

Scientific paper

Synthesis, Crystal Structures and Antimicrobial Activity of Oxidovanadium(V) Complexes with Hydrazone and Pyrone Ligands

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Abstract

A pair of oxidovanadium(V) complexes, [VOLL¹] (1) and [VOLL²] (2), where L is the dianionic form of the hydrazone ligand *N*²-(5-chloro-2-hydroxybenzylidene)pivalohydrazide (H₂L), L¹ and L² are the deprotonated forms of 3-hydroxy-2-methyl-4*H*-pyran-4-one (HL¹; maltol) and 2-ethyl-3-hydroxy-4*H*-pyran-4-one (HL²; ethyl maltol), respectively, have been prepared and characterized by elemental analyses, IR and UV-Vis spectroscopy, and single-crystal X-ray crystallographic determination. The V atoms in the complexes are in octahedral coordination, with the hydrazone ligand coordinated to the V atoms through the phenolate O, imino N and enolate O atoms, and with the pyrone ligands coordinated to the V atoms through two O atoms. The effect of the compounds on the antimicrobial activity against *Staphylococcus aureus*, *Escherichia coli*, and *Candida albicans* was studied.

Keywords: Hydrazone; Pyrone; Vanadium complex; Crystal structure; Antimicrobial activity

1. Introduction

Schiff bases are important ligands in coordination chemistry.¹ In recent years, metal complexes of Schiff bases have attracted remarkable attention due to their versatile biological activity, such as antifungal, antibacterial and antitumor.² Schiff base complexes derived from salicylaldehyde and its derivatives with primary amines, bearing the NNO, NNS, NOO or NSO donor sets, have particular biological activities.³ Hydrazones, bearing the typical functional group CH=N–NH–C(O), are a kind of Schiff base compounds. Hydrazones show interesting and remarkable biological activities, especially antimicrobial application.⁴ Pyrone compounds are bidentate ligands in various metal complexes.⁵ Most complexes with pyrone ligands have interesting biological properties.⁶ The search of literature indicates that vanadium complexes with pivalol substituted hydrazones are limited. In the present work, two oxidovanadium(V) complexes, [VOLL¹] (1) and [VOLL²] (2), where L is the dianionic form of the hydrazone ligand *N*²-(5-chloro-2-hydroxybenzylidene)pivalohydrazide (H₂L), L¹ and L² are the deprotonated form of 3-hydroxy-2-methyl-4*H*-pyran-4-one (HL¹; maltol)

and 2-ethyl-3-hydroxy-4*H*-pyran-4-one (HL²; ethyl maltol), respectively, are reported.

2. Experimental

2.1. Material and Methods

5-Chlorosalicylaldehyde, pivalohydrazide, 3-hydroxy-2-methyl-4*H*-pyran-4-one (maltol) and 2-ethyl-3-hydroxy-4*H*-pyran-4-one (ethyl maltol) were purchased from Fluka. Other reagents and solvents were analytical grade and used without further purification. Elemental (C, H, and N) analyses were made on a Perkin-Elmer Model 240B automatic analyser. Infrared (IR) spectra were recorded on an IR-408 Shimadzu 568 spectrophotometer. UV-Vis spectra were recorded on a Lambda 35 spectrometer. X-ray diffraction was carried out on a Bruker SMART 1000 CCD area diffractometer.

2.2. Synthesis of H₂L

5-Chlorosalicylaldehyde (0.156 g, 1.0 mmol) and pivalohydrazide (0.116 g, 1.0 mmol) were dissolved in

methanol (30 mL). The mixture was stirred at ambient temperature for 1 h to give a colorless solution. The solvent was removed to give colorless solid product of H₂L, which was re-crystallized from ethanol. The yield is 93%. IR data (KBr, cm⁻¹): 1623 (C=N). UV-Vis data in methanol [10⁻³ mol L⁻¹; λ_{max} (nm), ε (L mol⁻¹ cm⁻¹): 230, 15,570; 290, 14,310; 366, 10,205. Anal. Calcd. (%) for C₁₂H₁₅ClN₂O₂: C, 56.6; H, 5.9; N, 11.0. Found (%): C, 56.4; H, 6.0; N, 11.1.

