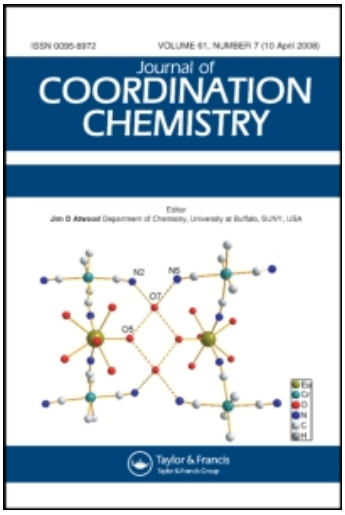

Journal of Coordination Chemistry
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713455674

Monooxovanadium(V) 2-phenylphenoxides: synthesis, characterization, and antimicrobial potential

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Online publication date: 07 April 2010

To cite this Article Sharma, Neeraj , Thakur, Maridula and Chaudhry, S. C.(2010) 'Monooxovanadium(V) 2-phenylphenoxides: synthesis, characterization, and antimicrobial potential', Journal of Coordination Chemistry, 63: 6, 1071 — 1079
To link to this Article: DOI: 10.1080/00958971003710377
URL: http://dx.doi.org/10.1080/00958971003710377

Journal of Coordination Chemistry
Vol. 63, No. 6, 20 March 2010, 1071–1079

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Monooxovanadium(V) 2-phenylphenoxides: synthesis, characterization, and antimicrobial potential

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(*Received 23 July 2009; in final form 11 November 2009*)

Monooxovanadium(V) complexes of the composition $VOCl_{3-n}(L)_n$ (where L = 2-phenylphenoxide ion; $n = 1$–3) (**1**–**3**) have been synthesized in quantitative yields by the reaction of $VOCl_3$ with 2-phenylphenol in toluene. The characterization of the complexes has been accomplished by elemental analysis, molar conductance measurements, IR, ^{1}H-NMR, electronic, mass spectral, and thermal studies. The ligands as well as the complexes have been screened for their *in vitro* antimicrobial activity against the pathogenic bacteria *Escherichia coli* and *Staphylococcus aureus* and fungi *Candida albicans*, *Aspergillus niger*, and *Fusarium oxysporum* by a twofold serial dilution. An increase in the biocidal activity was observed for the vanadium complexes. The minimum inhibitory concentration (MIC) values were 6.25–25 $\mu g\,mL^{-1}$ for complexes, relative to that of the free ligand of 25–50 $\mu g\,mL^{-1}$.

Keywords: Monooxovanadium(V) complexes; 2-Phenylphenol; Antimicrobial activity

1. Introduction

The discovery of the vanadium-dependent enzymes, namely, haloperoxidase [1, 2], nitrogenase [3], and nitrate reductase [4] has provided a new thrust in the research of this element. Vanadium's insulin-mimetic behavior [5–10] and a number of therapeutic effects [11] including anti-tumor and anti-inflammatory activities have stimulated renewed interest in vanadium coordination chemistry. The potential of vanadium complexes as catalysts in industry [12–15], organic synthesis [16], in material sciences, and magneto-structural study of polynuclear species [17, 18] has also drawn considerable attention. Substituted phenols constitute an important group of organic ligands which provide complexes with rich structural diversity and pronounced antioxidant, catalytic, and biological relevance [19]. Vanadium alkoxides and aryloxides owing to their applications as catalysts for polymerization [20], dinitrogen fixation [21–23], oxidation [24], and potential shape-selective transformations [25] have been the subject of much research. Literature survey reveals that compared to the synthesis of

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ISSN 0095-8972 print/ISSN 1029-0389 online
DOI: 10.1080/00958971003710377

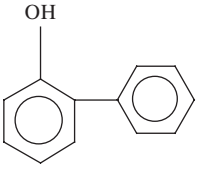


Figure 1. Structure of 2-phenylphenol.

