

RESEARCH ARTICLE

# Synthesis, structural characterisation and antibacterial activity of bis(1-phenyl-1,3-butanedionato) non-oxovanadium(IV) hydroxamates

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## Abstract

Complexes with a composition of  $VCl_{2-n}(bzac)_2(HL^{1,2})_n$  (I – IV) (where  $bzac = 1\text{-phenyl-1,3-butanedionato anion } (C_6H_5COCHCOCH_3^-)$ ;  $HL^1 = C_6H_5OCH_2C(O)NHO^-$ ,  $HL^2 = C_6H_5CH=CHC(O)NHO^-$ ,  $n = 1$  and  $2$ ) have been synthesised from the reactions of  $(VCl_2(bzac)_2)$  with equi- and bi-molar amounts of potassium phenoxyacetohydroxamate and cinnamohydroxamate ( $KHL^{1,2}$ ) in THF + MeOH solvent medium. The complexes have been characterised by elemental analyses, molar conductivity, magnetic measurements, IR, electronic and mass spectral studies. The physicochemical and spectral studies suggest a distorted octahedral geometry around vanadium in the complexes. The antibacterial activities of the newly synthesised complexes, vanadium precursor and ligands have been screened *in vitro* against six bacterial species. The complexes have shown higher antibacterial effect than the free ligands.

**Keywords:** Potassium phenoxyaceto-hydroxamate; potassiumcinnamohydroxamate; non-oxovanadium (IV) complexes; spectral studies; antibacterial activity

## Introduction

Over the years, an immense research interest has been directed towards coordination chemistry of vanadium, owing to the promising insulin-mimetic, appetite-suppressing and antihypertensive effects exhibited by vanadium complexes [1]. The literature contains numerous reports on the chemistry of the vanadyl ion,  $VO^{2+}$ , however, of late the chemistry of easily synthesisable dichloro vanadium (IV) chelate complexes from  $VO^{2+}$ , constituted appropriate precursors for alkylation, reduction and substitution reactions and have been reported to be functionalised with a variety of ligands. Several octacoordinated non-oxovanadium (IV) complexes with multidentate N, O donor ligands [2] and biphenylphenols have been reported [3]. Of the numerous ligands known to form vanadium complexes, hydroxamic acids (naturally occurring or synthetic) constitute an important family of organic bioligands [4]. The naturally occurring hydroxamic acids (siderophores) are involved in the microbial transport of iron [5]. Hydroxamic acids, with the NHOH moiety are constituents of antibiotics, antifungal agents, food additives, tumour inhibitors and growth factors [6–8], because of their low toxicities they have a wide spectrum of activities

in biological systems [9–11]. The powerful biological activity of structurally heterogeneous hydroxamic acids is related to their ability to form stable chelates with a variety of metal ions [12,13]. Metal complexes of hydroxamic acids have also attracted considerable attention because of their tautomerisation [14] exhibiting hydroxamic or hydroximic acid forms. The theoretical and experimental studies have suggested that the hydroxamic form is prevalent in free acids [15] or metal hydroxamates [16]. Fewer studies have described vanadium (IV) and (V) hydroxamate complexes as compared to many other transition metal hydroxamates [17–21] whereas vanadyl sulphate, ammonium vanadate and vanadyl acetylacetonate have been exploited as precursors [22]. We have been interested in exploring the use of dichlorovanadium (IV)  $\beta$ -diketonates as precursors for the synthesis of new complexes. There has been much research interest in coordination compounds of biologically active ligands, so in continuation of our earlier work [23] and as a result of the biological importance of vanadium and hydroxamate ligands, we report the synthesis and structural characterisation of mixed ligand non-oxovanadium (IV) complexes containing 1-phenyl-1,3-butanedionato and hydroxamate ligands from the reactions

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of  $VCl_2(bzac)_2$  with potassium phenoxyacetohydroxamate and cinnamohydroxamate (Figures 1 and 2). The newly synthesised complexes have been structurally characterised and assayed for their antibacterial activity, as the development of effective antibacterial drugs is a matter of great concern and the results are described.

