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Tris(ethylenediammonium) bis[(2-aminoethyl)ammonium] bis[bis(μ_5-hydrogen phosphato)penta-μ_2-oxido-decaoxidopentamolybdenum(VI)] decahydrate

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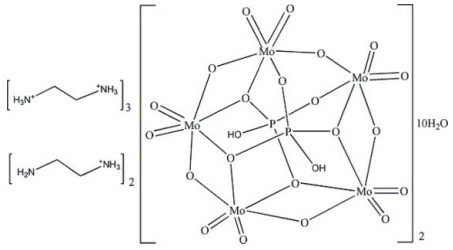
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.012 Å; R factor = 0.041; wR factor = 0.114; data-to-parameter ratio = 13.3.

The title compound, $(C_2H_{10}N_2)_3(C_2H_9N_2)_2[Mo_5(HPO_4)_2O_{15}]\cdot 10H_2O$, was prepared under hydrothermal conditions at pH 5.0. The structure contains mono- and diprotonated ethylenediamine cations, $[Mo_5O_{15}(HPO_4)_2]^{4-}$ anions and uncoordinated water molecules. The $[Mo_5O_{15}(HPO_4)_2]^{4-}$ heteropolyoxometallate anion is made up of five MoO_6 octahedra sharing an edge and forming a ring, which is closed by common corners of the terminal MoO_6 octahedron. The ring is topped on both sides by two slightly distorted PO_4 tetrahedra, sharing three corners with three MoO_6 octahedra. The terminal oxygen atoms of the PO_4 units are protonated. Together with the anions, the water molecules and the ethylenediammonium cations are involved in N—H···O and O—H···O hydrogen bonding, forming a three-dimensional supramolecular network.

Related literature

For background to polyoxometalates, see: Coronado & Gomez-Garcia (1998); Niu *et al.* (2009); Ruether *et al.* (2003). For the structure of $(C_2H_{10}N_2)_2[Mo_5O_{15}(HPO_4)_2]$, see: Sun *et al.* (2003). For structures containing the $[Mo_5O_{15}(PO_4)_2]^{6-}$ anion, see: Gong *et al.* (2006); Skibsted *et al.* (2000). For the bond-valence method, see: Brown (2002).



Experimental

Crystal data

$(C_2H_{10}N_2)_3(C_2H_9N_2)_2[Mo_5(HPO_4)_2O_{15}]\cdot 10H_2O$
M_r = 2312.06
Triclinic, $P\overline{1}$
a = 10.0045 (11) Å
b = 10.6625 (12) Å
c = 15.1884 (19) Å
α = 87.405 (2)°
β = 73.119 (1)°
γ = 77.978 (1)°
V = 1516.2 (3) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 2.23 mm^{-1}
T = 298 K
0.38 × 0.34 × 0.30 mm

Data collection

Siemens SMART CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.485, T_{max} = 0.554
7582 measured reflections
5253 independent reflections
4015 reflections with $I > 2\sigma(I)$
R_{int} = 0.033

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.114
S = 1.04
5253 reflections
396 parameters
5 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.28 e Å^{-3}
$\Delta\rho_{min}$ = −1.07 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N5—H5E···O20^{i}	0.90	2.66	3.075 (8)	109
N5—H5E···O28^{ii}	0.90	2.01	2.796 (9)	144
N5—H5D···O10	0.89	2.45	3.069 (8)	127
N5—H5D···O6	0.89	2.01	2.846 (8)	156
N5—H5C···O21^{i}	0.89	2.17	3.046 (8)	170
N4—H4E···O1^{i}	0.89	1.93	2.806 (8)	167
N4—H4D···O12^{iii}	0.89	2.65	3.357 (8)	137
N4—H4D···O22^{iii}	0.89	2.60	3.099 (8)	116
N4—H4D···O4^{iii}	0.89	2.08	2.907 (8)	155
N4—H4C···O25	0.89	1.92	2.803 (8)	171
N3—H3D···O17^{i}	0.87	2.25	3.117 (8)	176
N3—H3C···O15^{iv}	0.89	1.87	2.732 (7)	162
N2—H2E···O23^{iii}	0.90	2.56	3.030 (8)	113
N2—H2E···O5^{v}	0.90	1.84	2.699 (8)	159
N2—H2D···O20^{v}	0.89	2.14	2.924 (8)	146
N2—H2C···O6^{iii}	0.90	2.49	3.310 (8)	151
N2—H2C···O12^{iii}	0.90	2.35	3.084 (8)	139
N1—H1C···O7^{iv}	0.90	2.46	3.259 (8)	149
N1—H1C···O16^{iv}	0.90	2.28	3.011 (8)	138
N1—H1B···O28^{vi}	0.89	1.93	2.819 (8)	173
N1—H1A···O5^{v}	0.90	1.92	2.772 (8)	159
O28—H28B···O23^{vii}	0.86	2.39	3.157 (8)	149
O28—H28A···O27	0.84	2.31	2.740 (10)	112
O27—H27B···O17^{i}	0.87	2.46	2.912 (10)	113
O27—H27B···O22^{vii}	0.87	2.11	2.916 (10)	155
O27—H27A···O10^{viii}	0.87	2.03	2.875 (9)	163
O26—H26B···O19^{i}	0.84	2.40	3.064 (8)	136
O26—H26B···O17^{i}	0.84	2.36	2.874 (8)	120
O26—H26A···O14	0.84	2.11	2.858 (8)	148
O25—H25B···O21^{i}	0.84	1.97	2.808 (7)	170
O25—H25B···O4^{i}	0.84	2.57	3.083 (7)	120
O25—H25A···O11	0.85	1.93	2.745 (7)	163
O24—H24B···O25^{viii}	0.86	2.08	2.868 (9)	151
O24—H24A···O1^{iv}	0.86	1.97	2.795 (8)	159
O5—H5F···O28^{ii}	0.84	2.02	2.845 (8)	168
O1—H1F···N3^{ix}	0.85	2.18	2.766 (8)	126

Symmetry codes: (i) $x-1, y, z$; (ii) $-x+1, -y+1, -z$; (iii) $-x+1, -y+2, -z+1$; (iv) $-x+1, -y+1, -z+1$; (v) $x-1, y, z+1$; (vi) $x, y, z+1$; (vii) $x-1, y-1, z$; (viii) $x, y-1, z$; (ix) $x+1, y, z$.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve

structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WM2326).

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