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{2,6-Dinitro-6-[(2-oxidophenyl)iminomethyl]-phenolato}(ethanol)(ethanolato)oxidovanadium(V) at 100 K

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The central V atom of the title complex, $[V(C_{13}H_7N_3O_6)(C_2H_5O)O(C_2H_6O)]$, is positioned within a distorted octahedral coordination polyhedron formed by five O-donor atoms and one imine N atom. An axially bound ethanol solvent molecule forms an intermolecular hydrogen bond with the iminophenolate O atom of a neighboring complex, linking molecules into centrosymmetric dimers.

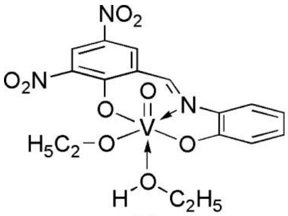
Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.006 Å
R factor = 0.055
wR factor = 0.160
Data-to-parameter ratio = 12.7

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

Comment

The title compound, (I), was prepared in order to study its reactivity toward dibasic tridentate Schiff base auxiliaries in ligand exchange reactions (Hartung *et al.*, 2007). It was crystallized from EtOH–pentane and investigated by X-ray diffraction at 100 K, in order to verify its structure.



(I)

In (I) (Fig. 1), the donor atoms form a distorted octahedral coordination polyhedron around the central V atom, which is displaced by 0.292 (2) Å toward O1, from the mean plane of atoms O2–O4/N1. A separation of 1.590 (3) Å indicates a double bond between V1 and O1. Longer distances are interpreted as single bonds connecting phenoxide O atoms O2 and O3 and alkoxide O atom O4 to V1 (Table 1; Rehder, 1999, Hartung *et al.*, 2004). The shortest of the V—O single bonds refers to alkoxide binding *trans* to the V1—N1 bond, which is a common structural motif in Schiff base-derived vanadium(V) complexes of the investigated type (Clague *et al.*, 1993, Bashirpoor *et al.*, 1997; Hartung *et al.*, 2004). The magnitude of the V1—N1 bond indicates a low affinitiy between the imine N atom and the central atom in neutral oxovanadium(V) complexes.

The H atom attached to O5 was located in a difference Fourier map restricting the data set to $2\theta < 40°$. Refinement of the structure, using all data with the O—H distance fixed, provides an O5—H5O···H2*A* hydrogen bond that links pairs of molecules into centrosymmetric dimers. The chelate ligand in (I) is virtually planar, except for the nitro group connected to C9. This entity is tilted from the plane of the aryl substituent, presumably for reasons of steric and polar repulsion between atom O3 and atoms O8 and O9.

Experimental

To a solution of 2-{[(2-hydroxy-3,5-dinitrophenyl)imino]methyl}phenol (303 mg, 1.00 mmol) in anhydrous EtOH (25 ml) was added $VO(OEt)_3$ (202 mg, 1.00 mmol) at 298 K. Stirring was continued for 24 h at 298 K. The solution was then concentrated under reduced pressure. The remaining residue was crystallized from EtOH–pentane (2:1 *v*/*v*) to afford crystals of (I) (m.p. > 623 K) suitable for X-ray diffraction. Analysis calculated for $C_{17}H_{18}N_3O_9V$: C 44.46, H 3.95, N 9.15%; found: C 43.88, H 3.76, N 9.15%; IR (KBr): 962 cm^{-1} (V═O).

