(*D*–H···*A*)**	***d*(*D*–H)**	***d*(H···*A*)**	***d*(*D*···*A*)**	**Angle**
1				
C3–H3···O2$^{\#1}$	0.93	2.49(3)	3.338(5)	151(5)
C13–H13B···O3$^{\#2}$	0.97	2.38(3)	3.314(5)	162(5)
2				
N2–H2···O1$^{\#3}$	0.90(1)	2.34(4)	3.137(4)	148(5)
O4–H4···O8$^{\#4}$	0.85(1)	1.88(1)	2.735(5)	178(7)
N4–H4A···O5$^{\#5}$	0.90(1)	2.38(5)	3.073(5)	134(5)
O8–H8···O2$^{\#6}$	0.85(1)	2.17(1)	3.016(4)	172(6)
C19–H19A···O6$^{\#7}$	0.97	2.54(2)	3.115(4)	118(5)
C20–H20B···O4$^{\#6}$	0.97	2.48(2)	3.442(4)	171(5)

Symmetry codes: #1: 1 – *x*, –*y*, 1 – *z*; #2: *x*, ½ – *y*, –½ + *z*; #3: –*x*, –*y*, –*z*; #4: 1 + *x*, *y*, –1 + *z*; #5: –*x*, 1 – *y*, 1–*z*; #6: *x*, *y*, 1 + *z*; #7: 1 + *x*, *y*, *z*.

3. 4. IR and UV-vis Spectra of the Complexes

The weak absorption at 3372 cm^{-1} and the sharp absorption at 3206 cm^{-1} of complex **2** are attributed to the stretching vibrations of O-H and N-H bonds, respectively. The intense bands at 1630 cm^{-1} for **1** and 1632 cm^{-1} for **2** are assigned to the vibrations of the azomethine groups, ν(C=N).[14] The characteristic of the spectra of both complexes is the exhibition of bands at 943 and 927 cm^{-1} for **1**, and 935 and 967 cm^{-1} for **2**, corresponding to the V=O stretching vibrations.[15] This agrees well with the bond lengths of C=N bonds in the crystal structures determined by single crystal X-ray diffraction. The two bands are close to each other in the spectrum of **1**, while those in complex **2** are not too close. The weak bands in the range 400–650 cm^{-1} are assigned to the vibrations of the V–O and V–N bonds.

In the UV-Vis spectra of the complexes, the bands at 360–370 nm are attributed to the azomethine chromophore π-π* transitions. The bands at higher energy (220–260 nm) are associated with the benzene π-π* transitions.[16]

3. 5. Catalytic Properties of the Complexes

The percentage of conversion of styrene, selectivity for styrene oxide, yield of styrene oxide and reaction time to obtain maximum yield using both the oxidants are given in Table 4. The data reveals that the complexes as catalysts convert styrene most efficiently in the presence of both oxidants. Nevertheless, the catalysts are selective towards the formation of styrene epoxides despite the formation of by-products which have been identified by GC-MS as benzaldehyde, phenylacetaldehyde, styrene epoxides derivative, alcohols *etc.* From the data it is also clear that the complexes exhibit excellent efficiency for styrene epoxide yield. When the reactions are carried out with PhIO and NaOCl, styrene conversions of complexes **1** and **2** were about 88% and 84%, and 83% and 80%, respectively. It is evident that between PhIO and NaOCl, the former acts as a better oxidant with respect to both styrene conversion and styrene epoxide selectivity. The epoxide yields for the complexes **1** and **2** using PhIO and NaOCl as oxidants are 87% and 83%, and 79% and 75%, respectively.

Table 4. Catalytic epoxidation results of complexes **1** and **2***

	1	**1**	**2**	**2**
Oxidant	PhIO	NaOCl	PhIO	NaOCl
Conversion (%)	88	84	83	80
Epoxide yield (%)	87	83	79	75
Selectivity (%)	97	95	89	91

* The time is 2 h for PhIO, and 3 h for NaOCl.

4. Conclusion

A new mononuclear oxidovanadium(V) complex and a new dinuclear oxidovanadium(V) complex derived

from tridentate Schiff bases have been synthesized and characterized. Single crystal X-ray analysis indicates that the V atoms in the mononuclear and dinuclear complexes are in distorted square pyramidal and octahedral coordination, respectively. The complexes have effective catalytic property for the epoxidation of styrene, with conversions over 80% and selectivity over 89%.

Supplementary Material

CCDC 2334633 for **1** and 2334634 for **2** contain the supplementary crystallographic data for this paper. These data can be obtained free of charge *via* http://www.ccdc.cam.ac.uk/conts/retrieving.html, or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: (+44) 1223-336-033; or e-mail: deposit@ccdc.cam.ac.uk.

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Povzetek

Sintetizirali smo dva nova oksidovanadijeva(V) kompleksa, $[VO_2L^1]$ (**1**) in $[V_2O_2(\mu\text{-}O)_2L^2)]$ (**2**), kjer sta L^1 in L^2 deprotonirani obliki 5-bromo-2-(((2-(pirolidin-1-il)etil)imino)metil)fenola (HL^1) in 5-bromo-2-(((2-((2-hidroksietil)amino) etil)imino)metil)fenola (HL^2), ter ju okarakterizirali s fizikalno-kemijskimi metodami in rentgensko monokristalno analizo. Rentgenska analiza kaže, da je atom vanadija v kompleksu **1** v kvadratno piramidalni koordinaciji, v kompleksu **2** pa v oktaedrični koordinaciji. Kristalni strukturi kompleksov sta stabilizirani z vodikovimi vezmi. Določili smo tudi katalitične lastnosti kompleksov za epoksidacijo stirena.

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