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The Molecular and Crystal Structures of (η^7-Cycloheptatrienyl)molybdenum(II)-μ-bromo-μ-dihydroxo-(η^7-cycloheptatrienyl)molybdenum(II) Tetrafluoroborate Tetrahydrofuran Solvent

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The molecular and crystal structures of (η^7-cycloheptatrienyl)molybdenum(II)-μ-bromo-μ-dihydroxo-(η^7-cycloheptatrienyl)molybdenum(II) tetrafluoroborate as a tetrahydrofuran solvate have been determined by the analysis of three-dimensional X-ray diffraction data. Crystals are monoclinic, space group $P2_1/a$ with cell dimensions $a = 18.936(18)$, $b = 11.530(14)$, $c = 9.955(4)$ Å, $\beta = 90.06(6)°$ *and* $Z = 4$. *Intensity data were recorded at room temperature on a Syntex $P2_1$ autodiffractometer using graphite monochromated Mo K_α radiation. The structure was solved by Patterson and Fourier techniques, and refined by least-squares to a conventional R factor of 0.106. The crystal structure is built of equal numbers of $|(C_7H_7)Mo(Br)(OH)_2Mo(C_7H_7)|^+$ cations, BF_4^- anions and C_4H_8O solvent molecules. The cation features a metal–metal bond, 2.882(2) Å, and cycloheptatrienyl rings which, although individually planar, are bent symmetrically away from the bromide bridge to subtend a mutual dihedral angle of* ca. *17°. The tetrafluoroborate anion and tetrahydrofuran solvent molecule show no unusual geometrical features the latter adopting an envelope conformation with β-carbon apical. The role of these species in locating the hydroxy-H atoms of the cation through possible intermolecular hydrogen bonding is discussed.*

Introduction

Reaction of the cycloheptatrienyl tricarbonyl molybdenum cation $|Mo(CO)_3(\eta^7\text{-}C_7H_7)|^+$ with arenes affords the mixed sandwich molecules $|Mo(\eta^7\text{-}C_7H_7)(\eta^6\text{-arene})|^+$ [1] and such products are themselves convenient starting materials for a number of other η^7-cycloheptatrienylmolybdenum compounds [1, 2]. In one of a series of related experiments $|Mo(CO)_3(\eta^7\text{-}C_7H_7)|^+BF_4^-$ was reacted [3] with $C_8H_7SiMe_3$ in tetrahydrofuran (THF) to yield an emerald green crystalline ionic product of uncertain formulation, although recording elemental analyses of C 34.0 and H 3.82% and shown (^{1}H n.m.r.) to contain a cycloheptatrienyl fragment. In view of the ease and speed of present-day diffraction experiments the salt was accordingly subjected to a single-crystal X-ray analysis.

Experimental

Crystal Data

$C_{18}H_{24}BBrF_4Mo_2O_3$, $M = 646.96$, monoclinic, $a = 18.956(18)$, $b = 11.530(14)$, $c = 9.955(4)$ Å, $\beta = 90.06(6)°$, $U = 2173(3)$ Å^3, $D_m = 1.96$ (flotation in 1,2-dibromoethane and chloroform), $Z = 4$, $D_c = 1.977$, $F(000) = 1264$. Mo K_α radiation, $\lambda_{\alpha 1} = 0.70926$, $\lambda_{\alpha 2} = 0.71354$ Å, μ (Mo K_α) = 31.1 cm^{-1}. Preliminary Weissenberg and precession photographs showed systematic absences consistent with space group $P2_1/a$ (C_{2h}^5, No 14).

