Scientific paper

Synthesis, Characterization and X-Ray Crystal Structures of Oxidovanadium(V) Complexes Derived from *N*'-(2-Hydroxy-5-methylbenzylidene)-4-methylbenzohydrazide with Antibacterial Activity

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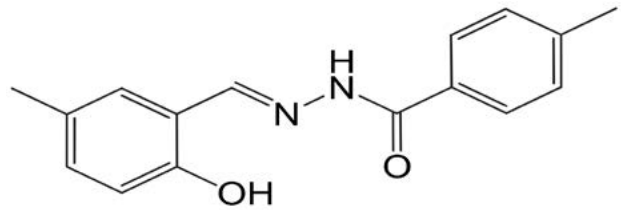
Abstract

A dinuclear oxidovanadium(V) complex $[V_2O_2L_2(OMe)_2]$ (**1**) was synthesized from *N*'-(2-hydroxy-5-methylbenzylidene)-4-methylbenzohydrazide (H_2L) and $VO(acac)_2$ in MeOH. Reaction of complex **1** with 3-hydroxy-2-methyl-4-pyrone (HL') afforded a mononuclear oxidovanadium(V) complex [VOLL'] (**2**). The hydrazone and both complexes were characterized by IR, UV and ^{1}H NMR spectroscopy, as well as X-ray single crystal determination. X-ray powder diffraction of the complexes was performed. The V atoms in the two complexes are in octahedral coordination. The molecules of complex **2** are linked through non-classical hydrogen bonds of type C–H···O to form one-dimensional chains running along the *a* axis. The biological assay indicates that the complexes have good antimicrobial activities on the bacteria strains *P. aeroginosa*, *S. aureus*, *B. subtilis* and *E. coli*.

Keywords: Hydrazone; Oxidovanadium complex; X-Ray crystal structure; Antibacterial activity.

1. Introduction

Vanadium compounds have received considerable attention for their various biological properties like normalizing the blood glucose levels and modeling haloperoxidases.[1] Hydrazones are a special kind of Schiff bases, which possess the characteristic functional group CH=N–NH–C(O). The compounds are well known for their facile synthesis and excellent coordinate capability to a number of metal ions. Hydrazones and metal complexes have been widely studied on their broadly biological applications.[2] Recently, we have reported the structures and biological activities of some vanadium complexes.[3] The solvents such as MeOH and EtOH usually coordinate to the V atoms through neutral or deprotonated form as terminal ligands.[4] Interestingly, they can act as bridging ligands to form dinuclear complexes.[5] When administered with bidentate ligands, it can obtain vanadium complexes with mixed ligands.[6] Maltol is used as food additive, flavor and fragrance. Maltol complexes of vanadium can regulate alkaline phosphatase activity and osteoblast-like cell growth.[7] In this paper, a new dinuclear oxidovanadium(V) complex $[V_2O_2L_2(OMe)_2]$ (**1**) and a new mononuclear oxidovanadium(V) complex [VOLL'] (**2**), where H_2L is *N*'-(2-hydroxy-5-methylbenzylidene)-4-methylbenzohydrazide (Scheme 1), and HL' is maltol, are presented.



Scheme 1. H_2L

2. Experimental

2. 1. Materials and Measurements

5-Methylsalicylaldehyde, 4-methylbenzohydrazide and maltol were purchased from Sigma-Aldrich

and used as received. Solvents and other reagents were commercial obtained. Elemental analyses for C, H and N were performed with a Perkin-Elmer elemental analyzer. IR spectra were recorded on a Nicolet AVATAR 360 spectrometer as KBr pellets. ^{1}H NMR spectrum was carried out on a Bruker 500 MHz instrument. Powder X-ray diffraction was performed with a Bruker D8 Advance X-ray diffractometer using Cu Kα radiation (λ = 1.548 Å) generated at 40 kV and 40 mA. The powder XRD spectra were recorded in a 2θ range of 2–50° using a 1D Lynxeye detector under ambient conditions. X-ray single crystal structures for the complexes were collected at 298(2) K using a Bruker D8 VENTURE PHOTON diffractometer with Mo*K*α radiation (λ = 0.71073 Å).

