Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$U_{eq} = (1/3)\Sigma_i\Sigma_jU_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
Sn	0.1633 (1)	0.1513 (1)	0.2226 (1)	0.042 (1)
C	0.0474 (5)	0.4195 (3)	0.1497 (2)	0.041 (1)
O	0.1844 (4)	0.4714 (3)	0.1908 (2)	0.058 (1)
O(1)	0.0128 (4)	0.2930 (2)	0.1463 (1)	0.050 (1)
N	−0.3560 (4)	0.6542 (3)	0.0001 (2)	0.044 (1)
C(1)	−0.1817 (5)	0.7039 (3)	0.0311 (2)	0.050 (1)
C(2)	−0.0471 (5)	0.6340 (3)	0.0805 (2)	0.045 (1)
C(3)	−0.0933 (4)	0.5015 (3)	0.0975 (2)	0.036 (1)
C(4)	−0.2726 (4)	0.4475 (3)	0.0647 (2)	0.036 (1)
C(5)	−0.4015 (4)	0.5260 (3)	0.0172 (2)	0.035 (1)
C(6)	0.0030 (8)	−0.0053 (4)	0.1654 (3)	0.092 (2)
C(7)	0.4641 (6)	0.1836 (4)	0.1960 (3)	0.068 (1)
C(8)	0.0313 (6)	0.2288 (5)	0.3173 (2)	0.067 (1)

Table 2. *Selected geometric parameters* (Å, °)

Sn—O(1)	2.145 (2)	Sn—O(1^i)	2.519 (2)
Sn—C(6)	2.101 (5)	Sn—C(7)	2.103 (4)
Sn—C(8)	2.108 (4)	N—C(5)	1.353 (4)
C(1)—C(2)	1.385 (5)	C(2)—C(3)	1.393 (4)
C—O	1.230 (4)	C(3)—C(4)	1.383 (4)
C—O(1)	1.281 (4)	C(4)—C(5)	1.391 (4)
C—C(3)	1.501 (4)	C(5)—C(5^{ii})	1.483 (5)
N—C(1)	1.329 (4)		
O(1)—Sn—C(6)	89.6 (2)	O(1)—Sn—C(7)	98.7 (1)
C(6)—Sn—C(7)	116.7 (2)	O(1)—Sn—C(8)	93.8 (1)
C(6)—Sn—C(8)	115.8 (2)	C(7)—Sn—C(8)	125.8 (2)
O(1)—Sn—O^i	175.1 (1)	C(6)—Sn—O^i	85.8 (1)
C(7)—Sn—O^i	84.9 (1)	C(8)—Sn—O^i	86.6 (1)
C—C(3)—C(2)	121.0 (3)	C—C(3)—C(4)	120.8 (3)
O—C—O(1)	124.1 (3)	C(2)—C(3)—C(4)	118.1 (3)
O—C—C(3)	121.8 (3)	C(3)—C(4)—C(5)	119.7 (3)
O(1)—C—C(3)	114.1 (3)	N—C(5)—C(4)	122.2 (3)
C(1)—N—C(5)	117.3 (3)	N—C(5)—C(5^{ii})	116.1 (3)
N—C(1)—C(2)	124.3 (3)	C(4)—C(5)—C(5^{ii})	121.7 (3)
C(1)—C(2)—C(3)	118.3 (3)		

Symmetry codes: (i) $\frac{1}{2} - x, y - \frac{1}{2}, \frac{1}{2} - z$; (ii) $-1 - x, 1 - y, -z$.

The intensity data were evaluated by a peak-profile procedure (Diamond, 1969) and then corrected for Lp and absorption effects. The structure was solved by standard Patterson methods, subsequently completed by a combination of a least-squares technique and Fourier syntheses (*SHELXTL-Plus*; Sheldrick, 1991), and refined by a full-matrix least-squares technique based on *F*. Neutral-atom scattering factors were used and an anomalous-dispersion correction was applied (Stewart, 1970). The *SHELXTL-Plus* package was used for data reduction and producing the graphics, while the final geometrical calculations were carried out with a locally modified version of *PARST* (Nardelli, 1983) on a DEC MicroVAX/3400 computer of the Centro Interdipartimentale per la Diffrattometria a Raggi-X.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: NA1192). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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μ-Oxo-bis[(5,10,15,20-tetraphenylporphyrinato)oxomolybdenum(V)]

