*Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)] = 0.0329$
$wR(F^2) = 0.1014$
$S = 1.167$
1145 reflections
125 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0716P)^2 + 0.2061P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.058$
$\Delta\rho_{max} = 0.786$ e Å$^{-3}$
$\Delta\rho_{min} = -0.426$ e Å$^{-3}$
Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.13 (1)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

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

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

| | $x$ | $y$ | $z$ | $U_{eq}$ |
|---|---|---|---|---|
| Mn1 | 0 | 0 | 0 | 0.0173 (3) |
| O1*W* | 0.0862 (4) | −0.2491 (3) | 0.2147 (3) | 0.0380 (6) |
| O2*W* | 0.2657 (3) | −0.0641 (3) | −0.2429 (3) | 0.0285 (5) |
| S1 | 0.19778 (8) | 0.22659 (8) | 0.24955 (8) | 0.0161 (3) |
| O1 | 0.3129 (3) | 0.3974 (3) | 0.1826 (3) | 0.0241 (5) |
| O2 | −0.0039 (3) | 0.2683 (3) | 0.3871 (3) | 0.0293 (5) |
| O3 | 0.3172 (3) | 0.0568 (3) | 0.3436 (3) | 0.0281 (5) |
| O4 | 0.1639 (3) | 0.1906 (3) | 0.0710 (2) | 0.0260 (5) |
| N | 0.3315 (4) | 0.4954 (3) | −0.2427 (3) | 0.0231 (5) |
| C | 0.5491 (4) | 0.5816 (4) | 0.4185 (4) | 0.0223 (6) |

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

| | | | |
|---|---|---|---|
| Mn1—O1*W*$^i$ | 2.160 (2) | S1—O1 | 1.478 (2) |
| Mn1—O4$^i$ | 2.179 (2) | S1—O4 | 1.487 (2) |
| Mn1—O2*W* | 2.189 (2) | N—C$^{ii}$ | 1.477 (3) |
| S1—O3 | 1.462 (2) | C—C$^{iii}$ | 1.519 (5) |
| S1—O2 | 1.467 (2) | | |
| O1*W*$^i$—Mn1—O1*W* | 180.0 | O3—S1—O2 | 110.3 (1) |
| O1*W*—Mn1—O4$^i$ | 87.03 (8) | O3—S1—O1 | 110.3 (1) |
| O1*W*—Mn1—O4 | 92.97 (8) | O2—S1—O1 | 110.3 (1) |
| O4$^i$—Mn1—O4 | 180.0 | O3—S1—O4 | 110.3 (1) |
| O1*W*—Mn1—O2*W* | 94.25 (9) | O2—S1—O4 | 108.8 (1) |
| O4$^i$—Mn1—O2*W* | 88.02 (8) | O1—S1—O4 | 106.8 (1) |
| O4—Mn1—O2*W* | 91.98 (8) | S1—O4—Mn1 | 138.2 (1) |
| O1*W*—Mn1—O2*W*$^i$ | 85.75 (9) | N$^{ii}$—C—C$^{iii}$ | 109.5 (3) |
| O2*W*—Mn1—O2*W*$^i$ | 180.0 | | |

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

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O1*W*—H1O1*W*···O1$^i$ | 0.88 (4) | 1.87 (4) | 2.736 (3) | 169 (3) |
| O1*W*—H2O1*W*···O2$^{ii}$ | 0.74 (5) | 2.02 (5) | 2.756 (3) | 172 (5) |
| O2*W*—H1O2*W*···O3$^{iii}$ | 0.70 (4) | 2.05 (4) | 2.729 (3) | 162 (4) |
| O2*W*—H2O2*W*···O3$^{iv}$ | 0.78 (6) | 2.07 (6) | 2.838 (3) | 168 (5) |
| N—H1N···O4 | 0.86 (4) | 1.98 (4) | 2.832 (3) | 172 (4) |
| N—H2N···O2$^v$ | 0.86 (4) | 2.02 (4) | 2.837 (3) | 156 (3) |
| N—H3N···O1$^{vi}$ | 0.91 (4) | 2.02 (4) | 2.880 (3) | 156 (3) |

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

Data were corrected for Lorentz–polarization and absorption effects. The structure was solved by Patterson methods. Refinement was by full-matrix least-squares methods.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: *MolEN* (Fair, 1990). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1976). 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: DU1123). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## References

