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The Crystal and Molecular Structure of μ-(η⁵:η⁵-Fulvalene)-μ-hydrido-μ-hydroxyl-bis(η⁵-cyclopentadienylmolybdenum) Hexafluorophosphate Hemihydrate

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The title complex crystallizes in the monoclinic space group $P2_1$ with $a = 10 \cdot 248$ (6), $b = 12 \cdot 814$ (7), $c = 9 \cdot 995$ (5) Å, $\beta = 107 \cdot 35$ (2)°, $Z = 2$. The structure was determined from four-circle diffractometer data and refined by least-squares calculations to $R = 0 \cdot 073$ for 2004 reflexions. The cation consists of two Mo(η^5-C_5H_5) moieties which are bridged by the H atom, the hydroxyl ligand, and the pentafulvalene ligand, which is η^5-bonded to both Mo atoms.

Introduction

The working up of the reaction products from the treatment of the tetramer [Mo(η^5-C_5H_5)$_2$HLi]$_4$ (Francis, Green, Luong-thi & Moser, 1976) with nitrous oxide yields three compounds whose mass spectra and analytical data are identical and correspond to the stoichiometry [$C_{10}H_{10}Mo$]$_2$. Prolonged treatment of each of these compounds with aqueous HPF$_6$ gives the same compound, a brown crystalline solid of stoichiometry $C_{20}H_{21}F_{12}Mo_2O_{1 \cdot 5}P_2$ whose crystal structure we report. This work has been the subject of a preliminary publication (Green, Cooper, Couldwell & Prout, 1977).

Experimental

Brown needles of [Mo$_2$(η^5-C_5H_5)$_2$(μ-η^5-C_5H_4-η^5-C_5H_4)(μ-H)(μ-OH)]2PF$_6 \cdot \frac{1}{2}H_2O$, were kindly supplied by Dr N. J. Cooper and Dr M. L. H. Green. The compound is sensitive to air and moisture and was mounted under dry nitrogen in a glass capillary. It was not possible to measure its density.

After survey photography the selected crystal (0·13 × 1·5 × 0·06 mm) was set up on a Hilger & Watts PDP8-controlled four-circle diffractometer and cell dimensions and orientation matrix were obtained by least squares from the setting angles of 20 reflexions.

The intensity of each independent reflexion with $\sin \theta / \lambda < 0 \cdot 66$ was measured with an $\omega/2\theta$ scan and the ordinate analysis method (Watson, Shotton, Cox & Muirhead, 1970) with 50 steps of 0·02°. Mo $K\alpha$ radiation from a graphite monochromator was used. Reflexions with $I < 3\sigma$, where σ is the standard deviation based on simple counting statistics, and whose apparent centre was more than 0·14° from the predicted position were excluded from subsequent calculations. Lorentz and polarization corrections were applied together with an empirical absorption correction (North, Phillips & Mathews, 1968) to yield a set of 2006 independent structure amplitudes.

Crystal data

$C_{20}H_{21}F_{12}Mo_2O_{1 \cdot 5}P_2$, $M_r = 767 \cdot 2$. Monoclinic, $a = 10 \cdot 248$ (6), $b = 12 \cdot 814$ (7), $c = 9 \cdot 995$ (5) Å, $\beta =$

107·35 (2)°, $U = 1252·8$ Å³. Systematic extinctions $0k0$: $k = 2n + 1$. Space group $P2_1$ or $P2_1/m$; $P2_1$ from structure analysis. $D_c = 2·03$ g cm⁻³ for $Z = 2$. Mo $K\alpha$ radiation, $\lambda = 0·71069$ Å, $\mu = 12$ cm⁻¹.

