

The N(1)—H(1) bond is aimed approximately halfway between O(2') and O(1') of adjacent molecules and forms a bifurcated hydrogen bond. The most important interlayer hydrogen bond involves O(5')—H(5') and carbonyl O(6) [O(5')—O(6) = 2.66 (2) Å].

The base-stacking pattern resembles those of 9-methyl- and 9-ethyladenine (Fig. 14*d,e*, Bugg, Thomas, Sundaralingam & Rao, 1971). Base stacking seems to be an important packing factor and it may have a determining effect in stabilizing this particular type of Hg—Cl chain, in which the pitch ($c/2 \sim 3.3$ Å) is equal to a normal interbase distance.

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Di- μ -chloro-bis[dichloro(1,1,1,5,5,5-hexafluoropentane-2,4-dionato)oxomolybdenum(V)]

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Abstract. $C_{10}H_2F_{12}O_6Cl_4Mo_2$, $M_r = 779.8$, monoclinic, $a = 5.972$ (5), $b = 12.733$ (8), $c = 14.704$ (10) Å, $\beta = 91.23$ (7)°, $U = 1117.9$ Å³, $Z = 2$, $d_c = 2.32$ g cm⁻³, Mo $K\alpha$ radiation, $\lambda = 0.7107$ Å, $\mu = 13.9$ cm⁻¹. Space group $P2_1/n$ from systematic absences $h0l$, $h + l = 2n + 1$ and $0k0$, $k = 2n + 1$. The centrosymmetric dimer contains a dichloro bridge [Mo—Cl 2.369 (4), 2.460 (4) Å]. The octahedral environment around each Mo is completed by a terminal O [Mo—O 1.648 (9) Å], a terminal Cl [Mo—Cl 2.305 (5) Å] and the bidentate hexafluoropentane-2,4-dionate ligand [Mo—O 2.07 (1), 2.17 (1) Å]. 1077 independent reflections measured on a diffractometer have been refined to an R of 0.062.

Introduction. Crystals of $Mo_2Cl_4O_2(hfac)_2$, $hfacH = 1,1,1,5,5,5$ -hexafluoropentane-2,4-dione, were prepared as follows. $hfacH$ (10 cm³) was distilled into $MoOCl_4$ (2 g) *in vacuo* and the resulting deep-red solution was filtered through the sintered disc of a double ampoule. Deep-red crystals formed over several months in the dark at 0°C. Excess $hfacH$ was removed and a suitable crystal was found for X-ray analysis. The crystal, 0.2 × 0.3 × 0.4 mm, was mounted with the a^* axis parallel to the instrument axis of a General Electric XRD-5 apparatus, which was used to measure diffraction intensities and cell dimensions. It was equipped with a manual goniostat, scintillation counter and pulse-height

Table 1. Positional parameters ($\times 10^4$) with estimated standard deviations in parentheses

	x	y	z
Mo	163 (2)	1179 (1)	172 (1)
Cl(1)	2648 (5)	-153 (3)	693 (2)
Cl(2)	2947 (7)	2351 (4)	582 (3)
O(1)	-1405 (15)	2437 (8)	-463 (6)
O(2)	1791 (15)	1063 (7)	-1127 (6)
O(3)	-1402 (15)	1198 (8)	1082 (6)
C(1)	-1131 (22)	2790 (11)	-1313 (9)
C(2)	104 (20)	2423 (11)	-1976 (8)
C(3)	1452 (21)	1579 (12)	-1863 (8)
C(4)	-2725 (35)	3737 (15)	-1354 (13)
C(5)	2796 (26)	1195 (14)	-2612 (10)
F(1)	-2604 (19)	4132 (9)	-2212 (8)
F(2)	-2135 (28)	4508 (11)	-852 (11)
F(3)	-4859 (19)	3521 (10)	-1241 (10)
F(4)	2887 (26)	186 (9)	-2680 (8)
F(5)	2255 (21)	1604 (12)	-3420 (6)
F(6)	4975 (18)	1424 (10)	-2531 (7)

discriminator. Zr-filtered Mo $K\alpha$ radiation was used. 1455 independent reflections were measured with $2\theta < 45^\circ$ and 1077 reflections with intensities $I > 2\sigma(I)$ were used in subsequent calculations. No absorption correction was made. The positions of the Mo atoms were obtained from a Patterson function and Fourier syntheses were used to locate the positions of the remaining atoms. The structure was refined by full-matrix least squares, with all atoms anisotropic, to an R of 0.062. The weighting scheme, chosen to give average values of wA^2 for groups of reflections independent of values of F_o and $\sin \theta/\lambda$, was $w^{1/2} = 1$ for $F_o < 30$ and $w^{1/2} = 30/F_o$ for $F_o > 30$. Calculations were made on a CDC computer at the University of London Computer Centre, with the XRAY system of programs (Stewart, 1972). Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974). The zero-weighted reflections showed no large discrepancies. In the final cycle of refinement all shifts were less than 0.02σ . The final atomic positions are given in Table 1, bond lengths and angles in Table 2.*

