

Data collection

Syntex P4 automated diffractometer	792 observed reflections
$\theta/2\theta$ scans	$[F > 6.0\sigma(F)]$
Absorption correction:	$R_{\text{int}} = 0.0355$
semi-empirical from ψ scans (XEMP; Siemens, 1991)	$\theta_{\text{max}} = 23.0^\circ$
$T_{\text{min}} = 0.25$, $T_{\text{max}} = 0.68$	$h = -1 \rightarrow 6$
1368 measured reflections	$k = -1 \rightarrow 11$
931 independent reflections	$l = -12 \rightarrow 12$
	3 standard reflections
	monitored every 97 reflections
	intensity decay: <5%

Refinement

Refinement on F	$\Delta\rho_{\text{max}} = 1.80 \text{ e } \text{\AA}^{-3}$
$R = 0.052$	$\Delta\rho_{\text{min}} = -1.74 \text{ e } \text{\AA}^{-3}$
$wR = 0.063$	Extinction correction: none
$S = 1.20$	Atomic scattering factors
931 reflections	from <i>International Tables</i>
86 parameters	for <i>X-ray Crystallography</i>
$w = 1/[\sigma^2(F) + 0.0008F^2]$	(1974, Vol. IV, Table
$(\Delta/\sigma)_{\text{max}} = 0.006$	2.2B)

Table 3. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2) for $\text{LiNi}_2\text{P}_3\text{O}_{10}$

	x	y	z	U_{eq}
Ni1	0.0366 (1)	0.0691 (1)	0.3363 (1)	0.007 (1)
Li1	0	0	0	0.030 (4)
P1	0.4637 (3)	1/4	0.1452 (1)	0.006 (1)
P2	-0.6669 (3)	1/4	0.8209 (1)	0.006 (1)
P3	-0.3979 (3)	1/4	0.5346 (1)	0.006 (1)
O11	0.2891 (6)	0.0985 (3)	0.1597 (3)	0.008 (1)
O12	0.7700 (9)	1/4	0.2381 (4)	0.009 (1)
O21	-0.4319 (10)	1/4	0.7100 (4)	0.011 (1)
O22	-0.4459 (9)	1/4	0.9756 (4)	0.008 (1)
O23	-0.8400 (7)	0.0985 (3)	0.8107 (3)	0.010 (1)
O31	-0.2283 (6)	0.0972 (3)	0.5129 (3)	0.008 (1)
O32	-0.7074 (9)	1/4	0.4462 (4)	0.009 (1)

Table 4. Selected geometric parameters (\AA) for $\text{LiNi}_2\text{P}_3\text{O}_{10}$

Ni1—O12 ⁱ	2.059 (3)	P1—O11 ^{vii}	1.513 (3)
Ni1—O23 ⁱⁱ	2.040 (3)	P1 ^{viii} —O22	1.624 (4)
Ni1—O31 ⁱⁱⁱ	2.044 (3)	P1—O12	1.525 (4)
Ni1—O31	2.132 (3)	P2—O21	1.560 (4)
Ni1—O11	2.095 (3)	P2—O22	1.592 (4)
Ni1—O32 ^{iv}	2.072 (3)	P2—O23	1.489 (3)
Li1—O11	1.982 (3)	P2—O23 ^{vii}	1.489 (3)
Li1—O23 ^v	2.100 (3)	P3—O21	1.594 (4)
Li1—O11 ^{vi}	1.982 (3)	P3—O32	1.521 (4)
Li1—O23 ⁱⁱⁱ	2.100 (3)	P3—O31	1.521 (3)
P1—O11	1.513 (3)	P3—O31 ^{vii}	1.521 (3)

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

A variable scan rate was used with a scan width of 1.2° below $K\alpha_1$ and 1.2° above $K\alpha_2$. Refinement was completed using full-matrix least-squares methods.

