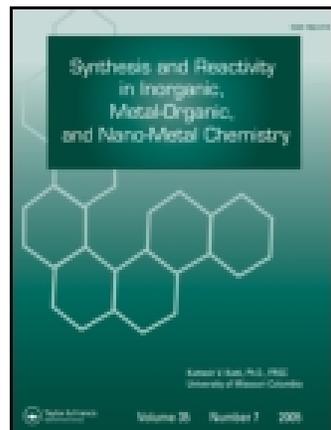


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New Vanadium Complexes with Aroylhydrazone Ligands: Synthesis, Structures, and Biochemical Properties

Ji-Lai Liu^a, Ming-Hui Sun^b & Jing-Jun Ma^c

^a Hebei Changshan Biochemical Pharmaceutical Co., Ltd., Shijiazhuang, P. R. China

^b Department of Economics and Management, Hebei North University, Zhangjiakou, P. R. China

^c College of Sciences, Agricultural University of Hebei, Baoding, P. R. China

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New Vanadium Complexes with Aroylhydrazone Ligands: Synthesis, Structures, and Biochemical Properties

Ji-Lai Liu¹, Ming-Hui Sun², and Jing-Jun Ma³

¹Hebei Changshan Biochemical Pharmaceutical Co., Ltd., Shijiazhuang, P. R. China

²Department of Economics and Management, Hebei North University, Zhangjiakou, P. R. China

³College of Sciences, Agricultural University of Hebei, Baoding, P. R. China

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The aroylhydrazone Schiff base ligands [*N'*-(2-hydroxynaphthyl)ethylidene]-4-nitrobenzohydrazide (H_2L^a) and *N'*-(3,5-dichloro-2-hydroxybenzylidene)-3-nitrobenzohydrazide (H_2L^b) reacting with $VO(acac)_2$ in methanol gave the complexes $[VOL^a(OMe)] \cdot 0.5MeOH \cdot 0.6H_2O$ (1) and $[VOL^b(OMe)(MeOH)]$ (2). Both complexes were characterized by elemental analyses and IR spectroscopic method in the solid state. Single crystal X-ray analysis was performed, which indicates that both complexes are mononuclear. The V atom in (1) is in a square pyramidal geometry, and that in (2) is in an octahedral geometry. V^{IV} in $VO(acac)_2$ was oxidized to V^V in the complexes by aerial oxygen during the synthesis and crystallization. Antibacterial activities of the aroylhydrazone ligands and the complexes have been studied on the strains *Bacillus subtilis*, *Escherichia coli*, and *Staphylococcus aureus*.

Keywords: aroylhydrazone, biochemical property, crystal structure, vanadium complex

Introduction

The presence of vanadium(IV) and vanadium(V) compounds in many vanadium-dependent enzymes, for instance, nitrogenases and haloperoxidases,^[1–3] as well as insulin mimicking,^[4,5] has stimulated the search for structural and functional models based on vanadium complexes. Moreover, oxovanadium complexes have been attracted tremendous attention for their versatile catalytic properties in organic synthesis.^[6–8] In recent years, the remarkable biological activity of aroylhydrazones and the dependence of their mode of chelation with transition metal ions in living systems have been of significant interest.^[9–12] Vanadium complexes of aroylhydrazones have also been reported to possess various antimicrobial activities.^[13–15] Aiming at obtaining new vanadium complexes with biological properties, in this article, two new vanadium complexes, $[VOL^a(OMe)] \cdot 0.5MeOH \cdot 0.6H_2O$ (1) and $[VOL^b(OMe)(MeOH)]$ (2), were synthesized, characterized, and studied their activities against *Bacillus subtilis* (*B. subtilis*), *Escherichia coli* (*E. coli*), and *Staphylococcus aureus* (*S. aureus*).