## Material and methods

Reagent-grade solvents were dried and distilled prior to use. All other chemicals were reagent grade.  $VCl_2(bzac)_2$  was prepared from  $VO(bzac)_2$  using published methods [24, 25] under nitrogen atmosphere and its formation and purity was checked by C, H, Cl and V microanalysis and IR spectral data. The potassium phenoxyacetohydroxamate and cinnamohydroxamate were synthesised using published methods [26]. The vanadium content in the complexes was determined as  $V_2O_5$  while chlorine was determined by Volhard's method [42]. The carbon, hydrogen and nitrogen analysis were obtained on Eager 300 NCH System Elemental Analyser (Chandigarh). The molar conductances ( $10^{-3}$  M solutions in methanol) were obtained at  $25 \pm 0.1^\circ\text{C}$  on Elico Conductivity Bridge Type CM-82T (Shimla). The magnetic susceptibilities were measured at room temperature by Guoy's method using  $Hg[Co(NCS)_4]$  as calibrant [43]. IR spectra of complexes were recorded as KBr pellets on a Nicolet-5700 FTIR spectrophotometer (Shimla). The pellets were prepared in a dry box to avoid the action of moisture. The electronic spectra of complexes were recorded on a Varian Cary-100 Bio UV-VIS spectrophotometer (Shimla) using methanol as solvent. The FAB-mass spectra were recorded on a Jeol SX  $10^2/DA-6000$  mass spectrometer/data system (Lucknow) using Argon/Xenon (6 KV, 10 mA). The accelerating voltage was 10 KV and m-nitrobenzylalcohol (NBA) was used as the matrix. All bacteria used were clinical isolates taken from the Post-Graduate Institute of Medical Education and Research, Chandigarh, India.

## Synthesis

### Preparation of $VCl_{2-n}(bzac)_2(HL^{1,2})_n$

In a typical reaction to a solution of potassium phenoxyacetohydroxamate (0.46 g, 2 mmol / 0.92 g, 4 mmol) / potassium cinnamohydroxamate (0.45 g, 2 mmol / 0.90 g, 4 mmol) in

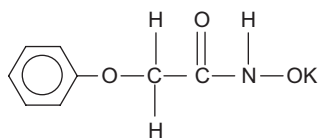


Figure 1. Structure of phenoxyacetohydroxamic acid ( $HL^1$ ).

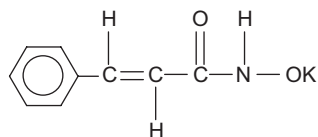


Figure 2. Structure of cinnamohydroxamic acid ( $HL^2$ ).

methanol (20 mL), a solution of  $VCl_2(bzac)_2$  (1 g, 2 mmol) in THF (20 mL) was added in separate experiments. The reaction mixture was stirred for 1 h and was then refluxed for 4–5 h. The white residue obtained during the course of the reaction was filtered and identified as KCl. The filtrate was distilled off to remove excess of solvent. The concentrate was then dried under vacuum by repeatedly treating with petroleum ether whereupon light-green and violet-blue coloured solids were obtained. These were recrystallised from dichloromethane.

## Antibacterial activity test

The precursor  $VCl_2(bzac)_2$ , potassium phenoxyacetohydroxamate ( $KHL^1$ ), potassium cinnamohydroxamate ( $KHL^2$ ) and vanadium (IV) complexes derived from these ligands of composition  $VCl_{2-n}(bzac)_2(HL^{1,2})_n$  (where  $bzac = 1$ -phenyl-1,3-butanedionate anion ( $C_6H_5COCHCOCH_3^-$ );  $HL^1 = C_6H_5OCH_2C(O)NHO^-$ ,  $HL^2 = C_6H_5CH=CHC(O)NHO^-$ ,  $n=1$  and 2) were screened *in vitro* for their antibacterial activity on selected bacteria *Escherichia coli*, *Staphylococcus aureus*, *Proteus mirabilis*, *Pseudomonas aeruginosa*, *Staphylococcus epidermidis* and *Shigella flexneri* using the minimum inhibitory concentration (MIC) method. All the samples were tested in triplicate.

