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N'-(3-Bromo-2-Hydroxybenzylidene)-4nitrobenzohydrazide and Its Oxovanadium(V) Complex: Synthesis, Crystal Structures, and Catalytic Property

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N'-(3-Bromo-2-Hydroxybenzylidene)-4nitrobenzohydrazide and Its Oxovanadium(V) Complex: Synthesis, Crystal Structures, and Catalytic Property

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A new benzohydrazone compound, N'-(3-bromo-2-hydro xybenzylidene)-4-nitrobenzohydrazide (H₂L), and its oxovanadium(V) complex, 2[VOL(BHA)]·CH₃CN, where HBHA is benzohydroxamic acid, was prepared and structurally characterized by physicochemical methods and single-crystal X-ray diffraction. The benzohydrazone compound crystallizes in the monoclinic space group $P2_1/c$, with a = 11.2211(15), b = 13.5515(18), c = 9.2850(10)Å, $\beta = 90.554(2)^{\circ}$, $V = 1411.8(30 \text{ Å}^3, Z = 4, R_1 = 0.0757$, and $wR_2 = 0.1920$. The oxovanadium(V) complex crystallizes in the triclinic space group P-1, with a = 11.3754(13), b = 14.7186(19), c = 15.611(2) Å, $\alpha = 83.952(2)^\circ$, $\beta = 72.346(2)^\circ$, $\gamma = 69.848(2)^\circ$, $V = 2338.1(5) \text{ Å}^3$, Z = 2, $R_1 = 0.0886$, and $wR_2 = 0.2245$. X-ray structural analysis indicates that the benzohydrazone ligand coordinates to the V atom through the phenolate O, imino N, and enolic O atoms. The V atom in the complex is coordinated by one benzohydrazone ligand and one BHA ligand, as well as one oxo O atom, forming an octahedral coordination. The crystal structures of the ligand and the complex are stabilized by hydrogen bonds. The complex is an effective catalyst for sulfoxidation.

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Keywords benzohydrazone, crystal structure, oxovanadium complex, sulfoxidation

INTRODUCTION

Schiff-base transition metal complexes have attracted much attention in metallo-organic and coordination chemistry because of their interesting structures and diverse range of applications,^[1–4] especially for their catalytic properties.^[5–7] Sulfoxides

are widely used as drug intermediates in pharmaceutical industries, and also sulfinyl groups have been used as auxiliaries in a variety of highly diastereoselective carbon-carbon bond forming reactions, including the synthesis of α -branched amines, α - and β -amino acids, aziridines, and amino phosphonic acids.^[8,9] Sulfoxidation catalyzed by complexes of transition metals such as titanium, manganese, iron, molybdenum, and vanadium have produced interesting results in the synthesis of various sulfoxides.^[10–12] As a continuation to explore effective catalysts used in sulfoxidation, in the present paper, a new benzohydrazone compound, *N'*-(3-bromo-2-hydroxybenzylidene)-4nitrobenzohydrazide (H₂L; Scheme 1), and its oxovanadium(V) complex, 2[VOL(BHA)]·CH₃CN, where HBHA is benzohydroxamic acid, were prepared and investigated for their peroxidic oxidation on sulfides.



SCH. 1. H₂L.

EXPERIMENTAL

All chemicals were of reagent grade and used as purchased. Microanalyses of C, H, N were performed on a Perkin-Elmer-2400 Series II CHN analyzer (Yunnan Normal University, China). FT-IR spectra (4000–200 cm⁻¹) of the ligand and the complex were recorded on a Perkin-Elmer Spectrum RX I FT-IR system (Yunnan Normal University, China) with solid KBr pellets. ¹H NMR spectra were recorded on Bruker Avance 200 spectrometer (Yunnan Normal University, China).

Synthesis of H₂L

Equimolar quantities of 3-bromosalicylaldehyde (0.201 g, 1 mmol) and 4-nitrobenzohydrazide (0.181 g, 1 mmol) were mixed in 50 mL methanol. The mixture was stirred at reflux for

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30 min and the solvent was evaporated to give yellow crystalline product of H_2L , which was used without further purification. Anal. Calcd. for $C_{14}H_{10}BrN_3O_4$: C, 46.2; H, 2,8; N, 11.5. Found: C, 46.1; H, 2.7; N, 11.6%.

Synthesis of the Oxovanadium Complex

 H_2L (36.4 mg, 0.1 mmol) and benzohydroxamic acid (13.7 mg, 0.1 mmol) were dissolved in 15 mL methanol. The solution was then added dropwise to a 10 mL gently warmed methanol solution of VO(acac)₂ (26.5 mg, 0.1 mmol). The mixture was refluxed for 30 min and cooled to room temperature.