2. 2. Synthesis of H_2L

5-Methylsalicylaldehyde (1.0 mmol, 0.14 g) and 4-methylbenzohydrazide (1.0 mmol, 0.15 g) were respectively dissolved in 20 mL MeOH and mixed together. The mixture was reflux for 20 min give a colorless solution. The solvent was removed by distillation to obtain white solid, which was re-crystallized from MeOH to give colorless crystalline product. Yield: 0.24 g (89%). Analysis: Found: C 71.45%, H 6.12%, N 10.53%. Calculated for $C_{16}H_{16}N_2O_2$: C 71.62%, H 6.01%, N 10.44%. IR data (KBr, cm^{-1}): *ν* 3439 (m, OH), 3293 and 3205 (w, NH), 1648 (s, C=O), 1617 (s, C=N). UV-Vis in MeOH (λ, nm (ε, L mol^{-1} cm^{-1})): 240 (23,750), 290 (32,022), 300 (31,120), 333 (17,020), 396 (4,250). ^{1}H NMR (d_6-DMSO, ppm) δ 12.04 (s, 1H, OH), 11.09 (s, 1H, NH), 8.61 (s, 1H, CH=N), 7.88 (d, 2H, ArH), 7.36 (d, 3H, ArH), 7.14 (d, 1H, ArH), 6.86 (d, 1H, ArH), 2.41 (s, 3H, CH_3), 2.27 (s, 3H, CH_3).

2. 3. Synthesis of $[V_2O_2L_2(OMe)_2]$ (1)

[VO(acac)$_2$] (0.10 mmol, 26 mg) and H_2L (0.10 mmol, 27 mg) were respectively dissolved in MeOH (20 mL) and mixed together. The mixture was stirred at room temperature for 30 min to give a deep brown solution. The mixture was filtered and with the filtration allowed to slow evaporate for a week. Brown block like single crystals were formed. The crystals were isolated by filtration. Yield: 19 mg (53%). Analysis: Found: C 55.87%, H 4.78%, N 7.58. Calculated for $C_{34}H_{34}N_4O_8V_2$: C 56.05%, H 4.70%, N 7.69%. IR data (KBr, cm^{-1}): *ν* 1610 (s, C=N), 961 (V=O). UV-Vis in MeOH (λ, nm (ε, L mol^{-1} cm^{-1})): 227 (21,320), 268 (23,215), 325 (15,630), 406 (4,655). Molar conductivity in MeOH at concentration of 10^{-4} mol L^{-1}: 26 Ω^{-1} cm^2 mol^{-1}.

2. 4. Synthesis of [VOLL'] (2)

Maltol (0.20 mmol, 26 mg) was dissolved in MeOH (15 mL) and added dropwise to a 10 mL methanolic solution of complex **1** (0.10 mmol, 73 mg). The mixture was stirred at room temperature for 30 min to give a deep brown solution. The mixture was filtered and with the filtration allowed to slow evaporate for a week. Brown block like single crystals were formed. The crystals were isolated by filtration. Yield: 43 mg (47%). Analysis: Found: C 57.78%, H 4.10%, N 6.19. Calculated for $C_{22}H_{19}N_2O_6V$: C 57.65%, H 4.18%, N 6.11%. IR data (KBr, cm^{-1}): *ν* 1608 (s, C=N), 970 (V=O). UV-Vis in MeOH (λ, nm (ε, L mol^{-1} cm^{-1})): 221 (22,175), 273 (28,190), 327 (14,370), 410 (3,575). Molar conductivity in MeOH at concentration of 10^{-4} mol L^{-1}: 35 Ω^{-1} cm^2 mol^{-1}.

2. 5. X-ray Crystallography

The collected data by the diffractometer were reduced with SAINT.[8] Multi-scan absorption corrections were applied with SADABS.[9] Structures of both complexes were solved by direct method and refined by full-matrix least-squares method against F^2 with SHELXTL.[10] The non-hydrogen atoms were refined anisotropically, while the hydrogen atoms were placed in idealized positions and constrained to ride on their parent atoms. The crystallographic data are listed in Table 1. Coordinate bond lengths and bond angles are summarized in Table 2.