For both compounds, data collection: XSCANS (Siemens, 1991); cell refinement: XSCANS; data reduction: XSCANS; program(s) used to solve structures: SHELXS86 (Sheldrick, 1990); program(s) used to refine structures: SHELXTL-Plus (Sheldrick, 1992); molecular graphics: XP (Siemens, 1990).

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Lists of structure factors, anisotropic displacement parameters and complete geometry have been deposited with the IUCr (Reference: BR1124). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

- Averbuch-Pouchot, M. T. & Durif, A. (1975). *J. Appl. Cryst.* **8**, 564.
 Averbuch-Pouchot, M. T. & Durif, A. (1985). *Acta Cryst.* **C41**, 1553–1555.
 Averbuch-Pouchot, M. T., Durif, A., Coing-Boyat, J. & Guitel, J. C. (1977). *Acta Cryst.* **B33**, 203–205.
 Averbuch-Pouchot, M. T., Durif, A. & Guitel, J. C. (1975). *Acta Cryst.* **B31**, 2482–2486.
 Bagieu-Bucher, M., Durif, A. & Averbuch-Pouchot, M. T. (1976). *J. Appl. Cryst.* **9**, 412.
 Corbridge, D. E. C. (1960). *Acta Cryst.* **13**, 263–269.
 Corbridge, D. E. C. & Tromans, E. R. (1958). *Anal. Chem.* **30**, 1101–1110.
 Davies, D. R. & Corbridge, D. E. C. (1958). *Acta Cryst.* **11**, 315–319.
 Dyroff, D. R. (1965). Thesis, University of California, USA.
 Jouini, O., Dabbabi, M., Averbuch-Pouchot, M. T., Durif, A. & Guitel, J. C. (1984). *Acta Cryst.* **C40**, 728–730.
 Lee, J. D. (1968). *J. Chem. Soc.* pp. 2881–2882.
 Sheldrick, G. M. (1990). *Acta Cryst.* **A46**, 467–473.
 Sheldrick, G. M. (1992). *SHELXTL-Plus*. Release 4.21/V. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
 Siemens (1990). *XP. Interactive Molecular Graphics Program*. Version 4.3. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
 Siemens (1991). *XSCANS User's Manual*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

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Zigzag Chain Structure of Hexaammonium Enneamolybdate Pentahydrate

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Abstract

The title compound, $(\text{NH}_4)_6[\text{Mo}_9\text{O}_{30}]\cdot 5\text{H}_2\text{O}$, contains an $\{[\text{Mo}_8\text{O}_{26}(\text{MoO}_4)]^{6-}\}_n$ anion with a zigzag chain structure, in which two adjacent Mo_8O_{26} moieties are linked by an MoO_4 tetrahedron.

