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# Epoxidation of alkenes by an oxidovanadium(IV) tetradentate Schiff base complex as an efficient catalyst with *tert*-butyl hydroperoxide

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#### Abstract

A new asymmetrical tetradentate  $N_2O_2$  Schiff base ligand was synthesized from 2hydroxyacetophenon, 2-hydroxynaphthaldehyde and 1,2 phenylenediimine. The new oxidovanadium(IV) Schiff base complex,  $V^{IV}OL$  (L = N-2-hydroxyacetophenon-N'-2hydroxynaphthaldehyde-1,2 phenylenediimine), was prepared by reaction of Schiff base ligand with vanadyl acetylacetonate. The Schiff base ligand (L) and the oxidovanadium(IV) complex were characterized by spectroscopic methods. The crystal structure of the complex was determined by the single crystal X-ray analysis. The complex crystallized in the orthorhombic system, having one V<sup>4+</sup> ion coordinating in an approximately square pyramidal N<sub>2</sub>O<sub>3</sub> geometry by two azomethine N atoms and phenolic oxygens from Schiff base ligand in a square plane and one oxygen atom in an apical position. Electrochemical properties of the complex were examined by means of cyclic voltammetry. The catalytic activity of the oxidovanadium(IV) Schiff base complex was tested in the epoxidation of cyclooctene.

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#### **Keywords:**

X-Ray crystallography, asymmetry, vanadium Schiff base complexes, electrochemistry, catalyst.

#### 1. Introduction

In the Schiff base compounds which are among the privileged ligands, the carbonyl group of ketone or aldehyde are replaced by an imine group due to simple one-pot condensation synthesis in an alcoholic solvent. For more than a century, metal complexes of the Schiff base ligands have been rigorously studied and used in different fields including biological research [1-6], chemical analysis [7], non-linear optics [8], magneto chemistry [9], photo physical studies [10] and catalysis [11].

In research studies, simple metal complexes related to different oxidants such as alkyl hydroperoxides,  $H_2O_2$  or  $O_2$  are used in order to imitate the metalloenzyme catalyzed oxidation [12-14]. The oxidant is activated by the central metal and transfer the oxygen atom to substrate from metal-oxo species [15-17].

Between transition metal complexes, use of vanadium complexes as oxidative catalysts is of great interest and a number of intriguing -oxo-transfer reactions are derived by oxidovanadium complexes of Schiff base compounds [18, 19]. Furthermore, the molecular oxygen for oxygenation reactions can also be activated by vanadium complexes [20, 21].

In catalytic oxidation, the use of high valent vanadium is encouraged due to (a) the different possible coordination numbers, (b) the easily interchangeable vanadium oxidation states (where +4 and +5 are the most stable ones under aerobic conditions), (c) the Lewis acid character of the vanadium centers and (d) the high affinity of the metal toward oxygen [22-24].

Since epoxides are the key starting materials for a wide variety of products, the epoxidation of alkenes is considered to be one of the most widely studied reactions in organic chemistry. An area of strong research activity is the catalytic epoxidation of olefins by transition metal complexes [25, 26]. In recent years the structural study, synthesis and catalytic activity of oxidovanadium Schiff base complexes in oxidation of different organic substrates are described in a great number of publications [27-29]. The central metal is surrounded by N<sub>2</sub>O<sub>2</sub> coordination environment. In this work, in order to investigate the catalytic activity of oxidovanadium Schiff base complex as catalyst, we synthesized a new asymmetrical tetradentate Schiff base ligand and its metal complex "Scheme 1"characterized by FT-IR, <sup>1</sup>H NMR and UV-Vis spectroscopies. Subsequently, the crystal structure of this new oxidovanadium (IV) Schiff base complex is also determined by X-ray crystallography. We also reported catalytic epoxidation of cyclooctene, cyclohexene, 1-octene, styrene, indene and norbornene in CHCl<sub>3</sub> using *tert*-butyl hydroperoxide as oxidant. Electrochemical behavior of V<sup>IV</sup>O complex also have been investigated. The oxidation potential of V<sup>IV</sup>O complex was measured by cyclic voltammetry.