metal complexes with a large number of substituted phenols, 2-phenylphenol, a broad spectrum biocide has remained unexplored despite its biocidal effects for control of post-harvest diseases of several varieties of fruits [26, 27]. Interest in 2-phenylphenol also stems from the fact that the ortho alkyl/aryl substituents show interesting coordination behavior, wherein the intermolecular dehydrogenation of the alkyl groups or the hydrogenation of the aryl rings or chelation *via* μ-arene interaction may result. In continuation of our earlier work on the synthesis of monooxovanadium(V) aryloxides [28–30], and considering the biological relevance of both vanadium and 2-phenylphenol, this work is aimed at the synthesis of the new monooxovanadium(V) complexes of 2-phenylphenol (figure 1) in order to have an insight into the coordination behavior of the ligand. As the activity of the bioactive ligands is known to increase on coordination with the metal ions, the complexes have also been screened against some pathogenic fungi, *Candida albicans*, *Aspergillus niger*, and *Fusarium oxysporum* and bacteria, namely, *Escherichia coli* and *Staphylococcus aureus* for assaying their antimicrobial activity.

2. Experimental

2.1. *Materials and methods*

All the solvents were of A.R. grade and dried by standard methods. 2-Phenylphenol (Merck) was recrystallized from benzene and the purity was checked by its sharp melting point (57°C). $VOCl_3$ was used as received.

Vanadium in the complexes was determined gravimetrically as V_2O_5 while chlorine was determined by Volhard's method. The elemental analyses were performed on a Carlo-Erba 1108 Elemental Analyzer. The conductivity measurements in nitrobenzene were made on an Elico Conductivity Bridge type CM-82T. FT-IR spectra of complexes were collected on a Nicolet 5700 spectrophotometer (4000–200 cm^{-1}) as KBr pellets or nujol mulls in CsI optics. The ^{1}H-NMR spectra were recorded on a BRUKERAVANCE II 400 Spectrometer using $CDCl_3$ as solvent and TMS as an internal reference. The UV-Vis spectra were recorded on a Varian CARY 100 BIO UV-Vis spectrophotometer. The FAB mass spectra were recorded at room temperature on a JEOL SX 102/DA-600 mass spectrometer/data system using Ar/Xe (6 kV, 10 mA) as the FAB gas. The accelerating voltage was 10 kV. The *m*-nitrobenzylalcohol (NBA) was used as the matrix. The thermograms were recorded on a simultaneous TG-DTA SHIMADZU DT-60 Thermal analyzer (heating rate, 20°C min^{-1}, reference, Al_2O_3; thermocouple, Pt/Pt–Rh 10% and atmosphere, air).

2.2. *Synthesis*

2.2.1. [$VOCl_2(L)$] (1). In a typical reaction, an equimolar amount of 2-phenylphenol (1.794 g, 0.01055 mol) in dry toluene (10 mL) was added to a solution of $VOCl_3$ (1 mL, 1.826 g, 0.01055 mol) in the same solvent (10 mL). The reaction mixture was refluxed until no more evolution of the HCl gas was observed (18 h) and then filtered. The filtrate was concentrated by distilling off the excess solvent to one-third of its initial volume. The concentrate was then treated with petroleum ether wherein a black complex was obtained. It was dried under vacuum and recrystallized from toluene (2.71 g, 75%), decomposition temperature 47°C, Anal. Calcd for $C_{12}H_9O_2Cl_2V$ (formula weight, 307) (%): C, 46.90; H, 2.93; Cl, 23.13; V, 16.61. Found: C, 47.30; H, 2.70; Cl, 23.46; V, 16.89; $\Lambda_m = 6.81\,Scm^2\,mol^{-1}$; $\nu_{max}\,(cm^{-1})$: 1618(s), 1578(w), 1477(m), 1432(m), 1209(s), 1109(m), 993(vs), 758(s), 698(s), 570(s), 526(w), 346(w); ^{1}H-NMR: δ_H (400 MHz, $(CD_3)_2SO$) 6.88 (d, 1H, *J* 8 Hz, 6-H), 7.00–7.06 (t, 1H, 4-H), 7.21–7.25 (t, 1H, 5-H), 7.27 (s, 1H, 4′-H), 7.38 (d, 1H, *J* 8 Hz, 3-H), 7.48 (s, 2H, 3′-H), 7.58 (d, 2H, *J* 8 Hz, 2′-H).