Discussion. We have investigated the reactions of $MOCl_4$ ($M = Mo, W$) with acacH (pentane-2,4-dione) and hfacH. Interaction of $WOCl_4$ with acacH led to the isolation of a salt, characterized by X-ray diffraction as 3-acetyl-2,4,6-trimethylpyrylium dichlorodioxo-(pentane-2,4-dionato)tungstate(VI), while interaction of $MoOCl_4$ with undiluted acacH led to the unexpected dimerization of the β -diketone to form 1,3,5,7-tetramethyl-2,4,6,8-tetraoxoadamantane (Drew, Fowles, Rice & Shanton, 1974; Fowles, Rice & Shanton, 1977).

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33002 (6 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

However, the addition of acacH to a chloroform solution of $MoOCl_4$ led to the isolation of the neutral adduct $MoOCl_3 \cdot acacH$ (Larson & Moore, 1966). On heating in benzene, this latter compound was converted into $MoOCl_2(acac)$. Reaction of $MoOCl_4$ with hfacH led directly to $MoOCl_2(hfac)$ whose structure is reported here.

The structure consists of a centrosymmetric dimer (Fig. 1) in which Mo atoms in distorted octahedral environments are bridged *via* two Cl atoms. A review of Mo^{IV} , Mo^V and Mo^{VI} structural chemistry (Spivack & Dori, 1975) shows that this type of arrangement is characteristic for compounds of the halides and oxyhalides of Mo^V . For example, $MoCl_3$, $MoOCl_3$ and $MoOBr_3$ all contain octahedral Mo bridged by edge-sharing of halogens to give dimeric or polymeric structures. Mo^V compounds which contain more than one O (or S) per Mo most often bridge through O (or S).

In the present dimer, the Mo_2Cl_2 group is perforce planar. The Mo-Cl-Mo angles are only $78.3(1)^\circ$, a value very much less than those in $MoOCl_3$ [$96.7(5)$, $103.2(6)^\circ$] (Drew & Tomkins, 1970), but even so it seems unlikely that there is any metal-metal bonding in the dimer [Mo...Moⁱ 3.049(1) Å]. The Mo-Cl_b distances [2.460(4), 2.369(4) Å] are comparable to values in $MoOCl_3$ [2.34(2), 2.44(2), 2.46(2), 2.78(2) Å, the latter being *trans* to terminal O]. There

Table 2. Molecular dimensions

Distances are in ångströms, angles in degrees. Superscripts (i) refers to the equivalent position $-x, -y, -z$.

Mo-Cl(1)	2.369 (4)	O(1)-C(1)	1.34 (2)
Mo-Cl(2)	2.305 (5)	C(1)-C(2)	1.32 (2)
Mo-Cl(1 ⁱ)	2.460 (4)	C(2)-C(3)	1.35 (2)
Mo-O(1)	2.068 (10)	C(3)-O(2)	1.28 (2)
Mo-O(2)	2.169 (9)	C(1)-C(4)	1.54 (2)
Mo-O(3)	1.648 (9)	C(3)-C(5)	1.46 (2)
Cl(1)-Mo-Cl(2)	86.3 (1)	C(4)-F(1)	1.36 (2)
Cl(1)-Mo-Cl(1 ⁱ)	101.7 (1)	C(4)-F(2)	1.27 (2)
Cl(1)-Mo-O(1)	167.0 (3)	C(4)-F(3)	1.32 (2)
Cl(1)-Mo-O(2)	86.9 (3)	C(5)-F(4)	1.29 (2)
Cl(1)-Mo-O(3)	96.4 (3)	C(5)-F(5)	1.33 (2)
Cl(2)-Mo-Cl(1 ⁱ)	163.6 (1)	C(5)-F(6)	1.34 (2)
Cl(2)-Mo-O(1)	86.2 (3)	Mo-O(1)-C(1)	128 (1)
Cl(2)-Mo-O(2)	86.5 (3)	O(1)-C(1)-C(2)	131 (1)
Cl(2)-Mo-O(3)	101.3 (4)	O(1)-C(1)-C(4)	102 (1)
Cl(1 ⁱ)-Mo-O(1)	83.1 (3)	C(2)-C(1)-C(4)	127 (1)
Cl(1 ⁱ)-Mo-O(2)	79.7 (3)	C(1)-C(2)-C(3)	122 (1)
Cl(1 ⁱ)-Mo-O(3)	92.0 (3)	C(2)-C(3)-O(2)	127 (1)
O(1)-Mo-O(2)	82.0 (3)	C(2)-C(3)-C(5)	121 (1)
O(1)-Mo-O(3)	95.4 (4)	O(2)-C(3)-C(5)	113 (1)
O(2)-Mo-O(3)	171.6 (4)		
Mo Cl(1)-Mo ⁱ	78.3 (1)		
C(1)-C(4)-F(1)	106 (1)	C(3)-C(5)-F(4)	115 (1)
C(1)-C(4)-F(2)	115 (2)	C(3)-C(5)-F(5)	115 (1)
C(1)-C(4)-F(3)	116 (1)	C(3)-C(5)-F(6)	114 (1)
F(1)-C(4)-F(2)	103 (2)	F(4)-C(5)-F(5)	109 (1)
F(1)-C(4)-F(3)	105 (2)	F(4)-C(5)-F(6)	100 (1)
F(2)-C(4)-F(3)	110 (2)	F(5)-C(5)-F(6)	102 (1)