Table 1. Crystallographic data and refinement parameters for the complexes

	1	**2**
Chemical formula	$C_{34}H_{34}N_4O_8V_2$	$C_{22}H_{19}N_2O_6V$
M_r	728.53	458.33
Crystal color, habit	Brown, block	Brown, block
Crystal system	Monoclinic	Monoclinic
Space group	$P2_1/n$	$P2_1/c$
Unit cell parameters		
a (Å)	8.7163(12)	14.319(2)
b (Å)	20.5798(16)	10.295(2)
c (Å)	9.5130(12)	14.335(2)
β (°)	105.194(2)	114.437(2)
V (Å^3)	1646.8(3)	1923.9(5)
Z	2	4
D_{calc} (g cm^{-3})	1.469	1.582
T (K)	298(2)	298(2)
μ (mm^{-1})	0.626	0.561
F(000)	752	944
Number of unique data	3049	3228
Number of observed data [$I > 2\sigma(I)$]	1597	1395
Number of parameters	220	283
Number of restraints	0	0
R_1, wR_2 [$I > 2\sigma(I)$]	0.0537, 0.0982	0.0762, 0.1867
R_1, wR_2 (all data)	0.1294, 0.1226	0.1491, 0.2323
Goodness of fit on F^2	0.977	0.909

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

1			
V1–O1	1.823(3)	V1–O2	1.933(3)
V1–O3	1.581(3)	V1–O4	1.827(3)
V1–N1	2.093(3)	V1–O4A	2.373(3)
O3–V1–O1	101.18(14)	O3–V1–O4	102.17(13)
O1–V1–O4	106.12(12)	O3–V1–O2	99.38(13)
O1–V1–O2	151.25(12)	O4–V1–O2	88.81(11)
O3–V1–N1	96.84(14)	O1–V1–N1	83.55(13)
O4–V1–N1	156.35(13)	O2–V1–N1	74.21(12)
O3–V1–O4A	176.23(12)	O1–V1–O4A	81.37(11)
O4–V1–O4A	74.36(11)	O2–V1–O4A	79.23(10)
N1–V1–O4A	86.18(11)		
2			
V1–O1	1.865(4)	V1–O2	1.800(4)
V1–O3	1.531(4)	V1–O4	2.207(4)
V1–O5	1.831(4)	V1–N2	2.068(5)
O3–V1–O1	96.7(2)	O3–V1–O5	99.8(2)
O1–V1–O5	95.6(2)	O3–V1–O2	100.8(2)
O1–V1–O2	154.0(2)	O5–V1–O2	100.0(2)
O3–V1–N2	99.4(2)	O1–V1–N2	75.4(2)
O5–V1–N2	159.6(2)	O2–V1–N2	83.0(2)
O3–V1–O4	175.9(2)	O1–V1–O4	81.0(2)
O5–V1–O4	77.1(2)	O2–V1–O4	82.5(2)
N2–V1–O4	83.4(2)		

Symmetry operation for A: 1 – *x*, 1 – *y*, 1 – *z*.

3. Results and Discussion

Complex **1** was synthesized by reaction $[VO(acac)_2]$ with H_2L in MeOH (Scheme 2). Reaction of complex **1** with maltol afforded complex **2** (Scheme 2). Complex **2** can also be directly synthesized by reaction $[VO(acac)_2]$, H_2L and maltol in MeOH. The V^{IV} in $[VO(acac)_2]$ was oxidized to V^{V} by oxygen in air during the reaction and crystallization. Molar conductivities of the complexes at the concentration of 10^{-4} mol L^{-1} are 26–35 Ω^{-1} cm^2 mol^{-1}, prove them as non-electrolytes.[11] The experimental and simulated powder XRD patterns of the complexes are shown in Figures 1 and 2, which confirm the purity of the bulk materials.

