Orthorhombic
Pbca
$a = 11.487$ (2) Å
$b = 12.067$ (1) Å
$c = 18.967$ (3) Å
$V = 2629.0$ (5) Å^3
$Z = 8$
$D_x = 3.29$ Mg m^{-3}

Cell parameters from 25 reflections
$\theta = 6.4\text{-}22.6°$
$\mu = 22.19$ mm^{-1}
Room temperature
Prism
0.2 × 0.18 × 0.12 mm
Yellow

Data collection

Enraf-Nonius CAD-4 diffractometer
ω-2θ scans
Absorption correction: by integration from crystal shape
$T_{\min} = 0.098$, $T_{\max} = 0.218$
2571 measured reflections
2511 independent reflections

1497 observed reflections [$I > 2\sigma(I)$]
$R_{\text{int}} = 0.0082$ (based on standard reflections)
$\theta_{\max} = 28°$
$h = 0 \rightarrow 14$
$k = 0 \rightarrow 14$
$l = 0 \rightarrow 23$
2 standard reflections
frequency: 120 min
intensity variation: <2.5%

Refinement

Refinement on F
$R = 0.038$
$wR = 0.048$
$S = 1.09$
1497 reflections
118 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F)/4|F_o|^2 + 0.03|F_o|^2 + 8.0]$
$(\Delta/\sigma)_{\max} = 0.06$

$\Delta\rho_{\max} = 2.27$ e Å^{-3}
$\Delta\rho_{\min} = -1.89$ e Å^{-3}
Extinction correction: Becker & Coppens (1975)
Extinction coefficient: 0.1013×10^5
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

The structure was solved by direct and difference Fourier methods and refined by full-matrix least-squares calculations employing programs amended by Lundgren (1982). The H atoms could not be located in the difference Fourier maps and they were not included in the structure-factor calculations.

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Lists of structure factors, anisotropic displacement parameters and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71796 (27 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AB1105]

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Hexakis(*tert*-butylammonium) Heptamolybdate(VI)–Water (1/7)

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Abstract

The asymmetric unit of $[(CH_3)_3CNH_3]_6[Mo_7O_{24}].7H_2O$ consists of a heptamolybdate anion, six *tert*-butylammonium cations and seven water molecules. The crystal packing is stabilized by electrostatic interactions and an extensive network of hydrogen contacts involving the O atoms from the polyanion, the water molecules and the N atoms, and some C atoms of the cations. Some of these hydrogen bonds may play an important role in the UV-induced photochromism of this compound.

Comment

This work was undertaken in order to obtain a deeper insight into the effect of the cation on the crystal packing, the strength of the intermolecular contacts and the chemical properties of organoammonium polyoxomolybdates. Several studies on polyoxometallates of V, Mo and W have been carried out in recent years because of their applications in photochemistry, catalysis and biomedicine

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(Papaconstantinou, 1989; Day & Kemplerer, 1985; Hill & Bouchard, 1985; Pope & Müller, 1991; Hill, Weeks & Schinazi, 1990, 1992). One of these inorganic metal–oxygen clusters is the heptamolybdate anion whose organic salts show interesting photochemical properties and antitumour activity (Yamase & Tomita, 1990; Martínez de Pancorbo *et al.*, 1988).

In this context and as a part of our extensive studies of molybdenum–alkylamine systems in aqueous solution (Román, Luque, Aranzabe & Gutiérrez-Zorrilla, 1992), we have studied the reaction between *tert*-butylamine and molybdenum trioxide (Román, San José, Luque & Gutiérrez-Zorrilla, 1993). One of the compounds obtained was hexakis(*tert*-butylammonium) heptamolybdate(VI)–water (1/7), the synthesis and chemical characterization of which have been reported recently by us (Román, San José, Aranzabe & Luque, 1992).

The asymmetric unit contains one discrete $[Mo_7O_{24}]^{6-}$ polyanion, six *tert*-butylammonium cations and seven water molecules. The heptamolybdate anion (Fig. 1) is built up of seven distorted MoO_6 octahedra sharing edges and corners, showing the well known butterfly-like shape.

