technetate. ions. There is no evidence of any strong interactions and all intermolecular contacts are greater than the van der Waals distances.

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The Structure of Ammonium Decamolybdate $(NH_4)_8Mo_{10}O_{34}$

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Abstract. $(NH_4)_8Mo_{10}O_{34}$, $M_r = 1647·7$, triclinic, $P\bar{1}$, $a = 7·750$ (1), $b = 10·889$ (1), $c = 11·038$ (1) Å, $\alpha = 73·13$ (1), $\beta = 80·82$ (1), $\gamma = 81·71$ (1)°, $V = 875·25$ Å^3, $Z = 1$, $D_x = 3·126$, $D_m = 3·12$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·7107 Å, $\mu = 3·51$ mm^{-1}, $F(000) = 780$, room temperature, final $R = 0·051$ for 3642 independent reflections. The title compound is formed by reaction between molybdenum trioxide and aqueous ammonia solution. The anionic asymmetric unit, Mo_5O_{17}, contains one MoO_4 tetrahedron connected to one of four MoO_6 octahedra by sharing of one common corner. The Mo_4O_{14} unit is built up of four edge-bridge condensed octahedra with seven terminal O atoms. Linkage of the asymmetric unit with its centrosymmetric counterpart gives rise to the $Mo_{10}O_{34}^{8-}$ anion. The overall structural framework is built up by a number of close contacts of the cation N atoms with the anion terminal oxygens. The metal–oxygen bond lengths conform with those of similar compounds.

Introduction. Molybdenum chemistry research has been given considerable attention because of the widespread versatility and the varied industrial applications of Mo compounds (Mitchell, 1973; Stiefel, 1977; Braithwaite, 1978). Ammonium molybdates are compounds of great interest for applications demanding high-purity molybdenum trioxide. Several structures have been determined for compounds formed in the ternary system NH_3–MoO_3–H_2O (Böschen, Buss & Krebs, 1974; Evans, Gatehouse & Leverett, 1975; Armour, Drew & Mitchell, 1975; Vivier, Bernard & Djomaa, 1977; Garin & Blanc, 1985). The anion

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Table 1. *Atomic coordinates and equivalent isotropic thermal parameters for non-hydrogen atoms*

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

	x	y	z	B_{eq}(Å^2)
Mo(1)	0·9220 (3)	0·1707 (2)	0·2541 (2)	1·00
Mo(2)	0·8181 (2)	0·7230 (2)	0·0765 (2)	0·78
Mo(3)	0·8081 (2)	0·4258 (2)	0·0242 (2)	0·63
Mo(4)	0·8948 (3)	0·4627 (2)	0·3097 (2)	0·89
Mo(5)	0·6117 (3)	0·1608 (2)	0·5488 (2)	1·11
O(1)	0·749 (1)	0·360 (1)	0·201 (1)	1·17
O(2)	0·802 (2)	0·140 (2)	0·426 (1)	1·58
O(3)	1·037 (1)	0·317 (1)	0·309 (1)	0·91
O(4)	1·018 (1)	0·272 (1)	0·079 (1)	1·04
O(5)	1·105 (2)	0·059 (1)	0·277 (2)	1·98
O(6)	0·782 (2)	0·101 (1)	0·197 (1)	1·49
O(7)	0·987 (1)	0·539 (1)	0·108 (1)	0·74
O(8)	0·644 (2)	0·829 (1)	0·011 (1)	1·21
O(9)	0·927 (2)	0·819 (1)	0·131 (1)	1·89
O(10)	0·701 (1)	0·583 (1)	−0·009 (1)	1·07
O(11)	0·724 (1)	0·617 (1)	0·228 (1)	1·20
O(12)	0·694 (2)	0·345 (1)	−0·041 (2)	1·76
O(13)	0·740 (2)	0·416 (1)	0·439 (1)	1·54
O(14)	1·024 (2)	0·555 (1)	0·353 (1)	1·66
O(15)	0·679 (2)	0·210 (2)	0·667 (1)	2·27
O(16)	0·433 (2)	0·264 (2)	0·482 (1)	2·51
O(17)	0·534 (2)	0·016 (1)	0·621 (1)	1·99
N(1)	0·590 (2)	−0·232 (2)	0·776 (2)	2·04
N(2)	0·094 (2)	0·199 (2)	0·592 (2)	2·91
N(3)	0·603 (2)	0·475 (2)	−0·325 (2)	1·81
N(4)	0·730 (2)	0·091 (2)	0·922 (2)	1·67

