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Scientific paper

Syntheses, Structures and Insulin-Like Activity of Two Oxidovanadium(V) Complexes with Similar Nicotinohydrazone Ligands

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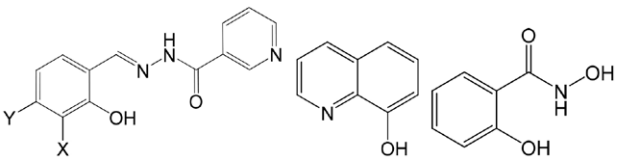
Abstract

Two new oxidovanadium(V) complexes, [VOL^1(HQ)] (**1**) and [VOL^2(SAH)] (**2**), were prepared by the reaction of [VO(acac)$_2$] (where acac = acetylacetonate) with *N*'-(3-ethoxy-2-hydroxybenzylidene)nicotinohydrazide (H_2L^1) and 8-hydroxyquinoline (HHQ), and *N*'-(2-hydroxy-4-methoxybenzylidene)nicotinohydrazide (H_2L^2) and salicylhydroxamic acid (HSAH), respectively, in methanol. Crystal and molecular structures of the complexes were determined by elemental analysis, infrared spectroscopy and single crystal X-ray diffraction. The V atoms in both complexes are in octahedral coordination. Thermal stability of the complexes was studied. Both complexes can decrease the blood glucose level in alloxan-diabetic mice, but the blood glucose level in the treated normal mice was not altered.

Keywords: Nicotinohydrazone ligand; oxovanadium complex; crystal structure; thermal property; insulin-like activity

1. Introduction

It was reported that inorganic vanadium salts exhibited insulin-like activity at 40 years ago.[1] Interestingly, a pharmacological advantage of the vanadium salts is that it can be orally administered with long-term insulin-like activity *in vivo*.[1b,2] However, inorganic vanadium salts are considered as less active and more toxic when compared with vanadium complexes with various types of ligands.[3] Thus, a number of vanadium complexes have been prepared to improve the stability and membrane permeability of the vanadyl cation or decrease the toxicity of the vanadate anion.[4] Metal complexes with nicotinohydrazones have received particular attention in biological and medicinal chemistry.[5] 8-Hydroxyquinoline (HHQ) and salicylhydroxamic acid (HSAH) are widely known bidentate ligands in coordination chemistry.[6] However, only two HQ coordinated oxovanadium complexes and one SAH coordinated oxidovanadium complex with hydrazone ligands have been reported so far.[7] In the present paper, two new oxovanadium(V) complexes with hydrazone and HQ or SAH ligands, [VOL^1(HQ)] (**1**) and [VOL^2(SAH)] (**2**) (H_2L^1 = *N*'-(3-ethoxy-2-hydroxybenzylidene)nicotinohydrazide, H_2L^2 = *N*'-(2-hydroxy-4-methoxybenzylidene)nicotinohydrazide; Scheme 1), have been presented.



Scheme 1. The hydrazone, HHQ and HSAH ligands. H_2L^1: X = OEt, Y = H; H_2L^2: X = H, Y = OMe.

2. Experimental

2. 1. Materials and Measurements

Commercially available 3-ethoxysalicylaldehyde, 4-methoxysalicylaldehyde and nicotinohydrazide were purchased from Sigma-Aldrich and used without further purification. Other solvents and reagents were made in China and used as received. H_2L^1 and H_2L^2 were prepared according to the literature method.[8] C, H and N elemental analyses were performed with a Perkin-Elmer elemental analyser. Infrared spectra were recorded on a Nicolet AVATAR 360 spectrometer as KBr pellets in the (4000–400) cm^{-1} region. Thermal stability analysis was performed on a Perkin-Elmer Pyris Diamond TG-DTA thermal analyses system. Molar conductivity data were determined with a DDS-11A conductometer.

