The structure was solved by direct methods and was initially refined using *SHELXTL/PC* (Sheldrick, 1990*b*) in order to obtain the analytical-absorption-corrected data generated by the program. These data were then used for the refinement using *SHELXL*93 (Sheldrick, 1993). During the refinement, one of the triethylammonium cations displayed disorder at atoms C7 (C7′) and C11 (C11′). The site occupancy factors of these disordered atoms were refined and found to be close to 0.5 and so were fixed at 0.5 for further refinement as there was a large correlation between the displacement factors and the site occupancy factors in the refinement calculation. The bond lengths in the disordered triethylammonium cation were restrained with reference to the non-disordered triethylammonium cation using the *DFIX* (*SHELXL*93) instruction. All H atoms were generated geometrically and allowed to ride on their respective C atoms, except for those attached to the N and O atoms of the triethylammonium cations and water molecules, respectively, which were located from the difference Fourier maps. All H atoms were refined with fixed $U_{\rm iso} = 0.08$ Å^2.

Data collection: *XSCANS* (Siemens, 1994). Cell refinement: *XSCANS*. Data reduction: *XSCANS*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990*a*). Program(s) used to refine structure: *SHELXL*93. Molecular graphics: *SHELXTL/PC* (Sheldrick, 1990*b*). Software used to prepare material for publication: *SHELXL*93.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AS1175). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1996). C**52**, 509–512

(Acetato-*O*,*O*′)[tris(2-aminoethyl)amine-*N*,*N*′,*N*″,*N*‴]nickel(II) Perchlorate

HOONG-KUN FUN,[a] BOON-CHUAN YIP,[a] ZHONG-LIN LU,[b] CHUN-YING DUAN,[b] YU-PENG TIAN[b] AND XIAO-ZENG YOU[b]

[a]*X-ray Crystallography Laboratory, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia, and* [b]*Coordination Chemistry Institute, State Key Laboratory of Coordination Chemistry, Centre for Advanced Studies in Science and Technology of Microstructures, Nanjing University, Nanjing 210093, People's Republic of China. E-mail: hkfun@cs.usm.my*

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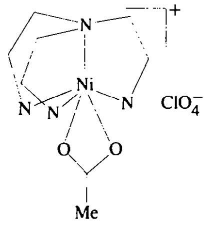
Abstract

The X-ray structure analysis of the title compound, $[\mathrm{Ni(C_2H_3O_2)(C_6H_{18}N_4)}]\mathrm{ClO_4}$, reveals it to consist of a distorted octahedral entity, with the Ni$^{\rm II}$ atom coordinated by four N atoms from a tris(2-aminoethyl)amine (tren) ligand and two O atoms from an acetate anion. The acetate anion coordinates to Ni$^{\rm II}$ as a bidentate ligand forming a four-membered ring, with the two O atoms chelating in different manners, resulting in different C—O and Ni—O bond distances.

Comment

Tripodal complexes of transition metals have been investigated widely since, in addition to their special chemical, physical and structural properties (Gou, You, Yu & Lu, 1993), they may also serve as candidates for magnetic resonance imaging agents (Smith & Raymond, 1985) and as models for unique coordination polyhedra (Fleisher, Gebaba & Tasher, 1970). We report here the crystal structure of the Ni$^{\rm II}$ complex (acetato-*O*,*O*′)[tris(2-aminoethyl)amine-*N*,*N*′,*N*″,*N*‴]nickel(II) perchlorate, (I), which contains a tetradentate tripod (tren) ligand and a bidentate acetate ligand.



(I)

An *ORTEP* plot (Johnson, 1965) of the title compound with the numbering scheme is shown in Fig. 1. The Ni atom is octahedrally coordinated by a tetradentate tren ligand and a bidentate acetate anion ligand. The tren Ni—N bond distances range from 2.066 (3) to 2.094 (4) Å, which agree with values observed for the dimeric cation [(tren)Ni(NCBH$_3$)]$_2^{2+}$ (Segal & Lippard, 1977). There are two different cations in the crystal, identified as *A* and *B*. The difference in the C—O bond distances in *A* (0.001 Å) is smaller than that in *B* (0.042 Å), while the difference in the Ni—O bond distances in *A* (0.142 Å) is larger than that in *B* (0.063 Å). This indicates that the acetate group coordinates to Ni^{II} in two different manners. Analogues of *A* and *B* have been found in diaquabis(phenoxyacetato)zinc(II) (Smith, O'Reilly, Kennard, Stadnicka & Oleksyn, 1981) and diaquabis(phenylthioacetato)zinc(II) (Mak, Yip, Smith, O'Reilly & Kennard, 1984). The acute O—Ni—O bond angle of 61.0 (1)° shows the coordination sphere to be significantly different from regular octahedral. The complex can thus be described as having distorted octahedral geometry.