#### 2. Experimental

#### 2.1 Chemicals and apparatus

All chemicals and solvents were purchased from commercial sources without any further purification. Fourier Transform Infrared (FTIR) spectra of the synthesized samples were recorded on a FT-IR JASCO 680 spectrophotometer in the range of 4000-400 cm<sup>-1</sup> with potassium bromide pellets. The UV-Vis spectra were obtained by a UV-Vis JASCO model V-570 spectrophotometer in the 800-200 nm range. The <sup>1</sup>HNMR spectrum was recorded in DMSO-

*d6* on Bruker-500 MHz and elemental analysis was performed using a CHN-O-Heraeus elemental analyzer.

The electrochemical experiments were carried out using a potentiostat/galvanostat Autolab Model PGSTAT-302N and NOVA 1.7 software coupled with a Pentium (IV) personal computer. A glassy carbon electrode was used as a working electrode (metrohm glassy carbon, 0.0314 cm<sup>2</sup>). An Ag/AgCl/KCl 3.0M and platinum wire were used as reference and counter respectively. Voltammetric measurements were performed at room temperature in DMF solution with 0.1 M tetrabutylammonium perchlorate as the supporting electrolyte. The X-ray single crystal structure analysis was obtained by using Bruker SMART APEX II-2009 CCD area detector diffractometer. Determination of products from the catalytic testing was carried out using gas chromatographic (GC) technique. Reaction mixture was analyzed by 6890N Technologies from Agilent Company with FID detector and N<sub>2</sub> as the carrier gas. Column chromatography with 28.5 m length, 320  $\mu$ m inner diameter and 0.25  $\mu$ m thickness (the HP+5 types) was used.

#### 2.2 Procedures for synthesis of the tridentate Schiff base ligand

The tridentate Schiff base ligand used in this research was prepared as previously reported [30] by condensation between 2-Hydroxyacetophenone and 1, 2-phenylenediamine (1:1mmol ratio) in 20 mL methanol as a solvent. The solution was refluxed for 3h and then the solvent was evaporated under the reduced pressure. Water was added to the precipitate for dissolving phenylenediamine. The resulting yellow solid was collected and washed with water and then recrystallized from methanol.

#### 2.3. General procedure for Synthesis of the unsymmetrical tetradentate Schiff base ligand

2-hydroxynaphthaldehyde 1 mmol was added to a solution of tridentate Schiff base ligand 1 mmol disolved in 10 mL methanol. The solution was stirred for 3h. The resulting yellow precipitate of Schiff base ligand was separated by vacuum filtration, washed with cold methanol and dried in vacuum desiccator.

Anal, calc. for  $C_{25}H_{20}N_2O_2$ : C, 78.94; H, 5.26; N, 7.36%. Found; C, 78.12; H, 5.02; N, 7.29%. FT-IR (KBr cm<sup>-1</sup>)  $\nu_{max}$ : 1617 (C=N), 1482 (C=C), 1192 (C-O). <sup>1</sup>H NMR (DMSO.*d*6,  $\delta$ , ppm): 2.3 (s, 3H, CH<sub>3</sub>), 6.8-8.5 (m, 14H, Ar-H), 9.7 (s, 1H, HC=N), 14.1 (s, 1H, OH) and 15.3 (s, 1H, OH). UV-Vis,  $\lambda_{max}$  (nm) ( $\epsilon$ ,M<sup>-1</sup>cm<sup>-1</sup>) (Ethanol): 474 (8466), 454 (11333), 390 (12666), 338 (19333), 264 (45333), 232 (73333).