2. 2. Synthesis of [VOL^1(HQ)] (1)

A methanolic solution (10 mL) of [VO(acac)$_2$] (0.1 mmol, 26.5 mg) was added to a methanolic solution (10 mL) of H_2L^1 (0.1 mmol, 28.5 mg) and HHQ (0.1 mmol, 14.5 mg) with stirring. The mixture was stirred for 30 min at room temperature to give a deep brown solution. The resulting solution was allowed to stand in air for a few days. Brown block-shaped crystals suitable for X-ray single crystal diffraction were formed at the bottom of the vessel. The isolated products were washed three times with cold ethanol, and dried in air. The yield was 55%. Anal. calc. for $C_{24}H_{19}N_4O_5V$: C, 58.31; H, 3.87; N, 11.33; found: C, 58.12; H, 3.78; N, 11.46%.

2. 3. Synthesis of [VOL^2(SAH)] (2)

This complex was prepared according to the same method as that described for **1**, with H_2L^1 replaced by H_2L^2 (0.1 mmol, 27.1 mg), and HHQ replaced by HSAH (0.1 mmol, 15.3 mg). The yield was 63%. Anal. calc. for $C_{21}H_{17}N_4O_7V$: C, 51.65; H, 3.51; N, 11.47; found: C, 51.56; H, 3.62; N, 11.38%.

2. 4. X-Ray Crystallography

Diffraction intensities for the complexes were collected at 298(2) K using a Bruker D8 VENTURE PHOTON diffractometer with Mo*K*a radiation (l = 0.71073 Å). The collected data were reduced using the SAINT program,[9] and multi-scan absorption corrections were performed using the SADABS program.[10] The structures were solved by direct methods and refined against F^2 by full-matrix least-squares methods using the SHELXTL.[11] All of the non-hydrogen atoms were refined anisotropically. The amino hydrogen atom in complex **2** was located from a difference Fourier map and refined isotropically, with N–H distance restrained to 0.90(1) Å. The remaining hydrogen atoms were placed in idealized positions and constrained to ride on their parent atoms. The crystallographic data for the complexes are summarized in Table 1.

Table 1. Crystallographic data and refinement parameters for the complexes

Parameter	Value	
	1	**2**
Chemical formula	$C_{24}H_{19}N_4O_5V$	$C_{21}H_{17}N_4O_7V$
Formula weight	494.4	488.3
Crystal system	Monoclinic	Monoclinic
Space group	$P2_1/n$	$P2_1/n$
Unit cell parameters		
a / Å	9.0157(5)	11.0094(8)
b / Å	11.4002(6)	17.7842(12)
c / Å	21.6406(11)	12.6825(10)
β / °	97.130(2)	101.446(2)
V / Å^3	2207.0(2)	2433.8(3)
Z	4	4
D_{calc} / g cm^{-3}	1.488	1.333
T / K	298(2)	298(2)
μ / mm^{-1}	0.495	0.453
F(000)	1016	1000
Unique reflections	4095	4526
Observed reflections [$I > 2\sigma(I)$]	3380	3562
Parameters	308	303
Restraints	0	1
R_1, wR_2 [$I > 2\sigma(I)$]	0.0377, 0.0943	0.0621, 0.2062
R_1, wR_2 (all data)	0.0503, 0.1021	0.0797, 0.2229
Goodness of fit on F^2	1.041	1.105

2. 5. Glucose-Lowering Assay

Male Kunming mice, weighing about 25–30 g, were obtained from Experimental Animal Center, Shandong Lukang Pharmaceutical Co., Ltd of China, and maintained on a light/dark cycle. All animals were allowed free access to food and water. Temperature and relative humidity were maintained at 25 °C and 50%. Mice were acclimatized for a week prior to induction of diabetes. Diabetes was induced by a single intra-peritoneal injection of freshly prepared alloxan (200 mg kg^{-1} body weight) in 0.9% saline. The control mice were injected with an equal volume of vehicle. After a week, blood was collected from the tail vein and serum samples were analyzed for blood glucose. Animals showing fasting (12 h) blood glucose higher than 11.1 mmol L^{-1} were considered to be diabetic and used for the study.

