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Syntheses, Crystal Structures, and Antibacterial Activity of Mononuclear Vanadium(V) Complexes with Aroylhydrazone and Pyrone as Mixed Ligands[1]

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Abstract—Two mononuclear vanadium(V) complexes, [VOL^1(KA)] (**I**) and [VOL^2(EM)] (**II**), where L^1 and L^2 are the dianionic form of *N*′-(3-ethoxy-2-hydroxybenzylidene)-4-methoxybenzohydrazide (H_2L^1) and 4-bromo-*N*′-(3-ethoxy-2-hydroxybenzylidene)benzohydrazide (H_2L^2), respectively; KA and EM are the monoanionic form of kojic acid and ethyl maltol, respectively, were prepared. The complexes were characterized by elemental analysis, IR and UV-Vis spectra, and single crystal X-ray determination (CIF files CCDC nos. 1823339 (**I**) and 1823341 (**II**)). The crystal of complex **I** crystallizes in the triclinic space group $P\bar{1}$ with unit cell a = 7.7813(16), b = 11.297(2), c = 14.3390(18) Å, α = 90.449(2)°, β = 100.402(2)°, γ = 108.366(2)°, V = 1173.8(4) Å^3, Z = 2. The crystal of complex **II** crystallizes in the monoclinic space group $P2_1/n$ with unit cell a = 8.6935(5), b = 30.9695(18), c = 18.4469(11) Å, β = 99.366(2)°, V = 4900.3(5) Å^3, Z = 8. The V atoms in the complexes are in octahedral geometry. The antimicrobial activities of the compounds against *K. pneumoniae*, *S. aureus*, *P. aeroginosa*, *E. coli*, and *B. subtilis* were investigated.

Keywords: aroylhydrazone, vanadium complex, crystal structure, antibacterial activity

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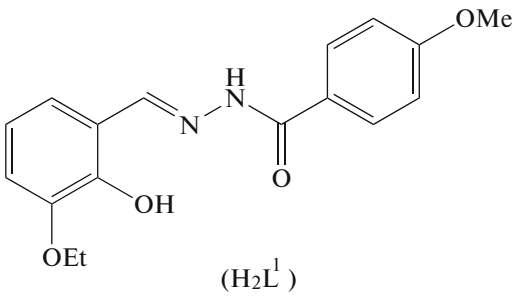
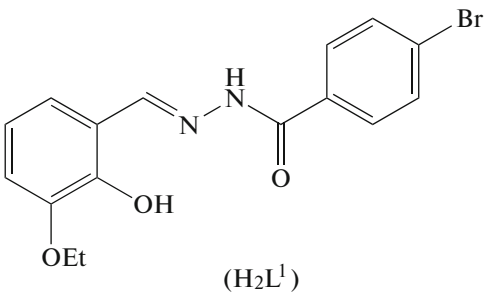
INTRODUCTION

Transition metal complexes with hydrazones are considered to be among the most important stereochemical models in coordination chemistry due to their preparative accessibility and structural variety [1–3], as well as interesting biological properties [4–6]. The bioinorganic chemistry of vanadium also receives great attention because of significant biological application, such as antimicrobial [7, 8], antitumor [9], and insulin mimetic activities [10, 11], as well as their potential to model vanadium-containing nitrogenases [12] and haloperoxidases [13, 14]. In addition, pyrone compounds have effective antibacterial activity [15, 16]. In the present work, we have chosen $VO(Acac)_2$ as the precursor to investigate its reactivity with biologically important aroylhydrazone and pyrone ligands, *N*′-(3-ethoxy-2-hydroxybenzylidene)-4-metho-xybenzohydrazide (H_2L^1), 4-bro-mo-*N*′-(3-ethoxy-2-hydroxybenzylidene)benzohydrazide (H_2L^2), kojic acid (KA) and ethyl maltol (EM). The newly synthesized complexes, [VOL^1(KA)] (**I**) and [VOL^2(EM)] (**II**), have been screened for their antibacterial activity with a view to explore their use as potential biocidal agents.