2. 3. Synthesis of Complex 1

5-Chlorosalicylaldehyde (15.6 mg, 0.10 mmol) was dissolved in methanol (10 mL) and methanol solution (10 mL) of pivalohydrazide (11.6 mg, 0.10 mmol) was added dropwise. Mixture was stirred at ambient temperature for 1 h. Then, 3-hydroxy-2-methyl-4*H*-pyran-4-one (12.6 mg, 0.10 mmol) and VO(acac)₂ (26.5 mg, 0.10 mmol) dissolved in methanol (10 mL) was added. The mixture was stirred for 1 h at ambient temperature to give a deep brown solution. Brown block-shaped single crystals suitable for X-ray diffraction were formed by slow evaporation of the solution in air for several days. The yield is 45%, 20.0 mg (based on V). IR data (KBr, cm⁻¹): 1612 (C=N), 973 (V=O). UV-Vis data in methanol [10⁻³ mol L⁻¹; λ_{max} (nm), ε (L mol⁻¹ cm⁻¹): 275, 16,230; 330, 5,230; 470, 1,575. Anal. Calcd. (%) for C₁₈H₁₈ClN₂O₆V: C, 48.6; H, 4.1; N, 6.3. Found (%): C, 48.7; H, 4.0; N, 6.2.

2. 4. Synthesis of Complex 2

Complex 2 was prepared by the same method as described for complex 1, with 3-hydroxy-2-methyl-4*H*-pyran-4-one replaced by 3-hydroxy-2-ethyl-4*H*-pyran-4-one (14.0 mg, 0.10 mmol). The yield is 51%, 23.4 mg (based on V). IR data (KBr, cm⁻¹): 1612 (C=N), 980 (V=O). UV-Vis data in methanol [10⁻³ mol L⁻¹; λ_{max} (nm), ε (L mol⁻¹ cm⁻¹): 275, 17,530; 330, 6,020; 470, 4,225. Anal. Calcd. (%) for C₁₉H₂₀ClN₂O₆V: C, 49.7; H, 4.4; N, 6.1. Found (%): C, 49.9; H, 4.3; N, 6.2.

2. 5. X-Ray Structure Determination

Data were collected from selected crystals mounted on glass fibres. The data for the two complexes were processed with SAINT⁷ and corrected for absorption using SADABS.⁸ Multi-scan absorption corrections were applied with ψ-scans.⁹ The structures were solved by direct methods using the program SHELXS-97 and were refined by full-matrix least-squares techniques on *F*² using anisotropic displacement parameters. The structures were refined by SHELXL-97 program.¹⁰ All hydrogen atoms were placed at the calculated positions. Idealized H atoms were refined with isotropic displacement parameters set to 1.2 (1.5 for methyl groups) times the equivalent isotropic *U* values of the parent atoms. The structure of complex 1 contains large solvent accessible void which might accommodate a disordered methanol molecule. The crystallo-

Table 1. Crystallographic data and refinement parameters for complexes 1 and 2

Parameters	1	2
Habit, color	Block, brown	Block, brown
Molecular formula	C ₁₈ H ₁₈ ClN ₂ O ₆ V	C ₁₉ H ₂₀ ClN ₂ O ₆ V
Formula weight	444.73	458.76
Temperature, K	298(2)	298(2)
Radiation (λ, Å)	MoK _α (0.71073)	MoK _α (0.71073)
Crystal system	Hexagonal	Triclinic
Space group	<i>R</i> -3	<i>P</i> -1
Unit cell dimensions:		
<i>a</i> , Å	40.9930(12)	10.2988(6)
<i>b</i> , Å	40.9930(12)	10.5588(7)
<i>c</i> , Å	7.3018(9)	11.3944(7)
α, °	90	63.0460(10)
β, °	90	75.2270(10)
γ, °	120	71.1960(10)
<i>V</i> , Å ³	10626.2(14)	1036.77(11)
<i>Z</i>	18	2
ρ _{calcd} , g cm ⁻³	1.251	1.470
<i>F</i> (000)	4104	472
Reflections collected	18661	5504
Independent reflections	4387	3812
Data/parameters	2809/253	3123/266
Final <i>R</i> indices (<i>I</i> > 2σ(<i>I</i>))	<i>R</i> ₁ = 0.0512, <i>wR</i> ₂ = 0.1215	<i>R</i> ₁ = 0.0393, <i>wR</i> ₂ = 0.1100
<i>R</i> indices (all data)	<i>R</i> ₁ = 0.0951, <i>wR</i> ₂ = 0.1437	<i>R</i> ₁ = 0.0497, <i>wR</i> ₂ = 0.1188
Goodness-of-fit on <i>F</i> ²	1.050	1.044

Table 2. Selected bond lengths (Å) and bond angles (°) for complexes **1** and **2**