Comment

The two MoO_5 trigonal bipyramids in the γ - $[\text{Mo}_8\text{O}_{26}]^{4-}$ anion (Niven, Cruywagen & Heyns, 1991) coordinate with a variety of oxygen- or nitrogen-donating ligands to form MoO_6 octahedra, as exemplified by the structures of $[\text{Mo}_8\text{O}_{26}(\text{OH})_2]^{6-}$ (Isobe, Marumo, Yamase & Ikawa, 1978), $[\text{Mo}_8\text{O}_{26}(\text{HCO}_2)_2]^{6-}$ (Adams, Klemperer & Liu, 1979), $[\text{Mo}_8\text{O}_{26}(\text{C}_5\text{H}_5\text{N})_2]^{4-}$ (McCarron, Whitney & Chase, 1984), $[\text{Mo}_8\text{O}_{26}(\text{MoO}_4)_2]^{8-}$ (Garin & Costamagna, 1988; Touboul, Idoura & Tolédano, 1984; Bharadwaj, Ohashi, Sasada, Sasaki & Yamase, 1986), $[\text{Mo}_8\text{O}_{24}(\text{OCH}_3)_4]^{4-}$ (McCarron & Harlow, 1983), $[\text{Mo}_8\text{O}_{22}(\text{OH})_4(\text{OC}_6\text{H}_4\text{CH}=\text{NPr}-2)_2]^{2-}$ ($\text{OC}_6\text{H}_4\text{CH}=\text{NPr}-2 = N$ -propylsalicylideneimine) (Kamenar, Korpar-Colig, Penavic & Cindric, 1990) and $[\text{Mo}_8\text{O}_{24}(\text{OH})_2(\text{MetO})_2]^{4-}$ (MetO = methioninate) (Kamenar *et al.*, 1990). In addition, two polymeric forms of the γ -octamolybdates have been discovered. The first example is $\{(\text{NH}_4)_6[\text{Mo}_8\text{O}_{26}(\text{O})]\}_n$ (Böschen, Buss & Krebs, 1974), in which the Mo_8O_{26} repeat units are linked by a μ_2 -O atom to form infinite chains of eight edge-sharing MoO_6 octahedra. The second example is $\{\text{Eu}_2(\text{H}_2\text{O})_{12}[\text{Mo}_8\text{O}_{26}(\text{O})]\}_n$ (Yamase & Naruke, 1991), in which the nine-coordinate Eu^{3+} ion is linked by two O atoms of an Mo_8O_{26} unit and one O atom of a neighbouring unit in the infinite chain $\{[\text{Mo}_8\text{O}_{26}(\text{O})]^{6-}\}_n$. The Mo_8O_{26} moieties of these chain compounds are arranged parallel to each other. We report here a novel example of $\{[\text{Mo}_8\text{O}_{26}(\text{MoO}_4)]^{6-}\}_n$, in which two adjacent Mo_8O_{26} moieties are linked by MoO_4^{2-} to form a polymeric structure based on an infinite zigzag chain. To our knowledge, this is the first example of a zigzag chain structure for the polymeric octamolybdates.

An ORTEPII (Johnson, 1976) plot of the $\{(\text{Mo}_8\text{O}_{26})_2(\text{MoO}_4)\}^{10-}$ unit, in which centrosymmetric Mo_8O_{26} moieties are linked by a MoO_4 tetrahedron, is shown in Fig. 1. The discrete $[\text{Mo}_8\text{O}_{26}(\text{MoO}_4)_2]^{8-}$ anion has been found in $[(\text{CH}_3)\text{NH}_3]_8[\text{Mo}_8\text{O}_{26}(\text{MoO}_4)_2]\cdot 2\text{H}_2\text{O}$ (hereafter MAM; Bharadwaj *et al.*, 1986), where the γ - $[\text{Mo}_8\text{O}_{26}]^{4-}$ ion coordinates to two MoO_4 tetrahedra at the positions of the O14 and O14ⁱ atoms in Fig. 1. Another isomer of the discrete anion, in which MoO_4 groups are attached at the positions of the O7 and O7ⁱ

atoms in Fig. 1, is found in $(\text{NH}_4)_6[\text{Mo}_8\text{O}_{26}(\text{MoO}_4)_2]$ and $\text{Ti}_8[\text{Mo}_8\text{O}_{26}(\text{MoO}_4)_2]$ (Garin & Costamagna, 1988; Touboul *et al.*, 1984).