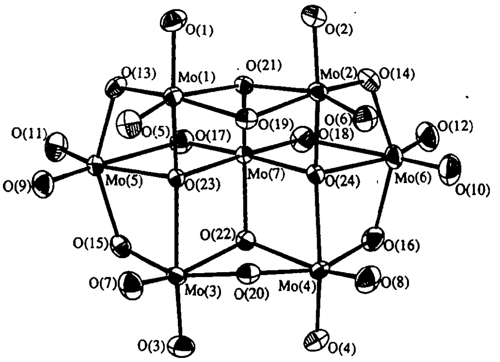

Fig. 1. View of the heptamolybdate(VI) anion showing the atom-labelling scheme.

The Mo—O distances and the O—Mo—O angles are in the ranges previously observed for this polyoxoanion except for the Mo(6)—O(18) bond [2.590 (3) Å] which is unusually long, though comparable with the Mo(5)—O(17) distance in $(H_3dien)_2[Mo_7O_{24}].4H_2O$ (Román, Luque, Aranzabe & Gutiérrez-Zorrilla, 1992). The polyanion shows two types of Mo···Mo distances: (*a*) those between octahedra sharing edges (3.196–3.465 Å) and (*b*) octahedra sharing corners (4.159–4.188 Å). The Mo—O distances can be classified into four types: short, *s* [1.706 (4)–1.745 (3) Å], intermediate, *i* [1.904 (3)–1.999 (3) Å], long, *l* [2.128 (3)–2.311 (3) Å] and very long, *vl* [2.534 (3)–2.590 (3) Å]. In each MoO_6 octahedron we find two distances of each of the types *s*, *i* and *l*, except for the Mo(5) and Mo(6) octahedra where one of the *l* bonds is replaced by a *vl* bond.

A view of the unit cell is shown in Fig. 2. The polyanions are located at two different *y* levels, while the cations and water molecules are placed between these layers of heptamolybdate anions forming an extensive network of hydrogen contacts; the most significant of these are contacts of types: N—H···O, N—H···O*w* [N···O = 2.763 (4)–3.244 (5) Å]; O*w*—H···O, O*w*—H···O*w* [O···O = 2.724 (6)–3.131 (9) Å]; C—H···O, C—H···O*w* [C···O = 3.232 (9)–3.494 (7) Å].

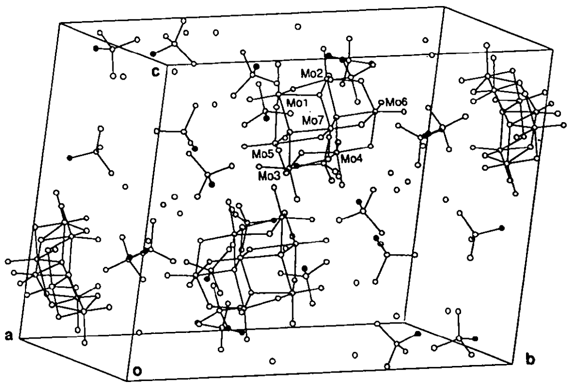

Fig. 2. View of the unit cell for $[(CH_3)_3CNH_3]_6[Mo_7O_{24}].7H_2O$.

Experimental

Crystal data

$(C_4H_{12}N)_6[Mo_7O_{24}].7H_2O$
$M_r = 1626.55$
Monoclinic
$P2_1/n$
$a = 14.023$ (9) Å
$b = 23.232$ (6) Å
$c = 18.212$ (7) Å
$\beta = 102.33$ (6)°
$V = 5796$ (4) Å^3
$Z = 4$
$D_x = 1.864$ Mg m^{-3}
$D_m = 1.85$ (1) Mg m^{-3}
D_m measured by flotation in a $CHBr_3/CCl_4$ mixture