Experimental

Materials and Methods

1-(2-Hydroxynaphthalen-2-yl)ethanone, 3,5-dichlorosalicylaldehyde, 4-nitrobenzohydrazide, 3-nitrobenzohydrazide,

and vanadium(IV) oxide acetylacetonate ($[VO(acac)_2]$) were purchased from Merck and Fluka, and used as received. The aroylhydrazone Schiff base ligands [*N'*-(2-hydroxynaphthyl)ethylidene]-4-nitrobenzohydrazide (H_2L^a) and *N'*-(3,5-dichloro-2-hydroxybenzylidene)-3-nitrobenzohydrazide (H_2L^b) were prepared in over 90% yields by condensation reactions of 1-(2-hydroxynaphthalen-2-yl)ethanone with 4-nitrobenzohydrazide, and 3,5-dichlorosalicylaldehyde with 3-nitrobenzohydrazide, respectively, in methanolic media, according to the literature method.^[16,17] All other chemicals and solvents used in this work were of analytical grade available commercially and used without further purification. Elemental analyses (carbon, hydrogen, and nitrogen) of the compounds were obtained from a Carlo ERBA Model EA 1108 analyzer. Infrared spectra were collected by using KBr pellets on a Jasco-5300 FT-IR spectrophotometer (Leici Instrument Co., Shanghai, China). Solution electrical conductivity was measured with a DDS-11A conductivity meter.

Synthesis of $[VOL^a(OMe)] \cdot 0.5MeOH \cdot 0.6H_2O$ (1)

$[VO(acac)_2]$ (0.01 mmol, 2.65 g) dissolved in methanol (30 mL) was added dropwise to a stirred methanolic solution (30 mL) of H_2L^a (0.01 mol, 3.49 g). The mixture was gently refluxed for 2 h, then the most of the solvent was evaporated by distillation. After cooling, the resulting brown solid was filtered off, washed with cold methanol, and dried in a vacuum containing anhydrous $CaCl_2$. Yield: 2.13 g (47%). Anal. Calcd. for $C_{41}H_{38}N_6O_{14}V_2$ (%): C, 52.35; H, 4.07; N, 8.93. Found: C, 52.16; H, 3.95; N, 9.02. IR data (cm^{-1} , KBr): 3386 (br), 1602 (s), 1563 (m), 1527 (m), 1481 (m), 1332 (w), 1287

Address correspondence to Jing-Jun Ma, College of Sciences, Agricultural University of Hebei, Baoding 071001, P. R. China. E-mail: majingjun71@yahoo.cn

(m), 1255 (s), 1217 (m), 972 (s), 755 (m), 454 (w). Brown block-like single crystals of the complex, suitable for single crystal X-ray diffraction, were obtained by slow evaporation of a methanol solution containing the complex.

Synthesis of [VOL^b(OMe)(MeOH)] (2)

Complex (2) was prepared by an analogous method to that for complex (1), except for the replacement of H₂L^a by H₂L^b (0.01 mol, 3.53 g). Yield: 2.72 g (56%). Anal. Calcd. for C₁₆H₁₄Cl₂N₃O₇V (%): C, 39.86; H, 2.93; N, 8.72. Found: C, 39.97; H, 3.05; N, 8.60. IR data (cm⁻¹, KBr): 3351 (w), 1611 (s), 1555 (m), 1519 (m), 1473 (m), 1341 (m), 1293 (m), 1252 (s), 976 (s), 877 (w), 751 (m), 672 (w), 471 (w).

X-Ray Crystallography

Suitable X-ray quality crystals of the complexes (1) and (2) were pick up under a microscope and investigated in a diffraction experiment at 298(2) K on a Bruker Apex II diffractometer with monochromated Mo K α radiation ($\lambda = 0.71073$ Å) obtained from a graded multilayer X-ray optics. The structures were solved by direct methods with SHELXS-97,^[18] and refined with full-matrix least-square techniques on F^2 with SHELXL-97.^[18] The C- and O-bonded hydrogen atoms were calculated in an idealized geometry, riding on their parent atoms, with distances restrained to 0.93–0.96 Å for C–H and 0.82 Å for O–H. The crystal data and refinement parameters are listed in Table 1.

