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## Key indicators

Single-crystal X-ray study  
 $T = 296$  K  
Mean  $\sigma(C-C) = 0.008$  Å  
 $R$  factor = 0.028  
 $wR$  factor = 0.069  
Data-to-parameter ratio = 13.7For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

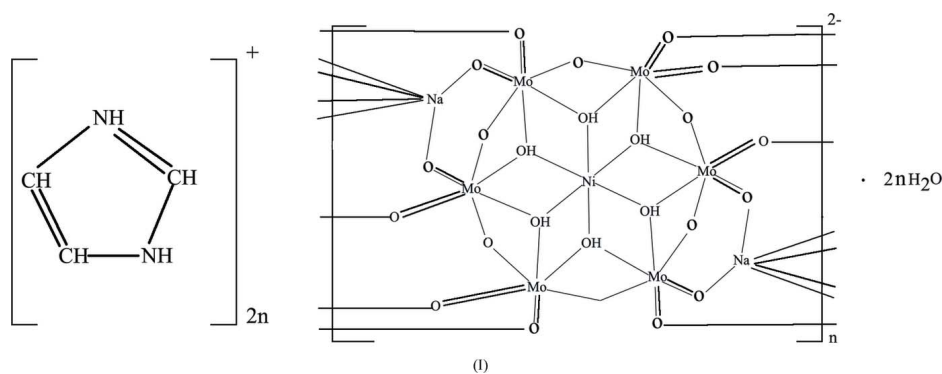
## Poly[bis(imidazolium) hexahydroxooctadeca-oxodisodiohexamolybdonickelate dihydrate]

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The title compound,  $(C_3H_5N_2)_2[NiNa_2Mo_6O_{18}(OH)_6] \cdot 2H_2O$ , features inversion-generated hexamolybdonickelate anions (Ni site symmetry  $\bar{1}$ ). The anions are linked by the octahedrally coordinated sodium cations into a three-dimensional network containing one-dimensional channels. The imidazolium cations are embedded in the channels and interact with the inorganic framework by way of  $N-H \cdots O$  hydrogen bonds.

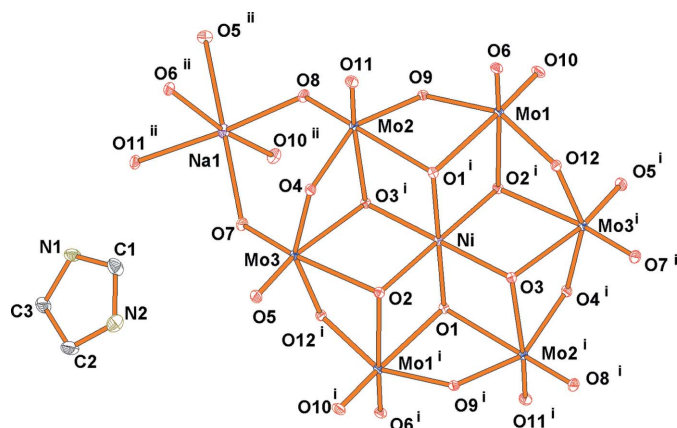
## Comment

The introduction of inorganic or organic cation in the synthesis of polyoxometalate (POM) clusters has been demonstrated as an efficient strategy for obtaining new POM family (Liu, Li *et al.*, 2006). To extend our most recent work, where hexamolybdonickelate units are linked by  $Na^+$  and guanidinium cations forming a three-dimensional network (Liu, Zhou *et al.*, 2006), we obtained the title compound, (I), by the introduction of imidazole into the reaction system, in which the anions of these two compounds are isostructural.