The experimental animals were randomly divided into 8 groups with 4 mice each according to the blood glucose. Group 1, normal control group: normal mice treated with 0.5% carboxymethyl cellulose (CMC). Groups 2 and 3, treated normal groups: normal mice treated with 20 mg V kg^{-1} complexes. Group 4, diabetic control group: alloxan diabetic mice treated with 0.5% CMC. Groups 5–8, treated diabetic groups: alloxan diabetic mice treated with the complexes at doses of 10 and 20 mg V kg^{-1} intragastric administration. The complexes were administered as suspensions in 0.5% CMC. The substances were administered intragastrically once a day at the volume of 10 mL kg^{-1} for 2 weeks.

3. Results and Discussion

3. 1. General

Replacement of two acetylacetonate ligands of [VO(acac)$_2$] by hydrazone and 8-hydroxyquinoline or salicylhydroxamate ligands in methanol resulted in the formation of two structurally similar complexes. The complexes are soluble in DMF, DMSO, methanol, ethanol, and acetonitrile. Molar conductance of complexes **1** and **2** at the concentration of 10^{-4} mol L^{-1} are 16 Ω^{-1} cm^2 mol^{-1}

and 23 Ω^{-1} cm^2 mol^{-1}, respectively, indicating they are non-electrolytes.[12]

3. 2. Crystal Structure Description of the Complexes

The molecular structures and atom numbering schemes of complexes **1** and **2** are shown in Figures 1 and 2, respectively. Selected bond lengths and angles are given in Table 2. The V atoms in the complexes are in octahedral coordination, with the three donor atoms of the nicotinohydrazone ligands and the hydroxy O atom of the HQ ligand (for **1**) or SAH ligand (for **2**) defining the equatorial plane, and with one oxo O atom and the pyridine N atom of the HQ ligand (for **1**) or the carbonyl O atom of the SAH ligand (for **2**) occupying the axial positions. The distances between atoms V(1) and O(5) in **1**, and V(1) and O(7) in **2** are 1.58–1.59 Å, indicating they are typical V=O double bonds. The V(1)–N(4) bond in **1** and V(1)–O(5) bond in **2** are significantly longer than

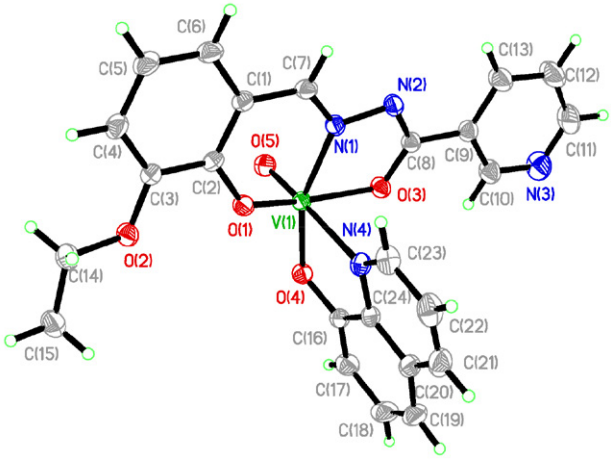


Figure 1. ORTEP plot of the molecular structure of complex **1**. Displacement ellipsoids of non-hydrogen atoms are drawn at the 30% probability level.

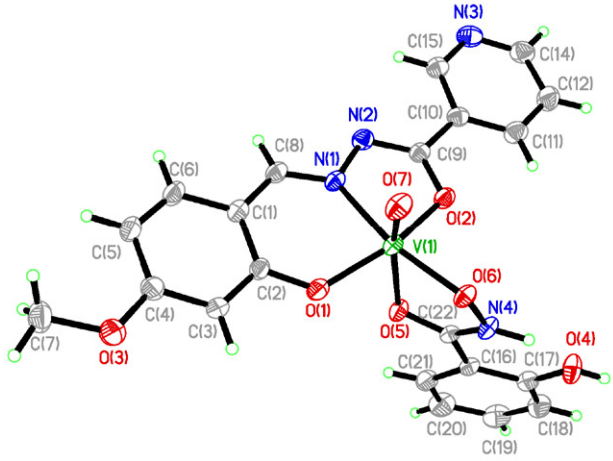


Figure 2. ORTEP plot of the molecular structure of complex **2**. Displacement ellipsoids of non-hydrogen atoms are drawn at the 30% probability level.