Benghozlen, M. H., Kamoun, S., Paulush, H. & Pabst, I. (1994). *Z. Kristallogr.* **209**, 382.
Cox, D. E., Frazer, B. C. & Will, G. (1965). *Acta Cryst.* **19**, 854–857.
Enraf–Nonius (1989). *CAD*-4 *Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.
Fair, C. K. (1990). *MolEN. An Interactive Intelligent System for Crystal Structure Analysis*. Enraf–Nonius, Delft, The Netherlands.
Healy, C. P., Patrick, M. J. & White, H. A. (1984). *Aust. J. Chem.* T**37**, 1105–1109.
Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.
Sheldrick, G. M. (1985). *SHELXL*86. *Program for the Solution of Crystal Structures*. University of Göttingen, Germany.
Sheldrick, G. M. (1993). *SHELXL*93. *Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.

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# Tetrakis(triethylammonium) Octamolybdate(VI) Dihydrate

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## Abstract

The X-ray structure analysis of the title compound, $(C_6H_{16}N)_4[Mo_8O_{26}].2H_2O$, has confirmed the presence of the $[Mo_8O_{26}]^{4-}$ anion. Eight edge- and corner-sharing $MoO_6$ octahedra constitute the anion, which has short terminal Mo—O bonds [1.684 (3)–1.711 (3) Å], bonds of intermediate length [1.749 (3)–1.990 (3) Å] and long bonds [2.140 (2)–2.472 (3) Å]. The structure consists of $\beta$-$Mo_8O_{26}$ polyanions, triethylammonium

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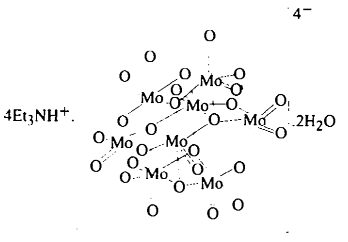
cations and water molecules of crystallization. The water molecules and triethylammonium cations are linked to the terminal and bridging O atoms of the anion by hydrogen bonds. Hydrogen bonds also exist between the water molecules and the triethylammonium cations.

## Comment

Studies of organoammonium isopolymolybdate compounds have aroused considerable interest in recent years in view of their photochemical and photochromic properties in solution as well as in the solid state (Xu, You & Wang, 1994). There are three types of octamolybdate structure, *i.e.* the $\alpha$ form, the $\beta$ form and the $\gamma$ form. Crystal structures of the $\alpha$-form octamolybdate have been reported for $(Bu_4N)_4[Mo_8O_{26}]$ (Fuchs & Hartl, 1976; Hsieh, Shaikh & Zubieta, 1987) and $(PPrPh_3)_4[Mo_8O_{26}].H_2O.MeCN$ (Day, Friedrich, Klemperer & Shum, 1977). The $\beta$-form structure has been observed for $(NH_4)_4[Mo_8O_{26}].nH_2O$ ($n$ = 4 or 5; Lindqvist, 1952) and $(C_5H_{10}NH_2)_4[Mo_8O_{26}].4H_2O$ (Xu, You & Wang, 1994). The $\gamma$ form has been observed for the first time recently in $[Me_3N(CH_2)_6NMe_3]_2[Mo_8O_{26}].2H_2O$ (Niven, Cruywagen & Heyns, 1991).

The title compound, (I), is colorless in the dark and is light pink when exposed to sunlight. In order to ascertain the mechanism of the photochromic reaction, the crystal structure of (I) has been determined.



(I)