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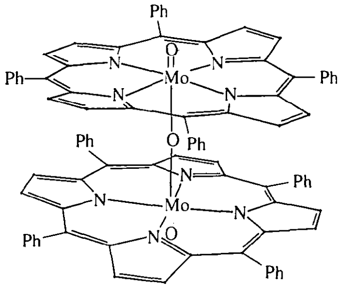
Abstract

The crystal structure of a new solvated crystal form of the title complex, [{Mo(O)(TPP)}$_2$O].1.5H$_2$O.0.5CH$_2$Cl$_2$ (TPP = $C_{44}H_{28}N_4$), has been determined. The dimeric molecule is in a general position, and the overall structural features are extremely similar to those of the previously reported non-solvated form.

Comment

The μ-oxo form of Mo^V porphyrinates, [Mo(O)-(Porph)]$_2$O, is now well known, since the structural characterization of the TPP (Johnson & Scheidt, 1978) and OEP (Kim, Sparapany & Ibers, 1987) derivatives. However, the behavior of this dimeric molecule in solution is rather subtle. Ledon, Bonnet, Brigandat & Varescon (1980) suggested an equilibrium in solution between the μ-oxo dimer and its dissociated monomer Mo(O)(Porph)(OH). They further pointed out that the isolation of solid material would depend upon the solvent used in recrystallization: aromatic solvents such as benzene led to the μ-oxo complex, while the monomeric Mo(O)(Porph)(OH) was the major component isolated from CH_2Cl_2. In the course of our extended studies of the novel singly hydroxo-bridged metalloporphyrins, this monomeric hydroxide complex drew our attention since it could be a precursor for the desired $[Mo(O)(porph)]_2(OH)^+$ complex. In our hands, however, the only isolable crystalline material from CH_2Cl_2 is the starting material μ-oxo complex, (I).



(I)

Unlike the previously reported TPP and OEP derivatives, this new crystal form has a full dimeric molecule in the asymmetric unit. Therefore, the O═Mo—O—Mo═O linkage is very close to being linear, but not exactly linear. By comparison, the two non-solvated TPP and OEP derivatives have linear bridges, required by the crystallographic symmetry. The two Mo═O bonds have an average bond length of 1.704 (4) Å, and the μ-oxo bridge has an average Mo—O bond length of 1.936 (16) Å. The average Mo—N distance is found to be 2.096 (6) Å. Each of the Mo^V atoms is almost in the mean plane of its 24-atom porphinato core (Fig. 3). The two porphyrin rings have an inter-ring spacing of *ca* 3.85 Å and are twisted by 28.6° with respect to each other. The four pairs of phenyl rings have an almost perpendicular orientation with an average phenyl–phenyl dihedral angle of 71° (Fig. 1). These structural features are quite similar to those of the reported TPP derivative.

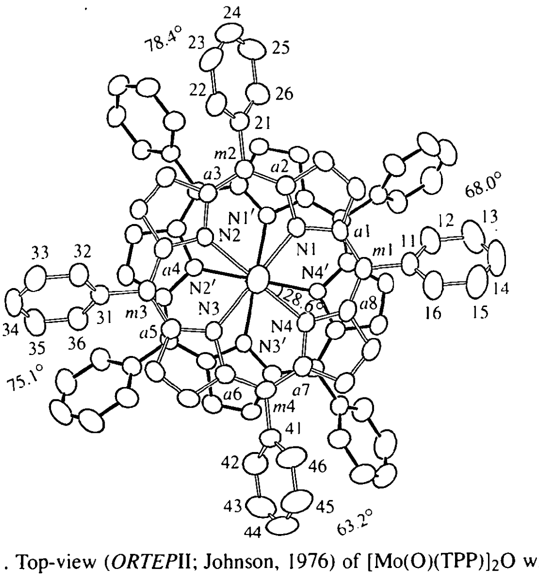


Fig. 1. Top-view (*ORTEP*II; Johnson, 1976) of $[Mo(O)(TPP)]_2O$ with labeling schemes. Displacement ellipsoids are drawn at the 50% probability level. Porphyrin H atoms are omitted and C atoms are labeled by their numbers only, for clarity.