## MIC determination by two-fold serial dilution

The MIC assay [27] was performed in a 96-well microtitre plate. For MIC assay of each test drug; a stock solution of 1mg/ mL of each drug was prepared in DMSO and a row of twelve wells was used out of which the last two wells were taken as control where no drug was added. To each of the ten wells, 100  $\mu\text{L}$  of the Muller-Hinton broth was added, except the first well which received 200  $\mu\text{L}$  of broth containing 500  $\mu\text{g}/\text{mL}$  concentration of the test drug in DMSO. From the first well (containing test drug), 100  $\mu\text{L}$  broth was withdrawn using a sterile tip, then added to the 100  $\mu\text{L}$  of the broth in the second well and the contents mixed four times. Next, 100  $\mu\text{L}$  was withdrawn from the second well and added to the third well and so on. In this way, a range of two-fold serial dilutions were prepared giving concentrations ranging from 500  $\mu\text{g}/\text{mL}$  to 0.98  $\mu\text{g}/\text{mL}$ . The broth in each of the wells was inoculated with 2  $\mu\text{L}$  of the bacterial culture and the contents were mixed by ten clockwise and ten anti-clockwise rotations on a flat surface. The plate was then incubated at  $35^\circ\text{C}$  and the observations for growth of bacteria were recorded after 24 h.

## Results and Discussion

### Chemistry

The interaction of  $VCl_2(bzac)_2$  with equi- and bimolar amounts of potassium phenoxyacetohydroxamate and potassium cinnamohydroxamate in THF + methanol solvent medium led to the formation of complexes  $VCl_{2-n}(bzac)_2(HL^{1,2})_n$  in quantitative yields according to Scheme 1.

The complexes were light green and bluish-violet in colour, microcrystalline and soluble in common organic

solvents such as methanol, chloroform, dichloromethane and acetonitrile, etc. The molar conductance values of the complexes ( $10^{-3}$ M solutions) (Table 1) in methanol in the 4.24 to 5.12  $\text{Scm}^2 \text{mole}^{-1}$  range suggested their non-electrolytic nature. The magnetic moment values (measured at room temperature) of the complexes were in the 1.71–1.75 BM range which suggested they were of a paramagnetic nature.

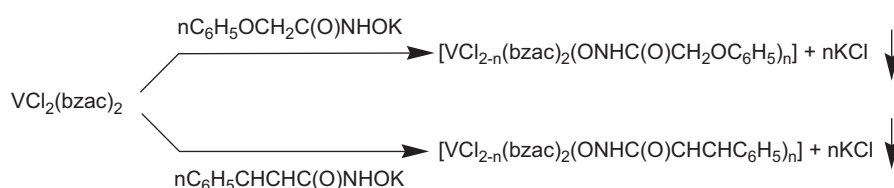
### IR spectra

A comparison of the IR spectra of  $\text{VCl}_2 \cdot n(\text{bzac})_2(\text{HL}^{1,2})_n$  with those of  $\text{VCl}_2(\text{bzac})_2$  and potassium phenoxyacetohydroxamate and cinnamohydroxamate has been useful in providing information regarding the formation of complexes. The absorption bands occurring in the 1680–1597  $\text{cm}^{-1}$  and 1650–1560  $\text{cm}^{-1}$  regions in potassium phenoxyacetohydroxamate and cinnamohydroxamate respectively have been attributed to  $\nu(\text{C}=\text{O})$  mode. The newly synthesised vanadium (IV) complexes exhibited bands due to  $\nu(\text{C}=\text{O})$  mode in 1663–1592  $\text{cm}^{-1}$  and 1660–1552  $\text{cm}^{-1}$  range respectively. No change in absorption band was observed due to  $\nu(\text{C}-\text{N})$  mode (occurring at  $\sim 1370 \text{ cm}^{-1}$  in free ligands upon complexation). The bands occurring at 3297  $\text{cm}^{-1}$  and 3229  $\text{cm}^{-1}$  assigned to  $\nu(\text{N}-\text{H})$  mode in KHL<sup>1</sup> and KHL<sup>2</sup> respectively were observed to appear at  $\sim 3292^{-1}$  and  $\sim 3232 \text{ cm}^{-1}$  in the respective complexes. This observation is indicative of the fact that  $-\text{NH}$  group is retained and does not participate in bonding. The bands due to  $\nu(\text{N}-\text{O})$  mode occurring at 938  $\text{cm}^{-1}$  and 958  $\text{cm}^{-1}$  in free ligands have been found to move to higher wave numbers and appeared at the  $\sim 975 \text{ cm}^{-1}$  and  $\sim 971 \text{ cm}^{-1}$  bands in respective series of complexes, suggesting that both the phenoxyacetohydroxamate and cinnamohydroxamate ions coordinated via the oxygen atom of the  $-\text{NHO}$  group only [19]. The monodentate nature of the ligands thus inferred is in consonance with the observation that hydroxamate ligands which behave as bidentate ligands in binary complexes act as monodentate ligands

