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Bis(tetramethylammonium) nonadecaoxohexamolybdenum(VI) monohydrate

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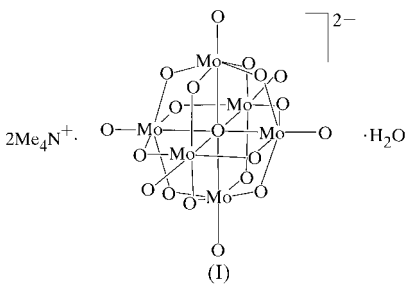
The six Mo atoms in the title compound, $(C_4H_{12}N)_2[Mo_6O_{19}].H_2O$, form a standard octahedral cage through bridging O atoms. The $[Mo_6O_{19}]^{2-}$ anion as a whole has O_h symmetry with three crystallographic fourfold axes aligned along Mo—O—Mo. There exist weak O···O hydrogen bonds (O100···O3 2.951 Å) between the terminal O3 atoms of the anions and O100 atoms of the solvate hydrates in the unit cell.

Comment

The title compound, (I), has the same anion as the following compounds: $C_{12}H_{37}N_9P_3^+$ (Allcock *et al.*, 1973), $C_{20}H_{40}MoN_4S_8^+$ (Garner *et al.*, 1978), $C_{12}H_{24}O_6K^+.H_2O$ (Nagano & Sasaki, 1979), Ph_4As^+ (Clegg *et al.*, 1982), $C_{16}H_{36}N^+$ (Dahlstrom *et al.*, 1982; Rheingold *et al.*, 1993; Boyle *et al.*, 1998), $C_{22}H_{22}O_2P^+$ and $C_{22}H_{22}P^+$ (Arzoumanian *et al.*, 1985), $C_{12}H_{24}O_6.H_3O^+$ (Shoemaker *et al.*, 1986),



(I)

$C_{39}H_{33}Mo_2O_4P_2^+$ (Riera *et al.*, 1988), $C_{14}H_{21}ClN_2Rh^+$ (Zhang *et al.*, 1989), $C_{10}H_8S_8^+$ (Triki *et al.*, 1991), $C_6H_4S_4^+$ (Attanasio *et al.*, 1991; Triki *et al.*, 1992), $C_{20}H_{30}N_{10}Re_{24}^+$ (Bernstein & Dunbar, 1992), $C_{15}H_{35}C_{11}FeN_5O_{52}^+$ (Lu *et al.*, 1992), $C_{20}H_{16}S_4^+$ (Triki *et al.*, 1994), $C_8H_{20}N^+$ (Liu *et al.*, 1995), $C_{14}H_{20}FeN^+$ (Veya & Kochi, 1995), $C_{16}H_{19}N_2^+$ (Xu *et al.*, 1995), $C_{20}H_{21}N_2^+$ (Xu *et al.*, 1995), $C_{23}H_{17}O^+$ (Xu *et al.*, 1996), $C_{20}H_{28}NaO_8^+$ (Lu *et al.*, 1996), $C_{16}H_{16}N_2S_2^+$ (Bellec *et al.*, 1997), $C_{72}H_{60}AgP_4^+$ (Long *et al.*, 1997) and $C_{36}H_{30}NP_2^+$ (Hoppe *et al.*, 1997).

Experimental

The title compound was obtained in an attempt to synthesize new polyoxomolybdates in both reaction systems of $Na_2MoO_4.2H_2O$ (2.0 g, 8.3 mmol), $(CHO)_2$ (3 ml), 3.5% HCl (1 ml), NH_4VO_3 (1.0 g, 8.6 mmol), $[(CH_3)_4N]Br$ (1.0 g, 6.5 mmol) and H_2O (60 ml), or of $Na_2MoO_4.2H_2O$ (2.15 g, 8.9 mmol), NH_4VO_3 (0.5 g, 4.3 mmol), $[(CH_3)_4N]Cl$ (2.0 g, 18.2 mmol), $NH_2OH.HCl$ (3.14 g, 45.2 mmol), Al_2O_3 (234.51 mg, 2.3 mmol) and H_2O (110 ml).

Crystal data

$(C_4H_{12}N)_2[Mo_6O_{19}]\cdot H_2O$
M_r = 1043.93
Cubic, $Fm\bar{3}m$
a = 13.8148 (16) Å
V = 2636.5 (5) Å^3
Z = 4
D_x = 2.635 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 15.4–16.6°
μ = 2.852 mm^{-1}
T = 293 (2) K
Octahedron, orange–red
0.53 × 0.48 × 0.45 mm

Data collection

Enraf–Nonius CAD-4 II diffractometer
ω–2θ scans
1396 measured reflections
167 independent reflections
149 reflections with $I > 2\sigma(I)$
R_{int} = 0.047
θ_{max} = 25.88°
$h = -9 \rightarrow 16$
$k = -15 \rightarrow 12$
$l = -16 \rightarrow 9$
3 standard reflections
every 300 reflections
frequency: 120 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.034
$wR(F^2)$ = 0.095
S = 1.326
167 reflections
21 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.0431P)^2 + 29.6855P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.70 e Å^{-3}
$\Delta\rho_{min}$ = −0.40 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0013 (2)

Table 1
Selected geometric parameters (Å, °).

Mo1—O3	1.654 (10)	Mo1—O1	2.3023 (12)
Mo1—O2	1.916 (3)		
O3—Mo1—O2	103.17 (17)	O2—Mo1—O1	76.83 (17)
O3—Mo1—O1	180.000 (2)		

The H atom on C1 was located and refined isotropically. The water H atoms were not located.

Data collection: Enraf–Nonius *CONTROL* software; cell refinement: *MolEN*/*VAX* (Fair, 1990); data reduction: *MolEN*/*VAX* (Fair, 1990); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); software used to prepare material for publication: *SHELXL*97 (Sheldrick, 1997).

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