Fig. 1. Dessin de la structure vue selon [010] et noms des atomes. Les traits en pointillé représentent les liaisons hydrogène.

Howard-Lock, Lock, Lippert & Rosenberg, 1982). Structure du *cis*-diammine-*cis*-dichloro-*trans*-dihydroxoplatine(IV) mono(peroxyde d'hydrogène) (Vollano, Blatter & Dabrowiak, 1984). Structure de trois variétés isomères de diamminedichlorodihydroxoplatine(IV) (Kuroda, Neidle, Ismail & Sadler, 1983).

Références

FAGGIANI, R., HOWARD-LOCK, H. E., LOCK, C. J. L., LIPPERT, B. & ROSENBERG, B. (1982). *Can. J. Chem.* **60**, 529–534.

FRENZ, B. A. (1982). *SDP Structure Determination Package.* B. A. Frenz & Associates, Inc., College Station, Texas, Etats-Unis, et Enraf–Nonius, Delft, Pays-Bas.

International Tables for X-ray Crystallography (1974). Tome IV, pp. 99, 149. Birmingham: Kynoch Press. (Distributeur actuel D. Reidel, Dordrecht.)

KURODA, R., NEIDLE, S., ISMAIL, M. I. & SADLER, P. J. (1983). *Inorg. Chem.* **22**, 3620–3624.

VOLLANO, J. F., BLATTER, E. E. & DABROWIAK, J. C. (1984). *J. Am. Chem. Soc.* **106**, 2732–2733.

WALKER, N. & STUART, D. (1983). *Acta Cryst.* A**39**, 159–166.

Structure of Guanidinium Molybdate

BY TOMOJI OZEKI, HIKARU ICHIDA AND YUKIYOSHI SASAKI

Department of Chemistry, Faculty of Science, The University of Tokyo, Tokyo 113, Japan

Abstract. $2CH_6N_3^+.MoO_4^{2-}$, $M_r = 280 \cdot 12$, monoclinic, $P2_1/m$, $a = 10 \cdot 657$ (2), $b = 7 \cdot 006$ (1), $c = 6 \cdot 567$ (2) Å, $\beta = 98 \cdot 12$ (2)°, $U = 485 \cdot 4$ (2) Å^3, $Z = 2$, $D_x = 1 \cdot 91$ Mg m^{-3}, $\lambda(Mo\ K\alpha) = 0 \cdot 71069$ Å, $\mu = 1 \cdot 32$ mm^{-1}, $F(000) = 432$, $T = 298$ K, $R = 0 \cdot 034$ and $wR = 0 \cdot 038$ for 1365 independent reflections with $F_o > 2 \cdot 5\sigma(F_o)$. The distances between O and Mo atoms increase with the number of interacting H atoms: $1 \cdot 757$ (3), $1 \cdot 762$ (3) and $1 \cdot 776$ (3) Å for the O atoms interacting with two, three and four H atoms, respectively. These H atoms lie between $2 \cdot 03$ (4) and $2 \cdot 30$ (4) Å from the O atoms. The O(3)—Mo—O(3^i) angle is $113 \cdot 2$ (1)°, whereas the other three O—Mo—O angles are $108 \cdot 5$ (2) to $108 \cdot 9$ (1)°. The mean distances for the C—N and N—H bonds in the guanidinium cations are $1 \cdot 325$ (6) and $0 \cdot 83$ (5) Å, respectively.

