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Tris(propane-1,3-diammonium) di-μ_5-phosphato-pentamolybdate(VI) tetrahydrate

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Key indicators

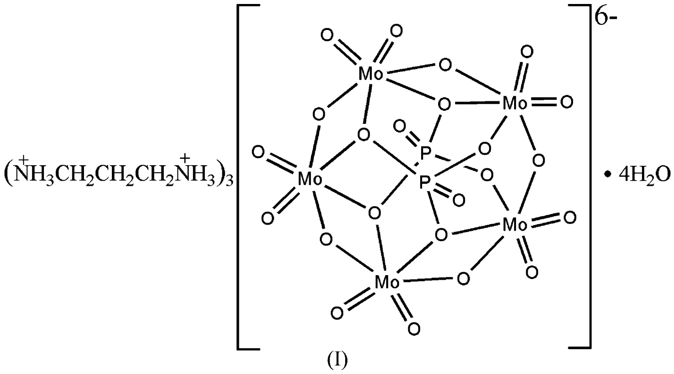
Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.005 Å
R factor = 0.033
wR factor = 0.092
Data-to-parameter ratio = 17.4

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The anions of the title compound, $(C_3H_{12}N_2)[Mo_5O_{15}(PO_4)_2]\cdot 4H_2O$, consist of five MoO_6 octahedra and two PO_4 tetrahedra. The octahedra form a ring. The PO_4 tetrahedra are located above and below the ring and connected to it by three O atoms. Anions and cations are connected through an extensive network of hydrogen bonds

Comment

The design and synthesis of inorganic–organic hybrid materials has provoked significant interest owing to their rich structural chemistry and potential applications in catalysis, biology and electrolytic materials (Sauer & Frébort, 2003; Bissessur & Gallant, 2003). This class of compounds is expected to have interesting chemical and physical properties (Stalick & Quicksall, 1976). The organic cations play an important role, not only in determining the crystal packing, but also in catalytic properties such as particle size, porosity and thermal stability (Aranzabe *et al.*, 1997). We report here the X-ray crystal structure of (I).



(I)

The asymmetric unit of (I) comprises three propanediammonium cations, four water molecules and one diphosphopentamolybdate(VI) anion. The diphosphopentamolybdate anion, $[P_2Mo_5O_{23}]^{6-}$, is built up of five MoO_6 octahedra and two PO_4 tetrahedra. The octahedra form a ring. The two PO_4 tetrahedra are attached to the ring, one above and the other below, each having three O atoms in common with the ring (Fig. 1). Each octahedron has four bridging O atoms and two terminal O atoms. The Mo—O bond lengths can be classified into three groups: (*a*) short, Mo—O_t, terminal O atoms, 1.701 (3)–1.740 (3) Å; (*b*) medium, Mo—O_b, O atoms being shared by two Mo atoms, 1.897 (2)–1.953 (2) Å; (*c*) long, Mo—O_b, O atoms being shared by P and Mo atoms, 2.194 (2)–2.383 (2) Å. The tetrahedral coordination around phosphorus is almost regular, with P—O bond distances

