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Synthesis, Crystal Structures, and Antifungal Activities of N'-(3,5-Dichloro-2-hydroxybenzylidene)-2-hydroxy-3-methylbenzohydrazide and its Oxovanadium(V) Complex [N'-(3,5-Dichloro-2-oxidobenzylidene- κ O)-2-hydroxy-3-methylbenzohydrazidato- κ^2 N',O](methanol- κ O)(methanolato- κ O)oxidovanadium(V)

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Synthesis, Crystal Structures, and Antifungal Activities of *N'*-3,5-Dichloro-2-hydroxybenzylidene)-2-hydroxy-3-methylbenzohydrazide and its Oxovanadium(V) Complex [*N'*-(3,5-Dichloro-2-oxidobenzylidene- κO)-2-hydroxy-3-methylbenzohydrazidato- $\kappa^2 N', O$](methanol- κO)(methanolato- κO)oxidovanadium(V)

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A new hydrazine ligand, *N'*-3,5-dichloro-2-hydroxybenzylidene)-2-hydroxy-3-methylbenzohydrazide, and its oxovanadium(V) complex, [*N'*-(3,5-dichloro-2-oxidobenzylidene- κO)-2-hydroxy-3-methylbenzohydrazidato- $\kappa^2 N', O$](methanol- κO)(methanolato- κO)oxidovanadium(V), were prepared and were structurally characterized by elemental analysis, infrared (IR) spectra, and single-crystal x-ray diffraction. The hydrazine ligand coordinates to the V atom through the phenolic O, imino N, and ethanolic O atoms. The V atom in the complex is in an octahedral coordination, with the three donor atoms of the hydrazine ligand and one methanolato O atom defining the equatorial plane, and with one methanol O atom and one oxo O atom occupying the axial positions. The displacement of the V atom from the equatorial plane toward the axial oxo O atom is 0.308(2) Å. The hydrazine ligand and the complex showed moderate to strong antifungal activities against the fungi *Aspergillus flavus*, *Fusarium*, and *Candida albicans*.

Keywords antifungal activity, crystal structure, hydrazine, oxovanadium(V) complex, synthesis

INTRODUCTION

The coordination chemistry of oxovanadium complexes with multidentate ligands has attracted much interest for both their structures and their biological activities.^[1–5] In recent years, a large number of oxovanadium complexes have been reported; however, the number of oxovanadium complexes with hydrazine ligands is relatively small.^[6–8] As described in this article, a

new hydrazine ligand, *N'*-3,5-dichloro-2-hydroxybenzylidene)-2-hydroxy-3-methylbenzohydrazide (Scheme 1), was prepared. Based on the ligand, a new oxovanadium(V) complex, [*N'*-(3,5-dichloro-2-oxidobenzylidene- κO)-2-hydroxy-3-methylbenzohydrazidato- $\kappa^2 N', O$](methanol- κO)(methanolato- κO)oxidovanadium(V), was successfully prepared. Both the hydrazine ligand and the complex were characterized by elemental analysis, infrared (IR) spectra, and single-crystal x-ray diffraction. The antifungal activities of the compounds were investigated.

EXPERIMENTAL

Materials and Measurements

3,5-Dichlorosalicylaldehyde and 2-hydroxy-3-methylbenzohydrazide were purchased from Aldrich. Other chemicals and solvents were of reagent grade and were used as obtained. Elemental analyses were performed on a Carlo-Erba EA micro-analyzer. Infrared (IR) spectra were recorded as KBr discs on a Bruker IFS-66V spectrometer. The ¹H-nuclear magnetic resonance (NMR) spectrum of the hydrazine ligand was recorded on a Bruker 400-MHz spectrometer and CDCl₃ was used as the solvent.