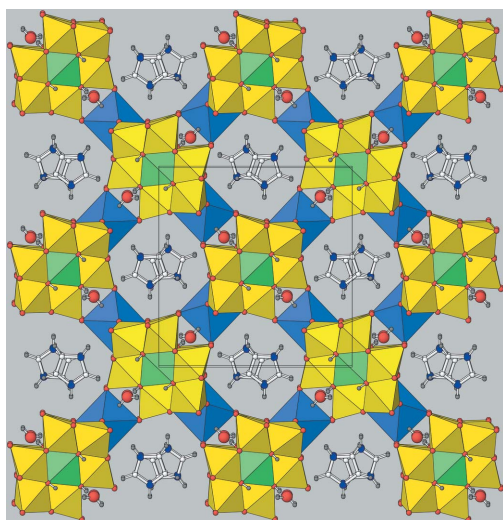
The title compound, (I), is composed of  $[Ni(OH)_6Mo_6O_{18}]^{4-}$  anionic clusters, sodium cations, imidazolium cations and uncoordinated water molecules, as shown in Fig. 1. The  $[Ni(OH)_6Mo_6O_{18}]^{4-}$  cluster adopts a  $\beta$ -type Anderson structure, made up of seven edge-sharing octahedra: six *cis*- $Mo(OH)_2O_4$  groups arranged hexagonally around the central  $Ni(OH)_6$  octahedron (Ni site symmetry  $\bar{1}$ ). The Ni–O and Mo–O distances vary between 2.024 (3)–2.043 (3) Å and 1.705 (3)–2.276 (3) Å, respectively (Table 1). Bond-valence sum calculations (Brown & Altermatt, 1985) yielded BVS values of 5.90–6.04 for the Mo atoms and 1.91 for Ni, in agreement with the expected values of 6 and 2, respectively.

The  $Na^+$  cation is bonded to six terminal O atoms from three neighbouring  $[Ni(OH)_6Mo_6O_{18}]^{4-}$  polyanions to result



**Figure 1**

A fragment of the polymeric structure of (I) showing the metal coordination polyhedra. Displacement ellipsoids are drawn at the 30% probability level, and H atoms and uncoordinated water molecules have been omitted for clarity. [Symmetry codes: (i)  $-x, 1 - y, 1 - z$ ; (ii)  $-x, y - \frac{1}{2}, \frac{3}{2} - z$ .]



**Figure 2**

The packing of (I) viewed down [100] in polyhedral representation. Colour key: Ni(OH)<sub>6</sub> octahedra green, Mo(OH)<sub>2</sub>O<sub>4</sub> yellow, NaO<sub>6</sub> blue, C atoms white, H grey, O red, N blue.

in an NaO<sub>6</sub> octahedron. Thus, the polyanions and Na<sup>+</sup> cations form a three-dimensional framework containing one-dimensional channels (Fig. 2) propagating in [100]. The imidazolium cations occupy the channels and interact with the inorganic framework by way of N—H...O hydrogen bonds. The uncoordinated water molecule interacts with nearby cluster O atoms by way of O—H...O hydrogen bonds and also accepts a similar link from the hydroxyl group of the adjacent [Ni(OH)<sub>6</sub>Mo<sub>6</sub>O<sub>18</sub>]<sup>4-</sup> unit (Table 2).

## Experimental

Na<sub>2</sub>MoO<sub>4</sub>·2H<sub>2</sub>O (1 g, 4 mmol) was dissolved in water (25 ml) with stirring. NiSO<sub>4</sub>·6H<sub>2</sub>O (0.050 g, 0.2 mmol) and imidazole (0.014 g, 0.2 mmol) were added to the mixture and the pH was adjusted to about 2.5–2.6 by adding 6 M HCl. The mixture was refluxed at 353 K for 3 h and then cooled to room temperature. Slow evaporation of the

solvent at room temperature led to prismatic crystals of (I) suitable for X-ray diffraction after three weeks.

### Crystal data

(C<sub>3</sub>H<sub>5</sub>N<sub>2</sub>)<sub>2</sub>[NiNa<sub>2</sub>Mo<sub>6</sub>O<sub>18</sub>(OH)<sub>6</sub>]-  
2H<sub>2</sub>O  
*M<sub>r</sub>* = 1244.57  
Monoclinic, *P*2<sub>1</sub>/*c*  
*a* = 7.1305 (7) Å  
*b* = 14.0039 (14) Å  
*c* = 14.7479 (15) Å  
*β* = 102.874 (1)°

*V* = 1435.6 (2) Å<sup>3</sup>  
*Z* = 2  
*D<sub>x</sub>* = 2.879 Mg m<sup>-3</sup>  
Mo *Kα* radiation  
*μ* = 3.32 mm<sup>-1</sup>  
*T* = 296 (2) K  
Prism, green  
0.25 × 0.21 × 0.14 mm

