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Isopropoxy[2′-(2-methoxybenzoyl)-2-oxidobenzohydrazidato]oxovanadium(V)

Li-Juan Chen,[a,b] Ming-Xing Yang[a] and Shen Lin[a]*

[a]Deptartment of Chemistry, Fujian Normal University, Fuzhou 350007, People's Republic of China, and [b]State Key Laboratory of Structural Chemistry, Fujian Institute of Research on the Structure of Matter, The Chinese Academy of Science, Fuzhou, Fujian 350002, People's Republic of China

Correspondence e-mail: shenlin_fz@163.com

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.039
wR factor = 0.117
Data-to-parameter ratio = 17.3

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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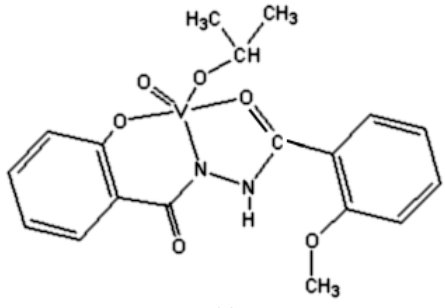
The V^V ion in the title complex, $[V(C_{15}H_{12}N_2O_4)(C_3H_7O)O]$, is in a distorted square-pyramidal environment. Two O atoms and one N atom from the tridentate hydrazidate ligand, and an O atom from the depronated solvent molecule, define the basal plane, and the oxo O atom occupies the apical position.

Comment

Vanadium is a trace metal in diverse living forms (Bhattacharyya *et al.*, 2002). It plays an active role in many biologically important reactions, such as halogenation of organic substrates and activation or fixation of nitrogen through an alternative pathway (Butler & Walker, 1993). In order to gain an insight into the intricate roles of vanadium in biological systems, it is necessary to acquire information about the stereochemistry and reactivity of its coordination compounds that contain a biologically relevant ligand donor set (Rehder, 1991). Most Schiff base ligands are coordinated to the metal center through their O/N atoms and are similar to the coordination environments of the biological system. A number of oxovanadium complexes with hydroxamate and hydrazone ligands have been studied (Rath *et al.*, 1999; Rajak *et al.*, 2000; Pal & Pal, 2001). However, there are only a few reports of vanadium complexes with salicylhydrazidate ligands (Palacios *et al.*, 1997). We present here the synthesis and crystal structure of a novel VO^{3+} complex, (I), with the *N*-*o*-methoxybenzoylsalicylhydrazidate ligand.



(I)

The molecular structure of the title complex is shown in Fig. 1. The V atom adopts a distorted square-pyramidal coordination environment. Atoms O1, O3 and N1 from the dianionic tridentate hydrazidate ligand and atom O6 from the depronated solvent molecule define the basal plane, with an r.m.s deviation of 0.020 Å, and oxo atom O5 occupies the apical position. The V atom deviates by 0.4510 (9) Å from the basal plane towards the oxo O atom. Bond lengths and angles around atom V1 (Table 1) are comparable with reported

values (Liu & Gao, 1998; Tsiamis *et al.*, 2000; Tsuchimoto *et al.*, 1999).

As shown in Fig. 1, there exists an intramolecular hydrogen bond between the uncoordinated hydrazine group of the ligand and the O atom of the methoxy group [N2···O4 = 2.616 (3) Å and N2—H2N···O4 = 128°].

Experimental

The material $VO(acac)_2$ (acac = acetylacetonate) was synthesized according to the reported procedure of Gao *et al.* (1998). The ligand *N*-*o*-methoxybenzoylsalicylhydrazidate (H_2L) was prepared by condensing salicylhydrazide with *o*-methyoxybenzoic acid in chloroform. The title compound was prepared by reacting H_2L (0.1 mmol) with $[VO(acac)_2]$ (0.1 mmol) in 2-propanol solvent with stirring. The mixed solution was filtered and then kept at ambient temperature. Dark-red crystals of the title complex were formed after one week.