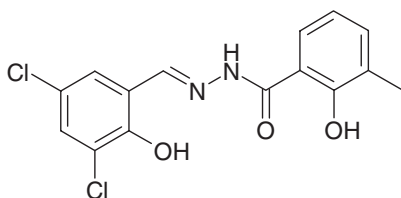
Synthesis of *N'*-3,5-Dichloro-2-hydroxybenzylidene)-2-hydroxy-3-methylbenzohydrazide

A methanol solution (30 mL) of 3,5-dichlorosalicylaldehyde (2.0 mmol, 380 mg) was added dropwise to a stirred methanol solution (20 mL) of 2-hydroxy-3-methylbenzohydrazide (2.0 mmol, 332 mg). The mixture was stirred at room temperature for 1 h to give a clear colorless solution. The solution was evaporated to give a colorless solid product of the hydrazine ligand. Yield: 96%. Anal.: Calcd. (%) for C₁₅H₁₂Cl₂N₂O₃: C, 53.1; H, 3.6; N, 8.3. Found (%): C, 53.3; H, 3.6; N, 8.2. ¹H-NMR data (CDCl₃, ppm): δ = 2.13 (s, 3H), 6.92 (q, 1H), 7.31

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SCH. 1. *N'*-3,5-Dichloro-2-hydroxybenzylidene)-2-hydroxy-3-methylbenzohydrazide.

(d, 1H), 7.47 (s, 1H), 7.54 (s, 1H), 7.66 (d, 1H), 8.17 (s, 1H), 8.72 (s, 1H), 10.1 (b, 2H). Single crystals of the compound were obtained by recrystallization of the product in methanol.

Synthesis of [*N'*-(3,5-Dichloro-2-oxidobenzylidene- κ O)-2-hydroxy-3-methylbenzohydrazidato- κ^2 *N',O*](methanol- κ O)(methanolato- κ O)oxidovanadium(V)

To a stirred methanol solution (5 mL) of the hydrazine ligand (0.5 mmol, 169 mg) was added dropwise a methanol solution (5 mL) of VO(acac)₂ (0.5 mmol, 132 mg). The mixture was stirred for 1 h at room temperature to give a brown solution. Brown crystals of the complex that were suitable for x-ray diffraction were obtained after 1 week for slow evaporation. Yield: 53%. Anal.: Calcd. (%) for C₁₇H₁₇Cl₂N₂O₆V: C, 43.7; H, 3.7; N, 6.0. Found (%): C, 43.5; H, 3.8; N, 6.1.

X-Ray Crystallography

Each single crystal of the compounds was mounted on a glass fiber. Intensity data were collected on a Bruker Smart 1000 CCD diffractometer. Intensities and lattice parameters were obtained with graphite-monochromated Mo K α radiation ($\lambda = 0.71073$ Å). All data were corrected for Lorentz and polarization effects. Empirical absorption corrections were also applied for both the crystal structures obtained.^[9] Complex scattering factors were taken from the program package SHELXTL.^[10] The structures of the compounds were solved by direct methods, which revealed the position of all non-hydrogen atoms. Both the structures were refined on F^2 by a full-matrix least-squares procedure using anisotropic displacement parameters for all non-hydrogen atoms. The amino H atom in the hydrazine ligand and the methanol H atom in the complex were located from difference Fourier maps and refined isotropically, with N–H and O–H distances restrained to 0.90(1) and 0.85(1) Å, respectively. The remaining hydrogen atoms were located in their calculated positions and refined using a riding model. The details of the x-ray crystal data and the structure solution and refinement for both compounds are given in Table 1. Selected bond lengths and angles are given in Table 2. Crystallographic data for both compounds have been deposited in the Cambridge Crystallographic Data Centre (CCDC 817822 and 817823).