in the presence of secondary ligands. This observation is contrary to previous reports describing O, O; N, N and N, O coordination in various hydroxamic acids [28]. The two to three absorption bands occurring in the 560–465  $\text{cm}^{-1}$  region have been assigned to  $\nu(\text{V}-\text{O})$  mode [29] in complexes which is obvious for the mixed-ligand complexes. In the far-IR spectra of the  $\text{VCl}_2(\text{bzac})_2(\text{HL}^{1,2})$  complexes, the absorption bands appearing at  $\sim 365 \text{ cm}^{-1}$  may be attributed to the  $\nu(\text{V}-\text{Cl})$  mode [30]. The absorption bands due to  $\nu_{\text{as}}(\text{C}=\text{O})$  and  $\nu_{\text{s}}(\text{C}=\text{O})$  modes in  $\text{VCl}_2(\text{bzac})_2$  occur in 1590–1490  $\text{cm}^{-1}$  and 1380–1310  $\text{cm}^{-1}$  regions respectively. It may be mentioned here that we were unable to make unambiguous assignments for the absorption bands due to the oylacetate ion in newly synthesised complexes due to the occurrence of some common absorption bands of hydroxamate and benzoylacetate ions in the same region.

### Electronic spectra

The electronic absorption spectra of potassium phenoxyacetohydroxamate and cinnamohydroxamate had sharp bands at 226, 257 nm and 221, 250 nm respectively in MeOH. These bands may be attributed to ligand  $\pi \rightarrow \pi^*$  transitions. The light-green and bluish-violet solutions of the mixed-ligand complexes of composition  $\text{VCl}(\text{bzac})_2(\text{HL}^1)$  and  $\text{V}(\text{bzac})_2(\text{HL}^1)_2$  displayed bands in 700–800 nm, 500–600 nm range and at 348 nm were assigned to LMCT benzoylacetate( $\pi^*$ )/hydroxamate ligand ( $\pi^*$ )  $\rightarrow$  vanadium ( $d\pi$ ) charge transfer spectra,  ${}^2E_g \leftarrow {}^2T_{2g}$  transition in octahedral geometry and MLCT vanadium ( $d\pi$ )  $\rightarrow$  benzoylacetate( $\pi^*$ )/hydroxamate ligand ( $\pi^*$ ) transitions respectively. In analogy to previous reports on non-oxovanadium(IV) complexes [2,31–33] the electronic spectra of bluish-violet solutions of  $\text{VCl}(\text{bzac})_2(\text{HL}^2)$  and  $\text{V}(\text{bzac})_2(\text{HL}^2)_2$  exhibited bands at 500–600 nm, 400–500 nm and  $\sim 315 \text{ nm}$  assigned to  ${}^2E_g \leftarrow {}^2T_{2g}$  transition in octahedral geometry, benzoylacetate/cinnamohydroxamate



**Scheme 1.** Schematic view of synthesis of bis(1-phenyl-1,3-butanedionato)non-oxovanadium(IV) hydroxamates (where  $n = 1, 2$ ).