Table 2 shows the Mo—O bond distances in the $\{[\text{Mo}_8\text{O}_{26}(\text{MoO}_4)]^{6-}\}_n$ anion. Each MoO_6 octahedron in the Mo_8O_{26} moieties has two short *cis*, two medium *trans* and two long *cis* Mo—O bonds in the ranges 1.698(6)–1.765(5) Å for the short bonds, 1.880(5)–2.042(5) Å for the medium bonds and 2.121(5)–2.447(5) Å for the long bonds. All the Mo—O(terminal) distances are in the short range, which is indicative of double bonding. There are two other short Mo—O distances, namely Mo3—O9 1.764(5) and Mo7—O27 1.765(5) Å, for which the O atoms are bicoordinate. These values agree very well with those reported for MAM. The long Mo—O distances of each MoO_6 octahedron are due to the *trans* influence of the terminal O atoms. The Mo···Mo distances between the MoO_4 tetrahedron and the neighbouring MoO_6 octahedra are Mo4···Mo5 3.513(1) and Mo5···Mo6 3.600(1) Å, which are shorter than the corresponding distance [3.661(1) Å] for MAM (Bharadwaj *et al.*, 1986). In addition, the Mo—O distances between Mo5 and the non-bridging O atoms, O15 and O16, are 1.733(6) and 1.709(6) Å, respectively, and are shorter than those in MAM [1.743(5)–1.752(4) Å; Bharadwaj *et al.*, 1986]. The O15 and O16 atoms are 2.82–3.85 Å away from the nearest terminal O atoms: O15···O5 3.647(8), O15···O20 3.053(8), O15···O24ⁱⁱ 3.603(8), O16···O5 3.003(8), O16···O7 3.852(8), O16···O18 3.365(8), O16···O24ⁱⁱ 2.823(8) Å. The least-squares plane containing Mo2, Mo3, Mo2ⁱ and Mo3ⁱ in the Mo_8O_{26} moiety intersects with the corresponding plane containing Mo7, Mo8, Mo7ⁱⁱ and Mo8ⁱⁱ in the adjacent Mo_8O_{26} moiety at an angle of 65.0(1)°. This angle indicates that the configuration of the two adjacent Mo_8O_{26} moieties is not parallel but zigzag. Fig. 2 shows the crystal structure of $(\text{NH}_4)_6[\text{Mo}_8\text{O}_{26}(\text{MoO}_4)]\cdot 5\text{H}_2\text{O}$ viewed along the *a* axis. All of the ammonium N atoms have four to six contacts to the anion and water O atoms with N···O distances of 2.78–3.00 Å, probably extending the hydrogen-bond network across the $\{[\text{Mo}_8\text{O}_{26}(\text{MoO}_4)]^{6-}\}_n$ chains.

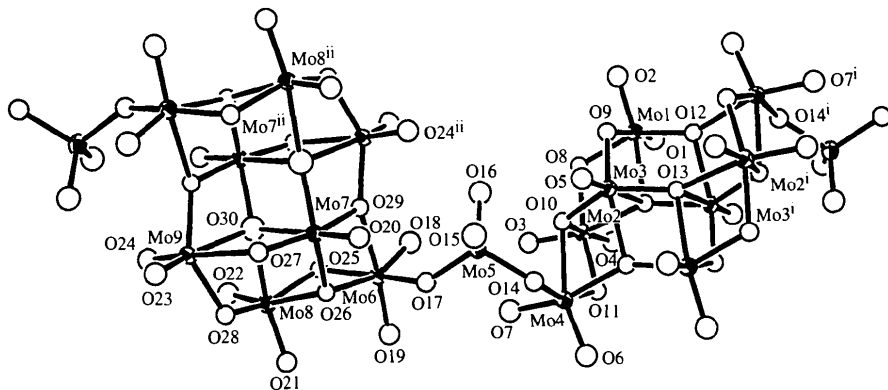


Fig. 1. An ORTEPII (Johnson, 1976) plot of the $\{(\text{Mo}_8\text{O}_{26})_2(\text{MoO}_4)\}^{10-}$ anion with the atom-numbering scheme.