The two [Ni(tren)(O$_2$CCH$_3$)]$^+$ cations in the asymmetric unit are linked *via* hydrogen bonds as shown in the packing diagram viewed down the *b* axis (Fig. 2). These hydrogen bonds are listed in Table 3.

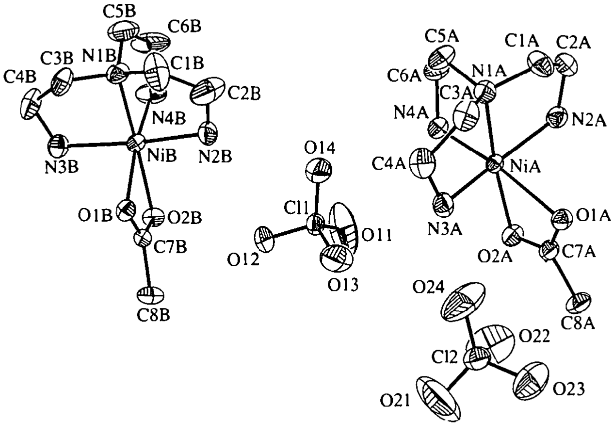


Fig. 1. *ORTEP* plot (Johnson, 1965) of complex (I) showing 30% probability displacement ellipsoids.

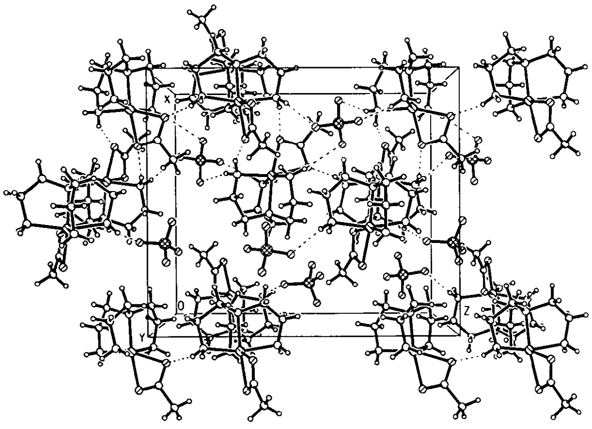


Fig. 2. Packing diagram of (I) viewed down the *b* axis.

Experimental

The title complex was synthesized according to the procedure described by Raymond & Basolo (1966), using Ni(OAc)$_2$.4H$_2$O and NaClO$_4$ instead of CuCl$_2$.2H$_2$O and KSCN. Prismatic purple crystals of the title complex were obtained by slow evaporation of an ethanol solution at room temperature.

Crystal data

[Ni($C_2H_3O_2$)($C_6H_{18}N_4$)]ClO_4
M_r = 363.44
Orthorhombic
$P2_12_12_1$
a = 13.078 (2) Å
b = 14.721 (2) Å
c = 15.4370 (10) Å
V = 2972.0 (6) Å^3
Z = 8
D_x = 1.625 Mg m^{-3}

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 45 reflections
θ = 5–12.5°
μ = 1.515 mm^{-1}
T = 293 (2) K
Prismatic
0.7 × 0.6 × 0.5 mm
Purple

Data collection

Siemens *P*4 four-circle diffractometer
θ–2θ scans
Absorption correction: empirical (*SHELXTL/PC*; Sheldrick, 1990*b*)
T_{min} = 0.653, T_{max} = 0.711
4720 measured reflections
4501 independent reflections

4270 observed reflections [$I > 2\sigma(I)$]
R_{int} = 0.0158
θ_{max} = 27.49°
$h = -1 \rightarrow 16$
$k = -1 \rightarrow 19$
$l = -1 \rightarrow 20$
3 standard reflections monitored every 97 reflections intensity decay: none

Refinement

Refinement on F^2
$R(F)$ = 0.0375
$wR(F^2)$ = 0.1078
S = 1.045
4501 reflections
362 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F_o^2) + (0.0673P)^2 + 1.5525P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.551 e Å^{-3}
$\Delta\rho_{min}$ = −0.448 e Å^{-3}

