Table 2. *Bond distances* (Å) *and angles* (°) *in the* MoO_4^{2-} *anion with e.s.d.'s in parentheses*

Mo–O(1)	1·776 (3)	O(1)–Mo–O(2)	108·5 (2)
Mo–O(2)	1·757 (3)	O(1)–Mo–O(3)	108·9 (1)
Mo–O(3)	1·762 (2)	O(2)–Mo–O(3)	108·7 (1)
		O(3)–Mo–O(3^i)	113·2 (1)

Symmetry operation: (i) $x, \frac{1}{2}-y, z$.

1365 unique reflections ($R_{int} = 0·026$); structure solved by the heavy-atom method; H atoms located from difference Fourier synthesis; refinement (on F) performed by block-diagonal least-squares program in *UNICS*III (Sakurai & Kobayashi, 1979), using anisotropic thermal parameters for non-H atoms and isotropic thermal parameters for H atoms; final $R = 0·034$, $wR = 0·038$, $S = 1·018$, $(\Delta/\sigma)_{max} = 0·05$, $-0·86 < \Delta\rho < 0·56$ e Å^{-3}; $w^{-1} = \sigma^2(F_o) + (0·015\,|F_o|)^2$; complex scattering factors from *International Tables for X-ray Crystallography* (1974); all calculations on HITAC M-680H/M-682H and S-810 computers at the Computer Centre of the University of Tokyo. Final atomic parameters are presented in Table 1 and bond lengths and angles of the MoO_4^{2-} anion in Table 2. Fig. 1 shows a view of the unit cell.*

Related literature. In the K_2MoO_4 (Gatehouse & Leverett, 1969) and $Na_2MoO_4.2H_2O$ (Matsumoto, Kobayashi & Sasaki, 1975) crystals, the MoO_4^{2-} anion has one large O–Mo–O angle [112·7 (8) and 112·7 (3)°, respectively], while the other O–Mo–O angles are between 107·8 and 109·7°. The Mo–O distances in K_2MoO_4 and $Na_2MoO_4.2H_2O$ are 1·74–1·78 and 1·752–1·788 Å, respectively.

* Lists of structure factors, anisotropic thermal parameters and complete lists of bond lengths and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44193 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

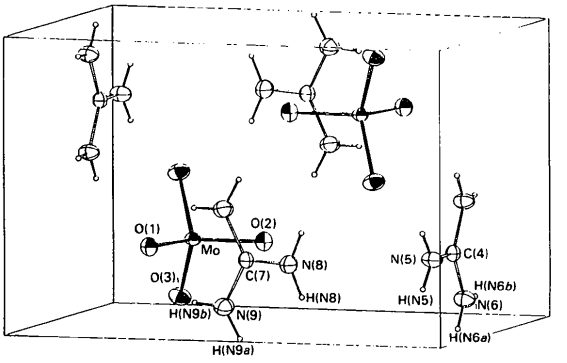

Fig. 1. An *ORTEP* (Johnson, 1976) view of the unit cell. Thermal ellipsoids are scaled to enclose 30% probability for non-H atoms.

References

GATEHOUSE, B. M. & LEVERETT, P. (1969). *J. Chem. Soc. A*, pp. 849–854.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

JOHNSON, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

MATSUMOTO, K. Y., KOBAYASHI, A. & SASAKI, Y. (1975). *Bull. Chem. Soc. Jpn*, **48**, 1009–1013.

SAKURAI, T. & KOBAYASHI, K. (1979). *Rikagaku Kenkyusho Hokoku*, **55**, 69–77.

Acta Cryst. (1987). C**43**, 2221–2222

Ammonium Nonamolybdenionickelate(IV) Hexahydrate

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Abstract. $(NH_4)_6[NiMo_9O_{32}].6H_2O$, $M_r = 1650·5$, trigonal, $R32$, $a = 15·932$ (2), $c = 12·413$ (2) Å, $V = 2728·7$ Å^3, $Z = 3$, $D_x = 3·013$, $D_m = 2·99$ (2) Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 3·52$ mm^{-1}, $F(000) = 2364$, $T = 291$ K, $R = 0·028$, $wR = 0·033$ for 592 observed reflections. The compound is isostructural with $(NH_4)_6[MnMo_9O_{32}].7$–$8H_2O$ [Allmann & D'Amour (1975). *Z. Kristallogr.* **141**, 342–353], except that N(2) is present as two adjacent half-atoms related by a crystal diad axis. The anion consists of one NiO_6 and nine MoO_6 octahedra sharing edges and has point symmetry 32. The Ni^{IV}–O bond length is 1·870 (6) Å.

