

Rui-Qin Fang and
Xian-Ming Zhang*School of Chemistry & Materials Science, Shanxi
Normal University, Linfen 041004, Shanxi
Province, People's Republic of ChinaCorrespondence e-mail:
zhangxm@dns.sxtu.edu.cn

Key indicators

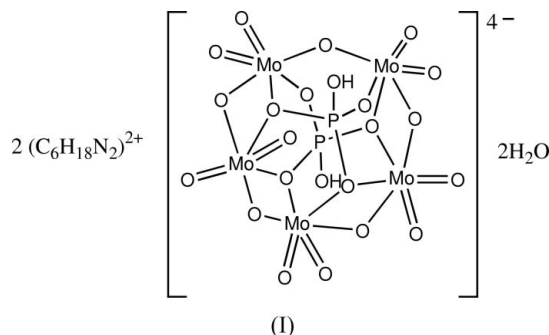
Single-crystal X-ray study
 $T = 298\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.005\text{ \AA}$
 R factor = 0.021
 wR factor = 0.052
Data-to-parameter ratio = 16.3For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.Bis(hexane-1,6-diaminium) di- μ_5 -hydrogen-
phosphato-penta- μ_2 -oxo-pentakis[dioxo-
molybdenum(VI)] dihydrate

The reaction of hexane-1,6-diamine with sodium molybdate and phosphoric acid yields the title salt, $(\text{C}_6\text{H}_{18}\text{N}_2)_2[\text{Mo}_5(\text{HPO}_4)_2\text{O}_{15}]\cdot 2\text{H}_2\text{O}$. Two pentamolybdate anions are linked by hydrogen bonds to form a dimer; the dimers are incorporated into the network structure through hydrogen bonds involving the hexane-1,6-diaminium cations and uncoordinated water molecules.

Received 16 August 2005
Accepted 5 September 2005
Online 14 September 2005

Comment

Pentamolybdates with five-unit MoO_6 octahedra have been reported for pentakis(trioxomolybdophosphate) polyanions in which octahedra 1 and 2, 2 and 3, 3 and 4, and 4 and 5 share an edge, and octahedra 1 and 5 have only one common vertex (Harrison *et al.*, 1997*a,b*). The reaction of 2,5-dimethylpiperazine with phosphoric acid yields the 2,5-dimethylpiperazinium salt of the anionic cluster, which has a hydroxyl group on the P atom (Zhang *et al.*, 2004). With hexane-1,6-diamine as the structure-directing reagent, a similar reaction affords the title pentamolybdate compound, $(\text{C}_6\text{H}_{18}\text{N}_2)_2[\text{Mo}_5\text{O}_{15}(\text{HPO}_4)_2]\cdot 2\text{H}_2\text{O}$ (I) (Fig. 1). The tetraanions are linked *via* hydrogen bonds involving the H atom of the hydrogenphosphate group into a dimer (Fig. 2). The dimers are incorporated into the network structure through hydrogen bonds involving the hexane-1,6-diamine cations and uncoordinated water molecules.



Experimental

A mixture of sodium molybdate dihydrate (0.192 g, 0.88 mmol), hexane-1,6-diamine (0.055 g, 0.47 mmol), 1,10-phenanthroline monohydrate (0.059 g, 0.30 mmol), 85% phosphoric acid (0.17 g, 1.47 mmol) and water (6 ml) in the molar ratio 3:1.6:1:4.9:1100 was sealed in a 15 ml Teflon-lined stainless-steel bomb, which was heated at 468 K for 6 days. The bomb was cooled to room temperature, affording prismatic crystals in 50% yield. Anal. Calc. for $\text{C}_{12}\text{H}_{42}\text{Mo}_5\text{N}_4\text{O}_{25}\text{P}_2$: C 12.17, H 3.58, N 4.73%. Found: C 12.02, H 3.72, N 4.59%.