Data Collection and Reduction

A single crystal, *ca.* 0.05 × 0.02 × 0.006 cm was mounted on a quartz fibre and set on a Syntex $P2_1$ four-circle diffractometer. 15 reflections, $8 < 2\theta < 21°$, were selected from a rotation photograph and centred in 2θ, ω, and χ, and from the real-space vectors and intervector cosines generated by the autoindexing program the unit cell was chosen by inspection [4]. For data collection $0 \leqslant h \leqslant 25$, $0 \leqslant k \leqslant 17$, $\bar{15} \leqslant l \leqslant 15$ (with equivalent Okl and Ok$\bar{l}$ reflections afterwards merged), and $2.9 \leqslant 2\theta \leqslant 50.0°$ (graphite monochromated Mo K_α X-radiation). Three check reflections were monitored once every 33 but subsequent [5] analysis of their net intensities as individual functions of time indicated no crystal decomposition or machine variance over the *ca.* 135 hr. X-ray exposure. Reflections were scanned from 1.0° below $K_{\alpha 1}$ to 1.0° above $K_{\alpha 2}$ at rates varying between 0.03367 and 0.48333° s^{-1}.

Of the 3848 reflections scanned, 3284 were adjudged 'observed' ($I > 1.0\sigma$) and used to solve and refine the structure. Absorption ($1.25 < A^* < 1.84$), Lorentz, and polarisation corrections were applied.

Solution and Refinement

Analysis of a three-dimensional Patterson synthesis readily revealed the presence of a triangle of heavy atoms of approximately similar size, which were all initially described as Mo atoms. Examination of their post-refinement isotropic thermal parameters and internuclear separations then indicated a probable Mo_2X heterotriangle consistent with the location, *via* a subsequent electron density different synthesis, of only two η^7-C_7 rings, one to each molybdenum.

Thus the incorrectly assigned heavy atom occupies a bridging function across the Mo–Mo bond although the uncertainty surrounding its identity led to parameters of the $Mo_2(C_7)_2$ moiety only being refined. The resultant ΔF map showed the BF_4^- anion, the five-membered solvent ring (at this stage all five atoms were taken to be carbon) and three bridging atoms in the cation – two of approximately carbon weight, the third (the original heavy atom bridge) approximately equivalent in scattering power to five carbon atoms.

The trimethyl silylcyclo-octatetraene used in the reaction was prepared [3] from C_8H_7Br and Me_3SiCl. Thus it was possible that the heavy bridging function could be a bromine atom, and this subsequently proved to be borne out by a sensible U_j value under refinement. The light bridges could be feasibly be F or OH by analogy with established $|(\eta^7\text{-}C_7H_7)Mo(\mu\text{-}X)_3Mo(\eta^7\text{-}C_7H_7)|^+$ compounds [6] (X = halogen or OR) although the former would necessarily involve the unusual degradation of the tetrafluoroborate anion, whereas the latter simply requires the presence of moisture in either reactant or, more probably, in the THF. Even though the crystallographic analysis subsequently proved to be insufficiently accurate either to locate hydrogen atoms from ΔF maps or to refine them from theoretical starting positions, additional evidence for hydroxy- as opposed to fluoro-bridges derives from various intermolecular parameters and is presented later.

As the refinement converged, the position of the oxygen atom in the solvent molecule became apparent from inspection of the thermal parameters. Cycloheptatrienyl-H and tetrahydrofuran-H atoms were introduced into calculated positions (the former in the plane of the seven C atoms) with $r_{C-H} = 1.00$ Å, $U_H = 0.05$ Å^2. Although held invariant under refinement, hydrogen atom positional parameters were thereafter updated every four cycles.

F_o moduli were weighted according to $\omega = (xy)^{-1}$ with $x = b/\sin\theta$ if $\sin\theta < b$, $x = 1$ if $\sin\theta \geqslant b$, and $y = F_o/a$ if $F_o > a$, $y = 1$ if $F_o \leqslant a$ which a and b took values of 70.0 and 0.23, respectively, and refinement continued until the mean shift:error of the 235 variables was <0.005. Final residuals are R 0.106, R_ω 0.129. The maximum residue revealed by an ultimate electron density difference map of 0.38 Å resolution was *ca.* 1.8 eÅ^{-3} near 0.16, 0.05, 0.04.

Atomic scattering factors for neutral atoms were taken from reference 7 (Mo, Br, F, B), reference 8 (O, C) and reference 9 (H), with appropriate correction for anomalous dispersion [10]. The final positional and thermal parameters obtained are listed in Table I. A comparison of $10|F_o|$ *vs.* $10F_c$ at the termination of refinement has been deposited with the Editor, together with H atom positional parameters.

