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(N-Hydroxy-N-phenylbenzamidato-κ^2O,O')-[3-methoxysalicylaldehyde (2,4-dichlorophenoxyacetyl)hydrazonato-κ^3O,N,O']-oxovanadium(V)

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The V^V atom in the title complex, $[V(C_{16}H_{12}C_{12}N_2O_4)(C_{13}H_{10}NO_2)O]$, is coordinated by two O atoms and one N atom of the tridentate hydrazone ligand, and by two O atoms of the bidentate hydroxamate co-ligand, thus defining a distorted octahedral VO(ONO)(ON) geometry.

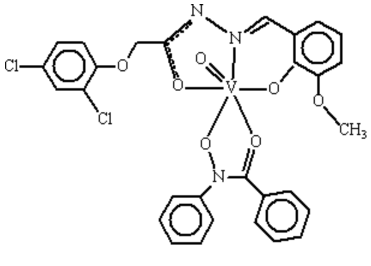
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.003 Å
R factor = 0.042
wR factor = 0.115
Data-to-parameter ratio = 17.0

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

Comment

The presence of oxovanadium(V) at the active sites of some biological enzymes, and the discovery of the biological relevance of hydrazone–vanadium complexes, has resulted in increased interest in the study of their structural properties (Sangeetha & Pal, 2000; Kurzak *et al.*, 1992). In previous work, we have reported the structures of some vanadium(V) complexes containing tridentate hydrazone ligands, in which the hydrazone ligands are formed by condensing benzoylhydrazine or its derivatives with acetylacetone, salicylaldehyde or 3-methoxysalicylaldehyde (Huo, Gao, Liu, Li & Ng, 2004; Huo, Gao, Liu, Zhao & Ng, 2004; Chen *et al.*, 1999; Gao *et al.*, 1998; Liu & Gao, 1998). Here, we present a new oxovanadium(V) complex, (I), $VOL(C_{13}H_{10}NO_2)$ [H_2L = 3-methoxysalicylaldehyde (1,4-dichlorophenoxyacetyl)-hydrazone], obtained from the reaction of N-(3-methoxy salicylaldehyde)-N'-(1,4-dichlorophenoxyacetyl)hydrazone, N-hydroxy-N-phenylbenzamide and $VO(acetylacetonate)_2$ in ethanol solution.



(I)

As shown in Fig. 1, the vanadium(V) atom in (I) is coordinated by two O atoms and one N atom of the tridentate hydrazone ligand, and two O atoms of the bidentate hydroxamate co-ligand, to form a distorted octahedral VO(ONO)(ON) configuration. The equatorial plane is defined by atoms O2, O3 and N1 of the fully deprotonated tridentate hydrazone ligand and the oxime atom O6 of the hydroxamate co-ligand. The axial oxo atom O1 is in a *trans* position relative to the carbonyl atom O7 of the hydroxamate co-ligand, with an O1—V1—O7 angle of 171.34 (7)°. The V atom deviates by 0.27 (2) Å from the equatorial plane towards

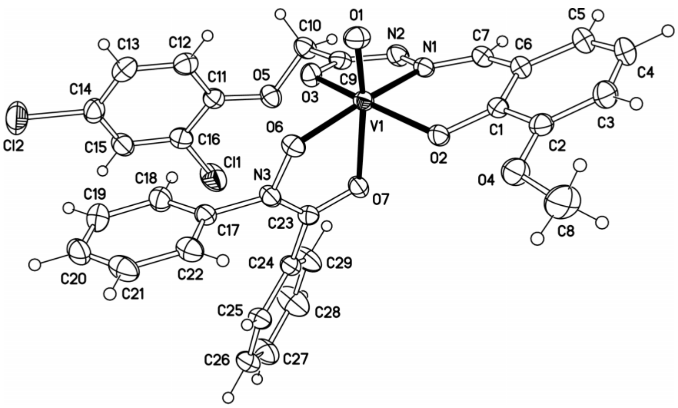


Figure 1
A view of the structure of (I), with 30% probability displacement ellipsoids.

atom O1. The V1—O1 bond distance of 1.5810 (16) Å is within the normal range of values observed in the structures of VO^{3+} complexes (Chen *et al.*, 1999). The bond distances to atom V1 in the equatorial plane follow the order phenoxide O < oxime O < enolic O < imine N. The axial V1—O7 distance of 2.1816 (15) Å is longer than the equatorial V—O bond lengths, due to the strong *trans* influence of the oxo group. The bond lengths (Table 1) indicate some degree of electron delocalization of the hydrazone ligand. The O3—C9—C10—O5 torsion angle is 58.1 (3)°, suggesting that the phenoxyacetate group has some conformational flexibility.

