

Aquaoxobis(quinoline-2-carboxylato-*N,O*)-vanadium(IV) ethanol hemisolvate

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Key indicators

Single-crystal X-ray study

 $T = 296$ KMean $\sigma(\text{C}-\text{C}) = 0.013$ Å R factor = 0.067 wR factor = 0.245

Data-to-parameter ratio = 15.9

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

The title compound, $[\text{VO}(\text{C}_{10}\text{H}_6\text{NO}_2)_2(\text{H}_2\text{O})]_2 \cdot \text{C}_2\text{H}_5\text{OH}$, contains six-coordinate V^{IV} atoms. The central V^{IV} atom in each of the two crystallographically independent molecules has a distorted octahedral coordination geometry, involving O atoms of the oxo and aqua ligands, together with two O and two N atoms of two organic ligands.

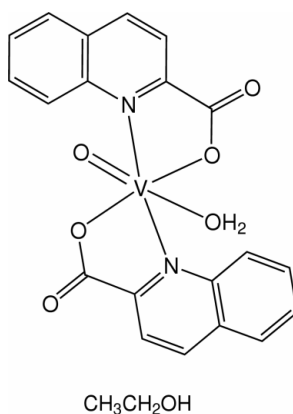
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Comment

Quinoline-2-carboxylic acid, a tryptophan metabolite, forms complexes with various transition metals (Martell & Smith, 1974). Crystal structures have been determined for the Cu^{II} (Haendler, 1986), Mn^{II} (Haendler, 1996; Okabe & Koizumi, 1997), Fe^{II} (Okabe & Makino, 1998), Co^{II} (Okabe & Makino, 1999) and Ni^{II} complexes (Odoko *et al.*, 2001). Vanadate ions have an insulin-mimetic effect in living animals (Bhattacharyya & Tracey, 2001) and in intact cell systems (Kanamori *et al.*, 2001; Sasagawa *et al.*, 2002). Therefore, we aimed to prepare complexes of quinoline-2-carboxylic acid and vanadium and determine their structures. We have characterized the title oxovanadium(IV) complex, (I).



(I)

The molecular structure of (I) is shown in Fig. 1. There are two independent complex molecules in the asymmetric unit, with similar structures. In the complex, two organic ligand molecules and one water molecule coordinate to the oxovanadium(IV) centre. The central V^{IV} atom has a distorted octahedral coordination geometry, involving the O atoms of oxo and aqua ligands, together with two O and two N atoms of the quinoline-2-carboxylate ligands. The carboxyl groups of the ligands are ionized and are essentially coplanar with the quinoline ring planes, as shown by relevant torsion angles [$\text{O}2\text{A}-\text{C}11\text{A}-\text{C}2\text{A}-\text{N}1\text{A}$ 2.4 (9°), $\text{O}2\text{B}-\text{C}11\text{B}-\text{C}2\text{B}-$

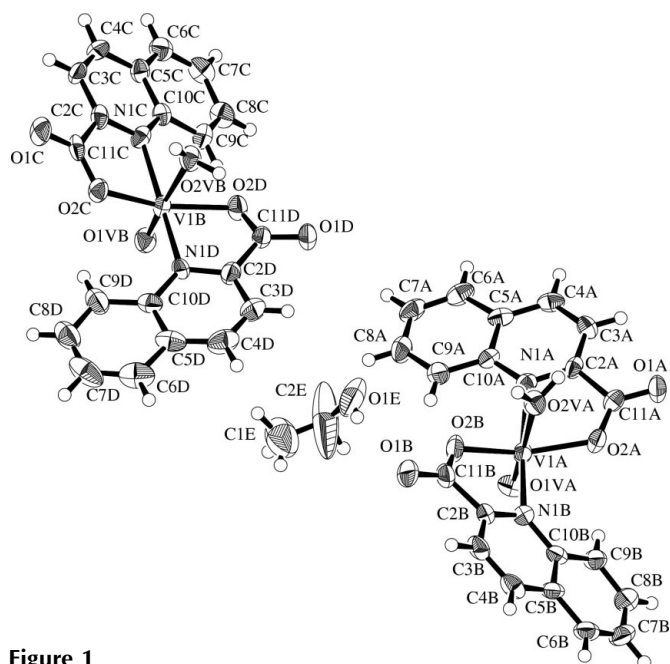


Figure 1
ORTEPII (Johnson, 1976) drawing of the asymmetric unit of the title compound, with the atomic numbering scheme. Ellipsoids correspond to 50% probability.

$N1B - 1 (1)^\circ$, $O2C - C11C - C2C - N1C - 5.0 (9)^\circ$ and $O2D - C11D - C2D - N1D - 2 (1)^\circ$. The quinoline-2-carboxylate ligands and the central V^{IV} form five-membered chelate rings. Such coplanarity and five-membered ring formation are usually present in metal compounds of quinoline-2-carboxylic acid (Okabe & Makino, 1999). Planar five-membered rings with similar bond lengths and angles are also found in a vanadium(III) complex with picolinic acid (pyridine-2-carboxylic acid), $[V^{III}(\text{pic})_3] \cdot H_2O$ (Chatterjee *et al.*, 1997), which has a similar coordination geometry.

Experimental

The green prism crystal of (I) used for X-ray analysis was obtained by slow evaporation from a 4:1 mixture of quinoline-2-carboxylic acid dissolved in ethanol and $VOSO_4$ dissolved in a small amount of water.

