The First Observation of γ-Octamolybdate: Synthesis, Crystal and Molecular Structure of $[Me_3N(CH_2)_6NMe_3]_2[Mo_8O_{26}]\cdot 2H_2O$ *

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The compound $[Me_3N(CH_2)_6NMe_3]_2[Mo_8O_{26}]\cdot 2H_2O$ crystallizes from an aqueous solution of sodium molybdate and $[Me_3N(CH_2)_6NMe_3]Cl_2$, slightly acidified with hydrochloric acid. The crystals are monoclinic, space group $P2_1/n$ with $a = 12.42(1)$, $b = 12.897(3)$, $c = 15.74(1)$ Å, $\beta = 106.41(5)°$ and $Z = 2$. The structure was determined by direct methods and refined by least squares to $R = 0.030$ for 3153 observed reflections and 315 parameters. The $[Mo_8O_{26}]^{4-}$ anion is composed of six MoO_6 octahedra interlinked along edges and two MoO_5 trigonal bipyramids each sharing two edges with the octahedra. This is different from the previously observed α and β isomers of $[Mo_8O_{26}]^{4-}$ and lends credence to one of the proposed mechanisms for their interconversion.

The polyoxoanions of molybdenum may be considered analogous to metal oxide surfaces, and as such have attracted interest as catalysts.[1] They are also used as spectroanalytical reagents and as imaging agents for electron microscopy.[1,2] There is further academic interest in their chemistry because of their complex structural and reactivity patterns and the interconversions among these large anions in solution.[1] A particularly intriguing example is the octamolybdate $[Mo_8O_{26}]^{4-}$ which has been observed as two isomers, α and β, each of which can be viewed as distorted arrays of cubic close-packed (c.c.p.) oxygen atoms with molybdenum atoms occupying interstitial sites.

Crystal structures of the α form have been reported for tetrabutylammonium octamolybdate $[NBu_4]_4[Mo_8O_{26}]$[3] and $[PPrPh_3]_4[Mo_8O_{26}]\cdot H_2O\cdot MeCN$.[4] As shown in Fig. 1(*a*) the α form has approximate D_{3d} symmetry and consists of a ring made up of six MoO_6 octahedra (as in $[TeMo_6O_{24}]^{6-}$)[5] linked to one MoO_4 tetrahedron above and another below its octahedral cavity. A large variation in Mo–O distances allows the structure to be viewed as a loose addition compound between two $MoO_4{}^{2-}$ units and a (neutral) Mo_6O_{18} ring made up of distorted MoO_4 tetrahedra, sharing corners.[6] The β form comprising eight edge-sharing MoO_6 octahedra as shown in Fig. 1(*b*) has been observed for $[NH_4]_4[Mo_8O_{26}]\cdot nH_2O$ ($n = 4$ or 5),[7] $[NMe_4]_2Na_2[Mo_8O_{26}]\cdot 2H_2O$, incorrectly reported as $[NMe_4]_2[Mo_8O_{25}]\cdot 5H_2O$,[8] $[NBu_4]_3K[Mo_8O_{26}]\cdot 2H_2O$,[9] $[NH_2Me_2]_4[Mo_8O_{26}]$,[10] $[Hepy]_4[Mo_8O_{26}]$ (epy = 3-ethylpyridine)[11] and $[C_5H_9N_2]_4[Mo_8O_{26}]\cdot 4H_2O$.[12] The β form of the octamolybdate may be represented by two $(O^{2-})(Mo_4O_{12})$ since these subunits can be obtained by breaking only bonds whose lengths exceed 2.22 Å and hence have bond orders less than 0.2.[13]

A relatively facile isomerization process in which the structurally disparate α and β isomers are interconverted has been reported to occur in acetonitrile for α-$[NBu_4]_4[Mo_8O_{26}]$ and β-$[NBu_4]_3K[Mo_8O_{26}]\cdot 2H_2O$.[9] The authors proposed a mechanism for the rapid isomerization of the α and β forms *via* a γ intermediate which has six MoO_6 octahedra and two MoO_5 trigonal bipyramids, shown as an idealized structure in Fig. 2. An alternative isomerization mechanism based upon concerted