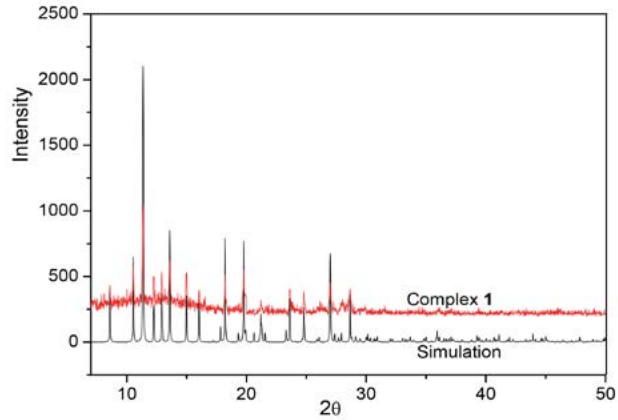
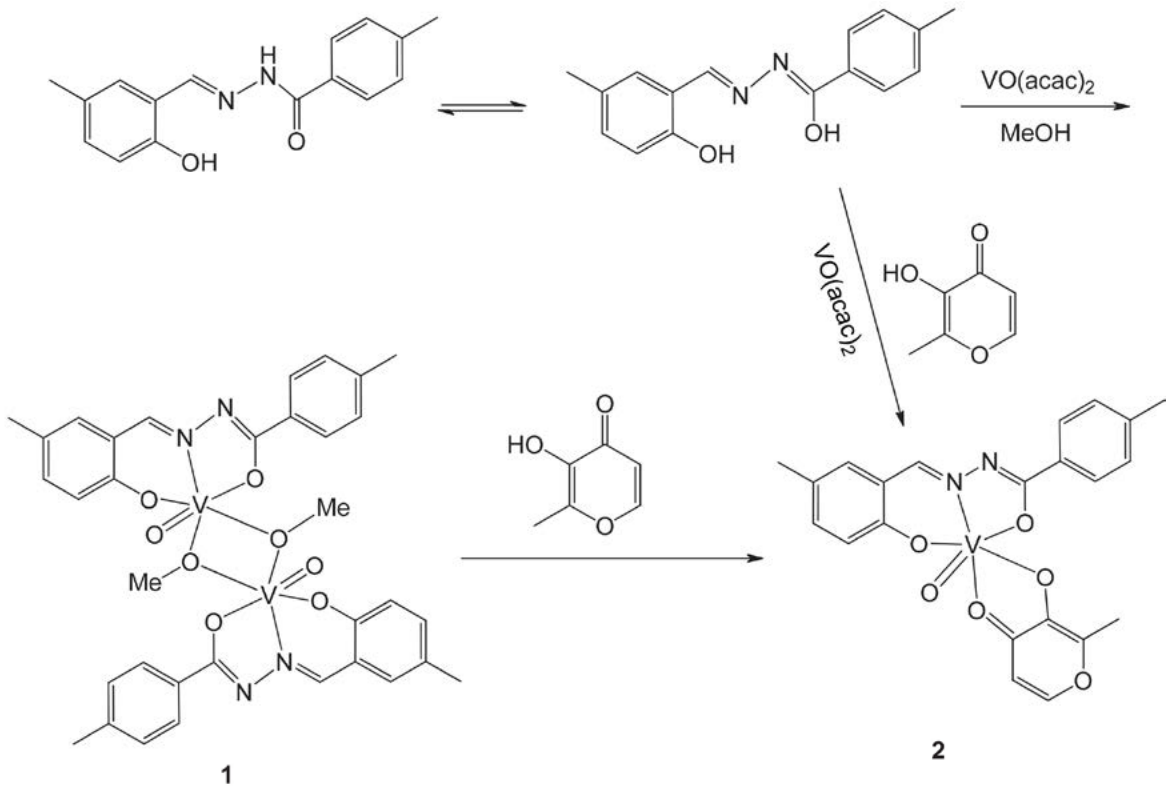


Figure 1. Experimental and simulated powder XRD patterns of complex **1**.



