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Acta Cryst. (1989). **C45**, 946–947

Structure of Tetraammonium Hexahydrogenhexamolybdocuprate(II) Tetrahydrate

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(Received 2 November 1988; accepted 12 December 1988)

Abstract. $(\text{NH}_4)_4[\text{H}_6\text{CuMo}_6\text{O}_{24}]\cdot 4\text{H}_2\text{O}$, $M_r = 1173.4$, monoclinic, $P2_1/c$, $a = 11.421$ (1), $b = 11.126$ (2), $c = 11.919$ (2) Å, $\beta = 107.22$ (1)°, $V = 1446.6$ (4) Å³,

$Z = 2$, $D_m = 2.73$, $D_x = 2.69$ Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 3.32$ mm⁻¹, $F(000) = 1126$, $T = 298$ K, $R = 0.060$ for 1734 independent reflections. The discrete polyanion $[\text{H}_6\text{CuMo}_6\text{O}_{24}]^{4-}$ is isostructural with $[\text{H}_6\text{CrMo}_6\text{O}_{24}]^{3-}$ [Perloff (1970). *Inorg. Chem.* **9**, 2228–2239]. Mo–O distances range from 1.70 to 2.25 Å, whereas Cu–O distances range from 2.02 to 2.12 Å, exhibiting no typical tetragonal distortion from the Jahn–Teller effect.

Table 1. *Positional and equivalent isotropic thermal parameters*

Fractional coordinates are multiplied by 10⁴ for the Cu and Mo atoms, by 10³ for the remaining atoms.

$$B_{\text{eq}} = \frac{1}{3}\pi^2 \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i> _{eq} (Å ²)
Mo(1)	1515 (1)	2413 (1)	1333 (1)	1.9 (1)
Mo(2)	3042 (1)	0058 (1)	0755 (1)	2.1 (1)
Mo(3)	1514 (1)	–2361 (1)	–0523 (1)	1.8 (1)
Cu	0	0	0	1.6 (1)
O(1)	141 (1)	116 (1)	–015 (1)	1.9 (4)
O(2)	143 (1)	–104 (1)	089 (1)	1.9 (4)
O(3)	–002 (1)	–112 (1)	–134 (1)	1.8 (4)
O(4)	262 (1)	108 (1)	192 (1)	2.1 (4)
O(5)	256 (1)	–100 (1)	–058 (1)	1.8 (4)
O(6)	002 (1)	–306 (1)	–034 (1)	1.8 (4)
O(7)	153 (1)	296 (1)	267 (1)	3.6 (6)
O(8)	244 (1)	339 (1)	090 (1)	3.0 (5)
O(9)	397 (1)	095 (1)	022 (1)	3.4 (6)
O(10)	405 (1)	–080 (1)	178 (1)	4.1 (6)
O(11)	246 (1)	–328 (1)	049 (1)	2.9 (1)
O(12)	154 (1)	–297 (1)	–183 (1)	2.7 (5)
O(13)	013 (1)	044 (1)	355 (1)	4.9 (7)
O(14)*	518 (2)	173 (2)	543 (3)	4.0 (10)
O(15)*	500 (2)	196 (2)	293 (3)	4.6 (11)
N(1)	311 (1)	474 (1)	–088 (1)	3.4 (7)
N(2)	340 (1)	–472 (1)	260 (1)	2.8 (6)

* Occupancies of these atoms are 0.5.

Experimental. Preparation: Rosenheim (1916); faintly blue rhombic crystals from a mixed aqueous solution of $\text{CuSO}_4\cdot 5\text{H}_2\text{O}$ and $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24}\cdot 4\text{H}_2\text{O}$ in the ratio 1:6, adjusted pH 4–5; D_m by flotation in dibromoethane and diiodomethane; crystal 0.10 × 0.10 × 0.02 mm. AFC-5R diffractometer; cell parameters from 22 indices in θ range 12.0–14.8°; data collected by θ – 2θ scan mode up to 55° in 2θ ; $-14 \leq h \leq 14$, $0 \leq k \leq 14$ and $0 \leq l \leq 15$; 3516 reflections measured; three standard reflections, no decay; empirical absorption correction

Table 2. *Selected interatomic distances (Å)*

Mo(1)–O(1)	2.23 (1)	Mo(2)–O(1)	2.22 (1)	Mo(3)–O(2)	2.25 (1)
Mo(1)–O(3)	2.24 (1)	Mo(2)–O(2)	2.25 (1)	Mo(3)–O(3)	2.22 (1)
Mo(1)–O(4)	1.94 (1)	Mo(2)–O(4)	1.96 (1)	Mo(3)–O(5)	1.95 (1)
Mo(1)–O(6)	1.94 (1)	Mo(2)–O(5)	1.92 (2)	Mo(3)–O(6)	1.94 (1)
Mo(1)–O(7)	1.70 (1)	Mo(2)–O(9)	1.70 (1)	Mo(3)–O(11)	1.70 (1)
Mo(1)–O(8)	1.70 (1)	Mo(2)–O(10)	1.70 (1)	Mo(3)–O(12)	1.70 (1)
Cu–O(1)	2.12 (1)	Cu–O(2)	2.03 (1)	Cu–O(3)	2.02 (1)