Similar procedure was adopted for the synthesis of $VOCl(L)_2$ and $VO(L)_3$ by the reaction of $VOCl_3$ (1 mL, 1.826 g, 0.01055 mol) with two and three equivalents of 2-phenylphenol (3.588 g, 0.021105 mol/5.382 g, 0.03165 mol) whereupon shining black to brownish-black complexes were obtained.

2.2.2. [$VOCl(L)_2$] (2). (1.79 g, 69%), decomposition temperature 56°C, Anal. Calcd for $C_{24}H_{18}O_3ClV$ (formula weight, 440.5) (%): C, 65.38; H, 4.09; Cl, 8.06; V, 11.58. Found: C, 5.71; H, 4.16; Cl, 8.22; V, 11.82; $\Lambda_m = 4.61\,Scm^2\,mol^{-1}$; $\nu_{max}\,(cm^{-1})$: 1583(s), 1476(s), 1431(s), 1269(w), 1203(vs), 1110(m), 991(s), 886(s), 826(s), 756(s), 697(s), 562(s), 437(w), 348(w); ^{1}H-NMR: δ_H (400 MHz, $(CD_3)_2SO$) 6.98 (d, 2H, *J* 8 Hz, 6-H), 7.11–7.15 (t, 2H, 4-H), 7.24 (d, 2H, *J* 8 Hz, 4′-H), 7.28 (d, 2H, *J* 8 Hz, 5-H), 7.36–7.39 (t, 2H, 3-H), 7.49 (d, 4H, *J* 8 Hz, 3′-H, 5′-H), 7.56 (d, 4H, *J* 8 Hz, 2′-H, 6′-H).

2.2.3. [$VO(L)_3$] (3). (1.62 g, 72%), decomposition temperature 60°C, Anal. Calcd for $C_{36}H_{27}O_4V$ (formula weight, 574) (%): C, 75.26; H, 4.70; V, 8.88. Found: C, 75.52; H, 4.98; V, 9.09; $\Lambda_m = 4.21\,Scm^2\,mol^{-1}$; $\nu_{max}\,(cm^{-1})$: 1595(s), 1498(s), 1471(s), 1450(s), 1426(s), 1246(s), 1204(s), 1106(s), 1075(w), 1045(w), 1016(s), 942(s), 889(s), 760(s), 696(m), 675(m), 542(w), 507(s), 481(s), 423(s); ^{1}H-NMR: δ_H (400 MHz, $(CD_3)_2SO$) 6.98 (d, 3H, *J* 8 Hz, 6-H), 7.03 (d, 3H, *J* 8 Hz, 4-H), 7.13–7.18 (t, 3H, 5-H), 7.23 (d, 3H, *J* 8 Hz, 4′-H), 7.34 (d, 3H, *J* 8 Hz, 3-H), 7.39 (d, 6H, *J* 8 Hz, 3′-H, 5′-H), 7.47 (s, 6H, 2′-H, 6′-H).

2.3. In vitro *antimicrobial assay*

In vitro antibacterial and antifungal activity of monooxovanadium(V) complexes were studied against the phytopathogenic fungi *C. albicans*, *A. niger*, and *F. oxysporum* and the bacteria Gram(−ve) *E. coli* and Gram(+ve) *S. aureus* by the minimum inhibitory concentration (MIC) method. MIC is the lowest concentration of the antimicrobial agent that prevents the development of visible growth after overnight incubation [31]. All the samples were tested in triplicate. The MIC of compounds against the test

bacteria namely, Gram(–ve) and fungi, was determined by reported methods [32]. All the test cultures were streaked on SCDA and incubated overnight at 37°C. A stock solution of 4 mg mL^{-1} of each compound was prepared in DMSO and appropriately diluted to give final concentrations of 100, 50, 25, 12.5, 6.25, 3.12, 1.56, 0.78, 0.39, 0.19, and 0.09 μg mL^{-1}. Standard bactericides such as tetracycline, chloramphenicol, and kanamycin and fungicides cycloheximide, carbendazim, and fluconazole were also diluted in a similar manner. Three hundred and twenty microliters of each dilution was added to 20 mL molten and cooled MHA (separate flasks were taken for each dilution). After thorough mixing, the medium was poured into sterilized petri plates and the plates were incubated at 37 ± 1°C for 24 h for bacteria and for 7 days at 25 ± 1°C and 36 h at 37 ± 1°C for *A. niger* and *C. albicans*, respectively.

