this nomenclature the present compound, although having an effective coordination of seven, would have a characteristic coordination of three by virtue of three primarily covalently bonded N atoms. Examples of characteristic three coordination are uncommon; the present cation is the only recorded example of a complex with three monodentate ligands. In the case of the bidentate complex tris(1,8-naphthyridine)-mercury(II) bis(perchlorate) (Epstein, Dewan, Kepert & White, 1974) each naphthyridine forms one short and one long bond to Hg, giving a planar configuration of the three shorter bonds similar to that in the title compound. Planar three coordination is also present in (2,2′-bipyridyl)methylmercury (Carty & Gatehouse, 1976), arising from the unsymmetrical bidentate character of the ligand.

The CCOO groups of the trifluoroacetate ions are planar as expected; O(1) and O(2) of one of these ions are coplanar with Hg and N(3) whilst those of the other are rotated 15° relative to this plane. Both trifluoroacetate ions have high U values for the F atoms, suggesting disorder. All the bonds from the anions are to the same Hg complex cation. There is no indication of any bonding between neighbouring structural units.

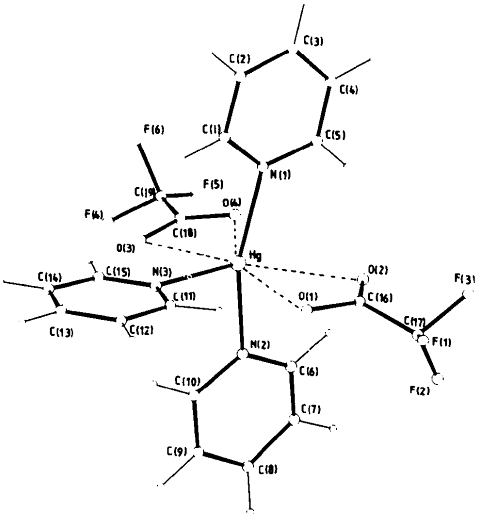

Fig. 1. A unit of the structure of tris(pyridine)mercury(II) bis(trifluoroacetate) showing the coordination around Hg.

We thank the Science Research Council for the provision of a research studentship to one of us (JH).

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μ-(1–4-η: 11–14-η-1,3,11,13-Tetradecatetraene-5,10-diol)-bis(tricarbonyliron)

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Abstract. $[(CO)_3Fe(C_4H_5)CHOHC_2H_4]_2$, $C_{20}H_{22}Fe_2O_8$, monoclinic, $P2_1/c$, $a = 14{\cdot}321\,(12)$, $b = 6{\cdot}114\,(3)$, $c = 12{\cdot}565\,(10)$ Å, $\beta = 105{\cdot}20\,(5)°$ at −35 °C, $D_c(-35\,°C) = 1{\cdot}581$, $D_m(21\,°C) = 1{\cdot}57$ g cm^{-3}, $Z = 2$. The molecule is composed of a fully extended 1,3,11,13-tetradecatetraene-5,10-diol ligand π bonded with two $Fe(CO)_3$ fragments in the customary butadiene–iron tricarbonyl fashion. Full-matrix least-

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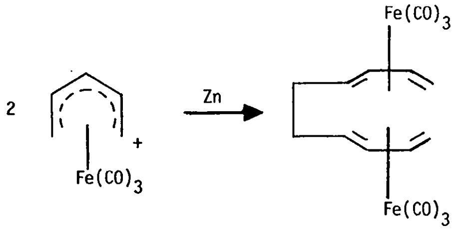
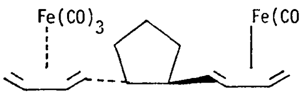
squares refinement using the 2248 reflections with $4 < 2\theta < 55°$ which have $I_o/\sigma(I_o) > 2{\cdot}0$ has converged with a conventional R index (on $|F|$) of 0·024.

Introduction. It has been previously demonstrated (Mahler, Gibson & Pettit, 1963) that pentadienyliron tricarbonyl cations upon treatment with zinc couple, as shown, to form a new C—C bond.



More recently, the extension (Sapienza, Riley, Davis & Pettit, 1976) of this procedure has provided a method for the preparation of metal-complexed medium-sized cycloalkanes; specifically, compounds containing five-, six- or ten-membered rings. Removal of the $Fe(CO)_3$ fragments by mild oxidative addition then affords the uncomplexed cyclic hydrocarbon.