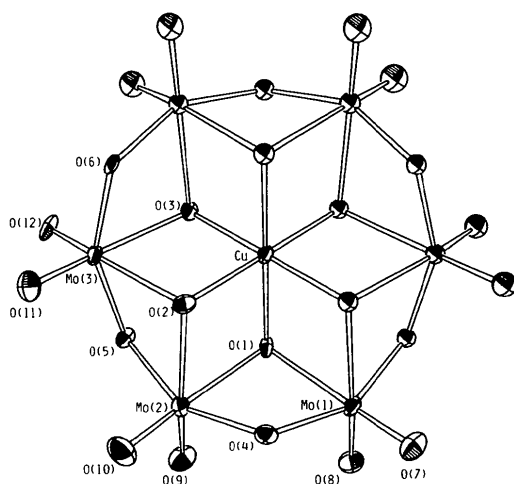


Fig. 1. The anion in $(\text{NH}_4)_4[\text{H}_6\text{CuMo}_6\text{O}_{24}]\cdot 4\text{H}_2\text{O}$. Thermal ellipsoids are scaled to enclose 30% probability.

(max. 1.23 and min. 0.87). Positions of Cu and Mo atoms from Patterson map, N and O atoms from Fourier syntheses; full-matrix least-squares refinement on F ; H atoms not located; all atoms anisotropic, final $R = 0.060$, $wR = 0.075$ and $S = 1.40$ for 1734 observed reflections [$|F| > 3\sigma(F)$], 187 parameters, $w = 1/\sigma^2(F)$, $(\Delta/\sigma)_{\text{max}} = 0.03$, maximum $\Delta\rho = 1.41 \text{ e } \text{\AA}^{-3}$ possibly at site fractionally occupied by disordered H_2O . All calculations using *TEXSAN* (Swepston, 1986) with scattering factors from *International Tables for X-ray Crystallography* (1974). Atomic parameters

are listed in Table 1* and interatomic distances are given in Table 2. Fig. 1 shows an *ORTEP* (Johnson, 1976) drawing of the structure.

Related literature. In $[\text{H}_6\text{CrMo}_6\text{O}_{24}]^{3-}$ Cr–O and Mo–O are in the ranges 1.97–1.99 and 1.70–2.35 Å, respectively (Perloff, 1970). In $[\text{TeMo}_6\text{O}_{24}]^{6-}$ Te–O and Mo–O are in the ranges 1.93–1.94 and 1.69–2.32 Å, respectively (Evans, 1974). In $[\text{IMo}_6\text{O}_{24}]^{7-}$ I–O and Mo–O are in the ranges 1.88–1.89 and 1.70–2.37 Å, respectively (Kondo, Kobayashi & Sasaki, 1980).

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51676 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1989). **C45**, 947–949

Structure of the Bis[bis(diphenylphosphino)methane]digold(I) Cation in $[\text{Au}_2(\text{dppm})_2](\text{BF}_4)_2$

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(Received 9 September 1988; accepted 14 November 1988)

Abstract. Bis- μ -[bis(diphenylphosphino)methane-*P,P'*]-digold(I) bis(tetrafluoroborate), $[\text{Au}_2(\text{C}_{25}\text{H}_{22}\text{P}_2)_2](\text{BF}_4)_2$, $[\text{Au}_2\{\text{C}_6\text{H}_5\sigma\text{PCH}_2\text{P}(\text{C}_6\text{H}_5)_2\}_2](\text{BF}_4)_2$, $M_r = 1336.4$, monoclinic, $P2_1/a$, $a = 15.047(5)$, $b = 17.462(3)$, $c = 10.532(2)$ Å, $\beta = 117.59(2)^\circ$, $V = 2452(1)$ Å³, $Z = 2$, $D_x = 1.810 \text{ g cm}^{-3}$, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 61.56 \text{ cm}^{-1}$, $F(000) = 1288$, $T = 298 \text{ K}$. Final $R = 0.040$ for 2587 unique observed

reflections. This is the first structure of an $\text{Au}_2(\text{dppm})_2^{2+}$ cation in which the anion is not coordinated to an Au atom. The Au...Au separation is 2.931(1) Å.

Experimental. Addition of AgBF_4 to $[\text{Au}_2(\text{dppm})_2]\text{Cl}_2$ in tetrahydrofuran gave a precipitate which, on dissolving in CH_3CN and removing the solvent, gave $[\text{Au}_2(\text{dppm})_2](\text{BF}_4)_2$ in 93% yield. Analysis: C 45.29 (calculated 44.94), H 3.47 (3.32%). M.p. 493 K with decomposition. A colorless crystal, obtained by re-

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