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Aqua(ethylenediamine-*N*,*N*′-diacetato-*O*,*O*′)oxovanadium(IV)

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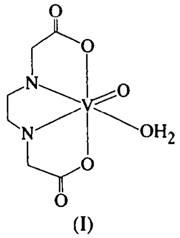
Abstract

In the title compound, $[VO(C_6H_{10}N_2O_4)(H_2O)]$, the V atom is situated in a distorted octahedral geometry. The ethylenediamine-*N*,*N*′-diacetate (edda) ligand coordinates to the vanadium center in the symmetric α-*cis* fashion, which differs from the β-*cis* coordination mode in $(NH_4)[VO_2(edda)]$ in the solid state. The distance from the vanadium center to the N atom *trans* to the oxo ligand is significantly elongated as a result of the strong *trans* influence of the oxo ligand.

Comment

Ethylenediamine-*N*,*N*′-diacetate (edda) can coordinate to a metal center by either an α-*cis* or β-*cis* coordination mode (α and β denote two O atoms in *trans* and *cis* positions in edda, respectively; *cis* refers to two remaining coordination sites). The preparative studies of cobalt(III) complexes with edda and its *N*-alkyl-substituted analogs have shown that the α-*cis* isomer is more stable than the β-*cis* one (Legg & Cooke, 1965; Gailey, Igi & Douglas, 1975). However, both isomers of the dioxovanadium(V) complex with edda, $[VO_2(edda)]^-$, exist in solution in nearly equal concentrations (Amos & Sawyer, 1972). Very recently, $(NH_4)[VO_2(edda)]$ was reported to adopt the β-*cis* configuration in the solid state (Crans, Keramidas, Mahroof-Tahir, Anderson & Miller, 1996). It is therefore of interest to determine the structure of the oxovanadium(IV) complex with edda, (I), in which the oxo ligand and the water molecule occupy *cis* positions, similar to the two oxo ligands in the dioxovanadium(V) complex.



(I)

The V atom is situated in a distorted octahedral geometry (Fig. 1). The two carboxylato O atoms (O1 and O3), the N1 atom of the edda ligand and the coordinated water molecule (O5) form an equatorial plane. The oxo ligand (O6) and the N2 atom of edda occupy the axial positions. Thus, the edda ligand coordinates to the vanadium center in the more stable α-*cis* coordination mode, which is distinct from the β-*cis* coordination in $(NH_4)[VO_2(edda)]$.

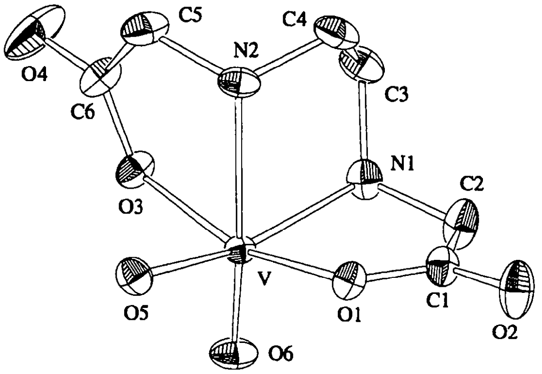


Fig. 1. Perspective view of the title anion with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms have been omitted for clarity.

The V—O6 distance of 1.599 (2) Å implies double-bond character (Table 1). The distance from the vanadium center to the N atom *trans* to the oxo ligand (V—N2) is significantly longer than that to the N atom in the *cis* position (V—N1), reflecting the strong *trans* influence of the oxo ligand [2.328 (2) *versus* 2.104 (2) Å].

Strong intermolecular hydrogen bonding [2.608 (3) Å] occurs between the carbonyl O atom (O2) and the aqua ligand (O5). Weaker hydrogen bonding (3.0–3.5 Å) also occurs between N1 and O1, N1 and O2, O4 and O5, O6 and O1, and O6 and N2.

Experimental

$VOSO_4$ (1.2 g) and H_2edda (0.9 g) were dissolved in 30 ml of water. $BaCO_3$ (1 g) was added to the above solution, CO_2 gas was evolved and $BaSO_4$ precipitated. After the precipitate was filtered off, the filtrate was kept over $CaCl_2$. The blue crystals deposited were collected by filtration.

