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

As(1)—O(1)	1·652 (1)	As(1)—O(2)	1·707 (2)
As(1)—O(3)	1·708 (2)	As(1)—C(1)	1·928 (2)
O(4)—C(7)	1·220 (3)	C(1)—C(2)	1·385 (3)
C(1)—C(6)	1·399 (3)	C(2)—C(3)	1·396 (3)
C(3)—C(4)	1·378 (4)	C(4)—C(5)	1·370 (4)
C(5)—C(6)	1·398 (3)	C(6)—C(7)	1·489 (3)
C(7)—C(8)	1·484 (3)	C(8)—C(9)	1·398 (3)
C(8)—C(13)	1·399 (3)	C(9)—C(10)	1·388 (4)
C(10)—C(11)	1·371 (5)	C(11)—C(12)	1·374 (4)
C(12)—C(13)	1·375 (4)		

O(1)—As(1)—O(2)	110·6 (1)	O(1)—As(1)—O(3)	114·9 (1)
O(2)—As(1)—O(3)	101·9 (1)	O(1)—As(1)—C(1)	116·5 (1)
O(2)—As(1)—C(1)	100·7 (1)	O(3)—As(1)—C(1)	110·3 (1)
As(1)—C(1)—C(2)	116·9 (2)	As(1)—C(1)—C(6)	122·3 (2)
C(2)—C(1)—C(6)	120·8 (2)	C(1)—C(2)—C(3)	119·7 (2)
C(2)—C(3)—C(4)	119·7 (2)	C(3)—C(4)—C(5)	120·5 (2)
C(4)—C(5)—C(6)	121·2 (2)	C(1)—C(6)—C(5)	118·0 (2)
C(1)—C(6)—C(7)	120·2 (2)	C(5)—C(6)—C(7)	121·9 (2)
O(4)—C(7)—C(6)	118·9 (2)	O(4)—C(7)—C(8)	120·1 (2)
C(6)—C(7)—C(8)	120·9 (2)	C(7)—C(8)—C(9)	122·8 (2)
C(7)—C(8)—C(13)	118·5 (2)	C(9)—C(8)—C(13)	118·5 (2)
C(8)—C(9)—C(10)	120·0 (3)	C(9)—C(10)—C(11)	120·6 (2)
C(10)—C(11)—C(12)	119·8 (3)	C(11)—C(12)—C(13)	120·9 (3)
C(8)—C(13)—C(12)	120·2 (2)		

Fig. 1. View of the title molecule with atom numbering.

Related literature. For the preparation of the compound see Aeschlimann & McCleland (1924), who also assigned the structure correctly; for spectroscopic studies see Parmar & Saluja (1988). For spectroscopic studies of related compounds see Parmar, Basra, Malhotra & Sandhu (1980, 1981).

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Structure of Pentacarbonyl(cycloheptatrienyl)(cyclopentadienyl)dimolybdenum(*Mo–Mo*)

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Abstract. $[Mo_2(C_5H_5)(C_7H_7)(CO)_5]$, $M_r = 488\cdot2$, monoclinic, $P2_1/c$, $a = 9\cdot949$ (2), $b = 8\cdot568$ (1), $c = 20\cdot580$ (5) Å, $\beta = 110\cdot00$ (3)°, $V = 1648\cdot5$ Å³, $Z = 4$, $D_x = 1\cdot97$ Mg m⁻³, $\lambda(Mo\,K\alpha) = 0\cdot71069$ Å, $\mu = 1\cdot67$ mm⁻¹, $F(000) = 952$, $T = 294$ K, $R = 0\cdot0222$ for 2393 unique reflections with $F > 3\sigma(F)$. The molecule consists of $Mo(\eta\text{-}C_7H_7)(CO)_2$ and $Mo(\eta\text{-}C_5H_5)(CO)_3$ fragments linked by an unsupported Mo–Mo bond of length 3·160 (1) Å.

Experimental. The title compound was prepared by reaction of $[Mo(\eta\text{-}C_7H_7)(NCMe)(CO)_2][BF_4]$ with Li-$[Mo(\eta\text{-}C_5H_5)(CO)_3]$ (Breeze, Ricalton & Whiteley,

1987); crystallization by slow diffusion of hexane into a CH_2Cl_2 solution of the compound afforded dark-green crystals. Crystal size $0\cdot075 \times 0\cdot16 \times 0\cdot28$ mm, CAD-4 diffractometer, cell parameters were derived from the setting angles of 25 reflections ($19 < 2\theta < 25°$), no absorption or extinction correction, no significant systematic drift in one standard reflection measured at $2\frac{1}{2}$ h intervals during data collection. Scan mode $\omega/2\theta$, scan width $= (0\cdot6 + 0\cdot35\tan\theta)°$, $(\sin\theta/\lambda)_{max} = 0\cdot595$ Å⁻¹, scan time 7 to 120 s, $h\,0{\rightarrow}11$, $k\,0{\rightarrow}10$, $l\,{-}24{\rightarrow}22$. 3104 measured reflections, all unique, 2393 with $F > 3\sigma(F)$ used in refinement. Structure solved by the heavy-atom method, refinement was by partitioned