**Table 1.** Analytical Data of non-oxovanadium(IV) complexes.

Complex (Molecular formula)	Color	Yield g (%)	Decomp. Temp. °C	Elemental Analysis					Molar cond. $\Lambda_m$ $\text{Scm}^2\text{s}^{-1}$	Magnetic moment, $\mu$ B.M.	Mol. Wt. Obs. (Calc.)
				V	Cl	C	H	N			
$[\text{VCl}(\text{bzac})_2(\text{HL}^1)]$ ( $\text{VC}_{28}\text{H}_{26}\text{O}_7\text{N}$ )(I)	Light green	1.05 (81)	107	8.55 (8.88)	6.13 (6.17)	58.36 (58.48)	4.39 (4.52)	2.16 (2.44)	4.24	1.72	574 (574.5)
$\text{V}(\text{bzac})_2(\text{HL}^1)_2$ ( $\text{VC}_{36}\text{H}_{34}\text{O}_{10}\text{N}_2$ )(II)	Bluish Violet	1.34 (84)	110	7.05 (7.23)	—	61.14 (61.27)	4.62 (4.82)	3.70 (3.97)	4.76	1.71	705 (705)
$\text{VCl}(\text{bzac})_2(\text{HL}^2)$ ( $\text{VC}_{27}\text{H}_{26}\text{O}_6\text{N}$ )(III)	Bluish Violet	1.13 (89)	115	8.47 (8.93)	6.18 (6.22)	60.72 (60.99)	4.41 (4.55)	2.17 (2.45)	4.85	1.75	570 (570.5)
$[\text{V}(\text{bzac})_2(\text{HL}^2)_2]$ ( $\text{VC}_{38}\text{H}_{34}\text{O}_8\text{N}_2$ )(IV)	Bluish Violet	1.30 (83)	112	7.03 (7.31)	—	65.34 (65.42)	4.53 (4.88)	3.96 (4.01)	5.12	1.73	697 (697)

ligand ( $\pi^*$ )  $\rightarrow$  vanadium ( $d\pi$ ) and MLCT vanadium ( $d\pi$ )  $\rightarrow$  benzoylacetate( $\pi^*$ ) /hydroxamate ligand ( $\pi^*$ ) transitions respectively.

### Mass spectra

The major FAB-MS peaks observed for the precursor  $VCl_2(bzac)_2$  and its derivatives with hydroxamate ligands are given in Table 2. Although the mass spectra of  $VCl_2(bzac)_2$  did not display any molecular ion peak, the fragment ions observed at  $m/e$  472, 411, 245, 205 and 534 corresponding to  $[VCl_2(bzac)_2 + CO]^+$ ,  $[VCl(bzac)_2 + 2H]^+$ ,  $[VCl(bzac) - 2H]^+$ ,  $[VCl_2(bzac) - C_6H_5]^+$  and  $[V(bzac)_3]^+$  respectively clearly showed the formation of precursor [34,35]. The most intense peak at  $m/e$  154 seemed to be that of the matrix.

The FAB-MS spectra of  $VCl(bzac)_2(HL^1)$  (I),  $V(bzac)_2(HL^1)_2$  (II),  $[VCl(bzac)_2(HL^2)]$  (III) and  $V(bzac)_2(HL^2)_2$  (IV) showed base peaks at 379, 154, 375 and 375 corresponding to  $[V(bzac)(HL^1) + H]^+$ , matrix,  $[V(bzac)(HL^2) + H]^+$  and  $[V(HL^2)_2]^+$  ions in respective complexes. The fragment ions occurring at  $m/e$  401, 423 and 461 may be attributed to  $[VO(HL^1)_2 + 2H]^+$ ,  $[VO(HL^1)_2 + Na + H]^+$ ,  $[VCl(HL^1)_2 + CH_3CO]^+$  in the case of complex (I). The fragment ions at 678 and 701 corresponding to  $[V(bzac)_2(HL^1)_2 - CO + H]^+$  and  $[V(bzac)_2(HL^1)_2 - 4H]^+$  appeared in complex (II).