1			
V1–O1	1.8563(18)	V1–O2	1.9256(19)
V1–O3	1.8715(16)	V1–O4	2.2710(18)
V1–O6	1.5782(19)	V1–N1	2.0905(19)
O6–V1–O1	97.55(10)	O6–V1–O3	97.92(9)
O1–V1–O3	107.93(8)	O6–V1–O2	101.02(10)
O1–V1–O2	152.43(9)	O3–V1–O2	89.55(8)
O6–V1–N1	95.84(9)	O1–V1–N1	83.99(8)
O3–V1–N1	160.34(8)	O2–V1–N1	74.04(7)
O6–V1–O4	173.68(9)	O1–V1–O4	81.16(8)
O3–V1–O4	76.72(7)	O2–V1–O4	82.44(7)
N1–V1–O4	90.19(7)		
2			
V1–O1	1.848(3)	V1–O2	1.927(2)
V1–O3	1.863(2)	V1–O4	2.258(2)
V1–O6	1.582(3)	V1–N1	2.098(3)
O6–V1–O1	100.45(13)	O6–V1–O3	97.37(12)
O1–V1–O3	96.71(10)	O6–V1–O2	94.86(13)
O1–V1–O2	155.01(11)	O3–V1–O2	100.81(10)
O6–V1–N1	100.20(12)	O1–V1–N1	83.22(11)
O3–V1–N1	162.15(11)	O2–V1–N1	74.66(11)
O6–V1–O4	172.57(12)	O1–V1–O4	85.42(11)
O3–V1–O4	77.29(9)	O2–V1–O4	81.25(10)
N1–V1–O4	84.93(10)		

graphic data for the complexes are listed in Table 1, selected bond lengths and bond angles for complexes **1** and **2** are given in Table 2.

2. 6. Antimicrobial Assay

Qualitative determination of antimicrobial activity was done using the disk diffusion method as described in the literature.¹¹ Suspensions in sterile peptone water from 24-h cultures of microorganisms were adjusted to 0.5 McFarland. Muller-Hinton Petri dishes of 90 mm were inoculated using these suspensions. Paper disks (6 mm in diameter) containing 10 µL of the substance to be tested (at a concentration of 2048 µg mL⁻¹ in DMSO) were placed in a circular pattern in each inoculated plate. Incubation of the plates was done at 37 °C for 18–24 h. Reading of the results was done by measuring the diameters of the inhibition zones generated by the test substance. Tetracycline was used as a reference substance. Determination of MIC was done using the serial dilutions in liquid broth method. The materials used were 96-well plates, suspensions of microorganism (0.5 McFarland), Muller-Hinton broth (Merck) and stock solutions of each substance to be tested (2048 µg mL⁻¹ in DMSO). The following concentrations of the substances to be tested were obtained in the 96-well plates: 1024, 512, 256, 128, 64, 32, 16, 8, 4 and 2 µg mL⁻¹. After incubation at 37 °C for 18–24 h, the MIC for each tested substance was determined by microscopic observation of

microbial growth. It corresponds to the well with the lowest concentration of the tested substance where microbial growth was clearly inhibited.