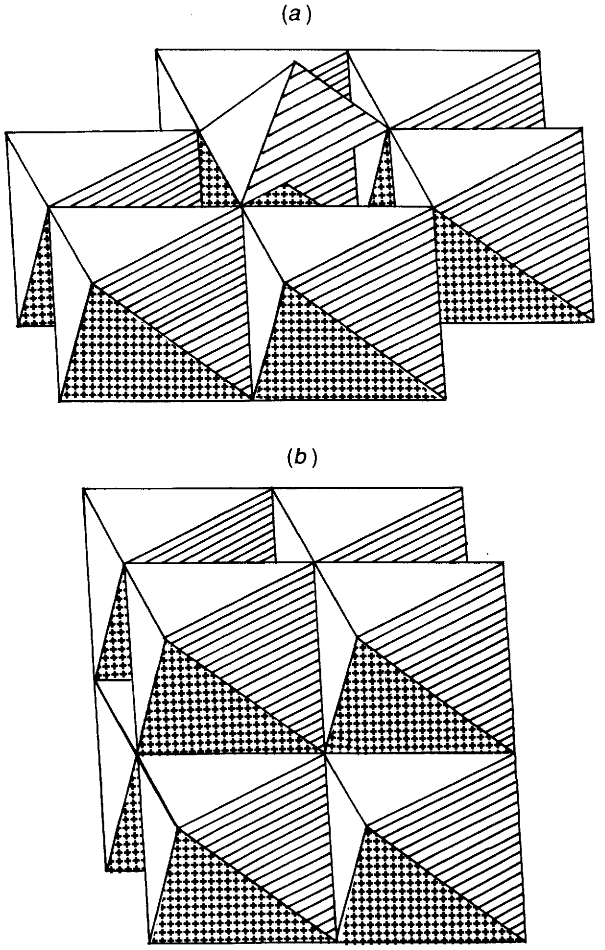

Fig. 1 Idealized molecular structures of (*a*) α- and (*b*) β-$[Mo_8O_{26}]^{4-}$, showing the molybdenum–oxygen polyhedra

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* *Supplementary data available:* see Instructions for Authors, *J. Chem. Soc., Dalton Trans.*, 1991, Issue 1, pp. xviii–xxii.

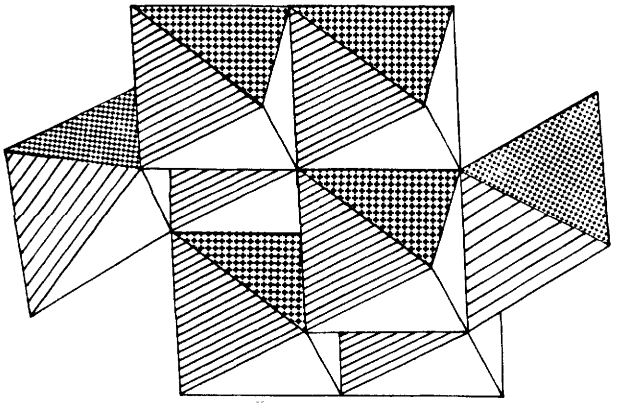

Fig. 2 Idealized molecular structure of γ-$[Mo_8O_{26}]^{4-}$, showing the molybdenum–oxygen octahedra and trigonal bipyramids

Table 1 Fractional atomic coordinates ($\times 10^4$) with estimated standard deviations (e.s.d.s) in parentheses for $[Me_3N(CH_2)_6NMe_3]_2$-$[Mo_8O_{26}]\cdot 2H_2O$

