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Key indicators

Single-crystal X-ray study

$T = 292\text{ K}$

Mean $\sigma(\text{C}-\text{C}) = 0.006\text{ \AA}$

R factor = 0.036

wR factor = 0.097

Data-to-parameter ratio = 17.3

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

Bis(hexaethylpararosanilinium) hexamolybdate

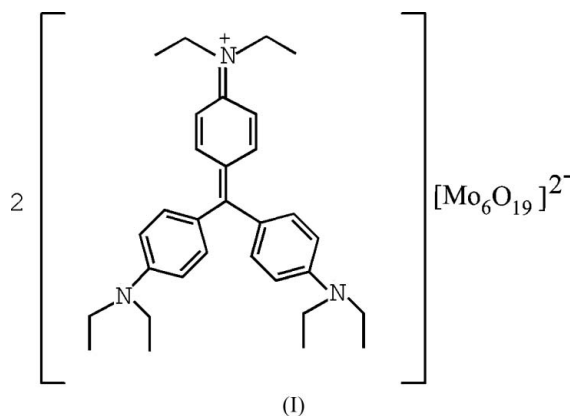
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The title compound, $(\text{C}_{31}\text{H}_{42}\text{N}_3)_2[\text{Mo}_6\text{O}_{19}]$, contains an $[\text{Mo}_6\text{O}_{19}]^{2-}$ hexamolybdate anion, which is built up from six distorted MoO_6 octahedra sharing common edges and one common vertex at the central O atom, and two hexaethylpararosaniline cations to balance the charge. The Mo—O distances lie in the range 1.670 (3)–2.325 (2) Å.

Comment

Polyoxometalates are a rich class of inorganic cluster systems that are remarkable for their molecular and electronic structural diversity and their significance in catalysis, medicine, and materials science (Gouzerh & Proust, 1998; Hill *et al.*, 1998; Pope & Muller, 1991). We report here the crystal structure of one such compound, (I).



The asymmetric unit of (I) consists of a hexamolybdate dianion and two hexaethylpararosaniline cations (Fig. 1). The hexamolybdate subunit $[\text{Mo}_6\text{O}_{19}]^{2-}$ is highly symmetrical and can be described as an octahedral arrangement of MoO_6 octahedra. Thus, each octahedron shares an edge with four octahedra and a vertex with the fifth octahedron. Consequently, there are six terminal oxo-groups, twelve doubly bridging oxo groups, and one μ_6 oxo group at the center of the cluster. The Mo—O distances are significantly different due to multiple bonding character and range from 1.670 (3) to 2.325 (2) Å, which are in agreement with those reported for $(\text{Bu}_4\text{N})_2[\text{Mo}_6\text{O}_{19}]$ (Dahlstrom & Zubietta, 1982). Weak C—H...O interactions are observed in the crystal structure of (I) (Table 2).

Experimental

A solution of $\text{Na}_2\text{MoO}_4 \cdot 2\text{H}_2\text{O}$ (2.42 g, 10 mmol) in H_2O (10 ml) was mixed with acetonitrile (40 ml), and HCl (37%, 5 ml); ethyl violet

(2.61 g, 5 mmol) in water (25 ml) was added. The resulting mixture was refluxed for 2 h at 348 K. After filtration, the pale yellow solution obtained was allowed to stand at room temperature. Well shaped yellow block-like crystals were obtained by slow evaporation of the solvent over a period of about two weeks.

Crystal data

(C₃₁H₄₂N₃)₂[Mo₆O₁₉]

M_r = 1792.99

Triclinic, *P* $\bar{1}$

a = 12.4002 (13) Å

b = 17.4038 (18) Å

c = 17.7311 (18) Å

α = 93.366 (2)°

β = 109.955 (2)°

γ = 98.015 (2)°

V = 3538.4 (6) Å³

Z = 2

D_x = 1.683 Mg m^{−3}

Mo *K*α radiation

μ = 1.10 mm^{−1}

T = 292 (2) K

Block, yellow

0.38 × 0.24 × 0.24 mm

Data collection

Bruker SMART CCD area-detector
diffractometer

φ and ω scans

Absorption correction: multi-scan
(*SADABS*; Bruker, 2000)