Mo *Kα* radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 8$–$18°$
$\mu = 1.51$ mm^{-1}
$T = 296$ (2) K
Prism
0.40 × 0.20 × 0.20 mm
Colourless

Data collection

CAD-4 diffractometer
ω-2θ scans
Absorption correction: refined from ΔF (*DIFABS*; Walker & Stuart, 1983)
$T_{min} = 0.86$, $T_{max} = 1.39$
10 091 measured reflections
10 091 independent reflections

8698 observed reflections [$I > 3\sigma(I)$]
$\theta_{max} = 25°$
$h = -16 \rightarrow 16$
$k = 0 \rightarrow 27$
$l = 0 \rightarrow 21$
2 standard reflections monitored every 100 reflections
intensity variation: none

Refinement

Refinement on F
$R = 0.037$
$wR = 0.040$
$S = 0.33$
8698 reflections
614 parameters
H-atom parameters not refined
$(\Delta/\sigma)_{\max} = 0.837$
$\Delta\rho_{\max} = 0.64$ e Å^{-3}
$\Delta\rho_{\min} = -0.75$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
Mo(1)	0.91123 (2)	0.07704 (1)	0.33115 (2)	0.0199 (1)
Mo(2)	0.74580 (2)	0.00560 (1)	0.39958 (2)	0.0212 (1)
Mo(3)	0.72526 (2)	0.08000 (1)	0.11807 (2)	0.0223 (1)
Mo(4)	0.56175 (2)	0.01022 (1)	0.18781 (2)	0.0217 (1)
Mo(5)	0.95650 (2)	0.05644 (1)	0.16841 (2)	0.0209 (1)
Mo(6)	0.61312 (2)	−0.09292 (1)	0.30684 (2)	0.0269 (1)
Mo(7)	0.79943 (2)	−0.03703 (1)	0.23059 (2)	0.0170 (1)
O(1)	0.9923 (2)	0.0648 (1)	0.4155 (2)	0.034 (1)
O(2)	0.8307 (2)	−0.0014 (1)	0.4839 (2)	0.034 (1)
O(3)	0.6522 (2)	0.0709 (2)	0.0303 (2)	0.040 (1)
O(4)	0.4989 (2)	0.0036 (1)	0.0953 (2)	0.031 (1)
O(5)	0.9158 (2)	0.1500 (1)	0.3176 (2)	0.037 (1)
O(6)	0.6437 (2)	0.0313 (2)	0.4256 (2)	0.035 (1)
O(7)	0.7503 (2)	0.1523 (1)	0.1245 (2)	0.039 (1)
O(8)	0.4766 (2)	0.0346 (2)	0.2346 (2)	0.040 (1)
O(9)	0.9819 (2)	0.1278 (1)	0.1576 (2)	0.035 (1)
O(10)	0.5177 (2)	−0.0773 (2)	0.3487 (2)	0.044 (1)
O(11)	1.0422 (2)	0.0195 (1)	0.1309 (2)	0.035 (1)
O(12)	0.6166 (3)	−0.1679 (1)	0.3019 (2)	0.042 (1)
O(13)	1.0093 (2)	0.0496 (1)	0.2750 (2)	0.025 (1)
O(14)	0.7185 (2)	−0.0776 (1)	0.3923 (2)	0.030 (1)
O(15)	0.8434 (2)	0.0508 (1)	0.0870 (1)	0.026 (1)
O(16)	0.5522 (2)	−0.0744 (1)	0.2049 (2)	0.031 (1)
O(17)	0.9004 (2)	−0.0447 (1)	0.1902 (2)	0.030 (1)
O(18)	0.7612 (2)	−0.1059 (1)	0.2443 (2)	0.032 (1)