Atom	*X*/*a*	*Y*/*b*	*Z*/*c*
Mo(1)	1 068(0)	4 261(0)	5 662(0)
Mo(2)	1 756(0)	6 278(0)	4 555(0)
Mo(3)	−1 530(0)	6 399(0)	6 287(0)
Mo(4)	602(0)	7 557(0)	5 854(0)
O(1)	−2 354(3)	5 816(3)	3 596(3)
O(2)	−960(3)	6 928(3)	5 049(3)
O(3)	−807(3)	4 291(3)	4 541(2)
O(4)	−128(3)	5 985(3)	3 720(3)
O(5)	2 004(4)	6 506(4)	3 560(3)
O(6)	1 048(3)	7 514(3)	4 776(3)
O(7)	3 022(3)	6 436(3)	5 312(3)
O(8)	−1 787(3)	5 281(3)	5 475(3)
O(9)	−2 588(4)	7 285(4)	6 053(3)
O(10)	−1 663(5)	5 763(4)	7 200(3)
O(11)	−141(4)	6 984(4)	6 720(3)
O(12)	1 902(4)	7 483(4)	6 595(3)
O(13)	235(5)	8 817(4)	5 850(4)
N(1)	1 667(4)	5 215(4)	9 075(3)
C(11)	1 542(7)	5 075(7)	8 103(5)
C(12)	1 509(7)	4 160(5)	9 453(6)
C(13)	2 814(5)	5 621(6)	9 548(5)
C(1)	752(5)	5 913(5)	9 214(4)
C(2)	817(5)	7 041(5)	8 968(4)
C(3)	−138(6)	7 612(5)	9 193(5)
C(4)	−49(6)	8 782(5)	9 155(4)
C(5)	−204(6)	9 230(5)	8 235(4)
C(6)	−196(6)	10 408(5)	8 332(4)
N(2)	−177(4)	11 044(4)	7 543(3)
C(21)	−1 159(6)	10 804(7)	6 794(5)
C(22)	−175(8)	12 144(5)	7 810(6)
C(23)	884(7)	10 858(7)	7 305(6)
O(31)	6 506(5)	9 185(4)	6 465(4)

translations of close-packed fragments along molecular mirror planes has also been proposed.[14]

We report here the isolation of a new octamolybdate $[Me_3N(CH_2)_6NMe_3]_2[Mo_8O_{26}]\cdot 2H_2O$ and the results of its crystallographic analysis which allow us to identify it as containing the γ form of the octamolybdate.

Results and Discussion

Final fractional atomic coordinates for the non-hydrogen atoms are reported in Table 1, and all bond lengths and angles in Tables 2 and 3 respectively. A projection of the entire structure down (100) is illustrated in Fig. 3. The asymmetric unit comprises four Mo and thirteen O atoms of the anion, one cation $[Me_3N(CH_2)_6NMe_3]^{2+}$ and one water of crystallization. The Mo_4O_{13} moiety is adjacent to a centre of symmetry which generates the other half of the anion which therefore has C_i symmetry, and allows the general formulation of the compound as $[Me_3N(CH_2)_6NMe_3]_2[Mo_8O_{26}]\cdot 2H_2O$.

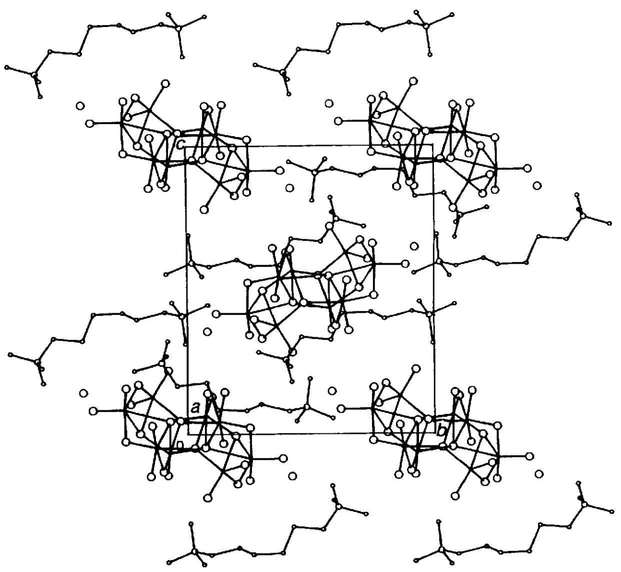

Fig. 3 Packing diagram of the structure showing the anions, cations and water of crystallization

