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Key indicators

Single-crystal X-ray study
 T = 186 K
 Mean $\sigma(\text{C}-\text{C}) = 0.012 \text{ \AA}$
 R factor = 0.035
 wR factor = 0.097
 Data-to-parameter ratio = 17.7

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

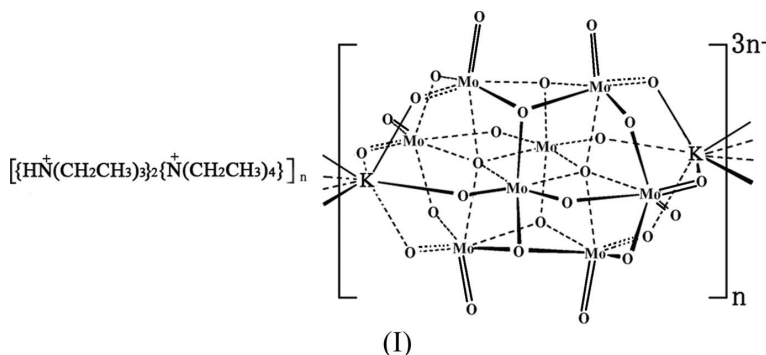
Poly[bis(triethylammonium) tetraethylammonium hexacosaoxopotassiooctamolybdate]

The title compound, $\{(\text{C}_6\text{H}_{16}\text{N})_2(\text{C}_8\text{H}_{20}\text{N})[\text{KM}_8\text{O}_{26}]\}_n$, contains polyanionic zigzag chains comprising centrosymmetric $[\text{Mo}_8\text{O}_{26}]^{4-}$ units linked by K^+ cations which also lie on centres of inversion.

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Comment

Polyoxometalates (POMs) have attracted interest for some time, stimulated by their wide range of topologies and driven by potential applications in catalysis, magnetism, medicinal chemistry and materials science (Hill, 1998; Coronado & Gomez-Garcia, 1998; Yamase, 1998). Introduction of organic ligands into POMs can lead to new types of frameworks and can improve their physicochemical properties.



The title compound, (I) (Fig. 1), consists of $[\text{Mo}_8\text{O}_{26}]^{4-}$ anions linked by K^+ cations into polyanionic chains. The centrosymmetric $[\text{Mo}_8\text{O}_{26}]^{4-}$ unit is constructed from eight linked $\{\text{MoO}_6\}$ octahedra, and can be viewed as two equivalent connected $[\text{Mo}_4\text{O}_{13}]^{2-}$ units. The O atoms can be divided into four sets according to their bonding environment: terminal O with Mo—O distances of 1.688 (4)–1.708 (4) Å, di-bridging O with Mo—O distances of 1.747 (4)–2.288 (4) Å, tri-bridging O with Mo—O distances of 1.945 (4)–2.322 (4) Å and penta-bridging O with Mo—O distances of 2.155 (4)–2.440 (4) Å. All Mo—O distances are within expected values, and bond valence sum calculations (Brown & Altermatt, 1985) suggest that the oxidation states of the Mo atoms are in the range 5.94–5.99, in good agreement with the anticipated value of 6. The K^+ cations lie in approximately cubic environments on centres of inversion [K—O in the range 2.795 (4)–2.844 (5) Å] and link the $[\text{Mo}_8\text{O}_{26}]^{4-}$ anions into zigzag one-dimensional chains running along the *c* axis (Fig. 2). Similar chains, linked by Na^+ cations, have been described recently (Li *et al.*, 2006; Liu *et al.*, 2006), with a linear rather than zigzag geometry. In (I), tetraethylammonium and triethylammonium cations (derived from decomposition of tetraethylammonium) lie