(H_2L^1)



(H_2L^1)

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EXPERIMENTAL

Materials and methods. 3-Ethoxysalicylaldehyde, 4-methoxybenzohydrazide, and 4-bromobenzohydrazide were purchased from Alfa Aesar. All other chemicals and solvents were of analytical grade and used as obtained. Microanalyses of the complexes were performed with a Vario EL III CHNOS elemental analyzer. The infrared spectra were recorded as KBr pellets with an FTS-40 spectrometer. UV-Vis spectra were recorded on a Lambdar 900 spectrometer. ^{1}H and ^{13}C NMR data were recorded on a Bruker 300 MHz spectrometer.

Synthesis of H_2L^1. 3-Ethoxysalicylaldehyde (0.15 g, 1.0 mmol) and 4-methoxybenzohydrazide (0.17 g, 1.0 mmol) were stirred at reflux in methanol (30 mL) for 30 min. Then, the solvent was evaporated by distillation, to give white solid product. The solid was recrystallized from methanol to give colorless crystalline product. The yield was 0.32 g (87%).

IR data (KBr; ν, cm^{-1}): 3542 s, 3408 m, 3242 m, 3063 w, 2980 w, 2930 w, 2897 w, 2838 w, 1643 s, 1614 s, 1564 m, 1514 m, 1468 s, 1368 m, 1310 m, 1256 s, 1181 m, 1115 w, 1077 m, 1022 m, 960 w, 885 w, 839 w, 739 m, 673 m, 619 w. UV-Vis data (methanol; λ, nm (ε, M^{-1} cm^{-1})): 300 (17,900), 340 (5,890). ^{1}H NMR (300 MHz, d^6-DMSO; δ, ppm): 12.00 (s., 1H, O*H*), 11.11 (s., 1H, N*H*), 8.64 (s., 1H, C*H*=N), 7.96 (d., 2H, Ar*H*), 7.12 (d., 2H, Ar*H*), 7.08–7.02 (m., 2H, Ar*H*), 6.86 (t., 1H, Ar*H*), 4.09 (q., 2H, CH_2), 3.86 (s., 1H, OCH_3), 1.37 (t., 3H, CH_3). ^{13}C NMR (75 MHz, d^6-DMSO; δ, ppm): 163.82, 162.18, 147.88, 147.54, 147.04, 129.56, 124.82, 121.19, 118.97, 118.56, 115.29, 113.80, 64.16, 55.45, 14.75.

For $C_{17}H_{18}N_2O_4$

Anal. calcd., %	C, 65.0	H, 5.8	N, 8.9
Found, %	C, 64.8	H, 5.9	N, 8.8

Synthesis of H_2L^2. This compound was prepared by the similar method as described for H_2L^1 with 4-methoxybenzohydrazide replaced by 4-bromobenzohydrazide (0.22 g, 1.0 mmol). The yield was 0.25 g (80%).

IR data (KBr; ν, cm^{-1}): 3558 m, 3429 m, 3200 w, 3067 w, 2980 w, 2930 w, 2880 w, 1655 s, 1606 s, 1564 m, 1472 s, 1393 m, 1285 s, 1246 s, 1151 w, 1077 m, 1010 w, 960 w, 881 w, 845 w, 731 m, 665 w, 460 w. UV-Vis data (methanol; λ, nm (ε, M^{-1} cm^{-1})): 300 (15,200), 340 (4,750). ^{1}H NMR (300 MHz, d^6-DMSO; δ, ppm): 12.16 (s., 1H, O*H*), 10.89 (s., 1H, N*H*), 8.67 (s., 1H, C*H*=N), 7.92 (d., 2H, Ar*H*), 7.78 (d., 2H, Ar*H*), 7.18 (d., 1H, Ar*H*), 7.05 (d., 1H, Ar*H*), 6.86 (t., 1H, Ar*H*), 4.15 (q., 2H, CH_2), 1.37 (t., 3H, CH_3). ^{13}C NMR (75 MHz, d^6-DMSO; δ, ppm): 161.84, 148.51, 147.53, 147.06, 131.94, 131.58, 129.71, 125.73, 120.95, 119.06, 118.98, 115.38, 64.17, 14.73.

For $C_{16}H_{15}N_2O_3Br$

Anal. calcd., %	C, 52.9	H, 4.2	N, 7.7
Found, %	C, 52.8	H, 4.1	N, 7.8

Synthesis of complex I. H_2L^1 (31.4 mg, 0.1 mmol), kojic acid (14.2 mg, 0.1 mmol) and $VO(Acac)_2$ (26.5 mg, 0.1 mmol) were stirred in methanol (20 mL) for 30 min. The solution was stand still at ambient temperature to slow evaporation of the solvent, yielding deep brown crystals of **I**. The yield was 23.4 mg (45%).

