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## Structure of Bis(dimethylammonium) Bis(tetrabutylammonium) β-Octamolybdate

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**Abstract.** X-ray structure analysis has confirmed the presence of the [Mo<sub>8</sub>O<sub>26</sub>]<sup>4-</sup> anion in [NH<sub>2</sub>(CH<sub>3</sub>)<sub>2</sub>]<sub>2</sub>-[N(C<sub>4</sub>H<sub>9</sub>)<sub>4</sub>]<sub>2</sub>[Mo<sub>8</sub>O<sub>26</sub>]: monoclinic, *P*<sub>2</sub><sub>1</sub>/*c*, *M*<sub>r</sub> = 1760.63, *a* = 10.287 (2), *b* = 14.033 (4), *c* = 21.241 (4) Å, β = 99.24 (2)°, *V* = 3026.4 Å<sup>3</sup>, *Z* = 2, *D*<sub>x</sub> = 1.93 g cm<sup>-3</sup>, λ(Mo Kα) = 0.7107 Å, μ = 16.4 cm<sup>-1</sup>, *F*(000) = 1752, *T* = 294 (1) K, *R* = 0.044, *wR* = 0.069 for 5137 observed reflections. Eight MoO<sub>6</sub> distorted octahedra share edges with short terminal Mo—O bonds (1.694–1.741 Å), bonds of intermediate length (1.889–2.001 Å) and long bonds (2.165–2.465 Å). The structure contains the β-Mo<sub>8</sub>O<sub>26</sub> polyanion.

**Introduction.** In previous papers reporting different types of octamolybdate anions (Roman, Gutierrez-

Zorrilla, Martínez-Ripoll & Garcia-Blanco, 1987), it seems that in the solid state the most frequent type is the β-isomer. In a previous paper, we reported that α-[Mo<sub>8</sub>O<sub>26</sub>] was transformed to β-[Mo<sub>8</sub>O<sub>26</sub>] in methanol (Wang, Xu & Wang, 1992). In order to find a mechanism for the isomerization of α- and β-[Mo<sub>8</sub>O<sub>26</sub>]<sup>4-</sup>, we continue to study the transformation of α- and β-Mo<sub>8</sub>O<sub>26</sub>. The crystal investigated in this study was grown when trying to prepare a suitable single crystal of triethylammonium octamolybdate from DMF solutions (DMF = *N,N*-dimethylformamide) of the tetrabutylammonium salt. Unexpectedly, crystals of the double salt were obtained. The dimethylammonium ion might have come from the free amine as an impurity in the DMF (DMF was used without subsequent purification). Alternatively, DMF may have been dissociated by water molecules absorbed from the

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atmosphere to give  $\text{NH}(\text{CH}_3)_2$  and formic acid (Wilson, McKee, Penfold & Wilkins, 1984; Piggott, Wong, Hursthouse & Short, 1988).

**Experimental.** 1.0 mmol  $\alpha\text{-[NBu}_4\text{]}_4\text{[Mo}_8\text{O}_{26}\text{]}$  (Filowitz, Ho, Klemperer & Shum, 1979) and  $10\text{ cm}^3$   $\text{NEt}_3$  were dissolved in DMF. The reaction mixture was heated to reflux and stirred for 2 h. After layering ether, large transparent crystals of the title compound appeared.

A selected single crystal of approximate dimensions  $0.20 \times 0.25 \times 0.35$  mm was mounted on a glass fibre. The intensity data were collected on an Enraf-Nonius CAD-4 diffractometer using graphite-monochromated Mo  $K\alpha$  radiation. The scan mode was  $\omega\text{-}2\theta$  in the range  $2 \leq 2\theta \leq 52^\circ$  ( $-13 \leq h \leq 13$ ,  $0 \leq k \leq 17$ ,  $0 \leq l < 26$ ). 6370 independent reflections were collected, of which 5137 with  $I > 3\sigma(I)$  were used for further computation. All data were corrected for Lorentz and polarization effects, and empirical absorption corrections, based on the  $\psi$  scan, were applied (maximum 0.998, minimum 0.956). Three standard reflections, monitored every 90 min, showed 3% variation in intensity.

The structure analysis was performed on a PDP11/44 computer with the *SDP* programs (Enraf-Nonius, 1985). The positions of all Mo atoms were determined by direct methods. The positions of the other non-H atoms were revealed by difference Fourier synthesis. The H atoms, located in a difference map, were included as fixed contributions.