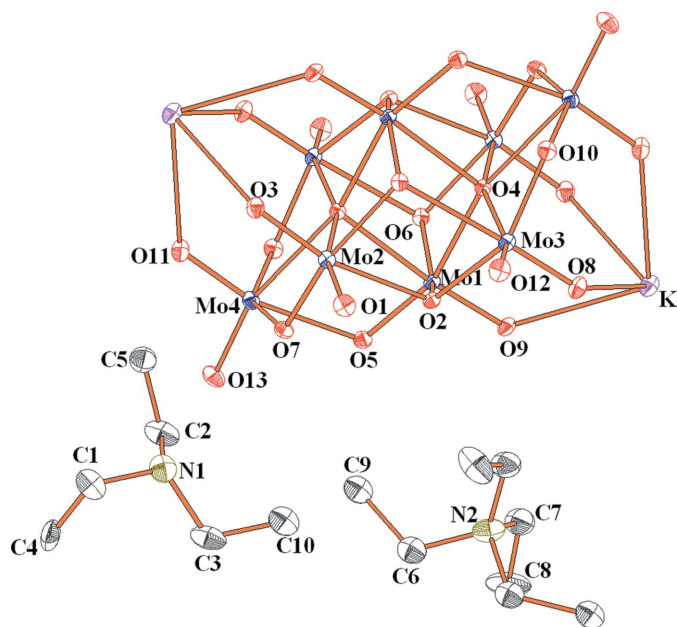


Figure 1

The contents of the asymmetric unit, together with symmetry-generated atoms to complete each structural component, showing displacement ellipsoids at the 50% probability level. H atoms have been omitted. Unlabelled C atoms of the tetraethylammonium cation are generated by $(-x, y, \frac{1}{2} - z)$ and unlabelled atoms of the polyoxoanion are generated by $(-x, 1 - y, -z)$.

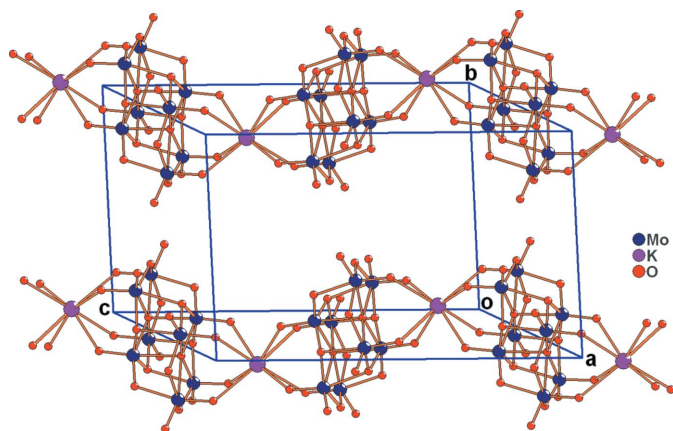


Figure 2

A view of the one-dimensional zigzag chain structure in (I).

between the polyanion chains (Fig. 3), forming N—H...O hydrogen bonds and C—H...O contacts (Table 1).

Experimental

All reagents and solvents were purchased from Siping Chemical Reagent Company and used without further purification. H_2MoO_4 (0.8 g) was dissolved in a 0.2 M KOH (25 ml) solution with stirring. Tetraethylammonium chloride (0.08 g) was added slowly and the pH was adjusted to 4.2–4.3 by addition of 6 M HCl (aq). The resulting slurry was refluxed at 363 K for about 1.5 h and then cooled to room temperature. The filtrate was transferred into a 50 ml beaker, and slow evaporation of the solvent at room temperature yielded crystals of (I) after one week.

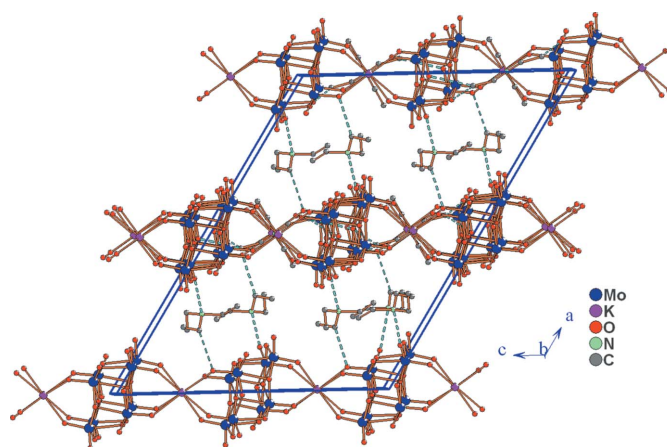


Figure 3

Projection of (I) along the *b* axis. N—H...O and C—H...O interactions are displayed as dashed lines. H atoms have been omitted.

