Bingchuan Yang*, Xueyan Lv and Rutao Liu

The crystal structure of tetrakis(1-isopropyl-1*H*-imidazolium) octamolybdate, $C_{24}H_{44}Mo_8N_8O_{26}$

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.22 × 0.20 × 0.19 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	2.16 mm ⁻¹
Diffractometer, scan mode:	Bruker SMART APEX II, φ and ω
$\theta_{\max}$, completeness:	25.0°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	11,330, 4105, 0.051
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2\sigma(I_{\text{obs}})$, 3229
$N(\text{param})_{\text{refined}}$:	302
Programs:	BRUKER [1], SHELX [2]

Abstract

$C_{24}H_{44}Mo_8N_8O_{26}$, monoclinic, $P2_1/n$ (no. 14), $a = 10.3214(11)$ Å, $b = 21.810(2)$ Å, $c = 10.9371(11)$ Å, $V = 2340.4(4)$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.0423$, $wR_{\text{ref}}(F^2) = 0.0893$, $T = 298(2)$ K.

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Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

A mixture of ammonium molybdate (0.25 mmol), 1-isopropylimidazole (2.0 mmol) in H₂O (15 mL) was stirred for 60 min at room temperature. Then the solution was sealed in a 25 mL Teflon-lined autoclave at 100 °C for three days. After the reaction was completed, the block crystals were obtained. Yield: 37.6%. **Anal. Calcd** for $C_{24}H_{44}Mo_8N_8O_{26}$: C, 17.70; H, 2.72; N, 6.88; found: C, 17.92; H, 2.64; N, 6.63.

Experimental details

Hydrogen atoms were assigned with common isotropic displacement factors $U_{\text{iso}}(\text{H}) = 1.2$ times U_{eq} (C, imidazole ring and methanetriyl carbon) and $U_{\text{iso}}(\text{H}) = 1.5$ times U_{eq} (C, methyl carbon). All the H atoms were refined as riding on their parent atom.

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***Corresponding author: Bingchuan Yang**, School of Environmental Science and Engineering, Shandong University, Qingdao 266237, China; and School of Chemistry and Chemical Engineering, Liaocheng University, Liaocheng 252000, Shandong, China, E-mail: yangbingchuan@lcu.edu.cn. <https://orcid.org/0000-0003-1195-5833>

Xueyan Lv, School of Environmental Science and Engineering, Shandong University, Qingdao 266237, China; School of Chemistry and Chemical Engineering, Liaocheng University, Liaocheng 252000, Shandong, China

Rutao Liu, School of Environmental Science and Engineering, Shandong University, Qingdao 266237, China

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($\text{\AA}^2$).

Atom	x	y	z	U_{iso}^*/U_{eq}
C1	0.9294 (8)	0.4192 (4)	0.1540 (8)	0.048 (2)
H1	1.017262	0.417630	0.211551	0.057*
C2	0.8211 (9)	0.4426 (5)	0.1782 (9)	0.058 (3)
H2	0.818436	0.459938	0.254995	0.070*
C3	0.7589 (9)	0.4089 (5)	−0.0176 (9)	0.056 (3)
H3	0.705776	0.398919	−0.100715	0.067*
C4	0.9822 (9)	0.3704 (4)	−0.0352 (9)	0.050 (2)
H4	1.055829	0.348749	0.028821	0.060*
C5	1.0446 (12)	0.4225 (5)	−0.0894 (13)	0.094 (4)
H5A	0.973395	0.447891	−0.142947	0.142*
H5B	1.098033	0.406104	−0.139595	0.142*
H5C	1.101831	0.446595	−0.020102	0.142*
C6	0.9069 (11)	0.3247 (4)	−0.1352 (9)	0.069 (3)
H6A	0.861853	0.295461	−0.096868	0.103*
H6B	0.970331	0.303889	−0.168979	0.103*
H6C	0.840541	0.345777	−0.203670	0.103*
C7	0.1229 (9)	0.6633 (4)	0.5614 (9)	0.049 (2)
H7	0.207674	0.660072	0.548753	0.059*
C8	0.0163 (8)	0.6255 (4)	0.5140 (8)	0.048 (2)
H8	0.012559	0.591634	0.461441	0.058*
C9	−0.0433 (8)	0.6955 (4)	0.6279 (9)	0.046 (2)
H9	−0.094966	0.718005	0.668154	0.055*
C10	0.1657 (9)	0.7610 (4)	0.6927 (9)	0.055 (3)
H10	0.114919	0.783012	0.741239	0.067*
C11	0.1838 (13)	0.8035 (5)	0.5930 (13)	0.101 (5)
H11A	0.237867	0.783967	0.546953	0.151*
H11B	0.228876	0.840092	0.633506	0.151*
H11C	0.096211	0.813949	0.534122	0.151*
C12	0.2986 (10)	0.7395 (6)	0.7878 (11)	0.089 (4)
H12A	0.279615	0.713965	0.851672	0.134*
H12B	0.350941	0.774401	0.828673	0.134*
H12C	0.349351	0.716518	0.743237	0.134*
Mo1	0.32461 (5)	0.52157 (3)	0.44087 (5)	0.02029 (15)
Mo2	0.47563 (6)	0.66385 (3)	0.44107 (6)	0.02752 (17)
Mo3	0.42473 (6)	0.41297 (3)	0.29025 (6)	0.02489 (16)
Mo4	0.57701 (6)	0.55273 (3)	0.29281 (5)	0.02444 (16)
N1	0.8908 (6)	0.3980 (3)	0.0319 (6)	0.0372 (16)
N2	0.7149 (7)	0.4360 (4)	0.0682 (7)	0.062 (2)
H2'	0.632522	0.447790	0.057114	0.074*
N3	0.0835 (6)	0.7076 (3)	0.6321 (6)	0.0374 (16)
N4	−0.0836 (7)	0.6464 (3)	0.5574 (7)	0.0477 (19)
H4'	−0.162767	0.629819	0.541237	0.057*
O1	0.1678 (4)	0.4898 (2)	0.4064 (4)	0.0309 (12)
O2	0.3741 (4)	0.5299 (2)	0.6278 (4)	0.0236 (10)
O3	0.2944 (4)	0.5998 (2)	0.4063 (4)	0.0279 (11)
O4	0.3752 (4)	0.5014 (2)	0.2878 (4)	0.0229 (10)
O5	0.5546 (4)	0.5580 (2)	0.4958 (4)	0.0237 (10)
O6	0.4664 (5)	0.6511 (2)	0.6112 (5)	0.0291 (11)
O7	0.3763 (5)	0.7268 (2)	0.3893 (5)	0.0445 (14)
O8	0.6382 (5)	0.6903 (2)	0.4794 (5)	0.0376 (13)
O9	0.4701 (5)	0.6243 (2)	0.2804 (4)	0.0295 (11)
O10	0.7381 (5)	0.5820 (2)	0.3377 (5)	0.0363 (13)
O11	0.5427 (5)	0.5344 (2)	0.1348 (4)	0.0357 (12)
O12	0.4590 (5)	0.4063 (2)	0.1494 (5)	0.0396 (13)
O13	0.2655 (5)	0.3843 (2)	0.2625 (5)	0.0352 (12)