#### 2.4. Synthesis of the vanadyl Schiff base complex

The methanolic solution (10 mL) of tetradentate Schiff base ligand (1mmol) was added to 10mL of a methanolic solution of VO(acac)<sub>2</sub> (1mmol). The yellow color of solution turned to dark green immediately and the reaction mixture was stirred and refluxed for 1h. As a result, a green precipitate was formed which after filtration and washing with methanol recrystallized from ether and dichloromethane.

Anal, calc. for  $C_{25}H_{18}N_2VO_3$ : C, 67.42; H, 4.04; N, 6.29%. Found; C, 64.96; H, 3.90; N, 6.01%. FT-IR (KBr cm<sup>-1</sup>)  $\nu_{max}$ : 1617, 1599 (C=N), 1529 (C=C), 1202 (C-O), 978 (V=O). UV-Vis,  $\lambda_{max}$  (nm) ( $\epsilon$ ,M<sup>-1</sup>cm<sup>-1</sup>) (Ethanol): 451 (17692), 402(18461), 362 (20000), 316 (18461), 246 (50769), 221 (47692).

#### 2.5. X-Ray Crystallography Analysis of the vanadyl Schiff base complex

By using graphite monochromated Mo-K<sub> $\alpha$ </sub> radiation, the X-ray diffraction measurements were collected on a STOE IPDS-2T diffractometer. For the purpose of collecting data, the black crystal vanadyl complex with a dimension of  $0.16 \times 0.39 \times 0.39$  mm was chosen and mounted on a glass fiber. Using least-squares refinement of diffraction data from 9695 unique reflections, cell constants and an orientation matrix for data collection were gained.

In a series of  $\omega$  scans in 1° oscillations and integrated using the Stoe X-AREA software package, data were obtained at a temperature of 100(2) K to a maximum 20 value of 60.3°. The numerical absorption coefficient ( $\mu$ ) for Mo-K<sub> $\alpha$ </sub> radiation is 0.710 mm<sup>-1</sup>.

numerical absorption correction was applied using X-RED and X-SHAPE softwares [31]. The data were modified for Lorentz and polarization effects. Direct methods were used to solve the structure [32] and different Fourier maps and full-matrix least-squares refinement against  $F^2$ , were performed using anisotropic displacement parameters [33]. All of hydrogen atoms were placed by difference Fourier map and then refined isotropically. Atomic factors were from International Tables for X-ray Crystallography. All purification were performed using the X-STEP32 crystallographic software package [34].

The experimental details, crystal data, and refinement results were summarized in Table 1 for vanadyl complexs. It is notable that, the asymmetric unit also contains solvents molecules, which could not be modeled. Therefore, the diffraction contribution of the solvent molecules was removed by the subroutine SQUEEZE in PLATON.

#### 3. Results and discussion

#### 3.1 FT-IR characterization

Several bands are observed within the FT-IR spectra of free Schiff base ligand and it's complex. The FT-IR spectrum of ligand shows a sharp band in the 1617 cm<sup>-1</sup>, related to the stretching vibration frequency of imine. In the Shiff base complex, coordination of Schiff base ligand to vanadyl ion causes a decrease in the C=N bond order and it's vibration frequency shifts to a lower frequency [35, 36] and is shown in 1599 cm<sup>-1</sup>. The exhibition of the sharp band in the 978 cm<sup>-1</sup>, corresponding to the V=O stretching vibration is the characteristic of the FT-IR spectrum of vanadyl complex. The appearance of this particular band in this region indicates the formation of vanadyl Schiff base complex.

#### 3.2. Electronic spectra

In the sections 2.3 and 2.4, the UV-Vis spectral data in ethanol are listed. All the bands which are lower than 400 nm are related to  $\pi \rightarrow \pi^*$  transition of aromatic ring within the Schiff base ligand at 232-390 nm and the complex at 221-362 nm (Fig 1). The bonds at 454 and the shoulder at 474 belong to the Schiff base ligand are attributed to  $\pi \rightarrow \pi^*$  transition related to azomethine group [30, 37, 38]. It has to be noted that these transitions in the vanadyl complex take place at 451 and 402 nm.