Experimental. The title compound was obtained as colorless tabular crystals by slowly evaporating a 10 ml aqueous solution of $2 \cdot 0$ g $Na_2MoO_4.2H_2O$ and $1 \cdot 5$ g $(CH_6N_3)_2CO_3$. Crystal $0 \cdot 55 \times 0 \cdot 13 \times 0 \cdot 05$ mm; Rigaku AFC-5R diffractometer on RU-1000 X-ray generator of High Intensity X-ray Facilities, Engineering Research Institute, Faculty of Engineering, The University of Tokyo; Mo $K\alpha$ radiation (graphite monochromatized); ω–2θ scan at 8° min$^{-1}(\omega)$; Lorentz–

Table 1. *Positional parameters* ($\times 10^4$; $\times 10^5$ *for* Mo; $\times 10^3$ *for* H) *and thermal parameters* (Å^2; B_{eq} *for non*-H *atoms*; B_{iso} *for* H *atoms*)

$$B_{eq} = \tfrac{8}{3}\pi^2\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}/B_{iso}
Mo	23782 (4)	25000	22645 (5)	1·68 (1)
O(1)	2022 (3)	2500	4826 (4)	2·34 (7)
O(2)	4034 (3)	2500	2352 (6)	2·95 (9)
O(3)	1765 (2)	400 (3)	1023 (3)	2·79 (6)
C(4)	8827 (4)	2500	4131 (6)	1·80 (9)
N(5)	8853 (4)	2500	6148 (6)	2·59 (10)
N(6)	8835 (3)	853 (4)	3137 (4)	2·52 (6)
C(7)	5008 (4)	2500	7895 (7)	2·22 (10)
N(8)	6201 (4)	2500	8748 (8)	3·11 (11)
N(9)	4395 (3)	857 (4)	7484 (5)	2·85 (7)
H(N5)	877 (4)	156 (6)	671 (6)	3·6 (9)
H(N6a)	881 (3)	−7 (6)	369 (5)	2·7 (8)
H(N6b)	874 (4)	79 (7)	179 (6)	4·4 (10)
H(N8)	652 (4)	141 (6)	882 (6)	4·3 (10)
H(N9a)	477 (3)	−18 (6)	757 (5)	3·5 (9)
H(N9b)	358 (4)	90 (7)	694 (6)	4·6 (10)

polarization corrections; absorption correction not applied; lattice constants by least squares from 84 reflections $(12 < \theta < 16°)$; $(\sin\theta)/\lambda < 0 \cdot 7035$ Å^{-1} $(-14 \leq h \leq 14, -9 \leq k \leq 9, 0 \leq l \leq 9)$; three standard reflections monitored every 100 reflections varied within $\pm 1 \cdot 5\%$; of the 3046 measured reflections, 2663 reflections with $F_o > 2 \cdot 5\sigma(F_o)$ were averaged to give

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$_2$MoO$_4$ (Gatehouse & Leverett, 1969) and Na$_2$MoO$_4$.2H$_2$O (Matsumoto,

---

* 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.
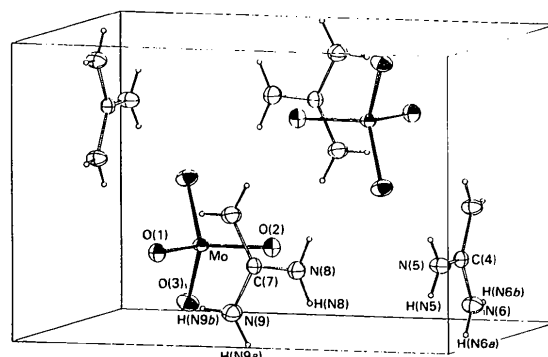

Fig. 1. An *ORTEP* (Johnson, 1976) view of the unit cell. Thermal ellipsoids are scaled to enclose 30% probability for non-H atoms.

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$_2$MoO$_4$ and Na$_2$MoO$_4$.2H$_2$O are 1·74–1·78 and 1·752–1·788 Å, respectively.

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.

Ammonium Nonamolybdenionickelate(IV) Hexahydrate

BY TIMOTHY J. R. WEAKLEY

Chemistry Department, Dundee University, Dundee DD1 4HN, Scotland

Abstract. (NH$_4$)$_6$[NiMo$_9$O$_{32}$].6H$_2$O, $M_r = 1650.5$, 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$_9$O$_{32}$].7–8H$_2$O [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.