Table 1. *Atomic coordinates* $(\times 10^4)$ *and equivalent isotropic thermal parameters* (Å^2)

$$B_{eq} = \tfrac{8}{3}\pi^2 \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i . \mathbf{a}_j .$$

	x	y	z	B_{eq}
Mo(1)	3052·5 (3)	2593·6 (3)	712·9 (1)	2.74
Mo(2)	2077·1 (3)	2242·7 (3)	2014·5 (1)	2.64
C(3)	3228 (4)	276 (4)	881 (2)	3.7
O(4)	3335 (3)	−1037 (3)	956 (2)	5.7
C(5)	960 (4)	2136 (4)	312 (2)	4.0
O(6)	−235 (3)	1887 (4)	65 (2)	5.9
C(7)	4158 (3)	2094 (4)	2233 (2)	3.2
O(8)	5383 (3)	1929 (3)	2469 (1)	4.8
C(9)	2748 (4)	3673 (4)	2796 (2)	3.6
O(10)	3149 (3)	4476 (3)	3270 (1)	5.3
C(11)	1442 (4)	4239 (4)	1528 (2)	3.8
O(12)	934 (3)	5413 (3)	1300 (2)	5.8
C(13)	3849 (7)	2627 (6)	−197 (3)	6.8
C(14)	5026 (5)	2347 (5)	386 (3)	5.5
C(15)	5496 (4)	3119 (5)	1008 (2)	4.8
C(16)	4897 (5)	4335 (4)	1231 (2)	4.9
C(17)	3729 (5)	5213 (4)	869 (3)	5.5
C(18)	2826 (6)	5068 (5)	207 (3)	6.7
C(19)	2818 (6)	3840 (8)	−279 (2)	8.0
C(20)	665 (4)	−5 (5)	1529 (2)	4.6
C(21)	1698 (4)	−448 (4)	2152 (2)	4.0
C(22)	1440 (4)	379 (4)	2681 (2)	4.2
C(23)	256 (4)	1348 (5)	2382 (2)	5.2
C(24)	−225 (4)	1116 (5)	1670 (2)	5.2

full-matrix least squares and difference Fourier synthesis, least-squares refinement on *F*. H atoms were introduced at calculated positions and positional and isotropic thermal parameters refined individually, all non-H atoms were refined anisotropically. In the final cycles a weighting scheme, $w = 1/(1·31 − 0·042F + 0·00066F^2)^{1/2}$, was adopted to give uniform wA^2 distribution over the *F* range. $R = 0·0222$, $wR = 0·0270$, $S = 1·081$, $(A/\sigma)_{max} = 0·30$ for positional parameters, 0·42 for thermal parameters, $(\Delta\rho)_{max} = 0·5$, $(\Delta\rho)_{min} = −0·3$ e Å^{-3}. Scattering factors from *International Tables for X-ray Crystallography* (1974). With the exception of *ORTEP* (Johnson, 1965) all computer programs were written in this Department. Table 1 gives atom parameters* and Table 2 bond lengths and angles. Fig. 1 shows a view of the molecule.

Related literature. The title compound is the first crystallographically characterized, metal–metal bonded binuclear complex which incorporates the dicarbonyl-(cycloheptatrienyl)molybdenum fragment; comparative structural data for this fragment are provided by $[Mo(\eta\text{-}C_7H_7)(CO)_2(\sigma\text{-}C_6H_5)]$ (Churchill & O'Brien, 1969) and $[Mo(\eta\text{-}C_7H_7)(CO)_2Br]$ (Ziegler, Sasse & Nuber, 1975). The Mo—Mo bond length is shorter than that in closely related binuclear complexes
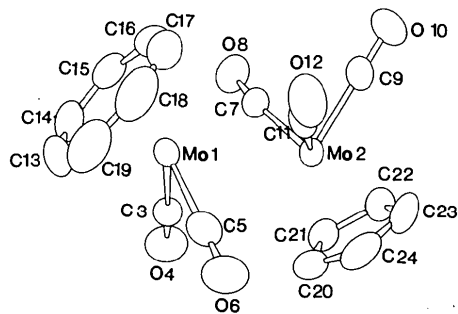
Table 2. *Bond lengths* (Å) *and angles* (°)