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

1			
V(1)–O(1)	1.8606(14)	V(1)–O(3)	1.0619(15)
V(1)–O(4)	1.8501(14)	V(1)–O(5)	1.5862(16)
V(1)–N(1)	2.0777(17)	V(1)–N(4)	2.3416(18)
O(5)–V(1)–O(4)	99.57(7)	O(5)–V(1)–O(1)	99.77(8)
O(4)–V(1)–O(1)	107.28(6)	O(5)–V(1)–O(3)	99.01(8)
O(4)–V(1)–O(3)	88.15(6)	O(1)–V(1)–O(3)	153.21(7)
O(5)–V(1)–N(1)	96.70(7)	O(4)–V(1)–N(1)	158.03(7)
O(1)–V(1)–N(1)	84.23(6)	O(3)–V(1)–N(1)	74.74(6)
O(5)–V(1)–N(4)	175.26(7)	O(4)–V(1)–N(4)	75.87(6)
O(1)–V(1)–N(4)	82.99(6)	O(3)–V(1)–N(4)	79.70(6)
N(1)–V(1)–N(4)	87.39(7)		
2			
V(1)–O(1)	1.855(3)	V(1)–O(2)	1.947(3)
V(1)–O(5)	2.218(3)	V(1)–O(6)	1.885(3)
V(1)–O(7)	1.585(3)	V(1)–N(1)	2.084(3)
O(7)–V(1)–O(1)	99.53(14)	O(7)–V(1)–O(6)	92.80(13)
O(1)–V(1)–O(6)	110.89(12)	O(7)–V(1)–O(2)	102.68(14)
O(1)–V(1)–O(2)	149.95(13)	O(6)–V(1)–O(2)	88.08(11)
O(7)–V(1)–N(1)	94.18(14)	O(1)–V(1)–N(1)	84.21(12)
O(6)–V(1)–N(1)	162.05(12)	O(2)–V(1)–N(1)	74.26(11)
O(7)–V(1)–O(5)	166.78(13)	O(1)–V(1)–O(5)	81.35(11)
O(6)–V(1)–O(5)	74.73(10)	O(2)–V(1)–O(5)	81.60(10)
N(1)–V(1)–O(5)	99.02(11)		

the other coordinate bonds, yet, it is not uncommon for such complexes.[13] The bond lengths in both complexes are comparable to each other, and also similar to those observed in the mononuclear oxidovanadium(V) complexes with octahedral coordination.[13] The angular distortion in the octahedral environment around V comes from the five- and six-membered chelate rings taken by the nicotinohydrazone ligands. For the same reason, the *trans* angles significantly deviate from the ideal values of 180°. Distortion of the octahedral coordination can be observed from the coordinate bond angles, ranging from 74.74(6)° to 107.28(6)° for the perpendicular angles, and from 153.21(7)° to 175.26(7)° for the diagonal angles for **1**, and from 74.26(11)° to 110.89(12)° for the perpendicular angles, and from 149.95(13)° to 162.05(12)° for the diagonal angles for **2**. The displacement of the V atoms from the equatorial plane is 0.30 Å for **1** and 0.23 Å for **2**. The dihedral angles between the benzene ring and the pyridine ring of the hydrazone ligands are 6.2(3)° in **1** and 6.3(5)° in **2**. In the crystal structure of **2**, the adjacent two complex molecules are linked by O–H···N hydrogen bonds [O(4)–H(4A)···N(3)^{i}: O(4)–H(4A) = 0.82 Å, H(4A)···N(3)^{i} = 1.92 Å, O(4)···N(3)^{i} = 2.736(4) Å, O(4)–(H4A)···N(3)^{i} = 172°; symmetry code: i) –½ + *x*, 1½ – *y*, ½ + *z*], to form a dimer. The dimers are further linked by N–H···O hydrogen bonds [N(4)–H(4)···O(6)^{ii}: N(4)–H(4) = 0.90(1) Å, H(4)···O(6)^{ii} = 2.14(4) Å, N(4)···O(6)^{ii} = 2.838(4) Å; symmetry code: ii) 1 – *x*, 2 – *y*, 1 – *z*], to form one dimensional chain.