The structure was refined with unit weights by full-matrix least-squares methods with anisotropic thermal factors for all non-H atoms. Refinement converged to a final  $R = 0.044$  and  $wR = 0.069$  for the 5137 observed reflections and 334 parameters.  $(\Delta/\sigma)_{\text{max}} = 0.24$ ,  $(\Delta\rho)_{\text{max}} = 0.83$ ,  $(\Delta\rho)_{\text{min}} = -0.4\text{ e \AA}^{-3}$ . Atomic scattering factors and  $f'$ ,  $f''$  values were obtained from *International Tables for X-ray Crystallography* (1974, Vol. IV). Table 1 shows the final atomic coordinates.\*

**Discussion.** The important bond distances and angles are summarized in Table 2.\*

The octamolybdate polyanion has a crystallographic centre of inversion. The polyanion shows the same configuration as in the salts of ammonium (Lindqvist, 1950; Weakley, 1982), disodium bis(tetramethylammonium) (Fuchs & Knopnadel, 1982), melaminium (Kroenke, Fackler & Mazany, 1983),

Table 1. *Atomic parameters and equivalent isotropic thermal parameters* ( $\text{\AA}^2$ )

$$B_{\text{eq}} = (4/3)[a^2B_{11} + b^2B_{22} + c^2B_{33} + ac(\cos\beta)B_{13}].$$

	<i>x</i>	<i>y</i>	<i>z</i>	$B_{\text{eq}}$
Mo(1)	0.61662 (9)	0.24501 (6)	0.49895 (4)	2.67 (2)
Mo(2)	0.56830 (8)	0.10599 (6)	0.61526 (4)	2.12 (1)
Mo(3)	0.32177 (8)	-0.05807 (6)	0.58194 (4)	2.05 (1)
Mo(4)	0.36873 (8)	0.07775 (6)	0.46606 (4)	1.75 (1)
O(1)	0.5800 (9)	0.3603 (5)	0.4803 (4)	4.3 (2)
O(2)	0.7801 (7)	0.2451 (7)	0.5333 (4)	4.3 (2)
O(3)	0.5022 (8)	0.1232 (5)	0.6826 (3)	3.2 (2)
O(4)	0.7358 (7)	0.1075 (6)	0.6408 (4)	3.4 (2)
O(5)	0.3170 (7)	-0.0388 (5)	0.6606 (3)	2.8 (1)
O(6)	0.1602 (7)	-0.0563 (5)	0.5459 (3)	2.9 (1)
O(7)	0.2056 (6)	0.0692 (5)	0.4358 (3)	2.8 (1)
O(8)	0.5428 (7)	0.2269 (5)	0.5757 (3)	2.6 (1)
O(9)	0.3661 (7)	-0.1887 (5)	0.5817 (3)	2.7 (1)
O(10)	0.4054 (7)	0.1983 (5)	0.4606 (3)	2.4 (1)
O(11)	0.4498 (6)	0.0350 (5)	0.3947 (3)	2.1 (1)
O(12)	0.3624 (6)	0.0750 (5)	0.5574 (3)	1.9 (1)
O(13)	0.5956 (6)	0.0712 (4)	0.5110 (3)	1.8 (1)
N(1)	0.3956 (8)	0.4409 (6)	0.6636 (4)	2.2 (2)
C(1)	0.297 (1)	0.5050 (8)	0.6908 (5)	3.2 (2)
C(2)	0.278 (1)	0.6042 (9)	0.6632 (7)	4.1 (3)
C(3)	0.172 (1)	0.6534 (9)	0.6967 (6)	4.1 (3)
C(4)	0.145 (1)	0.754 (1)	0.6739 (8)	5.3 (3)
C(5)	0.535 (1)	0.4795 (8)	0.6837 (5)	3.0 (2)
C(6)	0.647 (1)	0.4175 (9)	0.6655 (6)	3.8 (3)
C(7)	0.778 (1)	0.465 (1)	0.6856 (7)	5.4 (3)
C(8)	0.888 (1)	0.402 (1)	0.6676 (8)	6.5 (4)
C(9)	0.368 (1)	0.4375 (8)	0.5909 (5)	3.0 (2)
C(10)	0.229 (1)	0.403 (1)	0.5617 (6)	5.0 (3)
C(11)	0.217 (1)	0.414 (1)	0.4890 (6)	5.5 (4)
C(12)	0.089 (2)	0.375 (2)	0.454 (1)	11.3 (7)
C(13)	0.382 (1)	0.3398 (7)	0.6894 (5)	2.7 (2)
C(14)	0.425 (1)	0.3301 (9)	0.7612 (5)	3.5 (2)
C(15)	0.358 (1)	0.247 (1)	0.7884 (6)	3.9 (3)
C(16)	0.209 (1)	0.269 (1)	0.7923 (8)	6.3 (4)
N(2)	-0.020 (1)	0.4137 (8)	0.0923 (5)	4.2 (2)
C(17)	0.020 (1)	0.445 (2)	0.1591 (7)	6.7 (4)
C(18)	0.056 (1)	0.334 (1)	0.071 (1)	6.5 (4)