Structure solution and refinement

The structure was solved by Patterson and Fourier techniques, and during refinement the sum of the coordinates along the polar axis was set to zero to determine the origin (Carruthers, 1975). Refinement was by least squares with a large-block approximation to the normal matrix: a 2 × 2 block was calculated from the derivatives of the scale and dummy overall temperature factor, two blocks from those of the positional parameters of the cation and remaining atoms respectively, and a further two from those of the corresponding temperature factors. All atoms, except the water O atom, which was refined isotropically, had anisotropic temperature factors. Difference syntheses permitted the location of some of the H atoms in their expected positions, but they were not included in the final model. The refinement converged at $R = 0·09$, but the temperature factors of the anions and their geometry were poor. The P—F lengths were, therefore, constrained (Waser, 1963; Rollett, 1969) to their mean (recalculated between each cycle) with an estimated standard deviation of 0·02 Å; a similar procedure with an e.s.d. of 1° was adopted for the F—P—F angles. These means converged to 1·55 Å and 90° (or 180°) respectively, satisfactory values for PF_6^- groups. In addition, the difference in mean square displacement along the bond direction of the two atoms forming each bond was constrained to be zero with an e.s.d. of 0·005 Å². Two low-angle reflexions, 020 and 20$\bar{1}$, were given zero weight in the last cycles of refinement because of suspected extinction effects. Each reflexion was assigned a weight ω $= 1/\Sigma_{r=1}^n A_r T_r(X)$ where n is the number of coefficients, A_r, for a Chebyshev series, T_r is the polynomial function, and X is $F_o/F_s(\text{max})$. Three coefficients, A_r, were used with values 93·9, 131·3 and 48·0 (Rollett, 1965) and the final R was 0·073 for 2004 reflexions. All calculations were performed on the Oxford University ICL 1906A computer with the Oxford *CRYSTALS* package (Carruthers, 1975). Complex neutral-atom scattering factors were taken from *International Tables for X-ray Crystallography* (1974).

Results and discussion

The final atomic positional parameters are given in Table 1.* Projections of the crystal and molecular

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division, as Supplementary Publication No. SUP 32425 (20 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

structures are shown in Figs. 1, 2 and 3. Bond distances and angles, with estimated standard deviations calculated from the full variance–covariance matrix, and the equations of important molecular planes are given in Tables 2 and 3.

The asymmetric unit of the crystal contains one cation, two anions and half a water molecule all at general positions (Fig. 1), with the water molecule hydrogen-bonded to the O atom of the bridging hydroxyl group, $O(1) \cdots O(2)$, 2·51 Å. There are three short $O(2) \cdots F$ contacts, two to F(26) and F(25) of 2·97 and 3·17 Å, and one to F(13) of 2·95 Å. These contacts are much longer than in well-authenticated $O—H \cdots F$ bonds, e.g. 2·68 Å in $FeSiF_6 \cdot 6H_2O$ (Hamilton, 1962). The short contacts are not thought to represent necessarily true $OH \cdots F$ hydrogen bonds though it is recognized that there will be strong ion–dipole and dipole–dipole interactions between the anions and the water molecule.

Although the ill-resolved structural features of the

Table 1. *Fractional atomic coordinates* (×10³) *and isotropic temperature factor*

	x	y	z	U_{iso}
Mo(1)	213·1 (1)	258·0 (3)	7·2 (1)	
Mo(2)	401·6 (1)	233·0 (3)	306·1 (1)	
O(1)	193 (1)	214·7 (9)	200 (1)	
C(11)	124 (2)	413 (1)	73 (2)	
C(12)	72 (2)	406 (2)	−82 (2)	
C(13)	191 (2)	410 (1)	−126 (2)	
C(14)	312 (3)	414 (1)	−10 (2)	
C(15)	270 (2)	416 (1)	115 (2)	
C(21)	192 (3)	83 (2)	−33 (2)	
C(22)	288 (3)	118 (2)	−95 (3)	
C(23)	218 (3)	186 (2)	−207 (3)	
C(24)	82 (3)	194 (1)	−207 (2)	
C(25)	66 (2)	135 (2)	−105 (2)	
C(31)	506 (2)	389 (1)	290 (2)	
C(32)	546 (2)	362 (2)	443 (2)	
C(33)	424 (2)	357 (2)	488 (2)	
C(34)	318 (2)	387 (2)	375 (2)	
C(35)	361 (2)	404 (1)	252 (2)	
C(41)	473 (3)	69 (2)	255 (3)	
C(42)	590 (3)	128 (2)	341 (3)	
C(43)	566 (3)	145 (2)	472 (4)	
C(44)	442 (4)	103 (2)	469 (3)	
C(45)	383 (3)	52 (1)	334 (3)	
P(1)	674·2 (4)	260·0 (4)	903·9 (5)	
F(11)	727 (2)	160 (1)	851 (2)	
F(12)	623 (2)	362 (1)	957 (2)	
F(13)	816 (1)	278 (2)	1008 (1)	
F(14)	531 (1)	245 (2)	800 (1)	
F(15)	631 (1)	197 (1)	1014 (1)	
F(16)	718 (2)	324 (1)	792 (1)	
P(2)	−61·1 (5)	421·2 (4)	−541·2 (5)	
F(21)	−192 (1)	490 (1)	−605 (1)	
F(22)	70 (1)	354 (1)	−478 (1)	
F(23)	−28 (1)	425 (1)	−685 (1)	
F(24)	−94 (1)	417 (1)	−398 (1)	
F(25)	−149 (1)	321 (1)	−589 (1)	
F(26)	26 (2)	522 (1)	−492 (2)	
O(2)	979 (2)	156 (2)	251 (2)	0·051 (5)