Mo(1)—Mo(2)	3·160 (1)	C(19)—C(13)	1·429 (8)	
Mo(1)—C(3)	2·013 (3)	Mo(2)—C(7)	1·966 (3)	
Mo(1)—C(5)	1·998 (4)	Mo(2)—C(9)	1·949 (3)	
Mo(1)—C(13)	2·270 (4)	Mo(2)—C(11)	1·973 (3)	
Mo(1)—C(14)	2·292 (4)	Mo(2)—C(20)	2·392 (4)	
Mo(1)—C(15)	2·339 (4)	Mo(2)—C(21)	2·369 (3)	
Mo(1)—C(16)	2·322 (4)	Mo(2)—C(22)	2·331 (3)	
Mo(1)—C(17)	2·334 (4)	Mo(2)—C(23)	2·320 (3)	
Mo(1)—C(18)	2·338 (4)	Mo(2)—C(24)	2·360 (4)	
Mo(1)—C(19)	2·245 (4)	C(7)—O(8)	1·156 (4)	
C(3)—O(4)	1·136 (4)	C(9)—O(10)	1·149 (4)	
C(5)—O(6)	1·142 (4)	C(11)—O(12)	1·152 (4)	
C(13)—C(14)	1·380 (7)	C(20)—C(21)	1·394 (5)	
C(14)—C(15)	1·374 (6)	C(21)—C(22)	1·395 (5)	
C(15)—C(16)	1·356 (6)	C(22)—C(23)	1·399 (6)	
C(16)—C(17)	1·371 (6)	C(23)—C(24)	1·391 (6)	
C(17)—C(18)	1·357 (7)	C(24)—C(20)	1·402 (6)	
C(18)—C(19)	1·451 (8)			
C(13)—Mo(1)—C(14)	35·2 (2)	C(20)—Mo(2)—C(21)	34·0 (1)	
C(14)—Mo(1)—C(15)	34·5 (2)	C(21)—Mo(2)—C(22)	34·5 (1)	
C(15)—Mo(1)—C(16)	33·8 (1)	C(22)—Mo(2)—C(23)	35·0 (1)	
C(16)—Mo(1)—C(17)	34·2 (2)	C(23)—Mo(2)—C(24)	34·6 (2)	
C(17)—Mo(1)—C(18)	33·8 (2)	C(24)—Mo(2)—C(20)	34·3 (1)	
C(18)—Mo(1)—C(19)	36·9 (2)	C(7)—Mo(2)—C(9)	79·4 (1)	
C(19)—Mo(1)—C(13)	36·9 (2)	C(7)—Mo(2)—C(11)	107·5 (1)	
C(3)—Mo(1)—C(5)	84·0 (1)	C(9)—Mo(2)—C(11)	80·4 (1)	
Mo(1)—C(3)—O(4)	177·7 (3)	Mo(2)—C(17)—O(8)	168·5 (3)	
Mo(1)—C(5)—O(6)	178·1 (3)	Mo(2)—C(9)—O(10)	177·8 (3)	
C(3)—Mo(1)—Mo(2)	78·1 (1)	Mo(2)—C(11)—O(12)	169·7 (3)	
C(5)—Mo(1)—Mo(2)	75·7 (1)	C(7)—Mo(2)—Mo(1)	66·1 (1)	
C(13)—C(14)—C(15)	130·1 (4)	C(9)—Mo(2)—Mo(1)	121·4 (1)	
C(14)—C(15)—C(16)	129·5 (4)	C(11)—Mo(2)—Mo(1)	68·0 (1)	
C(15)—C(16)—C(17)	128·9 (4)	C(20)—C(21)—C(22)	107·8 (3)	
C(16)—C(17)—C(18)	129·9 (4)	C(21)—C(22)—C(23)	108·0 (4)	
C(17)—C(18)—C(19)	127·3 (4)	C(22)—C(23)—C(24)	108·3 (4)	
C(18)—C(19)—C(13)	127·7 (5)	C(23)—C(24)—C(20)	107·6 (4)	
C(19)—C(13)—C(14)	126·2 (4)	C(24)—C(20)—C(21)	108·4 (4)	

Fig. 1. *ORTEP* diagram and atomic numbering.

containing unsupported Mo—Mo single bonds, $[Mo_2(\eta\text{-}C_5H_5)_2(CO)_6]$, 3·235 Å (Adams, Collins & Cotton, 1974) and $[Mo_2(\eta\text{-}C_5Me_5)_2(CO)_6]$, 3·281 Å (Clegg, Compton, Errington & Norman, 1988). Additional features of note are the *trans* arrangement of the cyclopentadienyl and cycloheptatrienyl ligands and, in the tricarbonyl(cyclopentadienyl)molybdenum fragment, the bending at C of the two carbonyls located *cis* to the Mo—Mo bond.