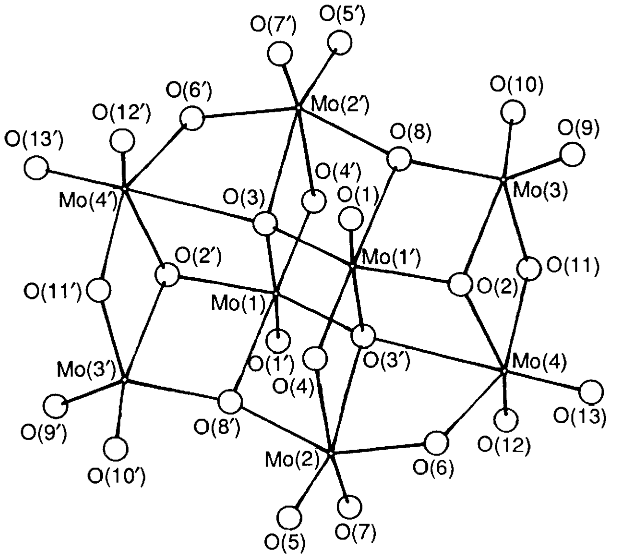

Fig. 4 Perspective view of the $[Mo_8O_{26}]^{4-}$ anion with atomic numbering

The $[Mo_8O_{26}]^{4-}$ *Anion.*—The anion, with atomic numbering, is illustrated in Fig. 4. Small circles represent molybdenums and large circles oxygens. Nuclei with primed labels are related to nuclei with the same label, unprimed, by inversion symmetry. There are six edge-sharing MoO_6 octahedra and two five-co-ordinate MoO_5 trigonal bipyramids. Each MoO_6 octahedron [Mo(1), Mo(2) and Mo(4)] has two short *cis*-, two medium *trans*- and two long *cis*-Mo–O bonds in the ranges 1.687(5)–1.747(5) Å for short, 1.881(4)–2.013(4) Å for medium and 2.154(4)–2.495(4) Å for long bonds. Within the specified ranges, the trigonal bipyramids Mo(3) have two short, two medium and one long Mo–O bond. As expected, all the terminal Mo–O distances are in the short range, which is indicative of double bonding.[15] There is one other short and therefore doubly bonded Mo–O, namely Mo(1)–O(4′), 1.747(5) Å, for which the oxygen is two-co-ordinate, bridging between Mo(1) and Mo(2′). Hence each molybdenum has two Mo=O double

Table 2 Bond lengths (Å) with e.s.d.s in parentheses for $[Me_3N(CH_2)_6NMe_3]_2[Mo_8O_{26}]\cdot 2H_2O$