The solution and refinement of this structure employed programs of the XRAY-72 system [11]. implemented on the University of London CDC 7600 computer.

Description and Discussion of the Structure

The analysis establishes the molecular formula as $|(\eta^7\text{-}C_7H_7)Mo(\mu\text{-}Br)(\mu\text{-}OH)_2Mo(\eta^7\text{-}C_7H_7)|^+BF_4^-\cdot C_4H_8O$. Calculated analyses of C 33.42 and H 3.74% agree well with those observed.

Neither ion nor the tetrahydrofuran solvent molecule have space-group imposed symmetry. Interatomic distances (uncorrected for thermal effects) are presented in Table II, interbond angles in Table III, and details of molecular planes appear in Table IV.

The $|(\eta^7\text{-}C_7H_7)Mo(\mu\text{-}Br)(\mu\text{-}OH)_2Mo(\eta^7\text{-}C_7H_7)|^+$ Cation

Figure 1 is a perspective view of the cation and demonstrates the atomic numbering scheme adopted (the hydrogen atoms of the cycloheptatrienyl rings carry the same number as the carbon to which they are bound).

Assuming that the cycloheptatrienyl ring acts as the 6e donor $C_7H_7^+$ and interacts with three vacant metal orbitals, the cation may formally be described as a 7-co-ordinate molybdenum(II) complex with each metal atom providing the cap of the other's distorted capped octahedral geometry.

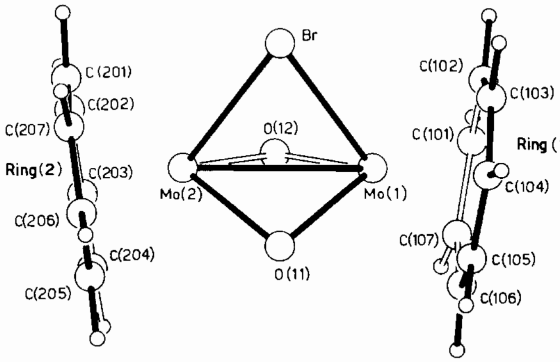


Figure 1. Perspective view of the cation $[(\eta^7\text{-}C_7H_7)Mo(\mu\text{-}Br)(\mu\text{-}OH)_2Mo(\eta^7\text{-}C_7H_7)]^+$.

Within the cycloheptatrienyl functions, the small ranges of (i) C–C distances around the rings, 1.33(4) to 1.46(4), mean 1.37(4) Å, and (ii) internal ring angles, 126.3(31) to 130.8(30), mean 128.6(14)°, allow the carbon sequences to be described as

TABLE I. Positional[a] and Thermal[b] Parameters.

Atom	x	y	z	U
Mo(1)	15247(8)[c]	4705(15)	49504(14)	*
Mo(2)	14642(9)	14765(16)	23027(14)	*
Br	8938(12)	24625(19)	44245(20)	*
O(11)	1022(8)	22(12)	3185(13)	*
O(12)	2277(6)	1165(13)	3672(12)	*
C(101)	2370(15)	268(26)	6528(29)	82(7)[d]
C(102)	1837(18)	937(29)	7076(32)	93(9)
C(103)	1125(17)	678(29)	7076(32)	92(8)
C(104)	788(16)	−240(28)	6553(32)	90(8)
C(105)	1037(16)	−1157(27)	5802(30)	86(8)
C(106)	1724(16)	−1410(26)	5501(29)	84(7)
C(107)	2310(16)	−790(27)	5748(30)	87(8)
C(201)	1426(14)	3174(24)	1214(27)	73(6)
C(202)	2081(16)	2784(27)	1068(31)	88(8)
C(203)	2298(17)	1689(29)	745(32)	91(8)
C(204)	1913(16)	745(26)	433(29)	84(8)
C(205)	1180(15)	658(26)	363(28)	80(7)
C(206)	668(16)	1460(28)	610(31)	88(8)
C(207)	763(18)	2572(31)	963(34)	98(9)
B	3846(18)	2272(31)	−1597(35)	81(8)
F(1)	3962(18)	2492(29)	−221(35)	190(11)
F(2)	4427(15)	2844(25)	−2063(28)	162(9)
F(3)	3225(12)	2888(20)	−1852(23)	130(7)
F(4)	3761(16)	1164(28)	−1568(31)	176(10)
O(1)	3478(9)	138(26)	3121(23)	*
C(2)	3660(21)	−664(35)	2164(40)	114(11)
C(3)	4402(23)	−489(38)	1877(43)	124(12)
C(4)	4698(22)	−29(38)	3045(32)	121(12)
C(5)	4122(22)	707(37)	3460(41)	120(12)