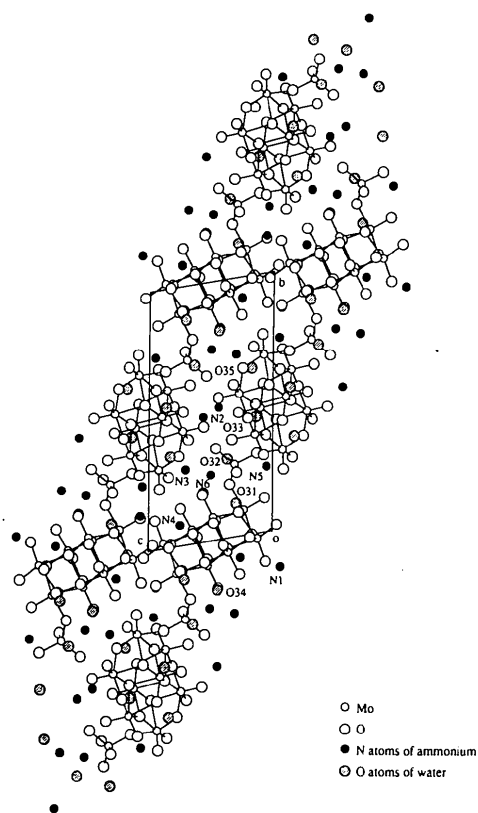


Fig. 2. A packing diagram for $(\text{NH}_4)_6[\text{Mo}_8\text{O}_{26}(\text{MoO}_4)].5\text{H}_2\text{O}$ viewed along the a axis.

Experimental

5 g of $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24}.4\text{H}_2\text{O}$ was dissolved in 13 ml of water and 0.8 ml CH_3COOH was added dropwise with stirring. The solution, at pH 4, was warmed and kept at 318 K for 20 min and then cooled to room temperature. Colourless crystals were produced after 3 d, together with a white powder.

Crystal data

$(\text{NH}_4)_6[\text{Mo}_9\text{O}_{30}].5\text{H}_2\text{O}$

$M_r = 1541.75$

Triclinic

$P\bar{1}$

$a = 10.622(2) \text{ \AA}$

$b = 18.234(1) \text{ \AA}$

$c = 10.400(2) \text{ \AA}$

$\alpha = 95.31(1)^\circ$

$\beta = 120.16(1)^\circ$

$\gamma = 96.62(1)^\circ$

$V = 1703(1) \text{ \AA}^3$

$Z = 2$

$D_x = 3.01 \text{ Mg m}^{-3}$

Data collection

Rigaku AFC-5S diffractometer

Mo $K\alpha$ radiation

$\lambda = 0.71069 \text{ \AA}$

Cell parameters from 25 reflections

$\theta = 10\text{--}12.5^\circ$

$\mu = 3.24 \text{ mm}^{-1}$

$T = 298 \text{ K}$

Thin plate

$0.3 \times 0.15 \times 0.01 \text{ mm}$

Colourless

$R_{\text{int}} = 0.018$

$\theta_{\text{max}} = 27.5^\circ$

$\omega/2\theta$ scans

Absorption correction:

refined from ΔF

(DIFABS; Walker &

Stuart, 1983)

$T_{\text{min}} = 0.84$, $T_{\text{max}} = 0.97$

7807 measured reflections

7422 independent reflections

6280 observed reflections

$[I > 2.0\sigma(I)]$

$h = -13 \rightarrow 11$

$k = -23 \rightarrow 23$

$l = 0 \rightarrow 13$

3 standard reflections

monitored every 100

reflections

intensity decay: 1.6%

Refinement

Refinement on F

$R = 0.0414$

$wR = 0.0337$

$S = 1.826$

6280 reflections

246 parameters

H atoms not located

$w = 1/\sigma^2(F)$

$(\Delta/\sigma)_{\text{max}} = 0.0109$

$\Delta\rho_{\text{max}} = 2.46 \text{ e \AA}^{-3}$

(0.71 \AA from O33)

$\Delta\rho_{\text{min}} = -1.18 \text{ e \AA}^{-3}$

(0.61 \AA from O33)

Extinction correction: none

Atomic scattering factors

from *International Tables*

for X-ray Crystallography

(1974, Vol. IV)

Table 1. Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

B_{iso} for O and N, $B_{\text{eq}} = (8\pi^2/3)\sum_i\sum_j U_{ij}a_i^*a_j^*$ for Mo atoms.