#### 3.3. <sup>1</sup>H NMR spectra

The <sup>1</sup>HNMR data of the asymmetrical tetradentate Schiff base ligand is presented in section 2.3. The single peak at about 2.3 ppm, is related to the hydrogens of methyl. However, in the range of 6.88-8.50 ppm, the aromatic hydrogens are observed. The imine hydrogen is assigned at 9.70 ppm. At 14.12 and 15.35 ppm, the phenolic hydrogens of 2-hydroxyacetophenone and 2-

hydroxy naphthaldehyde are found, respectively. The <sup>1</sup>H NMR techniques confirmed the structure of synthesized ligand as well.

#### 3.4 Description of the molecular structure of the vanadyl complex

The structure of the vanadyl complex with the atom–numbering scheme is presented in Fig 2. The crystallographic data shows that the vanadium (IV) ion, in the five-coordinated geometry is surrounded by two oxygen and two nitrogen atoms from Schiff base ligand and oxo ligand. The geometry around the vanadium(IV) ion is distorted squre-pyramidal, as indicated by the unequal metal-ligand bond distances and angles: i.e. V-N1 = 2.075(3), V-N2 = 2.041(3), V-O1 = 1.916(3), V-O2 = 1.926(2), V-O3 = 1.604(2) Å and O3-V-O1, O3-V-O2, O3-V-N2, O2-V-N1, O1-V-N2, O3-V-N1, O1-V-N1, O2-V-N2, N2-V1-N1, O1-V-O2 angles are distributed in the range of  $111.59(13)^\circ$ ,  $110.83(11)^\circ$ ,  $106.90(13)^\circ$ ,  $146.67(11)^\circ$ ,  $141.25(11)^\circ$ ,  $102.06(11)^\circ$ , 88.65(11)°, 86.29(11)°, 79.06(11)° and 84.31(10)°, respectively. All distances and angles in vanadyl complex are in agreement with the same distances in other vanadium (IV) complexes [39-42]. Crystal data and experimental details are listed in Table 1 and 2. The structure of the noted complex is identified by X-ray diffraction and crystallized in the Orthorhombic, space group Pca2 (1).

#### 3.5 Electrochemical investigation

The cyclic voltammetry of VOL complex is carried out in DMF solution at ambient temperature.

The typical cyclic voltammogram of [VOL] complex in the potential range of 0.0 to 1.0 V (vs Ag/AgCl) is shown in Fig 3. At about +0.50 V, an oxidation peak is observed. In a fully-

reversible one-electron step, [VOL] is oxidized to the mono cation  $[VOL]^+$ . The electron is separated from the nonbonding orbitals and the V(V) complex is created. Upon reversal of the scan direction, the V(V) complex is reduced to V(IV) at lower potentials about 0.45 V. Multiple scans result in nearly superposable cyclic voltammograms. Thereby showing that the five coordinatione geometry is stable in both oxidation states, at least on the cyclic voltammetry time scale. In the vanadyl Schiff base complex redox process is the one-electron transfer reaction. The calculation for the formal potential ( $E_{1/2}$  (IV/V)) for the V(IV/V) redox couple is the average of the cathodic ( $E_{pc}$ ) and anodic peak ( $E_{pa}$ ) which is equal to 0.47 V.

#### 3.6 Catalytic epoxidation of olefins

The new synthesized vanadyl Schiff base complex VOL was examined in the epoxidation of cyclooctene, cyclohexene, styrene, norbornene, 1-octene and indene in order to study it's catalytic activity.