Table 1. Crystallographic data for the complexes.

	(1)	(2)
Chemical formula	C ₄₁ H ₃₈ N ₆ O ₁₄ V ₂	C ₁₆ H ₁₄ Cl ₂ N ₃ O ₇ V
Formula weight	938.64	482.14
Crystal system	Triclinic	Monoclinic
Space group	<i>P</i> -1	<i>P</i> 2 ₁ / <i>c</i>
Unit cell dimensions		
<i>a</i> (Å)	7.492(2)	12.973(11)
<i>b</i> (Å)	14.131(3)	18.279(15)
<i>c</i> (Å)	21.771(2)	7.986(7)
α (°)	75.572(2)	90
β (°)	89.807(2)	90.03(6)
γ (°)	79.842(1)	90
<i>V</i> (Å ³)	2195.2(8)	1894(3)
<i>Z</i>	2	4
ρ (g cm ⁻³)	1.420	1.691
μ (mm ⁻¹)	0.498	0.852
<i>T</i> _{min} , <i>T</i> _{max}	0.9157, 0.9246	0.7841, 0.8025
Reflections collected	14,768	14,885
Reflections unique	7820	4065
Reflections observed [<i>I</i> > 2 σ (<i>I</i>)]	4339	3006
Parameters	610	268
Restraints	30	1
<i>R</i> ₁ , <i>wR</i> ₂ [<i>I</i> > 2 σ (<i>I</i>)]	0.0960, 0.2387	0.0874, 0.2094
<i>R</i> ₁ , <i>wR</i> ₂ (all data)	0.1594, 0.3006	0.1082, 0.2191
Goodness of fit on F^2	1.012	1.099

Table 2. MIC values ($\mu\text{g mL}^{-1}$) of the tested compounds.

	<i>B. subtilis</i>	<i>E. coli</i>	<i>S. aureus</i>
H ₂ L ^a	>100	25	>100
H ₂ L ^b	12.5	6.25	25
(1)	12.5	12.5	12.5
(2)	1.56	6.25	3.13
Penicillin	0.78	100	3.13
Kanamycin	0.39	6.25	1.56

Antibacterial Assay

The antibacterial activities were tested against *B. subtilis*, *E. coli*, and *S. aureus* using Mueller-Hinton medium. The MICs (minimum inhibitory concentrations) of the test compounds were determined by a colorimetric method using the dye MTT [3-(4,5-dimethylthiazol-2-yl)-2,5-diphenyl tetrazolium bromide].^[19] A stock solution of the synthesized compound (50 $\mu\text{g mL}^{-1}$) in dimethyl sulfoxide (DMSO) was prepared and quantities of the test compounds were incorporated in a specified quantity of sterilized liquid Mueller-Hinton medium. A specified quantity of the medium containing the compound was poured into microtitration plates. A suspension of the microorganism was prepared to contain approximately 10⁵ cfu mL⁻¹ and applied to microtitration plates with serially diluted compounds in DMSO to be tested and incubated at 37°C for 24 h. After the MICs were visually determined on each of the microtitration plates, 50 μL of PBS (phosphate buffered saline 0.01 mol L⁻¹, pH 7.4: Na₂HPO₄·12H₂O 2.9 g, KH₂PO₄ 0.2 g, NaCl 8.0 g, KCl 0.2 g, distilled water 1000 mL) containing 2 mg of MTT/mL was added to each well. Incubation was continued at room temperature for 4–5 h. The content of each well was removed and 100 μL of isopropanol containing 5% 1 mol L⁻¹ HCl was added to extract the dye. After 12 h of incubation at room temperature, the optical density (OD) was measured with a microplate reader at 550 nm. The antibiotics kanamycin and penicillin were used as standard drugs. The observed MIC values are given in Table 2.