IR data (KBr; ν, cm^{-1}): 3521 m, 3450 m, 1597 s, 1497 m, 1451 m, 1405 w, 1339 m, 1256 s, 1181 m, 1081 w, 1027 w, 981 m, 935 w, 881 w, 856 w, 764 w, 736 w, 669 w, 631 w, 540 w, 490 w. UV-Vis data (acetonitrile; λ, nm (ε, M^{-1} cm^{-1})): 220 (17,210), 257 (10,630), 336 (5,350), 435 (2,720).

For $C_{23}H_{21}N_2O_9V$

Anal. calcd., %	C, 53.1	H, 4.1	N, 5.4
Found, %	C, 53.2	H, 4.1	N, 5.2

Synthesis of complex II. Th is complex was prepared by the similar method as described for **I** with H_2L^1 replaced by H_2L^2 (36.3 mg, 0.1 mmol), and kojic acid replaced by ethyl maltol (14.0 mg, 0.1 mmol). The yield was 0.30 g (53%).

IR data (KBr; ν, cm^{-1}): 1607 s, 1553 w, 1515 w, 1473 w, 1450 m, 1396 m, 1343 m, 1257 s, 1185 w, 1098 w, 978 m, 895 w, 843 w, 771 w, 732 m, 593 m, 525 w, 480 w. UV-Vis data (acetonitrile; λ, nm (ε, M^{-1} cm^{-1})): 280 (15,320), 356 (5,670), 475 (3,300).

For $C_{23}H_{20}BrN_2O_7V$

Anal. calcd., %	C, 48.7	H, 3.6	N, 4.9
Found, %	C, 48.5	H, 3.5	N, 5.0

X-ray crystal structure determination. Data were collected on a Bruker SMART 1000 CCD area diffractometer using a graphite monochromator and MoK_α radiation (λ = 0.71073 Å) at 298(2) K. The data were corrected with the SADABS programs and refined on F^2 with the Siemens SHELXL software [17, 18]. The structures were solved by direct methods and difference Fourier syntheses. All non-hydrogen atoms were refined anisotropically. Hydrogen atoms were placed in calculated positions and included in the last cycles of refinement. Crystal data and details of the data collection and refinement

Table 1. Crystallographic data for complexes **I** and **II**

Parameter	Value	
	I	**II**
Chemical formula	$C_{23}H_{21}N_2O_9V$	$C_{23}H_{20}N_2O_7BrV$
Fw	520.4	567.3
Crystal shape/colour	Block/brown	Block/brown
Crystal size, mm	0.27 × 0.23 × 0.22	0.12 × 0.08 × 0.06
Crystal system	Triclinic	Monoclinic
Space group	$P\bar{1}$	$P2_1/n$
a, Å	7.7813(16)	8.6935(5)
b, Å	11.297(2)	30.9695(18)
c, Å	14.3390(18)	18.4469(11)
α, deg	90.449(2)	90
β, deg	100.402(2)	99.366(2)
γ, deg	108.366(2)	90
V, Å^3	1173.8(4)	4900.3(5)
Z	2	8
μ(MoK_α), cm^{-1}	0.479	5.690
T_{min}	0.8816	0.5484
T_{max}	0.9020	0.7264
ρ_c, g cm^{-3}	1.472	1.538
Reflections/parameters	4196/319	6537/617
Unique reflections	2275	3526
Restraints	0	0
Goodness of fit on F^2	1.000	1.026
R_{int}	0.0687	0.0723
R_1 ($I > 2\sigma(I)$)	0.0661	0.0607
wR_2 ($I > 2\sigma(I)$)	0.1436	0.1324
R_1 (all data)	0.1433	0.1372
wR_2 (all data)	0.1784	0.1623
Largest peak and deepest hole, e Å^{-3}	0.461 and –0.294	0.464 and –0.492

are listed in Table 1. Selected coordination bond lengths and angles are listed in Table 2.