Crystal data

(C₆H₁₈N₂)₂[Mo₅(HPO₄)₂O₁₅]·2H₂O
M_r = 1184.14
 Triclinic, *P* $\bar{1}$
a = 11.0814 (13) Å
b = 11.9500 (14) Å
c = 14.2178 (17) Å
 α = 103.038 (2)°
 β = 100.388 (2)°
 γ = 103.746 (1)°
V = 1726.4 (4) Å³

Z = 2
D_x = 2.278 Mg m⁻³
 Mo *K*α radiation
 Cell parameters from 3258 reflections
 θ = 2.2–28.3°
 μ = 1.96 mm⁻¹
T = 298 (2) K
 Block, white
 0.30 × 0.30 × 0.19 mm

Data collection

Bruker APEX area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
T_{min} = 0.592, *T_{max}* = 0.708
 18401 measured reflections

7331 independent reflections
 6893 reflections with *I* > 2σ(*I*)
R_{int} = 0.018
 θ_{max} = 27.0°
h = -13 → 14
k = -15 → 15
l = -18 → 18

Refinement

Refinement on *F*²
R [*F*² > 2σ(*F*²)] = 0.021
wR (*F*²) = 0.052
S = 1.06
 7331 reflections
 451 parameters
 H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.021P)^2 + 1.6423P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 ($\Delta\sigma$)_{max} = 0.001
 $\Delta\rho_{max}$ = 0.64 e Å⁻³
 $\Delta\rho_{min}$ = -0.46 e Å⁻³

Table 1 Hydrogen-bond geometry (Å, °).

<i>D</i> — <i>H</i> ··· <i>A</i>	<i>D</i> — <i>H</i>	<i>H</i> ··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> — <i>H</i> ··· <i>A</i>
N4—H4C···O11 ⁱ	0.89	2.05	2.917 (3)	163
N4—H4C···O13 ⁱ	0.89	2.60	3.077 (3)	114
N4—H4C···O7 ⁱ	0.89	2.66	3.207 (3)	121
N4—H4D···O13 ⁱⁱ	0.89	2.07	2.955 (3)	173
N4—H4E···O22	0.89	2.02	2.871 (3)	160
N1—H1C···O9 ⁱⁱⁱ	0.89	2.08	2.961 (3)	171
N1—H1D···O17	0.89	2.01	2.831 (3)	153
N1—H1D···O5	0.89	2.53	3.088 (3)	122
N1—H1E···O10 ⁱ	0.89	1.98	2.871 (3)	175
N2—H2C···O3 ^{iv}	0.89	2.03	2.912 (3)	171
N2—H2C···O1 ^{iv}	0.89	2.62	3.169 (3)	121
N2—H2C···O15 ^{iv}	0.89	2.66	3.099 (3)	112
N2—H2D···O2W ^v	0.89	1.98	2.853 (3)	167
N2—H2E···O15 ^{vi}	0.89	2.12	2.915 (3)	149
N3—H3C···O1W ^{vii}	0.89	2.16	2.940 (3)	146
N3—H3D···O20 ^{viii}	0.89	2.00	2.826 (3)	153
N3—H3E···O21 ⁱⁱⁱ	0.89	2.13	2.987 (3)	163
O2W—H2WA···O18 ^{ix}	0.85 (1)	1.99 (1)	2.835 (3)	174 (4)
O1W—H1WA···O2W ^{iv}	0.85 (1)	1.93 (1)	2.764 (3)	169 (4)
O1W—H1WB···O14 ^v	0.85 (1)	1.93 (1)	2.771 (3)	175 (3)
O2W—H2WB···O9	0.85 (1)	1.94 (1)	2.787 (3)	174 (4)
O1—H1···O1W ^x	0.85	1.82	2.639 (3)	163
O5—H2···O7 ⁱ	0.85	1.81	2.650 (2)	177

Symmetry codes: (i) -*x*+1, -*y*+1, -*z*+1; (ii) *x*, *y*+1, *z*; (iii) *x*-1, *y*, *z*; (iv) *x*, *y*, *z*+1; (v) -*x*+1, -*y*, -*z*+1; (vi) -*x*, -*y*, -*z*+1; (vii) *x*-1, *y*, *z*-1; (viii) -*x*, -*y*+1, -*z*; (ix) *x*+1, *y*, *z*; (x) *x*, *y*, *z*-1.