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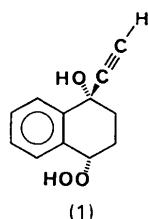
1-Ethynyl-4-hydroperoxy-1,2,3,4-tetrahydro-1-naphthol

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Abstract. $C_{12}H_{12}O_3$, $M_r = 204.2$, monoclinic, $P2_1/c$, $a = 11.815$ (2), $b = 7.673$ (2), $c = 12.580$ (3) Å, $\beta = 115.06$ (2)°, $V = 1033.1$ (9) Å^3, $Z = 4$, $D_x = 1.313$ g cm^{-3}, $\lambda(Cu\,K\alpha) = 1.54184$ Å, $\mu = 7.34$ cm^{-1}, $F(000) = 432$, $T = 299$ K, $R = 0.065$ for 1420 observations (of 2131 unique data). The stereochemistry of the hydroxyl and hydroperoxyl groups is *cis*. The hydroperoxyl O—O bond distance is 1.476 (2) Å. The hydrogen-bonding pattern consists of two intermolecular interactions: a hydroperoxyl donor to the hydroxyl group, with an O···O distance of 2.818 (2) Å and an O—H···O angle of 168 (2)°, and a hydroxyl donor to O(3) of the hydroperoxyl group, with a distance and angle of 2.670 (2) Å and 170 (2)°, respectively.

Experimental. Colorless needles, m.p. 426–428 K, of 1-ethynyl-4-hydroperoxy-1,2,3,4-tetrahydro-1-naphthol (1) were isolated

(1)

from a mixture of 1-tetralone and 1-ethynyl-1,2,-3,4-tetrahydro-1-naphthol in benzene, which was allowed to evaporate slowly over a period of 3 weeks. The rate of autooxidation of tetralins is increased by the

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Table 1. *Coordinates and equivalent isotropic thermal parameters*

$$B_{eq} = \tfrac{4}{3}(a^2\beta_{11} + b^2\beta_{22} + c^2\beta_{33} + ac\beta_{13}\cos\beta).$$

	x	y	z	$B_{eq}(\text{Å}^2)$
O(1)	0.1313 (2)	−0.0616 (3)	0.1309 (2)	5.26 (6)
O(2)	0.1673 (2)	0.2560 (3)	0.4530 (2)	4.83 (5)
O(3)	0.0869 (2)	0.2583 (3)	0.5163 (2)	5.29 (6)
C(1)	0.2937 (2)	0.0282 (4)	0.3128 (2)	3.22 (6)
C(2)	0.3753 (2)	0.0570 (5)	0.2595 (2)	3.83 (7)
C(3)	0.4782 (2)	0.1620 (5)	0.3112 (2)	4.42 (8)
C(4)	0.5045 (2)	0.2409 (5)	0.4170 (2)	4.21 (8)
C(5)	0.4244 (3)	0.2145 (5)	0.4706 (2)	3.94 (7)
C(6)	0.3190 (2)	0.1100 (4)	0.4190 (2)	3.33 (6)
C(7)	0.2333 (2)	0.0902 (4)	0.4803 (2)	4.00 (7)
C(8)	0.1440 (3)	−0.0599 (5)	0.4339 (3)	5.18 (8)
C(9)	0.0840 (2)	−0.0611 (5)	0.3019 (3)	5.01 (8)
C(10)	0.1830 (2)	−0.0931 (4)	0.2550 (2)	3.72 (7)
C(11)	0.2239 (2)	−0.2761 (4)	0.2748 (2)	3.80 (7)
C(12)	0.2504 (3)	−0.4217 (5)	0.2874 (3)	5.52 (9)

presence of a ketone (Robertson & Waters, 1948). Crystal size 0.12 × 0.16 × 0.40 mm, space group from systematic absences $h0l$ with l odd and $0k0$ with k odd, cell dimensions from setting angles of 25 reflections having $25 < \theta < 30°$. Data collection on Enraf–Nonius CAD-4 diffractometer, Cu $K\alpha$ radiation, graphite monochromator, ω–2θ scans designed for $I = 50\sigma(I)$, subject to max. scan time = 120 s, scan rates varied 0.59–3.28° min^{-1}. Data having $2 < \theta < 75°$, $0 \le h \le 14$, $0 \le k \le 9$, $-15 \le l \le 15$ measured. Data corrected for background, Lorentz, polarization, decay and absorption effects. Absorption corrections were based on ψ scans, with a minimum relative transmission coefficient of 69.90%. Standard reflections 200, 060, 004 indicated a 14.1% decay and a linear correction was applied. $R_{int} = 0.033$ for averaging $0kl$ and $0k\bar{l}$ data, 2131 unique data, 1420