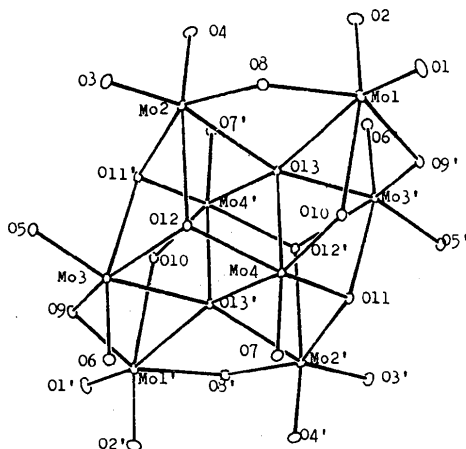
dimethylammonium (Wilson *et al.*, 1984; Piggott *et al.*, 1988), bis(triethylammonium) bis(tetrabutylammonium) (Wang *et al.*, 1992), 2-ethylpyridinium (Roman, Martínez-Ripoll & Jaud, 1982), 3-ethylpyridinium (Roman, Jaud & Galy, 1981), 4-ethylpyridinium (Roman, Vegas, Martínez-Ripoll & Garcia-Blanco, 1982), 3-aminopyridinium (Roman & Gutierrez-Zorrilla, 1985) and anilinium (Roman, Gutierrez-Zorrilla, Martínez-Ripoll, Estaban-Calderon & Garcia-Blanco, 1985) cations.

The  $[\text{Mo}_8\text{O}_{26}]^{4-}$  anion is built up of eight  $\text{MoO}_6$  distorted octahedra sharing edges and corners. They can be described alternatively as two cyclic  $\text{Mo}_4\text{O}_{12}$  units formed from distorted  $\text{MoO}_4$  tetrahedra, cross-linked by long Mo—O bonds and by additional long bonds from Mo to two extra  $\text{O}^{2-}$  ions [O(13), O(13')]. The Mo—O bond lengths vary from 1.694 Å for one of the nonbridging Mo—O bonds to 2.465 Å for one of the bonds to the unusual five-coordinate O atom [O(13)] that sits near the centre of each  $\text{Mo}_4\text{O}_{12}$  half of the anion, in the plane of the four Mo atoms. In the idealized octamolybdate anion there are three different types of Mo atom: four  $\text{Mo}^a$  [Mo(2), Mo(3), Mo(2'), Mo(3')], two  $\text{Mo}^b$  [Mo(1), Mo(1')] and two  $\text{Mo}^c$  [Mo(4), Mo(4')] (see Fig. 1). The Mo—O bonds can be classified in three types by their bond lengths: short terminal Mo—O bonds

\* Lists of observed and calculated structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55603 (31 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CR0415]

Table 2. Mo—O and O—O distances (Å) and O—Mo—O bond angles (°) in the β-Mo<sub>8</sub>O<sub>26</sub> anion