TABLE 1 Crystal data and structure refinement for H_2L and the complex

	H_2L	Complex
Empirical formula	$C_{14}H_{10}BrN_3O_4$	C ₄₄ H ₃₁ Br ₂ N ₉ O ₁₄ V ₂
Formula weight	364.2	1171.5
Temperature (K)	298(2)	298(2)
Wavelength (Å)	0.71073	0.71073
Crystal system	Monoclinic	Triclinic
Space group	$P2_{1}/c$	<i>P</i> -1
Crystal color/shape	Yellow/block	Brown/block
Unit cell dimensions		
<i>a</i> (Å)	11.2211(15)	11.3754(13)
<i>b</i> (Å)	13.5515(18)	14.7186(19)
<i>c</i> (Å)	9.2850(10)	15.611(2)
α (/°)	90	83.952(2)
β (/°)	90.554(2)	72.346(2)
γ (/°)	90	69.848(2)
Volume (Å ³)	1411.8(3)	2338.1(5)
Ζ	4	2
Density (calculated, g cm^{-3})	1.713	1.664
Absorption coefficient (mm^{-1})	2.934	2.186
<i>F</i> (000)	728	1172
θ range for data collection (°)	1.81-26.49	2.72-25.00
Reflections collected	10088	14070
Independent reflections	2817	7425
Observed reflections $[I > 2\sigma(I)]$	1818	4431
Restraints	2	0
Parameters	205	641
Max. and min.	0.5778 and	0.7642 and 0.8445
transmission	0.6354	
Goodness-of-fit on F^2	1.023	1.016
<i>R</i> _{int}	0.0699	0.0463
Final <i>R</i> indices $[I > 2\sigma(I)]$	0.0757	0.0886
<i>R</i> indices (all data)	0.1920	0.2245

The resulting deep brown solution was filtered and left undisturbed. After a few days, brown block shaped X-ray diffraction quality single crystals were formed and isolated by filtration. Yield: 33.7 mg (60%). Anal. Calcd. for $C_{44}H_{31}Br_2N_9O_{14}V_2$: C, 45.1; H, 2.7; N, 10.8. Found: C, 44.9; H, 2.7; N, 10.7%.

Crystallographic Data Collection and Structure Refinements

Diffraction quality single crystals of H₂L and the complex were mounted on a Bruker AXS SMART diffractometer (Yunnan Normal University, China) with a graphite monochromator and Mo K α radiation ($\lambda = 0.71073$ Å). The crystallographic data collection was performed using multi-scan technique at 298(2) K. Data collection and the unit cell refinement were performed using CrysAlisPRO software.^[13] The structures of the compounds were solved by direct method procedures with SHELXS,^[14] and refined by full-matrix least squares based on F^2 with SHELXL.^[14] The non-hydrogen atoms were refined with anisotropic factors. The amino H atoms in H₂L and the complex were located in difference Fourier maps and refined isotropically, with N–H distances restrained to 0.90(1) Å. The remaining hydrogen atoms were positioned geometrically treated as riding on their parent atoms. The data collection and refinement parameters are listed in Table 1. Selected bond lengths and angles are given in Table 2.

Catalytic Oxidation

The oxovanadium complex (0.001 M) and phenyl methyl sulfide (0.1 M) were dissolved at room temperature in a mixture of CH_2Cl_2 and CH_3OH (6:4) together with 1,3,5-trimethoxybenzene (0.1 M) as internal standard. The resulting solution was cooled to 283 K and H_2O_2 (35% w/w) added dropwise (0.125 M). An aliquot of the reaction solution (2.0 mL) was quenched with 5 mL of a stock solution of Na_2SO_3 (0.1 M) at 60 min and 2 h, and extracted with dichlormethane (three times 4 mL). From the collected organic phases the solvent was removed under reduced pressure to complete dryness and the residue redissolved in deuterated chloroform (0.6 mL) and analyzed by ¹H NMR to determine the yields with reference to the internal standard 1,3,5-trimethoxybenzene.

RESULTS AND DISCUSSION

The condensation of 3-bromosalicylaldehyde with 4nitrobenzohydrazide in methanol with 1:1 stoichiometric ratio gave yellow crystalline product of H_2L . The benzohydrazone ligand react with VO(acac)₂ in the presence of benzohydroxamic acid yielded the oxovanadium complex. Elemental analyses were found to be in good agreement with the formulae calculated from the X-ray determination.