In the epoxidation of cyclooctene, different parameters i.e. the effect of type of oxidants, quantity of them, solvent and catalyst quantity were tested. The vanadyl Schiff base complex in the epoxidation of cyclooctene and the result of it's catalytic activity in different solvent with the presence of *tert*-butylhydroperoxide (TBHP) is shown in Fig. 4 and Table 3. It is evident that the catalytic activity of vanadyl Schiff base complex in CH<sub>3</sub>Cl and CH<sub>2</sub>Cl<sub>2</sub>, with low coordination ability, is greater than CH<sub>3</sub>CN, C<sub>3</sub>H<sub>6</sub>O and CH<sub>3</sub>OH with high coordination ability [43]. Different reactions were used in order to gain the suitable oxidant in the epoxidation of cyclooctene (Table 4). High yield was gained in the presence of TBHP in CHCl<sub>3</sub>. By altering the amount of oxidant, the reaction was also studied (Table 5). Two mmol of oxidant offered the maximum epoxidation yield among the 0.5, 0.75, 1, 2 and 2.5 mmol of TBHP. The quantity of Schiff base complex also

checked on the epoxidation of cyclooctene (Table 6). In the presence of TBHP, different quantity of Schiff base complex (0.005, 0.01 and 0.02 mmol) were used in the epoxidation of cyclooctene in the CHCl<sub>3</sub> and the maximum epoxidation yield was made by 0.01 mmol of complex.

Our further investigation concerned the oxidation of cyclohexene, styrene, norbornene, 1octene and indene. In a typical experiment, a mixture of 0.01 mmol vanadyl Schiff base complex, freshly distilled CH<sub>3</sub>Cl (3 mL), 1 mmol freshly distilled olefin and 2 mmol of TBHP was refluxed at 50°C for several times. The reaction products were monitored at periodic time using gas chromatography. By using homogeneous catalyst, the product distributions in the oxidation of olefins were shown in Table 7.

The same results were obtained from catalytic activities of some five coordination oxidovanadium (IV) Schiff base complexes with tetradentate  $N_2O_2$  or tridentate  $NO_2$  ligands in cyclooctene epoxidation reaction [44-46]. The results showed that the cyclooctene epoxidation reaction with TBHP had higher conversion than  $H_2O_2$  [44]. The catalytic activities of two oxovanadium (IV) Schiff base complexs in cyclooctene epoxidation reaction with chloroform, acetonitrile and dichloromethane were investigated by Rayati and showed that the highest conversion obtained in the chloroform [46]. In summary based on the reported results and also present work, the high epoxidation yield by oxidovanadium Schiff base complexes was obtained in the presence of TBHP in CHCl<sub>3</sub>.

Furthermore, the mechanistic aspects of the epoxidation reaction of alkenes were reviewed. The proposed mechanism of epoxidation reaction by oxidovanadium (IV) Schiff base complex in the presence of TBHP was showed in Scheme 2 [47]. The observed result confirmed that the first step of mechanism involves the oxidation of vanadium (IV) Schiff base complex with TBHP and the interaction of this complex with TBHP resulted in vanadium (V) species [48]. In the next

step involves the transfer of TBHP proton to the terminal oxygen atom of Vanadyl group and coordination of t-BuOO<sup>-</sup> to vanadium (V) as a Lewis acidic metal center and resulted in formation of the oxidoperoxidovanadium (V) intermediate.

#### 4. Conclusions

In the present research, as a part of our progressing studies on the catalyst, we demonstrated the synthesis and characterization of new oxidovanadium Schiff base complex (IV). The elemental analysis (CHN), UV-Vis and FT-IR spectroscopy showed that the vanadyl complex was synthesized succesfully. The single crystal X-ray analysis showed that V(IV) placed in a distorted five coordinated square pyramidal  $N_2O_3$  geometry. Fuethermore, the catalytic activity of this complex was tested in epoxidation of alkenes such as cyclooctene, cyclohexene, styrene, norbornene, 1-octene and indene using *tert*-butyl hydroperoxide as oxygen source in chloroform. Based on the results, the vanadyl Schiff base complex was very active in epoxidation of alkenes in optimized conditions. Moreover, electrochemical behavior of V<sup>IV</sup>O complex was also investigated.

