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The Structure of Bis(tetraethylammonium) Bis(μ-acetato-*O*,*O*')-tri-μ-chloro-pentachloro-μ_3-oxo-*cyclo*-trimolybdate(3*Mo*–*Mo*), $2C_8H_{20}N^+.C_4H_6Cl_8Mo_3O_5^{2-}$

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Abstract. $M_r = 966{\cdot}04$, orthorhombic, $Pna2_1$, $a = 17{\cdot}815$ (1), $b = 16{\cdot}629$ (2), $c = 12{\cdot}003$ (1) Å, $V = 3556$ (1) Å^3, $Z = 4$, $D_m = 1{\cdot}80$, $D_x = 1{\cdot}804$ g cm^{-3}, graphite-monochromatized Mo $K\alpha$, $\lambda = 0{\cdot}71073$ Å, $\mu = 16{\cdot}564$ cm^{-1}, $F(000) = 1928$, $T = 295$ K, final $R = 0{\cdot}048$ for 2584 reflections. In the mono-oxo-capped trinuclear Mo cluster anion, the three Mo atoms [Mo(1), Mo(2), Mo(3)], the μ_3-O atom [O(1)] and three μ-Cl atoms [Cl(1), Cl(2), Cl(3)] form an apex-deficient cubane-like configuration. The symmetry of the anion is C_s.

Introduction. The title crystal was one of the trinuclear Mo clusters (Huang Jinling, Shang Maoyu, Huang Jianquan & Lu Jiaxi, 1982; Shang Maoyu, Huang Jinling & Lu Jiaxi, 1984) synthesized for a systematic study of middle-valence Mo clusters to get a better understanding of possible structure configurations as well as the metal–metal bonding characters.

Experimental. Black octahedron-like crystals produced by $MoCl_3.3H_2O$, Ac_2O and Et_4NI in EtOH saturated with HCl gas; D_m by flotation in xylene/bromoform: $0{\cdot}19 \times 0{\cdot}30 \times 0{\cdot}33$ mm, Enraf–Nonius CAD-4 diffractometer, 25 reflections for measuring lattice parameters, $2 \le 2\theta \le 50°$, h 0–21, k 0–20, l 0–14, 3661 data, all independent, 2584 with $I \ge 3\sigma(I)$, Enraf–Nonius crystallographic computing package (Frenz, 1980), PDP 11/70 computer; Lp correction, empirical absorption correction based on ψ scans of 9 Bragg reflections, transmission factors 0·8979–0·9991; heavy-atom method, full-matrix refinement on F, Mo, Cl anisotropic, O, N, C isotropic, H not determined; final $R = 0{\cdot}048$ for observed reflections, $R_w = 0{\cdot}051$, $w = 1$; max. $\Delta/\sigma = 0{\cdot}52$, $\Delta\rho$ excursions $\pm 0{\cdot}7$ e Å^{-3}; atomic scattering factors from *International Tables for X-ray Crystallography* (1974).*

Discussion. Table 1 gives the atomic coordinates and Table 2 the interatomic distances and angles. Figs. 1 and 2 show the structure and packing of the cluster, respectively.

Three independent Mo atoms form a triangular configuration with an average side length of 2·60 (1) Å corresponding to a Mo–Mo bond order of one (Müller, Josters & Cotton, 1980). One μ_3-O atom [O(1)] and three μ-Cl atoms [Cl(1), Cl(2), Cl(3)] bind the three Mo atoms together to form an apex-deficient cubane-like

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39089 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

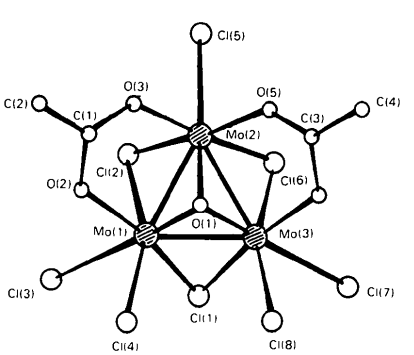

Fig. 1. Structure of the cluster with atom numbering.

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Table 1. *Atomic coordinates and isotropic temperature factors*