3. Results and discussion

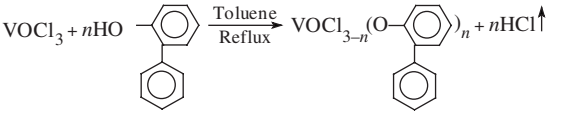
$VOCl_3$ mediates the oxidative coupling of various phenols to dimers or higher oligomers and aromatic ethers of utility in the synthesis of many natural products [33]. The reduction of vanadium(V) to vanadium(IV) from oxidation of the phenol to a quinone [34] indicates that bonding of phenols to strongly oxidizing metal ions is difficult [35, 36]. Nevertheless, several vanadium(V) phenolate complexes stable toward reduction are known [37].

The formation of $VOCl_{3-n}(L)_n$ in fairly good yields (69–75%) has been effected by the reaction of $VOCl_3$ with 2-phenylphenol in appropriate molar ratios in toluene (scheme 1). The carbon, hydrogen, chlorine, and vanadium analyses conform to the proposed formulations.

The complexes are brownish-black to black solids and soluble in common organic solvents. The molar conductance values of millimolar solution of the complexes in nitrobenzene (ranging from 4.21 to 6.81 Scm^2 mol^{-1}) show that all the complexes are non-ionic. Molar conductances for 1:1 electrolyte in nitrobenzene lie in the 20–30 Scm^2 mol^{-1} range [38].

3.1. *IR spectra*

The formation of the complexes has been ascertained from the comparison of the IR spectra of the complexes with that of 2-phenylphenol from 4000 to 200 cm^{-1}. The absorption at 3653 cm^{-1} due to the phenolic ν(OH) in 2-phenylphenol was absent in the complexes, suggesting deprotonation. The absorptions due to ν(C–O) occur at 1410–1310 cm^{-1} and 1260–1180 cm^{-1} in free phenols [39, 40]. The bands at



(where $n = 1$ to 3)

Scheme 1. Synthesis of monooxovanadium(V) 2-phenylphenoxides.

1327–1269 cm^{-1} in 2-phenylphenol assigned to ν(C–O) occur at 1250–1180 cm^{-1} in the complexes, indicating phenolic oxygen bonding. This was further supported by bands at 570–520 cm^{-1} assigned to ν(V–O). The ν(V=O) occurs at 1035 cm^{-1} [41] in $VOCl_3$ and 1020–960 cm^{-1} in numerous vanadyl salts and complexes [42]. The ν(V=O) appeared at 993, 991, and 992 cm^{-1} in the $VOCl_2(L)$, $VOCl(L)_2$, and $VO(L)_3$, respectively. The lowering in ν(V=O) may be attributed to the decreased terminal oxo $p\pi \rightarrow$ vanadium $d\pi$ interactions resulting from increased electron density at vanadium upon bonding through 2-phenylphenoxide. For $VOCl_2(L)$ and $VOCl(L)_2$, absorptions at 346 and 348 cm^{-1} may be attributed to ν(V–Cl) [43]. The bands at 830–780 cm^{-1} in $VOCl_2(L)$ and $VOCl(L)_2$ have been attributed to V–O–V [44], suggesting dimers.

3.2. *^{1}H-NMR spectra*

A comparison of the room temperature ^{1}H-NMR spectra of the complexes with the free 2-phenylphenol, substantiated the formation of the complexes. The ^{1}H-NMR spectra of 2-phenylphenol displayed signals at δ 5.16, δ 6.96–7.28, and δ 7.40–7.53 ppm (Supplementary material). The ^{1}H-NMR spectra of the complexes did not display a signal at δ 5.16 ppm, indicating deprotonation and bonding through phenolic oxygen. The aromatic protons of the phenolic ring as well as the phenyl substituent undergo downfield shifts in complexes which may be ascribed to deshielding of these protons due to the transfer of the electron density from the phenolic ring to vanadium.