*These atoms refined anisotropically[e], producing:

Atom	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Mo(1)	440(9)	505(10)	303(8)	1(7)	−11(6)	64(7)
Mo(2)	490(9)	560(10)	277(7)	−12(8)	−22(6)	25(7)
Br	719(14)	576(13)	477(11)	99(11)	47(9)	−5(9)
O(11)	75(9)	55(8)	36(6)	0(7)	−4(6)	−8(6)
O(12)	38(7)	81(10)	31(6)	−6(6)	−1(5)	9(6)
O(1)	46(9)	198(25)	112(16)	38(13)	−6(10)	−67(17)

[a] Positional parameters are in fractional co-ordinates of the unit cell edges, $\times 10^5$ for Mo and Br atoms and $\times 10^4$ for other atoms. [b] Thermal parameters are in $Å^2$, $\times 10^4$ for Mo and Br atoms, $\times 10^3$ for other atoms. [c] Estimated standard deviations, shown in parentheses throughout this paper, are right-adjusted to the least significant digit in the preceding number. [d] The isotropic temperature factor is in the form $\exp\{-8\pi^2 U(\sin^2\vartheta)/\lambda^2\}$. [e] The anisotropic temperature factor is in the form $\exp\{-2\pi^2(U_{11}a^{*2}h^2 + U_{22}b^{*2}k^2 + U_{33}c^{*2}l^2 + 2U_{12}a^*b^*hk + 2U_{13}a^*c^*hl + 2U_{23}b^*c^*kl)\}$.

essentially regular heptagons, planar to within 0.021 Å {ring(1)} and 0.013 Å {ring (2)} . Molybdenum atoms (1) and (2) lie *ca.* 1.604 and 1.590 Å above their respective ring planes, and in projection are displaced only slightly (0.035 and 0.026 Å) from the centre. Metal–carbon distances show good internal consistency, range 2.21(3)–2.27(3), mean 2.25(4) Å, and, as might be expected, are progressively (though not significantly) shorter than the corresponding mean distance in typical $Mo^I(\eta^7\text{-}C_7H_7)$ [12. 13] and $Mo^0(\eta^7\text{-}C_7H_7)$ [14–16] complexes.

Except for the relative conformation of the cycloheptatrienyl rings (almost staggered about the Mo–Mo vector, although fluxional in solution [3]) the cation has effective C_{2v} symmetry about an axis from the centre of the metal–metal bond to the bromide bridge. The C_7H_7 rings do not lie parallel to this axis, however, each tipping away by about 8.5° in opposite directions.

This distortion is believed to be primarily the result of the inequivalence of the metal-to-bridge-atom distances. In a situation {Figure 2(i)} in whicb

TABLE II. Interatomic Distances (Å).