#### 4. Supplementary material

CCDC No. 1513755 contains the supplementary crystallographic data for V<sup>IV</sup>OL (L = N-2hydroxyacetophenon-N'-2-hydroxynaphthaldehyde-1,2 phenylenediimine),. These data can be obtained at www.ccdc.cam.ac.uk/deposit {or from the Cambridge Crystallographic Data Center 12, Union Road Cambridge CB2 1EZ, UK; Fax: (internet) +44-1223/336-033; E. mail: deposit@ccdc.cam.ac.uk).

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MA

Scheme 1. The structure of Schiff base ligand and its complex.



**Scheme 2.** The proposed mechanism for the epoxidation of alkenes by the oxovanadium Schiff base complex in the presence of TBHP.

![](_page_18_Figure_1.jpeg)

Fig. 1. Electronic spectra of Schiff base ligand (a) and [VO(L)] Complex (b, dashed line).

ROFF

![](_page_19_Figure_1.jpeg)

Fig. 2. The labeled diagram of [VO(L)] Complex showing 50% probability thermal ellipsoids.

![](_page_20_Figure_1.jpeg)

Fig. 3. Cyclic voltamogram of [VO(L)] Complex in DMF at room temperature and Scan rate:

![](_page_21_Figure_1.jpeg)

**Fig. 4.** The catalytic epoxidation of cyclooctene in different solvent in the presence of TBHP by the vanadyl Schiff base complex in the reflux condition. Reaction condition: (3 ml) solvent, (1 mmol) cyclooctene, (2 mmol) oxidant and (0.01 mmol) Vanadyl Schiff base complex.

	Compound	(A)
	Formula	$C_{25}H_{18}N_2O_3V$
	Formula weight	445.35
	Temperature /K	100(2)
	Wavelength λ /Å	0.710
	Crystal system	Orthorhombic
	Space Group	Pca2(1)
	Crystal size /mm <sup>3</sup>	0.16 × 0.39 ×
	0	39
	a /Å	7.8551(7)
	b/Å	25.291(2)
	c /Å	22.775(2)
	α/°	90
	β /°	90
	γ /°	90
0	Density (calc.) /g cm <sup>-1</sup>	1.308
	$\boldsymbol{\theta}$ ranges for data collection	0.805-25.99
~	F(000)	1832
	Absorption coefficient	0.466
	Index ranges	-9 ≤ h ≤ 9

**Table 1.** Crystallographic and structure refinements data for [VO (L)] Complex.

	-31 ≤ k ≤ 31				
	-28 ≤ l ≤ 28				
Reflections collected	8887				
Parameters, restrains	561, 1				
Final $R_1$ , $w R_2^a$ (Obs. data)	0.0355 , 0.0918				
Final $R_1$ , $w R_2^a$ (All data)	0.0394 , 0.0890				
Goodness of fit on F <sup>2</sup> (S)	1.021				
$R_1 = \Sigma   F_0  -  F_c   / \Sigma  F_0 , wR_2 = [\Sigma($	$w(F_o^2 - F_c^2)^2) / \Sigma w(F_o^2)^2]^1$				

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<sup>a</sup>  $R_1 = \Sigma ||F_0| - |F_c|| / \Sigma |F_0|$ ,  $wR_2 = [\Sigma (w(F_0^2 - F_c^2)^2) / \Sigma w(F_0^2)^2]^1$ 

Bond distances		Bond angels		_
V1-N1	2.075(3)	03-V1-01	111.59(13)	
V1-N2	2.041(3)	03-V1-02	110.83(11)	
V1-01	1.916(3)	01-V1-02	84.31(10)	
V1-02	1.926(2)	O3-V1-N2	106.90(13)	
V1-03	1.604(2)	01-V1-N2	141.25(11)	
01-C1	1.335(4)	O2-V1-N2	86.29(11)	
O2-C24	1.308(4)	O3-V1-N1	102.06(11)	
N1-C7	1.315(4)	01-V1-N1	88.65(11)	
N2-C13	1.414(4)	O2-V1-N1	146.67(11)	
N1-C8	1.419(4)	N2-V1-N1	79.06(11)	
N2-C14	1.319(4)			

Table 2. Selected bond distances (Å) and bond angles (°) for [VO (L)] Complex.