Mo(1)–O(3)	2.494(4)	Mo(3)–O(2)	2.356(5)
Mo(1)–O(1′)	1.694(4)	Mo(3)–O(8)	1.893(4)
Mo(1)–O(2′)	1.881(4)	Mo(3)–O(9)	1.701(5)
Mo(1)–O(3′)	1.907(4)	Mo(3)–O(10)	1.701(5)
Mo(1)–O(4′)	1.747(5)	Mo(3)–O(11)	1.829(5)
Mo(1)–O(8′)	2.291(5)	Mo(4)–O(2)	2.154(4)
Mo(2)–O(4)	2.368(4)	Mo(4)–O(6)	1.929(5)
Mo(2)–O(5)	1.705(5)	Mo(4)–O(11)	1.992(6)
Mo(2)–O(6)	1.900(4)	Mo(4)–O(12)	1.707(5)
Mo(2)–O(7)	1.696(4)	Mo(4)–O(13)	1.687(5)
Mo(2)–O(3′)	2.213(4)	Mo(4)–O(3′)	2.495(4)
Mo(2)–O(8′)	2.013(4)	N(2)–C(23)	1.489(9)
N(1)–C(11)	1.504(9)	N(2)–C(6)	1.494(9)
N(1)–C(12)	1.520(9)	C(1)–C(2)	1.513(9)
N(1)–C(13)	1.503(8)	C(2)–C(3)	1.521(10)
N(1)–C(1)	1.513(9)	C(3)–C(4)	1.515(9)
N(2)–C(21)	1.469(9)	C(4)–C(5)	1.520(9)
N(2)–C(22)	1.480(9)	C(5)–C(6)	1.527(9)

Table 3 Bond angles (°) for $[Me_3N(CH_2)_6NMe_3]_2[Mo_8O_{26}]\cdot 2H_2O$

O(4′)–Mo(1)–O(8′)	162.0	O(10)–Mo(3)–O(11)	101.0
O(3′)–Mo(1)–O(8′)	72.7	O(9)–Mo(3)–O(11)	113.2
O(3′)–Mo(1)–O(4′)	99.4	O(9)–Mo(3)–O(10)	104.7
O(2′)–Mo(1)–O(8′)	73.9	O(8)–Mo(3)–O(11)	121.8
O(2′)–Mo(1)–O(4′)	103.7	O(8)–Mo(3)–O(10)	99.5
O(2′)–Mo(1)–O(3′)	135.9	O(8)–Mo(3)–O(9)	113.1
O(1′)–Mo(1)–O(8′)	93.1	O(2)–Mo(3)–O(11)	73.5
O(1′)–Mo(1)–O(4′)	104.7	O(2)–Mo(3)–O(10)	164.1
O(1′)–Mo(1)–O(3′)	104.8	O(2)–Mo(3)–O(9)	91.1
O(1′)–Mo(1)–O(2′)	104.9	O(2)–Mo(3)–O(8)	72.2
O(3)–Mo(1)–O(8′)	86.3	O(13)–Mo(4)–O(3′)	165.0
O(3)–Mo(1)–O(4′)	76.1	O(12)–Mo(4)–O(3′)	88.5
O(3)–Mo(1)–O(3′)	77.7	O(12)–Mo(4)–O(13)	105.0