Bond Distances in Co-ordination Sphere

	Mo(1)–Mo(2)	2.882(2)	
Mo(1)–Br	2.641(4)	Mo(2)–Br	2.632(3)
Mo(1)–O(11)	2.064(13)	Mo(2)–O(11)	2.070(14)
Mo(1)–O(12)	2.082(13)	Mo(2)–O(12)	2.086(12)
Mo(1)–C(101)	2.25(3)	Mo(2)–C(201)	2.24(3)
Mo(1)–C(102)	2.26(3)	Mo(2)–C(202)	2.27(3)
Mo(1)–C(103)	2.26(3)	Mo(2)–C(203)	2.23(3)
Mo(1)–C(104)	2.27(3)	Mo(2)–C(204)	2.21(3)
Mo(1)–C(105)	2.26(3)	Mo(2)–C(205)	2.22(3)
Mo(1)–C(106)	2.27(3)	Mo(2)–C(206)	2.26(3)
Mo(1)–C(107)	2.22(3)	Mo(2)–C(207)	2.27(3)

Bond Distances around Cycloheptatrienyl Rings

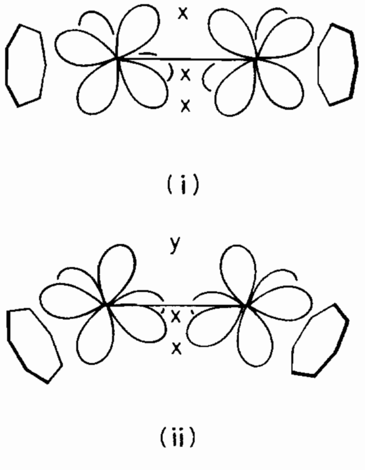
C(101)–C(102)	1.38(4)	C(201)–C(202)	1.33(4)
C(102)–C(103)	1.38(5)	C(202)–C(203)	1.37(5)
C(103)–C(104)	1.34(5)	C(203)–C(204)	1.35(4)
C(104)–C(105)	1.38(4)	C(204)–C(205)	1.39(4)
C(105)–C(106)	1.37(4)	C(205)–C(206)	1.36(4)
C(106)–C(107)	1.34(4)	C(206)–C(207)	1.34(5)
C(107)–C(101)	1.45(4)	C(207)–C(201)	1.46(4)

Bond Distances in Tetrafluoroborate Anion

B–F(1)	1.41(5)	B–F(3)	1.40(4)
B–F(2)	1.36(5)	B–F(4)	1.29(5)

Bond Distances in Tetrahydrofuran Molecule

O(1)–C(2)	1.37(5)	C(4)–C(5)	1.44(6)
C(2)–C(3)	1.45(6)	C(5)–O(1)	1.42(5)
C(3)–C(4)	1.39(6)		



(i)

(ii)

Figure 2. Representations of the sets of octahedrally disposed metal orbitals in (i) $M(\mu\text{-}X)_3M$ and (ii) $M(\mu\text{-}Y)(\mu\text{-}X)_2M$ species.

each metal atom uses three *fac*-orbitals of an octahedral set to bind three equivalent bridging functions, X, essentially D_{3h} molecular symmetry is preserved with no tipping of the non-bridging groups, even though X may be too small to support the ideal $X\hat{M}M$ angle of 69.3° and hence distort the facial lobes inwards. Such a situation has been independently investigated by Alcock [17] and Smart [18] using the symmetrical bridged cation $|(\eta^7\text{-}C_7H_7)Mo(\mu\text{-}Cl)_3Mo(\eta^7\text{-}C_7H_7)|^+$. In $M(\mu\text{-}Y)(\mu\text{-}X)_2M$ systems, however, an in-plane tilt of both metal orbital sets (and hence the *trans* non-bridging groups*) is required to maximise overlap with the unique group Y {Figure 2(ii)}.

The degree of tilt, θ, is approximately given by $(Y\hat{M}M - X\hat{M}M)$. For $|(\eta^7\text{-}C_7H_7)Mo(\mu\text{-}Br)(\mu\text{-}OH)_2Mo(\eta^7\text{-}C_7H_7)|^+$ θ(mean) calculates to +10.6°, the positive sign representing tipping of the C_7 rings away from the bromide bridge, and is in good agreement with that found, +8.5°.

Comparison with the tri(μ-chloro) cation is completed by examination of the sequence of (i) Mo–

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*Although the metal lobes *trans* to the bridging groups are necessarily eclipsed with respect to the metal–metal vector it does not, of course, follow that η-bonded functions such as cycloheptatrienyl rings must also be eclipsed.

TABLE III. Bond Angles (°).