Scheme 2. The synthetic procedure for the two complexes

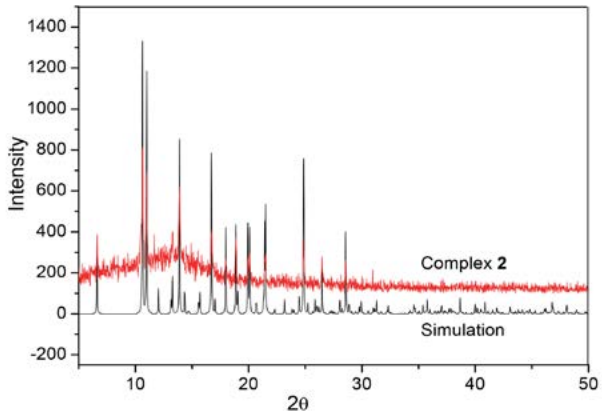


Figure 2. Experimental and simulated powder XRD patterns of complex **2**.

3. 1. Crystal Structure Description of $[V_2O_2L_2(OMe)_2]$ (1)

Molecular structure of complex **1** is presented in Figure 3. The molecule possesses crystallographic inversion center symmetry. The two V atoms with a distance of 3.363(2) Å are bridged by two methanolate ligands. The V atoms in the complex are in octahedral coordination, which are coordinated by the three donor atoms (N1, O1, O2) of the dianionic hydrazone ligand, two oxygens (O4 and O4A) from two methanolate ligands, and one oxo oxygen (O3). The methanolate O4 atom is coordinated *trans* to the oxo oxygen, which gives a long distance than V1–O4. This is not unusual for the *trans* effect of oxo group.[3–5] The V1–O3 bond length of 1.581(3) Å is within normal values for reported oxidovanadium(V) complexes.[4] The V–O and V–N bond lengths are comparable to the dinuclear vanadium complexes bridged by methanolate or ethanolate ligand.[5] The angular distortion about V coordinate bonds in the octahedral geometry is due to the strain of the four- and five-membered chelate rings V1–O4–V1A–O4A and V1–N1–N2–C8–O2 taken by the hydrazone ligand, with angles of 74.36(11)° and 74.21(12)°, respectively. The bond angles in the octahedral coordination are 74.36(11)–106.12(12)° for the perpendicular and 151.25(12)–176.23(12)° for the diagonal angles, also show distortion about the octahedral geometry. The V atom displaced out of the plane defined by O1, N1, O2 and O4 toward the oxo group by 0.336(2) Å. The benzene rings C1–C6 and C9–C14 form a dihedral angle of 5.9(3)°.

3. 2. Crystal Structure Description of [VOLL'] (2)

Molecular structure of complex **2** is presented in Figure 4. The V atom in the complex is in octahedral coordination, which are coordinated by the three donor atoms (N1, O1, O2) of the dianionic hydrazone ligand, two donor atoms (O4 and O5) of the maltolate ligand, and one oxo oxygen (O3). The carbonyl O4 atom is coordinated *trans* to the oxo oxygen, which gives a long distance than V1–O5. This is not unusual for the *trans* effect of oxo group.[3–5] The V1–O3 bond length of 1.531(4) Å is comparable to that of complex **1**, and within normal values for reported oxidovanadium(V) complexes.[5] The angular distortion about V coordinate bonds in the octahedral geometry is due to the strain of the five-membered chelate rings V1–O4–C17–C18–O5 and V1–N2–N1–C8–O2 taken by the hydrazone and maltolate ligands, with angles of 77.1(2)° and 75.4(2)°, respectively. The bond angles in the octahedral coordination are 75.4(2)–100.8(2)° for the perpendicular and 154.0(2)–175.9(2)° for the diagonal angles, also show distortion about the octahedral geometry. The V atom displaced out of the plane defined by O1, N1, O2 and O5 toward the oxo group by 0.303(2) Å. The benzene rings C1–C6 and C9–C14 form a dihedral angle of 3.2(3)°.