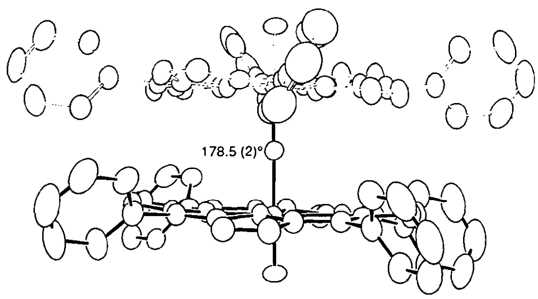


Fig. 2. Edge-view (*ORTEP*II; Johnson, 1976) of $[Mo(O)(TPP)]_2O$. Displacement ellipsoids are drawn at the 50% probability level. Porphyrin H atoms are omitted for clarity.

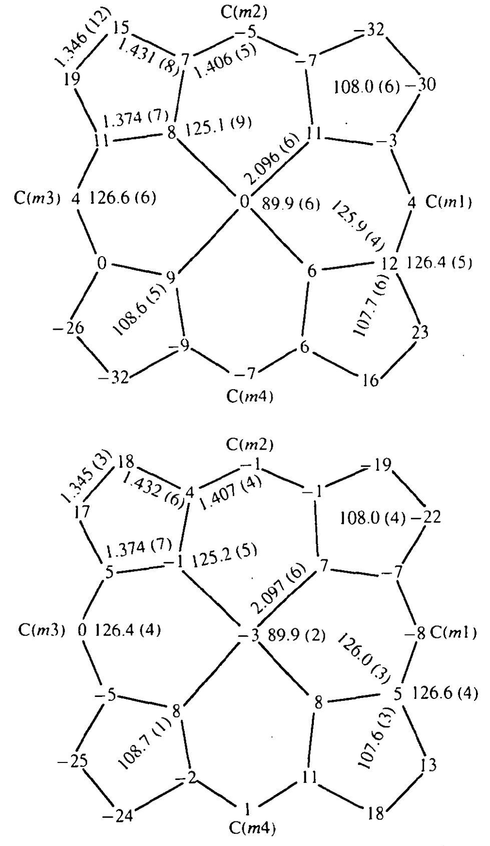


Fig. 3. Formal diagrams of the porphinato cores (top for ring 1, bottom for ring 2) of $[Mo(O)(TPP)]_2O$, displaying the average values for the bond parameters. The numbers in parentheses are the estimated standard deviations calculated on the assumption that the average values were all drawn from the same population. Also displayed are the perpendicular displacements, in units of 0.01 Å, of each atom from the 24-atom mean plane of the core. For both rings, a positive value is a displacement towards the other ring of the dimer.

Experimental

The title compound was crystallized by diffusion of hexanes into a dichloromethane solution.

Crystal data

$[Mo_2O_3(C_{44}H_{28}N_4)_2]$.-
 $1.5H_2O.0.5CH_2Cl_2$
$M_r = 1534.78$
Monoclinic
$P2_1/n$
$a = 14.105\,(4)$ Å
$b = 32.281\,(4)$ Å
$c = 18.473\,(3)$ Å
$\beta = 107.380\,(10)°$
$V = 8027.2\,(28)$ Å^3
$Z = 4$
$D_x = 1.270$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 250 reflections
$\theta = 5.1–20.8°$
$\mu = 0.40$ mm^{-1}
$T = 293\,(2)$ K
Rectangular prism
$0.50 \times 0.30 \times 0.25$ mm
Black

Data collection

Enraf–Nonius FAST area-detector diffractometer
Ellipsoid-mask fitting scans
Absorption correction: refined from ΔF (*DIFABS*; Walker & Stuart, 1983)
 $T_{\min} = 0.852$, $T_{\max} = 1.000$

22990 measured reflections
9009 independent reflections
7977 observed reflections $[I > 2\sigma(I)]$
$R_{\rm int} = 0.0753$
$\theta_{\max} = 22.58°$
$h = -12 \rightarrow 14$
$k = -34 \rightarrow 28$
$l = -18 \rightarrow 18$