	x	y	z	$B_{\text{iso}}/B_{\text{eq}}$
Mo1	1.14739 (8)	-0.05520 (4)	0.35422 (8)	1.86 (2)
Mo2	0.82078 (7)	-0.02037 (4)	0.12146 (7)	1.86 (2)
Mo3	1.03597 (7)	0.09269 (4)	0.48056 (7)	1.60 (2)
Mo4	0.68298 (7)	0.10210 (4)	0.23128 (8)	1.87 (2)
Mo5	0.88131 (8)	0.28433 (4)	0.30654 (8)	2.06 (2)
Mo6	0.72850 (7)	0.31048 (4)	-0.07940 (7)	1.77 (2)
Mo7	0.91794 (7)	0.48640 (4)	0.10453 (7)	1.55 (2)
Mo8	0.63906 (7)	0.44425 (4)	-0.27349 (7)	1.81 (2)
Mo9	0.83393 (7)	0.61325 (4)	-0.14701 (8)	1.75 (2)
O1	1.0982 (6)	-0.1454 (3)	0.2594 (6)	2.9 (1)
O2	1.3236 (6)	-0.0250 (3)	0.3928 (6)	2.9 (1)
O3	0.7680 (6)	0.0213 (3)	-0.0343 (6)	2.7 (1)
O4	0.7606 (6)	-0.1138 (3)	0.0532 (6)	2.7 (1)
O5	1.0960 (6)	0.1841 (3)	0.5665 (6)	2.6 (1)
O6	0.5382 (6)	0.0960 (3)	0.2605 (6)	2.9 (1)
O7	0.6226 (6)	0.1340 (3)	0.0642 (6)	2.9 (1)
O8	1.0281 (5)	-0.0115 (3)	0.1819 (6)	2.0 (1)
O9	1.1896 (6)	0.0680 (3)	0.4761 (6)	2.0 (1)
O10	0.9064 (5)	0.0946 (3)	0.2749 (5)	1.8 (1)
O11	0.6684 (5)	-0.0012 (3)	0.1651 (6)	2.0 (1)
O12	0.8235 (5)	0.0782 (3)	0.4583 (5)	1.9 (1)
O13	1.0525 (5)	0.0394 (3)	0.6397 (5)	1.7 (1)
O14	0.7832 (6)	0.2090 (3)	0.3451 (6)	2.2 (1)
O15	0.9599 (7)	0.3567 (3)	0.4600 (7)	3.4 (1)
O16	1.0178 (6)	0.2519 (3)	0.2893 (6)	3.0 (1)
O17	0.7573 (6)	0.3138 (3)	0.1306 (6)	2.2 (1)
O18	0.8025 (6)	0.2336 (3)	-0.0882 (6)	2.8 (1)
O19	0.5427 (6)	0.2752 (3)	-0.1606 (6)	2.7 (1)
O20	0.9428 (6)	0.4811 (3)	0.2777 (6)	2.4 (1)
O21	0.4616 (6)	0.4082 (3)	-0.3200 (6)	2.5 (1)
O22	0.6290 (6)	0.4478 (3)	-0.4417 (6)	2.7 (1)
O23	0.8193 (6)	0.6977 (3)	-0.0740 (6)	2.7 (1)
O24	0.7846 (6)	0.6185 (3)	-0.3289 (6)	2.6 (1)
O25	0.7191 (5)	0.3500 (3)	-0.2457 (5)	1.9 (1)
O26	0.7222 (5)	0.4304 (3)	-0.0315 (5)	1.7 (1)
O27	0.8836 (5)	0.5780 (3)	0.0802 (5)	1.9 (1)
O28	0.6507 (5)	0.5462 (3)	-0.2014 (5)	2.0 (1)
O29	1.0442 (5)	0.6217 (3)	-0.0563 (5)	1.8 (1)
O30	1.1179 (5)	0.5040 (3)	0.1427 (5)	1.8 (1)
O31	0.3070 (7)	0.1298 (3)	0.2941 (7)	3.7 (1)
O32	0.558 (1)	0.3089 (5)	0.364 (1)	8.4 (3)
O33	0.580 (1)	0.4535 (5)	0.139 (1)	8.7 (3)
O34	0.6032 (9)	0.1907 (4)	0.5601 (9)	6.0 (2)
O35	0.7019 (8)	0.6458 (4)	0.1737 (9)	5.6 (2)