3. 3. IR Spectra

Complexes **1** and **2** exhibit typical bands at 963 cm^{-1} and 975 cm^{-1}, respectively, assigned to the V=O vibration.[14] The bands due to $\nu_{C=O}$ were absent in the complexes, but new C–O stretches appeared at 1266 cm^{-1} for **1** and 1250 cm^{-1} for **2**. This suggests occurrence of *keto*-imine tautomerization of the ligands during complexation. The intense $\nu_{C=N}$ absorptions are observed at 1602 cm^{-1} for **1** and **2**.[15] The weak peaks in the low wave numbers in the region (400–650) cm^{-1} may be attributed to V–O and V–N bonds in the complexes.

3. 4. Thermal Property

Differential thermal (DT) and thermal gravimetric analyses (TGA) were conducted to examine the stability of the complexes (Figures 3 and 4). For **1**, the complex decomposed from 170 °C to 490 °C, corresponding to the loss of the nicotinohydrazone and HQ ligands and the formation of V_2O_5. The total observed weight loss of 82.7% is close to the calculated value of 81.6%. For **2**, the complex decomposed from 170 °C to 510 °C, corresponding to the loss of the nicotinohydrazone and SAH ligands and the formation of V_2O_5. The total observed weight loss of 82.3% is close to the calculated value of 81.4%.

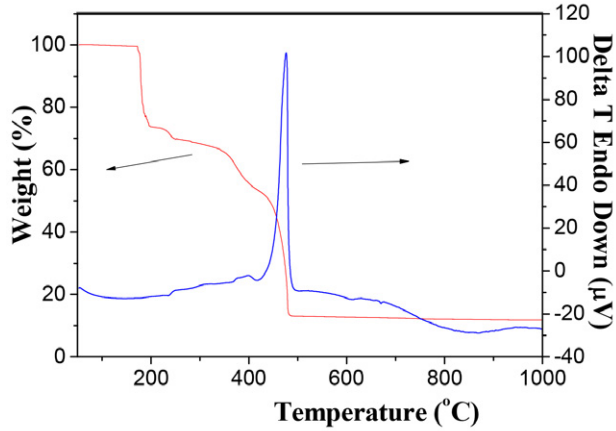


Figure 3. DT-TGA curves of complex **1**.

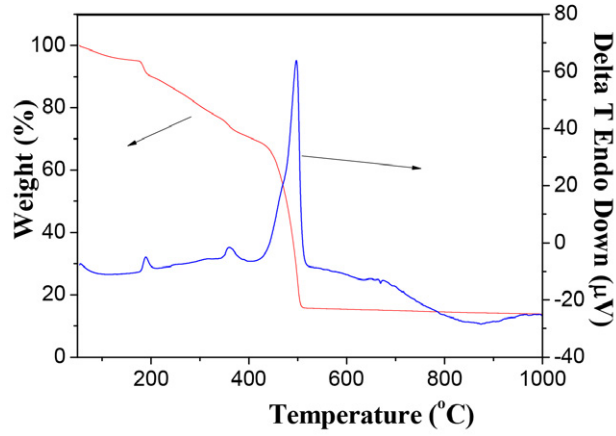


Figure 4. DT-TGA curves of complex **2**.

3. 5. Insulin-Like Activity of the Complexes

The results are listed in Table 3, which showed that both complexes had blood glucose-lowering effect at doses of 10.0 and 20.0 mg V kg^{-1}. Both complexes can decrease the blood glucose level in alloxan-diabetic mice, whereas the blood glucose level in the treated normal mice (20.0 mg V kg^{-1} by intragastric administration for 2 weeks) was not altered as compared with the untreated normal mice. The alloxan-diabetic mice exhibited significant hyperglycemia. After 2-week administration with the complexes, the blood glucose level was decreased compared with the diabetic control group. The glucose-lowering effect of both complexes is similar to each other. $VOSO_4$ was also assayed as comparison, and showed similar activities as compared to the complexes.