Extinction correction: *SHELXTL/PC* (Sheldrick, 1990*b*)
Extinction coefficient: 0.0072 (6)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)
Absolute configuration: Flack (1983) parameter = −0.02 (2)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$U_{eq} = (1/3)\Sigma_i\Sigma_jU_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
Ni*A*	0.11239 (4)	0.01904 (3)	0.61183 (3)	0.03396 (13)
N1*A*	−0.0444 (3)	0.0005 (3)	0.6216 (2)	0.0457 (8)
N2*A*	0.1065 (3)	−0.1056 (3)	0.5468 (3)	0.0527 (9)

N3*A*	0.0809 (3)	0.1523 (3)	0.6481 (2)	0.0509 (9)
N4*A*	0.1169 (3)	−0.0398 (2)	0.7333 (2)	0.0426 (7)
C1*A*	−0.0721 (5)	−0.0575 (4)	0.5482 (4)	0.0686 (15)
C2*A*	0.0011 (5)	−0.1340 (5)	0.5351 (4)	0.077 (2)
C3*A*	−0.0875 (4)	0.0945 (4)	0.6150 (4)	0.0613 (13)
C4*A*	−0.0290 (4)	0.1617 (4)	0.6673 (4)	0.0663 (14)
C5*A*	−0.0670 (4)	−0.0396 (5)	0.7071 (4)	0.0638 (14)
C6*A*	0.0207 (4)	−0.0903 (4)	0.7441 (4)	0.0614 (13)
C7*A*	0.2421 (3)	0.0790 (3)	0.5086 (3)	0.0442 (9)
C8*A*	0.3251 (4)	0.1162 (5)	0.4518 (4)	0.069 (2)
O1*A*	0.1502 (2)	0.0777 (2)	0.4826 (2)	0.0453 (7)
O2*A*	0.2648 (2)	0.0478 (2)	0.5826 (2)	0.0496 (7)
Ni*B*	0.09592 (4)	0.14815 (3)	1.22076 (3)	0.03588 (14)
N1*B*	−0.0592 (3)	0.1229 (3)	1.2303 (3)	0.0571 (10)
N2*B*	0.0752 (4)	0.1095 (3)	1.0926 (3)	0.0619 (11)
N3*B*	0.0767 (4)	0.2077 (3)	1.3429 (2)	0.0568 (10)
N4*B*	0.1118 (4)	0.0160 (3)	1.2639 (4)	0.075 (2)
C1*B*	−0.0961 (5)	0.1335 (6)	1.1390 (5)	0.101 (3)
C2*B*	−0.0281 (6)	0.0994 (9)	1.0754 (4)	0.130 (5)
C3*B*	−0.0980 (4)	0.1979 (4)	1.2875 (4)	0.0666 (14)
C4*B*	−0.0316 (5)	0.2084 (6)	1.3650 (4)	0.078 (2)
C5*B*	−0.0723 (6)	0.0314 (5)	1.2656 (7)	0.101 (3)
C6*B*	0.0131 (5)	−0.0259 (5)	1.2621 (8)	0.115 (3)
C7*B*	0.2082 (3)	0.2735 (3)	1.1693 (3)	0.0387 (8)
C8*B*	0.2801 (4)	0.3442 (4)	1.1338 (4)	0.0650 (14)
O1*B*	0.1142 (2)	0.2862 (2)	1.1711 (2)	0.0440 (6)
O2*B*	0.2455 (2)	0.1980 (2)	1.1966 (2)	0.0477 (7)
Cl1	0.18410 (9)	0.17474 (7)	0.87741 (7)	0.0469 (2)
O11	0.2644 (6)	0.1162 (6)	0.8632 (5)	0.176 (4)
O12	0.2012 (4)	0.2202 (3)	0.9567 (3)	0.0719 (11)
O13	0.1797 (5)	0.2395 (4)	0.8112 (3)	0.111 (2)
O14	0.0899 (5)	0.1291 (5)	0.8763 (3)	0.121 (2)
Cl2	0.16277 (11)	0.37571 (10)	0.51916 (9)	0.0625 (3)
O21	0.1974 (14)	0.4453 (6)	0.5583 (7)	0.278 (8)
O22	0.2344 (6)	0.3018 (5)	0.5303 (6)	0.154 (3)
O23	0.1576 (5)	0.3943 (5)	0.4288 (3)	0.110 (2)
O24	0.0743 (4)	0.3369 (7)	0.5472 (5)	0.148 (3)