N1	0.9798 (7)	-0.1439 (4)	-0.0641 (7)	2.6 (1)
N2	1.2040 (8)	0.4858 (4)	0.5618 (8)	3.2 (1)
N3	0.9465 (9)	0.2929 (5)	0.7010 (9)	4.1 (2)
N4	0.5127 (8)	0.0817 (4)	0.7432 (8)	3.0 (1)
N5	0.4308 (8)	0.2556 (4)	0.0486 (8)	3.5 (2)
N6	1.3260 (8)	0.2585 (4)	0.5001 (8)	3.4 (2)

Table 2. Selected geometric parameters (Å)

Mo1—O1	1.712 (6)	Mo5—O16	1.709 (6)
Mo1—O2	1.714 (6)	Mo5—O17	1.815 (5)
Mo1—O8	1.915 (5)	Mo6—O17	2.042 (5)
Mo1—O9	2.350 (5)	Mo6—O18	1.701 (6)
Mo1—O12 ⁱ	1.910 (5)	Mo6—O19	1.719 (6)
Mo1—O13 ⁱ	2.207 (5)	Mo6—O25	1.896 (5)
Mo2—O3	1.712 (5)	Mo6—O26	2.213 (5)
Mo2—O4	1.698 (6)	Mo6—O29 ⁱⁱ	2.217 (5)
Mo2—O8	1.944 (5)	Mo7—O20	1.699 (5)
Mo2—O10	2.291 (5)	Mo7—O26	1.912 (5)
Mo2—O11	1.946 (5)	Mo7—O27	1.765 (5)
Mo2—O13 ⁱ	2.238 (5)	Mo7—O29 ⁱⁱ	2.121 (5)
Mo3—O5	1.700 (6)	Mo7—O30	1.935 (5)
Mo3—O9	1.764 (5)	Mo7—O30 ⁱⁱ	2.429 (5)
Mo3—O10	1.880 (5)	Mo8—O21	1.721 (5)
Mo3—O12	2.132 (5)	Mo8—O22	1.706 (5)
Mo3—O13	1.939 (5)	Mo8—O25	1.985 (5)
Mo3—O13 ⁱ	2.447 (5)	Mo8—O26	2.261 (5)
Mo4—O6	1.705 (6)	Mo8—O28	1.906 (5)
Mo4—O7	1.710 (6)	Mo8—O30 ⁱⁱ	2.253 (5)
Mo4—O10	2.205 (5)	Mo9—O23	1.711 (6)
Mo4—O11	1.905 (5)	Mo9—O24	1.706 (5)
Mo4—O12	2.190 (5)	Mo9—O27	2.320 (5)
Mo4—O14	2.040 (5)	Mo9—O28	1.954 (5)
Mo5—O14	1.824 (5)	Mo9—O29	1.919 (5)
Mo5—O15	1.733 (6)	Mo9—O30	2.254 (5)

Symmetry codes: (i) $2 - x, -y, 1 - z$; (ii) $2 - x, 1 - y, -z$.

Pairs of atoms further than 3.97 Å apart were assigned to be pairs of N atoms of ammonium ions, as distances between two N atoms should be longer than 3.7 Å (Seimons & Templeton, 1954).

All calculations were carried out using *TEXSAN* (Molecular Structure Corporation, 1989) software running on a MicroVAXII computer.