RESULTS AND DISCUSSION

The hydrazine ligand and the oxovanadium(V) complex are stable in air at room temperature. They are soluble in methanol, ethanol, and acetonitrile. The elemental analyses are in good

TABLE 1
Crystallographic data

Compound	Hydrazine ligand	Complex
Empirical formula	C ₁₆ H ₁₆ Cl ₂ N ₂ O ₄	C ₁₇ H ₁₇ Cl ₂ N ₂ O ₆ V
Formula weight	371.21	467.2
Crystal shape/color	Block/colorless	Block/brown
Crystal size (mm)	0.10 × 0.10 × 0.07	0.23 × 0.21 × 0.20
Crystal system	Triclinic	Triclinic
Space group	<i>P</i> -1	<i>P</i> -1
<i>a</i> (Å)	7.338(2)	7.835(2)
<i>b</i> (Å)	10.624(3)	11.695(3)
<i>c</i> (Å)	11.151(3)	12.306(2)
α (°)	98.377(3)	106.723(3)
β (°)	102.777(3)	101.504(3)
γ (°)	91.353(3)	99.250(3)
<i>V</i> (Å ³)	837.3(4)	1029.2(4)
<i>Z</i>	2	2
<i>D</i> _{calc} (g/cm ³)	1.472	1.507
<i>T</i> (K)	298(2)	298(2)
μ (mm ⁻¹)	0.411	0.777
<i>F</i> (000)	384	476
Range of <i>h, k, l</i>	−9/9, −13/12, −14/14	−9/9, −13/14, −15/15
Max. and min. transmission	0.960 and 0.972	0.842 and 0.860
Unique reflections	3562	4340
Observed reflections	2269	2575
<i>I</i> > 2 σ (<i>I</i>)		
Data/restraints/parameters	3562/14/225	4340/1/260
Goodness-of-fit on F^2	1.027	1.062
<i>R</i> _{int}	0.0325	0.0368
<i>R</i> ₁ [<i>I</i> ≥ 2 σ (<i>I</i>)]	0.0664	0.0659
<i>wR</i> ₂ [<i>I</i> ≥ 2 σ (<i>I</i>)]	0.1497	0.1080
<i>R</i> ₁ (all data)	0.1023	0.1194
<i>wR</i> ₂ (all data)	0.1690	0.1224

agreement with the chemical formulas proposed for the compounds.

The hydrazine compound *N'*-3,5-dichloro-2-hydroxybenzylidene)-2-hydroxy-3-methylbenzohydrazide is a flexible multidentate ligand, which can coordinate to the metal ions through the two phenolic O, imino N, and enolic O atoms. The V atoms usually adopt octahedral and square-pyramidal coordination.^[11–13] The methanol was proved to be a preferred solvent for the preparation of such complexes.

Structure of the Hydrazine Ligand

The molecular structure of the hydrazine ligand is shown in Figure 1. The compound consists of a hydrazine molecule and a methanol molecule of crystallization. The hydrazine molecule

TABLE 2
Selected bond lengths (Å) and angles (°) for the hydrazine ligand and the complex

Hydrazine ligand			
Bond lengths			
O1–C2	1.348(4)	N1–C7	1.270(4)
N1–N2	1.371(4)	N2–C8	1.364(4)
O2–C8	1.238(4)	C8–C9	1.473(4)
Complex			
Bond lengths			
O1–C2	1.331(4)	N1–C7	1.294(4)
N1–N2	1.400(4)	N2–C8	1.318(4)
O2–C8	1.315(4)	C8–C9	1.486(5)
V1–O1	1.885(3)	V1–O2	1.978(2)
V1–O4	2.336(3)	V1–O5	1.769(2)
V1–O6	1.591(3)	V1–N1	2.150(3)
Bond angles			
O1–V1–O2	152.39(11)	O1–V1–N1	82.46(11)
O1–V1–O4	81.46(11)	O2–V1–O4	80.22(10)
O2–V1–N1	74.58(10)	N1–V1–O4	81.63(10)
O5–V1–O1	100.13(11)	O5–V1–O2	96.84(10)
O5–V1–O4	80.02(10)	O5–V1–N1	160.84(11)
O6–V1–O1	99.72(14)	O6–V1–O2	97.80(13)
O6–V1–O4	177.30(11)	O6–V1–O5	102.10(12)
O6–V1–N1	96.10(12)		