O(19)	0.7882 (2)	0.0784 (1)	0.3648 (2)	0.023 (1)
O(20)	0.6281 (2)	0.0823 (1)	0.1808 (2)	0.025 (1)
O(21)	0.8614 (2)	−0.0153 (1)	0.3318 (1)	0.020 (1)
O(22)	0.7022 (2)	−0.0126 (1)	0.1468 (1)	0.021 (1)
O(23)	0.8320 (2)	0.0587 (1)	0.2204 (1)	0.019 (1)
O(24)	0.6720 (2)	−0.0105 (1)	0.2850 (1)	0.020 (1)
N(11)	0.1900 (3)	0.0629 (2)	0.3814 (2)	0.036 (1)
C(12)	0.2727 (3)	0.0458 (2)	0.3442 (3)	0.033 (1)
C(13)	0.2700 (4)	0.0881 (3)	0.2803 (3)	0.056 (2)
C(14)	0.3678 (4)	0.0497 (2)	0.4024 (3)	0.046 (2)
C(15)	0.2549 (5)	−0.0154 (3)	0.3164 (5)	0.074 (3)
N(21)	0.8566 (3)	0.0627 (2)	0.9362 (2)	0.029 (1)
C(22)	0.9045 (3)	0.1188 (2)	0.9249 (2)	0.033 (1)
C(23)	0.8544 (5)	0.1658 (2)	0.9596 (4)	0.055 (2)
C(24)	0.8937 (5)	0.1264 (3)	0.8408 (3)	0.052 (2)
C(25)	1.0121 (4)	0.1133 (3)	0.9635 (3)	0.055 (2)
N(31)	0.3992 (3)	0.0776 (2)	0.9789 (2)	0.033 (1)
C(32)	0.3343 (3)	0.1109 (2)	1.0192 (3)	0.034 (1)
C(33)	0.4001 (5)	0.1471 (3)	1.0794 (3)	0.059 (2)
C(34)	0.2699 (4)	0.1491 (2)	0.9606 (3)	0.048 (2)
C(35)	0.2744 (4)	0.0677 (3)	1.0526 (4)	0.054 (2)
N(41)	0.0063 (3)	0.2499 (2)	0.1534 (2)	0.039 (1)
C(42)	0.1117 (4)	0.2531 (2)	0.1941 (3)	0.043 (2)
C(43)	0.1692 (5)	0.2156 (3)	0.1498 (4)	0.060 (2)
C(44)	0.1187 (6)	0.2294 (4)	0.2742 (4)	0.072 (2)
C(45)	0.1444 (5)	0.3155 (3)	0.1941 (6)	0.078 (3)
N(51)	0.6816 (3)	0.1688 (2)	0.2896 (2)	0.040 (1)
C(52)	0.6047 (4)	0.1970 (2)	0.3238 (3)	0.042 (1)
C(53)	0.6552 (6)	0.2097 (4)	0.4062 (5)	0.082 (3)
C(54)	0.5247 (5)	0.1547 (4)	0.3244 (5)	0.078 (3)
C(55)	0.5814 (8)	0.2535 (4)	0.2865 (7)	0.137 (6)
N(61)	0.9913 (3)	0.0696 (2)	0.5669 (2)	0.036 (1)
C(62)	0.9673 (3)	0.1301 (2)	0.5871 (2)	0.034 (1)
C(63)	0.9465 (5)	0.1285 (3)	0.6650 (3)	0.058 (2)
C(64)	1.0574 (7)	0.1657 (3)	0.5882 (6)	0.085 (3)
C(65)	0.8800 (7)	0.1510 (3)	0.5305 (4)	0.090 (3)
O(25*w*)	0.2562 (3)	0.1599 (2)	0.4860 (2)	0.056 (1)
O(26*w*)	0.5757 (4)	0.1023 (2)	0.5318 (2)	0.059 (1)
O(27*w*)	0.4601 (3)	0.1997 (2)	0.5095 (3)	0.057 (1)
O(28*w*)	0.6549 (3)	0.0829 (3)	0.8744 (3)	0.067 (2)
O(29*w*)	0.4965 (5)	0.1690 (3)	0.9058 (4)	0.099 (3)
O(30*w*)	0.6612 (5)	0.2338 (3)	1.0041 (5)	0.095 (3)
O(31*w*)	0.3470 (6)	0.2402 (2)	0.8153 (5)	0.106 (3)