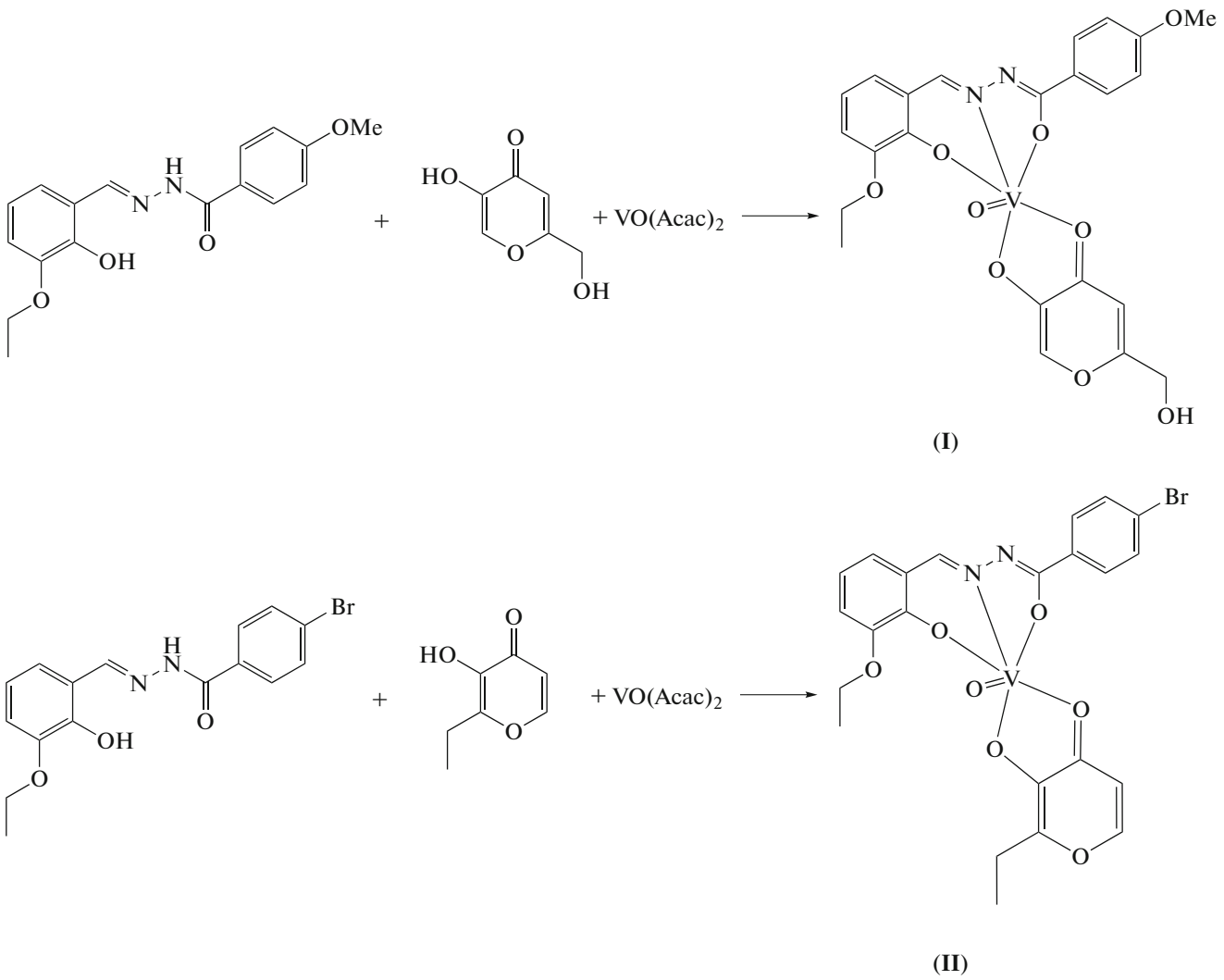
Supplementary material for structures **I** and **II** has been deposited with the Cambridge Crystallographic Data Centre (CCDC nos. 1823339 and 1823341; deposit@ccdc.cam.ac.uk or http://www.ccdc.cam.ac.uk).

RESULTS AND DISCUSSION

The complexes were readily prepared by the reaction of equimolar quantities of the aroylhydrazone ligands with $VO(Acac)_2$ in the presence of kojic acid or ethyl maltol in methanol (Scheme 1).

