Table 3. *Cavity volume* (Å^3) *of crystals of mode* I

Crystal	Volume
S-cn-dmp (298K)	17·97
S-cn-dmp (223K)	15·54
R-cn-*S*-mba (298K)	14·53
S-cn-*S*-mba (298K)	12·23
R-cn-pyrr (298K)	11·55

oxime, 2·370 (1) Å (Tomotake, Uchida, Ohashi, Sasada, Ohgo & Baba, 1984), and slightly shorter than those observed in [(*R*)-1-cyanoethyl](tributylphosphine)cobaloxime, 2·316 Å (Kurihara, Uchida, Ohashi, Sasada, Ohgo & Baba, 1983) and [(*R*)-1-cyanoethyl](diethylphenylphosphine)cobaloxime, 2·313 (1) Å (Tomotake, Uchida, Ohashi, Sasada, Ohgo & Baba, 1984).

The crystal structure at 223 K is shown in Fig. 3. There is one molecule in the asymmetric unit and the reactive cyanoethyl group is isolated from the other cyanoethyl group. The racemization occurring in such a crystal packing is classified as mode I. There is no unusually short contact between the molecules.

In order to examine why the present crystal is so rapidly racemized by X-ray exposure, the cavity for the cyanoethyl group was calculated (Ohashi, Uchida, Sasada & Ohgo, 1983) and the volume compared with the corresponding volumes of other crystals of mode I in Table 3. Although for the present crystal, the cavity at 298 K is not exact since the structure is not the initial one but that racemized at the 60% level, its volume, 17·97 Å^3, is significantly greater than those of the other crystals. Since the volume at 223 K, 15·54 Å^3, is still greater than those of the other cavities, the present crystal may be racemized by X-ray exposure at 223 K. Experiments to obtain the rate constants at various temperatures are in progress.

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Structure of (η^5-$C_5H_4CH_2CH_2OH)_2Mo_2(CO)_6$, Hexacarbonylbis-[$\eta^5$-1-(2-hydroxyethyl)cyclopentadienyl]dimolybdenum

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Abstract. $C_{20}H_{18}Mo_2O_8$, M_r = 578·2, orthorhombic, $P2_12_12_1$, a = 13·874 (2), b = 16·870 (4), c = 9·131 (2) Å, V = 2137 (1) Å^3, Z = 4, D_x = 1·80 Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, μ = 1·19 mm^{-1}, F(000) = 1144, T = 294 K, R = 0·036 for 1915 independent data [$I \geq 3\sigma(I)$]. Each Mo atom exhibits 'four-legged piano stool' bond geometry, with the other Mo atom at a basal position (Mo—Mo 3·211 Å). The molecule is close to being centrosymmetric if the orientations of the terminal

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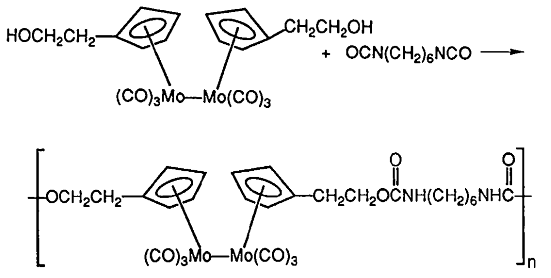
OH groups are neglected. Hydrogen bonds [O···O 2·69, 2·72 (1) Å] link the molecules into a three-dimensional network.

Introduction. In a recent paper (Tenhaeff & Tyler, 1990), we reported on the synthesis of several polyurethanes containing $Cp(CO)_3Mo–Mo(CO)_3Cp$ units along the backbone ($Cp = \eta^5\text{-}C_5H_4R$). A sample synthesis is shown below.



Metal–metal bonds can be photochemically cleaved (Meyer & Caspar, 1985), so the presence of the Mo–Mo unit makes these polymers photochemically reactive. As indicated, the key reagent in the synthesis of these polymers is the organometallic 'diol' $(\eta^5\text{-}C_5H_4\text{-}CH_2CH_2OH)_2Mo_2(CO)_6$. In this communication we report the structure of this complex.