Table 2. *Selected geometric parameters* (Å, °)

Mo(1)—O(1)	1.728 (3)	Mo(4)—O(20)	1.932 (3)
Mo(1)—O(5)	1.717 (3)	Mo(4)—O(22)	2.311 (3)
Mo(1)—O(13)	1.987 (3)	Mo(4)—O(24)	2.141 (3)
Mo(1)—O(19)	1.950 (3)	Mo(5)—O(9)	1.716 (3)
Mo(1)—O(21)	2.257 (2)	Mo(5)—O(11)	1.732 (4)
Mo(1)—O(23)	2.128 (3)	Mo(5)—O(13)	1.932 (3)
Mo(2)—O(2)	1.738 (3)	Mo(5)—O(15)	1.932 (3)
Mo(2)—O(6)	1.709 (3)	Mo(5)—O(17)	2.534 (3)
Mo(2)—O(14)	1.969 (3)	Mo(5)—O(23)	2.158 (3)
Mo(2)—O(19)	1.944 (3)	Mo(6)—O(10)	1.714 (4)
Mo(2)—O(21)	2.287 (3)	Mo(6)—O(12)	1.745 (3)
Mo(2)—O(24)	2.155 (3)	Mo(6)—O(14)	1.936 (3)
Mo(3)—O(3)	1.718 (3)	Mo(6)—O(16)	1.920 (3)
Mo(3)—O(7)	1.715 (3)	Mo(6)—O(18)	2.590 (3)
Mo(3)—O(15)	1.982 (3)	Mo(6)—O(24)	2.158 (3)
Mo(3)—O(20)	1.957 (3)	Mo(7)—O(17)	1.741 (3)
Mo(3)—O(22)	2.253 (2)	Mo(7)—O(18)	1.721 (3)
Mo(3)—O(23)	2.184 (3)	Mo(7)—O(21)	1.931 (3)
Mo(4)—O(4)	1.733 (3)	Mo(7)—O(22)	1.904 (3)
Mo(4)—O(8)	1.706 (4)	Mo(7)—O(23)	2.285 (2)
Mo(4)—O(16)	1.999 (3)	Mo(7)—O(24)	2.305 (3)
O(1)—Mo(1)—O(5)	104.7 (2)	O(8)—Mo(4)—O(24)	96.9 (2)
O(1)—Mo(1)—O(13)	90.4 (2)	O(16)—Mo(4)—O(20)	155.6 (2)
O(1)—Mo(1)—O(19)	100.5 (2)	O(16)—Mo(4)—O(22)	85.1 (2)
O(1)—Mo(1)—O(21)	89.1 (2)	O(16)—Mo(4)—O(24)	73.3 (1)
O(1)—Mo(1)—O(23)	157.0 (1)	O(20)—Mo(4)—O(22)	73.8 (2)
O(5)—Mo(1)—O(13)	101.1 (2)	O(20)—Mo(4)—O(24)	88.6 (2)
O(5)—Mo(1)—O(19)	95.3 (2)	O(22)—Mo(4)—O(24)	72.6 (2)
O(5)—Mo(1)—O(21)	163.9 (2)	O(9)—Mo(5)—O(11)	104.8 (2)
O(5)—Mo(1)—O(23)	95.1 (2)	O(9)—Mo(5)—O(13)	98.7 (2)
O(13)—Mo(1)—O(19)	157.4 (1)	O(9)—Mo(5)—O(15)	97.7 (2)
O(13)—Mo(1)—O(21)	87.0 (1)	O(9)—Mo(5)—O(17)	172.8 (2)
O(13)—Mo(1)—O(23)	74.2 (2)	O(9)—Mo(5)—O(23)	103.5 (2)
O(19)—Mo(1)—O(21)	73.6 (1)	O(11)—Mo(5)—O(13)	101.9 (2)
O(19)—Mo(1)—O(23)	89.0 (2)	O(11)—Mo(5)—O(15)	100.9 (2)
O(21)—Mo(1)—O(23)	73.5 (1)	O(11)—Mo(5)—O(17)	82.3 (2)
O(2)—Mo(2)—O(6)	104.1 (2)	O(11)—Mo(5)—O(23)	151.6 (2)
O(2)—Mo(2)—O(14)	93.1 (2)	O(13)—Mo(5)—O(15)	147.4 (2)
O(2)—Mo(2)—O(19)	99.8 (2)	O(13)—Mo(5)—O(17)	80.3 (2)
O(2)—Mo(2)—O(21)	91.5 (2)	O(13)—Mo(5)—O(23)	74.6 (2)
O(2)—Mo(2)—O(24)	159.4 (2)	O(15)—Mo(5)—O(17)	80.0 (2)
O(6)—Mo(2)—O(14)	101.5 (2)	O(15)—Mo(5)—O(23)	74.3 (2)
O(6)—Mo(2)—O(19)	96.9 (2)	O(17)—Mo(5)—O(23)	69.3 (2)
O(6)—Mo(2)—O(21)	162.8 (2)	O(10)—Mo(6)—O(12)	105.5 (3)
O(6)—Mo(2)—O(24)	94.0 (2)	O(10)—Mo(6)—O(14)	98.0 (2)
O(14)—Mo(2)—O(19)	154.2 (2)	O(10)—Mo(6)—O(16)	98.9 (2)
O(14)—Mo(2)—O(21)	84.5 (2)	O(10)—Mo(6)—O(18)	174.4 (1)
O(14)—Mo(2)—O(24)	73.6 (1)	O(10)—Mo(6)—O(24)	105.0 (2)
O(19)—Mo(2)—O(21)	73.0 (2)	O(12)—Mo(6)—O(14)	101.5 (2)
O(19)—Mo(2)—O(24)	87.2 (1)	O(12)—Mo(6)—O(16)	100.7 (2)
O(21)—Mo(2)—O(24)	72.0 (2)	O(12)—Mo(6)—O(18)	80.1 (1)
O(3)—Mo(3)—O(7)	105.0 (2)	O(12)—Mo(6)—O(24)	149.5 (2)
O(3)—Mo(3)—O(15)	93.5 (2)	O(14)—Mo(6)—O(16)	147.3 (2)
O(3)—Mo(3)—O(20)	101.3 (3)	O(14)—Mo(6)—O(18)	80.1 (1)
O(3)—Mo(3)—O(22)	90.7 (2)	O(14)—Mo(6)—O(24)	74.2 (2)
O(3)—Mo(3)—O(23)	158.5 (2)	O(16)—Mo(6)—O(18)	80.4 (1)
O(7)—Mo(3)—O(15)	100.6 (2)	O(16)—Mo(6)—O(24)	74.4 (2)
O(7)—Mo(3)—O(20)	95.2 (2)	O(18)—Mo(6)—O(24)	69.4 (1)
O(7)—Mo(3)—O(22)	163.0 (2)	O(17)—Mo(7)—O(18)	105.9 (2)
O(7)—Mo(3)—O(23)	93.9 (2)	O(17)—Mo(7)—O(21)	101.0 (3)
O(15)—Mo(3)—O(20)	154.8 (1)	O(17)—Mo(7)—O(22)	101.5 (2)
O(15)—Mo(3)—O(22)	84.9 (1)	O(17)—Mo(7)—O(23)	82.5 (2)
O(15)—Mo(3)—O(23)	72.8 (2)	O(17)—Mo(7)—O(24)	170.2 (1)