In the crystal structure of the complex, the vanadium complexes are linked through intermolecular C–H···O hydrogen bonds (Table 3), to form one dimensional chains running along the *a* axis (Figure 5).

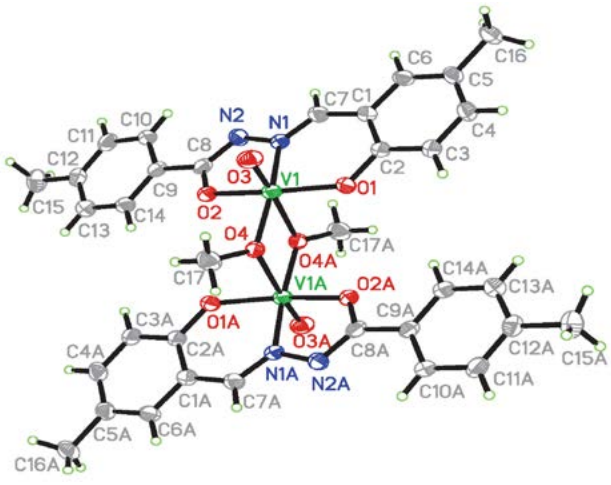


Figure 3. ORTEP plot of molecular structure of complex **1**. Displacement ellipsoids of non-hydrogen atoms are drawn at the 30% probability level.

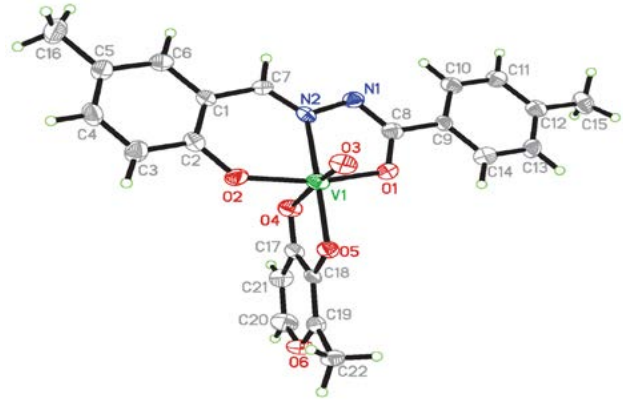


Figure 4. ORTEP plot of molecular structure of complex **2**. Displacement ellipsoids of non-hydrogen atoms are drawn at the 30% probability level.

Table 3. Hydrogen bond distances (Å) and bond angle (°) for complex **2**

D–H···*A*	*d*(*D*–H), Å	*d*(H···*A*), Å	*d*(*D*···*A*), Å	Angle (*D*–H···*A*), °
C21–H21···O1^{i}	0.93	2.56	3.243	131

Symmetry code: i) 1 – *x*, ½ + *y*, ½ – *z*.

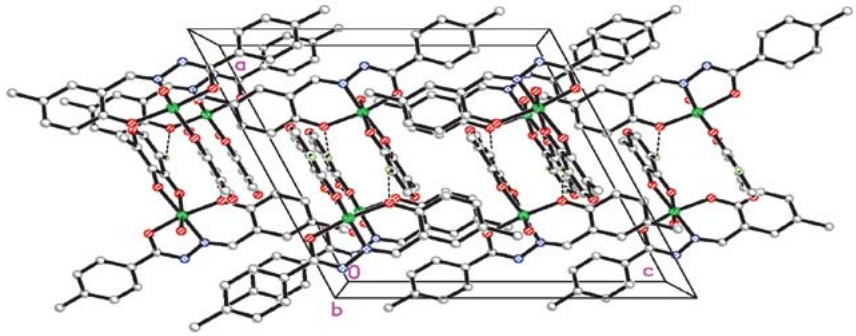


Figure 5. Molecular packing diagram of complex **2**, viewed along the *b* axis. Hydrogen atoms not related to hydrogen bonding are omitted. Hydrogen bonds are shown as dashed lines.