An *ORTEP* plot (Johnson, 1965) with the atomic numbering for the polyanion is shown in Fig. 1. The anions can be described as two cyclic $Mo_4O_{12}$ units which are cross-linked by long Mo—O bonds and by additional long bonds from Mo to two extra $O^{2-}$ ions (*i.e.* O7 and O7*a*). The Mo—O bond lengths vary from 1.684 (3) Å, for one of the non-bridging Mo—O bonds, to 2.472 (3) Å, for one of the Mo—O bonds formed by the five-coordinate O atoms (O7 and O7*a*) which are near the centers of each half of the anion where the four Mo atoms are coplanar. In the idealized octamolybdate anion there are three different types of molybdenum octahedra: (*a*) octahedra formed by atoms Mo1 and Mo1*a*, which, being closest to the centroid of the polyanion, are the least distorted; (*b*) octahedra formed by atoms Mo4 and Mo4*a*, which are the most distorted since they are furthest from the centroid; and (*c*) octahedra formed by atoms Mo2, Mo2*a*, Mo3 and Mo3*a*, which have an intermediate degree of distortion (Xu, You & Wang, 1994). This is probably due to the effects of the neighbouring cations. The Mo—O bonds can be classified into three types according to their length, *i.e.* short terminal Mo—O bonds [1.684 (3)–1.711 (3) Å], bonds of intermediate length [1.749 (3)–1.990 (3) Å] and long bonds [2.140 (2)–2.472 (3) Å]. Comparing the title compound with $(C_5H_{10}NH_2)_4[Mo_8O_{26}].4H_2O$, it can be seen that there are slight differences in the Mo—O bond lengths due to the different cation–polyanion interactions. Most of the bond distances of the title compound are longer than those of $(C_5H_{10}NH_2)_4[Mo_8O_{26}].4H_2O$, except for the distances Mo1—O12, Mo2—O11, Mo2—O12, Mo3—O7 and Mo4—O1, which are shorter. The greatest difference is for the Mo3—O12 bond, which is 0.068 Å longer in the title compound than in $(C_5H_{10}NH_2)_4[Mo_8O_{26}].4H_2O$. The smallest difference is seen for the Mo2—O11 bond, which is 0.001 Å shorter in the title compound than in $(C_5H_{10}NH_2)_4[Mo_8O_{26}].4H_2O$.

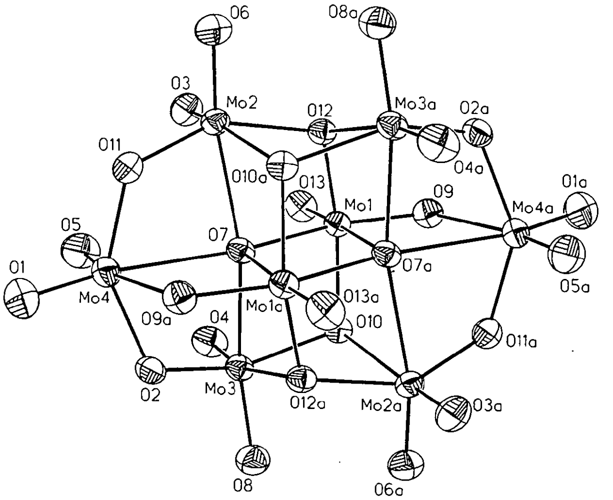


Fig. 1. *ORTEP* plot (Johnson, 1965) of the title polyanion showing the atom-numbering scheme and 40% probability displacement ellipsoids.

The title structure consists of centrosymmetric $\beta$-octamolybdate anions, *i.e.* $[Mo_8O_{26}]^{4-}$, triethylammonium cations and water molecules of crystallization. The packing diagram viewed down the *b* axis is shown in Fig. 2. O1*W*···O2 [2.761 (4) Å] and O1*W*···O3 [2.777 (5) Å] hydrogen bonds link the water molecules to the terminal and bridging O atoms of the polyanion. Two of the inversion-related triethylammonium cations are linked to terminal and bridging O atoms of the $[Mo_8O_{26}]^{4-}$ anion through N1···O6 [3.124 (5) Å] and C4···O12 [3.337 (6) Å] hydrogen bonds. The other two inversion-related disordered

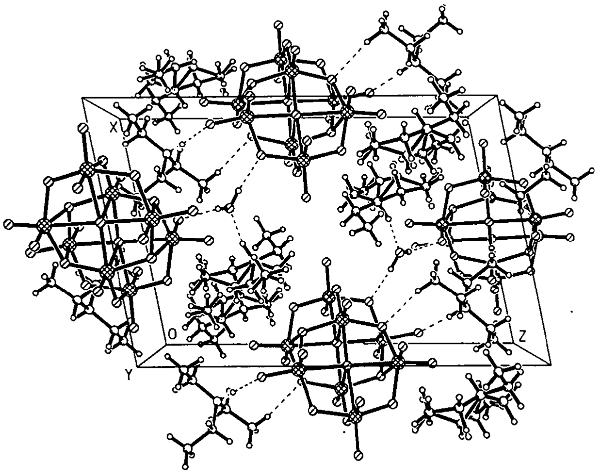


Fig. 2. Packing of the title molecules viewed down the *b* axis.

triethylammonium cations have no direct interaction with the polyanion but are linked to the water molecules by N2···O1*W* [2.735 (9) Å] hydrogen bonds.

## Experimental

The synthesis of the title compound was carried out by the reaction of 2.8 g (19 mmol) of $MoO_3$ and 3.0 ml (20 mmol) of triethylamine in a 40 ml aqueous solution for 10 min under reflux. After standing for 5 d at room temperature, the initially colorless block of crystalline product was formed and a crystal of suitable size was selected for X-ray diffraction analysis.

