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Poly[diammonium diaquahexahydroxyoctadeca-oxodisodiohexamolybdonickelate(II)]

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Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(Na–O) = 0.015 Å
R factor = 0.056
wR factor = 0.098
Data-to-parameter ratio = 14.3

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

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The title compound, $(NH_4)_2[Na_2Ni(OH)_6Mo_6O_{18}(H_2O)_2]$, features inversion-generated hexamolybdonickelate(II) anions (Ni site symmetry $\overline{1}$). The anions are linked by the five-coordinate sodium cations into layers. Adjacent layers are linked through N—H···O and O—H···O hydrogen bonds involving the ammonium ions and water molecules, thus forming a three-dimensional network.

Comment

Varying the inorganic or organic cation employed in the synthesis is an efficient strategy for producing novel polyoxometalates (Liu, Li *et al.*, 2006). To extend our recent work, where hexamolybdonickelate(II) units are linked by cations forming three-dimensional networks (Liu, Zhou *et al.*, 2006), we obtained the title compound, (I), in which the anions of these compounds are isostructural.

The title compound, (I), is composed of $[Ni(OH)_6Mo_6O_{18}]^{4-}$ anionic clusters, sodium and ammonium cations, and water molecules as shown in Fig. 1. The $[Ni(OH)_6Mo_6O_{18}]^{4-}$ cluster adopts a β-type Anderson structure, made up of seven edge-sharing octahedra: six MoO_6 groups arranged hexagonally around the central $Ni(OH)_6$ octahedron (Ni site symmetry $\overline{1}$). The Ni—O and Mo—O distances vary between 2.015 (7)—2.023 (7) Å and 1.659 (7)—2.250 (7) Å, respectively (Table 1). Bond valence sum calculations (Brown & Altermatt, 1985) indicated oxidation states of 5.95–6.07 for the Mo atoms and 1.97 for Ni in good agreement with the expected values of 6 and 2, respectively.

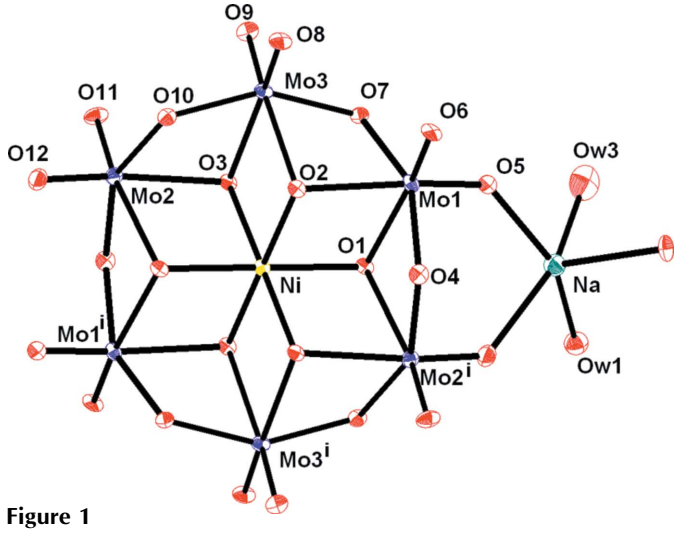

Figure 1
Fragment of (I) showing the polyoxometallate cluster and one attached NaO_5 grouping. Displacement ellipsoids are drawn at the 30% probability level (H atoms omitted for clarity). [Symmetry code: (i) $1-x, -y, -z$.]

The Na^+ cation is bonded to three terminal O atoms from two neighboring $[Ni(OH)_6Mo_6O_{18}]^{4-}$ polyanions and two water molecules, resulting in an NaO_5 square-based pyramid. Thus, the polyanions and Na^+ cations form a 4,4-connected layered structure. These are further linked through ammonium ions and water molecules by hydrogen bonds, thus forming a three-dimensional framework (Fig. 2).

Experimental

$(NH_4)_6Mo_7O_{24}\cdot 6H_2O$ (2 g, 0.16 mmol) and $NiSO_4\cdot 6H_2O$ (0.050 g, 0.2 mmol) were dissolved in water (25 ml) with stirring. NaCl (0.10 g, 1.7 mmol) was then added to the transparent solution and the pH was adjusted to about 2.5–2.6 by adding 4 *M* HCl. The mixture was refluxed at 343 K for about 5 h and then cooled to room temperature; the solution was filtered into a 50 ml beaker. Slow evaporation of the solvent at room temperature led to green block-shaped crystals of (I) suitable for X-ray diffraction after two weeks.