### Data collection

Bruker SMART APEX-II CCD  
diffractometer  
*φ* and *ω* scans  
Absorption correction: multi-scan  
(SADABS; Bruker, 2002)  
*T<sub>min</sub>* = 0.445, *T<sub>max</sub>* = 0.630

7331 measured reflections  
2814 independent reflections  
2703 reflections with *I* > 2σ(*I*)  
*R<sub>int</sub>* = 0.029  
*θ<sub>max</sub>* = 26.0°

### Refinement

Refinement on *F*<sup>2</sup>  
*R* [*F*<sup>2</sup> > 2σ(*F*<sup>2</sup>)] = 0.028  
*wR* [*F*<sup>2</sup>] = 0.069  
*S* = 1.01  
2814 reflections  
206 parameters  
H-atom parameters constrained

*w* = 1/[σ<sup>2</sup>(*F<sub>o</sub>*<sup>2</sup>) + (0.0206*P*)<sup>2</sup>  
+ 9.957*P*]  
where *P* = (*F<sub>o</sub>*<sup>2</sup> + 2*F<sub>c</sub>*<sup>2</sup>)/3  
(Δ/σ)<sub>max</sub> < 0.001  
Δρ<sub>max</sub> = 0.80 e Å<sup>-3</sup>  
Δρ<sub>min</sub> = -0.92 e Å<sup>-3</sup>  
Extinction correction: SHELXL97  
Extinction coefficient: 0.0231 (5)

**Table 1**

Selected bond lengths (Å).

Ni—O3	2.024 (3)	Mo2—O1 <sup>i</sup>	2.253 (3)
Ni—O2	2.037 (3)	Mo3—O7	1.711 (3)
Ni—O1	2.043 (3)	Mo3—O5	1.722 (3)
Mo1—O10	1.705 (3)	Mo3—O12 <sup>i</sup>	1.934 (3)
Mo1—O6	1.727 (3)	Mo3—O4	1.953 (3)
Mo1—O12	1.915 (3)	Mo3—O3 <sup>i</sup>	2.213 (3)
Mo1—O9	1.947 (3)	Mo3—O2	2.276 (3)
Mo1—O2 <sup>i</sup>	2.225 (3)	Na1—O10 <sup>ii</sup>	2.302 (4)
Mo1—O1 <sup>i</sup>	2.238 (3)	Na1—O6 <sup>iii</sup>	2.363 (4)
Mo2—O11	1.713 (3)	Na1—O11 <sup>iii</sup>	2.386 (4)
Mo2—O8	1.719 (3)	Na1—O7	2.402 (4)
Mo2—O4	1.936 (3)	Na1—O8	2.417 (4)
Mo2—O9	1.953 (3)	Na1—O5 <sup>iv</sup>	2.471 (4)
Mo2—O3 <sup>i</sup>	2.215 (3)		

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

**Table 2**

Hydrogen-bond geometry (Å, °).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
O3—H3...O13W	0.91	1.77	2.659 (5)	166
O13W—H2W...O4 <sup>v</sup>	0.86	2.09	2.725 (5)	130
O13W—H1W...O6 <sup>vi</sup>	0.85	2.14	2.932 (5)	156
O13W—H1W...O10 <sup>vi</sup>	0.85	2.39	2.938 (5)	123
N1—H1N...O9 <sup>iii</sup>	0.86	1.87	2.712 (5)	165
N2—H2N...O6 <sup>v</sup>	0.86	2.14	2.965 (6)	161

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

The C- and N-bound H atoms were positioned geometrically (C—H = 0.93 Å, N—H = 0.86 Å) and refined as riding with *U*<sub>iso</sub>(H) = 1.2*U*<sub>eq</sub>(carrier). The O-bound H atoms were located in difference maps and refined as riding in their as-found relative positions (O—H = 0.80–0.91 Å) with *U*<sub>iso</sub>(H) = 1.2*U*<sub>eq</sub>(O).

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINTE* (Bruker, 2002); data reduction: *SAINTE*; 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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