Refinement

Refinement on F^2
$R(F) = 0.0466$
$wR(F^2) = 0.1421$
$S = 1.097$
8989 reflections
938 parameters
H atoms idealized with riding model
$w = 1/[\sigma^2(F_o^2) + (0.0792P)^2 + 20.0322P]$
 where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.031$
$\Delta\rho_{\max} = 0.836$ e Å^{-3}
$\Delta\rho_{\min} = -0.574$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
Mo(1)	0.90976 (3)	0.166336 (13)	0.54939 (3)	0.0293 (2)
N(1)	0.8880 (3)	0.11426 (13)	0.4778 (3)	0.0364 (11)
N(2)	0.9528 (3)	0.20123 (12)	0.4689 (2)	0.0324 (10)
N(3)	0.9173 (3)	0.22000 (12)	0.6142 (2)	0.0330 (10)
N(4)	0.8562 (3)	0.13283 (13)	0.6261 (2)	0.0330 (10)
C(*a*1)	0.8664 (4)	0.0751 (2)	0.4984 (3)	0.0381 (14)
C(*a*2)	0.9212 (4)	0.1110 (2)	0.4154 (3)	0.0375 (14)
C(*a*3)	0.9650 (4)	0.1857 (2)	0.4029 (3)	0.0328 (13)
C(*a*4)	0.9767 (4)	0.2425 (2)	0.4740 (3)	0.0341 (13)
C(*a*5)	0.9549 (4)	0.2575 (2)	0.6016 (3)	0.0335 (13)
C(*a*6)	0.9089 (4)	0.2204 (2)	0.6871 (3)	0.0332 (13)
C(*a*7)	0.8455 (4)	0.1480 (2)	0.6926 (3)	0.0358 (13)
C(*a*8)	0.8265 (4)	0.0924 (2)	0.6183 (3)	0.0389 (14)
C(*b*1)	0.8867 (5)	0.0466 (2)	0.4457 (4)	0.050 (2)
C(*b*2)	0.9195 (5)	0.0681 (2)	0.3961 (4)	0.048 (2)
C(*b*3)	0.9938 (4)	0.2199 (2)	0.3643 (3)	0.0371 (13)
C(*b*4)	0.9998 (4)	0.2541 (2)	0.4062 (3)	0.0344 (13)
C(*b*5)	0.9718 (4)	0.2821 (2)	0.6685 (3)	0.0407 (14)
C(*b*6)	0.9437 (4)	0.2593 (2)	0.7201 (3)	0.0395 (14)
C(*b*7)	0.8038 (5)	0.1161 (2)	0.7269 (3)	0.0455 (15)
C(*b*8)	0.7900 (5)	0.0822 (2)	0.6813 (3)	0.047 (2)
C(*m*1)	0.8336 (4)	0.0650 (2)	0.5606 (3)	0.0388 (14)
C(*m*2)	0.9539 (4)	0.1443 (2)	0.3795 (3)	0.0356 (13)
C(*m*3)	0.9802 (4)	0.2687 (2)	0.5360 (3)	0.0327 (13)
C(*m*4)	0.8738 (4)	0.1877 (2)	0.7224 (3)	0.0370 (13)
C(11)	0.8094 (4)	0.0208 (2)	0.5693 (4)	0.0425 (15)