The water and phosphate H atoms were located in a Fourier map and refined freely. The H atoms belonging to organic groups were placed at calculated positions (N—H = 0.89, C—H = 0.97 Å) and refined using the riding model approximation, with *U_{iso}* = 1.2 or 1.5 times *U_{eq}*(parent atom).

Data collection: SMART (Bruker, 2001); cell refinement: SAINT (Bruker, 2001); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine

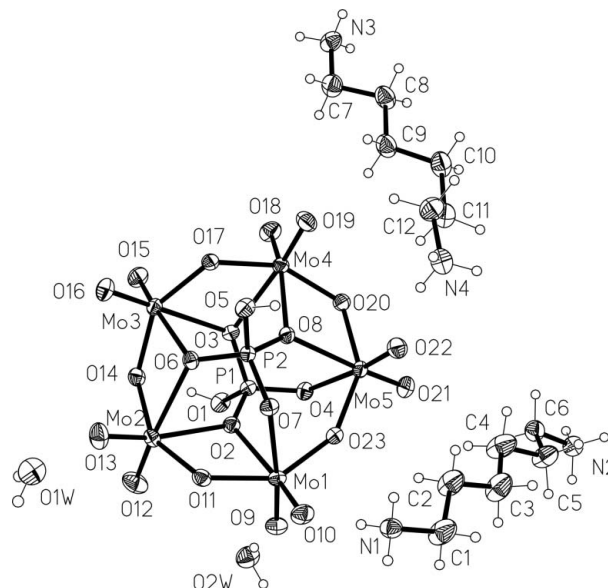


Figure 1 ORTEP (Johnson, 1976) plot of (I), with displacement ellipsoids drawn at the 50% probability level. H atoms are drawn as spheres of arbitrary radii.

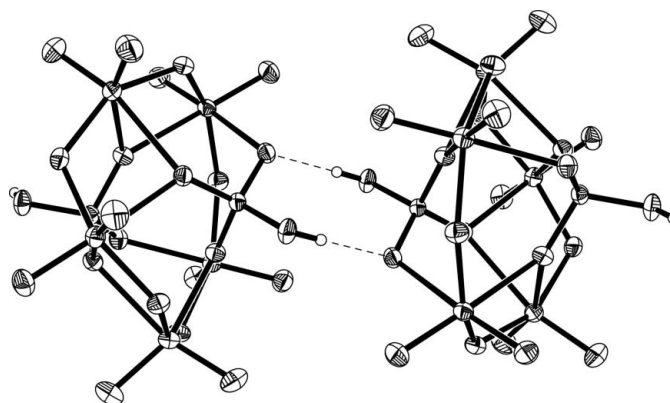


Figure 2 ORTEP (Johnson, 1976) plot illustrating the octamolybdate dimer formed by hydrogen bonds (dashed lines). H atoms are drawn as spheres of arbitrary radii.

structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEP (Johnson, 1976); software used to prepare material for publication: SHELXL97.

This work was financially supported by NSFC (20401011) and Youth Foundation of Shanxi (20041009).

References

Bruker (2001). SADABS, SAINT and SMART. Bruker AXS, Inc., Madison, Wisconsin, USA.
 Harrison, W. T. A., Dussack, L. L. & Jacobson, A. J. (1997a). Acta Cryst. C53, 200–202.
 Harrison, W. T. A., Dussack, L. L. & Jacobson, A. J. (1997b). Acta Cryst. C53, 856–959.
 Johnson, C. K. (1976). ORTEP. Report ORNL-5138. Oak Ridge National Laboratory, Oak Ridge, Tennessee.
 Sheldrick, G. M. (1997). SHELXS97 and SHELXL97. University of Göttingen, Germany.
 Zhang, X.-M., Fang, R.-Q., Wu, H.-S. & Ng, S. W. (2004). Acta Cryst. E60, m171–m173.