O(20)—Mo(3)—O(22)	74.8 (1)	O(18)—Mo(7)—O(21)	101.2 (2)
O(20)—Mo(3)—O(23)	86.7 (2)	O(18)—Mo(7)—O(22)	101.6 (2)
O(22)—Mo(3)—O(23)	72.0 (1)	O(18)—Mo(7)—O(23)	171.6 (2)
O(4)—Mo(4)—O(8)	104.5 (3)	O(18)—Mo(7)—O(24)	83.9 (2)
O(4)—Mo(4)—O(16)	91.5 (2)	O(21)—Mo(7)—O(22)	141.9 (1)
O(4)—Mo(4)—O(20)	99.5 (2)	O(21)—Mo(7)—O(23)	76.5 (1)
O(4)—Mo(4)—O(22)	87.5 (2)	O(21)—Mo(7)—O(24)	75.6 (2)
O(4)—Mo(4)—O(24)	155.6 (2)	O(22)—Mo(7)—O(23)	76.5 (1)
O(8)—Mo(4)—O(16)	99.9 (2)	O(22)—Mo(7)—O(24)	77.0 (2)
O(8)—Mo(4)—O(20)	98.2 (2)	O(23)—Mo(7)—O(24)	87.7 (1)
O(8)—Mo(4)—O(22)	166.7 (2)		

