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Ethanol(ethoxy)[3-methoxysalicylaldehyde (4-methoxybenzoyl)hydrazonato-κ^3O,N,O']-oxovanadium(V)

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The six-coordinated V^V atom in the title complex, [VO*L*(OCH_2CH_3)(CH_3CH_2OH)] [H_2L = 3-methoxysalicylaldehyde (4-methoxybenzoyl)hydrazone] or [VO(C_2H_5O)($C_{16}H_{14}N_2O_4$)(C_2H_6O)], displays a distorted octahedral VO(ONO)(O)(O) coordination. The equatorial plane is defined by one N and two O atoms of the tridentate hydrazone ligand and the ethoxy O atom, while the axial oxo group is *trans* to the ethanol O atom. Adjacent molecules are linked by intermolecular hydrogen bonds into a chain structure along the *c* axis.

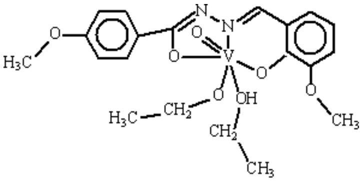
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.005 Å
Disorder in main residue
R factor = 0.060
wR factor = 0.148
Data-to-parameter ratio = 17.8

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

Comment

The study of vanadium hydrazone complexes has attracted the interest of many researchers owing to their possible function as bioenzyme models (Sangeetha & Pal, 2000; Kurzak *et al.*, 1992). We have previously reported the structures of some vanadium(V) complexes containing tridentate hydrazone ligands, in which the hydrazone ligands are formed by condensing benzoylhydrazine with acetylacetone, salicylaldehyde or 3-methoxysalicylaldehyde (Chen *et al.*, 1999; Gao *et al.*, 1998; Liu & Gao, 1998). Recently, we have obtained a new oxovanadium(V) complex, [VO*L*(OCH_2CH_3)(CH_3-CH_2OH)] [H_2L = 3-methoxysalicylaldehyde (4-methoxybenzoyl)hydrazone], (I), which was prepared by the reaction of H_2L and VO(acetylacetonate)$_2$ in ethanol solution. As part of a continuing study of oxovanadium–hydrazonate complexes, we report here the synthesis and structure of the title complex, (I).



(I)

The molecular structure of (I) is illustrated in Fig. 1. The V^V atom is coordinated by five O atoms and one N atom to form a distorted octahedral O_5N configuration. The equatorial plane is defined by atoms O1, O2 and N2 of the fully deprotonated tridentate hydrazone ligand and atom O6 of the ethoxy group (r.m.s. deviation = 0.05 Å). The oxo O7 atom and atom O5 of the ethanol molecule occupy the apical sites with a *trans* angle of 174.8 (1)°. The V atom deviates by 0.32 (4) Å out of the plane and toward the oxo O atom. The V—O7 bond distance of 1.586 (2) Å is within the normal range in the structures of VO^{3+} complexes. The bond distances in the equatorial plane

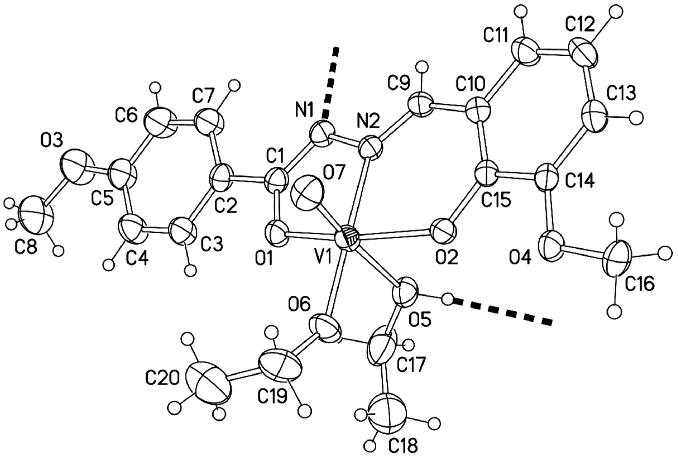


Figure 1

View of the title complex, with displacement ellipsoids shown at the 30% probability level. Hydrogen bonds are shown as dashed lines.

follow the order: alkoxide oxygen < phenoxide oxygen < enolic oxygen < imine nitrogen. Owing to the strong *trans* influence of the oxo ligand, the V—O5 distance of 2.399 (2) Å is the longest of all the V—O bond lengths. The five-membered chelate ring composed of atoms O1, C1, N1, N2 and V1 is essentially planar, with a mean deviation of 0.04 Å, while the six-membered chelate ring composed of atoms O2, C9, C10, C15, N2 and V1 is non-planar. In addition, a chain structure is formed by the intermolecular O—H···N hydrogen bonds of adjacent molecules along the *c* direction (Table 2).