$[Mo_2(\eta^5\text{-}C_5H_5)_2(\mu\text{-}\eta^5\text{-}C_5H_4\text{-}\eta^5\text{-}C_5H_4)(\mu\text{-}H)(\mu\text{-}OH)]2PF_6.\tfrac{1}{2}H_2O$

anions are probably the result of disorder, constrained refinement led to chemically reasonable values at convergence. As would be expected, the shorter P—F lengths are associated with the anion at P(1), that with greater apparent thermal motion as indicated by the anisotropic temperature factors.

Although there is no direct evidence for the H atom from electron density distribution, its presence is inferred from NMR measurements and it is required to bring each of the Mo atoms to the 18(e) configuration. The hypothetical position, marked by broken lines in the figures, is similar to those in the bent M—H—M systems, found by neutron diffraction, in $(\eta^5\text{-}C_5H_5)_2$-

Table 2. *Interatomic distances* (Å) *and bond angles* (°)

Mo(1)—Mo(2)	3·053 (2)		C(11)—C(12)	1·48 (3)
Mo(1)—O(1)	2·07 (1)		C(12)—C(13)	1·42 (3)
Mo(2)—O(1)	2·09 (1)		C(13)—C(14)	1·43 (3)
Mo(1)—C(11)	2·36 (2)		C(14)—C(15)	1·44 (3)
Mo(1)—C(12)	2·39 (2)		C(15)—C(11)	1·42 (3)
Mo(1)—C(13)	2·33 (2)		C(21)—C(22)	1·38 (3)
Mo(1)—C(14)	2·28 (2)		C(22)—C(23)	1·43 (4)
Mo(1)—C(15)	2·29 (2)		C(23)—C(24)	1·40 (4)
Mo(1)—C(21)	2·28 (2)		C(24)—C(25)	1·31 (3)
Mo(1)—C(22)	2·31 (2)		C(25)—C(21)	1·44 (3)
Mo(1)—C(23)	2·35 (3)		C(31)—C(32)	1·50 (3)
Mo(1)—C(24)	2·32 (2)		C(32)—C(33)	1·45 (3)
Mo(1)—C(25)	2·24 (2)		C(33)—C(34)	1·37 (3)
Mo(2)—C(31)	2·29 (2)		C(34)—C(35)	1·45 (3)
Mo(2)—C(32)	2·36 (2)		C(35)—C(31)	1·43 (3)
Mo(2)—C(33)	2·37 (2)		C(41)—C(42)	1·46 (4)
Mo(2)—C(34)	2·34 (2)		C(42)—C(43)	1·41 (4)
Mo(2)—C(35)	2·27 (2)		C(43)—C(44)	1·37 (4)
Mo(2)—C(41)	2·33 (2)		C(44)—C(45)	1·46 (4)
Mo(2)—C(42)	2·29 (2)		C(45)—C(41)	1·40 (4)
Mo(2)—C(43)	2·28 (3)		C(15)—C(35)	1·42 (3)
Mo(2)—C(44)	2·28 (2)			
Mo(2)—C(45)	2·35 (2)			
P(1)—F(11)	1·55		P(2)—F(21)	1·57
P(1)—F(12)	1·56		P(2)—F(22)	1·56
P(1)—F(13)	1·53		P(2)—F(23)	1·57
P(1)—F(14)	1·53		P(2)—F(24)	1·57
P(1)—F(15)	1·53		P(2)—F(25)	1·56
P(1)—F(16)	1·56		P(2)—F(26)	1·56

Mo(1)—O(1)—Mo(2)	94·3 (6)