O(3)–Mo(1)–O(2′)	72.3	O(11)–Mo(4)–O(3′)	85.2
O(3)–Mo(1)–O(1′)	177.2	O(11)–Mo(4)–O(13)	100.5
O(3′)–Mo(2)–O(8′)	72.6	O(11)–Mo(4)–O(12)	92.5
O(7)–Mo(2)–O(8′)	96.7	O(6)–Mo(4)–O(3′)	71.2
O(7)–Mo(2)–O(3′)	98.3	O(6)–Mo(4)–O(13)	99.7
O(6)–Mo(2)–O(8′)	148.9	O(6)–Mo(4)–O(12)	98.5
O(6)–Mo(2)–O(3′)	78.6	O(6)–Mo(4)–O(11)	153.5
O(6)–Mo(2)–O(7)	99.0	O(2)–Mo(4)–O(3′)	68.4
O(5)–Mo(2)–O(8′)	98.2	O(2)–Mo(4)–O(13)	99.4
O(5)–Mo(2)–O(3′)	156.2	O(2)–Mo(4)–O(12)	154.4
O(5)–Mo(2)–O(7)	104.6	O(2)–Mo(4)–O(11)	75.4
O(5)–Mo(2)–O(6)	103.7	O(2)–Mo(4)–O(6)	84.6
O(4)–Mo(2)–O(8′)	81.4	C(13)–N(1)–C(1)	111.5
O(4)–Mo(2)–O(3′)	71.5	C(12)–N(1)–C(1)	106.9
O(4)–Mo(2)–O(7)	169.7	C(12)–N(1)–C(13)	109.1
O(4)–Mo(2)–O(6)	78.4	C(11)–N(1)–C(1)	110.6
O(4)–Mo(2)–O(5)	85.7	C(11)–N(1)–C(13)	110.9
Mo(3)–O(2)–Mo(4)	93.1	C(11)–N(1)–C(12)	107.6
Mo(4)–O(2)–Mo(1′)	123.9	N(1)–C(1)–C(2)	116.0
Mo(3)–O(2)–Mo(1′)	105.6	C(1)–C(2)–C(3)	107.7
Mo(1)–O(3)–Mo(4′)	91.2	C(2)–C(3)–C(4)	113.8
Mo(1)–O(3)–Mo(2′)	95.8	C(3)–C(4)–C(5)	115.2
Mo(1)–O(3)–Mo(1′)	102.4	C(4)–C(5)–C(6)	106.7
Mo(2′)–O(3)–Mo(4′)	87.4	C(23)–N(2)–C(6)	110.1
Mo(1′)–O(3)–Mo(4′)	156.7	C(22)–N(2)–C(6)	106.8
Mo(1′)–O(3)–Mo(2′)	109.7	C(22)–N(2)–C(23)	107.2
Mo(2)–O(4)–Mo(1′)	115.5	C(21)–N(2)–C(6)	110.3
Mo(2)–O(6)–Mo(4)	116.6	C(21)–N(2)–C(23)	110.9
Mo(3)–O(8)–Mo(2′)	140.9	C(21)–N(2)–C(22)	111.4
Mo(3)–O(8)–Mo(1′)	107.7		
Mo(1′)–O(8)–Mo(2′)	103.0		
Mo(3)–O(11)–Mo(4)	118.0		