Results and Discussion

Synthesis and Characterization

Reaction of VO(acac)₂ with tridentate aroylhydrazone ligands H₂L^a and H₂L^b in methanol, respectively, under aerobic conditions afforded neutral oxovanadium(V) complexes (1) and (2). Most likely during the synthesis and crystallization of the complexes from oxovanadium(IV) starting material, the aerial oxygen acts as the oxidizing agent. Elemental analyses of the complexes are consistent with the general molecular formulae proposed by single crystal X-ray determination. In absolute methanolic solution, they are electrically non-conducting.

IR Spectroscopic Characteristics

Infrared spectra of the free aroylhydrazone ligands and their complexes gave information about the coordination. The free

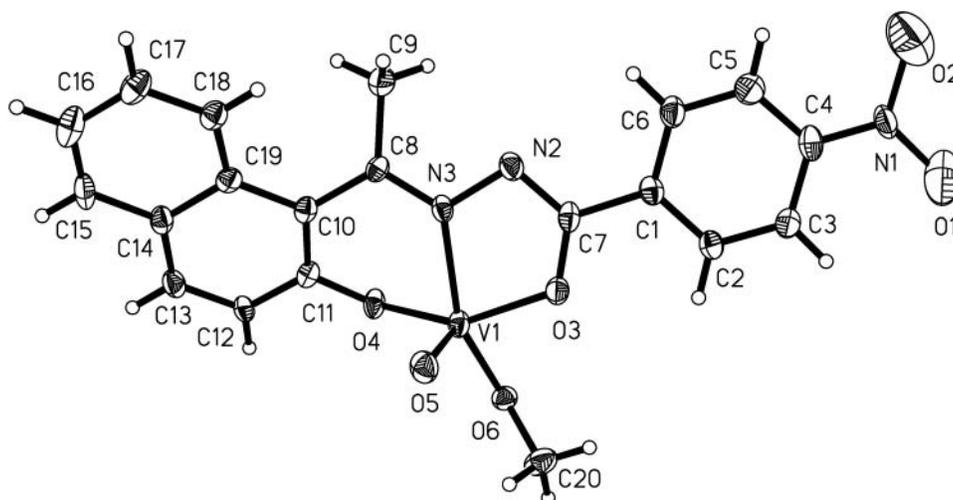


Fig. 1. Structure of the V1 molecule of complex (1) with the atom labeling scheme. All non-hydrogen atoms are represented at 30% probability thermal ellipsoids.

ligands display broad bands in the region $3350\text{--}3450\text{ cm}^{-1}$ and weak and sharp bands at about 3270 cm^{-1} . These bands are possibly due to the O—H and N—H stretches. However, the sharp bands are absent in the spectra of the complexes. The absence of the $\nu(\text{C}=\text{O})$ bands, present in the free aroylhydrazone ligands H_2L^a and H_2L^b at 1660 and 1651 cm^{-1} , respectively, indicates the enolization of the amide functionality upon coordination to the V atom. Strong bands observed at 1602 cm^{-1} for (1) and 1611 cm^{-1} for (2) can be attributed to the stretching vibrations of the azomethine groups, $\nu_{\text{C}=\text{N}}$.^[20] The spectra of the complexes display typical and intense bands at 972 cm^{-1} for (1) and 976 cm^{-1} for (2), which are assigned to the stretches of the V=O bonds.^[21]

Description of the Crystal Structure of (1)