TABLE 3
Hydrogen geometries

$D-H\cdots A^a$	$d(D-H, \text{Å})$	$d(H\cdots A, \text{Å})$	$d(D\cdots A, \text{Å})$	Angle ($D-H\cdots A, ^\circ$)
Hydrazine ligand				
N2–H2...O4 ^{#1}	0.90(1)	1.99(2)	2.860(5)	164(4)
O4–H4...N2 ^{#2}	0.82	2.42	2.860(5)	115
O3–H3...C12 ^{#3}	0.82	2.89	3.330(3)	116
O3–H3...O2	0.82	1.84	2.559(4)	146
O1–H1...N1	0.82	1.84	2.551(4)	144
C14–H14...O4 ^{#2}	0.93	2.54	3.446(4)	164
The complex				
O3–H3...N2	0.82	1.84	2.564(3)	147
O4–H4...O3 ^{#4}	0.84(1)	1.928(2)	2.755(3)	167(4)

^aSymmetry codes: #1: $x, 1 + y, z$; #2: $x, -1 + y, z$; #3: $x, y, 1 + z$; #4: $1 - x, -y, 1 - z$.

the formation of the planar geometry of the molecule. The hydrazine molecule adopts a *trans* configuration about the C7–N1 bond. The long C7–N1 bond [1.270(4) Å] and the short N1–N2 bond [1.371(4) Å] suggest the delocalization of the acetohydrazide moiety. All the bond distances in the compound are within normal ranges^[14] and are comparable to the similar compounds.^[15,16]

In the crystal structure of the compound (Figure 2), the methanol molecules are linked to the hydrazine molecules through intermolecular C–H...O, N–H...O, and O–H...N hydrogen bonds (Table 3). The molecules are further linked by intermolecular O–H...Cl hydrogen bonds, to form chains along

is approximately coplanar, with mean deviation from the least-squares plane defined by the non-hydrogen atoms of 0.050(1) Å, and with the dihedral angle between the C1–C6 and C9–C14 benzene rings of 3.8(2)°. The two intramolecular O1–H1...N1 and O3–H3...O2 hydrogen bonds (Table 3) may contribute to

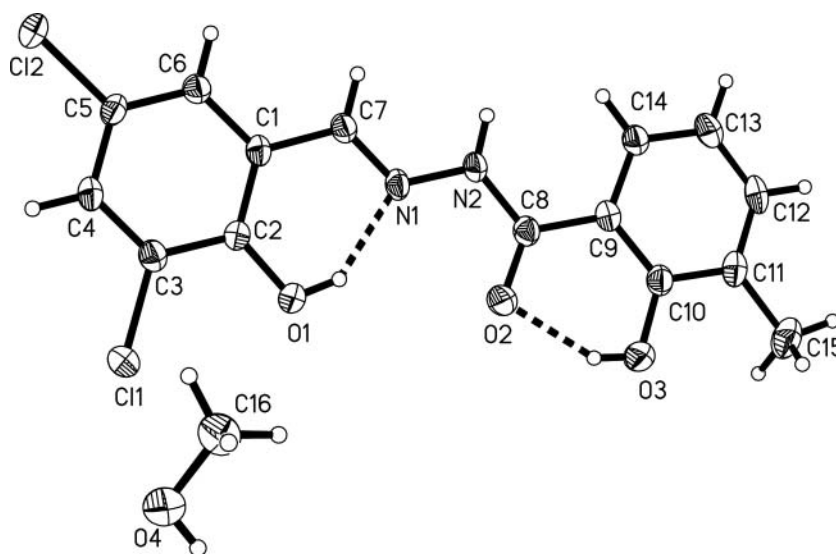


FIG. 1. Molecular structure of the hydrazine ligand with 30% probability ellipsoids.