3.3. *Electronic spectra*

The UV-Vis spectra of 2-phenylphenol exhibit three absorption bands at 280, 270, and 241 nm assignable to $\sigma \rightarrow \sigma^*$, $\pi \rightarrow \pi^*$, and $n \rightarrow \pi^*$ transitions, respectively. These bands shift to higher wavelengths and appear in 315–300, 275–266, and 250–246 nm range in complexes. In addition, **1**–**3** have a medium intensity band at 404, 406, and 410 nm, respectively, assigned to the ligand to metal charge transfer (LMCT) from the phenolate oxygen to an empty d orbital of vanadium. The LMCT transitions are characteristic of VO^{3+} with phenolate ligands.

3.4. *FAB-MS spectra*

The FAB-MS spectral data for $[VOCl_2(L)]$, $[VOCl(L)_2]$, and $[VO(L)_3]$ are given in the "Supplementary material". The fragment at *m/e* 307 for **1** corresponds to $[M–H]^+$. The fragment ions at *m/e* 611 and 577 beyond the molecular mass peak may be attributed to $[2M–H]^+$ and $[2M–Cl]^+$, thereby suggesting a dimeric nature. The most intense peak at *m/e* 217 is ascribed to $[VCl_2(OC_6H_5)]^+$. The formations of **2** and **3** have been inferred from fragments at *m/e* 425/427 and 559 attributed to $[VCl(L)_2 + H]^+$ and $[V(L)_3 + H]^+$, formed by the removal of oxygen from the respective complexes. For **2**, the *m/e* at 876 and 674 ascribed to $[2M–4H]^+$ and $[2M–Cl–3H]^+$ indicate the dimeric nature. The most intense fragment at *m/e* 338 has been attributed to $[(L)_2]^+$ in **2**. The most intense fragment at *m/e* 170 corresponds to $[HL]^+$ in **3**. Complex **3** did not display a fragment beyond its molecular ion peak, confirming its monomeric nature.

The well-defined fragmentation patterns have been observed for all the complexes (Supplementary material).

3.5. *Thermal studies*

The thermal data obtained from the TG and DTA curves are summarized in table 1 and the "Supplementary material". $[VOCl_2(L)]$, $[VOCl(L)_2]$, and $[VO(L)_3]$ have initial decomposition temperatures of 46°C, 57°C, and 60°C, respectively. $[VOCl_2(L)]$ and $[VOCl(L)_2]$ undergo decomposition in a single step while $[VO(L)_3]$ exhibited a two-step decomposition. The percentage weight loss for **1** and **2** was 76% and 78% against the calculated weight loss of 74% and 79% accounting for the formation of V_2O_5 as the final product. The weight loss of 70% and 14% in the first and second step, respectively, compared to the calculated weight loss of 70% and 15% in the respective steps in **3** accounted for the formation of $[VO_2(OC_6H_5)]$ as the probable intermediate which undergoes decomposition to yield V_2O_5. The formation of V_2O_5 supports the stoichiometry of the complexes. The thermal decomposition in TG is accompanied by an exothermic peak for **1** and **2** while both endothermic and exothermic peaks have been observed for **3**. The process of decomposition is given in table 1 and scheme 2.

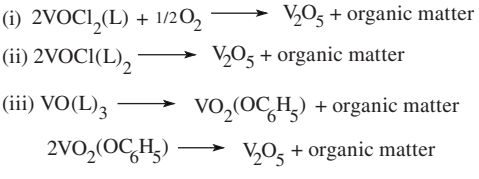
On the basis of IR and mass spectral studies, a five-coordinate environment around vanadium for dimeric $[VOCl_2(L)]$ and $[VOCl(L)_2]$ and a tetrahedral stereochemistry around vanadium in $[VO(L)_3]$ may be tentatively assigned.

3.6. *Antibacterial activity*

The biological activity of several vanadium complexes has been reported [45–55]. The ligand, 2-phenylphenol, and the newly synthesized monooxovanadium(V) complexes

Table 1. Thermal data of monooxovanadium(V) complexes.