Experimental

VO(acac)$_2$ (acac$^-$ = acetylacetonate) was synthesized according to Rowe & Jones (1957). The ligand H$_2$*L* was synthesized by condensing 3-methoxysalicylaldehyde with an equimolar amount of 4-methoxybenzoylhydrazine in ethanol. An ethanol solution (15 ml) of VO(acac)$_2$ (2 mmol) was added dropwise to an ethanol solution (15 ml) containing H$_2$*L* (2 mmol). The mixture was refluxed with stirring for 1.5 h, cooled slowly to room temperature and filtered. Deep-red crystals were isolated from the filtered solution after a few days. Analysis calculated for $C_{20}H_{25}N_2O_7V$: C 52.18, H 6.34, N 6.09%; found: C 52.37, H 6.45, N 6.00%.

Crystal data

$[VO(C_2H_5O)(C_{16}H_{14}N_2O_4)(C_2H_6O)]$
$M_r = 456.36$
Monoclinic, $P2_1/c$
$a = 19.705$ (4) Å
$b = 10.328$ (2) Å
$c = 11.148$ (2) Å
$\beta = 97.95$ (3)°
$V = 2247.0$ (8) Å^3
$Z = 4$
$D_x = 1.349$ Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 10711 reflections
$\theta = 3.6$–27.4°
$\mu = 0.48$ mm^{-1}
$T = 293$ (2) K
Prism, red
0.39 × 0.25 × 0.21 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{\min} = 0.834$, $T_{\max} = 0.905$
20434 measured reflections
5081 independent reflections
3359 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.061$
$\theta_{\max} = 27.5°$
$h = -24 \rightarrow 25$
$k = -13 \rightarrow 13$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.060$
$wR(F^2) = 0.148$
$S = 1.02$
5081 reflections
286 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0749P)^2 + 0.5402P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.36$ e Å^{-3}
$\Delta\rho_{\min} = -0.27$ e Å^{-3}

Table 1

Selected geometric parameters (Å, °).

V1—N2	2.129 (2)	N1—N2	1.412 (3)
V1—O1	1.958 (2)	N1—C1	1.301 (4)
V1—O2	1.844 (2)	N2—C9	1.287 (4)
V1—O5	2.399 (2)	O1—C1	1.296 (3)
V1—O6	1.761 (2)	O2—C15	1.338 (3)
V1—O7	1.586 (2)		
N2—V1—O5	81.51 (9)	O6—V1—O2	103.0 (1)
O1—V1—N2	73.91 (9)	O6—V1—O5	82.9 (1)
O1—V1—O5	78.67 (8)	O7—V1—N2	93.3 (1)
O2—V1—N2	83.68 (9)	O7—V1—O1	100.2 (1)
O2—V1—O1	149.7 (1)	O7—V1—O2	101.2 (1)
O2—V1—O5	78.10 (9)	O7—V1—O5	174.8 (1)
O6—V1—N2	161.4 (1)	O7—V1—O6	102.2 (1)
O6—V1—O1	93.1 (1)		

Table 2

Hydrogen-bonding geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O5—H21···N1$^{\rm i}$	0.85 (3)	2.07 (3)	2.908 (3)	174 (4)

Symmetry code: (i) $x, \frac{1}{2} - y, \frac{1}{2} + z$.

The ethoxy and ethanol groups are both disordered over two positions, but their occupancies were arbitrarily assumed to be 0.5. The C—O and C—C distances were restrained to be 1.50 (1) Å, and the 1,3-related O···C distance to 2.45 (1) Å. For the C atoms, the displacement parameters of the unprimed and primed atoms were set to be equal to each other; additionally, the displacement parameters were restrained to be approximately isotropic. The H atoms were placed in calculated positions and were allowed to ride on their parent C atoms [C—H = 0.93 Å and $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$ for the aromatic H atoms, and C—H = 0.96 Å and $U_{\rm iso}$(H) = 1.5$U_{\rm eq}$ for the methyl H atoms]. The hydroxyl H atom of ethanol was located in a difference map and refined with an O—H distance restraint of 0.85 (1) Å and $U_{\rm iso}$(H) = 1.5$U_{\rm eq}$(O).

Data collection: *RAPID-AUTO* (Rigaku, 1998); 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: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *SHELXL*97.

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References

Chen, W., Gao, S. & Liu, S.-X. (1999). *Acta Cryst.* C**55**, 531–533.

Gao, S., Weng, Z.-Q. & Liu, S.-X. (1998). *Polyhedron*, **17**, 3595–3606.

Higashi, T. (1995). *ABSCOR*. Rigaku Corporation, Tokyo, Japan.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Kurzak, B., Kozlowski, H. & Farkas, E. (1992). *Coord. Chem. Rev.* **114**, 169–200.

Liu, S.-X. & Gao, S. (1998). *Polyhedron*, **17**, 81–84.

Rigaku (1998). *RAPID-AUTO*. Rigaku Corporation, Tokyo, Japan.

Rigaku/MSC (2002). *CrystalStructure*. Rigaku/MSC Inc., 9009 New Trails Drive, The Woodlands, TX 77381, USA.

Rowe, R. & Jones, M. M. (1957). *Inorg. Synth.* **5**, 113–115.

Sangeetha, N. R. & Pal, S. (2000). *Bull. Chem. Soc. Jpn*, **73**, 357–363.

Sheldrick, G. M. (1997). *SHELXL*97 and *SHELXS*97. University of Göttingen, Germany.

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