$B_{eq} = \frac{1}{3}\sum_i\sum_j B_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	$B_{eq}/B(Å^2)$
Mo(1)	0·37344 (7)	0·22987 (8)	0·6412 (1)	3·18 (2)†
Mo(2)	0·43756 (6)	0·34501 (6)	0·7500 (0)	3·29 (2)†
Mo(3)	0·37303 (8)	0·22956 (8)	0·8593 (1)	3·38 (2)†
Cl(1)	0·2608 (2)	0·2162 (2)	0·7498 (9)	3·82 (6)†
Cl(2)	0·3408 (2)	0·3709 (2)	0·6149 (4)	4·33 (9)†
Cl(3)	0·2900 (2)	0·2134 (3)	0·4792 (4)	5·1 (1)†
Cl(4)	0·3966 (3)	0·0930 (2)	0·6157 (4)	4·9 (1)†
Cl(5)	0·4587 (2)	0·4895 (2)	0·7547 (10)	5·11 (8)†
Cl(6)	0·3426 (2)	0·3689 (2)	0·8868 (3)	3·92 (8)†
Cl(7)	0·2914 (3)	0·2117 (3)	1·0223 (3)	5·0 (1)†
Cl(8)	0·3993 (3)	0·0892 (2)	0·8859 (4)	4·9 (1)†
O(1)	0·4579 (4)	0·2275 (4)	0·7426 (13)	3·0 (1)
O(2)	0·4583 (7)	0·2541 (8)	0·5235 (11)	5·3 (3)
O(3)	0·5209 (6)	0·3434 (7)	0·6366 (10)	4·5 (2)
O(4)	0·4611 (5)	0·2461 (6)	0·9730 (8)	3·3 (2)
O(5)	0·5159 (5)	0·3425 (6)	0·8832 (8)	3·2 (2)
N(1)	0·3720 (6)	−0·0063 (6)	0·245 (2)	3·9 (2)
N(2)	0·3041 (6)	0·4356 (6)	0·251 (3)	4·1 (2)
C(1)	0·5220 (16)	0·3015 (17)	0·542 (3)	9·5 (8)
C(2)	0·5795 (9)	0·2969 (10)	0·460 (1)	4·3 (3)
C(3)	0·5111 (6)	0·2978 (7)	0·960 (1)	1·8 (2)
C(4)	0·5682 (10)	0·3132 (11)	1·060 (2)	4·7 (4)
C(5)	0·3322 (15)	−0·0673 (16)	0·182 (2)	8·9 (7)
C(6)	0·3838 (12)	−0·1273 (13)	0·120 (2)	6·5 (5)
C(7)	0·3090 (15)	0·0305 (15)	0·320 (2)	8·2 (6)
C(8)	0·2463 (9)	0·0718 (9)	0·262 (3)	5·7 (3)
C(9)	0·4124 (14)	0·0535 (16)	0·187 (2)	8·4 (7)
C(10)	0·4634 (14)	0·1079 (15)	0·229 (3)	9·7 (7)
C(11)	0·4223 (13)	−0·0499 (14)	0·330 (2)	7·2 (6)
C(12)	0·3905 (13)	−0·1010 (14)	0·419 (2)	7·0 (5)
C(13)	0·3579 (12)	0·3841 (13)	0·314 (2)	6·5 (5)
C(14)	0·3960 (11)	0·3165 (12)	0·262 (4)	7·7 (5)
C(15)	0·2697 (12)	0·5001 (13)	0·319 (2)	6·1 (5)
C(16)	0·3134 (11)	0·5344 (12)	0·420 (2)	5·4 (4)
C(17)	0·3561 (12)	0·476 (13)	0·155 (2)	6·6 (5)
C(18)	0·3288 (13)	0·5495 (14)	0·118 (2)	6·8 (5)
C(19)	0·2433 (12)	0·3885 (12)	0·193 (2)	6·1 (5)
C(20)	0·1911 (12)	0·3453 (13)	0·252 (7)	9·2 (5)

† B_{eq} values.

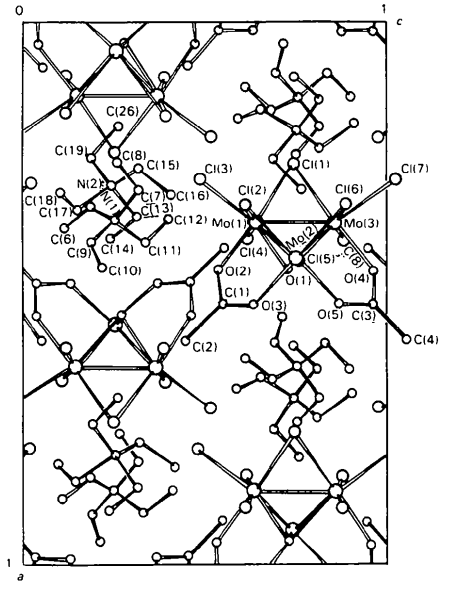

Fig. 2. Packing of the cluster.

Table 2. *Bond distances* (Å) *and angles* (°)

Mo(1)–Mo(2)	2·584 (2)	Mo(2)–O(5)	2·122 (9)
Mo(1)–Mo(3)	2·618 (1)	Mo(3)–Cl(1)	2·403 (4)
Mo(1)–Cl(1)	2·404 (4)	Mo(3)–Cl(6)	2·403 (5)
Mo(1)–Cl(2)	2·437 (6)	Mo(3)–Cl(7)	2·456 (5)
Mo(1)–Cl(3)	2·464 (5)	Mo(3)–Cl(8)	2·401 (6)
Mo(1)–Cl(4)	2·333 (6)		
Mo(1)–O(1)	1·936 (8)	Mo(3)–O(1)	2·062 (8)
Mo(1)–O(2)	2·108 (14)	Mo(3)–O(4)	2·098 (11)
Mo(2)–Mo(3)	2·594 (2)	O(2)–C(1)	1·40 (3)
Mo(2)–Cl(2)	2·405 (5)	O(3)–C(1)	1·33 (3)
Mo(2)–Cl(5)	2·433 (2)	O(4)–C(3)	1·247 (15)
Mo(2)–Cl(6)	2·391 (5)	O(5)–C(3)	1·191 (13)
Mo(2)–O(1)	1·989 (4)	C(1)–C(2)	1·43 (3)
Mo(2)–O(3)	2·014 (10)	C(3)–C(4)	1·59 (2)