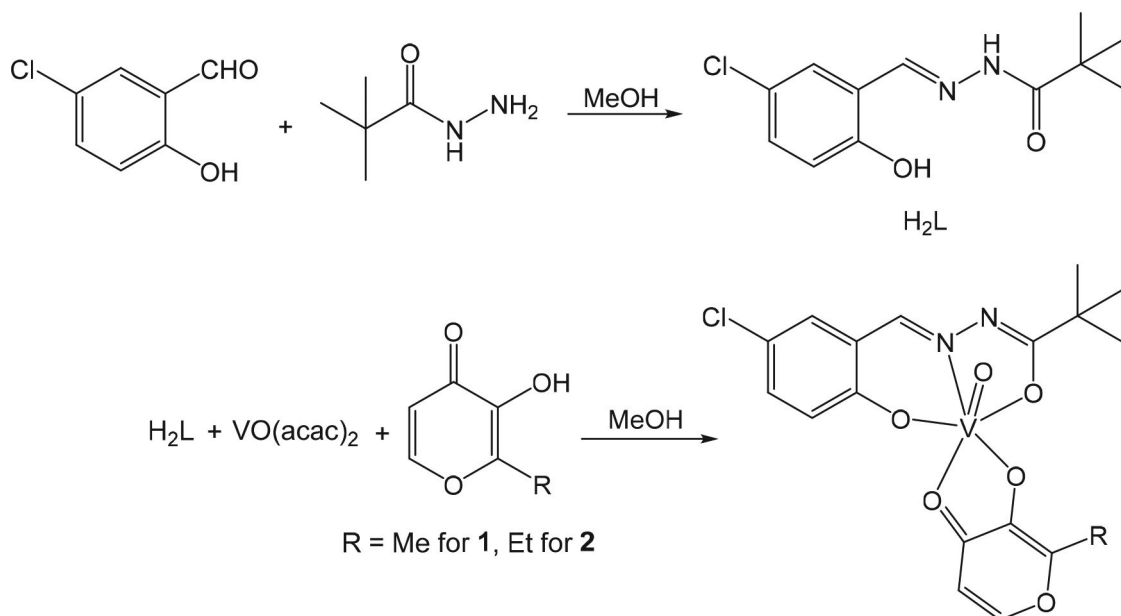
3. Results and Discussion

3. 1. Chemistry

The hydrazone compound H₂L was prepared by the condensation of equimolar quantities of 5-chlorosalicylaldehyde with pivalohydrazide in methanol. Complexes **1** and **2** were readily synthesized by reaction of the hydrazone with VO(acac)₂ in methanol in the presence of 3-hydroxy-2-methyl-4H-pyran-4-one and 2-ethyl-3-hydroxy-4H-pyran-4-one, respectively (Scheme 1). All the compounds are very stable at room temperature in the solid state, and soluble in common organic solvents, such as methanol, ethanol, and acetonitrile. The results of the elemental analyses are in accord with the composition suggested for the Schiff base and the complexes.

3. 2. IR and UV-Vis Spectra

For the IR spectrum of H₂L (Fig. S1), the typical band indicative of the azomethine group was observed at 1623 cm⁻¹, while in the complexes (Figs. S2 and S3), it was observed at 1612 cm⁻¹. The weak absorption at 3450 cm⁻¹ for H₂L can be attributed to the vibration of O–H group. The bands indicative of the V=O groups of the complexes



Scheme 1. The synthetic procedure of the complexes.

are observed at 973–980 cm^{-1} . UV-Vis spectra of H_2L and the complexes were measured in methanol. In the spectrum of the hydrazone (Fig. S4), the band centered at 365 nm is attributed to the azomethine chromophore $\pi-\pi^*$ transition. The band at higher energy (292 nm) is associated with the benzene $\pi-\pi^*$ transition. In the spectra of the complexes (Figs. S5 and S6), the bands at 470 nm are attributed to the intramolecular charge transfer transitions from the p_π orbital on the phenolate O to the empty d orbitals of the V atoms.¹²

3. 3. Structure Description of the Complexes

The molecular structures of complexes **1** and **2** are shown in Figs. 1 and 2, respectively. The V atoms are coordi-

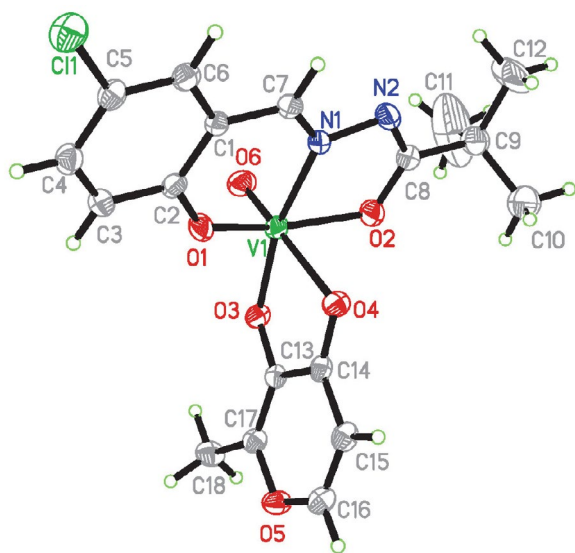


Fig. 1. Perspective view of complex **1** with 30% probability thermal ellipsoids.

ned by one hydrazone ligand, one pyrone ligand, and one oxo O group. The hydrazone ligand coordinated to the V atom through the phenolate O, imino N, and enolate hydroxyl O atoms. The pyrone ligands are coordinated to the V atoms through the deprotonated hydroxyl O and carbonyl O atoms. The V atoms are in octahedral coordination, with the three donor atoms of the hydrazone ligand, and the hydroxyl O atom of the pyrone ligand defining the equatorial plane, and with the carbonyl O atom of the pyrone ligand, and the oxo O group occupying the axial positions. In both complexes, the corresponding bond distances around the V atoms are comparable to each other, and also similar to the corresponding bonds observed in the similar vanadium complexes with hydrazones or pyrones.¹³ The *cis* and *trans* coordinate bond angles in the equatorial planes are range from 74.66(11) to 100.81(10) $^\circ$ and from 155.01(11) to 162.15(11) $^\circ$ for complex **1**, and