Table 2. Selected bond lengths (Å) and angles (deg) for complexes **I** and **II**

Bond	*d*, Å	Bond	*d*, Å
		I	
V(1)–O(9)	1.586(3)	V(1)–O(1)	1.852(3)
V(1)–O(6)	1.863(3)	V(1)–O(2)	1.927(3)
V(1)–N(1)	2.080(4)	V(1)–O(5)	2.300(3)
		II	
V(1)–O(13)	1.589(4)	V(1)–O(1)	1.851(5)
V(1)–O(5)	1.859(5)	V(1)–O(2)	1.936(4)
V(1)–N(1)	2.067(6)	V(1)–O(4)	2.279(5)
V(2)–O(14)	1.585(5)	V(2)–O(7)	1.856(5)
V(2)–O(11)	1.859(5)	V(2)–O(8)	1.933(4)
V(2)–N(3)	2.069(6)	V(2)–O(10)	2.267(5)
Angle	ω, deg	Angle	ω, deg
		I	
O(9)V(1)O(1)	98.43(15)	O(9)V(1)O(6)	100.48(15)
O(1)V(1)O(6)	104.08(13)	O(9)V(1)O(2)	101.32(14)
O(1)V(1)O(2)	152.31(14)	O(6)V(1)O(2)	91.23(13)
O(9)V(1)N(1)	96.10(15)	O(1)V(1)N(1)	83.91(14)
O(6)V(1)N(1)	160.21(14)	O(2)V(1)N(1)	74.90(14)
O(9)V(1)O(5)	178.26(14)	O(1)V(1)O(5)	81.59(12)
O(6)V(1)O(5)	77.84(12)	O(2)V(1)O(5)	79.25(12)
N(1)V(1)O(5)	85.63(12)		
		II	
O(13)V(1)O(1)	99.8(2)	O(1)3V(1)O(5)	98.2(2)
O(1)V(1)O(5)	104.2(2)	O(1)3V(1)O(2)	100.8(2)
O(1)V(1)O(2)	151.2(2)	O(5)V(1)O(2)	92.5(2)
O(13)V(1)N(1)	95.1(2)	O(1)V(1)N(1)	83.2(2)
O(5)V(1)N(1)	163.3(2)	O(2)V(1)N(1)	75.1(2)
O(13)V(1)O(4)	174.7(2)	O(1)V(1)O(4)	80.62(18)
O(5)V(1)O(4)	76.6(2)	O(2)V(1)O(4)	80.70(18)
N(1)V(1)O(4)	90.2(2)	O(14)V(2)O(7)	99.2(2)
O(14)V(2)O(11)	99.5(2)	O(7)V(2)O(11)	103.2(2)
O(14)V(2)O(8)	100.2(2)	O(7)V(2)O(8)	152.4(2)
O(11)V(2)O(8)	92.8(2)	O(14)V(2)N(3)	96.0(2)
O(7)V(2)N(3)	83.6(2)	O(11)V(2)N(3)	161.7(2)
O(8)V(2)N(3)	74.9(2)	O(14)V(2)O(10)	176.6(2)
O(7)V(2)O(10)	80.56(18)	O(11)V(2)O(10)	77.3(2)
O(8)V(2)O(10)	81.28(18)	N(3)V(2)O(10)	87.3(2)



Scheme 1.

The crystal structure of complex **I** with atom numbering scheme is shown in Fig. 1a. In structure **I**, there are two independent molecules. Two V atoms in the complex are in octahedral coordination with the phenolate O, imino N and enolate O atoms of the aroylhydrazone ligand, and the phenolate O atom of the KA ligand defining the equatorial plane, and with the carbonyl O atom of the KA ligand and the oxo O atom occupying the axial positions. The distance of V(1)–O(9) is 1.586(3) Å, indicating it is a typical double bond. The displacement of the V atom from the least-squares plane defined by the four equatorial donor atoms is 0.306(1) Å. The dihedral angle between the C(1)–C(6) and C(9)–C(14) rings of the aroylhydrazone ligand is 27.8(2)°. The bond lengths and angles in the complex are comparable to those observed in vanadium complexes with aroylhydrazone ligands [19, 20]. The *cis* coordinate bond angles are in the range 74.90(14)°–104.08(13)°, and the *trans* bond angles are in the range 152.31(14)°–178.26(14)°.

In the crystal structure of complex **I**, the molecules are linked through intermolecular O–H···O hydrogen bonds to form dimers (Fig. 2a, Table 3).