Intensity data were corrected for Lp effects. The positions of the seven Mo atoms were obtained by direct methods using *MULTAN*80 (Main *et al.*, 1980) and those of O, N and C atoms from subsequent Fourier syntheses. An absorption correction following the *DIFABS* procedure (Walker & Stuart, 1983) was applied at the conclusion of isotropic refinement. A weighting scheme of type $w = w_1.w_2$ with $w_1 = k_1/(a + b|F_o|)^2$ and $w_2 = k_2/(c + d\sin\theta/\lambda + e\sin^2\theta/\lambda)$ was used to obtain flat dependence in $\langle w\Delta F^2\rangle$ *versus* $\langle F_o\rangle$, and *versus* $\langle\sin\theta/\lambda\rangle$ (*PESOS*; Martínez-Ripoll & Cano, 1975); the coefficients used were $k_1 = 0.497$, $k_2 = 1.013$, $a = 4.916$, $b = -0.099$ for $|F_o| \leq 26.20$; $a = 2.513$, $b = 0.000$ for $26.20 < |F_o| \leq 52.81$; $a = 0.791$, $b = 0.028$ for $52.81 < |F_o| \leq 185.61$; $a = -9.380$, $b = 0.079$ for $|F_o| > 185.61$; $c = 23.116$, $d = -129.978$, $e = 185.590$ for $\sin\theta/\lambda \leq 0.37$; $c = 0.885$, $d = -0.553$, $e = 0.000$ for $\sin\theta/\lambda > 0.37$. Non-H atoms were refined anisotropically. The H atoms were positioned from stereochemical criteria and included as fixed contributors. Most calculations were carried out using the *XRAY*76 system (Stewart *et al.*, 1976) running on a MicroVAX 3800 computer. Geometrical calculations were made with *PARST* (Nardelli, 1983).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71830 (59 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: MU1070]

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2,2′-Bipyrimidine (bpm)-Bridged Dinuclear Complexes. 3. Structure of μ-(2,2′-Bipyrimidine-N^1,$N^{1\prime}$:N^3,$N^{3\prime}$)-bis(tetraaquazinc) Disulfate Dihydrate, $[Zn_2(\mu\text{-bpm})(H_2O)_8](SO_4)_2.2H_2O$

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Abstract

The title compound, $[Zn_2(\mu\text{-}C_8H_6N_4)(H_2O)_8]$.-$(SO_4)_2.2H_2O$, consists of $[Zn_2(\mu\text{-bpm})(H_2O)_8]^{4+}$ dinuclear units (bpm = 2,2′-bipyrimidine), sulfate counter anions and crystallization water molecules. The bpm ligand acts in a bis-chelating fashion and bridges the two metal ions. Each metal ion is in a distorted octahedral environment created by two N atoms from bpm and four O atoms from the water molecules.

Comment

2,2′-Bipyrimidine is a heterocyclic N-donor ligand which has been the subject of great interest in the last