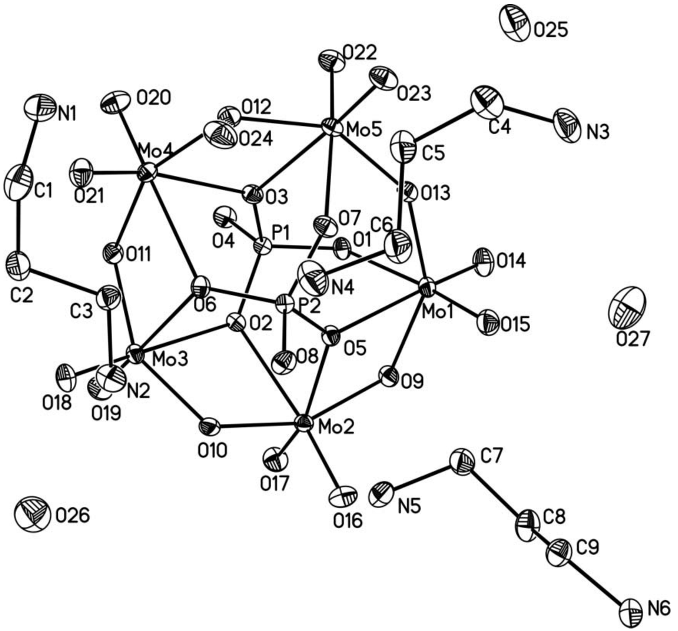


Figure 1
The asymmetric unit of (I), showing 40% probability displacement ellipsoids and the atom-numbering scheme. H atoms have been omitted.

ranging from 1.512 (2) to 1.560 (2) Å. The P—O bond distance to the terminal O atom [P1—O4 = 1.512 (2) Å and P2—O8 = 1.522 (2) Å] is shorter than those involving the O atoms shared by the Mo atoms. This feature is observed in comparable compounds (Aranzabe *et al.*, 1997; Strandberg, 1973).

The propanediammonium cations and water molecules participate in the formation of an extensive network of N—H···O, N—H···O_w, and O_w—H_w···O hydrogen bonds (Table 2).

Experimental

The title compound, (I), was prepared by reacting $Na_2MoO_4\cdot2H_2O$ with $Na_2HPO_4\cdot2H_2O$ and propanediamine (3:1:3) under microwave irradiation. The final pH was adjusted to a value of 5.6 by using HCl (1.0 *M*). Single crystals of (I) suitable for X-ray study were obtained by recrystallizion from water.

Crystal data

$(C_3H_{12}N_2)_3[Mo_5O_{15}(PO_4)_2]\cdot4H_2O$
M_r = 1210.14
Monoclinic, $P2_1/n$
a = 12.971 (3) Å
b = 15.608 (3) Å
c = 16.750 (3) Å
β = 98.398 (3)°
V = 3354.5 (12) Å^3

Z = 4
D_x = 2.396 Mg m^{-3}
Mo *Kα* radiation
μ = 2.02 mm^{-1}
T = 298 (2) K
Block, colourless
0.34 × 0.17 × 0.15 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.547, T_{max} = 0.751

21375 measured reflections
8130 independent reflections
7286 reflections with $I > 2\sigma(I)$
R_{int} = 0.024
θ_{max} = 28.3°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.092
S = 1.01
8130 reflections
466 parameters
H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.064P)^2 + 2.6086P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.72 e Å^{-3}
$\Delta\rho_{min}$ = −2.47 e Å^{-3}

Table 1
Selected bond lengths (Å).