Crystal data

$[V(C_{13}H_7N_3O_6)(C_2H_5O)O(C_2H_6O)]$
$M_r = 459.28$
Monoclinic, $C2/c$
$a = 21.109$ (2) Å
$b = 8.271$ (1) Å
$c = 23.249$ (3) Å
$\beta = 106.38$ (1)°
$V = 3894.4$ (8) Å^3
$Z = 8$
Mo $K\alpha$ radiation
$\mu = 0.57$ mm^{-1}
$T = 100$ (2) K
0.44 × 0.20 × 0.04 mm

Data collection

Oxford Diffraction Xcalibur diffractometer with Sapphire CCD detector
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2006)
$T_{min} = 0.789$, $T_{max} = 0.978$
10522 measured reflections
3781 independent reflections
2446 reflections with $I > 2\sigma(I)$
$R_{int} = 0.040$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.055$
$wR(F^2) = 0.160$
$S = 1.05$
3781 reflections
297 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max} = 1.12$ e Å^{-3}
$\Delta\rho_{min} = -0.56$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—V1	2.161 (3)	O3—V1	1.914 (3)
O1—V1	1.590 (3)	O4—V1	1.755 (3)
O2—V1	1.915 (3)	O5—V1	2.293 (3)
O1—V1—O4	102.36 (14)	O3—V1—N1	84.08 (12)
O1—V1—O3	97.34 (14)	O2—V1—N1	77.97 (12)
O4—V1—O3	99.10 (12)	O1—V1—O5	175.18 (13)
O1—V1—O2	99.18 (13)	O4—V1—O5	82.46 (11)
O4—V1—O2	93.41 (12)	O3—V1—O5	81.88 (11)
O3—V1—O2	156.62 (12)	O2—V1—O5	80.27 (11)
O1—V1—N1	95.52 (14)	N1—V1—O5	79.68 (11)
O4—V1—N1	161.23 (13)		
C13—C7—N1—C6	177.8 (4)	C10—C9—N3—O8	34.0 (6)
C10—C11—N2—O7	−8.5 (6)		

Table 2
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O5—H5*A*···O2^{i}	0.810 (10)	1.987 (14)	2.784 (4)	168 (4)

Symmetry code: (i) $-x+1, -y+1, -z$.

All H atoms, except for those attached to C14, C15, C16 and C17, were located in a difference Fourier map and refined giving C—H distances in the range 0.78 (4)–1.12 (4) Å. Their displacement parameters were constrained with U_{iso}(H) values set to 1.2U_{eq} of the parent atom. Atom H5*A* was refined with the O5—H distance restraint of 0.8 Å. H atoms attached to C14, C15, C16 and C17 were positioned geometrically and treated as riding atoms, with C—H distances in the range of 0.98–0.99 Å and with U_{iso}(H) set to 1.2U_{eq} of the parent atom. The highest peak in the residual electron density map is located 1.56 Å from O1.

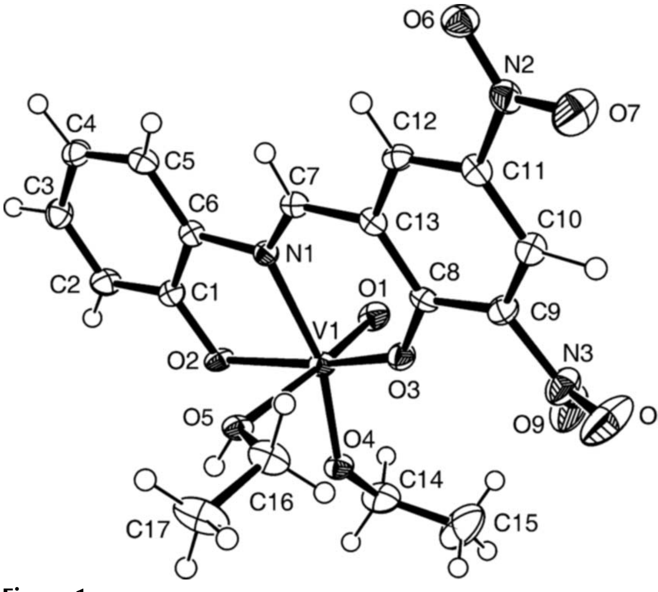


Figure 1
Molecular structure of (I). Displacement ellipsoids are plotted at the 50% probability level.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2006); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2006); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek 2003) and *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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