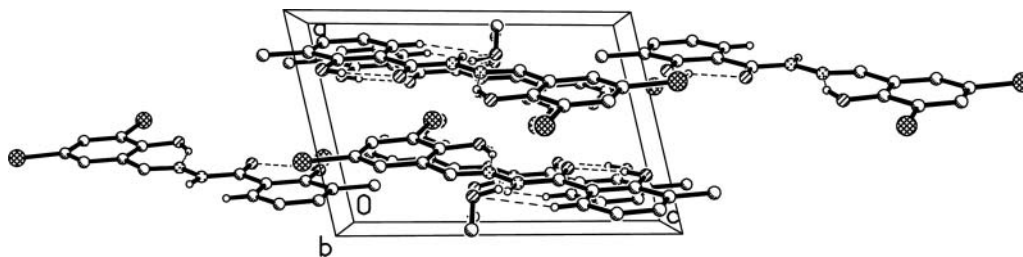


FIG. 2. Molecular packing diagram of the hydrazine ligand.

the unit cell c axis. In addition, there are weak $\pi \cdots \pi$ stacking interactions among the molecules.

Structure of the Oxovanadium(V) Complex

The molecular structure of the oxovanadium(V) complex is shown in Figure 3. The dianionic hydrazine ligand coordinates to the V atom through the phenolic O, imino N, and enolic O atoms, forming five-membered and six-membered chelate rings with bite angles of $74.6(1)$ and $82.5(1)^\circ$. The V atom in the complex is in an octahedral coordination, with the three donor atoms of the hydrazine ligand and one methanolato O atom defining the equatorial plane, and with one methanol O atom and one oxo O atom occupying the axial positions. The displacement of the V atom from the equatorial plane toward the axial oxo O atom is $0.308(2)$ Å. The coordinate bond lengths and angles in the present structure are similar to those reported elsewhere.^[11–13] The formation of the coordinate bonds with the V atom for the hydrazine ligand, together with the existence of the intramolecular $O3-H3 \cdots N2$ hydrogen bond (Table 3), leads to the planarity of the hydrazine ligand. The dihedral angle between the C1–C6 and C9–C14 benzene rings is $5.0(2)^\circ$.

In the crystal structure of the complex (Figure 4), the adjacent two complex molecules are linked together by the coordinated methanol molecules through two intermolecular O–H \cdots O hydrogen bonds (Table 3).

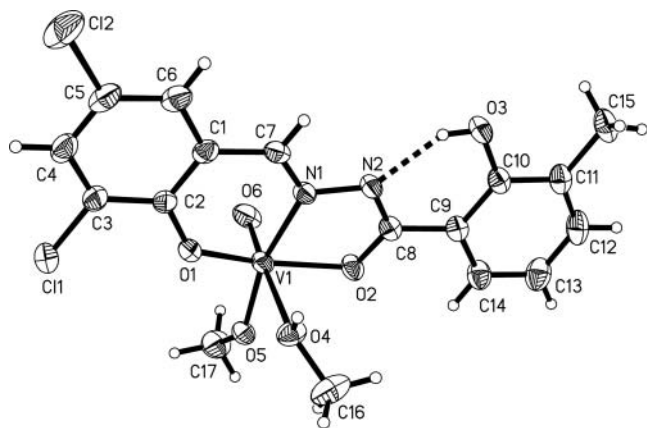


FIG. 3. Molecular structure of the complex with 30% probability ellipsoids.

IR Spectra

The typical band centered at 3427 cm^{-1} of the hydrazine ligand can be attributed to the phenolic O–H stretches. The $\nu(\text{C}=\text{O})$ absorption band at 1651 cm^{-1} and the sharp $\nu(\text{NH})$ band at 3217 cm^{-1} in the IR spectrum of the hydrazine ligand are absent in the complex, indicating the enolization of the amide functionality and subsequent proton replacement by the V atom. The middle absorption band at 1332 cm^{-1} in the spectrum of the complex is assigned to the $\nu(\text{C}=\text{O})$ (enolic) vibration. The intense absorption at 1627 cm^{-1} in the hydrazine ligand can be assigned to the $-\text{C}=\text{N}-$ group, which is observed at the lower frequency (1602 cm^{-1}) in the complex, indicating the coordination of the imino N atom to the V center. The band indicative of the $\text{V}=\text{O}$ vibration is at 917 cm^{-1} . And the lower frequencies in the range $430\text{--}480\text{ cm}^{-1}$ can be attributed to the V–O and V–N vibrations.

