

Triammonium hexahydroxidooctadeca-oxidohexamolybdogallate(III) heptahydrate

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Key indicators: single-crystal X-ray study; $T = 173$ K; mean $\sigma(\text{Mo}-\text{O}) = 0.004$ Å; H-atom completeness 19%; R factor = 0.040; wR factor = 0.110; data-to-parameter ratio = 15.7.

The title compound, $(\text{NH}_4)_3[\text{GaMo}_6(\text{OH})_6\text{O}_{18}] \cdot 7\text{H}_2\text{O}$, contains two centrosymmetric GaMo_6 B-type Anderson cluster units consisting of central GaO_6 octahedra surrounded by a hexagonal assembly of MoO_6 edge-sharing octahedra. Like other B-type Anderson clusters, where the central Mo atom is substituted with a di- or trivalent metal ion, the central six μ_3 -oxido bridges are protonated. The average Ga—O bond length is 1.97 (1) Å, whereas the average Mo—O distances are 2.29 (2), 1.94 (1) and 1.709 (5) Å, respectively, for Mo—(μ_3 -OH), Mo—(μ_2 -O) and Mo=O bonds. In the crystal structure, the $\text{Ga}(\mu_3\text{-OH})_6\text{Mo}_6\text{O}_{18}^{3-}$ polyanionic clusters are surrounded by NH_4^+ cations and solvent water molecules, forming an extended network of hydrogen bonds.

Related literature

The gallium-substituted B-type Anderson cluster has been observed previously in solution and the solid state (Rollins & Earley, 1959; Kitazumi *et al.*, 2003), but crystal structures have not been reported. Anderson–Evans clusters are well known and many papers dealing with their preparation have been published (Anderson, 1937; Lorenzo-Luis & Gili, 2000; Lee *et al.*, 2001, and references therein). A similar planar core of seven metals is observed in the recently reported structure of $[\text{Ga}_{13}(\mu_3\text{-OH})_6(\mu_2\text{-OH})_{18}(\text{H}_2\text{O})_{24}](\text{NO}_3)_{15}$ (Rather *et al.*, 2005). Research into this structure led to isolation of the title compound.

Experimental

Crystal data

$(\text{NH}_4)_3[\text{GaMo}_6(\text{OH})_6\text{O}_{18}] \cdot 7\text{H}_2\text{O}$ $b = 10.9651$ (7) Å
 $M_r = 1215.65$ $c = 11.7599$ (8) Å
 Monoclinic, $P2_1/c$ $\beta = 100.2120$ (10)°
 $a = 22.7642$ (15) Å $V = 2888.9$ (3) Å³

$Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 3.56$ mm⁻¹

$T = 173$ (2) K
 $0.38 \times 0.20 \times 0.03$ mm

Data collection

Bruker SMART APEX CCD diffractometer
 Absorption correction: multi-scan (SADABS; Sheldrick, 1995)
 $T_{\text{min}} = 0.345$, $T_{\text{max}} = 0.901$

15163 measured reflections
 6225 independent reflections
 4471 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.020$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.040$
 $wR(F^2) = 0.110$
 $S = 1.17$
 6225 reflections
 397 parameters
 6 restraints

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 1.02$ e Å⁻³
 $\Delta\rho_{\text{min}} = -1.10$ e Å⁻³

Table 1

Selected bond lengths (Å).

Ga1—O3	1.953 (3)	Mo3—O6	1.949 (4)
Ga1—O1	1.954 (4)	Mo3—O3	2.252 (4)
Ga1—O2	1.972 (3)	Mo3—O1 ⁱ	2.286 (4)
Ga2—O15	1.968 (3)	Mo4—O19	1.705 (4)
Ga2—O13	1.969 (3)	Mo4—O20	1.713 (4)
Ga2—O14	1.978 (3)	Mo4—O17	1.939 (3)
Mo1—O7	1.703 (4)	Mo4—O16	1.948 (3)
Mo1—O8	1.711 (4)	Mo4—O13	2.286 (3)
Mo1—O5	1.918 (4)	Mo4—O14	2.324 (3)
Mo1—O4	1.939 (4)	Mo5—O21	1.702 (4)
Mo1—O1	2.293 (4)	Mo5—O22	1.708 (3)
Mo1—O2	2.307 (4)	Mo5—O17	1.932 (4)
Mo2—O9	1.704 (4)	Mo5—O18	1.956 (3)
Mo2—O10	1.714 (4)	Mo5—O15	2.285 (3)
Mo2—O6	1.943 (4)	Mo5—O14	2.284 (3)
Mo2—O5	1.948 (4)	Mo6—O23	1.712 (3)
Mo2—O3	2.287 (4)	Mo6—O24	1.719 (4)
Mo2—O2	2.300 (3)	Mo6—O16 ⁱⁱ	1.917 (4)
Mo3—O12	1.711 (4)	Mo6—O18	1.944 (4)
Mo3—O11	1.715 (4)	Mo6—O15	2.273 (3)
Mo3—O4 ⁱ	1.918 (4)	Mo6—O13 ⁱⁱ	2.290 (3)

Symmetry codes: (i) $-x + 1, -y + 1, -z + 1$; (ii) $-x, -y + 1, -z + 1$.

