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Bis(tetramethylammonium) hexaaquacobalt(II) β-octamolybdate(VI)

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(N–C) = 0.014 Å
R factor = 0.039
wR factor = 0.105
Data-to-parameter ratio = 13.4

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

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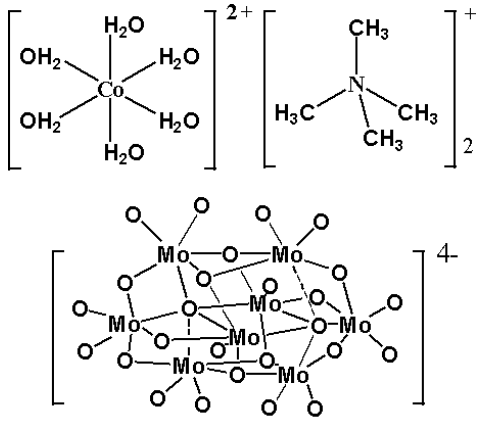
The title compound, $(C_4H_{12}N)_2[Co(H_2O)_6][\beta\text{–}Mo_8O_{26}]$, contains β-octamolybdate ($[\beta\text{-}Mo_8O_{26}]^{4-}$), tetramethylammonium and $[Co(H_2O)_6]^{2+}$ ions. The $[\beta\text{-}Mo_8O_{26}]^{4-}$ anion and $[Co(H_2O)_6]^{2+}$ cations lie on inversion centers, and the tetramethylammonium cations lie on twofold rotation axes.

Comment

More and more chemists are currently interested in topics concerning transition metal oxides (so-called polyoxometalates), owing mainly to their structural variety and promising potential applications in catalysis, biology, medicine and materials science (Wu *et al.*, 2002). Of the various polyoxometalate structures, some of the most interesting are members of the octamolybdate family, with a variety of structural isomers, including α-, β- and γ-octamolybdates *etc.* (Yang *et al.*, 2002).



(I)

In this paper, we report a new octamolybdate compound $(C_4H_{12}N)_2[Co(H_2O)_6][\beta\text{-}Mo_8O_{26}]$. X-ray crystallography shows that the title compound is discrete, consisting of $[\beta\text{-}Mo_8O_{26}]^{4-}$ anions, and tetramethylammonium and $[Co(H_2O)_6]^{2+}$ cations. The framework of this compound is similar to that of $(C_4H_{12}N)_2[Fe(H_2O)_6][Mo_8O_{26}]$ (Do *et al.*, 1999), the most significant differences between the two compounds being observed in the structures of the cations, the color and the space groups. The cobalt-centered cations and the anions lie on inversion centers, and the tetramethylammonium cations lie on twofold rotation axes.

Experimental

A mixture of $Na_2MoO_4{\cdot}2H_2O$ (0.120 g, 0.5 mmol), MoO_3 (0.20 g, 1.38 mmol), $NH_2OH{\cdot}HCl$ (0.15 g, 2.15 mmol), $(CH_3)_4NCl$ (0.11 g, 1.0 mmol), $Co(CH_3COO)_2$ (0.20 g, 1.13 mmol) and water (12 ml) was

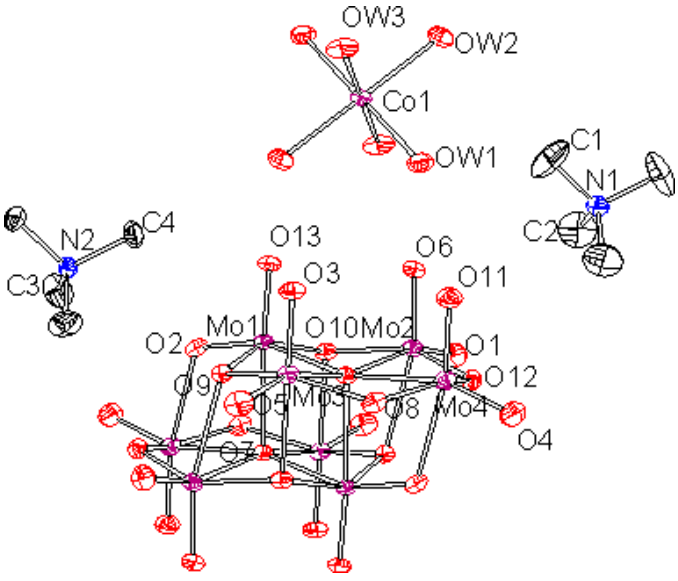


Figure 1
The asymmetric unit of (I), together with the symmetry-related other half of each ion. Displacement ellipsoids are drawn at the 30% probability level and H atoms have been omitted.

sealed in a 20 ml Teflon-lined stainless-steel reactor and heated to 433 K for 48 h. Red crystals of $(C_4H_{12}N)_2[Co(H_2O)_6][\beta\text{–}Mo_8O_{26}]$ suitable for X-ray analysis were obtained after the reaction was cooled to room temperature over a period of 48 h.