Comment

In the past decades, polyoxomolybdates, a subclass of polyoxometalates that featured intriguing structure and excellent properties in the areas of magnetism, biomedicine, materials science, catalysis and energy science [3, 4], have received increasing attention because of their extending application and properties in catalysis, analytical chemistry, and separation science, and so on [5, 6]. For example, a ligand-induced protocol for the selective synthesis of the first lacunary polyoxomolybdate in pyridine solvent was reported [7]. Wang's group synthesized four novel Mo_8O_{26} -based metal-organic frameworks derived from the bis(pyrazine)-bis(amide) *N*-ligands under hydrothermal conditions and found that the four octamolybdate-based frameworks exhibited excellent amperometric sensing properties toward four different metal and non-metal ions, such as, $Cr(VI)$, $Fe(III)$, BrO_3^- , and NO_2^- [8]. So far, various aluminium based polyoxomolybdates has been reported, but imidazole-decorated octamolybdate clusters are rarely reported [9]. Therefore, the design and assembly of novel imidazole-based octamolybdates with multifunctional performance is an imperative issue.

The crystal structure of the title 1-isopropylimidazole-based octamolybdate is shown in Figure. Single crystal X-ray diffraction analysis reveals that the asymmetric unit of the octamolybdate cluster structure consist of half one octamolybdate cluster and two 1-isopropyl-1*H*-imidazolium cations (1-*i*IM) with the proton attached to the N atom of the imidazole ring. Non-hydrogen atoms forming the asymmetric unit are shown as ellipsoids in the figure. Moreover, due to the rigidity of the ring, all the non-H atoms of imidazole ring lie nearly in the same plane. Although the crystallographically independent Mo atoms are in $\{MoO_6\}$ environments, they can still be divided into three types based on subtle differences in their coordination environments. Mo(1) coordinates to one terminal O1 atom (Ot), one μ_2 -bridging O3 atom (Ob), two μ_3 -bridging O2 and O4 atoms (Oc), two μ_5 -bridging O5 and O5 #1 (#1 = 1.5 − *x*, 1.5 + *y*, 1.5 − *z*) atoms (Od) and presents the $[MoO_6]$ coordinated octahedron environments. Conversely, for Mo(2), their coordination environments comprises two Ot atoms, three Ob atoms, one Od atom and also exhibits $[MoO_6]$ coordinated octahedron environments. In contrast to Mo1 and Mo2 modes, Mo(3) and Mo(4) atoms are surrounded by two Ot atoms, one Ob atom, two Oc atoms and one Od atom, respectively. Correspondingly, the Mo–O bond lengths are all in the normal range and are comparable with those of a reported $(HiIM)_4[(iIM)_2Mo_8O_{26}] \cdot 2iIM \cdot 2H_2O$ [10].

In addition, the supramolecular structure of title 1-*i*IM-based octamolybdate was formed by intermolecular

N–H...O hydrogen-bonding interactions between the octamolybdate cluster and protonated 1-isopropylimidazole molecules.

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