Table 2. *Interatomic distances* (Å) *and angles* (°) *for* $(NH_4)_8Mo_{10}O_{34}$

MoO_6 octahedra			
Mo(1)–O(1)	2·267 (9)	Mo(3)–O(1)	1·879 (10)
Mo(1)–O(2)	1·934 (11)	Mo(3)–O(4)	2·181 (9)
Mo(1)–O(3)	2·200 (12)	Mo(3)–O(7^{i})	1·978 (8)
Mo(1)–O(4)	2·012 (9)	Mo(3)–O(10)	1·754 (10)
Mo(1)–O(5)	1·730 (13)	Mo(3)–O(12)	1·693 (15)
Mo(1)–O(6)	1·698 (15)	Mo(3)–O(7)	2·420 (12)
Mo(2)–O(4^{i})	1·959 (10)	Mo(4)–O(1)	2·354 (12)
Mo(2)–O(7)	2·198 (9)	Mo(4)–O(3)	1·798 (10)
Mo(2)–O(8)	1·747 (13)	Mo(4)–O(7)	2·180 (10)
Mo(2)–O(9)	1·717 (15)	Mo(4)–O(11)	2·061 (9)
Mo(2)–O(10)	2·355 (12)	Mo(4)–O(13)	1·717 (12)
Mo(2)–O(11)	1·843 (9)	Mo(4)–O(14)	1·721 (15)
O(1)–Mo(1)–O(2)	88·6 (6)	O(1)–Mo(3)–O(4)	76·4 (4)
O(1)–Mo(1)–O(3)	71·6 (4)	O(1)–Mo(3)–O(7)	78·2 (4)
O(1)–Mo(1)–O(4)	71·9 (4)	O(1)–Mo(3)–O(10)	103·6 (5)
O(1)–Mo(1)–O(6)	88·9 (5)	O(1)–Mo(3)–O(12)	104·4 (5)
O(2)–Mo(1)–O(3)	82·9 (7)	O(4)–Mo(3)–O(7^{i})	72·2 (3)
O(2)–Mo(1)–O(5)	101·1 (8)	O(4)–Mo(3)–O(7)	80·8 (4)
O(2)–Mo(1)–O(6)	96·5 (7)	O(4)–Mo(3)–O(12)	97·3 (5)
O(3)–Mo(1)–O(4)	81·4 (4)	O(7)–Mo(3)–O(7^{i})	75·8 (4)
O(3)–Mo(1)–O(5)	94·2 (6)	O(7^{i})–Mo(3)–O(10)	97·4 (4)
O(4)–Mo(1)–O(5)	95·3 (6)	O(7^{i})–Mo(3)–O(12)	100·8 (6)
O(4)–Mo(1)–O(6)	93·3 (5)	O(10)–Mo(3)–O(7)	77·2 (4)
O(5)–Mo(1)–O(6)	105·0 (7)	O(10)–Mo(3)–O(12)	104·4 (5)
O(1)–Mo(1)–O(5)	161·9 (5)	O(1)–Mo(3)–O(7^{i})	141·8 (4)
O(2)–Mo(1)–O(4)	158·0 (7)	O(4)–Mo(3)–O(10)	157·5 (5)
O(3)–Mo(1)–O(6)	160·6 (5)	O(7)–Mo(3)–O(12)	176·4 (5)
O(4^{i})–Mo(2)–O(7)	72·2 (4)	O(1)–Mo(4)–O(3)	76·8 (4)
O(4^{i})–Mo(2)–O(8)	99·7 (5)	O(1)–Mo(4)–O(7)	74·4 (4)
O(4^{i})–Mo(2)–O(9)	95·6 (5)	O(1)–Mo(4)–O(11)	81·0 (4)
O(4^{i})–Mo(2)–O(10)	78·8 (4)	O(1)–Mo(4)–O(13)	87·5 (5)
O(7)–Mo(2)–O(9)	103·8 (5)	O(3)–Mo(4)–O(7)	88·7 (4)
O(7)–Mo(2)–O(10)	71·3 (4)	O(3)–Mo(4)–O(13)	103·8 (5)
O(7)–Mo(2)–O(11)	73·5 (4)	O(3)–Mo(4)–O(14)	103·2 (5)
O(8)–Mo(2)–O(9)	103·0 (6)	O(7)–Mo(4)–O(11)	70·0 (4)
O(8)–Mo(2)–O(10)	81·6 (5)	O(7)–Mo(4)–O(14)	91·9 (5)
O(8)–Mo(2)–O(11)	106·7 (5)	O(11)–Mo(4)–O(13)	90·7 (5)
O(9)–Mo(2)–O(11)	101·1 (5)	O(11)–Mo(4)–O(14)	94·7 (5)
O(10)–Mo(2)–O(11)	82·1 (5)	O(13)–Mo(4)–O(14)	105·6 (6)
O(4^{i})–Mo(2)–O(11)	144·5 (5)	O(1)–Mo(4)–O(14)	166·3 (4)
O(7)–Mo(2)–O(8)	152·6 (6)	O(3)–Mo(4)–O(11)	152·7 (5)
O(9)–Mo(2)–O(10)	173·3 (5)	O(7)–Mo(4)–O(13)	155·2 (5)
MoO_4 tetrahedron			
Mo(5)–O(2)	1·874 (3)	Mo(5)–O(16)	1·766 (15)
Mo(5)–O(15)	1·721 (18)	Mo(5)–O(17)	1·696 (11)
O(2)–Mo(5)–O(15)	110·5 (7)	O(15)–Mo(5)–O(16)	112·0 (9)
O(2)–Mo(5)–O(16)	112·8 (6)	O(15)–Mo(5)–O(17)	106·2 (7)
O(2)–Mo(5)–O(17)	109·2 (8)	O(16)–Mo(5)–O(17)	105·8 (8)
Possible hydrogen bonds			
N(1)–O(10)	2·793 (21)	N(1)–O(17)	2·761 (21)
N(2)–O(16)	2·811 (22)	N(3)–O(11)	2·797 (18)
N(4)–O(15)	2·801 (23)		
Shortest NH_4^+–NH_4^+ distance			
N(1)–N(3)	3·656 (34)		