Crystal data

$(C_4H_{12}N)_2[Co(H_2O)_6][Mo_8O_{26}]$
M_r = 1498.84
Monoclinic, $C2/c$
a = 16.0338 (10) Å
b = 13.6400 (8) Å
c = 16.6764 (10) Å
β = 96.779 (1)°
V = 3621.6 (4) Å^3
Z = 4
D_x = 2.749 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3856 reflections
θ = 2.0–25.1°
μ = 3.23 mm^{-1}
T = 293 (2) K
Prism, red
0.43 × 0.22 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.421, T_{max} = 0.525
5426 measured reflections
3195 independent reflections
2825 reflections with $I > 2\sigma(I)$
R_{int} = 0.021
θ_{max} = 25.1°
$h = -12 \rightarrow 19$
$k = -16 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.105
S = 1.10
3195 reflections
239 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0357P)^2 + 82.4908P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.018
$\Delta\rho_{max}$ = 0.68 e Å^{-3}
$\Delta\rho_{min}$ = −1.02 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Mo1—O13	1.681 (5)	Mo3—O8	1.898 (5)
Mo1—O2	1.746 (5)	Mo3—O9	2.002 (5)
Mo1—O10	1.950 (5)	Mo3—O7^{i}	2.338 (5)
Mo1—O9	1.956 (5)	Mo3—O10^{i}	2.339 (5)
Mo1—O7^{i}	2.162 (5)	Mo4—O4	1.699 (6)
Mo1—O7	2.348 (5)	Mo4—O11	1.700 (6)
Mo2—O1	1.701 (5)	Mo4—O12	1.926 (5)
Mo2—O6	1.701 (5)	Mo4—O8	1.931 (6)
Mo2—O12	1.882 (5)	Mo4—O2^{i}	2.295 (5)
Mo2—O10	1.992 (5)	Mo4—O7^{i}	2.409 (5)
Mo2—O9^{i}	2.327 (5)	Co1—*OW3*	2.063 (5)
Mo2—O7^{i}	2.350 (5)	Co1—*OW2*	2.076 (5)
Mo3—O5	1.699 (6)	Co1—*OW1*	2.104 (6)
Mo3—O3	1.701 (5)		
O13—Mo1—O2	104.7 (3)	O8—Mo3—O7^{i}	77.1 (2)
O13—Mo1—O10	101.7 (2)	O9—Mo3—O7^{i}	73.54 (18)
O2—Mo1—O10	97.2 (2)	O5—Mo3—O10^{i}	88.2 (2)
O13—Mo1—O9	100.3 (2)	O3—Mo3—O10^{i}	164.0 (2)
O2—Mo1—O9	96.4 (2)	O8—Mo3—O10^{i}	83.9 (2)
O10—Mo1—O9	150.2 (2)	O9—Mo3—O10^{i}	71.56 (18)
O13—Mo1—O7^{i}	98.0 (2)	O7^{i}—Mo3—O10^{i}	71.20 (17)
O2—Mo1—O7^{i}	157.3 (2)	O4—Mo4—O11	105.2 (3)
O10—Mo1—O7^{i}	78.67 (19)	O4—Mo4—O12	101.8 (3)
O9—Mo1—O7^{i}	78.57 (19)	O11—Mo4—O12	98.7 (3)
O13—Mo1—O7	173.8 (2)	O4—Mo4—O8	101.6 (3)
O2—Mo1—O7	81.4 (2)	O11—Mo4—O8	98.4 (3)
O10—Mo1—O7	78.01 (19)	O12—Mo4—O8	146.1 (2)
O9—Mo1—O7	77.93 (18)	O4—Mo4—O2^{i}	88.7 (2)
O7^{i}—Mo1—O7	75.9 (2)	O11—Mo4—O2^{i}	166.0 (2)
O1—Mo2—O6	105.0 (3)	O12—Mo4—O2^{i}	78.0 (2)
O1—Mo2—O12	101.5 (3)	O8—Mo4—O2^{i}	78.4 (2)
O6—Mo2—O12	101.1 (3)	O4—Mo4—O7^{i}	159.0 (2)
O1—Mo2—O10	101.2 (3)	O11—Mo4—O7^{i}	95.7 (2)
O6—Mo2—O10	96.9 (2)	O12—Mo4—O7^{i}	74.59 (19)
O12—Mo2—O10	146.1 (2)	O8—Mo4—O7^{i}	74.78 (19)
O1—Mo2—O9^{i}	89.5 (2)	O2^{i}—Mo4—O7^{i}	70.28 (17)
O6—Mo2—O9^{i}	163.4 (2)	*OW3*^{ii}—Co1—*OW3*	180
O12—Mo2—O9^{i}	83.4 (2)	*OW3*^{ii}—Co1—*OW2*^{ii}	88.9 (2)
O10—Mo2—O9^{i}	71.99 (19)	*OW3*—Co1—*OW2*^{ii}	91.1 (2)
O1—Mo2—O7^{i}	160.7 (2)	*OW3*^{ii}—Co1—*OW2*	91.1 (2)
O6—Mo2—O7^{i}	94.1 (2)	*OW3*—Co1—*OW2*	88.9 (2)
O12—Mo2—O7^{i}	76.8 (2)	*OW2*^{ii}—Co1—*OW2*	180
O10—Mo2—O7^{i}	73.42 (18)	*OW3*^{ii}—Co1—*OW1*	94.1 (3)
O9^{i}—Mo2—O7^{i}	71.21 (16)	*OW3*—Co1—*OW1*	85.9 (3)
O5—Mo3—O3	105.3 (3)	*OW2*^{ii}—Co1—*OW1*	89.7 (2)
O5—Mo3—O8	101.5 (3)	*OW2*—Co1—*OW1*	90.3 (2)
O3—Mo3—O8	101.5 (3)	*OW3*^{ii}—Co1—*OW1*^{ii}	85.9 (3)
O5—Mo3—O9	100.3 (3)	*OW3*—Co1—*OW1*^{ii}	94.1 (3)
O3—Mo3—O9	97.1 (2)	*OW2*^{ii}—Co1—*OW1*^{ii}	90.3 (2)
O8—Mo3—O9	146.4 (2)	*OW2*—Co1—*OW1*^{ii}	89.7 (2)
O5—Mo3—O7^{i}	159.4 (2)	*OW1*—Co1—*OW1*^{ii}	180
O3—Mo3—O7^{i}	95.1 (2)		