The FAB-MS peaks observed at  $m/e$  467, 439 and 374 corresponded to  $(VCl(bzac)_2 + 2NHO - 3H)^+$ ,  $[VCl(bzac)(HL^2) + 2CH_3]^+$  and  $[V(HL^2)(bzac)]^+$  and the fragment ions at 730, 683 and 566 to  $[V(bzac)_2(HL^2)_2 + NHO + 2H]^+$ ,  $[V(bzac)_2(HL^2)_2 - CH_3 + H]^+$ ,  $[V(bzac)_2(HL^2) + NHO]^+$ , showed the formation of (III) and (IV) complexes respectively.

Based upon physicochemical and FTIR, UV-VIS and mass spectral data coupled with magnetic moment measurements, a distorted-octahedral geometry around vanadium may tentatively be proposed (Figures 3 to 6).

### Anti bacterial activity

The literature contains numerous reports concerned with the antimicrobial activity of ligands and their complexes against pathogenic bacteria and plant pathogenic fungi [36, 37]. Hence, in the present work, the precursor  $VCl_2(bzac)_2$ , ligands and newly synthesised complexes were tested *in vitro* for their antibacterial activity against *E. coli*, *S. aureus*, *P. mirabilis*, *P. aeruginosa*, *S. epidermidis* and *S. flexneri*. From the MIC values given in Table 3 it is quite apparent that both the ligands have similar antibacterial activity (62.5 – 250  $\mu$ g/ mL) for all bacterial species except that  $HL^2$  is more effective against *P. aeruginosa* than that of  $HL^1$ . The  $VCl_2(bzac)_2$  has been observed to inhibit the bacterial growth in a concentration range of 125–250  $\mu$ g/mL.

The newly synthesised complexes have been found to be more active (15.6 – 62.5  $\mu$ g/mL) than free ligands  $HL^1$  and  $HL^2$ . The non-chloro vanadium(IV) complexes (II) and (IV) have shown pronounced activity at a MIC of 15.6  $\mu$ g/mL against *S. epidermidis* and *S. flexneri* respectively which is higher than that of the commercial antibiotic streptomycin used as a standard for the comparison of results. These complexes