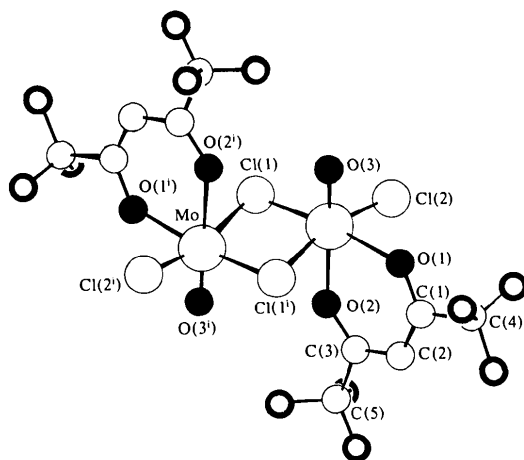


Fig. 1. The structure of Mo₂Cl₄O₂(hfac)₂.

seems no good reason for the range of Mo—Cl_b distances though perhaps there is some tendency towards five-coordination in the dimer.

The terminal Mo—O and Mo—Cl bond lengths [1.648 (9); 2.305 (5) Å] are comparable to values in MoOCl₃·SPPPh₃ [1.647 (3); 2.305 (1)–2.342 (1) Å] (Boorman, Garner, Mably & King, 1974) and in MoOCl₃L₂, L = PO(NMe₂)₃ [1.669 (5); 2.368 (2)–2.381 (2) Å] (Khodadad, Viossat & Rodier, 1977). The two bonds to hfac are significantly different [2.07 (1), 2.17 (1) Å], the longer bond being *trans* to terminal O. This difference of 0.10 Å is a common feature of molecules containing terminal O, for example 0.099 Å in MoOCl₃L₂, 0.11 Å in ReOCl₂(acac)(PPh₃) (Lock & Wan, 1975).

Least-squares-planes calculations for the dimer show

that the six-membered ring is almost planar, the maximum deviation of a contributing atom being 0.05 Å. The metal atom is 0.34 Å above the plane of Cl(1), Cl(1'), Cl(2) and O(1) towards the terminal O(3). The dimensions of the hfac ligand are as expected, although the F atoms are undergoing severe thermal motion.

No intermolecular distance is shorter than the sum of the van der Waals radii; the shortest being an O...F contact of 3.0 Å.

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Bis(dimethylphenylphosphine)trichlorostannioiodine

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Abstract. [P(C₆H₅)(CH₃)₂]₂AuSnCl₃, monoclinic, *P*2₁/*m*, *a* = 11.954 (2), *b* = 9.925 (2), *c* = 10.059 (1) Å, β = 111.40 (2)°, *Z* = 2, *D*_o = 2.05, *D*_c = 2.08 g cm⁻³. The Au atom is coordinated by the two phosphine ligands [Au—P, 2.310 (4), 2.318 (4) Å] and the SnCl₃ ligand [Au—Sn, 2.881 (1) Å], in a trigonal geometry distorted considerably towards linear

P—Au—P and an uncoordinated SnCl₃⁻ anion [P—Au—P, 153.8 (2)°].

Introduction. Initial cell dimensions were determined from precession photographs (Mo *K*_α radiation). Systematic absences (0*k*0 for *k* odd) indicated space group *P*2₁/*m* or *P*2₁; the centrosymmetric *P*2₁/*m*