Antifungal Study

The hydrazine ligand and the oxovanadium(V) complex were tested for their *in vitro* growth inhibition against the fungi *Aspergillus flavus*, *Fusarium*, and *Candida albicans* on sabour dextrose agar medium, according to the literature method.^[17] The test solutions of two different concentrations were prepared in dimethyl sulfoxide (DMSO). The percentage inhibition of fungal growth was determined on the growth in test plates compared to that of respective control plates, given by the Vincent equation: Percent inhibition = $100(C - T)/C$, where C is the diameter of fungal growth on the control plate, and T is the diameter of fungal growth on the test plate.^[18] The results are listed in Table 4.

The hydrazine ligand showed moderate antifungal activities against the three fungi *Aspergillus flavus*, *Fusarium*, and *Can-*

TABLE 4
Antifungal activity (% inhibition)

Compound ($\mu\text{g/L}$)	<i>Aspergillus flavus</i>		<i>Fusarium</i>		<i>Candida albicans</i>	
	100	200	100	200	100	200
Hydrazine ligand	27	43	40	55	32	63
Complex	53	76	62	81	65	75

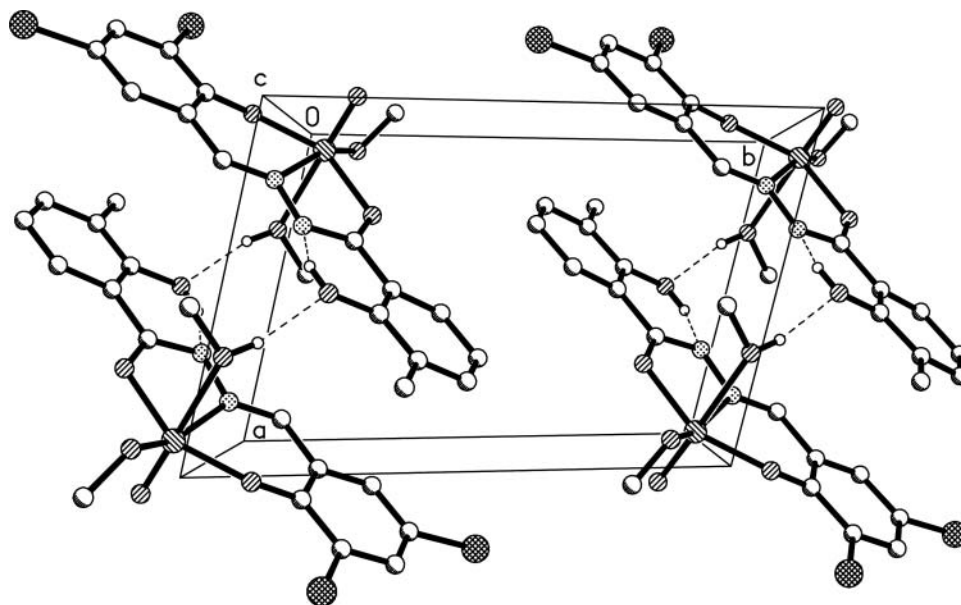


FIG. 4. Molecular packing diagram of the complex.

didia albicans. It is obvious that the antifungal activities of the oxovanadium(V) complex are much stronger than those of the hydrazine ligand. Such increased activity of the metal complexes can be explained on the basis of chelation theory.^[19] On chelation, the polarity of the metal ion will be reduced to a great extent due to the overlap of the ligand orbital. Furthermore, it increases the delocalization of π -electrons over the whole chelate ring and enhances the lipophilicity of the complexes.^[20] This increased lipophilicity leads to breakdown of the permeability barrier of the cell and thus retards the normal cell processes.^[21]

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