In di-Molybdenum Cation			
Mo(1)–Br–Mo(2)	66.26(8)	Ring C(101)–C(107)	
Mo(1)–O(11)–Mo(2)	88.4(6)	C–Mo(1)–C	
Mo(1)–O(12)–Mo(2)	87.8(5)	Range 34.4(12)–37.8(11)	
Mo(1)–Mo(2)–Br	57.03(8)	Mean 35.8(13)[a]	
Mo(1)–Mo(2)–O(11)	45.7(4)		
Mo(1)–Mo(2)–O(12)	45.9(3)	C–C–C	
Mo(2)–Mo(1)–Br	56.71(7)	Range 126.3(31)–130.8(30)	
Mo(2)–Mo(1)–O(11)	45.9(4)	Mean 128.5(15)	
Mo(2)–Mo(1)–O(12)	46.3(3)		
		Ring C(201)–C(207)	
		C–Mo(2)–C	
		Range 34.3(10)–37.3(11)	
		Mean 35.6(12)	
		C–C–C	
		Range 126.9(30)–130.6(29)	
		Mean 128.6(13)	
In Tetrafluoroborate Anion			
F(1)–B–F(2)	96.8(28)	F(2)–B–F(3)	111.7(29)
F(1)–B–F(3)	102.4(28)	F(2)–B–F(4)	126.0(33)
F(1)–B–F(4)	100.1(30)	F(3)–B–F(4)	113.9(30)
In Tetrahydrofuran Molecule			
O(1)–C(2)–C(3)	106.7(32)	C(4)–C(5)–O(1)	108.0(34)
C(2)–C(3)–C(4)	106.1(36)	C(5)–O(1)–C(2)	105.0(27)
C(3)–C(4)–C(5)	99.4(35)		

[a] The estimated standard deviation of the mean is given by the expression $\sigma^2 = \{\Sigma_{i=1}^{N}(x_i - \bar{x})^2\}$ (N – 1) where x_i is the ith and $\bar{x}$ the mean of N similar types of parameter.

TABLE IV. Least-squares Planes Data.

Each plane is represented by the equation Px + Qy + Rz = S, Å where x, y and z are the atomic co-ordinates. Atomic deviations (Å) are expressed in square brackets.

Plane (1):	P	Q	R	S
C(101)–C(107)	2.057	–5.919	8.473	5.834

|C(101) +0.026, C(102) –0.015, C(103) –0.008, C(104) +0.023, C(105) –0.020, C(106) +0.016, C(107) –0.021, Mo(1) –1.604|

Plane (2):				
C(201) – C(207)	–0.352	–3.102	9.586	0.107

|C(201) +0.022, C(202) –0.020, C(203) +0.002, C(204) +0.009, C(205) –0.005, C(206) +0.001, C(207) –0.009, Mo(2) +1.590|

Plane (3):				
C(3)O(1)C(2)C(5)	–4.004	7.938	–6.904	–3.462

|C(3) +0.015, O(1) +0.024, C(2) –0.024, C(5) –0.016, C(4) –0.544|

Planes (4) and (5) are the three-atom sequences BrO(11)O(12) and C(3)C(4)C(5) respectively. Important dihedral angles are (degrees)

(1)–(2)	17.1	(3)–(5)	36.3
(1)–(4)	8.7		
(2)–(4)	8.5		

X lengths, 2.073, 2.461 [18] and 2.636 Å, and (ii) either Mo–X–Mo angles, 88.1, *ca.* 77 [18] and 66.3° or X–Mo–Mo angles, 46.0, *ca.* 51.5 [18] and 56.9°, as the bridge atom X varies from OH to Cl to Br, which suggests that the structural authority of a chloride bridge lies approximately midway between that of a hydroxy and a bromide bridge. Not unexpectedly, therefore, the Mo–Mo distance in the Mo(μ-Br)(μ-OH)$_2$Mo species is somewhat shorter than in its Mo(μ-Cl)$_3$Mo analogue, 2.882(2) *vs.* 3.076/3.060(2) [18] Å, although all three values lie within the accepted range [19, 20].

