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

Cell parameters from 25 reflections
$\theta = 8–10°$
$\mu = 1.010$ 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 $I > 1.5\sigma(I)$
$R_{\rm int} = 0.021$
$\theta_{\max} = 30°$
$h = 0 \rightarrow 9$
$k = 0 \rightarrow 9$
$l = -14 \rightarrow 12$
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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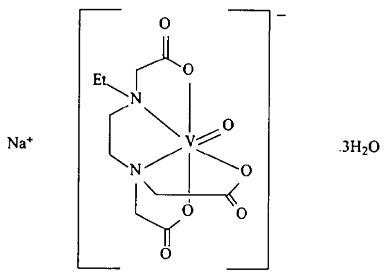
Abstract

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.



(I)

The V atom adopts a distorted octahedral structure (Fig. 1). Three carboxylato O atoms (O1, O3 and O5) together with the amino N atom (N2) of the eed3a ligand form the equatorial plane. The axial positions are occupied by the oxo ligand (O7) and the other amino N atom (N1) of eed3a. This coordination mode is similar to that found in the half unit of $[(VO)_2(ttha)]^{2-}$ (ttha is triethylenetetraamine-*N*,*N*,*N′*,*N″*,*N‴*,*N‴*-hexaacetate) (Fallon & Gatehouse, 1976).

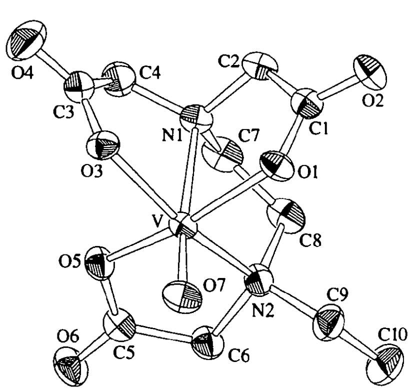


Fig. 1. Perspective view of the title anion with the atom-numbering scheme. The displacement ellipsoids are shown at the 50% probability level and H atoms have been omitted for clarity.

The V—O7 distance is 1.606 (1) Å and lies within the range expected for vanadyl groups of oxovanadium(IV) complexes. The distance from the V-atom center to the N atom *trans* to the oxo ligand [V—N1 2.301 (2) Å] is significantly longer than that to the N atom in the *cis* position [V—N2 2.142 (2) Å], reflecting the strong *trans* influence of the oxo ligand. The V atom is displaced towards the oxo ligand by 0.37 (7) Å from the best least-squares equatorial plane. As a result, the O1—V—O5 [161.54 (6)°] and O3—V—N2 [154.00 (6)°] angles become significantly smaller than the 180° expected for the idealized octahedral complex, but larger than those found in square-pyramidal oxovanadium(IV) complexes (140–145°; Borovik, Dewey & Raymond, 1993). The two bite angles of the O—N chelate rings [O1—V—N1 76.52 (5) and O3—V—N1 75.61 (6)°] are smaller than the other bite angle [O5—V—N2 80.70 (6)°]. These small bite angles are a consequence of the elongation of the V—N1 bond.

Experimental

Crystals of the title compound were prepared according to the literature (Kanamori *et al.*, 1994).

Crystal data

$Na[VO(C_{10}H_{15}N_2O_6)].3H_2O$
$M_r = 403.21$
Monoclinic
$P2_1/a$
$a = 17.329 (5)$ Å
$b = 13.631 (3)$ Å
$c = 6.943 (2)$ Å
$\beta = 101.40 (1)°$
$V = 1607.6 (6)$ Å^3
$Z = 4$
$D_x = 1.666$ Mg m^{-3}
D_m not measured
Mo $K\alpha$ radiation
$\lambda = 0.7107$ Å
Cell parameters from 25 reflections
$\theta = 21–25°$
$\mu = 0.698$ mm^{-1}
$T = 296$ K
Column
0.45 × 0.40 × 0.18 mm
Blue

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (North, Phillips & Mathews, 1968)
$T_{min} = 0.816$, $T_{max} = 0.882$
10 101 measured reflections
6025 independent reflections
4120 reflections with $I > 1.5\sigma(I)$
$R_{int} = 1.93$
$\theta_{max} = 30°$
$h = 0 \rightarrow 24$
$k = -19 \rightarrow 19$
$l = -9 \rightarrow 9$
3 standard reflections every 120 reflections intensity decay: 0.75%

Refinement

Refinement on F
$R = 0.0381$
$wR = 0.0617$
$S = 1.398$
4120 reflections
217 parameters
H-atom parameters not refined and water H atoms not located
$w = 1/[\sigma^2(F_o) + 0.00063|F_o|^2]$
$(\Delta/\sigma)_{max} = 0.0010$
$\Delta\rho_{max} = 0.48$ e Å^{-3}
$\Delta\rho_{min} = -0.28$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

