Crystal structure of diammonium bis[dimethyl(2-hydroxyethyl)ammonium octamolybdate diwater, $(C_4H_{12}NO)_2(NH_4)_2[Mo_8O_{26}] \cdot 2H_2O$

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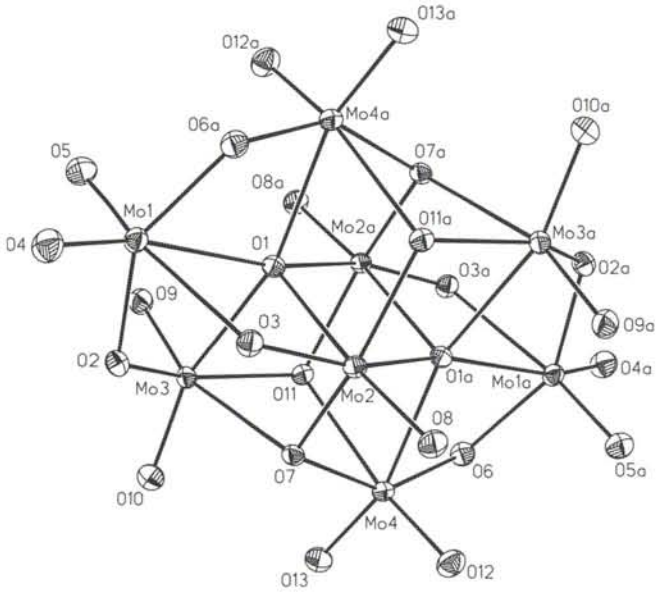

Abstract

$C_8H_{36}Mo_8N_4O_{30}$, triclinic, $P\bar{1}$ (No. 2), $a = 7.719(2)$ Å, $b = 9.693(2)$ Å, $c = 11.687(2)$ Å, $\alpha = 80.95(3)°$, $\beta = 81.75(3)°$, $\gamma = 84.59(3)°$, $V = 852.3$ Å^3, $Z = 1$, $R_{gt}(F) = 0.027$, $wR_{ref}(F^2) = 0.070$, $T = 293$ K.

Source of material

The compound was obtained with treatment of 2-dimethylaminoethanol, ammoniumheptamolybdate tetrahydrate and nitric acid in methanol solution.

Discussion

Every molybdenum atom is surrounded by six oxygen atoms. The geometric parameters in an octamolybdate group are very similar to those reported in [1]. The hydrogen bonds between the dimethyl(2-hydroxyethyl)ammonium cations, octamolybdate groups and the ammonium ions form a three-dimensional network where the donor and acceptor atom distances in N—H···O and O—H···O environments vary between 2.868 Å and 3.005 Å.

Table 1. Data collection and handling.

Crystal:	colourless, irregular, size 0.20 × 0.20 × 0.25 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	29.53 cm^{-1}
Diffractometer, scan mode:	Nicolet P3, ω/2θ
$2\theta_{max}$:	54°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	3692, 3692
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 3135
$N(param)_{refined}$:	232
Programs:	SHELXS-97 [2], SHELXL-97 [3], SHELXTL/PC [4]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(14)	2*i*	0.3549	0.1698	0.1660	0.048
H(2)	2*i*	0.682(9)	0.134(7)	0.396(6)	0.072
H(1)	2*i*	0.551(9)	0.215(7)	0.359(6)	0.072
H(1A)	2*i*	0.3165	0.8667	0.3409	0.047
H(1B)	2*i*	0.4604	0.9229	0.3849	0.047
H(1C)	2*i*	0.4941	0.7964	0.3283	0.047
H(1D)	2*i*	0.4573	0.9336	0.2586	0.047
H(2A)	2*i*	0.6966	0.1932	0.1787	0.037
H(1E)	2*i*	0.4491	0.2865	0.0142	0.039
H(1F)	2*i*	0.4587	0.1517	–0.0464	0.039
H(2B)	2*i*	0.7304	0.0784	0.0168	0.035
H(2C)	2*i*	0.7392	0.2254	–0.0611	0.035
H(3A)	2*i*	0.5912	0.4111	0.1047	0.072
H(3B)	2*i*	0.7780	0.4356	0.0356	0.072
H(3C)	2*i*	0.7507	0.4175	0.1725	0.072
H(4A)	2*i*	0.9846	0.2354	0.1728	0.073
H(4B)	2*i*	1.0081	0.2345	0.0375	0.073
H(4C)	2*i*	0.9664	0.0969	0.1234	0.073