structure of ammonium decamolybdate has been briefly described in earlier literature (Fuchs, Hartl, Hunnius & Mahjour, 1975). It was found that isolated decamolybdate ions are built up by connecting one Mo_8O_{28} unit to corners of two MoO_4 tetrahedra. However, atom positions and thermal parameters, as well as cation distribution, were not given. We now report the complete structure determination of the title compound.

Experimental. Powder and single crystals of ammonium decamolybdate were synthesized by heating at 353 K a mixture of MoO_3, NH_3 and distilled water in molar weight ratio, contained in a sealed glass vessel. Compound synthesis confirmed by usual analysis techniques. Lattice parameters refined from 32 diffractometer-centered reflections with conventional least-squares procedure. Nearly spherical polyhedral crystal 0·1 mm diameter, Philips PW 1100 automatic four-circle diffractometer, graphite monochromator; θ–2θ scan, scan speed 3·0° min^{-1}, $\theta_{max} = 30°$, $[(\sin\theta)/\lambda]_{max} = 0{\cdot}703$ Å^{-1}, $0 \le h \le 10$, $-12 \le k \le 12$, $-12 \le l \le 12$; 4794 independent reflections measured, 3642 observed with $I > 4\sigma(I)$, 986 unobserved, 166 rejected. Two strong standards (340, 233) measured every 240 reflections, their intensity variation within $\pm 1{\cdot}3\%$ during complete run. Correction for Lorentz, polarization and spherical absorption effects (transmission factors 0·391 to 0·446). Statistical tests based on $|E|$ values (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980) indicated centrosymmetric distribution of atoms; the same was observed from $N(z)$ test (Howells, Phillips & Rogers, 1950); Mo-atom positions from direct methods. N and O from difference Fourier synthesis; criterion of minimum ammonium–ammonium distance of 3·7 Å (Seimons & Templeton, 1954) to fix N atoms. 255 variables refined using F values; $R = 0{\cdot}051$, $wR = 0{\cdot}053$, $w = [\sigma^2(F_o) + 0{\cdot}011122F_o^2]^{-1}$, ten strong reflections down-weighted because of extinction effects, $S = 5{\cdot}4$, final max. $\Delta/\sigma = 0{\cdot}018$; atomic scattering factors and anomalous-dispersion corrections from *International Tables for X-ray Crystallography* (1974). All calculations were carried out with *SHELX*76 (Sheldrick, 1976).