Mean e.s.d.s: for O–Mo–O, 0.2; for C–N–C, N–C–C and C–C–C, 0.6°.

bonds; the resulting O=Mo=O angles are in the range 104.6(3)–105.0(3)°, which is considerably larger than the regular octahedral or regular trigonal-bipyramidal 90° *cis* groups; this is expected from the greater O···O repulsions between oxygens with short bonds to the metal atom and has previously been observed.[16] For Mo(4) we see the expected trend of increasing Mo–O for two- < three- < four-co-ordinate oxygens, but the trend is not so apparent for the other six-co-ordinate molybdenums: for Mo(1), the Mo(1)–O(3) (three-co-ordinated) [2.494(4) Å] is longer than Mo(1)–O(3′) (four-co-ordinated [1.907(4) Å]; similarly for Mo(2), the Mo(2)–O(4) (two-co-ordinated) [2.368(4) Å] is longer than both Mo(2)–O(8′) (three-co-ordinated) [2.013(4) Å] and Mo(2)–O(3′) (four-co-ordinated) [2.213(4) Å]. These variations are no doubt a consequence of departure from regular octahedral symmetry as evidenced by large deviations of the bond angles from 180 and 90° for *trans*- and *cis*-co-ordinated oxygens.

For Mo(3), the five-co-ordinate molybdenum, the Mo–O bond lengths increase for two- < three- co-ordinate oxygens. While deviations from any regular five-co-ordinate shape are clearly apparent, one O–Mo–O angle close to 180° [164.1(3)°], three O–Mo–O close to 120° [range 113.1(2)–121.8(2)°] and six O–Mo–O close to 90° [range 72.2(2)–104.7(3)°] enable identification of the co-ordination as trigonal bipyramidal rather than square-based pyramidal. The structure of the $[Mo_8O_{26}]^{4-}$ anion as determined here hence very closely approximates that of the γ intermediate proposed by Klemperer and Shum[9] in their study of the synthesis and interconversion in acetonitrile of the isomeric α- and β-$[Mo_8O_{26}]^{4-}$ anions, α$[NBu_4]_4[Mo_8O_{26}]$ and β$[NBu_4]K[Mo_8O_{26}]\cdot 2H_2O$. They noted there that the observed rapid isomerization under anhydrous conditions was difficult to reconcile with the then current mechanistic theories of polyoxoanion transformations[17] and proposed the γ intermediate, which like the α and β forms, consists of c.c.p. oxygens with molybdenums occupying tetrahedral and octahedral holes. They further proposed that the isomerization $\alpha \longrightarrow \beta$ and $\beta \longrightarrow \alpha$ occurs *via* unstable α–γ and β–γ structures which consist of close-packed oxygens with molybdenums occupying trigonal-bipyramidal and octahedral holes, with the oxygen close-packing based on interpenetrating hexagonally close-packed (h.c.p.) layers so that as the isomerization proceeds a shear plane arising from repeated c.c.p.–h.c.p. transformations becomes apparent.

The structure reported here hence confirms that such a γ form is indeed at an energy minimum, and that the mechanism for the α–β isomerism reported by Klemperer and Shum[9] is viable, possibly more so than that proposed by Masters *et al.*[14] Interestingly, Klemperer and Shum note that the isomerism may be catalysed by traces of water, from which they put forward an expectation of hydroxide co-ordination at the vacant sites on the two five-co-ordinate molybdenums. Our structure has water present, but its oxygen O(31) is 4.40 Å from Mo(3), too far for any co-ordination considerations; the anion hence retains its six- and five-co-ordinate molybdenums. The water is likely hydrogen bonded to either of the terminal oxygens on Mo(3) (see later) and hence its catalytic role in the isomerization cannot be discounted.

The $[Me_3N(CH_2)_6NMe_3]^{2+}$ *Cation and Water Molecule.*—The cation has bond lengths and angles which are all of the expected order of magnitude[18] and do not merit discussion. It is of course extremely interesting in the role it plays in stabilizing, in aqueous solution, the γ form proposed to be an intermediate in the cation-dependent α–β equilibrium in acetonitrile. Under the conditions of crystallization (pH *ca.* 6) the heptamolybdate ion, $[Mo_7O_{24}]^{6-}$, is the predominating polynuclear species in solution; significant concentrations of the octamolybdate ion only occur at much lower pH, *i.e.* in the range 2–3.[19] The α or β form is normally selectively crystallized from an aqueous molybdate solution acidified to pH 3–4 by addition of a particular cation.[9] The γ form on the other hand was captured during acidification of molybdate by the presence of the $[Me_3N(CH_2)_6NMe_3]^{2+}$ cation at a relatively high pH. Its

Table 4 Hydrogen-bonding parameters (distances in Å, angles in °) involving the water of crystallization

O–H*		H···O	
O(31)–H(311)	1.00(1)	H(311)···O(9^{I})	2.00(1)
O(31)–H(312)	1.00(1)	H(312)···O(10^{II})	1.04(1)
O–H–O		O–H···O	
H(311)–O(31)–H(312)	98(6)	O(31)–H(311)···O(9^{I})	141(2)
		O(31)–H(312)···O(10^{II})	142(2)
O···O			
O(31)···O(9)	2.847(9)		
O(31)···O(10)	2.892(9)		

* Constraint applied to O–H distance. Symmetry elements applied: **I** $x + 1, y, z$; **II** $-x + \frac{1}{2}, y + \frac{1}{2}, -z + \frac{3}{2}$.