IR Spectra

The sharp absorption bands located at 3222 cm⁻¹ in the infrared spectrum of H_2L and at 3289 cm⁻¹ in the infrared spectrum of the complex are attributed to the N–H stretches.

complex						
H ₂ L						
Bond lengths						
C7-N1	1.269(5)	N1-N2	1.386(5)			
N2-C8	1.343(6)	C8-O2	1.228(5)			
Bond angles						
C7-N1-N2	116.9(4)	N1-N2-C8	119.3(3)			
N2-C8-O2	123.0(4)					
the complex						
Bond lengths						
V1-01	1.858(6)	V1-O2	1.970(5)			
V1-N1	2.074(7)	V1-O5	2.202(6)			
V1-06	1.857(6)	V1-07	1.592(7)			
V2-08	1.879(6)	V2-O9	1.935(6)			
V2-O13	1.866(6)	V2-O14	1.586(6)			
V2-N5	2.079(7)	V2-O12	2.243(6)			
C7-N1	1.294(10)	N1-N2	1.388(9)			
N2-C8	1.305(11)	C8-O2	1.307(10)			
C28-N5	1.293(12)	N5-N6	1.406(9)			
N6-C29	1.296(11)	C29-O9	1.303(10)			
Bond angles						
07-V1-06	95.0(3)	07-V1-01	99.5(3)			
06-V1-01	107.6(3)	O7-V1-O2	100.5(3)			
O6-V1-O2	89.1(2)	O1-V1-O2	152.7(3)			
07-V1-N1	97.2(3)	O6-V1-N1	161.3(3)			
01-V1-N1	84.3(3)	O2-V1-N1	74.8(2)			
07-V1-05	171.0(3)	O6-V1-O5	76.1(2)			
01-V1-05	82.0(2)	O2-V1-O5	81.2(2)			
N1-V1-O5	91.8(2)	O14-V2-O13	97.1(3)			
O14-V2-O8	99.0(3)	O13-V2-O8	104.8(3)			
O14-V2-O9	101.4(3)	O13-V2-O9	91.3(3)			
O8-V2-O9	152.1(3)	O14-V2-N5	97.1(3)			
O13-V2-N5	162.0(3)	O8-V2-N5	83.8(3)			
O9-V2-N5	75.0(3)	O14-V2-O12	172.2(3)			
O13-V2-O12	75.2(2)	O8-V2-O12	82.3(2)			
O9-V2-O12	80.1(2)	N5-V2-O12	90.7(2)			
C7-N1-N2	117.5(7)	N1-N2-C8	109.2(7)			
N2-C8-O2	121.8(8)	C28-N5-N6	116.6(7)			
N5-N6-C29	108.7(7)	N6-C29-O9	121.9(8)			

 TABLE 2

 Selected bond lengths (Å) and bond angles (°) for H₂L and the complex

The broad and weak bands indicative of the ν_{O-H} vibrations of the hydroxyl groups of the ligand is centered at 3456 cm⁻¹. The strong band observed at 1610 cm⁻¹ is attributed to the conjugated C=N-N=C moiety of the benzohydrazone ligand. The middle band at 973 cm⁻¹ is assigned to the V=O stretch.^[15] The bands due to ν (V–O) and ν (V–N) are occurred at 443, 530, and 595 cm⁻¹, respectively.

Structure Description of H₂L and the Complex

Figures 1 and 2 give perspective views of H_2L and the oxovanadium(V) complex. H_2L crystallizes in the monoclinic space



FIG. 1. Molecular structure with atomic numbering for H_2L . Hydrogen bond is shown as a dashed line.

group $P2_1/c$ and the complex crystallizes in the triclinic space group P-1.

In the free benzohydrazone molecule, there is an intramolecular O1-H1...N1 hydrogen bond, which contributes to the planarity of the benzene ring and the methylidene unit. The dihedral angle between the two benzene rings is $14.3(3)^{\circ}$. The oxovanadium complex, derived from H₂L and benzohydroxamic acid, is a mononuclear compound. In the asymmetric unit of the complex, there are two independent complex molecules and an acetonitrile molecule of crystallization. The benzohydrazone ligands form one five- and one six-membered chelate rings with bite angles of $74.8(2)^{\circ}$ and $84.3(3)^{\circ}$ for one molecule and $75.0(3)^{\circ}$ and $83.8(3)^{\circ}$ for the other. The benzohydroxamic acid ligands form five-membered chelate rings with bite angles of $76.1(2)^{\circ}$ for one molecule and $75.2(2)^{\circ}$ for the other. The V1 and V2 atoms are in distorted octahedral O5N coordination spheres. For the V1 coordination, the equatorial plane is defined by the phenolate O1 atom, imine N1 atom, and enolic O2 atom of the benzohydrazone ligand, and the O6 atom of the benzohydroxamic acid ligand, and the axial positions are occupied by the O5 atom of the benzohydroxamic acid ligand and one oxo O7 atom. The displacement of the V1 atom from the equatorial plane towards the axial oxo group is 0.263(1) Å. For the V2



FIG. 2. Molecular structure with atomic numbering for the complex. Hydrogen bond is shown as a dashed line.