The hydrazone ligand and the hydroxamate co-ligand combine with the V^V atom to give one six-membered and two five-membered chelate rings. The two five-membered chelate rings, involving atoms O3/C9/N2/N1/V1 and O6/N3/C23/O7/V1, are basically planar, with mean deviations of 0.04 (2) and 0.07 (2) Å, respectively, whereas the six-membered chelate ring, involving atoms O2/C1/C6/C7/N1/V1, is non-planar, with an r.m.s. deviation of 0.18 (2) Å. The dihedral angle between the two five-membered chelate rings is 71.7 (7)°. The dihedral angles between the benzene rings C1–C6 and C11–C16, C11–C16 and C17–C22, C17–C22 and C24–C29, and C1–C6 and C24–C29 are 71.3 (6), 69.8 (6), 66.0 (6) and 52.5 (6)°, respectively.

Experimental

The $VO(acac)_2$ starting material ($acac^-$ = acetylacetonate) was synthesized according to the method of Rowe & Jones (1957). The ligand (H_2L), 3-methoxysalicylaldehyde (1,4-dichlorophenoxyacetyl)hydrazone, was synthesized by condensing 3-methoxysalicylaldehyde with an equimolar quantity of (1,4-dichlorophenoxyacetyl)hydrazine in ethanol. An ethanol solution (15 ml) of $VO(acac)_2$ (2 mmol) was added dropwise to an ethanol solution (15 ml) containing *N*-(3-methoxysalicylaldehyde)-*N*′-(1,4-dichlorophenoxyacetyl)hydrazone (2 mmol) and the mixture was refluxed with stirring for 1 h. An ethanol solution containing *N*-hydroxy-*N*-phenylbenzamide (2 mmol) was added slowly to the above solution with stirring for 25 min and then filtered. The resulting dark-red solid was dissolved in dichloromethane and filtered. Dark-red crystals of (I) were obtained by slow diffusion of hexane into the filtrate over several days. CHN analysis, calculated for $C_{29}H_{22}C_{l2}N_3O_7V$: C 53.89, H 3.43, N 6.50%; found: C 53.97, H 3.48, N 6.45%.

Crystal data

$[V(C_{16}H_{12}Cl_2N_2O_4)(C_{13}H_{10}NO_2)O]$
M_r = 646.34
Monoclinic, $C2/c$
a = 26.290 (2) Å
b = 14.445 (2) Å
c = 15.568 (2) Å
β = 107.30 (3)°
V = 5644.6 (15) $Å^3$
Z = 8

D_x = 1.521 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 22 925 reflections
θ = 3.4–27.5°
μ = 0.59 mm^{-1}
T = 293 (2) K
Prism, red
0.39 × 0.27 × 0.20 mm

Data collection

Rigaku R-AXIS RAPID area-detector diffractometer
ω scans
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
T_{min} = 0.802, T_{max} = 0.891
23 639 measured reflections

6460 independent reflections
5251 reflections with $I > 2\sigma(I)$
R_{int} = 0.026
θ_{max} = 27.5°
$h = -34 \rightarrow 34$
$k = -18 \rightarrow 18$
$l = -20 \rightarrow 18$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.042
$wR(F^2)$ = 0.115
S = 1.02
6460 reflections
380 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0608P)^2 + 5.141P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.48 e $Å^{-3}$
$\Delta\rho_{min}$ = −0.33 e $Å^{-3}$

Table 1
Selected geometric parameters (Å, °).

V1—N1	2.0672 (17)	V1—O7	2.1816 (15)
V1—O1	1.5810 (16)	N1—N2	1.397 (2)
V1—O2	1.8452 (14)	N1—C7	1.285 (3)
V1—O3	1.9737 (14)	N2—C9	1.291 (3)
V1—O6	1.8541 (14)	O3—C9	1.291 (2)
N1—V1—O7	88.04 (6)	O2—V1—O6	107.17 (6)
O1—V1—N1	99.88 (8)	O2—V1—O7	85.58 (6)
O1—V1—O2	98.71 (7)	O3—V1—N1	74.41 (6)
O1—V1—O3	99.46 (7)	O3—V1—O7	79.21 (6)
O1—V1—O6	96.27 (8)	O6—V1—N1	159.14 (7)
O1—V1—O7	171.34 (7)	O6—V1—O3	90.02 (6)
O2—V1—N1	83.41 (6)	O6—V1—O7	75.23 (6)
O2—V1—O3	153.37 (7)		

All H atoms were placed in calculated positions and were allowed to ride on their parent C atoms, with C—H = 0.93 Å and $U_{iso}(H) = 1.2U_{eq}$ for aromatic H atoms, and with C—H = 0.96 Å and $U_{iso}(H) = 1.5U_{eq}$ for methyl H atoms.

Data collection: *RAPID-AUTO* (Rigaku Corporation, 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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