C(12)	0.7366 (5)	0.0005 (2)	0.5146 (4)	0.056 (2)
C(13)	0.7151 (6)	−0.0407 (2)	0.5240 (6)	0.078 (2)
C(14)	0.7655 (7)	−0.0611 (2)	0.5890 (7)	0.090 (3)
C(15)	0.8389 (7)	−0.0415 (2)	0.6442 (5)	0.081 (2)
C(16)	0.8604 (5)	−0.0008 (2)	0.6345 (4)	0.059 (2)
C(21)	0.9836 (5)	0.1339 (2)	0.3103 (3)	0.0400 (14)
C(22)	1.0783 (5)	0.1427 (2)	0.3080 (4)	0.050 (2)
C(23)	1.1067 (7)	0.1333 (2)	0.2443 (5)	0.068 (2)
C(24)	1.0396 (8)	0.1158 (2)	0.1823 (5)	0.079 (3)
C(25)	0.9463 (7)	0.1067 (2)	0.1836 (4)	0.071 (2)
C(26)	0.9172 (5)	0.1153 (2)	0.2481 (4)	0.057 (2)
C(31)	1.0232 (4)	0.3111 (2)	0.5344 (3)	0.0380 (14)
C(32)	1.1169 (4)	0.3157 (2)	0.5262 (3)	0.0432 (14)
C(33)	1.1575 (5)	0.3542 (2)	0.5249 (4)	0.059 (2)
C(34)	1.1051 (6)	0.3889 (2)	0.5313 (4)	0.067 (2)
C(35)	1.0131 (6)	0.3851 (2)	0.5402 (4)	0.063 (2)
C(36)	0.9718 (5)	0.3465 (2)	0.5414 (3)	0.0461 (15)
C(41)	0.8672 (4)	0.1960 (2)	0.8001 (3)	0.0413 (15)
C(42)	0.8017 (5)	0.2249 (2)	0.8124 (4)	0.056 (2)
C(43)	0.7937 (6)	0.2311 (3)	0.8848 (5)	0.076 (2)
C(44)	0.8521 (7)	0.2084 (3)	0.9444 (5)	0.084 (3)
C(45)	0.9184 (7)	0.1809 (3)	0.9334 (4)	0.080 (2)
C(46)	0.9255 (6)	0.1741 (2)	0.8611 (4)	0.063 (2)
Mo(2)	0.63556 (3)	0.194634 (13)	0.44742 (3)	0.0282 (2)
N(1′)	0.6546 (3)	0.16342 (12)	0.3528 (2)	0.0338 (11)
N(2′)	0.6887 (3)	0.24930 (12)	0.4128 (2)	0.0315 (10)
N(3′)	0.6312 (3)	0.22382 (12)	0.5476 (2)	0.0318 (10)
N(4′)	0.5910 (3)	0.13875 (12)	0.4859 (2)	0.0304 (10)
C(*a*1′)	0.6217 (4)	0.1238 (2)	0.3311 (3)	0.0330 (13)
C(*a*2′)	0.6813 (4)	0.1819 (2)	0.2949 (3)	0.0325 (13)
C(*a*3′)	0.7186 (4)	0.2535 (2)	0.3490 (3)	0.0323 (13)
C(*a*4′)	0.7070 (4)	0.2859 (2)	0.4527 (3)	0.0318 (12)
C(*a*5′)	0.6458 (4)	0.2656 (2)	0.5623 (3)	0.0331 (13)
C(*a*6′)	0.5918 (4)	0.2066 (2)	0.6008 (3)	0.0354 (13)