V—O1	2.009 (1)	V—O7	1.606 (1)
V—O3	1.992 (1)	V—N1	2.301 (2)
V—O5	1.989 (1)	V—N2	2.142 (2)
O1—V—O5	161.54 (6)	O5—V—O7	101.40 (7)
O1—V—O7	96.09 (7)	O5—V—N2	80.70 (6)
O1—V—N1	76.52 (5)	O7—V—N1	172.61 (7)
O3—V—O7	104.88 (7)	O7—V—N2	100.15 (7)
O3—V—N1	75.61 (6)	N1—V—N2	80.48 (6)
O3—V—N2	154.00 (6)		

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: OA1015). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Clemizole Tetrachlorocuprate(II) and Clemizole Tetrachlorocobaltate(II)

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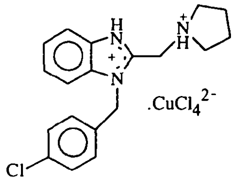
Abstract

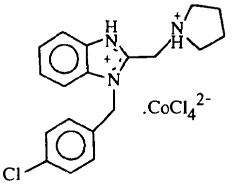
The crystal structures of 3-[4-(chlorophenyl)methyl]-2-(1-pyrrolidiniomethyl)-1*H*-benzimidazolium tetrachlorocopper(II), $(C_{19}H_{22}ClN_3)[CuCl_4]$, and 3-[4-(chlorophenyl)methyl]-2-(1-pyrrolidiniomethyl)-1*H*-benzimidazolium tetrachlorocobalt(II), $(C_{19}H_{22}ClN_3)[CoCl_4]$, show different conformations of the free radical dihydro cations of clemizole, a potent anti-allergic agent. The anions, $CuCl_4^{2-}$ and $CoCl_4^{2-}$, exhibit flattened and normal tetrahedral geometries, respectively.

Comment

Our continued interest in the conformations of antihistamines effective on H_1-receptors has led to the preparation of free radical dihydro cations of clemizole in order to investigate the influence of tetrachlorocopper(II) and tetrachlorocobalt(II) ions on the conformation of clemizole. In this paper, we report the crystal structures of clemizole tetrachlorocopper(II), (1), and clemizole tetrachlorocobalt(II), (2); the crystal structures of clemizole in its free form, (3) (Parvez & Sabir, 1996*a*), clemizole hydrochloride, (4) (Parvez, 1996), and a transition metal complex of clemizole, clemizoledichlorozinc(II), (5) (Parvez & Sabir, 1996*b*), have been reported recently from this laboratory.



(1)



(2)

The *ORTEP*II (Johnson, 1976) drawings of (1) and (2) are shown in Figs. 1 and 2, respectively. The molecular dimensions of the dihydro cations in (1) and (2) are normal, with identical corresponding bond distances and angles. The C1—N1 and C1—N2 distances in the two cations are equivalent, indicating electron delocalization in the imidazole rings. The remaining bond distances and angles are not very different from those observed in compounds (3)–(5).

The benzimidazole and phenyl rings are individually planar in the two cations and are inclined at angles of 70.5 (6) and 82.7 (9)° in (1) and (2), respectively; the corresponding angles in the crystal structures of (3)–(5) are 68.8 (6), 82.8 (9) and 81.1 (4)°, respectively.

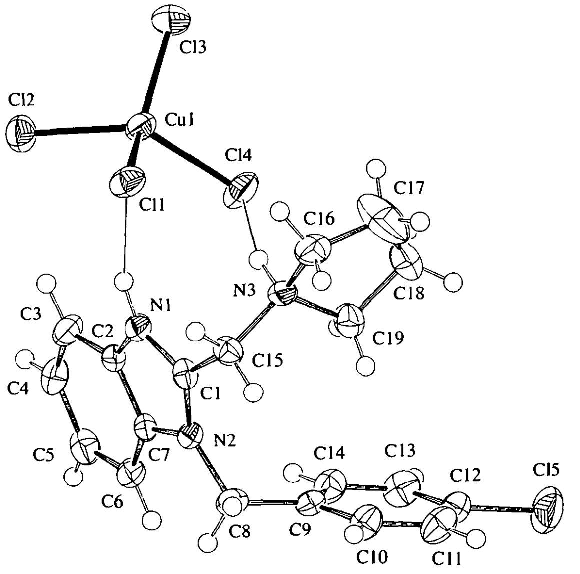


Fig. 1. *ORTEP*II (Johnson, 1976) drawing of (1) with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms have been assigned arbitrary radii.

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