Experimental. The compound was prepared as described elsewhere (Tenhaeff & Tyler, 1990). Suitable crystals for X-ray work were grown from a tetrahydrofuran–*n*-hexane solution.

Red block, dimensions 0·15 × 0·17 × 0·20 mm, mounted on a fiber and lightly coated with diluted polystyrene cement; Rigaku AFC6*R* diffractometer; initial cell parameters from 20 reflections in range $10·9 \leq 2\theta \leq 13·5°$, improved values from 25 strong reflections identified from rapid scan of shell $27 \leq 2\theta \leq 30°$; ω–2θ scans, scan speed 16° min^{-1} (on ω), scan width $(1·37 + 0·30\tan\theta)°$; range $2 \leq 2\theta \leq 50°$, $0 \leq h \leq 16$, $0 \leq k \leq 20$, $0 \leq l \leq 10$; three standard reflections every 200, no change; 2183 independent reflections scanned, 1915 with $I \geq 3\sigma(I)$ used in refinement.

MITHRIL E-map (Gilmore, 1984) showed Mo atoms at positions consistent with Patterson map; remaining non-H atoms from single cycle of *DIRDIF* (Beurskens *et al.*, 1984); *DIFABS* absorption correction (Walker & Stuart, 1983) after isotropic refinement (min., max. correction factors 0·87, 1·27); full-matrix least-squares refinement (on *F*), function minimized $\sum w(|F_o| - |F_c|)^2$, $w = 1/\sigma^2(F)$, $\sigma(F)$ from counting statistics, $p = 0·03$; non-H atoms anisotropic, H atoms (except OH) at calculated positions; convergence at R 0·036, wR 0·043, S 1·56, 271 parameters; max. Δ/σ 0·04 in last cycle; max., min. $\Delta\rho$ 0·55, $-0·53$ e $Å^{-3}$ in final difference map. The hydroxy H atoms were not directly located. They were omitted from the model, as more than one position for each atom was consistent with standard bond geometry. The enantiomer of the almost centrosymmetric molecule was also refined *ab initio*, with no significant change in the residuals or standard deviations of the atomic parameters. All programs were contained in the *TEXSAN* suite (Molecular Structure Corporation, 1989) and were run on a MicroVAX Series II/*RC* computer. Scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV, pp. 71, 148).

Table 1. *Atomic coordinates* ($\times 10^4$; Mo $\times 10^5$) *and isotropic equivalent thermal parameters* ($Å^2$)

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

	x	*y*	*z*	B_{eq}
Mo(1)	61460 (5)	60820 (4)	49957 (7)	2·55 (3)
Mo(2)	61990 (5)	73337 (4)	76437 (7)	2·65 (3)
O(1)	7487 (5)	6071 (5)	2249 (8)	6·0 (4)
O(2)	5897 (7)	7750 (4)	3632 (8)	6·3 (4)
O(3)	8199 (5)	5954 (4)	6343 (8)	5·1 (4)
O(4)	3328 (5)	4840 (5)	1740 (7)	5·0 (4)
O(5)	4903 (5)	7466 (6)	10398 (9)	7·1 (5)
O(6)	6427 (6)	5642 (4)	8982 (8)	6·1 (4)
O(7)	4151 (4)	7515 (5)	6283 (9)	5·8 (4)
O(8)	7715 (5)	9528 (4)	10842 (7)	4·6 (3)
C(1)	7008 (7)	6080 (6)	3282 (12)	4·3 (5)
C(2)	6001 (7)	7151 (5)	4220 (9)	3·7 (4)
C(3)	7424 (7)	6048 (6)	5892 (10)	3·6 (4)
C(4)	5721 (6)	4796 (5)	4400 (10)	3·3 (4)
C(5)	5076 (6)	5320 (5)	3656 (9)	2·9 (4)
C(6)	4518 (6)	5714 (5)	4726 (10)	3·3 (4)
C(7)	4810 (6)	5446 (6)	6122 (10)	3·5 (4)
C(8)	5561 (8)	4887 (6)	5939 (10)	4·0 (4)
C(9)	4955 (7)	5369 (6)	2029 (9)	4·1 (4)
C(10)	4318 (7)	4727 (6)	1415 (11)	4·4 (5)
C(11)	5374 (8)	7406 (7)	9345 (13)	5·2 (5)
C(12)	6317 (7)	6250 (5)	8400 (9)	3·8 (4)
C(13)	4925 (6)	7408 (6)	6732 (10)	3·9 (4)
C(14)	6680 (7)	8629 (5)	8113 (11)	3·9 (4)
C(15)	7276 (7)	8120 (5)	8976 (9)	3·1 (4)
C(16)	7827 (6)	7658 (5)	7987 (20)	3·5 (4)
C(17)	7588 (7)	7891 (6)	6560 (10)	4·0 (4)
C(18)	6882 (8)	8479 (6)	6618 (10)	4·1 (5)
C(19)	7342 (8)	8108 (7)	10605 (10)	4·3 (5)
C(20)	8021 (7)	8753 (6)	11166 (11)	4·3 (5)