**Table 2.** Mass-spectral data of non-oxovanadium(IV) complexes.

Complex	Major fragment ions (m/e %)
$VCl_2(bzac)_2$	$[V(bzac)_3 + CH_3CO]^+$ (577, 30.55); $[VO(bzac)_3 + H]^+$ (551, 36.11); $[VO(bzac)_3 - H]^+$ (549, 36.11); $[V(bzac)_3]^+$ (534, 30.55); $[VCl_2(bzac)_2 + CO]^+$ (472, 58.33); $[VCl(bzac)_2 + 2H]^+$ (411, 13.88); $[VO(bzac)_2 + H]^+$ (390, 41.67); $[V(bzac)_2]^+$ (373, 22.22); $[VO_2(bzac) + H]^+$ (245, 77.77); $[VO(bzac)]^+$ (228, 50.00); $[VCl_2(bzac) - C_6H_5]^+$ (205, 8.33).
$[VCl(bzac)_2(HL^1)]$ (I)	$[V(bzac)_3]^+$ (534, 11.11); $[VCl(HL^1)_2 + CH_3CO]^+$ (461, 22.22); $[VO(HL^1)_2 + Na + H]^+$ (423, 8.33); $[VO(HL^1)_2 + 2H]^+$ (401, 13.88); $[VO(bzac)_2 + H]^+$ (390, 75.00); $[V(bzac)(HL^1) + H]^+$ (379, 100); $[VCl(HL^1)_2 - C_6H_5]^+$ (341, 11.11); $[VO_2(bzac) + H]^+$ (245, 66.66); $[VO(bzac)]^+$ (228, 58.33); $[HL^1 + CO - 2H]^+$ (192, 16.66).
$[V(bzac)_2(HL^1)_2]$ (II)	$[V(bzac)_2(HL^1)_2 - 4H]^+$ (701, 8.33); $[V(bzac)_2(HL^1)_2 - CO + H]^+$ (678, 8.33); $[V(bzac)_2(HL^1)_2 - CONHO - H]^+$ (645, 8.33); $[VO(HL^1)_2 + CH_3CO - H + NBA_1]^+$ (577, 19.44); $[VO(bzac)_3 + H]^+$ (551, 22.22); $[VO(bzac)_3 - H]^+$ (549, 19.44); $[VO(HL^1)_2 + CH_3CO - H]^+$ (441, 16.66); $[VO(bzac)_2 + H]^+$ (390, 63.88); $[VO(bzac)_2]^+$ (389, 47.22); $[V(bzac)_2]^+$ (373, 38.88); $[VO_2(bzac) + H]^+$ (245, 61.11); $[VO(bzac)]^+$ (228, 41.66); $[HL^1 + CO - 3H]^+$ (191, 27.77).
$[VCl(bzac)_2(HL^2)]$ (III)	$[VO(bzac)_3 + H]^+$ (551, 19.44); $[VCl(bzac)_2 + 2NHO - 3H]^+$ (467, 11.11); $[VCl(bzac)(HL^2) + 2CH_3]^+$ (439, 11.11); $[VCl(bzac)_2 + 2H]^+$ (411, 19.44); $[VO(bzac)(HL^2) + H]^+$ (390, 36.11); $[V(bzac)(HL^2) + H]^+$ (375, 100); $[V(bzac)(HL^2)]^+$ (374, 27.77); $[VCl(bzac) + CO - H]^+$ (273, 8.33); $[VO_2(bzac) + H]^+$ (245, 33.33); $[VO(bzac)]^+$ (228, 16.66); $[VCl(bzac) - CO]^+$ (219, 5.55); $[HL^2 + CO + H]^+$ (191, 11.11).
$[V(bzac)_2(HL^2)_2]$ (IV)	$[V(bzac)_2(HL^2)_2 + NHO + 2H]^+$ (730, 11.11); $[V(bzac)_2(HL^2)_2 - CH_3 + H]^+$ (683, 13.88); $[V(bzac)_2(HL^2) + NHO]^+$ (566, 13.88); $[V(bzac)_2(HL^2) + 2CH_3]^+$ (565, 11.11); $[V(bzac)_2(HL^2) - C_6H_5 - 2H]^+$ (456, 8.33); $[V(bzac)_2(HL^2) - C_6H_5CO - 2H]^+$ (428, 19.44); $[VO(bzac)_2 + H]^+$ (390, 68.88); $[V(bzac)(HL^2) + H]^+$ (375, 100); $[V(bzac)_2 - CH_3]^+$ (358, 27.78); $[VO(HL^2) + NHO + H]^+$ (261, 8.33); $[VO_2(bzac) + H]^+$ (245, 66.66); $[VO(bzac)]^+$ (228, 58.33); $[HL^2 + CO + H]^+$ (191, 13.88).

$NBA_1 = 136$ ,  $NBA_2 = 137$ ,  $NBA_3 = 154$ ,  $NBA_4 = 289$  and  $NBA_5 = 307$

are also more effective against *P. mirabilis* compared to complexes (I) and (III). On the other hand, complexes (I) and (III) have shown higher antibacterial activity at MIC 31.2  $\mu$ g/mL

against the bacteria *S. flexneri* and *P. aeruginosa* respectively relative to complexes (II) and (IV). It is worth pointing out here that although all the complexes have shown similar antibacterial activity against *E. coli* and *S. aureus*, this observed trend is not the same for all these bacteria. The explanation to this observation may be ascribed to different liposolubility of complexes for different bacteria [38,39]. The enhancement in activity may be attributed to the coordination of hydroxamate ligands to the metal ion and an efficient diffusion of the metal complexes into bacterial cells [40,41]. The antibacterial activity of these complexes was compared with commercial antibiotic (streptomycin) and promising bioactivity was shown by the complexes studied.