Molecular structure and the atom numbering scheme for complex (1) is depicted in Figure 1. Selected bond lengths and angles are given in Table 3. The compound contains two mononuclear vanadium molecules, one methanol molecule, and one point two disordered water molecules. The coordination geometry around each vanadium atom can be described as distorted square pyramidal with the tridentate aroylhydrazone ligand coordinated in a meridional fashion, forming five- and six-membered chelate rings with bite angles of $74.4(2)^\circ$ (N3-V1-O3), $81.9(2)^\circ$ (N3-V1-O4), $74.5(2)^\circ$ (N6-V2-O9), and $81.8(2)^\circ$ (N6-V2-O10), typical for this type of ligand system.^[22,23] The aroylhydrazone ligands coordinate in their dianionic enolate form, which is consistent with the observed C7-O3 , C7-N2 , C27-O9 , and C27-N5 bond lengths of $1.298(9)$, $1.316(9)$, $1.294(8)$, and $1.319(9)\text{ \AA}$ in the complex. The three donor atoms of the aroylhydrazone ligand and the O atom of the deprotonated methanol ligand define the basal plane of the square pyramidal coordination. The apical position of the square pyramidal coordination is occupied by the oxo group. The V atoms deviate from the basal planes towards the oxo groups by $0.375(2)\text{ \AA}$ for V1 molecule and $0.383(2)\text{ \AA}$ for V2 molecule.

Description of the Crystal Structure of (2)

Molecular structure and the atom numbering scheme for complex (2) is depicted in Figure 2. Selected bond lengths and

Table 3. Selected bond lengths (\AA) and angles ($^\circ$) for the complexes.

(1)			
Bond lengths (\AA)			
V1–O3	1.908(5)	V1–O4	1.830(5)
V1–O5	1.578(5)	V1–O6	1.821(5)
V1–N3	2.110(6)	V2–O9	1.908(5)
V2–O10	1.829(5)	V2–O11	1.809(5)
V2–O12	1.582(5)	V2–N6	2.107(6)
Bond angles ($^\circ$)			
O5–V1–O6	103.7(3)	O5–V1–O4	101.7(3)
O6–V1–O4	104.9(2)	O5–V1–O3	101.0(3)
O6–V1–O3	89.7(2)	O4–V1–O3	149.1(2)
O5–V1–N3	98.2(2)	O6–V1–N3	155.1(2)
O4–V1–N3	81.9(2)	O3–V1–N3	74.4(2)
O12–V2–O11	103.9(3)	O12–V2–O10	101.8(3)
O11–V2–O10	104.5(2)	O12–V2–O9	101.5(2)
O11–V2–O9	89.7(2)	O10–V2–O9	148.8(2)
O12–V2–N6	98.5(2)	O11–V2–N6	154.8(2)
O10–V2–N6	81.8(2)	O9–V2–N6	74.5(2)
(2)			
Bond lengths (\AA)			
V1–O1	1.868(4)	V1–O2	1.966(4)
V1–O5	2.341(5)	V1–O6	1.750(5)
V1–O7	1.572(5)	V1–N1	2.134(5)
Bond angles ($^\circ$)			
O7–V1–O6	103.8(2)	O7–V1–O1	98.7(2)
O6–V1–O1	99.5(2)	O7–V1–O2	98.0(2)
O6–V1–O2	97.3(2)	O1–V1–O2	152.8(2)
O7–V1–N1	96.2(2)	O6–V1–N1	159.0(2)
O1–V1–N1	83.6(2)	O2–V1–N1	73.3(2)
O7–V1–O5	174.0(2)	O6–V1–O5	81.4(2)
O1–V1–O5	83.1(2)	O2–V1–O5	78.3(2)
N1–V1–O5	78.3(2)		