The crystal structure of complex **II** with atom numbering scheme is shown in Fig. 1b. The V atom in the complex is in octahedral coordination with the phenolate O, imino N and enolate O atoms of the aroylhydrazone ligand, and the phenolate O atom of the EM ligand defining the equatorial plane, and with the carbonyl O atom of the EM ligand and the oxo O atom occupying the axial positions. The distance of V(1)–O(13) is 1.589(4) Å, and that of V(2)–O(14) is 1.585(5) Å, indicating they are typical double bonds. The displacements of the V(1) and V(2) atoms from the least-squares planes defined by the respective four equatorial donor atoms are 0.285(1) and 0.294(1) Å. The dihedral angle between the C(1)–C(6) and C(9)–C(14) rings of the aroylhydrazone ligand is 37.2(3)°, and that between the C*(24)–C(29) and C(32)–C(37) rings of the other aroylhydrazone ligand is 49.0(3)°.

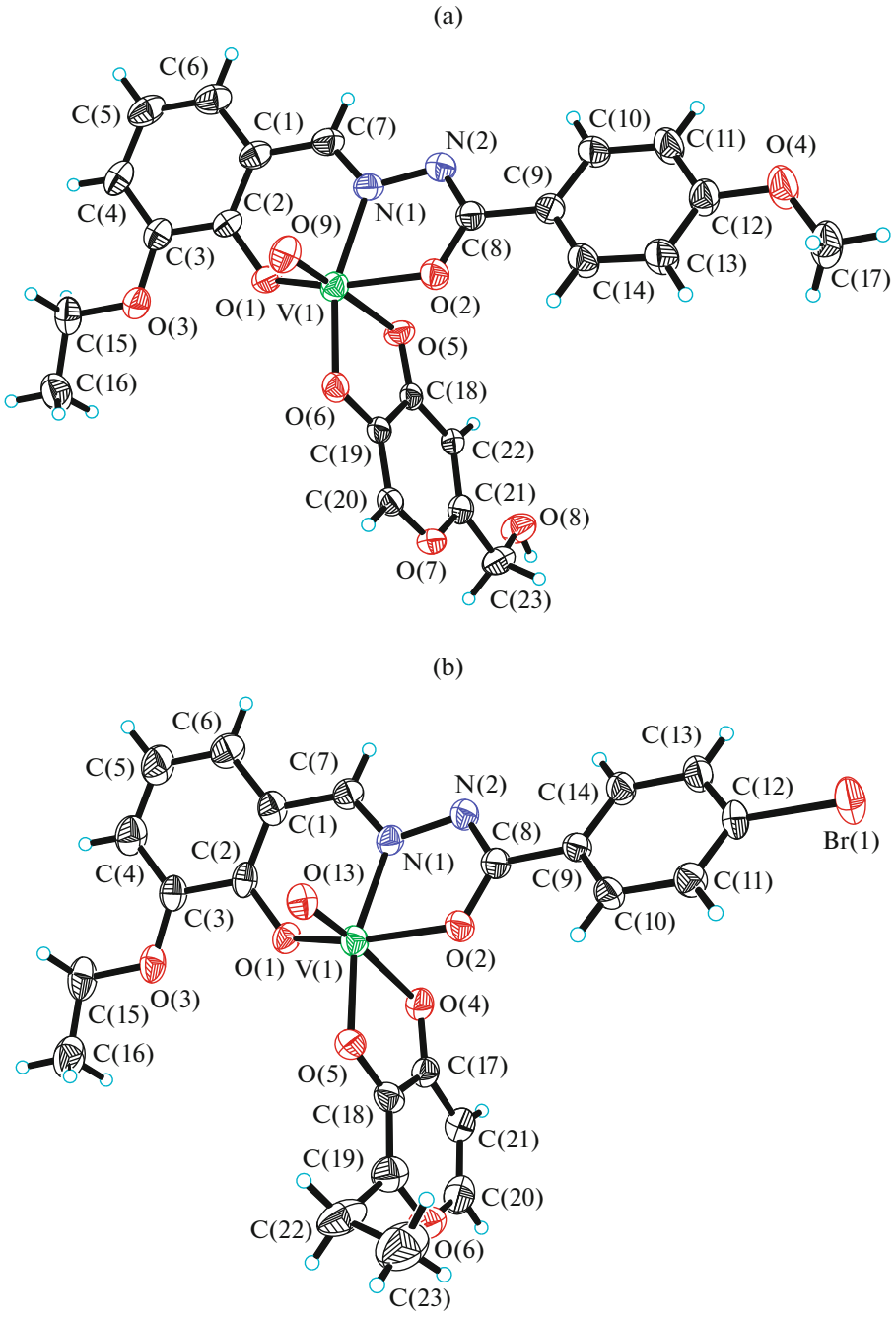


Fig. 1. ORTEP plots (30% probability level) and the numbering scheme for **I** and **II**.

