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Bis(tetramethylammonium) hexamolybdate hydrate, $[(CH_3)_4N]_2[Mo_6O_{19}]\cdot H_2O$

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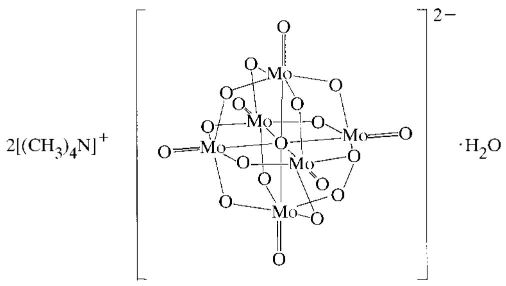
The crystal and molecular structure of the title compound, $(C_4H_{12}N)_2[Mo_6O_{19}]\cdot H_2O$, has been determined from X-ray diffraction data. The polyoxoanion $[Mo_6O_{19}]^{2-}$ is built up from six distorted MoO_6 octahedra sharing common edges and one common vertex at the central O atom, and has crystallographic $m3m$ (O_h) symmetry. The cation has crystallographic $\bar{4}3m$ symmetry.

Comment

In the attempt to synthetize a polyoxomolybdovanadate coordinated with glycine, the tetramethylammonium salt of the hexamolybdate anion has been isolated and structurally characterized. The crystal structure of the title compound, (I), is built up of tetramethylammonium cations, $[(CH_3)_4N]^+$, hexamolybdate anions, $[Mo_6O_{19}]^{2-}$, having a well known Lindquist-type of structure (Lindquist, 1953), and water molecules. Each Mo atom of the polyanion is coordinated by six O atoms (one terminal O_t, one central O_c and four bridging O_b) in a distorted octahedral arrangement, due to the displacement of Mo atoms toward the terminal O_t atoms. The six MoO_6 octahedra share a common vertex at the central O_c, and also each octahedron shares four edges with adjacent octahedra. Bond distances and angles within the $[Mo_6O_{19}]^{2-}$ anion are close to those already observed, for example, in compounds with tetrabutylammonium (Dahlstrom *et al.*, 1982; Rheingold *et al.*, 1992) and tetraphenylarsonium (Clegg *et al.*, 1982) cations, or in the complexes with 18-crown-6 ethers and potassium (Nagano & Sasaki, 1979) or oxonium (Shoemaker *et al.*, 1986) cations.



(I)

Experimental

A mixture of $(CH_3)_4NI$ (0.67 g), MoO_3 (0.50 g), V_2O_5 (0.20 g) and glycine (0.50 g) was dissolved in water (30 ml) and heated at reflux for 3 h. The undissolved solid (0.45 g) was filtered off and the resulting green solution (pH = 3) was left at room temperature. After 24 h, the solution changed colour to orange, and transparent orange crystals of the title complex were isolated.

Crystal data

$(C_4H_{12}N)_2[Mo_6O_{19}]\cdot H_2O$
$M_r = 1045.93$
Cubic, $Fm\bar{3}m$
$a = 13.824$ (5) Å
$V = 2641.8$ (17) Å^3
$Z = 4$
$D_x = 2.630$ Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters: using all available data
$\mu = 2.846$ mm^{-1}
$T = 293$ (2) K
Prism, orange
0.24 × 0.22 × 0.22 mm

Data collection

Nonius KappaCCD diffractometer
2° φ and ω scans
702 measured reflections
320 independent reflections
273 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.020$
$\theta_{\rm max} = 34.28°$
$h = -21 \rightarrow 21$
$k = -15 \rightarrow 15$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
$R(F) = 0.025$
$wR(F^2) = 0.064$
$S = 1.194$
320 reflections
20 parameters
H-atom parameters not refined

$w = 1/[\sigma^2(F_o^2) + (0.0238P)^2 + 6.8529P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.001$
$\Delta\rho_{\rm max} = 0.63$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.51$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 8.0 (18) × 10^{-4}

Table 1
Selected geometric parameters (Å).

Mo1—O1	1.679 (4)	Mo1—O3$^{\rm iii}$	1.9212 (15)
Mo1—O3$^{\rm i}$	1.9212 (15)	Mo1—O3	1.9212 (15)
Mo1—O3$^{\rm ii}$	1.9212 (15)	Mo1—O2	2.3041 (9)

Symmetry codes: (i) z, x, y; (ii) $-z, -x, y$; (iii) $-x, -y, z$.

Due to the symmetry of tetramethylammonium cation ($\bar{4}3m$), methyl H atoms were generated using the HFIX147 command. A peak corresponding to a water H atom was located in the difference density map at special position (e) of the space group $Fm\bar{3}m$ and was included in structure-factor calculations. However, due to the site symmetry and water-molecule geometry, it must be disordered around six equivalent positions.

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Data collection, cell refinement and data reduction: *DENZO* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure and prepare material for publication: *SHELXL*97 (Sheldrick, 1997).

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