Table 5 Crystal data, details of the data collection and final refinements for $[Me_3N(CH_2)_6NMe_3]_2[Mo_8O_{26}]\cdot 2H_2O$

Molecular formula	$C_{24}H_{64}Mo_8N_4O_{28}$
M	1624.30
Crystal symmetry	Monoclinic
Space group	$P2_1/n$
a/Å	12.42(1)
b/Å	12.897(3)
c/Å	15.74(1)
β/°	106.41(5)
U/Å^3	2419(3)
D_c/g cm^{-3} (for $Z = 2$)	2.23
F(000)	1592
μ(Mo-Kα)/cm^{-1}	20.44
Crystal dimensions/mm	0.06 × 0.13 × 0.16
Scan mode	ω–2θ
Scan width/°	0.90 + 0.35 tan θ
Aperture width/mm	1.12 + 1.05 tan θ
θ range scanned/°	1–25
Range of h, k, l	−14 to 14, 0–15, 0–18
Crystal decay (%)	2.3
Absorption correction (max., min., av. transmission)	99.95, 79.25, 91.36
No. of unique reflections collected	3761
No. of reflections with $I_{rel} > 2\sigma I_{rel}$	3153
No. of parameters	315
$R = \Sigma\|\|F_o\| - \|F_c\|\|/\Sigma\|F_o\|$	0.030
$R' = \Sigma w^{\frac{1}{2}}\|\|F_o\| - \|F_c\|\|/\Sigma w^{\frac{1}{2}}\|F_o\|$	0.029
w	$(\sigma^2 F)^{-1}$
S	1.41

existence in aqueous solution as a thermodynamically stable species in equilibrium with other polyanions is therefore still to be decided. The possibility of isolating γ-$[Mo_8O_{26}]^{4-}$ from an acidified solution already at equilibrium by addition of a suitable cation is currently being investigated.

The waters of crystallization, which are symmetry related are located such that they are hydrogen bonded to O(9) and O(10) of adjacent molybdenum cations, as shown in Table 4, giving some degree of polymerization to the whole structure.

Experimental

Crystal Preparation.—When an aqueous solution 0.4 mol dm^{-3} in both $Na_2[MoO_4]\cdot 2H_2O$ and $[Me_3(CH_2)_6NMe_3]Cl_2$ was treated with *ca.* 1.0 mol dm^{-3} HCl a cloudy white precipitate appeared momentarily as each drop of acid was added. The precipitate dissolved rapidly upon stirring. However, at pH *ca.* 6 very fine crystals became visible which could not be dissolved by stirring. Upon standing, a crop of very small but well defined crystals was rapidly formed.

X-Ray Structure Determination.—A suitable single crystal was irradiated with Mo-Kα (λ = 0.7107 Å) radiation on an Enraf-Nonius CAD4 diffractometer. Accurate cell parameters were obtained by least-squares refinement of the setting angles of 24 reflections (16 < θ < 17°). Data were collected with variable scan width, aperture width and speed. Reference reflections were periodically monitored for intensity and orientation control. The data were corrected for Lorentz polarization and an empirical absorption correction was applied.[20] Further details of the crystal and data collection are reported in Table 5.

Structure solution and refinement. The structure was solved by direct methods using SHELXS 86[21] and refined using SHELX 76.[22] In the final refinements, all non-hydrogen atoms were treated anisotropically; all hydrogens were treated isotropically with linked thermal parameters. The methylene hydrogens of the cation were placed in calculated positions and the hydrogens of the methyls treated as rigid groups. The water hydrogens were initially positioned as located in a difference map and subsequently constrained to ride at 1.00(1) Å from the parent oxygen. An appropriate weighting scheme was employed. Complex neutral atom scattering factors were taken from Cromer and Mann[23] for non-hydrogen atoms, and from Stewart *et al.*[24] for H, with dispersion corrections from Cromer and Liberman.[25] Molecular parameters were calculated using PARST[26] and drawings obtained using PLUTO.[27] All computations were performed on a VAX computer at the Computer Centre of the University of Cape Town. Final fractional atomic coordinates are reported in Table 1.

Additional material available from the Cambridge Crystallographic Data Centre comprises H-atom coordinates and thermal parameters.

Acknowledgements

The authors thank the Universities of Cape Town and Stellenbosch and the Foundation for Research Development for financial assistance.

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Received 4*th February* 1991; *Paper* 1/00520K