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Tris(ethylenediammonium) penta-μ_2-oxo-decaoxo-di-μ_5-phosphato-pentamolybdate(VI) trihydrate

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In the title compound, $(C_2H_{10}N_2)_3[Mo_5O_{15}(PO_4)_2]\cdot 3H_2O$, the heteropolymolybdate anion adopts its usual structure, consisting of five edge-sharing and corner-sharing distorted MoO_6 octahedra and two corner-sharing PO_4 tetrahedra. A large number of O—H···O and N—H···O hydrogen bonds helps to consolidate the crystal packing.

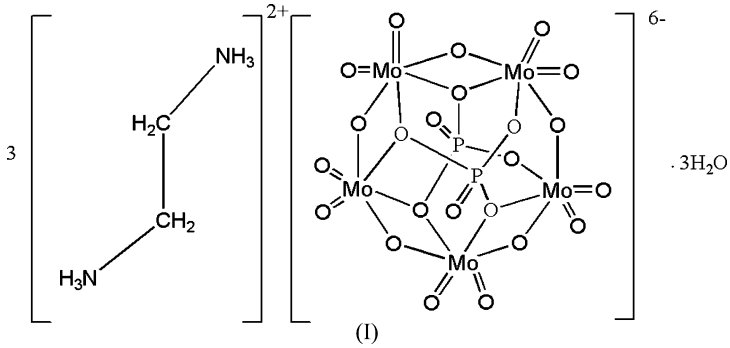
Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.005 Å
Disorder in solvent or counterion
R factor = 0.025
wR factor = 0.069
Data-to-parameter ratio = 11.4

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

Comment

The title compound, (I), arose as a side product during exploratory syntheses in the molybdenum(VI)/vanadium(V)/boron/phosphate system. Compound (I) consists of one heteropolymolybdate anion, three ethylenediammonium cations and three water molecules (Fig. 1). The $[Mo_5O_{23}P_2]^{6-}$ anion is built up from five MoO_6 octahedra and two PO_4 tetrahedra, as described previously (Lin *et al.*, 2002). The octahedra form a five-membered ring by edge- and corner-sharing and the two PO_4 tetrahedra are coordinated above and below the ring. All the Mo centers present a typical distorted octahedral coordination: the Mo—O bond lengths can be classified into three groups: short, 1.709 (3)–1.727 (3) Å; medium, 1.924 (3)–1.937 (3) Å; long, 2.278 (3)–2.386 (3) Å, similar to previously reported complexes containing the same anion (Aranzabe *et al.*, 1997; Lin *et al.*, 2002; Upreti *et al.*, 2005).



(I)

The packing of (I) is shown in Fig. 2. A large number of O—H···O and N—H···O hydrogen bonds help to consolidate the packing, forming a three-dimensional network. Details of these hydrogen bonds are provided in the CIF. In view of the significant disorder in this material, they should be regarded as probable.

Experimental

A mixture of V_2O_5, H_3BO_3, H_3PO_4, $(NH_4)_6Mo_7O_{24}\cdot 4H_2O$, ethylenediamine and water in a ratio of 1:2:2:1:4:404 was stirred for 40 min at room temperature. The mixture was then sealed in a 20 ml

Teflon-lined autoclave and heated at 383 K for 144 h. After cooling, colorless crystals of (I) were recovered.

Crystal data

$(C_2H_{10}N_2)_3[Mo_5O_{15}(PO_4)_2]\cdot 3H_2O$	V = 1520.8 (11) Å^3
M_r = 1150.05	Z = 2
Triclinic, $P\overline{1}$	D_x = 2.511 Mg m^{-3}
a = 9.014 (4) Å	Mo $K\alpha$ radiation
b = 10.699 (4) Å	μ = 2.22 mm^{-1}
c = 17.543 (7) Å	T = 298 (2) K
α = 78.611 (7)°	Block, colorless
β = 80.228 (6)°	0.50 × 0.40 × 0.20 mm
γ = 67.198 (6)°	

Data collection

Bruker SMART 1K CCD area-detector diffractometer	6233 measured reflections
ω scans	5211 independent reflections
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2000)	5075 reflections with $I > 2\sigma(I)$
T_{min} = 0.403, T_{max} = 0.665	R_{int} = 0.019
	θ_{max} = 25.1°