The bond lengths and angles in the complex are comparable to those observed in vanadium complexes with aroylhydrazone ligands [19, 20]. The *cis* coordinate bond angles are in the range 74.9(2)°–104.2(2)°, and the *trans* bond angles are in the range 151.2(2)°–176.6(2)°.

In the crystal structure of complex **II**, the molecules are stacked along the *x* axis (Fig. 2b).

The infrared spectra of the complexes are consistent with the structural data given in this paper. The broad and medium bands in the range 3520–3560 and 3400–3450 cm^{-1} can be attributed to the O–H stretching vibration of the hydroxyl groups of the free aroylhydrazones and complex **I**. As for the spectra of the two aroylhydrazones, the weak and sharp bands in the range 3200–3250 cm^{-1} can be attributed to the N–H stretching vibration. These bands are absent in the complexes, indicating the coordination through the enolate form of the aroylhydrazone ligands. The strong bands at 1643 and 1655 cm^{-1} in the spectra of

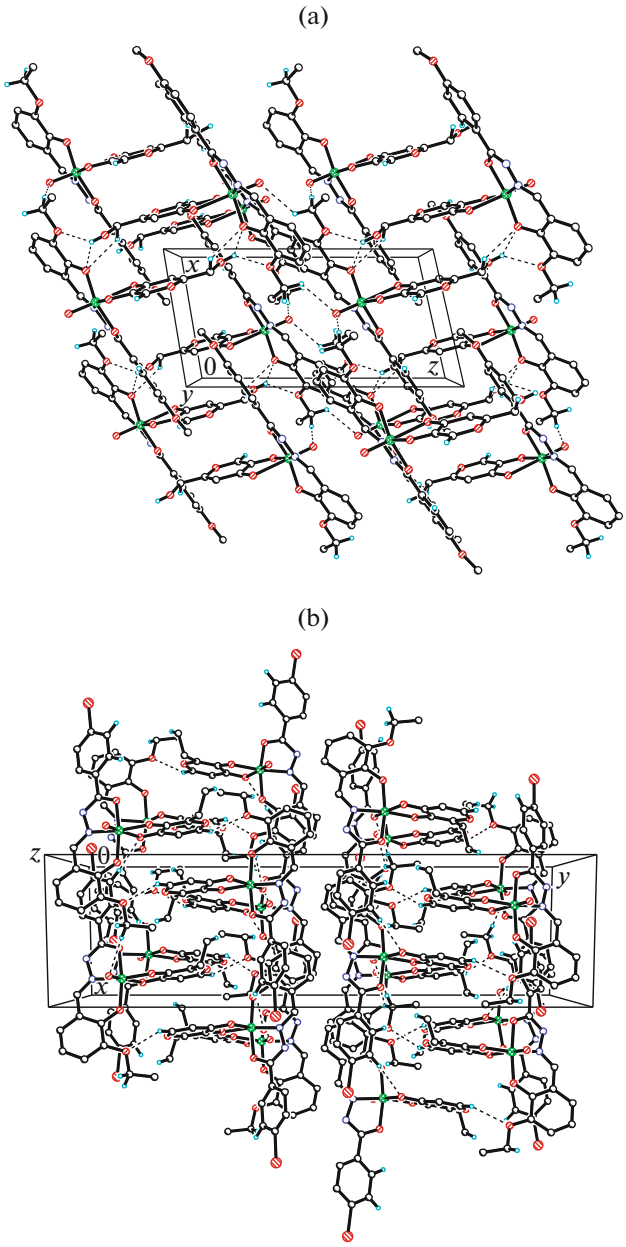


Fig. 2. Molecular packing diagram for **I** and **II**.