Mo1—O14	1.701 (3)	Mo4—O20	1.711 (3)
Mo1—O15	1.719 (3)	Mo4—O11	1.929 (2)
Mo1—O13	1.931 (2)	Mo4—O12	1.941 (2)
Mo1—O9	1.944 (2)	Mo4—O3	2.224 (2)
Mo1—O1	2.215 (2)	Mo4—O6	2.360 (2)
Mo1—O5	2.383 (2)	Mo5—O23	1.709 (2)
Mo2—O17	1.712 (3)	Mo5—O22	1.740 (3)
Mo2—O16	1.714 (3)	Mo5—O12	1.904 (2)
Mo2—O9	1.919 (2)	Mo5—O13	1.918 (2)
Mo2—O10	1.953 (2)	Mo5—O7	2.229 (2)
Mo2—O5	2.194 (2)	Mo5—O3	2.318 (2)
Mo2—O2	2.347 (2)	O1—P1	1.533 (2)
Mo3—O19	1.713 (2)	O2—P1	1.550 (2)
Mo3—O18	1.732 (2)	O3—P1	1.560 (2)
Mo3—O11	1.897 (2)	O4—P1	1.512 (2)
Mo3—O10	1.909 (2)	O5—P2	1.557 (2)
Mo3—O2	2.290 (2)	O6—P2	1.554 (2)
Mo3—O6	2.312 (2)	O7—P2	1.533 (2)
Mo4—O21	1.708 (3)	O8—P2	1.522 (2)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*A*···O1^{i}	0.89	1.96	2.826 (4)	164
N1—H1*B*···O18^{ii}	0.89	2.17	2.955 (4)	147
N1—H1*C*···O24	0.89	2.06	2.867 (5)	151
N1—H1*C*···O21	0.89	2.54	3.001 (4)	113
N2—H2*A*···O8	0.89	2.08	2.897 (4)	152
N2—H2*B*···O18	0.89	1.96	2.816 (4)	160
N2—H2*C*···O26	0.89	1.99	2.879 (5)	177
N3—H3*A*···O4^{iii}	0.89	1.79	2.674 (4)	175
N3—H3*B*···O25	0.89	1.98	2.773 (5)	148
N3—H3*C*···O27	0.89	2.03	2.900 (5)	164
N4—H4*A*···O13^{iv}	0.89	2.39	3.064 (5)	132
N4—H4*A*···O22^{iv}	0.89	2.53	3.327 (5)	150
N4—H4*B*···O8	0.89	2.35	2.895 (4)	119
N4—H4*B*···O7	0.89	2.53	3.414 (4)	174
N4—H4*C*···O14^{iv}	0.89	2.37	3.125 (5)	143
N5—H5*A*···O8	0.89	1.84	2.716 (4)	167
N5—H5*B*···O22^{iv}	0.89	2.27	3.008 (4)	141
N5—H5*B*···O23^{iv}	0.89	2.35	2.887 (4)	119
N5—H5*C*···O16^{v}	0.89	2.14	2.968 (4)	155
N5—H5*C*···O16	0.89	2.38	2.966 (4)	123
N6—H6*A*···O22^{vi}	0.89	2.34	2.934 (4)	125
N6—H6*B*···O4^{vi}	0.89	2.09	2.780 (4)	134
N6—H6*B*···O3^{vi}	0.89	2.44	3.276 (4)	158
N6—H6*C*···O19^{v}	0.89	2.00	2.867 (4)	163
O24—H1···O7	0.83 (2)	2.27 (2)	3.062 (4)	161 (5)
O24—H2···O9^{i}	0.84 (2)	1.87 (2)	2.700 (4)	172 (6)
O25—H3···O10^{vii}	0.84 (2)	2.01 (3)	2.760 (4)	148 (5)
O25—H4···O23	0.88 (2)	1.96 (2)	2.834 (4)	174 (6)
O26—H5···O7^{iv}	0.83 (2)	2.03 (3)	2.837 (4)	163 (6)
O26—H6···O15^{iv}	0.85 (2)	2.19 (4)	2.907 (4)	142 (6)
O27—H7···O17^{iii}	0.85 (2)	2.10 (3)	2.926 (4)	163 (7)
O27—H8···O20^{vi}	0.86 (2)	1.99 (2)	2.843 (4)	171 (7)

Symmetry codes: (i) $x-\frac{1}{2}, -y+\frac{3}{2}, z-\frac{1}{2}$; (ii) $-x+2, -y+1, -z$; (iii) $x-1, y, z$; (iv) $-x+\frac{3}{2}, y-\frac{1}{2}, -z+\frac{1}{2}$; (v) $-x+2, -y+1, -z+1$; (vi) $x-\frac{1}{2}, -y+\frac{3}{2}, z+\frac{1}{2}$; (vii) $-x+\frac{3}{2}, y+\frac{1}{2}, -z+\frac{1}{2}$.

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H atoms attached to C and N atoms were included in calculated positions, with C—H = 0.97 Å and N—H = 0.89 Å and included in the refinement in the riding-model approximation, with $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C) and 1.5$U_{\rm eq}$(N). H atoms attached to O atoms were located in a difference density map and refined isotropically.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL*.

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