### *Crystal data*

$(C_6H_{16}N)_4[Mo_8O_{26}].2H_2O$
$M_r = 1628.34$
Monoclinic
$P2_1/n$
$a = 11.478 (3)$ Å
$b = 13.027 (3)$ Å
$c = 17.550 (4)$ Å
$\beta = 101.29 (2)°$
$V = 2573.4 (11)$ Å$^3$
$Z = 2$
$D_x = 2.101$ Mg m$^{-3}$

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 40 reflections
$\theta = 7.5–12.5°$
$\mu = 1.967$ mm$^{-1}$
$T = 293 (2)$ K
Transparent block
0.62 × 0.56 × 0.46 mm
Initially colorless, but turned dark purple upon irradiation with X-rays

### *Data collection*

Siemens *P*4 four-circle diffractometer
$\theta$–$2\theta$ scans
Absorption correction: analytical
$T_{\min} = 0.329$, $T_{\max} = 0.454$
7334 measured reflections
5885 independent reflections
5407 observed reflections [$I > 2\sigma(I)$]

$R_{\rm int} = 0.0274$
$\theta_{\max} = 27.5°$
$h = -14 \rightarrow 14$
$k = 0 \rightarrow 16$
$l = 0 \rightarrow 22$
3 standard reflections monitored every 97 reflections
intensity decay: none

### *Refinement*

Refinement on $F^2$
$R(F) = 0.0337$
$wR(F^2) = 0.0938$
$S = 1.174$
5885 reflections
296 parameters
$w = 1/[\sigma^2(F_o^2) + (0.0387P)^2 + 4.7336P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.958$ e Å$^{-3}$
$\Delta\rho_{\min} = -0.931$ e Å$^{-3}$

Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.0108 (3)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

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

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

| | $x$ | $y$ | $z$ | $U_{\rm eq}$ |
|---|---|---|---|---|
| Mo1 | 0.35460 (3) | 0.05642 (2) | −0.02015 (2) | 0.02927 (10) |
| Mo2 | 0.52529 (3) | 0.09950 (3) | −0.14169 (2) | 0.03278 (10) |
| Mo3 | 0.54631 (3) | 0.15252 (2) | 0.12003 (2) | 0.03315 (10) |
| Mo4 | 0.72270 (3) | 0.19914 (3) | −0.00240 (2) | 0.03928 (11) |
| O1 | 0.8694 (3) | 0.2257 (3) | 0.0099 (2) | 0.0635 (10) |
| O2 | 0.7016 (2) | 0.1838 (2) | 0.1036 (2) | 0.0386 (6) |
| O3 | 0.4535 (3) | 0.2143 (2) | −0.1586 (2) | 0.0458 (7) |
| O4 | 0.4757 (3) | 0.2661 (2) | 0.0997 (2) | 0.0497 (7) |
| O5 | 0.6539 (4) | 0.3137 (3) | −0.0212 (2) | 0.0589 (9) |
| O6 | 0.5324 (3) | 0.0547 (3) | −0.2317 (2) | 0.0477 (7) |
| O7 | 0.5366 (2) | 0.1015 (2) | −0.00736 (14) | 0.0305 (5) |
| O8 | 0.5749 (3) | 0.1446 (2) | 0.2192 (2) | 0.0463 (7) |
| O9 | 0.2372 (2) | −0.0294 (2) | −0.0210 (2) | 0.0384 (6) |
| O10 | 0.4050 (2) | 0.0616 (2) | 0.09273 (15) | 0.0326 (5) |
| O11 | 0.6834 (3) | 0.1416 (2) | −0.1052 (2) | 0.0402 (6) |
| O12 | 0.3880 (2) | 0.0191 (2) | −0.12087 (14) | 0.0309 (5) |
| O13 | 0.2881 (3) | 0.1714 (2) | −0.0387 (2) | 0.0441 (7) |
| N1 | 0.6851 (3) | 0.0243 (3) | 0.3533 (2) | 0.0483 (9) |
| C1 | 0.7851 (5) | 0.0990 (4) | 0.3763 (3) | 0.0603 (13) |
| C2 | 0.8633 (7) | 0.0779 (6) | 0.4513 (4) | 0.095 (2) |
| C3 | 0.7285 (5) | −0.0821 (3) | 0.3403 (3) | 0.0633 (15) |
| C4 | 0.8028 (6) | −0.0880 (5) | 0.2815 (3) | 0.074 (2) |
| C5 | 0.6000 (6) | 0.0225 (5) | 0.4083 (3) | 0.078 (2) |
| C6 | 0.5483 (9) | 0.1231 (7) | 0.4172 (6) | 0.120 (3) |
| N2 | 1.1183 (6) | 0.0904 (6) | 0.1791 (6) | 0.125 (3) |
| C7† | 1.155 (4) | 0.0136 (12) | 0.2430 (7) | 0.221 (16) |
| C7′† | 1.2203 (13) | 0.063 (3) | 0.2425 (7) | 0.153 (9) |
| C8 | 1.1791 (13) | 0.0708 (12) | 0.3158 (8) | 0.199 (7) |
| C9 | 1.0795 (12) | 0.0159 (9) | 0.1141 (6) | 0.187 (7) |
| C10 | 1.0161 (10) | −0.0725 (9) | 0.1359 (8) | 0.150 (5) |
| C11† | 1.2064 (19) | 0.1760 (13) | 0.1983 (16) | 0.190 (12) |
| C11′† | 1.1513 (16) | 0.1653 (10) | 0.1218 (8) | 0.094 (4) |
| C12 | 1.1677 (13) | 0.2677 (10) | 0.1530 (10) | 0.249 (10) |
| O1*W* | 0.9083 (3) | 0.1576 (3) | 0.2125 (2) | 0.0641 (10) |

