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Aqua(oxalato-κ^2O,O')oxo(1,10-phenanthroline-κ^2N,N')vanadium(IV)

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Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.004 Å
R factor = 0.043
wR factor = 0.125
Data-to-parameter ratio = 14.4

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

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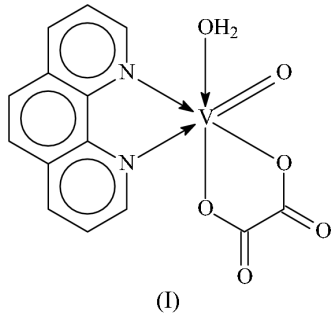
In the title complex, $[V(C_2O_4)O(C_{12}H_8N_2)(H_2O)]$, a 1,10-phenanthroline molecule and an oxalate dianion chelate to the V^{IV} atom, the distorted octahedral coordination of which is completed by a water molecule and an oxo O atom. The complex molecules are linked across a centre of inversion to form a hydrogen-bonded dimer, and these dimers are linked by another hydrogen bond into a linear chain.

Comment

This paper describes our continuing studies of vanadium–phenanthroline complexes, which result from the solvothermal reaction of vanadium(V) pentoxide with acids in the presence of 1,10-phenanthroline (Fu *et al.*, 2004). The oxalate dianion has been used as a chelating entity in a plethora of metal complexes. Curiously, however, the structural literature on vanadium oxalates lists only one example of a vanadium oxalate adduct with an α,α'-diimine, as noted from a search of the Cambridge Structural Database (Version 5.25; Allen, 2002), namely the mixed-valence V^{IV}–V^{V} anion, $\{[(C_{10}H_8N_2)(C_2O_4)VO]_2O\}^-$, the V atoms of which exist in octahedral geometries (Costisor *et al.*, 2001).

In the present study, the hydrothermal reaction of vanadium(V) pentoxide and oxalic acid probably gives vanadyl oxalate, $(C_2O_4)V^{IV}O$, which was isolated as the title aqua-coordinated phenanthroline complex, (I) (Fig. 1).



(I)

Two molecules of the complex are linked into a centrosymmetric hydrogen-bonded dimer, and adjacent dimers are connected into a linear chain through a second O—H···O hydrogen bond (Table 2, Fig. 2). The V—N bond *trans* to the V=O vanadyl grouping is significantly longer than the other V—N bond, by some 0.2 Å.

Experimental

Vanadium pentoxide (0.091 g, 0.5 mmol), oxalic acid (0.190 g, 1.5 mmol), 1,10-phenanthroline (0.10 g, 0.5 mmol) and water (7 ml)

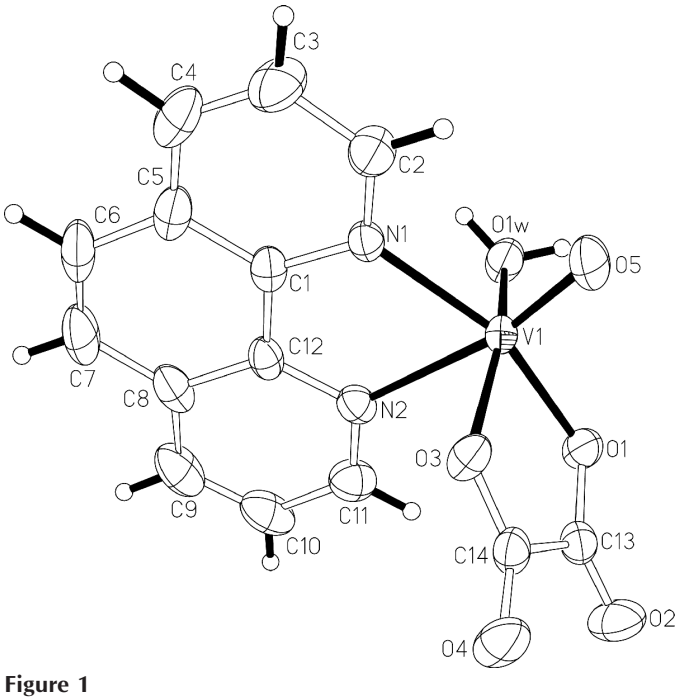


Figure 1
A view of the molecule of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

were placed in a Teflon-lined stainless steel bomb. The bomb was heated at 443 K for 6 d. Green crystals of (I) were obtained on slow cooling of the bomb. CHN elemental analysis, found: C 47.58, H 2.90, N 7.88%; calculated for $C_{14}H_{10}N_2O_6V$: C 47.61, H 2.85, N 7.93%.