Table 2. *Selected geometric parameters (Å, °)*

Ni*A*—N4*A*	2.066 (3)	Ni*B*—N1*B*	2.068 (4)
Ni*A*—N1*A*	2.074 (3)	Ni*B*—N2*B*	2.076 (4)
Ni*A*—N3*A*	2.082 (4)	Ni*B*—N3*B*	2.094 (4)
Ni*A*—O2*A*	2.087 (3)	Ni*B*—N4*B*	2.066 (4)
Ni*A*—N2*A*	2.093 (4)	Ni*B*—O1*B*	2.185 (3)
Ni*A*—O1*A*	2.229 (3)	Ni*B*—O2*B*	2.122 (3)
N1*A*—C1*A*	1.464 (7)	N1*B*—C1*B*	1.499 (8)
N1*A*—C5*A*	1.476 (6)	N1*B*—C3*B*	1.501 (7)
N1*A*—C3*A*	1.498 (6)	N1*B*—C5*B*	1.462 (9)
N2*A*—C2*A*	1.452 (7)	N2*B*—C2*B*	1.385 (9)
N3*A*—C4*A*	1.474 (7)	N3*B*—C4*B*	1.457 (8)
N4*A*—C6*A*	1.471 (6)	N4*B*—C6*B*	1.431 (8)
C1*A*—C2*A*	1.493 (9)	C1*B*—C2*B*	1.416 (11)
C3*A*—C4*A*	1.489 (8)	C3*B*—C4*B*	1.486 (9)
C5*A*—C6*A*	1.483 (7)	C5*B*—C6*B*	1.400 (10)
C7*A*—O2*A*	1.266 (5)	C7*B*—O1*B*	1.243 (5)
C7*A*—O1*A*	1.267 (6)	C7*B*—O2*B*	1.285 (5)
C7*A*—C8*A*	1.499 (6)	C7*B*—C8*B*	1.506 (6)
N4*A*—Ni*A*—N1*A*	84.68 (15)	N4*B*—Ni*B*—N1*B*	84.7 (2)
N4*A*—Ni*A*—N3*A*	99.00 (15)	N4*B*—Ni*B*—N2*B*	93.6 (2)
N1*A*—Ni*A*—N3*A*	84.8 (2)	N1*B*—Ni*B*—N2*B*	83.7 (2)
N4*A*—Ni*A*—O2*A*	104.70 (13)	N4*B*—Ni*B*—N3*B*	96.7 (2)
N1*A*—Ni*A*—O2*A*	170.61 (14)	N1*B*—Ni*B*—N3*B*	83.9 (2)
N3*A*—Ni*A*—O2*A*	93.2 (2)	N2*B*—Ni*B*—N3*B*	163.1 (2)
N4*A*—Ni*A*—N2*A*	94.0 (2)	N4*B*—Ni*B*—O2*B*	106.8 (2)
N1*A*—Ni*A*—N2*A*	83.3 (2)	N1*B*—Ni*B*—O2*B*	168.2 (2)
N3*A*—Ni*A*—N2*A*	161.5 (2)	N2*B*—Ni*B*—O2*B*	92.7 (2)
O2*A*—Ni*A*—N2*A*	96.2 (2)	N3*B*—Ni*B*—O2*B*	97.1 (2)
N4*A*—Ni*A*—O1*A*	165.45 (13)	N4*B*—Ni*B*—O1*B*	167.8 (2)
N1*A*—Ni*A*—O1*A*	109.62 (14)	N1*B*—Ni*B*—O1*B*	107.4 (2)
N3*A*—Ni*A*—O1*A*	85.38 (14)	N2*B*—Ni*B*—O1*B*	86.2 (2)
O2*A*—Ni*A*—O1*A*	61.04 (11)	N3*B*—Ni*B*—O1*B*	86.54 (15)
N2*A*—Ni*A*—O1*A*	85.32 (14)	O2*B*—Ni*B*—O1*B*	61.04 (11)
C1*A*—N1*A*—C5*A*	114.2 (4)	C5*B*—N1*B*—C1*B*	114.1 (7)
C1*A*—N1*A*—C3*A*	113.1 (4)	C5*B*—N1*B*—C3*B*	114.8 (5)
C5*A*—N1*A*—C3*A*	110.8 (4)	C1*B*—N1*B*—C3*B*	111.6 (5)
C1*A*—N1*A*—Ni*A*	105.4 (3)	C5*B*—N1*B*—Ni*B*	107.8 (4)
C5*A*—N1*A*—Ni*A*	108.4 (3)	C1*B*—N1*B*—Ni*B*	103.3 (4)
C3*A*—N1*A*—Ni*A*	104.2 (3)	C3*B*—N1*B*—Ni*B*	104.0 (3)
C2*A*—N2*A*—Ni*A*	110.3 (3)	C2*B*—N2*B*—Ni*B*	109.8 (4)
C4*A*—N3*A*—Ni*A*	109.6 (3)	C6*B*—N4*B*—Ni*B*	108.0 (4)
C6*A*—N4*A*—Ni*A*	106.9 (3)	C4*B*—N3*B*—Ni*B*	109.3 (4)
N1*A*—C1*A*—C2*A*	112.7 (5)	C2*B*—C1*B*—N1*B*	114.4 (5)
N2*A*—C2*A*—C1*A*	112.0 (5)	N2*B*—C2*B*—C1*B*	116.2 (7)
C4*A*—C3*A*—N1*A*	112.5 (4)	C6*B*—C5*B*—N1*B*	116.6 (6)
N3*A*—C4*A*—C3*A*	109.3 (4)	C5*B*—C6*B*—N4*B*	117.3 (6)
N1*A*—C5*A*—C6*A*	113.0 (4)	C4*B*—C3*B*—N1*B*	110.6 (5)
N4*A*—C6*A*—C5*A*	111.3 (4)	N3*B*—C4*B*—C3*B*	112.3 (4)
O2*A*—C7*A*—O1*A*	120.2 (4)	O1*B*—C7*B*—O2*B*	119.9 (4)
O2*A*—C7*A*—C8*A*	119.4 (4)	O1*B*—C7*B*—C8*B*	121.4 (4)
O1*A*—C7*A*—C8*A*	120.5 (4)	O2*B*—C7*B*—C8*B*	118.7 (4)