C(*a*7′)	0.5670 (4)	0.1336 (2)	0.5518 (3)	0.0330 (13)
C(*a*8′)	0.5757 (4)	0.1024 (2)	0.4470 (3)	0.0346 (13)
C(*b*1′)	0.6295 (4)	0.1172 (2)	0.2565 (3)	0.0389 (14)
C(*b*2′)	0.6654 (4)	0.1520 (2)	0.2346 (3)	0.0361 (13)
C(*b*3′)	0.7593 (4)	0.2941 (2)	0.3501 (3)	0.0397 (14)
C(*b*4′)	0.7509 (4)	0.3138 (2)	0.4119 (3)	0.0410 (14)
C(*b*5′)	0.6128 (4)	0.2746 (2)	0.6266 (3)	0.0407 (14)
C(*b*6′)	0.5801 (4)	0.2393 (2)	0.6493 (3)	0.0404 (14)
C(*b*7′)	0.5411 (4)	0.0909 (2)	0.5570 (3)	0.0404 (14)
C(*b*8′)	0.5449 (4)	0.0720 (2)	0.4928 (3)	0.0424 (14)
C(*m*1′)	0.5855 (4)	0.0957 (2)	0.3743 (3)	0.0360 (13)
C(*m*2′)	0.7118 (4)	0.2231 (2)	0.2930 (3)	0.0313 (12)
C(*m*3′)	0.6847 (4)	0.2940 (2)	0.5209 (3)	0.0343 (13)
C(*m*4′)	0.5637 (4)	0.1651 (2)	0.6041 (3)	0.0342 (13)
C(11′)	0.5467 (5)	0.0552 (2)	0.3376 (3)	0.0422 (14)
C(12′)	0.4480 (5)	0.0459 (2)	0.3229 (4)	0.059 (2)
C(13′)	0.4099 (7)	0.0078 (3)	0.2906 (4)	0.080 (2)
C(14′)	0.4731 (9)	−0.0201 (3)	0.2755 (5)	0.095 (3)
C(15′)	0.5716 (9)	−0.0117 (2)	0.2895 (5)	0.089 (3)
C(16′)	0.6089 (6)	0.0258 (2)	0.3206 (4)	0.059 (2)
C(21′)	0.7383 (4)	0.2371 (2)	0.2241 (3)	0.0334 (13)
C(22′)	0.8116 (4)	0.2174 (2)	0.2019 (3)	0.0388 (14)
C(23′)	0.8410 (4)	0.2317 (2)	0.1419 (3)	0.047 (2)
C(24′)	0.7968 (5)	0.2662 (2)	0.1026 (4)	0.054 (2)
C(25′)	0.7211 (5)	0.2856 (2)	0.1220 (3)	0.048 (2)
C(26′)	0.6922 (4)	0.2713 (2)	0.1824 (3)	0.0385 (14)
C(31′)	0.7079 (4)	0.3362 (2)	0.5532 (3)	0.0402 (15)
C(32′)	0.7702 (5)	0.3419 (2)	0.6258 (4)	0.049 (2)
C(33′)	0.7981 (6)	0.3808 (2)	0.6549 (5)	0.071 (2)
C(34′)	0.7636 (8)	0.4149 (3)	0.6121 (6)	0.089 (3)
C(35′)	0.6994 (7)	0.4108 (2)	0.5408 (6)	0.082 (3)
C(36′)	0.6707 (5)	0.3716 (2)	0.5108 (4)	0.057 (2)
C(41′)	0.5250 (5)	0.1533 (2)	0.6678 (3)	0.0403 (14)
C(42′)	0.5835 (5)	0.1566 (2)	0.7425 (4)	0.053 (2)