			TGA data			DTA data	
Complex	Initial decomposition temperature (°C)	Stages of decomposition	Decomposition range (°C)	% weight loss	Decomposition products	Peak temperature (°C)	Peak nature
$VOCl_2(L)$	46	Single	46–520	76.12	V_2O_5	441.10	Exo
$VOCl(L)_2$	57	Single	57–696	78.24	V_2O_5	475.24	Exo
$VO(L)_3$	60	Two	60–352	70.34	$VO_2(OC_6H_5)$	106.65	Endo
			352–525	14.09	V_2O_5	464.49	Exo



Scheme 2. Thermal decomposition of monooxovanadium(V) 2-phenylphenoxides.

Table 2. The *in vitro* antimicrobial activity of monooxovanadium(V) complexes (MIC in $\mu g\,mL^{-1}$).

	Bacteria		Fungi		
Ligand/complexes	*E. coli*	*S. aureus*	*C. albicans*	*A. niger*	*F. oxysporum*
HOL	25	50	50	25	25
$VOCl_2(L)$	25	12.5	12.5	12.5	25
$VOCl(L)_2$	12.5	12.5	12.5	6.25	12.5
$VO(L)_3$	12.5	6.25	6.25	6.25	6.25
Tetracycline	<3.12	<3.12	–	–	–
Chloramphenicol	<3.12	<3.12	–	–	–
Kanamycin	<3.12	<3.12	–	–	–
Cycloheximide	–	–	<3.12	<3.12	<3.12
Carbendazim	–	–	<3.12	<3.12	<3.12
Fluconazole	–	–	<3.12	<3.12	<3.12

were tested *in vitro* for their antibacterial activity against *S. aureus* and *E. coli* at different concentrations in DMSO using the MIC method (table 2 and "Supplementary material"). All the complexes possess more activity against *S. aureus*, with an MIC of 6.25–12.5 $\mu g\,mL^{-1}$, than the MIC shown for *E. coli* at 12.5–25 $\mu g\,mL^{-1}$. Complex **3** is most active but less than the conventional bactericides tetracycline, chloramphenicol, and kanamycin (MIC values <3.12 $\mu g\,mL^{-1}$). The enhanced antibacterial activity has been shown by the complexes relative to the free ligand.

3.7. *Antifungal activity*

In vitro antifungal screening of 2-phenylphenol as well as its three monooxovanadium(V) complexes were performed against *C. albicans*, *A. niger*, and *F. oxysporum* by the MIC method (table 2 and "Supplementary material"). The MIC of the complexes were in the range of 6.25–25 $\mu g\,mL^{-1}$, which suggests their potential as antifungal agents. The data indicates that **3** possesses better antifungal activity than **1** and **2**. The antifungal activity of the complexes is less than standard fungicides, namely, cycloheximide, carbendazim, and fluconazole (MIC values <3.12 $\mu g\,mL^{-1}$).

A comparison of the results of biological activity of the monooxovanadium(V) complexes with that of other reports [45–55] revealed the effectiveness of newly synthesized vanadium complexes as antimicrobial agents.

4. Conclusion

Monooxovanadium(V) complexes of 2-phenylphenol have been thoroughly characterized by physicochemical, spectral, and thermal techniques. The complexes showed higher antimicrobial activity (6.25–25 $\mu g\,mL^{-1}$) than the free ligand. Hence, oxovanadium(V) complexes have potential as biocides.

Acknowledgments

One of the authors (M. Thakur) is thankful to the University Grants Commission, New Delhi, India, for providing financial assistance in the form of Junior Research Fellowship. The authors thank Department of Science and Technology (DST), Government of India, New Delhi, for providing financial assistance for FT-IR and UV-Vis Spectrophotometer facility to the department, under its FIST program. The authors wish to thank Sophisticated Analytical Instrument Facility, CDRI, Lucknow, India, for recording the mass spectra. The authors also thank the Sophisticated Analytical Instrument Facility, Punjab University, Chandigarh, for recording ^{1}H-NMR and elemental analysis data. The authors thank the Department of Bio-sciences, Himachal Pradesh University, Summer Hill, Shimla, for carrying out the antimicrobial studies.

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