Table 2

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
O2—H2 ⁱⁱⁱ ···O10 ⁱⁱⁱ	0.84 (2)	2.00 (3)	2.822 (5)	166 (6)
O13—H13 ^{iv} ···O23 ^{iv}	0.84 (2)	1.86 (2)	2.691 (5)	173 (7)
O14—H14 ^v ···O22 ^v	0.84 (2)	1.98 (2)	2.808 (5)	168 (6)
O3—H3 ^{vi} ···O45 ^{vi}	0.85 (2)	1.76 (3)	2.602 (5)	169 (8)
O1—H1 ^{vii} ···O11 ^{vii}	0.85 (2)	1.87 (4)	2.682 (5)	159 (9)
O15—H15 ^{viii} ···O15 ^{viii}	0.83 (2)	1.87 (3)	2.645 (5)	155 (7)

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

Table 3

Short contact geometry (Å).

N1S···O24 ⁱⁱ	2.911 (7)	O2S···O12	2.797 (6)
N1S···O6S ^{vi}	2.784 (8)	O2S···O3S ^{xii}	2.830 (7)
N2S···O16	2.779 (6)	O2S···O6S ^{ix}	2.704 (7)
N2S···O21 ⁱⁱⁱ	2.979 (6)	O3S···O9	3.016 (6)
N2S···O24 ^{vii}	2.901 (6)	O3S···O19	3.034 (7)
N2S···O3S ^{viii}	2.899 (7)	O3S···O5S ^v	2.917 (8)
N2S···O7S	2.898 (7)	O4S···O3 ^{xiii}	2.602 (5)
N3S···O5 ⁱ	2.775 (6)	O4S···O6	2.773 (6)
N3S···O12 ^{xii}	2.907 (7)	O4S···O6 ^{xii}	2.934 (6)
N3S···O5S	2.801 (8)	O5S···O8	2.782 (6)
N3S···O7S ^d	3.030 (8)	O5S···O16 ^d	3.083 (6)
O1S···O18 ^x	2.794 (5)	O6S···O20	2.776 (6)
O1S···O18 ⁱⁱ	2.905 (6)	O6S···O7S	2.769 (7)
O2S···O4 ^{xi}	2.718 (5)	O7S···O7 ^{xiv}	2.973 (6)

Symmetry codes (i): $x, -y + \frac{1}{2}, z - \frac{1}{2}$; (ii): $-x, y - \frac{1}{2}, -z + \frac{1}{2}$; (iii): $x, -y + \frac{3}{2}, z + \frac{1}{2}$; (iv): $x, -y + \frac{1}{2}, z + \frac{1}{2}$; (v): $-x + 1, y + \frac{1}{2}, -z + \frac{3}{2}$; (vi): $x, -y + \frac{3}{2}, z - \frac{1}{2}$; (vii): $-x, y - \frac{1}{2}, -z + \frac{3}{2}$; (viii): $x, -y + \frac{1}{2}, z + \frac{1}{2}$; (ix): $1 - x, 1 - y, 1 - z$; (x): $x, -y + \frac{3}{2}, z - \frac{1}{2}$; (xi): $-x + 1, y - \frac{1}{2}, -z + \frac{1}{2}$; (xii): $-x + 1, -y, -z + 1$; (xiii): $x, -y + \frac{1}{2}, z - \frac{1}{2}$; (xiv): $x, -y + \frac{3}{2}, z + \frac{1}{2}$

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINTE* (Bruker, 2000); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXTL* (Bruker, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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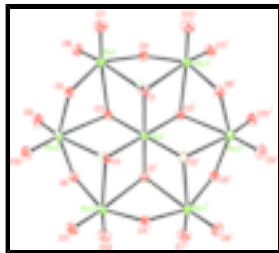
Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WM2161).

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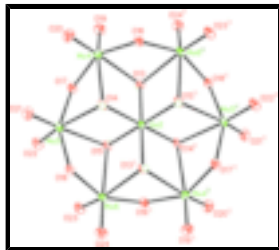
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