Crystal data

$[V(C_{15}H_{12}N_2O_4)(C_3H_7O)O]$	D_x = 1.467 Mg m^{-3}
M_r = 410.29	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 4227 reflections
a = 10.835 (8) Å	θ = 2.4–27.5°
b = 9.882 (8) Å	μ = 0.57 mm^{-1}
c = 17.7300 (12) Å	T = 293 (2) K
β = 101.84 (3)°	Prism, dark red
V = 1858 (2) Å^3	0.6 × 0.4 × 0.3 mm
Z = 4	

Data collection

Rigaku R-AXIS RAPID diffractometer	4226 independent reflections
	3336 reflections with $I > 2\sigma(I)$
ω scans	R_{int} = 0.028
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1999)	θ_{max} = 27.5°
	$h = 0 \rightarrow 14$
T_{min} = 0.655, T_{max} = 0.843	$k = 0 \rightarrow 12$
16 277 measured reflections	$l = -23 \rightarrow 22$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.066P)^2 + 0.1239P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.039	
$wR(F^2)$ = 0.117	
S = 1.08	$(\Delta/\sigma)_{max} < 0.001$
4226 reflections	$\Delta\rho_{max}$ = 0.48 e Å^{-3}
244 parameters	$\Delta\rho_{min}$ = −0.26 e Å^{-3}
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

V1—O5	1.5834 (16)	V1—O3	1.9824 (14)
V1—O6	1.7402 (16)	V1—N1	2.0307 (18)
V1—O1	1.8331 (16)		
O5—V1—O6	105.40 (9)	O1—V1—O3	149.26 (6)
O5—V1—O1	104.39 (8)	O5—V1—N1	103.54 (9)
O6—V1—O1	99.29 (9)	O6—V1—N1	149.35 (7)
O5—V1—O3	101.83 (8)	O1—V1—N1	82.98 (8)
O6—V1—O3	88.84 (7)	O3—V1—N1	75.49 (7)

H atoms were placed in calculated positions and were included in the refinement in the riding-model approximation [N—H = 0.86 Å, C—H = 0.93–0.98 Å and U_{iso}(H) = 1.2U_{eq}(parent atom) or 1.5U_{eq}(C_{methyl})].

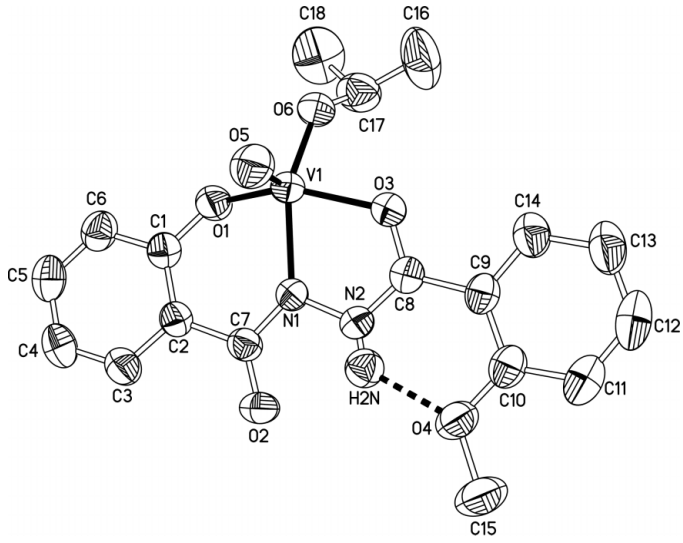


Figure 1
The structure of the title complex, showing 50% probability displacement ellipsoids and the atomic numbering scheme. The N—H···O hydrogen bond is shown as a dashed line. All H atoms except H2N have been omitted for clarity.

Data collection: *RAPID-AUTO* (Rigaku, 1999); cell refinement: *RAPID-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2002); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL*/*PC* (Sheldrick, 1998); software used to prepare material for publication: *SHELXL*97.

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