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**Table 3.** The solvent effect on the catalytic epoxidation of cyclooctene in the presence of TBHP by the vanadyl Schiff base complex in the reflux condition<sup>a</sup>.

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Solvent	Time (min)	Yield(٪)
acetone	120	1
methanol	120	2
acetonitrile	120	7
1, 2 dichloroethane	120	14
1, 2 dichloromethane	120	43
chloroform	120	93

<sup>a</sup> Reaction condition: (3 ml) solvent, (1 mmol) cyclooctene, (2 mmol) TBHP and (0.01 mmol) Vanadyl Schiff base complex.

**Table 4.** Epoxidation of cyclooctene in  $CHCl_3$  as solvent in the presence of different oxidant by the Vanadyl Schiff base complex in the reflux condition<sup>a</sup>.

SCRIPS

Solvent	oxidante	Time(min)	Yield(½)
chloroform	-	120	No
			reaction
chloroform	Sodium periodate	120	2
chloroform	H <sub>2</sub> O <sub>2</sub>	120	10
chloroform	UHP	120	41
chloroform	ТВНР	120	93

<sup>a</sup> Reaction condition: (3 ml) chloroform, (1 mmol) cyclooctene, (2 mmol) oxidant and (0.01 mmol) Vanadyl Schiff base complex.

**Table 5.** The effect of the amount of the oxidant in the epoxidation of cyclooctene in CHCl<sub>3</sub> as solvent in the presence of TBHP by the Vanadyl Schiff base complex in the reflux condition<sup>a</sup>.

SCRIP

 Run	mmol of oxidante	Time(min)	Yield(٪)
 1	0.5	120	80
2	0.75	120	10
3	1	120	4
4	2	120	93
5	2.5	120	20

<sup>a</sup> Reaction condition: (3 ml) chloroform, (1 mmol) cyclooctene, (0.01 mmol) Vanadyl Schiff base complex and (0.5, 0.75, 1, 2 and 2.5 mmol) oxidant.

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**Table 6.** The effect of the amount of the Vanadyl Schiff base complex in the epoxidation of cyclooctene in CHCl<sub>3</sub> as solvent in the presence of TBHP complex in the reflux condition<sup>a</sup>.

Run	mmol of catalyst	Time(min)	Yield(٪)
1	0	120	3
2	0.005	120	30
3	0.01	120	93
4	0.02	120	35

<sup>a</sup> Reaction condition: (3 ml) chloroform, (1 mmol) cyclooctene, (0.01 mmol) Vanadyl Schiff base complex and (0.5, 0.75, 1, 2 and 2.5 mmol) oxidant.

Entry	Alkene	Time(min)	Yield(٪)
1		120	93
2		480	85
3		360	76
4		120	60
5		480	50
6		480	40

**Table 7.** Epoxidation of alkenes in  $CHCl_3$  as solvent in the presence of TBHP by the Vanadyl Schiff base complex in the reflux condition <sup>a</sup>.

<sup>a</sup> Reaction condition: (3 ml) chloroform, (1 mmol) alkene, (0.01 mmol) Vanadyl Schiff base complex and 2 mmol TBHP.

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![](_page_30_Figure_1.jpeg)

- Novel vanadyl Schiff base complex was prepared and its structure were confirmed by different techniques
- The X-ray crystallography results show that the complex is five coordinated in the solid state
- The catalytic activity of the oxidovanadium(IV) Schiff base complex was tested in the epoxidation of cyclooctene