Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

$[(C_2H_5)_4N]_4[Mo_8O_{26}]$: a redetermination and correction

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Received 17 August 2005
Accepted 24 August 2005
Online 31 August 2005

Key indicators

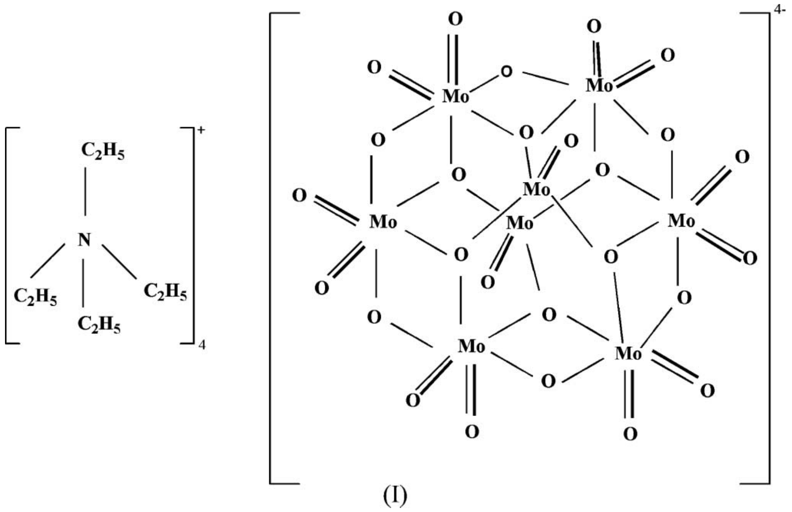
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.031 Å
R factor = 0.068
wR factor = 0.181
Data-to-parameter ratio = 15.4

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

The title compound, tetraethylammonium hexa-μ_3-oxo-hexa-μ_2-oxo-tetradecaoxooctamolybdate, contains discrete centrosymmetric α-$[Mo_8O_{26}]^{4-}$ anions and, for charge compensation, four tetraethylammonium cations per anion. The compound reported by Lu, Haung, Huang & Haung [(1989). *Jiegou Huaxue*, **8**, 23–26] is evidently isostructural with the title compound, although Lu *et al.* formulated their phase as having a composition of $\{[(C_2H_5)_4N]^+\}_2 \cdot 2H^+ \cdot [Mo_8O_{26}]^{4-}$.

Comment

Single crystals of the title compound, (I), were formed inadvertently in one of our exploratory hydrothermal syntheses aimed at new oxides in the quaternary $(C_2H_5)_4N/Mo/Sb/O$ system. Compound (I) contains discrete centrosymmetric α-$[Mo_8O_{26}]^{4-}$ anions (Fig. 1) and, contrary to the previous incorrect formulation (Lu *et al.*, 1989), four tetraethylammonium (TEA) cations for charge compensation. The structure reported by Lu *et al.* (1989) is evidently the same as (I), but they formulated its composition as $\{[(C_2H_5)_4N]^+\}_2 \cdot 2H^+ \cdot [Mo_8O_{26}]^{4-}$, *i.e.* containing two only TEA cations and two protons, the latter occurring in unspecified locations.



(I)

The α form of the $[Mo_8O_{26}]^{4-}$ anion is one of no fewer than eight structural modifications of this species (Allis *et al.*, 2004). The α form consists of a ring of six edge-shared MoO_6 octahedra, bicapped by two MoO_4 tetrahedra. The values of the molybdenum/oxygen bond lengths and angles in (I) agree with those reported for other α-$[Mo_8O_{26}]^{4-}$ anions in the literature (Fuchs & Hartl, 1976; Day *et al.*, 1977; Hsieh *et al.*, 1987; Burkholder & Zubieta, 2005).

Experimental

A mixture of $(C_2H_5)_4NOH$ (Fluka, 0.43 ml, 2.92 mmol), MoO_3 [Paxmy (India), 0.4236 g, 2.94 mmol], Sb_2O_3 [E-Merck (India), 0.1429 g, 0.490 mmol] and water (4.2 ml) was heated at 498 K in a 23 ml Teflon-lined acid digestion bomb for 3 d and the oven was then switched off. Colourless needle-shaped cyrstals of (I) were recovered from the bomb along with a yellow powder of unidentified composition. The initial and final pH values were 10 and 6, respectively. No attempts were made to synthesize (I) as a single phase.

Crystal data

$(C_8H_{20}N)_4[Mo_8O_{26}]$
$M_r = 1704.52$
Monoclinic, $P2_1/n$
$a = 11.836$ (6) Å
$b = 20.032$ (7) Å
$c = 12.089$ (12) Å
$\beta = 105.04$ (7)°
$V = 2768$ (3) Å^3
$Z = 2$
$D_x = 2.045$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 10$–15°
$\mu = 1.83$ mm^{-1}
$T = 293$ (2) K
Block cut from needle, colourless
0.3 × 0.2 × 0.2 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{min} = 0.550$, $T_{max} = 0.693$
5097 measured reflections
4851 independent reflections
3807 reflections with $I > 2\sigma(I)$
$R_{int} = 0.020$
$\theta_{max} = 25.0°$
$h = 0 \rightarrow 14$
$k = 0 \rightarrow 23$
$l = -14 \rightarrow 13$
2 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.068$
$wR(F^2) = 0.181$
$S = 1.18$
4851 reflections
316 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0376P)^2 + 76.2716P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 2.70$ e Å^{-3}
$\Delta\rho_{min} = -1.07$ e Å^{-3}

H atoms were placed in calculated positions [C—H = 0.96 (CH_3) and 0.97Å (CH_2)] and assigned U_{iso}(H) values of 1.2 and 1.5 times U_{eq}(C) for CH_2 and CH_3 groups, respectively. The highest peak and deepest hole in the final difference map are located 0.99 Å from Mo2 and 1.64 Å from O7, respectively.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (*WinGX*; Farrugia, 1999); program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

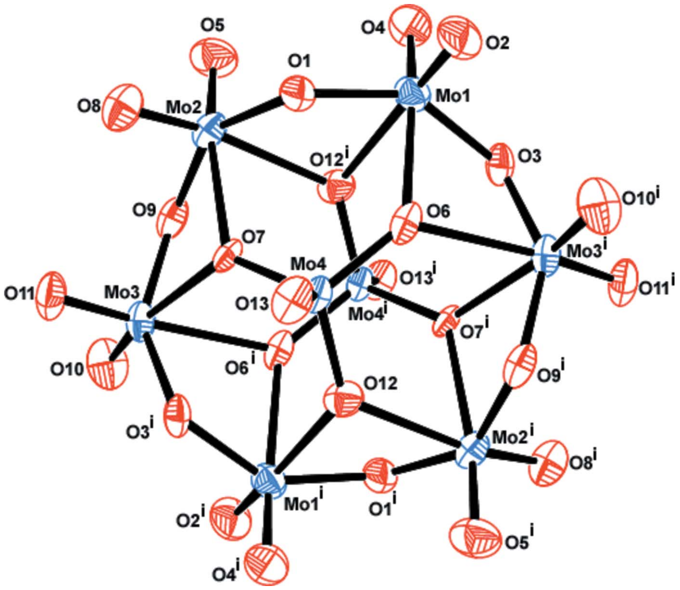


Figure 1
View of the α-$[Mo_8O_{26}]^{4-}$ anion in (I), showing 50% displacement ellipsoids. [Symmetry code: (i) $1 - x, 1 - y, 1 - z$.]

We thank the Sophisticated Analytical Instrument Facility of our institute for the data collection.

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