Mo(2)–Mo(1)–Mo(3)	59·83 (8)	Mo(1)–Mo(3)–Mo(2)	59·43 (8)
Cl(1)–Mo(1)–Cl(2)	87·82 (14)	Cl(1)–Mo(3)–Cl(6)	88·62 (14)
Cl(1)–Mo(1)–Cl(3)	85·0 (2)	Cl(1)–Mo(3)–Cl(7)	86·1 (2)
Cl(1)–Mo(1)–Cl(4)	97·30 (16)	Cl(1)–Mo(3)–Cl(8)	98·36 (16)
Cl(1)–Mo(1)–O(1)	107·9 (3)	Cl(1)–Mo(3)–O(1)	103·7 (3)
Cl(1)–Mo(1)–O(2)	168·9 (4)	Cl(1)–Mo(3)–O(4)	172·1 (3)
Cl(2)–Mo(1)–Cl(3)	82·0 (2)	Cl(6)–Mo(3)–Cl(7)	82·8 (2)
Cl(2)–Mo(1)–Cl(4)	164·6 (2)	Cl(6)–Mo(3)–Cl(8)	164·4 (2)
Cl(2)–Mo(1)–O(1)	106·63 (17)	Cl(6)–Mo(3)–O(1)	106·0 (2)
Cl(2)–Mo(1)–O(2)	84·3 (4)	Cl(6)–Mo(3)–O(4)	87·3 (3)
Cl(3)–Mo(1)–Cl(4)	84·0 (2)	Cl(7)–Mo(3)–Cl(8)	83·8 (2)
Cl(3)–Mo(1)–O(1)	164·4 (3)	Cl(7)–Mo(3)–O(1)	166·8 (3)
Cl(3)–Mo(1)–O(2)	85·7 (4)	Cl(7)–Mo(3)–O(4)	86·6 (3)
Cl(4)–Mo(1)–O(1)	85·7 (2)	Cl(8)–Mo(3)–O(1)	86·1 (2)
Cl(4)–Mo(1)–O(2)	88·4 (4)	Cl(8)–Mo(3)–O(4)	84·0 (3)
O(1)–Mo(1)–O(2)	82·4 (5)	O(1)–Mo(3)–O(4)	84·0 (4)
Mo(1)–Mo(2)–Mo(3)	60·74 (2)	Mo(1)–Cl(1)–Mo(3)	66·00 (4)
Cl(2)–Mo(2)–Cl(5)	87·1 (2)	Mo(1)–Cl(2)–Mo(3)	64·49 (13)
Cl(2)–Mo(2)–Cl(6)	85·75 (7)	Mo(2)–Cl(6)–Mo(3)	65·51 (13)
Cl(2)–Mo(2)–O(1)	106·1 (3)	Mo(1)–O(1)–Mo(2)	82·3 (2)
Cl(2)–Mo(2)–O(3)	94·3 (3)	Mo(1)–O(1)–Mo(3)	81·74 (15)
Cl(2)–Mo(2)–O(5)	169·3 (3)	Mo(2)–O(1)–Mo(3)	79·6 (2)
Cl(5)–Mo(2)–Cl(6)	86·0 (2)	Mo(1)–O(2)–C(1)	125·5 (13)
Cl(5)–Mo(2)–O(1)	160·54 (12)	Mo(2)–O(3)–C(1)	126·4 (12)
Cl(5)–Mo(2)–O(3)	85·1 (3)	Mo(3)–O(4)–C(3)	123·1 (8)
Cl(5)–Mo(2)–O(5)	84·3 (3)	Mo(2)–O(5)–C(3)	123·5 (7)
Cl(6)–Mo(2)–O(1)	108·9 (3)	O(2)–C(1)–O(3)	114·9 (19)
Cl(6)–Mo(2)–O(3)	171·0 (3)	O(2)–C(1)–C(2)	116·2 (19)
Cl(6)–Mo(2)–O(5)	87·2 (3)	O(3)–C(1)–C(2)	129·0 (18)
O(1)–Mo(2)–O(3)	79·8 (4)	O(4)–C(3)–O(5)	125·1 (10)
O(1)–Mo(2)–O(5)	83·9 (4)	O(4)–C(3)–C(4)	118·5 (9)
O(3)–Mo(2)–O(5)	91·4 (3)	O(5)–C(3)–C(4)	116·0 (9)

configuration. Furthermore, each Mo atom is coordinated by three additional atoms to complete a distorted octahedron. As a whole, the symmetry of the cluster anion is reduced from common C_{3v} to C_s by the two bridging acetate radicals.

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