Table 3. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3*A*—H3*NA*···O24	0.900 (5)	2.35 (1)	3.13 (1)	144.9 (5)
N3*A*—H3*NB*···O13	0.900 (6)	2.235 (7)	3.106 (7)	163.0 (5)
N4*A*—H4*NA*···O14	0.900 (5)	2.468 (7)	3.344 (7)	164.7 (4)
N2*B*—H2*ND*···O12	0.900 (6)	2.263 (6)	3.127 (6)	160.7 (5)
N4*A*—H4*NB*···O1*B*$^{\rm i}$	0.900 (5)	2.178 (5)	2.997 (5)	150.9 (4)
N2*A*—H2*NB*···O11$^{\rm i}$	0.900 (6)	2.418 (9)	3.302 (9)	167.2 (5)
N2*B*—H2*NC*···O2*A*$^{\rm ii}$	0.900 (7)	2.266 (6)	3.125 (6)	159.5 (5)
N3*B*—H3*NC*···O23$^{\rm iii}$	0.900 (7)	2.442 (8)	3.228 (8)	146.2 (5)
N3*B*—H3*ND*···O1A$^{\rm iii}$	0.900 (6)	2.171 (5)	3.040 (5)	162.1 (5)

Symmetry codes: (i) $\frac{1}{2}-x, -y, z-\frac{1}{2}$; (ii) $\frac{1}{2}-x, -y, \frac{1}{2}+z$; (iii) $x, y, 1+z$.

The structure was solved by direct methods and refined by full-matrix least squares. All the H atoms were fixed geometrically and allowed to ride on those atoms to which they are attached, with fixed $U_{\rm iso}$ = 0.08 Å^2.

Data collection: *XSCANS* (Siemens, 1994). Cell refinement: *XSCANS*. Data reduction: *XSCANS*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990*a*). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL*/*PC* (Sheldrick, 1990*b*). Software used to prepare material for publication: *SHELXL*93. Geometric calculations: *PARST* (Nardelli, 1983).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KH1049). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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A New Decavanadate Dihydrate Templated by Ethylenediamine

Cyril Ninclaus, Didier Riou and Gérard Férey

Laboratoire des Fluorures URA 449, Faculté des Sciences, Université du Maine, 72017 Le Mans CEDEX, France

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Abstract

$(C_2H_{10}N_2)_3[V_{10}O_{28}].2H_2O$, tris(ethylenediammonium) decavanadate dihydrate, crystallizes in the monoclinic system ($P2_1/n$). Its structure, solved by single-crystal X-ray diffraction with a final R value of 0.0237 for 5136 unique reflections, is characterized by a pseudo body-centred arrangement of the $[V_{10}O_{28}]^{6-}$ polyanions, between which are intercalated the ethylenediammonium cations and the water molecules.