Crystal data

$(NH_4)_2[Na_2Ni(OH)_6Mo_6O_{18}(H_2O)_2]$
M_r = 1214.54
Monoclinic, $P2_1/c$
a = 11.7792 (10) Å
b = 10.9353 (9) Å
c = 11.7793 (10) Å
β = 116.223 (4)°
V = 1361.1 (2) Å^3
Z = 2
D_x = 2.964 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 3.50 mm^{-1}
T = 296 (2) K
Block, light green
0.16 × 0.12 × 0.11 mm

Data collection

Bruker SMART APEX II CCD diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.605, T_{max} = 0.680
7181 measured reflections
2674 independent reflections
1396 reflections with $I > 2\sigma(I)$
R_{int} = 0.036
θ_{max} = 26.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.056
$wR(F^2)$ = 0.098
S = 0.95
2674 reflections
187 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.038P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.75 e Å^{-3}
$\Delta\rho_{min}$ = −0.67 e Å^{-3}

Table 1
Selected bond lengths (Å).

Ni—O1	2.015 (7)	Mo2—O10	1.922 (7)
Ni—O3	2.022 (7)	Mo2—O3	2.204 (7)
Ni—O2	2.023 (7)	Mo2—O1^{i}	2.250 (7)
Ni—O1^{i}	2.015 (7)	Mo3—O8	1.674 (7)
Ni—O3^{i}	2.022 (7)	Mo3—O9	1.702 (7)
Ni—O2^{i}	2.023 (7)	Mo3—O10	1.934 (7)
Mo1—O6	1.659 (7)	Mo3—O7	1.935 (7)
Mo1—O5	1.698 (8)	Mo3—O2	2.193 (7)
Mo1—O4	1.933 (7)	Mo3—O3	2.211 (7)
Mo1—O7	1.937 (7)	Na—O*W*3	2.045 (14)
Mo1—O2	2.196 (7)	Na—O*W*1	2.258 (16)
Mo1—O1	2.209 (7)	Na—O5	2.350 (13)
Mo2—O11	1.684 (8)	Na—O12^{i}	2.389 (13)
Mo2—O12	1.712 (8)	Na—O11^{ii}	2.442 (13)
Mo2—O4^{i}	1.909 (8)		

Symmetry codes: (i) $-x+1, -y, -z$; (ii) $x+1, -y+\frac{1}{2}, z+\frac{1}{2}$.

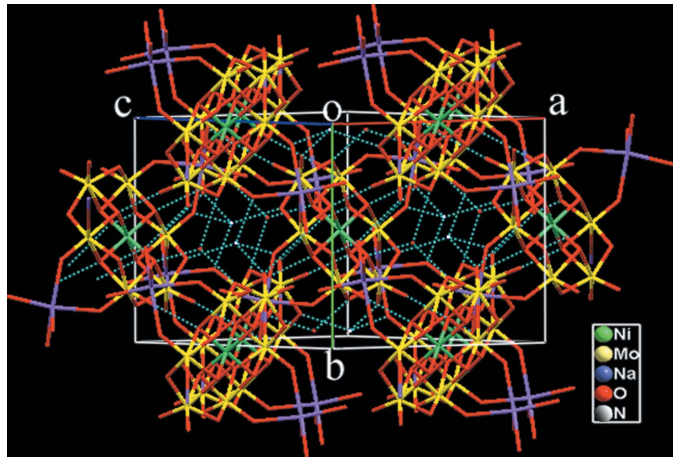

Figure 2
The packing for (I), with hydrogen bonds indicated by dashed lines.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N—HN1···O10	0.84	1.88	2.681 (12)	157
N—HN2···O9^{iii}	0.93	2.03	2.830 (12)	143
N—HN3···O5^{iv}	0.84	2.06	2.883 (12)	164
N—HN4···O12^{v}	0.84	2.01	2.826 (12)	164
O1—H1···O6^{vi}	0.87	1.98	2.847 (10)	169
O2—H2···O*W*2	0.88	1.82	2.649 (11)	156
O3—H3···O8^{vi}	0.87	1.91	2.781 (11)	171
O*W*1—H*W*1*B*···O10^{vii}	0.85	2.34	3.074 (14)	146
O*W*2—H*W*2*B*···O7^{iv}	0.91	2.18	2.864 (10)	131
O*W*3—H*W*3*A*···O9^{viii}	0.86	2.36	2.891 (13)	121
O*W*3—H*W*3*B*···O4^{vi}	0.84	2.13	2.690 (12)	124

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

All the H atoms were located in difference maps and refined in their as-found relative positions (O—H = 0.82–0.91 Å and N—H = 0.84–0.93 Å), using a riding model with U_{iso}(H) = 1.2U_{eq}(O,N).

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 1997); program(s) used to refine structure: *SHELXTL*; molecular graphics: *DIAMOND* (Brandenburg, 1999); software used to prepare material for publication: *SHELXTL*.

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