FIG. 3. Molecular packing diagram of H_2L , viewed along the *b*-axis. Hydrogen bonds are shown as dashed lines.

coordination, the equatorial plane is defined by the phenolate O8 atom, imine N5 atom, and enolic O9 atom of the benzohydrazone ligand, and the O13 atom of the benzohydroxamic acid ligand, and the axial positions are occupied by the O12 atom of the benzohydroxamic acid ligand and one oxo O14 atom. The displacement of the V2 atom from the equatorial plane towards the axial oxo group is 0.287(1) Å. The *cis* bond angles are in the range 74.8(2)–107.6(3)°, and the *trans* bond angles are in the range 152.1(3)–171.0(3)°, indicating the octahedral coordination is much distorted. The V–O and V–N bond lengths in the complex are similar to those observed in other similar oxovanadium(V) complexes with octahedral coordination.^[16–18] In the benzohydrazone ligands of the complex, the dihedral angles between the two benzene rings are 8.6(3)° and 20.7(3)°.

The coordination of H₂L to the V atoms can be reflected by the changes of the corresponding bond lengths and angles (Table 2). The bond lengths of C7=N1, C28=N5, N1–N2, and N5–N6 in the complex are much longer than the corresponding bonds observed in H₂L. However, the distances of C8–N2 and C29–N6 in the complex are much shorter than C8–N2 in H₂L, and the distances of C8–O2 and C29–O9 in the complex are much longer than C8–O2 in H₂L, indicating the enolization of the N2–C8–O2 and N6–C29–O9 groups in the complex.

In the crystal structure of H_2L , benzohydrazone molecules are linked through intermolecular N–H…O hydrogen bonds (Table 3), forming chains running along the *c*-axis (Figure 3). In the crystal structure of the oxovanadium complex, the acetonitrile molecules are linked to the oxovanadium complex molecules



FIG. 4. Molecular packing diagram of the complex, viewed along the *a*-axis. Hydrogen bonds are shown as dashed lines.



SCH. 2. The oxidation reaction.

through intermolecular N–H···N hydrogen bonds (Table 3; Figure 4).

Catalytic Property

The catalytic oxidation test of the complex on the oxidation of sulfide under homogeneous conditions in solution using methyl phenyl sulfide as a substrate is shown as Scheme 2. As oxidant hydrogen peroxide was used in a slight excess of 1.25

Distances (1) and angles () informing hydrogen bonaning of 1122 and the complex						
<i>D</i> -H···A	d(D-H)	$d(\mathbf{H}\cdots A)$	$d(D \cdots A)$	Angle $(D-H\cdots A)$		
H ₂ L						
O1-H1···N1	0.85(1)	1.99(5)	2.626(5)	131(6)		
$N2-H2\cdots O2^{i}$	0.90(1)	2.07(2)	2.911(4)	156(5)		
the complex						
N8–H8A…N9	0.86	2.11	2.932(13)	159		

TABLE 3 Distances (Å) and angles (°) involving hydrogen bonding of H_2L and the complex

Symmetry code for i: x, 3/2 - y, 1/2 + z.

equivalents based on the sulfide substrate. Reactions were run with 1 mol% of catalyst based on the substrate at a temperature of 10°C. NMR techniques have been used to monitor the formation of the sulfoxides with 1,3,5-trimethoxybenzene as internal standard to determine the yields. The reaction was started by the addition of hydrogen peroxide. A control reaction under the same condition without any complex present leads to less than 1% sulfide conversion within 4 h. In the presence of the complex conversion of 91% of sulfide to sulfoxide within 60 min reaction time were observed. After about 2 h the conversion of a total amount of sulfide was complete. Under the given conditions no over oxidation to the sulfone could be detected. It can be seen that the complex has effective catalytic property for the sulfoxidation.

CONCLUSION

In the present work, a new benzohydrazone compound N'-(3-bromo-2-hydroxybenzylidene)-4-nitrobenzohydrazide, and a new oxovanadium(V) complex derived from the benzohydrazone compound and benzohydroxamic acid have been successively prepared and structurally characterized. The benzohydrazone ligand coordinates to the V atom through the phenolate O, imino N, and enolic O atoms. The complex is an effective catalyst for the sulfoxidation reaction of sulfide to sulfoxide.

SUPPLEMENTARY MATERIALS

Crystallographic data for the complexes have been deposited with the Cambridge Crystallographic Data Centre (CCDC 901044 for H_2L and 901045 for the complex). Copies of these information may be obtained free of charge from the Director, CCDC, 12 Union Road, Cambridge, CB2 1EZ, UK (fax: +44–1223-336033; Email: deposit@ccdc.cam.ac.uk or http://www.ccdc.cam.ac.uk).

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