Symmetry codes: (i) $\frac{1}{2}-x,\frac{3}{2}-y,1-z$; (ii) $\frac{1}{2}-x,\frac{5}{2}-y,1-z$.

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

D—H···A	D—H	H···A	D···A	D—H···A
OW1—*HW1A*···O11	0.82	2.08	2.819 (8)	150
OW1—*HW1B*···O4^{iii}	0.82	2.10	2.852 (9)	153
OW2—*HW2A*···O13^{ii}	0.82	2.14	2.897 (7)	152
OW2—*HW2A*···O4^{iii}	0.82	2.42	2.916 (8)	120
OW2—*HW2B*···O3^{ii}	0.82	2.20	2.859 (8)	138
OW2—*HW2B*···O5^{iv}	0.82	2.28	2.889 (8)	131
OW3—*HW3B*···O6^{ii}	0.82	2.02	2.764 (8)	151
OW3—*HW3A*···O1^{v}	0.82	2.03	2.822 (8)	163

Symmetry codes: (ii) $\frac{1}{2}-x,\frac{5}{2}-y,1-z$; (iii) $\frac{1}{2}-x,\frac{1}{2}+y,\frac{3}{2}-z$; (iv) $\frac{1}{2}+x,\frac{1}{2}+y,z$; (v) $x-\frac{1}{2},\frac{1}{2}+y,z$.

All H atoms were placed at calculated positions (C—H = 0.96 Å and O—H = 0.8184–0.8200 Å) and treated using a riding model. Isotropic displacement parameters were refined for water H atoms and constrained for C-bound H atoms [U_{iso}(H) = 1.5U_{eq}(C)]. The aqua H atoms were located from difference maps and refined freely. In the final diffence map, the −1.020 Å^{-3} hole is 0.84 Å from atom Mo4 atom and the 0.680 Å^{-3} peak is 0.81 Å from atom Mo2.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SMART* and *SAINT* (Siemens, 1994); data reduction: *SAINT* and *XPREP* in *SHELXTL* (Siemens, 1994); program(s) used to solve structure: *SHELXTL*; program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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