Experimental. Preparation: Baker & Weakley (1966); red-black rhombohedra from water; D_m by flotation in $CHBr_3$–CHI_3; data crystal 0·25 × 0·25 × 0·25 mm.

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Table 1. *Final atomic coordinates* ($\times 10^4$) *and equivalent isotropic* (Ni, Mo) *or isotropic temperature factors* ($Å^2 \times 10^3$)

$$U_{eq} = \tfrac{1}{3}\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i . \mathbf{a}_j .$$

	x	y	z	U_{eq} or U_{iso}
Ni	0	0	0	9 (1)
Mo(1)	1998 (1)	0	0	14 (1)
Mo(2)	1390 (1)	867 (1)	2019 (1)	12 (1)
O(1)	636 (5)	−436 (4)	914 (5)	10 (1)
O(2)	2275 (5)	1143 (5)	888 (5)	15 (1)
O(3)	2341 (6)	−607 (5)	871 (6)	25 (2)
O(4)	1806 (6)	378 (6)	2946 (6)	24 (2)
O(5)	1739 (5)	1999 (5)	2517 (6)	20 (2)
O(6)	0	0	2607 (9)	16 (3)
N(1)	4426 (9)	0	0	35 (4)
N(2)*	2922 (12)	3077 (12)	219 (15)	21 (4)
Aq(1)	4048 (18)	5977 (18)	1553 (17)	72 (6)
Aq(2)	1273 (15)	−2749 (14)	1514 (16)	50 (5)

* Site half occupied.

Table 2. *Bond lengths and other interatomic distances* (Å) *and interbond angles* (°)

Ni–O(1)	1·870 (6)	Mo(2)–O(1^i)	2·189 (6)
Mo(1)–O(1)	2·230 (6)	Mo(2)–O(2)	1·880 (7)
Mo(1)–O(2)	1·980 (6)	Mo(2)–O(4)	1·700 (7)
Mo(1)–O(3)	1·712 (8)	Mo(2)–O(5)	1·715 (7)
Mo(2)–O(1)	2·266 (6)	Mo(2)–O(6)	2·070 (4)
Ni···Mo(1)	3·183 (2)	Mo(1)···Mo(2)	3·235 (2)
Ni···Mo(2)	3·167 (2)	Mo(2)···Mo(2^i)	3·355 (2)

O(1)–Ni–O(1^i)	87·0 (3)	O(2)–Mo(2)–O(4)	101·1 (3)
O(1)–Ni–O(1^{ii})	100·1 (4)	O(2)–Mo(2)–O(5)	102·3 (3)
O(1)–Ni–O(1^{iii})	86·7 (4)	O(2)–Mo(2)–O(6)	147·8 (3)
O(1)–Mo(1)–O(1^{iii})	70·3 (2)	O(4)–Mo(2)–O(5)	104·0 (4)
O(1)–Mo(1)–O(2)	74·8 (2)	O(4)–Mo(2)–O(6)	89·3 (3)
O(1)–Mo(1)–O(3)	92·2 (3)	O(5)–Mo(2)–O(6)	104·5 (3)
O(2)–Mo(1)–O(3)	100·4 (3)	Ni–O(1)–Mo(1)	101·5 (3)
O(3)–Mo(1)–O(3^{iii})	106·0 (5)	Ni–O(1)–Mo(2)	99·5 (3)
O(1)–Mo(2)–O(2)	75·8 (3)	Mo(1)–O(1)–O(2)	92·0 (3)
O(1)–Mo(2)–O(4)	98·0 (3)	Mo(1)–O(2)–Mo(2)	113·9 (4)
O(1)–Mo(2)–O(5)	157·8 (3)	Mo(2)–O(6)–Mo(2^i)	108·3 (3)
O(1)–Mo(1)–O(6)	72·7 (3)		