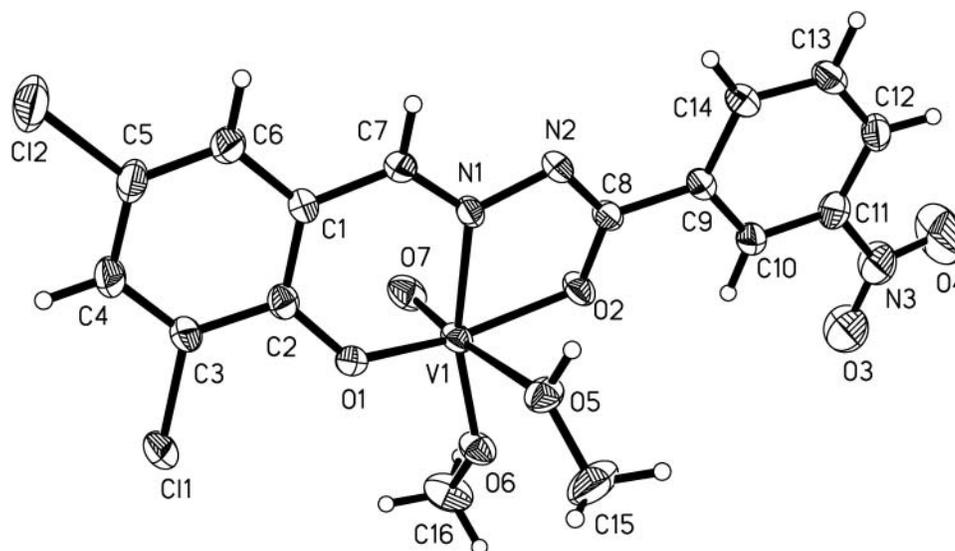


Fig. 2. Structure of complex (2) with the atom labeling scheme. All non-hydrogen atoms are represented at 30% probability thermal ellipsoids.

angles are given in Table 3. The coordination geometry around the vanadium atom can be described as a distorted octahedral with the tridentate aroylhydrazone ligand coordinated in a meridional fashion, forming five- and six-membered chelate rings with bite angles of $73.3(2)^\circ$ (N1–V1–O2) and $83.6(2)^\circ$ (N1–V1–O1), typical for this type of ligand system.^[22,23] The aroylhydrazone ligand coordinates in its dianionic enolate form, which is consistent with the observed C8–O2 and C8–N2 bond lengths of 1.292(7) and 1.302(8) Å in the complex. The three donor atoms of the aroylhydrazone ligand and the O atom of the deprotonated methanol ligand define the equatorial plane of the octahedral coordination. The axial positions of the octahedral coordination are occupied by the oxo group and the O atom of the methanol ligand. The V atom deviates from the equatorial plane towards the oxo groups by 0.315(2) Å.

Antibacterial Activities

The aroylhydrazone ligand H₂L^a has weak activity against *E. coli*, and no activity against *B. subtilis* and *S. aureus*. The aroylhydrazone ligand H₂L^b and the vanadium complexes have weak to medium activities against the three strains. In general, the vanadium complexes have stronger antibacterial activities than the aroylhydrazone ligands. Complex (2) has good activities against *B. subtilis* and *S. aureus*, with MIC values of 1.56 and 3.13 $\mu\text{g mL}^{-1}$, respectively, which are even close to the standard drugs penicillin and kanamycin. Detailed comparison among the compounds can simply conclude that chloro-substitute groups of the benzene rings may enhance the antibacterial activities of such compounds.

Conclusions

Two new vanadium(V) complexes were synthesized and characterized with the help of microanalytical, IR spectroscopic

and single crystal X-ray crystallographic measurements. The crystal structures of the complexes reveal that the V atoms are in distorted square pyramidal or octahedral coordination spheres. The vanadium complexes have interesting antibacterial activities on the strains *B. subtilis*, *E. coli*, and *S. aureus*.

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Supplemental Material

CCDC 934997 (1) and 934998 (2) contain the supplementary crystallographic data for the complex. These data can be obtained free of charge via <http://www.ccdc.cam.ac.uk/conts/retrieving.html>, or from the Cambridge Crystallographic Data Centre, 12 Union Road, Cambridge CB2 1EZ, UK; fax: (+44) 1223-336-033; or e-mail: deposit@ccdc.cam.ac.uk.

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