Crystal data

$[VO(C_{10}H_6NO_2)_2(H_2O)]_2 \cdot C_2H_5OH$	$Z = 2$
$M_r = 904.63$	$D_x = 1.577 \text{ Mg m}^{-3}$
Triclinic, $P\bar{1}$	Mo $K\alpha$ radiation
$a = 11.745 (3) \text{ \AA}$	Cell parameters from 25 reflections
$b = 16.727 (6) \text{ \AA}$	$\theta = 10.5\text{--}13.0^\circ$
$c = 10.425 (2) \text{ \AA}$	$\mu = 0.57 \text{ mm}^{-1}$
$\alpha = 106.94 (2)^\circ$	$T = 296.2 \text{ K}$
$\beta = 93.58 (2)^\circ$	Prism, green
$\gamma = 101.33 (3)^\circ$	$0.10 \times 0.10 \times 0.05 \text{ mm}$
$V = 1905.3 (10) \text{ \AA}^3$	

Data collection

Rigaku AFC-5R diffractometer	$R_{int} = 0.089$
ω - 2θ scans	$\theta_{max} = 27.5^\circ$
Absorption correction: ψ scan (North <i>et al.</i> , 1968)	$h = 0 \rightarrow 15$
$T_{min} = 0.945$, $T_{max} = 0.972$	$k = -21 \rightarrow 21$
9184 measured reflections	$l = -13 \rightarrow 13$
8754 independent reflections	3 standard reflections
2975 reflections with $I > 2\sigma(I)$	every 150 reflections
	intensity decay: 0.3%

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.067$
 $wR(F^2) = 0.245$
 $S = 1.09$
 8754 reflections
 550 parameters

H-atom parameters not refined
 $w = 1/[\sigma^2(F_o^2) + (0.081P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} = 0.001$
 $\Delta\rho_{max} = 0.87 \text{ e \AA}^{-3}$
 $\Delta\rho_{min} = -0.91 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters (\AA , $^\circ$).

$V1A - O1VA$	1.599 (6)	$V1B - O1VB$	1.602 (5)
$V1A - O2A$	2.005 (5)	$V1B - O2C$	1.982 (6)
$V1A - O2B$	1.990 (6)	$V1B - O2D$	2.020 (5)
$V1A - O2VA$	2.294 (5)	$V1B - O2VB$	2.251 (5)
$V1A - N1A$	2.191 (7)	$V1B - N1C$	2.155 (7)
$V1A - N1B$	2.145 (7)	$V1B - N1D$	2.139 (6)
$O1VA - V1A - O2A$	103.6 (3)	$O2C - V1B - O2VB$	79.2 (2)
$O1VA - V1A - O2B$	102.3 (3)	$O2C - V1B - N1C$	78.7 (2)
$O1VA - V1A - O2VA$	177.2 (2)	$O2C - V1B - N1D$	95.5 (2)
$O1VA - V1A - N1A$	93.4 (3)	$O2D - V1B - O2VB$	76.0 (2)
$O1VA - V1A - N1B$	94.9 (3)	$O2D - V1B - N1C$	103.5 (2)
$O2A - V1A - O2B$	154.0 (2)	$O2D - V1B - N1D$	78.9 (2)
$O2A - V1A - O2VA$	78.1 (2)	$O2VB - V1B - N1C$	86.5 (2)
$O2A - V1A - N1A$	78.2 (2)	$O2VB - V1B - N1D$	85.8 (2)
$O2A - V1A - N1B$	97.7 (2)	$N1C - V1B - N1D$	171.1 (2)
$O2B - V1A - O2VA$	76.0 (2)	$V1A - O2A - C11A$	119.4 (4)
$O2B - V1A - N1A$	102.2 (3)	$V1A - O2B - C11B$	118.8 (5)
$O2B - V1A - N1B$	78.1 (3)	$V1B - O2C - C11C$	119.2 (5)
$O2VA - V1A - N1A$	84.9 (2)	$V1B - O2D - C11D$	116.5 (5)
$O2VA - V1A - N1B$	86.9 (2)	$V1A - N1A - C2A$	112.2 (5)
$N1A - V1A - N1B$	171.4 (2)	$V1A - N1A - C10A$	128.5 (6)
$O1VB - V1B - O2C$	104.4 (3)	$V1A - N1B - C2B$	111.9 (5)
$O1VB - V1B - O2D$	100.4 (3)	$V1A - N1B - C10B$	130.0 (5)
$O1VB - V1B - O2VB$	176.4 (3)	$V1B - N1C - C2C$	112.1 (5)
$O1VB - V1B - N1C$	94.0 (3)	$V1B - N1C - C10C$	128.7 (5)
$O1VB - V1B - N1D$	93.9 (3)	$V1B - N1D - C2D$	112.2 (6)
$O2C - V1B - O2D$	154.9 (2)	$V1B - N1D - C10D$	129.2 (5)

H atoms, located from a difference map, were included in idealized positions (except for those on H_2O) and then fixed.

Data collection: *MSC/AFCC Diffractometer Control Software* (Molecular Structure Corporation & Rigaku Corporation, 1999); cell refinement: *MSC/AFCC Diffractometer Control Software*; data reduction: *TEXSAN* (Molecular Structure Corporation & Rigaku Corporation, 1999); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999) and *DIRDIF94* (Beurskens *et al.*, 1992); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *TEXSAN*.

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