Discussion. Atomic coordinates and equivalent isotropic thermal parameters are given in Table 1.* Table 2 lists the corresponding interatomic distances and angles for the MoO_6 and MoO_4 polyhedra. Figs. 1 and 2 respectively show an *ORTEP* plot (Johnson, 1976) and the coordination polyhedra of the asymmetric unit of the anion, $Mo_5O_{17}^{4-}$, projected along **c**. The main structural characteristic of the compound is the presence of both tetrahedral and octahedral coordination of Mo atoms. This is consistent with the well known fact that the π component of the Mo^{VI}—O bond stabilizes Mo^{VI} in varied oxygen coordinations (Goodenough, 1982).

The asymmetric unit consists of one MoO_4 tetrahedron and four MoO_6 octahedra. The tetrahedron is connected to one octahedron by sharing of one common corner, O(2). The remaining three oxygen atoms, O(15), O(16) and O(17), are unshared by other polyhedra. The Mo_4O_{14} unit, on the other side, is built up of four edge-bridge condensed octahedra with seven terminal O atoms. This arrangement has therefore a total of ten terminal oxygens with Mo—O bond lengths found in the range of 1·696 to 1·766 Å. The presence of terminal (O_t) and bridging (O_b) oxygens is consistent with the infrared spectrum, *i.e.* three strong bands located at 630, 650 and 700 cm^{-1} indicate Mo—O_b—Mo vibrations, while bands at 900 and 940 cm^{-1} correspond to Mo—O_t vibrations; a wide and intense band at 850 cm^{-1} takes into account the remaining Mo—O stretching frequencies in the crystal.

Two Mo_5O_{17} units are connected through O(4) and O(7), thus forming a centrosymmetric anion, $Mo_{10}O_{34}^{8-}$, as depicted in Fig. 3. The metal–oxygen distances in the tetrahedron vary from 1·696 to 1·874 Å, while for the octahedral coordination they span a larger range, from 1·693 to 2·420 Å. The observed distances and polyhedral distortions are typical of molybdenum(VI) oxides (Schröder, 1975). Mo atoms in octahedral sites show a general type of distortion typical of octamolybdate anions (Wilson, McKee, Penfold & Wilkins, 1984), *i.e.* the shortening of two *cis* O—Mo bonds with a consequential lengthening of the remaining four molybdenum–oxygen interactions. On the other hand, Mo in the tetrahedral site is bonded to three terminal oxygens with short Mo—O distances, while linkage to the octahedron through bridging O(2) exhibits bond lengthening.

Finally, the NH_4^+ cations preferentially occupy sites near the terminal O atoms of the tetrahedra, with a minimum ammonium–ammonium distance of 3·677 Å, which satisfies the shortest-distance criterion for cation location.

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44675 (19 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

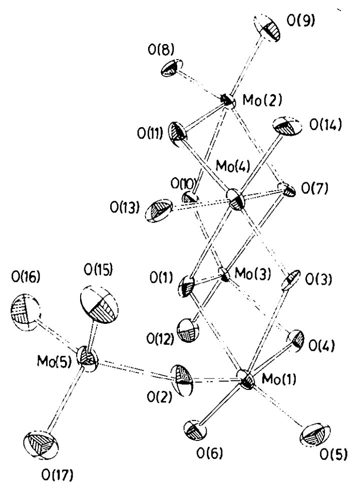

Fig. 1. *ORTEP* plot (Johnson, 1976) of the asymmetric unit, Mo_5O_{17}. The thermal ellipsoids are at 50% probability.