C(43′)	0.5465 (8)	0.1457 (2)	0.8019 (4)	0.075 (2)
C(44′)	0.4521 (9)	0.1331 (2)	0.7866 (6)	0.089 (3)
C(45′)	0.3912 (7)	0.1296 (2)	0.7134 (6)	0.078 (3)
C(46′)	0.4283 (5)	0.1397 (2)	0.6542 (4)	0.057 (2)
O(1)	1.0282 (3)	0.15141 (12)	0.5952 (2)	0.0491 (10)
O(2)	0.5147 (3)	0.20571 (12)	0.3999 (2)	0.0422 (9)
O(3)	0.7714 (2)	0.17983 (10)	0.4984 (2)	0.0317 (8)
O(4)	0.3430 (7)	0.1593 (3)	0.3842 (10)	0.271 (8)
O(5)	1	0	1	0.17 (3)
O(6)	0.892 (3)	0.0365 (12)	0.936 (2)	0.23 (2)
C	1.147 (3)	0.0236 (12)	0.773 (2)	0.26 (2)
Cl(1)	1.130 (2)	0.0779 (9)	0.7659 (15)	0.487 (14)
Cl(2)	1.250 (2)	0.0233 (9)	0.849 (2)	0.52 (2)

Table 2. *Selected geometric parameters (Å, °)*

Mo(1)—O(1)	1.701 (4)	Mo(2)—O(2)	1.707 (4)
Mo(1)—O(3)	1.947 (3)	Mo(2)—O(3)	1.925 (3)
Mo(1)—N(1)	2.104 (4)	Mo(2)—N(1′)	2.103 (4)
Mo(1)—N(2)	2.095 (4)	Mo(2)—N(2′)	2.090 (4)
Mo(1)—N(3)	2.090 (4)	Mo(2)—N(3′)	2.093 (4)
Mo(1)—N(4)	2.095 (4)	Mo(2)—N(4′)	2.102 (4)
O(1)—Mo(1)—O(3)	176.2 (2)	O(2)—Mo(2)—N(1′)	91.6 (2)
O(1)—Mo(1)—N(1)	91.3 (2)	O(2)—Mo(2)—N(2′)	93.4 (2)
O(1)—Mo(1)—N(2)	93.2 (2)	O(2)—Mo(2)—N(3′)	94.1 (2)
O(1)—Mo(1)—N(3)	94.3 (2)	O(2)—Mo(2)—N(4′)	90.0 (2)
O(1)—Mo(1)—N(4)	90.9 (2)	O(3)—Mo(2)—N(1′)	86.1 (2)
O(3)—Mo(1)—N(1)	86.4 (2)	O(3)—Mo(2)—N(2′)	87.9 (2)
O(3)—Mo(1)—N(2)	89.8 (2)	O(3)—Mo(2)—N(3′)	88.1 (2)
O(3)—Mo(1)—N(3)	87.9 (2)	O(3)—Mo(2)—N(4′)	88.62 (15)
O(3)—Mo(1)—N(4)	86.1 (2)	N(1′)—Mo(2)—N(2′)	90.2 (2)
N(1)—Mo(1)—N(2)	90.1 (2)	N(1′)—Mo(2)—N(3′)	174.2 (2)
N(1)—Mo(1)—N(3)	174.3 (2)	N(1′)—Mo(2)—N(4′)	89.7 (2)
N(1)—Mo(1)—N(4)	89.9 (2)	N(2′)—Mo(2)—N(3′)	89.9 (2)
N(2)—Mo(1)—N(3)	89.1 (2)	N(2′)—Mo(2)—N(4′)	176.5 (2)
N(2)—Mo(1)—N(4)	175.9 (2)	N(3′)—Mo(2)—N(4′)	89.8 (2)
N(3)—Mo(1)—N(4)	90.4 (2)	Mo(1)—O(3)—Mo(2)	178.5 (2)
O(2)—Mo(2)—O(3)	177.4 (2)		

The intensity data were collected at room temperature because of increased reflection breadth at lower temperature. The room temperature data hampered refinement of the diffuse solvent molecules. One water molecule (assigned occupancy 1.0) was within hydrogen-bonding distance (2.8 Å) of the terminal oxo atom O(2) of the dimer. A second water site contained two positions [O(5) and O(6)] 2.0 Å apart that were constrained to have total occupancy of 1.0 (actual total occupancy was 0.5). A third solvent site was partially occupied with one half molecule of CH_2Cl_2. Cell determination and intensity-data collection were performed on an Enraf–Nonius FAST area-detector diffractometer with an Mo rotating-anode source. Our detailed methods and procedures for small-molecule X-ray data collection with the FAST system have been described previously (Scheidt & Turowska-Tyrk, 1994). All reflections were used in least-squares refinement, including negative intensities.

Data collection: *MADNES* (Messerschmitt & Pflugrath, 1987). Cell refinement: *MADNES*. Data reduction: *MADNES*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *SHELXL*93.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: BK1173). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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A Trinuclear Cluster of Molybdenum(IV), $Mo_3(\mu_3\text{-}S)(\mu_2\text{-}S)_3(HC_2O_4)_2(C_2O_4)(H_2O)_3$

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Abstract

The three molybdenum centers in triaqua-$1\kappa O$;$2\kappa O$;$3\kappa O$-bis[oxalato(1−)]-$2\kappa^2 O,O''$;$3\kappa^2 O,O''$-oxalato(2−)-$1\kappa^2 O,O''$-μ_3-thio-tri-μ-thio-$1{:}2\kappa^2 S$;$1{:}3\kappa^2 S$;$2{:}3\kappa^2 S$-*triangulo*-trimolybdenum diethanol solvate, $[Mo_3S_4(C_2HO_4)_2(C_2O_4)(H_2O)_3].2C_2H_6O$, form an equilateral triangle with an average Mo—Mo distance of 2.736 (4) Å. Each Mo atom is in a distorted octahedral environment made up of two O atoms of an oxalate ligand, one O atom of a water molecule, one capping-sulfur and two bridging-sulfur ligands. The structure is further stabilized by intermolecular hydrogen bonding between the oxalate and water ligands.