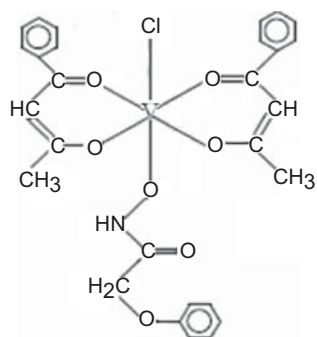


Figure 3. Proposed structure of  $[VCl(bzac)_2(HL^1)]$

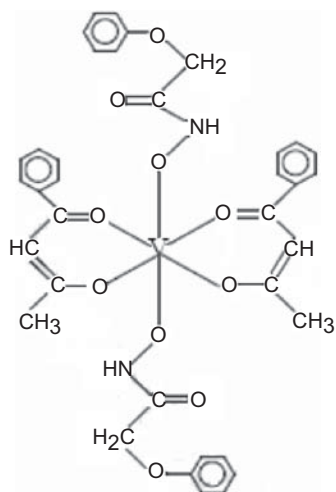


Figure 4. Proposed structure of  $[V(bzac)_2(HL^1)_2]$

## Conclusion

The  $VCl_2(bzac)_2$  has been exploited as precursor for the synthesis of mixed-ligand non-oxovanadium(IV) complexes derived from phenoxyacetohydroxamate and cinnamohydroxamate ligands. IR spectral studies depicted the unidentate nature of hydroxamate ligands in regard to coordination through the hydroxylamine oxygen (-NHO) only is displayed. The complexes have shown promising antibacterial activity against all the bacteria tested.

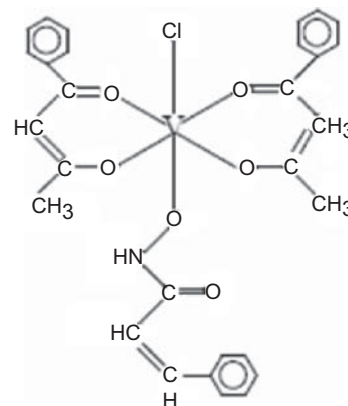


Figure 5. Proposed structure of  $[VCl(bzac)_2(HL^2)]$

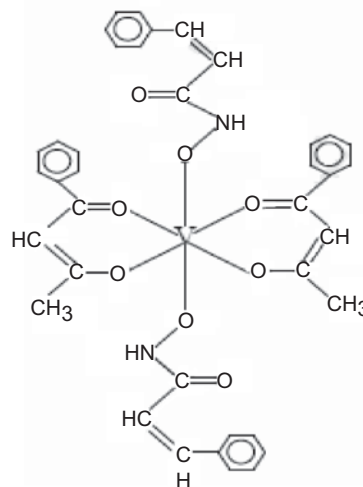


Figure 6. Proposed structure of  $[V(bzac)_2(HL^2)_2]$

Table 3. Antibacterial activity of ligands and vanadium (IV) complexes by MIC method in  $\mu\text{g/mL}$ .

Compound	<i>E. coli</i>	<i>S. aureus</i>	<i>P. mirabilis</i>	<i>P. aeruginosa</i>	<i>S. epidermidis</i>	<i>S. flexneri</i>
$C_6H_5OCH_2C(O)NHOK (HL^1)$	125	250	125	125	125	62.5
$C_6H_5CH=CHC(O)NHOK(HL^2)$	125	250	125	62.5	125	62.5
$VCl_2(bzac)_2$	250	250	125	250	125	125
$[VCl(bzac)_2(HL^1)]$ (I)	31.2	62.5	62.5	62.5	62.5	31.2
$[V(bzac)_2(HL^1)_2]$ (II)	31.2	62.5	31.2	62.5	15.6	62.5
$[VCl(bzac)_2(HL^2)]$ (III)	31.2	62.5	62.5	31.2	125	31.2
$[V(bzac)_2(HL^2)_2]$ (IV)	31.2	62.5	31.2	62.5	125	15.6
Streptomycin	31.2	62.5	62.5	31.2	62.5	31.2

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## Declaration of interest

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