Crystal data

$[V(C_2O_4)O(C_{12}H_8N_2)(H_2O)]$	$Z = 2$
$M_r = 353.18$	$D_x = 1.682$ Mg m^{-3}
Triclinic, $P\overline{1}$	Mo $K\alpha$ radiation
$a = 7.6328$ (7) Å	Cell parameters from 1906 reflections
$b = 9.7800$ (9) Å	$\theta = 2.8$–$24.3°$
$c = 9.8622$ (9) Å	$\mu = 0.75$ mm^{-1}
$\alpha = 89.351$ (1)°	$T = 295$ (2) K
$\beta = 73.954$ (1)°	Prism, green
$\gamma = 80.565$ (1)°	0.14 × 0.10 × 0.08 mm
$V = 697.5$ (1) Å^3	

Data collection

Bruker APEX CCD area-detector diffractometer	3105 independent reflections
	2406 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.034$
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)	$\theta_{max} = 27.5°$
	$h = -9 \rightarrow 9$
$T_{min} = 0.804$, $T_{max} = 0.943$	$k = -12 \rightarrow 12$
7736 measured reflections	$l = -12 \rightarrow 12$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0717P)^2 + 0.0065P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.043$	
$wR(F^2) = 0.125$	$(\Delta/\sigma)_{max} = 0.001$
$S = 1.05$	$\Delta\rho_{max} = 0.39$ e Å^{-3}
3105 reflections	$\Delta\rho_{min} = -0.26$ e Å^{-3}
216 parameters	
H atoms treated by a mixture of independent and constrained refinement	

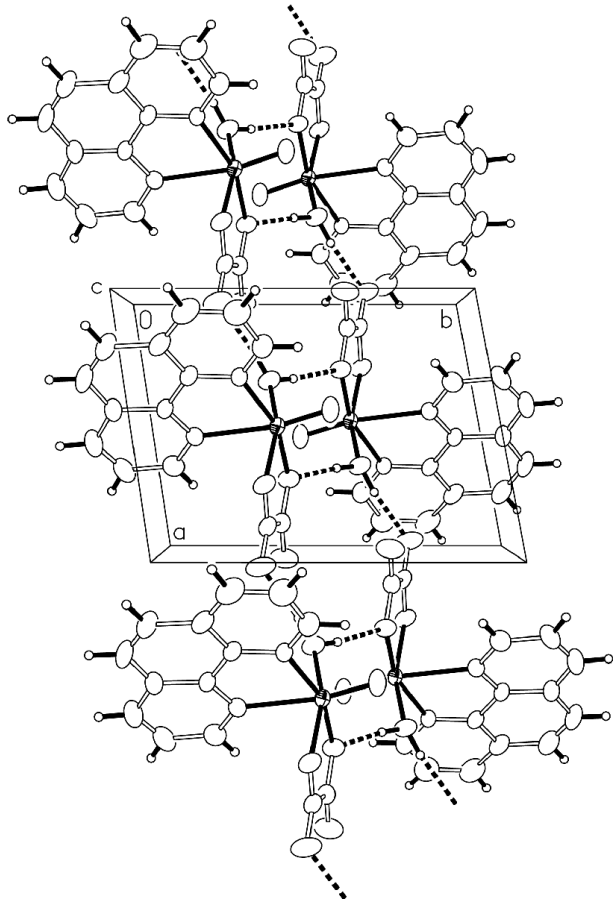


Figure 2
A view of the hydrogen-bonded ribbon structure of (I). Hydrogen bonds are indicated by dashed lines.

Table 1
Selected geometric parameters (Å, °).

V1—O1	1.990 (2)	V1—O1*W*	2.036 (2)
V1—O3	2.001 (2)	V1—N1	2.120 (2)
V1—O5	1.586 (2)	V1—N2	2.319 (2)
O1—V1—O3	80.8 (1)	O3—V1—N2	80.9 (1)
O1—V1—O5	105.3 (1)	O5—V1—O1*W*	98.5 (1)
O1—V1—O1*W*	88.7 (1)	O5—V1—N1	94.0 (1)
O1—V1—N1	160.4 (1)	O5—V1—N2	167.5 (1)
O1—V1—N2	87.2 (1)	O1*W*—V1—N1	91.6 (1)
O3—V1—O5	101.8 (1)	O1*W*—V1—N2	80.4 (1)
O3—V1—O1*W*	158.9 (1)	N1—V1—N2	73.6 (1)
O3—V1—N1	92.4 (1)		

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*W*—H1w1···O1^{i}	0.84 (1)	1.86 (1)	2.697 (3)	173 (3)
O1*W*—H1w2···O4^{ii}	0.86 (1)	1.85 (1)	2.671 (3)	160 (3)

Symmetry codes: (i) $1-x, 1-y, 1-z$; (ii) $x-1, y, z$.

The C-bound H atoms were placed in calculated positions (C—H = 0.93 Å) and were included in the refinement in the riding-model approximation, with $U_{iso}(H) = 1.2U_{eq}(C)$. The water H atoms were located in a difference map and refined with distance restraints of O—H = 0.85 (1) and H···H = 1.39 (1) Å.

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; 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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