H_2L^1 and H_2L^2 are assigned to the C=O groups, which are absent in the complexes. The strong bands at 1614 and 1606 cm^{-1} in the spectra of H_2L^1 and H_2L^2 are assigned to the azomethine groups [21, 22], which are shift to 1597 and 1607 cm^{-1} for the complexes. The bands indicative of the V=O bonds are observed at 981 cm^{-1} for **I** and 978 cm^{-1} for **II** [23].

The electronic spectra of the complexes are similar. The weak bands at 400–550 nm are attributed to intramolecular charge transfer transitions from the p_π orbital on the nitrogen and oxygen to the empty *d* orbitals of the metal [24]. The intense bands observed at 250–280 nm are assigned to intraligand π–π* transitions. The charge transfer LMCT bands are located at 320–370 nm.

The free aroylhydrazones and the two vanadium complexes were assayed for in vitro antibacterial activity against *K. pneumoniae*, *S. aureus*, *P. aeroginosa*, *E. coli*, and *B. subtilis* at 50 μg mL^{-1} using ethanol as solvent and control, and using tetracyclin as the standard drug. The minimum inhibitory concentrations (MIC) were determined by broth micro-dilution method [25]. The observed MIC values in μg mL^{-1} are reported in Table 4. The antibacterial activity was evaluated by measuring the zone of inhibition in mm. Eth-

Table 3. Geometric parameters of hydrogen bond for complexes **I** and **II***

D–H···A	Distance, Å			Angle D–H···A, deg
	D–H	H···A	D···A	
		I		
O(8)–H(8)···O(1)^{i}	0.82	2.49	3.053(4)	127
O(8)–H(8)···O(3)^{i}	0.82	2.31	2.951(5)	135
C(14)–H(14)···O(2)	0.93	2.42	2.755(5)	101
C(15)–H(15*A*)···O(9)^{ii}	0.97	2.49	3.416(5)	159
C(15)–H(15*B*)···O(9)^{iii}	0.97	2.53	3.199(5)	126
C(20)–H(20)···O(4)^{iv}	0.93	2.39	3.155(5)	140
C(23)–H(23*B*)···O(1)^{i}	0.97	2.44	3.098(5)	125
		II		
C(10)–H(10)···O(2)	0.93	2.43	2.762(6)	101
C(11)–H(11)···O(4)^{ii}	0.93	2.55	3.474(6)	173
C(15)–H(15*A*)···O(13)^{v}	0.97	2.57	3.525(6)	168
C(15)–H(15*B*)···O(14)^{vi}	0.97	2.50	3.183(6)	127
C(20)–H(20)···O(9)	0.93	2.55	3.361(6)	145
C(23)–H(23*A*)···O(6)	0.96	2.56	2.952(6)	105
C(34)–H(34)···O(10)^{v}	0.93	2.60	3.520(6)	173
C(38)–H(38*B*)···O(13)^{vii}	0.97	2.59	3.264(6)	126
C(43)–H(43)···O(3)	0.93	2.49	3.314(6)	147
C(46)–H(46*A*)···O(12)	0.96	2.50	2.899(6)	105

* Symmetry codes: ^{i} $-x, -y, -z$; ^{ii} $-1+x, y, z$; ^{iii} $-x, -y, 1-z$; ^{iv} $-1+x, -1+y, z$; ^{v} $1+x, y, z$; ^{vi} $1/2+x, 1/2-y, 1/2+z$; ^{vii} $-1/2+x, 1/2-y, -1/2+z$.

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

Compound	*K. pneumoniae*	*S. aureus*	*P. aeroginosa*	*E. coli*	*B. subtilis*
H_2L^1	45	>50	>50	43	38
H_2L^2	38	32	41	36	33
I	30	45	>50	35	29
II	25	27	33	32	27

anol had no antibacterial activity on the bacteria at the concentration studied. The results revealed that the free aroylhydrazones and the two complexes showed from weak to effective activities against the tested microorganisms. In general, H_2L^2 has stronger activities than H_2L^1, and the complexes showed higher activities than the free aroylhydrazones. Such an enhancement in the activity of metal complexes against certain specific microorganisms may be explained on the basis of Overtone's concept [26] and Tweedy's chelation theory [27]. The least MIC with 25 μg mL^{-1} was observed for complex **II** against *K. pneumoniae*.

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