*T*_{min} = 0.679, *T*_{max} = 0.778

32369 measured reflections

14494 independent reflections

10125 reflections with *I* > 2σ(*I*)

*R*_{int} = 0.061

θ_{\max} = 26.5°

Refinement

Refinement on *F*²

R [*F*² > 2σ(*F*²)] = 0.036

wR (*F*²) = 0.097

S = 1.09

14494 reflections

838 parameters

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0446P)^2 + 0.0065P]$

where $P = (F_o^2 + 2F_c^2)/3$

(Δ/σ)_{max} = 0.002

$\Delta\rho_{\max} = 0.74$ e Å^{−3}

$\Delta\rho_{\min} = -0.64$ e Å^{−3}

Table 1

Hydrogen-bond geometry (Å, °).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
C6—H6...O19 ⁱ	0.93	2.53	3.256 (5)	135
C7—H7A...O6 ⁱⁱ	0.97	2.45	3.035 (5)	118
C17—H17B...O8 ⁱ	0.97	2.47	3.418 (5)	166
C27—H27A...O15	0.97	2.50	3.346 (5)	146
C37—H37...O16 ⁱⁱⁱ	0.93	2.53	3.356 (5)	149
C38—H38A...O11 ^{iv}	0.97	2.42	3.260 (5)	145
C58—H58A...O18 ^v	0.97	2.53	3.336 (6)	141

Symmetry codes: (i) *x* − 1, *y* − 1, *z*; (ii) *x*, *y* − 1, *z*; (iii) −*x* + 1, −*y* + 1, −*z* + 1; (iv) −*x* + 2, −*y* + 1, −*z* + 1; (v) −*x* + 2, −*y* + 2, −*z* + 1.

The H atoms were positioned geometrically and constrained to ride on their parent atoms, with C—H = 0.93–0.97 Å and *U*_{iso}(H) =

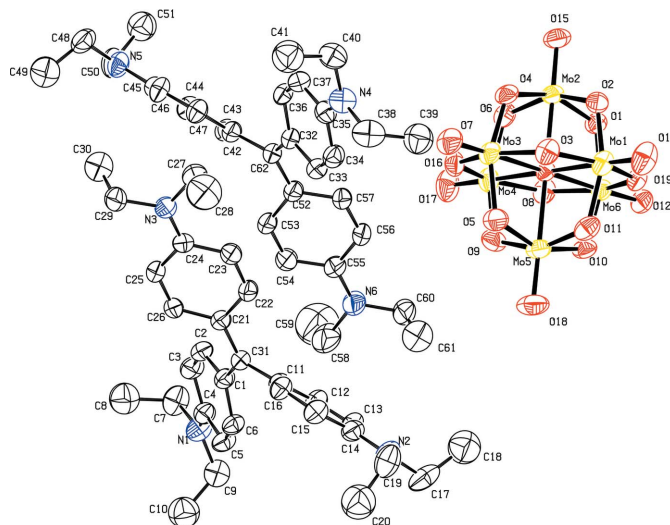


Figure 1

The asymmetric unit of (I), showing 50% probability displacement ellipsoids and labelling of the non-H atoms. H atoms have been omitted for clarity.

1.5*U*_{eq}(C) for methyl H atoms and 1.2*U*_{eq}(C) for others. The *U*^{ij} components of atom C59 were approximated to isotropic behaviour.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

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References

- Bruker (1997). *SHELXTL*. Version 5.10. Bruker AXS Inc., Madison, Wisconsin, USA.
- Bruker (2000). *SMART*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Dahlstrom, P. & Zubieta, J. (1982). *Cryst. Struct. Commun.* **11**, 463–466.
- Gouzerh, P. & Proust, A. (1998). *Chem. Rev.* **98**, 77–79.
- Hill, C. L. (1998). *Chem. Rev.* **98**, 1–6.
- Pope, M. T. & Muller, A. (1991). *Angew. Chem.* **30**, 34–37.
- Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.