	Mo(1)	O(1)	O(2)	O(8)	O(9')	O(10)	O(13)
O(1)	1.694 (8)		2.716 (12)	2.830 (11)	2.842 (11)	2.886 (11)	
O(2)	1.723 (9)	105.3 (4)		2.746 (11)	2.770 (11)		3.081 (11)
O(8)	1.922 (7)	102.8 (4)	97.6 (4)			2.651 (10)	2.683 (9)
O(9')	1.923 (7)	103.5 (4)	98.9 (4)	143.9 (3)		2.651 (10)	2.646 (9)
O(10)	2.290 (7)	91.6 (3)	163.1 (3)	77.4 (3)	77.6 (3)		2.736 (9)
O(13)	2.465 (6)	161.7 (3)	93.1 (3)	74.2 (3)	73.0 (3)	70.10 (22)	
	Mo(2)	O(3)	O(4)	O(8)	O(11')	O(12)	O(13)
O(3)	1.696 (7)		2.701 (11)	2.784 (10)	2.851 (10)	2.893 (10)	
O(4)	1.721 (8)	104.5 (4)		2.789 (11)	2.784 (10)		2.943 (10)
O(8)	1.893 (7)	101.6 (3)	100.9 (3)			2.812 (10)	2.683 (10)
O(11)	1.996 (7)	100.9 (3)	96.8 (3)	146.9 (3)		2.553 (9)	2.598 (9)
O(12)	2.314 (7)	91.0 (3)	162.6 (3)	83.3 (3)	72.2 (3)		2.736 (9)
O(13)	2.330 (6)	163.2 (3)	92.0 (3)	78.1 (3)	73.4 (2)	72.25 (22)	
	Mo(3)	O(5)	O(6)	O(9)	O(11')	O(12)	O(13')
O(5)	1.700 (7)		2.709 (10)	2.786 (10)	2.836 (10)	2.811 (10)	
O(6)	1.716 (7)	104.9 (3)		2.828 (10)		2.760 (10)	2.965 (9)
O(9)	1.889 (7)	101.7 (3)	103.3 (3)		2.861 (10)		2.646 (9)
O(11')	2.345 (7)	87.6 (3)	163.5 (3)	84.3 (3)		2.553 (9)	2.724 (9)
O(12)	2.001 (7)	98.6 (3)	95.6 (3)	147.6 (3)	71.5 (3)		2.590 (9)
O(13')	2.278 (6)	159.6 (3)	94.8 (3)	78.2 (3)	72.17 (22)	74.2 (2)	
	Mo(4)	O(7)	O(10)	O(11)	O(12)	O(13')	O(13)
O(7)	1.702 (7)		2.727 (10)	2.830 (9)	2.818 (9)	2.932 (9)	
O(10)	1.741 (7)	104.7 (9)		2.761 (4)	2.777 (10)		2.735 (9)
O(11)	1.938 (7)	101.9 (3)	97.2 (3)			2.598 (9)	2.724 (9)
O(12)	1.952 (7)	100.7 (3)	97.3 (3)	149.1 (3)		2.590 (9)	2.737 (9)
O(13')	2.165 (6)	97.9 (3)	157.3 (3)	78.5 (3)	77.8 (3)		2.790 (9)
O(13)	2.377 (6)	173.7 (3)	81.6 (3)	77.5 (2)	77.7 (2)	75.7 (2)	

Fig. 1. The [Mo<sub>8</sub>O<sub>26</sub>]<sup>4-</sup> polyanion. Thermal ellipsoids are drawn at the 50% probability level.

(1.694–1.741 Å), bonds of intermediate length (1.889–2.001 Å) and long bonds (2.165–2.465 Å).

The structure of the present anion matches those found in [NH<sub>4</sub>Et<sub>3</sub>]<sub>2</sub>[NBu<sub>4</sub>]<sub>2</sub>[Mo<sub>8</sub>O<sub>26</sub>].2H<sub>2</sub>O (Wang *et al.*, 1992) and [NH<sub>2</sub>(CH<sub>3</sub>)<sub>24</sub>]<sub>2</sub>[Mo<sub>8</sub>O<sub>26</sub>].2C<sub>3</sub>H<sub>7</sub>NO (Wilson *et al.*, 1984). It can be seen from these anions that there are slight differences in the Mo—O distances [except the Mo(1)—O(13) distance] owing to the different cation–polyanion interactions. The Mo(1)—O(13) distances vary from 2.414 to 2.485 Å with enlargement of the cation. The smaller the cation, the shorter is the distance Mo(1)—O(13) and the easier is the formation of β-octamolybdate.

Table 3 provides a comparison of the distortions of the MoO<sub>6</sub> octahedra in these three compounds. The degrees of distortion of the MoO<sub>6</sub> octahedra have been evaluated by the equation  $\Delta l = (1/6)\sum[(R_i + R)/R]^2$ , where  $R_i$  = individual Mo—O distance and  $R$  = mean Mo—O distance.

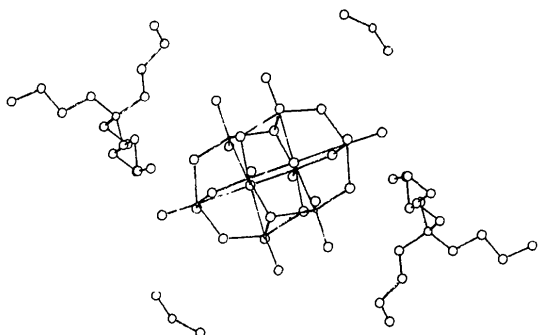
The Mo(4) octahedron is the least distorted because it is the most restrained. This fact can be explained by its central position in the polyanion. On the other hand, the Mo(1) octahedron is the most distorted since it is the most external. Mo(2) and Mo(3) present an intermediate degree of distortion.