Discussion. Atomic coordinates and equivalent isotropic thermal parameters are given in Table 1, and bond lengths and selected bond angles are given in Table 2.* A view of the molecule showing the numbering of atoms is shown in Fig. 1.

The $(\eta^5\text{-}C_5H_4\text{-}CH_2CH_2OH)_2Mo_2(CO)_6$ molecule lies on a general position, but its non-crystallographic symmetry is close to centrosymmetric. Corre-

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* Lists of structure factors, anisotropic thermal parameters, mean planes, full bond angles, torsion angles and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53441 (28 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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

The centroids of the cyclopentadienyl rings are denoted by Cp(1) and Cp(2).

Mo(1)—C(1)	1·97 (1)	Mo(2)—C(11)	1·93 (1)
Mo(1)—C(2)	1·948 (9)	Mo(2)—C(12)	1·96 (1)
Mo(1)—C(3)	1·95 (1)	Mo(2)—C(13)	1·96 (1)
Mo(1)—C(4)	2·313 (9)	Mo(2)—C(14)	2·324 (9)
Mo(1)—C(5)	2·314 (8)	Mo(2)—C(15)	2·340 (8)
Mo(1)—C(6)	2·356 (8)	Mo(2)—C(16)	2·345 (8)
Mo(1)—C(7)	2·376 (9)	Mo(2)—C(17)	2·362 (9)
Mo(1)—C(8)	2·34 (1)	Mo(2)—C(18)	2·35 (1)
O(1)—C(1)	1·15 (1)	O(5)—C(11)	1·17 (1)
O(2)—C(2)	1·15 (1)	O(6)—C(12)	1·17 (1)
O(3)—C(3)	1·16 (1)	O(7)—C(13)	1·16 (1)
O(4)—C(10)	1·42 (1)	O(8)—C(20)	1·41 (1)
C(4)—C(5)	1·43 (1)	C(14)—C(15)	1·43 (1)
C(4)—C(8)	1·43 (1)	C(14)—C(18)	1.42 (1)
C(5)—C(6)	1·41 (1)	C(15)—C(16)	1·42 (1)
C(5)—C(9)	1·50 (1)	C(15)—C(19)	1·49 (1)
C(6)—C(7)	1·41 (1)	C(16)—C(17)	1·40 (1)
C(7)—C(8)	1·41 (1)	C(17)—C(18)	1·39 (1)
C(9)—C(10)	1·51 (1)	C(19)—C(20)	1·53 (1)
Mo(1)—Cp(1)	2·003 (8)	Mo(2)—Cp(2)	2·013 (8)
Mo(1)—Mo(2)	3·211 (1)		
Cp(1)—Mo(1)—Mo(2)	119·7 (2)	Cp(2)—Mo(2)—Mo(1)	118·2 (2)
Cp(1)—Mo(1)—C(1)	113·7 (4)	Cp(2)—Mo(2)—C(11)	112·6 (4)
Cp(1)—Mo(1)—C(2)	125·8 (4)	Cp(2)—Mo(2)—C(12)	125·4 (4)
Cp(1)—Mo(1)—C(3)	128·2 (4)	Cp(2)—Mo(2)—C(13)	127·8 (4)
Mo(2)—Mo(1)—C(1)	125·8 (3)	Mo(1)—Mo(2)—C(11)	129·3 (3)
Mo(2)—Mo(1)—C(2)	88·1 (3)	Mo(1)—Mo(2)—C(12)	84·9 (3)
Mo(2)—Mo(1)—C(3)	71·6 (3)	Mo(3)—Mo(2)—C(13)	78·7 (3)
C(1)—Mo(1)—C(2)	77·0 (4)	C(11)—Mo(2)—C(12)	79·9 (4)
C(1)—Mo(1)—C(3)	71·6 (4)	C(11)—Mo(2)—C(13)	72·6 (4)
C(2)—Mo(1)—C(3)	105·8 (4)	C(12)—Mo(2)—C(13)	106·6 (4)
Hydrogen bonds			
O(4)···O(8)^{i}	2·69 (1)	O(4)···O(8)^{ii}	2·72 (1)