4. Conclusion

Two new mononuclear oxidovanadium(V) complexes derived from mixed ligands *N*'-(3-ethoxy-2-

Table 4 Effects of the complexes on blood glucose levels of mice

Group	Dose (mg V kg^{-1})[a]	Blood glucose (mmol L^{-1}) 0	7 d	14 d	21 d
normal mice	CMC	5.8±0.5	5.9±0.7	5.9±0.9	5.8±0.8
normal mice + **1**	20.00	5.7±0.8	5.8±1.0	5.9±1.1	5.9±0.8
normal mice + **2**	20.00	5.7±0.6	5.9±0.7	5.8±0.9	5.8±1.3
normal mice + $VOSO_4$	20.00	5.6±0.5	5.7±0.4	5.8±0.8	5.7±0.9
alloxan mice	CMC	16.7±1.5	16.0±1.2	15.7±1.4	15.5±1.5
alloxan mice + **1**	20.00	8.2±1.3	6.2±1.5	6.1±1.3	6.4±1.3
alloxan mice + **1**	10.00	9.3±1.5	6.7±1.1	6.9±1.2	7.0±1.4
alloxan mice + **2**	20.00	7.8±1.4	6.1±1.2	6.2±1.4	6.0±1.1
alloxan mice + **2**	10.00	9.7±1.2	6.6±1.3	6.5±1.0	6.3±1.3
alloxan mice + $VOSO_4$	20.00	8.3±1.1	6.4±0.8	6.3±0.5	6.2±1.0
alloxan mice + $VOSO_4$	10.00	9.5±1.0	6.8±1.1	6.7±0.9	6.1±0.7

[a] Data were expressed as mean±standard deviations.

hydroxybenzylidene)nicotinohydrazide and 8-hydroxyquinoline, and *N*'-(2-hydroxy-4-methoxybenzylidene) nicotinohydrazide and salicylhydroxamic acid, respectively, were prepared and structurally characterized. The V atoms are in octahedral coordination. Thermal stability of the complexes was studied. The bioassay indicated that both complexes have effective insulin-like activity on alloxan-diabetic mice, which deserve further study.

Supplementary Data

CCDC 979511 (**1**) and 979512 (**2**) contain the supplementary crystallographic data for the complexes. These data can be obtained free of charge *via* http://www.ccdc.cam.ac.uk/conts/retrieving.html, or from the Cambridge Crystallographic Data Center, 12 Union Road, Cambridge CB2 1EZ, UK; fax: (+44)1223-336-033; or e-mail: deposit@ccdc.cam.ac.un.

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Povzetek

Z reakcijo $[VO(acac)_2]$ (acac = acetilacetonat) z *N*'-(3-etoksi-2-hidroksibenziliden)nikotinohidrazidom (H_2L^1) in 8-hidroksikinolinom (HHQ) ter z *N*'-(2-hidroksi-4-metoksibenziliden)nikotinohidrazidom (H_2L^2) in salicilhidroksamsko kislino (HSAH) v metanolu smo pripravili dva nova oksidovanadijeva(V) kompleksa $[VOL^1(HQ)]$ (**1**) in $[VOL^2(SAH)]$ (**2**). Kristalno in molekulsko strukturo kompleksov smo določili z elementno analizo, infrardečo spektroskopijo in monokristalno rentgensko difrakcijo. Atomi vanadija v obeh kompleksih so v oktaedrični koordinaciji. Proučevali smo termično stabilnost kompleksov. Oba kompleksa lahko zmanjšata raven glukoze v krvi pri aloksan-diabetičnih miših, vendar se raven glukoze v krvi pri zdravljenih normalnih miših ni spremenila.

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