Crystal data

$[VO(C_6H_{10}N_2O_4)(H_2O)]$
$M_r = 259.11$

Mo $K\alpha$ radiation
$\lambda = 0.7107$ Å

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Monoclinic
Pc
$a = 8.307\ (2)$ Å
$b = 6.7435\ (9)$ Å
$c = 10.102\ (2)$ Å
$\beta = 118.576\ (6)^\circ$
$V = 496.9\ (1)$ Å^3
$Z = 2$
$D_x = 1.732$ Mg m^{-3}
D_m not measured

Cell parameters from 25 reflections
$\theta = 8–10^\circ$
$\mu = 1.010$ mm^{-1}
$T = 296$ K
Plate
0.38 × 0.30 × 0.20 mm
Blue

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scans (North, Phillips & Mathews, 1968)
$T_{\min} = 0.764$, $T_{\max} = 0.817$
1582 measured reflections
1498 independent reflections

1380 reflections with $I > 1.5\sigma(I)$
$R_{\rm int} = 0.021$
$\theta_{\max} = 30^\circ$
$h = 0 \rightarrow 9$
$k = 0 \rightarrow 9$
$l = -14 \rightarrow 12$
3 standard reflections every 120 reflections intensity decay: −2.60%

Refinement

Refinement on F
$R = 0.031$
$wR = 0.045$
$S = 1.417$
1380 reflections
134 parameters
H atoms not refined
$w = 1/[\sigma^2(F_o) + 0.0009|F_o|^2]$

$(\Delta/\sigma)_{\max} = 0.0030$
$\Delta\rho_{\max} = 0.29$ e Å^{-3}
$\Delta\rho_{\min} = -0.68$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

V—O1	1.986 (2)	V—O6	1.599 (2)
V—O3	2.011 (2)	V—N1	2.104 (2)
V—O5	2.053 (2)	V—N2	2.328 (2)
O1—V—O3	161.10 (9)	O5—V—O6	102.2 (1)
O1—V—O5	87.02 (8)	O5—V—N1	157.93 (9)
O1—V—O6	103.3 (1)	O5—V—N2	82.40 (8)
O1—V—N1	81.48 (8)	O6—V—N1	98.7 (1)
O3—V—O5	92.28 (9)	O6—V—N2	170.15 (10)
O3—V—O6	95.4 (1)	N1—V—N2	77.99 (8)
O3—V—N2	75.65 (8)		

The H atoms of the aqua ligand were not located.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1995). Program(s) used to solve structure: *TEXSAN*. Program(s) used to refine structure: *TEXSAN*. Molecular graphics: *TEXSAN*. Software used to prepare material for publication: *TEXSAN*.

Lists of atomic coordinates, displacement parameters, structure factors and complete geometry have been deposited with the IUCr (Reference: OA1014). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Sodium (*N*-Ethylethylenediamine-*N*,*N'*,*N'*-triacetato)oxovanadium(IV) Trihydrate

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Abstract

The V atom in the title compound, Na[VO($C_{10}H_{15}N_2O_6$)].3H_2O, adopts a distorted octahedral structure. The strong *trans* influence of the vanadyl oxo atom [V—O 1.606 (1) Å] is obvious in the V—N distances, *i.e.* 2.301 (2) Å for the position *trans* to the oxo ligand and 2.142 (2) Å for the position *cis*.

Comment

The importance of vanadium compounds to the physiological functions of living organisms is becoming increasingly recognized (Chasteen, 1990; Sigel & Sigel, 1995). Vanadium(IV) complexes particularly have attracted the attention of pharmacologists as well as chemists with regard to their insulin mimetic functions (Orvig, Thompson, Battell & McNeill, 1995). In order to establish the structure–function relationship, it is important to obtain detailed structures of vanadium(IV) complexes. Although many of the stability constants for oxovanadium(IV) complexes with polyaminocarboxylates have been determined (Felcman & Fraústo da Silva, 1983), only a few X-ray analyses are available. We report here the crystal structure of the oxovanadium(IV) complex, (I), with the pentadentate eed3a (*N*-ethylethylenediamine-*N*,*N'*,*N'*-triacetate) ligand.

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