† Occupancy = 0.5.

Table 2. *Selected bond lengths* (Å)

| | | | |
|---|---|---|---|
| Mo1—O13 | 1.684 (3) | Mo3—O4 | 1.691 (3) |
| Mo1—O9 | 1.749 (3) | Mo3—O8 | 1.711 (3) |
| Mo1—O12 | 1.942 (2) | Mo3—O2 | 1.904 (3) |
| Mo1—O10 | 1.953 (3) | Mo3—O10 | 1.990 (3) |
| Mo1—O7 | 2.140 (2) | Mo3—O7 | 2.314 (3) |
| Mo1—O7$^{\rm i}$ | 2.406 (3) | Mo3—O12$^{\rm i}$ | 2.358 (3) |
| Mo2—O6 | 1.701 (3) | Mo4—O5 | 1.690 (3) |
| Mo2—O3 | 1.705 (3) | Mo4—O1 | 1.691 (3) |
| Mo2—O11 | 1.884 (3) | Mo4—O11 | 1.924 (3) |
| Mo2—O12 | 1.985 (3) | Mo4—O2 | 1.933 (3) |
| Mo2—O7 | 2.336 (3) | Mo4—O9$^{\rm i}$ | 2.279 (3) |
| Mo2—O10$^{\rm i}$ | 2.348 (3) | Mo4—O7 | 2.472 (3) |

Symmetry code: (i) $1 - x, -y, -z$.

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.

## References

Day, V. W., Friedrich, M. F., Klemperer, W. G. & Shum, W. (1977). *J. Am. Chem. Soc.* **99**, 952–953.

Fuchs, J. & Hartl, H. (1976). *Angew. Chem. Int. Ed. Engl.* **15**, 375–376.

Hsieh, T.-C., Shaikh, S. N. & Zubieta, J. (1987). *Inorg. Chem.* **26**, 4079–4089.

Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.

Lindqvist, I. (1952). *Acta Cryst.* **5**, 667–670.

Niven, M. L., Cruywagen, J. J. & Heyns, J. B. B. (1991). *J. Chem. Soc. Dalton Trans.* pp. 2007–2011.

Sheldrick, G. M. (1990*a*). *Acta Cryst.* A**46**, 467–473.

Sheldrick, G. M. (1990*b*). *SHELXTL/PC. Structure Determination Software Programs.* Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Sheldrick, G. M. (1993). *SHELXL93. Program for the Refinement of Crystal Structures.* University of Göttingen, Germany.

Siemens (1994). *XSCANS. X-ray Single Crystal Analysis System.* Version 2.1. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Xu, X.-X., You, X.-Z. & Wang, X. (1994). *Polyhedron*, **13**, 1011–1014.

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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

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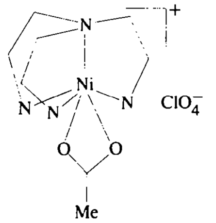
## Abstract

The X-ray structure analysis of the title compound, $[\rm Ni(C_2H_3O_2)(C_6H_{18}N_4)]ClO_4$, 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