Lists of structure factors, anisotropic displacement parameters, hydrogen-bond distances and complete geometry have been deposited with the IUCr (Reference: OH1091). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

- Adams, R. D., Klempner, W. G. & Liu, R. (1979). *J. Chem. Soc. Chem. Commun.* pp. 256–257.
- Bharadwaj, P. K., Ohashi, Y., Sasada, Y., Sasaki, Y. & Yamase, T. (1986). *Acta Cryst.* **C42**, 545–547.
- Böschchen, I., Buss, B. & Krebs, B. (1974). *Acta Cryst.* **B30**, 48–56.
- Garin, J. L. & Costamagna, J. A. (1988). *Acta Cryst.* **C44**, 779–782.
- Isobe, M., Marumo, F., Yamase, T. & Ikawa, T. (1978). *Acta Cryst.* **B34**, 2728–2731.
- Johnson, C. K. (1976). *ORTEP*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
- Kamenar, B., Korpar-Colig, B., Penavic, M. & Cindric, M. (1990). *J. Chem. Soc. Dalton Trans.* pp. 1125–1130.
- McCarron, E. M. III & Harlow, I. R. L. (1983). *J. Am. Chem. Soc.* **105**, 6179–6181.
- McCarron, E. M. III, Whitney, J. F. & Chase, D. B. (1984). *Inorg. Chem.* **23**, 3275–3280.

- Molecular Structure Corporation (1989). *TEXSAN. Single Crystal Structure Analysis Software*. MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.
- Niven, M. L., Cruywagen, J. J. & Heyns, B. B. (1991). *J. Chem. Soc. Dalton Trans.* pp. 2007–2011.
- Seimons, W. J. & Templeton, D. H. (1954). *Acta Cryst.* **7**, 194–198.
- Touboul, M., Idoura, C. & Tolédano, P. (1984). *Acta Cryst.* **C40**, 1652–1655.
- Walker, N. & Stuart, D. (1983). *Acta Cryst.* **A39**, 158–166.
- Yamase, T. & Naruke, H. (1991). *J. Chem. Soc. Dalton Trans.* pp. 285–292.

Acta Cryst. (1996). **C52**, 1872–1874

Triperchloratoytterbium(III) Monohydrate, $\text{Yb}(\text{ClO}_4)_3\cdot\text{H}_2\text{O}$

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Abstract

The crystal structure of triperchloratoytterbium(III) monohydrate, $\text{Yb}(\text{ClO}_4)_3\cdot\text{H}_2\text{O}$, has been determined. In this coordination compound, bidentate and tridentate perchlorato groups form a channelled three-dimensional network. The eightfold coordination of ytterbium is completed by one strongly bound water molecule lying inside the channels.

Comment

In spite of its well known weak coordination behaviour, ClO_4^- can compete with more basic molecules or ions such as H_2O or OH^- in complexing small-sized and polarizing cations like the lanthanide ions Ln^{3+} (Huskowska, Legendziewicz, Schleid & Meyer, 1992). This is demonstrated well by the compounds $\text{Nd}_2(\text{OH})_3(\text{ClO}_4)_3\cdot 5\text{H}_2\text{O}$ (Csöregi, Huskowska, Ertan, Legendziewicz & Kierkegaard, 1989) and $\text{Pr}_2(\text{OH})_3\text{H}_2\text{O}(\text{ClO}_4)_3$ (Schleid, Meyer, Oczko & Legendziewicz, 1991), and particularly by the title compound, $\text{Yb}(\text{ClO}_4)_3\cdot\text{H}_2\text{O}$.

The molecular structure of $\text{Yb}(\text{ClO}_4)_3\cdot\text{H}_2\text{O}$ comprises bidentate and tridentate perchlorato groups that form catenated rings including six Yb atoms (Yb—Yb distances range from 5.77 to 5.92 Å). These rings are linked to each other exclusively through tridentate groups to form large channels along the *c* axis that contain water molecules. The bridging bidentate