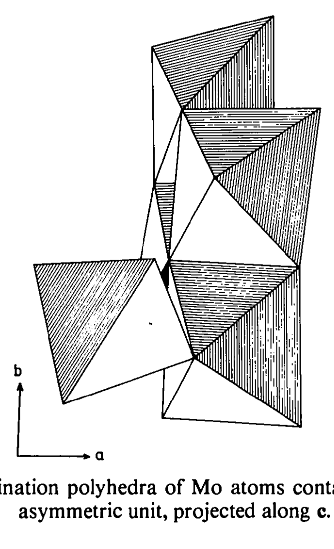

Fig. 2. Coordination polyhedra of Mo atoms contained within the asymmetric unit, projected along **c**.

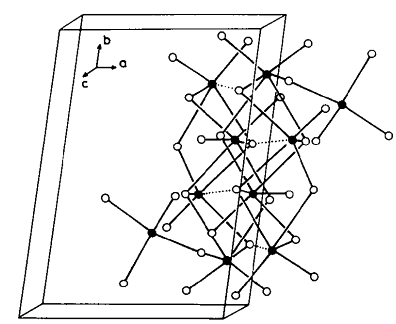

Fig. 3. The $Mo_{10}O_{34}^{8-}$ anion in the unit cell. Filled circles represent Mo atom, open circles O atoms and dotted lines linkage between centrosymmetrically related units.

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Distrontium Diantimonate(V). A Rietveld Refinement of Neutron Powder Diffraction Data

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Abstract. $Sr_2Sb_2O_7$: $M_r = 530.74$, orthorhombic, *Imma*. At $T = 300$ K: $a = 7.4557$ (2), $b = 10.3708$ (3), $c = 7.6860$ (1) Å, $V = 594.29$ (3) Å^3, $Z = 4$, $D_x = 5.932$ (2) Mg m^{-3}, $\mu R = 0.12$, $\lambda = 2.5804$ (1) Å, $R_I = 1.79$, $R_p = 3.48$, $R_{wp} = 4.59$%. The structure has been refined by Rietveld analysis of neutron powder diffraction data recorded at room temperature for 85 reflections. The structure is of the weberite type.

Introduction. The antimonates $Ca_2Sb_2O_7$ and $Cd_2Sb_2O_7$ crystallize with structures of both the pyrochlore and the weberite type, but $Sr_2Sb_2O_7$ is known only as a weberite (Knop, Demazeau & Hagenmuller, 1980). In relation to $Ba_2U_2O_7$ (Cordfunke & IJdo, 1988) with weberite structure and space group *Imma*, it was desirable to know the structure of $Sr_2Sb_2O_7$ in detail. There is some confusion about the space group of $Sr_2Sb_2O_7$. Bystrom (1945) reports its space group as *Imm*2 ($a = 7.28$, $b = 7.44$, $c = 10.18$ Å), Burchard & Rudorff (1979) as *Imnm*. Giuseppetti & Tadini (1978) report for the weberite Na_2MgAlF_7 the space group *Imma*. Knop, Cameron & Jochem (1982) state that descriptions of structures of weberites in space groups *Imm*2, $I2_12_12_1$ and *Imma* must be regarded as practically indistinguishable.

Experimental. AR starting materials $SrCO_3$ and Sb_2O_3 were thoroughly mixed in an agate mortar in the approximate ratios. The sample was heated in a platinum crucible at 1173 K for 7 days and at 973 K for 2 days. X-ray powder diffraction patterns were obtained using a Philips PW 1050 diffractometer. The systematic extinctions indicate space group *Imma* or *Im*2*a*. Since no single crystals were available, Rietveld's (1969) method was used for the refinement of neutron powder diffraction data; neutron powder profile recorded at the Petten High-Flux Reactor; $5 < 2\theta < 163°$ in steps of 0·1°; neutrons at 295 K from the (111) planes of a Cu crystal; pyrolytic graphite with a total thickness of 120 mm as a second-order filter; Soller slits, horizontal divergence 0·5°, placed between the reactor and the monochromator and in front of the four ^{3}He counters; sample holder (∅ = 14·46 mm) consisted of a 'V' tube, closed with Cu plugs with 'O' rings. No

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