Crystal data

$(\text{C}_6\text{H}_{16}\text{N})_2(\text{C}_8\text{H}_{20}\text{N})[\text{KM}_8\text{O}_{26}]$
 $M_r = 1557.27$
 Monoclinic, $C2/c$
 $a = 25.2665$ (13) Å
 $b = 10.9129$ (6) Å
 $c = 18.7274$ (10) Å
 $\beta = 121.666$ (1)°
 $V = 4395.0$ (4) Å³

$Z = 4$
 $D_x = 2.353$ Mg m⁻³
 Mo $K\alpha$ radiation
 $\mu = 2.39$ mm⁻¹
 $T = 186$ (2) K
 Prism, colourless
 $0.41 \times 0.17 \times 0.10$ mm

Data collection

Bruker SMART APEXII CCD diffractometer
 ω scans
 Absorption correction: multi-scan (SADABS; Bruker, 2002)
 $T_{\min} = 0.633$, $T_{\max} = 0.775$

11643 measured reflections
 4364 independent reflections
 3494 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.027$
 $\theta_{\max} = 26.1^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.035$
 $wR(F^2) = 0.097$
 $S = 1.03$
 4364 reflections
 247 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0459P)^2 + 30.4579P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.73$ e Å⁻³
 $\Delta\rho_{\min} = -0.74$ e Å⁻³

Table 1

Selected bond lengths (Å).

Mo1—O2	1.945 (4)	Mo3—O6 ⁱ	2.322 (4)
Mo1—O4	2.155 (4)	Mo3—O8	1.701 (4)
Mo1—O4 ⁱ	2.331 (4)	Mo3—O10	1.895 (4)
Mo1—O5	1.747 (4)	Mo3—O12	1.688 (4)
Mo1—O6	1.953 (4)	Mo4—O4 ⁱ	2.440 (4)
Mo1—O9	1.692 (4)	Mo4—O5	2.288 (4)
Mo2—O1	1.691 (4)	Mo4—O7	1.918 (4)
Mo2—O2	2.317 (4)	Mo4—O10 ⁱ	1.921 (4)
Mo2—O3	1.706 (4)	Mo4—O11	1.703 (4)
Mo2—O4 ⁱ	2.334 (4)	Mo4—O13	1.708 (4)
Mo2—O6 ⁱ	2.000 (4)	K—O3 ⁱ	2.797 (4)
Mo2—O7	1.898 (4)	K—O8	2.806 (5)
Mo3—O2	2.003 (4)	K—O9	2.795 (4)
Mo3—O4	2.343 (4)	K—O11 ⁱ	2.844 (5)

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

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>
N1—H1...O13	0.91	1.89	2.786 (8)	169
C2—H2A...O3 ^{iv}	0.97	2.77	3.221 (9)	109
C9—H9A...O7 ^v	0.96	2.59	3.192 (14)	121
C9—H9B...O5	0.96	2.55	3.263 (12)	131
C9—H9C...O7	0.96	2.76	3.374 (12)	123

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

Methyl and methylene H atoms were placed in calculated positions and were allowed to ride during subsequent refinement with C—H = 0.96 Å, $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$, and C—H = 0.97 Å, $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$, respectively. The N-bound H atom of the triethylammonium cation was visible in a difference Fourier map, but was placed in a calculated position with N—H = 0.91 Å and allowed to ride during subsequent refinement, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{N})$.

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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