The Crystal Structure

The asymmetric unit is completed by a tetrafluoroborate counter-ion and a tetrahydrofuran solvent molecule. The labelling of these species is given in Figure 3, the projection of the unit cell contents as seen along the *c* crystallographic axis.

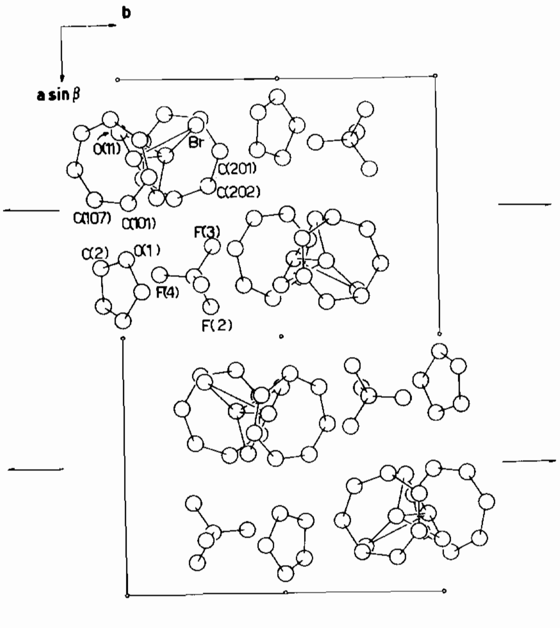

Figure 3. View of the crystal packing, seen along the *c* axis. Hydrogen atoms are not shown for the sake of clarity. Only the minimum symmetry elements necessary to describe the array are present.

The BF_4^- ion appears to be somewhat distorted towards a trigonal pyramidal geometry {F(1) apical} although the high degree of thermal activity of the fluorine atoms renders barely significant the bond length and angle variations. The THF molecule adopts an envelope conformation with an acute folding angle across C(3)...C(5) of *ca.* 36°. Although it, too, is comparatively poorly defined in that the accuracy in O–C and C–C distances is less than is desirable, we have used both these small molecules to predict, with some success, the location of the hydroxy-H atoms through intermolecular hydrogen bonding.

The two possible positions for each hydroxy-H atom are:

H(11)-bonded to O(11) at 0.95 Å with A–Ô–B = 109.47°.

A	0.1174	–0.0694	0.2809
B	0.0521	0.0033	0.3232

H(12)-bonded to O(12) at 0.95 Å with C–Ô–D = 109.47°

C	0.2503	0.1843	0.4002
D	0.2623	0.0628	0.3372

Evidence for A being the true location of H(11) begins with the recognition of an H...O bond between H(104′) at –x, –y, 1 – z and O(11), producing H(104′)–O(11)–Mo(1) and H(104′)–O(11)–Mo(2) angles of 114 and 109° respectively. If H(11) resides at position A the additional angle, H(104′)–O(11)–A, is also sensible (114°), whereas the alternative angle, H(104′)–O(11)–B is only 6° and H(104′)---B only *ca.* 1.5 Å. Thus H(104′) and H(11) at A complete an approximate tetrahedron about O(11) in which the two metal atoms are the additional vertices.

Interestingly H(11) at A is itself involved in two H---F contacts to F(2″) and F(3″) of the same BF_4 anion at ½ – x, y – ½, –z. H(11)---F(2″) is 2.16 Å subtending an angle H(11)–F(2″)–B″ of 94°, and H(11)---F(3″) is 2.21 Å with H(11)–F(3″)–B″ at 91°. Additionally the four atoms O(11), H(11), F(2″) and F(3″) are essentially co-planar. All these observations are mutually compatible and further evidence for the position of H(11).

The probable position of H(12) at C is arrived at indirectly. Neither O(12) nor H(12) at C is involved in hydrogen bonding. Position D, however, is unreasonably close (1.73 Å) to O(1) and lies effectively, and unacceptably, in the plane of O(1), C(2) and C(5).

We note finally that the atoms H(11) and H(12) thus positioned do not maintain the effective mirror plane through Mo(1), Mo(2) and Br.

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