Probable hydrogen bonds

N(1)···O(3)	3·15 (2)	N(2)···O(2)	2·84 (2)
N(1)···O(3^{iv})	3·14 (2)	N(2)···O(2^{iv})	2·82 (2)
N(1)···O(4^v)	2·80 (2)	N(2)···O(5^{vii})	2·89 (2)
N(1)···O(5^{vi})	2·97 (2)	N(2)···Aq(2^{iii})	2·98 (3)
Aq(1)···O(4^{viii})	2·96 (3)	Aq(2)···O(3)	3·02 (3)
Aq(1)···O(4^{ix})	2·87 (3)	Aq(2)···O(5^x)	3·06 (3)
Aq(1)···Aq(2^i)	2·75 (4)		

Symmetry code: (i) $-y, x-y, z$; (ii) $y, y-x, -z$; (iii) $x-y, -y, -z$; (iv) $y, x, -z$; (v) $\frac{1}{3}-y, -\frac{1}{3}+x-y, -\frac{1}{3}+z$; (vi) $-\frac{1}{3}-y, \frac{1}{3}+x-y, \frac{1}{3}+z$; (vii) $\frac{1}{3}+y-x, -\frac{1}{3}-x, -\frac{1}{3}+z$; (viii) $\frac{1}{3}+x, \frac{2}{3}+y, -\frac{1}{3}+z$; (ix) $\frac{1}{3}+x-y, -\frac{1}{3}+y, \frac{2}{3}-z$; (x) $y-x, -x, z$.

CAD-4*F* diffractometer; cell parameters from 25 reflections in θ range 12–19°; data collected by ω–2θ scans in θ range 1–25°, h, k −18 to 18, l 0 to 14; 3270 reflections, 630 unique, R_{int}=0·035; three standard reflections, no decay; empirical absorption correction by ψ-scans based on $\bar{4}44$, $\bar{5}76$ and $\bar{4}56$. Positions of Ni, Mo from Patterson, confirmed by direct methods; N, O atoms from difference syntheses; full-matrix least-squares refinement on F, function minimized $\sum w(\Delta F)^2$, H atoms not located, all other atoms anisotropic, final $R = 0{\cdot}028$, $wR = 0{\cdot}033$ for 592 observed reflections [$|F| \geq 3\sigma(F)$], 53 parameters, $w = 1/\sigma^2(F)$, max. (Δ/σ) in last cycles 0·013, max. $\Delta\rho$ in final difference map 0·88 [at 0,0,0·5, possibly a fractional H_2O molecule], min. −1·19 e Å^{-3}. All calculations used *SHELX*76 (Sheldrick, 1976) with scattering factors from *International Tables for X-ray Crystallography* (1974). Atomic coordinates are listed in Table 1 and derived dimensions in Table 2. A view of the anion is shown in Fig. 1.*

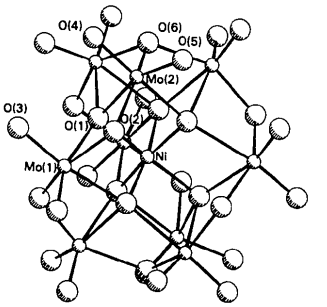

Fig. 1. The anion in $(NH_4)_6[NiMo_9O_{32}].6H_2O$. Atoms Ni and O(6) lie on a crystal triad axis, and Mo(1) on a diad.

Related literature. Bond lengths in $Ni^{IV}O_6$ octahedra in other polyoxometallate anions: $K_7[NiV_{13}O_{38}].18H_2O$. 1·82 (3), 1·89 (2) Å (Kobayashi & Sasaki, 1975); $(Na,K)_6[NiW_6O_{24}].12H_2O$, 1·978 (10) Å (av.) (Hau, 1970).

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44161 (6 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

ALLMANN, R. & D'AMOUR, H. (1975). *Z. Kristallogr.* **141**, 342–353.

BAKER, L. C. W. & WEAKLEY, T. J. R. (1966). *J. Inorg. Nucl. Chem.* **28**, 447–454.

HAU, H. H.-K. (1970). Doctoral Thesis, Boston Univ. *Diss. Abstr. Int.* B**31**, 2600–2601.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

KOBAYASHI, A. & SASAKI, Y. (1975). *Chem. Lett.* pp. 1123–1124.

SHELDRICK, G. M. (1976). *SHELX*76. *Program for crystal structure determination.* Univ. of Cambridge, England.