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Mo(1)	2*i*	0.08198(4)	0.21762(3)	0.73161(3)	0.0183(2)	0.0202(2)	0.0211(2)	–0.0010(1)	–0.0025(1)	0.0007(1)
Mo(2)	2*i*	0.20860(3)	0.51191(3)	0.54616(2)	0.0121(1)	0.0178(2)	0.0198(2)	–0.0020(1)	–0.0025(1)	–0.0027(1)
Mo(3)	2*i*	0.06982(4)	0.21828(3)	0.45530(3)	0.0177(2)	0.0155(1)	0.0210(2)	–0.0012(1)	–0.0012(1)	–0.0030(1)
Mo(4)	2*i*	0.19533(4)	0.50929(3)	0.27149(3)	0.0168(2)	0.0196(2)	0.0188(2)	–0.0014(1)	0.0007(1)	–0.0016(1)
O(1)	2*i*	–0.0380(3)	0.3808(2)	0.5790(2)	0.015(1)	0.019(1)	0.021(1)	0.0002(9)	–0.0017(9)	–0.0024(9)

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Table 3. Continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(2)	2*i*	0.1903(3)	0.1570(3)	0.5860(2)	0.021(1)	0.021(1)	0.025(1)	0.002(1)	–0.004(1)	–0.002(1)
O(3)	2*i*	0.2741(3)	0.3854(3)	0.6576(2)	0.017(1)	0.022(1)	0.027(1)	–0.0031(9)	–0.004(1)	–0.001(1)
O(4)	2*i*	0.2203(4)	0.1354(3)	0.8258(2)	0.030(1)	0.034(2)	0.029(1)	–0.000(1)	–0.006(1)	0.002(1)
O(5)	2*i*	–0.0937(3)	0.1195(3)	0.7573(2)	0.025(1)	0.031(1)	0.031(2)	–0.005(1)	–0.001(1)	–0.003(1)
O(6)	2*i*	0.0232(3)	0.6258(3)	0.1940(2)	0.025(1)	0.026(1)	0.019(1)	–0.002(1)	–0.004(1)	–0.002(1)
O(7)	2*i*	0.2567(3)	0.3967(2)	0.4201(2)	0.013(1)	0.019(1)	0.023(1)	–0.0006(9)	–0.0005(9)	–0.0038(9)
O(8)	2*i*	0.3693(3)	0.6235(3)	0.5120(2)	0.017(1)	0.027(1)	0.033(1)	–0.006(1)	–0.003(1)	–0.003(1)
O(9)	2*i*	–0.1169(3)	0.1306(3)	0.4884(2)	0.024(1)	0.022(1)	0.034(2)	–0.004(1)	–0.002(1)	–0.003(1)
O(10)	2*i*	0.1972(4)	0.1418(3)	0.3486(2)	0.031(1)	0.027(1)	0.030(1)	0.000(1)	0.001(1)	–0.006(1)
O(11)	2*i*	–0.0392(3)	0.3806(2)	0.3550(2)	0.018(1)	0.020(1)	0.017(1)	–0.0029(9)	–0.0018(9)	–0.0031(9)
O(12)	2*i*	0.3623(3)	0.6168(3)	0.2484(2)	0.022(1)	0.030(1)	0.034(2)	–0.007(1)	–0.003(1)	0.001(1)
O(13)	2*i*	0.2573(4)	0.3898(3)	0.1778(2)	0.033(2)	0.030(1)	0.025(1)	–0.000(1)	0.003(1)	–0.007(1)
O(14)	2*i*	0.4169(4)	0.1126(3)	0.1261(3)	0.038(2)	0.041(2)	0.037(2)	–0.005(1)	0.010(1)	–0.008(1)
OW(1)	2*i*	0.6053(5)	0.1426(4)	0.3475(3)	0.050(2)	0.060(2)	0.039(2)	–0.016(2)	–0.011(2)	–0.011(2)
N(1)	2*i*	0.4320(4)	0.8798(4)	0.3281(3)	0.025(2)	0.032(2)	0.036(2)	–0.006(1)	–0.000(1)	–0.004(2)
N(2)	2*i*	0.7530(4)	0.2347(4)	0.1099(3)	0.029(2)	0.038(2)	0.023(2)	–0.009(1)	–0.002(1)	0.002(1)
C(1)	2*i*	0.4923(5)	0.1887(4)	0.0191(3)	0.036(2)	0.036(2)	0.027(2)	–0.004(2)	–0.007(2)	–0.006(2)
C(2)	2*i*	0.6890(5)	0.1764(4)	0.0134(3)	0.033(2)	0.029(2)	0.025(2)	–0.003(2)	0.001(2)	–0.003(2)
C(3)	2*i*	0.7149(7)	0.3883(5)	0.1053(5)	0.055(3)	0.044(3)	0.049(3)	–0.010(2)	–0.008(2)	–0.014(2)
C(4)	2*i*	0.9453(6)	0.1970(6)	0.1110(5)	0.033(2)	0.059(3)	0.053(3)	–0.009(2)	–0.013(2)	0.004(2)

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