Symmetry codes: (i) $1 - x$, $-0·5 + y$, $0·5 - z$; (ii) $-0·5 + x$, $1·5 - y$, $1 - z$.

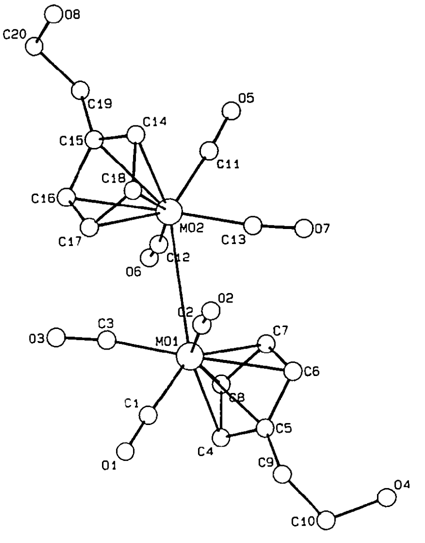

Fig. 1. The structure of the $(\eta^5\text{-}C_5H_4\text{-}CH_2CH_2OH)_2Mo_2(CO)_6$ complex showing the atom-numbering scheme.

sponding bond lengths and angles in the two halves of the molecule are of similar magnitude, and corresponding torsion angles are of similar magnitude and opposite sign. The structure is overall very similar to that of the accurately centrosymmetric 3-hydroxypropyl analog (Coolbaugh, Coots, Santarsiero & Grubbs, 1985) and also similar to that of the parent complex $Cp_2Mo_2(CO)_6$ (Wilson & Shoemaker, 1957). For comparison, the metal–metal bond lengths in these complexes are 3·213 (8) (3-hydroxypropyl analog), 3·222 (5) (parent complex), and 3·211 (1) Å (2-hydroxyethyl complex).

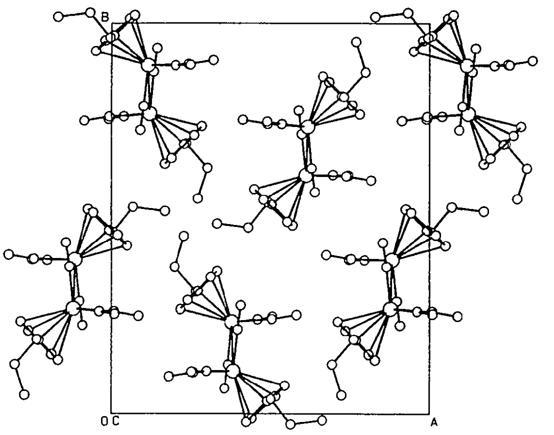

Fig. 2. A packing diagram of the $(\eta^5\text{-}C_5H_4\text{-}CH_2CH_2OH)_2\text{-}Mo_2(CO)_6$ complex.

The chief departure from local centrosymmetry involves the torsion angles about the CH_2—CH_2 bonds [C(5)—C(9)—C(10)—O(4), +73°; C(15)—C(19)—C(20)—O(8), +63°]; these angles favor a packing mode in which the molecules are linked into a three-dimensional network *via* intermolecular hydrogen bonds between the OH groups. A packing diagram is shown in Fig. 2.

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