Comment

We have recently described a new family of vanadyl vanadates intercalated by organic templates and formulated as $[(V^{IV}O)(V^{V}O_4)]_2$.diamine (Riou & Férey, 1995*a*,*b*). Their two-dimensional structures are built up from inorganic layers of corner-sharing $V^{V}O_4$ tetrahedra and $[V^{IV}_2O_8]$ units in the form of two square pyramids; the organic cations are intercalated between them. In these phases, it was thought that the deintercalation of the amines from the structures could lead to a new form of V_2O_5. The thermal degradation method failed, as did the *in situ* oxidation of the amine in aqueous medium by hydrogen peroxide using $VO(VO_4)$.0.5(ethylenediamine) as the starting compound. We have not obtained a new form of V_2O_5 but a new member in the long list of decavanadates templated by organic cations (Zurkova & Vavra, 1993; Sucha, Sivak & Schwendt, 1993): $(C_2H_{10}N_2)_3[V_{10}O_{28}].2H_2O$ (Fig. 1). In this compound, the centrosymmetric decavanadate group is built up from five independent atoms. Its topology is very close to that recently described by Averbuch-Pouchot (1994). In the cell, the $[V_{10}O_{28}]^{6-}$ anions located around the origin and around the centre of the cell describe a pseudo body-centred arrangement. The electroneutrality is ensured by the ethylenediammonium cations, which also form strong hydrogen bonds with the water molecules and with some O atoms.

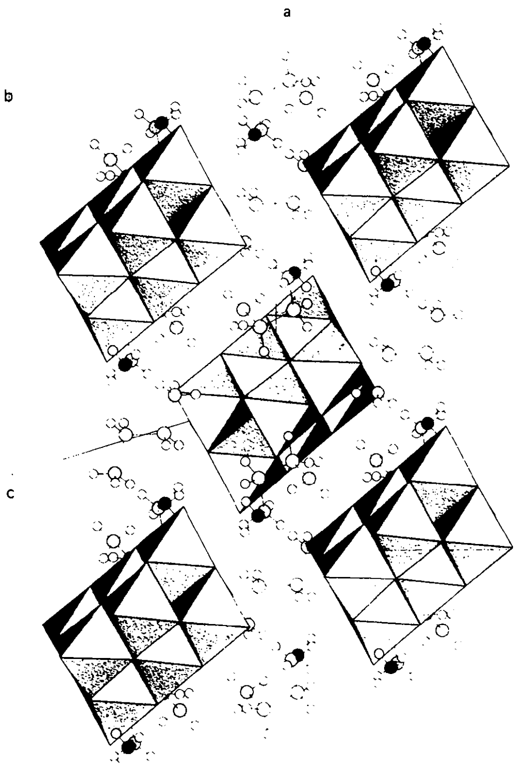

Fig. 1. Projection of the structure along the *b* axis. The N and C atoms are represented by dark and light shaded circles, respectively, O*W* by black circles and H by small circles.

Experimental

The title compound was prepared by dissolution then recrystallization at room temperature of $VO(VO_4).0.5(N_2C_2H_{10})$ (Riou & Férey, 1995*a*,*b*) (300 mg) in H_2O_2 (50 ml). In a first step, the vanadyl vanadate compound dissolved in 2–3 h to give an orange solution; after one day, small orange crystals were observed on the inner wall of the flask, their growth continuing until the decolouration of the solution. The density D_m was measured by multipycnometry (1305 micromeritics under He flow).

Crystal data

$(C_2H_{10}N_2)_3[V_{10}O_{28}].2H_2O$
$M_r = 590.0$
Monoclinic
$P2_1/n$
$a = 10.5096\,(8)$ Å
$b = 10.8730\,(10)$ Å
$c = 14.3200\,(10)$ Å
$\beta = 106.261\,(8)°$

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 38 reflections
$\theta = 14–16°$
$\mu = 3.119$ mm^{-1}
$T = 293$ K
Truncated parallelepiped

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