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0123P)^2 + 3.1377P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.026	$(\Delta/\sigma)_{max}$ = 0.003
$wR(F^2)$ = 0.070	$\Delta\rho_{max}$ = 0.64 e Å^{-3}
S = 1.12	$\Delta\rho_{min}$ = −0.71 e Å^{-3}
5211 reflections	Extinction correction: *SHELXL97*
459 parameters	Extinction coefficient: 0.0064 (3)
H-atom parameters constrained	

C– and N-bound H atoms were placed in idealized positions (C—H = 0.97 Å and N—H = 0.89 Å) and refined as riding with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(N). O-bound H atoms were located in difference maps, adjusted to idealized values (O—H = 0.84–0.86 Å) and refined as riding, with U_{iso}(H) = 1.5U_{eq}(O) The N3 and N5 ethylenediamine cations are disordered over two positions. Short contacts imply that the disordering of the two molecules is correlated; they were refined with the same major and minor components of 0.565 (7) and 0.435 (7), respectively.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2000); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2000); molecular graphics: *SHELXTL/PC* (Sheldrick, 1999); software used to prepare material for publication: *SHELXTL/PC*.

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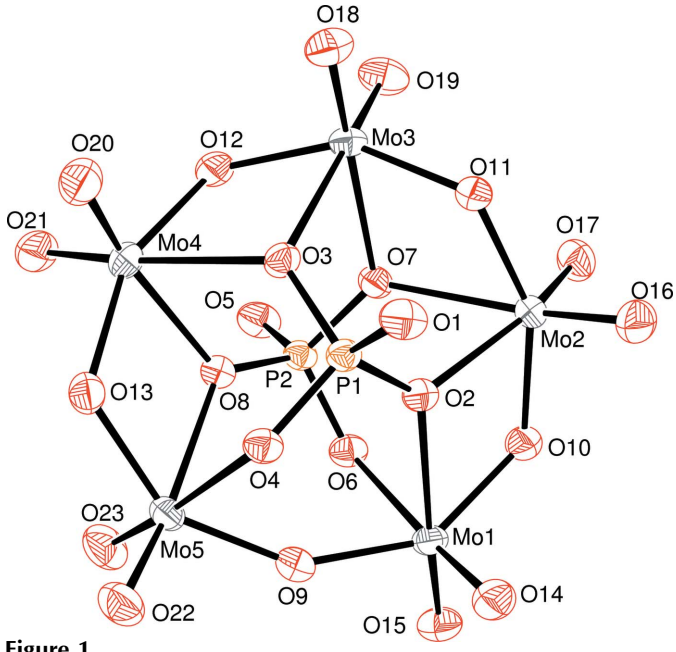

Figure 1
The $[Mo_5O_{23}P_2]^{6-}$ anion in (I), showing 50% displacement ellipsoids.

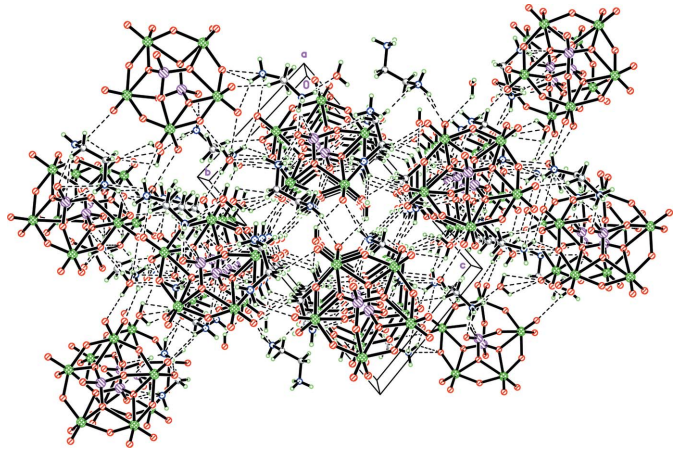

Figure 2
The packing of (I). The minor disorder components have been omitted for clarity. Colour key: O red, N blue, Mo green, P purple, C grey and H green. Dashed lines represent hydrogen bonds.

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