It can be seen from Table 3 that there are significant differences in the degree of distortion of these three compounds. When tetrabutylammonium in α-[NBu<sub>4</sub>]<sub>4</sub>[Mo<sub>8</sub>O<sub>26</sub>] was replaced by other small groups, for example, methyl-, ethyl- and propylammonium, K and Na, the α type was isomerized to β type (Day, Abdel-Meguid, Dabestani, Thomas, Pretzer & Muettterties, 1976). For the larger cations, the greater the degree of distortion, the easier the Mo(1)—O(13) bond is broken and the easier the α-octamolybdate is formed. As has been suggested (Day, Friedrich, Klemperer & Shum, 1977), the [Mo<sub>8</sub>O<sub>26</sub>]<sup>4-</sup> cluster can be viewed as consisting of two (O<sup>2-</sup>) (Mo<sub>4</sub>O<sub>12</sub>) subunits by breaking only bonds whose lengths exceed 2.22 Å and hence have bond orders less than 0.2. Such a viewpoint may be helpful in understanding some of the transformations and reactivities of the β-[Mo<sub>8</sub>O<sub>26</sub>]<sup>4-</sup> anion in solution.

The dimethylammonium N(2) atom shows short distances (in the range 2.88–3.15 Å) to terminal O

Table 3. Degree of distortion within the MoO<sub>6</sub> octahedron

Compound	Distortion $\Delta l$ ( $\times 10^4$ )	Mo(1)	Mo(2)	Mo(3)	Mo(4)
[NH <sub>2</sub> (CH <sub>3</sub> ) <sub>2</sub> ] <sub>2</sub> [NBu <sub>4</sub> ] <sub>2</sub> salt	$\Delta l$ ( $\times 10^4$ )	201	163	160	140
[HNEt <sub>3</sub> ][NBu <sub>4</sub> ] salt	$\Delta l$ ( $\times 10^4$ )	218	175	160	145
[NH <sub>2</sub> (CH <sub>3</sub> ) <sub>2</sub> ] <sub>4</sub> salt	$\Delta l$ ( $\times 10^4$ )	190	133	169	134


 Fig. 2. The crystal packing of [NH<sub>2</sub>(CH<sub>3</sub>)<sub>2</sub>]<sub>2</sub>[NBu<sub>4</sub>]<sub>2</sub>(Mo<sub>8</sub>O<sub>26</sub>).

atoms of the anions, which indicates the presence of hydrogen bonding to the [Mo<sub>8</sub>O<sub>26</sub>]<sup>4-</sup> anion. Some of these hydrogen bonds may be responsible for the photochromic properties in the solid state. Crystal packing is shown in Fig. 2.

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## Structure of Tetrakis( $\mu$ -acetato)-bis(4-pyridylmethanol)dicopper(II)

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**Abstract.** [Cu<sub>2</sub>(C<sub>2</sub>H<sub>3</sub>O<sub>2</sub>)<sub>4</sub>(C<sub>6</sub>H<sub>7</sub>NO)<sub>2</sub>],  $M_r = 581.53$ , triclinic,  $P\bar{1}$ ,  $a = 7.301$  (4),  $b = 8.216$  (4),  $c = 10.709$  (5) Å,  $\alpha = 91.407$  (1),  $\beta = 98.247$  (1),  $\gamma = 114.242$  (3)°,  $V = 577.27$  Å<sup>3</sup>,  $Z = 1$ ,  $D_m = 1.63$ ,  $D_x = 1.67$  Mg m<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.71069$  Å,  $\mu =$

$1.90$  mm<sup>-1</sup>,  $F(000) = 298$ ,  $T = 293$  K. Final  $R = 0.042$  for 1699 observed reflections. The crystal contains centrosymmetric binuclear molecules with four carboxylate bridges spanning a Cu...Cu separation of 2.647 (1) Å, mean Cu—O 1.982 (2) Å and Cu—N(apical) 2.160 (3) Å. The Cu atoms are displaced by 0.211 (1) Å from the plane containing four oxygen atoms towards the ligand 4-pyridylmethanol.

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