C(15)—C(11)—C(12)	109 (2)		C(35)—C(31)—C(32)	104 (2)
C(11)—C(12)—C(13)	104 (2)		C(31)—C(32)—C(33)	109 (2)
C(12)—C(13)—C(14)	112 (2)		C(32)—C(33)—C(34)	106 (2)
C(13)—C(14)—C(15)	107 (2)		C(33)—C(34)—C(35)	112 (2)
C(14)—C(15)—C(11)	108 (2)		C(34)—C(35)—C(31)	108 (2)
C(11)—C(15)—C(35)	128 (2)		C(31)—C(35)—C(15)	127 (2)
C(14)—C(15)—C(35)	123 (2)		C(34)—C(35)—C(15)	124 (2)
C(25)—C(21)—C(22)	106 (2)		C(45)—C(41)—C(42)	108 (2)
C(21)—C(22)—C(23)	107 (2)		C(41)—C(42)—C(43)	106 (2)
C(22)—C(23)—C(24)	108 (2)		C(42)—C(43)—C(44)	110 (2)
C(23)—C(24)—C(25)	109 (2)		C(43)—C(44)—C(45)	108 (3)
C(24)—C(25)—C(21)	111 (2)		C(44)—C(45)—C(41)	107 (2)

F(11)—P(1)—F(12)	179		F(21)—P(2)—F(22)	179
F(11)—P(1)—F(13)	90		F(21)—P(2)—F(23)	90
F(11)—P(1)—F(14)	91		F(21)—P(2)—F(24)	90
F(11)—P(1)—F(15)	91		F(21)—P(2)—F(25)	89
F(11)—P(1)—F(16)	89		F(21)—P(2)—F(26)	90
F(12)—P(1)—F(13)	90		F(22)—P(2)—F(23)	89
F(12)—P(1)—F(14)	89		F(22)—P(2)—F(24)	90
F(12)—P(1)—F(15)	90		F(22)—P(2)—F(25)	91
F(12)—P(1)—F(16)	91		F(22)—P(2)—F(26)	89
F(13)—P(1)—F(14)	179		F(23)—P(2)—F(24)	180
F(13)—P(1)—F(15)	91		F(23)—P(2)—F(25)	90
F(13)—P(1)—F(16)	89		F(23)—P(2)—F(26)	90
F(14)—P(1)—F(15)	90		F(24)—P(2)—F(25)	89
F(14)—P(1)—F(16)	90		F(24)—P(2)—F(26)	90
F(15)—P(1)—F(16)	180		F(25)—P(2)—F(26)	179

Table 3. *Least-squares planes*

In the equations, x, y and z represent fractional coordinates with respect to the crystallographic axes. The table gives the displacement (Å) of the specified atom from the plane.

Plane 1: ring C(11) to C(15)

Equation of plane: $-0·325x + 12·797y - 0·299z = 5·210$

C(11)	0·010	C(14)	−0·005
C(12)	−0·013	C(15)	−0·003
C(13)	0·011	Mo(1)*	−1·980

Normal to Mo(1) meets plane 0·11 Å from centroid.

Plane 2: ring C(21) to C(25)

Equation of plane: $0·507x + 9·890y + 5·900z = 0·736$

C(21)	−0·018	C(24)	−0·004
C(22)	0·016	C(25)	0·014
C(23)	−0·008	Mo(1)*	1·966

Normal to Mo(1) meets plane 0·09 Å from centroid.

Plane 3: ring C(31) to C(35)

Equation of plane: $-0·716x - 12·362y - 2·215z = -5·823$

C(31)	0·011	C(34)	−0·023
C(32)	−0·024	C(35)	0·006
C(33)	0·030	Mo(2)*	1·977

Normal to Mo(2) meets plane 0·11 Å from centroid.

Plane 4: ring C(41) to C(45)

Equation of plane : $-3·865x + 10·980y - 2·229z = -1·638$

C(41)	0·003	C(44)	0·017
C(42)	0·007	C(45)	−0·012
C(43)	−0·015	Mo(2)*	1·962

Normal to Mo(2) meets plane 0·06 Å from centroid.

Plane 5: Mo(1) Mo(2) Q(4) Q(3)†

Equation of plane: $9·395x + 1·686y - 6·330z = 2·365$

Q(4)	0·060	Mo(2)	−0·137
Q(3)	0·050	Q(2)*	0·209
Mo(1)	0·027	Q(1)*	0·262

* Atom not included in the plane calculation.

† Q(1) and Q(2) are the coordinates of the normals from Mo(1) to planes 1 and 2, respectively. Similarly, Q(3) and Q(4) are the coordinates of the normals from Mo(2) to planes 3 and 4.