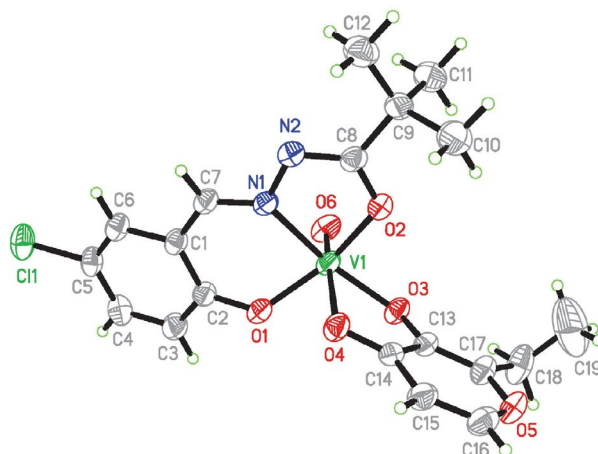


Fig. 2. Perspective view of complex **2** with 30% probability thermal ellipsoids.

from 74.04(7) to 107.93(8)° and from 152.43(9) to 160.34(8)° for complex **2**, respectively. In addition, the values for the axial *trans* O–V–O angles are 172.57(12)° and 173.68(9)°, respectively, for complexes **1** and **2**. All the above bond values indicating the distortion of the octahedral coordination from ideal geometry.

3. 4. Antimicrobial Activity

The antimicrobial activity results are summarized in Table 3. A comparative study of minimum inhibitory concentration (MIC) values of the hydrazone and the complexes indicates that the complexes have better activity than the free hydrazone. Generally, this is caused by the greater lipophilic nature of the complexes than the ligand. Such increased activity of the metal chelates can be explained on the basis of chelating theory.¹⁴ On chelating, the polarity of the metal atoms will be reduced to a greater extent due to the overlap of the ligand orbital and partial sharing of positive charge of the metal atoms with donor atoms. Further, it increases the delocalization of *p*-electrons over the whole chelate ring and enhances the lipophilicity of the complexes. This increased lipophilicity enhances the penetration of the complexes into lipid membrane and blocks the metal binding sites on enzymes of microorganisms.

From the results, it is obvious that the two complexes have higher antibacterial and antifungal activities against *Staphylococcus aureus*, *Escherichia coli*, and *Candida albicans* when compared to the free hydrazone. This phenomenon is in accordance with those reported in literature.¹⁵ The two complexes have in general the same activities against all the bacteria and fungi strains. The complexes have strong activity against *Escherichia coli*, with MIC value of 1.0 µg/mL, which is superior to tetracycline. The complexes have strong activity against *Staphylococcus aureus*, yet, they are weaker than tetracycline. It is interesting that both complexes have medium activity against *Candida albicans*, which is rarely seen in metal complexes.¹⁶

Table 3. MIC values (µg/mL) for the antimicrobial activities of the tested compounds

Compounds	<i>Staphylococcus aureus</i>	<i>Escherichia coli</i>	<i>Candida albicans</i>
H ₂ L	64	16	> 512
1	2.0	1.0	64
2	2.0	1.0	64
Tetracycline	0.32	2.12	> 1024

4. Material

CCDC 1896231 for **1**, and 1896233 for **2** contain the supplementary crystallographic data for this paper. These

data can be obtained free of charge at <http://www.ccdc.cam.ac.uk/const/retrieving.html> or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44(0)1223-336033 or email: deposit@ccdc.cam.ac.uk.

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Povzetek

Sintetizirali smo dva oksidovanadijeva(V) kompleksa [VOLL¹] (**1**) in [VOLL²] (**2**), kjer je L dianion hidrazona *N*²-(5-kloro-2-hidroksibenziliden)pivalohidrazida (H₂L), L¹ in L² sta deprotonirani obliki 3-hidroksi-2-metil-4*H*-piran-4-ona (HL¹; maltol) in 2-etil-3-hidroksi-4*H*-piran-4-ona (HL²; etil maltol), ter ju okarakterizirali z elementno analizo, IR in UV-Vis spektroskopijo in monokristalno rentgensko analizo. V kompleksih ima vanadijev atom oktaedrično koordinacijo s hidrazonskim ligandom koordiniranim na V atom preko fenolatnega O, iminskega N in enolatnega O atoma ter s pirovskim ligandom koordiniranim preko dveh O atomov. Določili smo tudi protimikrobno aktivnost obeh spojin proti *Staphylococcus aureus*, *Escherichia coli* in *Candida albicans*.