3. 3. Infrared and UV-Vis Spectra

The spectrum of H_2L showed vibrations which can be attributed to C=O, C=N, C–OH, and NH at 1648, 1617, 1154, 3290 and 3205 cm^{-1}, respectively.[12] The bands at 961 cm^{-1} for **1** and 961 cm^{-1} for **2** are assigned to the characteristic vibration of V=O bond.[13] In the spectra of the complexes, the $\nu_{C=O}$ and ν_{NH} are absent, but new C–O stretches appeared at 1283 cm^{-1} (**1**) and 1257 cm^{-1} (**2**). This indicates the presence of *keto*-imine tautomerization of the hydrazone ligand after complexation.[12] The $\nu_{C=N}$ observed at 1617 cm^{-1} in the spectrum of H_2L shifted to 1608–1610 cm^{-1} for the complexes upon coordination to V ions.[14]

In the UV-Vis spectra of H_2L and the complexes, the absorptions in the ranges 220–240 and 300–333 nm can be assigned as $\pi\rightarrow\pi^{\star}$ and $n\rightarrow\pi^{\star}$ transitions, respectively. The absorptions at 268 nm for **1** and 273 nm for **2** are due to LMCT of V=O, which is observed at 274 nm for [VO $(acac)_2$].[15]

3. 4. Antibacterial Activity

The hydrazone H_2L and the complexes were assayed for antibacterial activity against *P. aeroginosa*, *S. aureus*, *B. subtilis* and *E. coli* at 50 μg mL^{-1} using ethanol as solvent and control, and using tetracyclin as the standard drug. The minimum inhibitory concentrations (MICs) were determined by broth micro-dilution method.[16] The MIC values are listed in Table 4. The antibacterial activity was evaluated by measuring the zone of inhibition in mm. Ethanol has no antibacterial activity on the bacteria at the concentration studied. The results indicate that H_2L and the oxidovanadium complexes have weak to strong activities against the microorganisms. The two complexes showed stronger activities than H_2L. The least MIC value of 8 μg mL^{-1} was observed for the dinuclear complex **1** against *E. coli*. Complex **2** has good activities against *S. aureus* and *E. coli*, with the MIC values of 11 and 10 μg mL^{-1}, respectively. The present two complexes have better activity on *B. subtilis* and *E. coli*, while similar activity on *S. aureus* when compared to the vanadium(V) complexes with fluoro- and chloro-substituted benzohydrazone ligands.[12]

Table 4. Minimum inhibitory concentrations (MICs, μg mL^{-1}) of the compounds

Compound	*P. aeroginosa*	*S. aureus*	*B. subtilis*	*E. coli*
H_2L	> 50	45	39	27
1	> 50	13	17	8
2	> 50	11	14	10

4. Supplementary Material

CCDC–2285223 (1) and 2285222 (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 (CCDC), 12 Union Road, Cambridge CB2 1EZ, UK; fax: +44(0)1223-336033 or e-mail: deposit@ccdc.cam.ac.uk.

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Povzetek

Dvojedrni oksidovanadijev(V) kompleks $[V_2O_2L_2(OMe)_2]$ (**1**) smo sintetizirali iz *N*'-(2-hidroksi-5-metilbenziliden)-4-metilbenzohidrazida in $VO(acac)_2$ v MeOH. Pri reakciji kompleksa **1** s 3-hidroksi-2-metil-4-pironom (HL') smo dobili enojedrni oksidovanadijev(V) kompleks [VOLL'] (**2**). Hidrazon in oba kompleksa smo okarakterizirali z IR, UV in ^{1}H NMR spektroskopijo ter z rentgensko monokristalo analizo. Komplekse smo analizirali tudi z rentgensko praškovno difrakcijo. Vanadijevi atomi v obeh kompleksih so v oktaedrični koordinaciji. Molekule kompleksa **2** so povezane preko neklasičnih vodikovih vezi tipa C–H···O in tvorijo enodimenzionalne verige vzdolž osi *a*. Biološka evalvacija kaže, da imajo kompleksi učinkovito protimikrobno delovanje na bakterije *P. aeroginosa*, *S. aureus*, *B. subtilis* in *E. coli*.