Mo₂(CO)₄(H)[P(CH₃)₂] (Petersen, Dahl & Williams, 1974), and [HW₂(CO)₉NO] (Olsen, Koetzle, Kirtley, Andrews, Tipton & Bau, 1974).

The pentafulvalene ligand in this and related complexes behaves as two five-electron donors. The $M \cdots M$ separation, dihedral angle, and C—C separation between the C_5 rings follow the correlation suggested by Guggenberger & Tebbe (1976), *i.e.* the ligand folds to accommodate a Mo—Mo separation which is shorter than the optimum for a perfectly planar fulvalene (Fig. 2).

The cation has approximate C_s point symmetry, the mirror plane bisecting C(15)—C(35) and passing through H(1) and O(1) (Fig. 2). The planes, Table 3, containing the Mo atoms and the normals to the η^5-cyclopentadienyl rings fold towards each other about the Mo(1)—Mo(2) vector. The angle between the normals to these planes is 31·2°. Although the individual C_5 rings are planar, the whole fulvalene ligand is not

planar: the angle between planes C(11)—C(15) and C(31)—C(35) is 18·1°.

The OH group symmetrically bridges the two Mo atoms, as shown by the distances Mo(1)—O(1) 2·07 and Mo(2)—O(1) 2·09 Å. These are similar to values observed for Mo—O lengths in the bis-η^5-cyclopentadienylmolybdenum amino acid complexes, *e.g.* 2·10 Å in [(η⁵-C₅H₅)₂Mo(glycine)]Cl and [(η⁵-C₅H₅)₂-Mo(sarcosine)]Cl (Prout, Allison, Delbaere & Gore, 1972), and for terminal Mo—OH lengths in [(η⁵-C₅H₅)₂Mo(OH)NH₂CH₃]PF₆ (2·050 Å) (Prout, Cameron, Forder, Critchley, Denton & Rees, 1974), and [MoO(OH)(CN)₄]³⁻ (2·077 Å) (Robinson, Schlemper & Murmann, 1975). There are no OH-bridged Mo structures for comparison.

The observed data for di-μ-oxo complexes are Na₂Mo₂O₄(cysteine)₂ (1·93 Å) (Knox & Prout, 1969), Mo₂O₄(histidine)₂ (1·92 Å) (Delbaere & Prout, 1971) and Mo₂O₄(cysteine ethyl ester)₂ (1·93 Å) (Drew & Kay, 1971). In these complexes, the Mo—Mo lengths range from 2·552 to 2·569 Å, the Mo—O—Mo angle

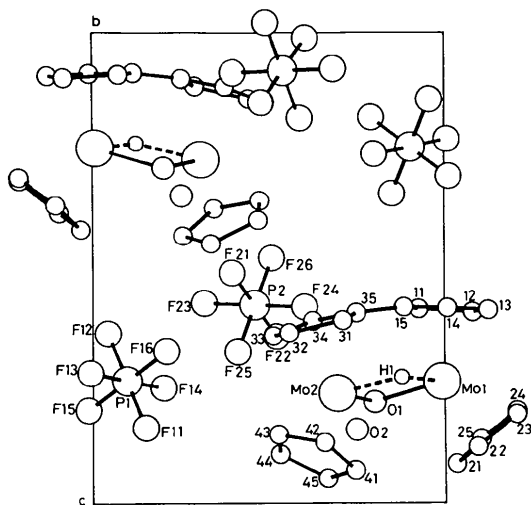Fig. 1. The crystal structure of μ-(η⁵:η⁵-fulvalene)-μ-hydrido-μ-hydroxyl-bis(η⁵-cyclopentadienylmolybdenum) hexafluorophosphate hemihydrate projected down a. For clarity, carbon atoms are denoted by their serial number only.
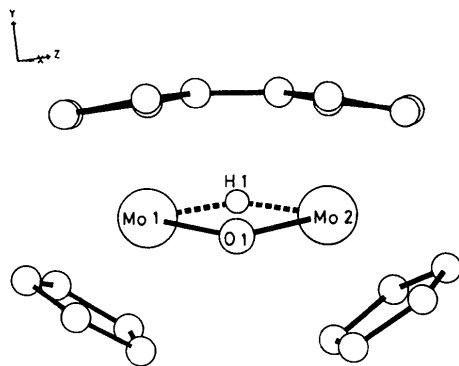

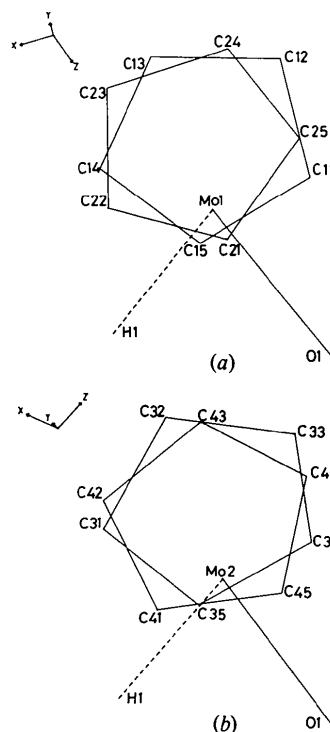Fig. 2. The molecular structure of [Mo₂(η⁵-C₅H₅)₂(μ-η⁵-C₅H₄-η⁵-C₅H₄)(μ-H)(μ-OH)]²⁺.

Fig. 3. The two halves of the cation projected down Z_R. [X_R, Y_R and Z_R have the following definition. In (a) the origin is taken as the Mo(1) atom, and $Q(1)$ and $Q(2)$ are the points where the normals at Mo(1) to the η⁵-C₅H₄ ring C(11)—C(15) and η⁵-C₅H₅ ring C(21)—C(25) pass through the best planes of those rings; then X_R is the normal to the plane containing $Q(1)$, $Q(2)$ and Mo(1), Y_R is the bisector of the angle $Q(1)$—Mo(1)—$Q(2)$, so that the positive direction forms an angle less than 90° with the bond Mo(1)—O(1), and Z_R is the perpendicular to the plane containing X_R and Y_R. Similarly for Mo(2) in (b).] This view corresponds very closely to the projection on the MX_2 plane used in many descriptions of bent metal bis-η⁵-cyclopentadienyls.

from 83·0 to 83·4°, and a metal—metal bond is suggested. In $(\eta^5\text{-}C_5H_5)_2Mo_2(H)(OH)(\text{fulvalene})^{2+}$ the Mo—Mo separation is 3·053 Å and the Mo—O—Mo angle is 94·3°. The $M\cdots M$ separation in multibridged systems is a poor criterion for assessing metal—metal bonding; however, an obtuse bridging angle is normally found in complexes with non-bonding metal—metal systems (Dahl, de Gil & Feltham, 1969), e.g. in $[(\eta^5\text{-}C_5H_5)Ti(OH)]_2(\text{fulvalene})$ (Ti$\cdots$Ti 3·195 Å, Ti—OH—Ti 98·8, 102·7°) (Guggenberger & Tebbe, 1976). The reduction of the bridging angle at the μ-OH from the more usual near-tetrahedral value is attributed to the formation of the Mo—H—Mo bond.

In the η^5-cyclopentadienyl rings the C—C bonds are in the range 1·31–1·50 Å [mean 1·44 (5) Å], while the C—C—C angles vary from 104 to 112° [mean 108 (2)°], with no systematic variation. These observations are consistent with other fulvalene ligand—metal structures where the C—C distances are considered to be equivalent (Churchill & Wormald, 1969; Guggenberger & Tebbe, 1973, 1976). The results for least-squares planes through the η^5-cyclopentadienyl rings are given in Table 3. The individual rings are planar within experimental error. In this structure the C_5 rings on both Mo atoms have a staggered conformation (Fig. 3); both eclipsed and staggered conformations are observed, and the choice of conformation has been attributed to crystal packing effects (Prout, Allison, Delbaere & Gore, 1972). The geometries of the two bis(η^5-cyclopentadienyl)molybdenum moieties are as commonly found in bent bis(η^5-cyclopentadienyl) systems (Prout, Cameron, Forder, Critchley, Denton & Rees, 1974). The Mo—C distances are in the range 2·24–2·39 Å [mean 2·32 (4) Å], the perpendicular distances from the Mo atoms to the rings vary from 1·962–1·980 Å [mean 1·971 (9) Å], and the perpendiculars to the η^5-cyclopentadienyl rings at the Mo atoms meet the rings close (0·09 Å mean) to their centroids making angles of 138·0° at Mo(1) and 135·3° at Mo(2).

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