The crystal structure determination of the only observed isomer of the five-membered-ring complex (I) (Sapienza *et al.*, 1976) established that the ring-closure reaction led only to *trans* stereochemistry, *i.e.* that the butadiene–$Fe(CO)_3$ groups are in a *trans* relationship across the ring. It was known that the dialcohols of the eventual five- and six-membered-ring compounds undergo an identical stereoselective ring-closure reaction (Sapienza, Cornell & Pettit, 1977). Speculation (Sapienza *et al.*, 1976) as to the other stereoselective aspect of this reaction sequence, *viz* the stage at which the relative disposition of the $Fe(CO)_3$ units on the butadiene ligands is determined, led to a desire to know the stereochemistry of the only isolated dialcohol–$Fe(CO)_3$ precursor. We have now determined the crystal structure of one of these dialcohol–$Fe(CO)_3$ species: that leading to the cyclohexyl ring system.



(I)

Single crystals of the title compound formed as yellow blocky prisms from a 2-propanol solution at room temperature. Preliminary X-ray diffraction experiments conducted with a Syntex $P2_1$ diffractometer indicated the symmetry and systematic reflection absences of space group $P2_1/c$ (No. 14). Crystal-data and X-ray diffraction data collection details are given in Table 1. Processing of the intensity data (with p = 0·02) was carried out as previously described (Riley & Davis, 1976).

Table 1. *Experimental summary for* [(CO)$_3$Fe(C$_4$H$_5$)CHOHC$_2$H$_4$]$_2$

Syntex $P2_1$ autodiffractometer equipped with graphite monochromator and Syntex LT-1 low-temperature flow system.
Radiation: Mo $K\alpha$, $\lambda = 0{\cdot}71069$ Å.
Mode: ω-scan technique, recentered automatically after each batch of 1000 reflections.
Scan range: symmetrically over 1·5° about the $K\alpha_{1,2}$ maximum.
Scan rate: variable, 2·0 to 5·0° min^{-1}.
Background: offset 1·0 and −1·0° in ω from $K\alpha_{1,2}$ maximum.
Check reflections: four remeasured after every 96 reflections; analysis* of 30 sets of check reflections indicated random fluctuation not exceeding 1·4% or 0·3 of an e.s.d. of the initial intensity set.
2432 reflections measured; $4° \leq 2\theta \leq 55°$.
Data-crystal faces: four faces of {100}, {$\bar{1}$02}, two broken faces.
Data-crystal dimensions: 0·36 × 0·53 × 0·88 mm.
Data-crystal volume: 0·166 mm^3.
Absorption coefficient: μ(Mo $K\alpha$) = 14·4 cm^{-1}.
Transmission-factor range: 0·47 to 0·61.

* Henslee & Davis (1975).

The structure was solved by standard heavy-atom procedures and refined by full-matrix least-squares methods using the 2248 data with $I_o/\sigma(I_o) > 2{\cdot}0$. In the final cycles of refinement the nonhydrogen atoms were treated anisotropically, and the H atoms isotropically. At convergence, $R = \sum||F_o| - |F_c||/\sum|F_o| = 0{\cdot}024$, $R_w = [\sum w(|F_o| - |F_c|)^2/\sum w|F_o|^2]^{1/2} = 0{\cdot}033$ and the standard deviation of an observation of unit weight, $[\sum w(||F_o| - |F_c||)^2/(m-s)]^{1/2}$, is 2·42 for m = 2248 observations ($|F_o|$) and s = 180 variables. The function minimized in refinement is $\sum w(|F_o| - |F_c|)^2$, where the weight w is $\sigma(|F_o|)^{-2}$, the reciprocal square of the standard deviation of each observation $|F_o|$. Neutral-atom scattering factors for Fe, O, C (*International Tables for X-ray Crystallography*, 1974) and H (Stewart, Davidson & Simpson, 1965) were used in these calculations. Corrections for the real (f') and imaginary (f'') parts of anomalous scattering were applied to the Fe scattering curve (*International Tables for X-ray Crystallography*, 1974). A structure factor calculation using all 2430 reflections measured during data collection gave R and R_w values of 0·026 and 0·033 respectively.* Examination of the data showed no indication of secondary extinction.

The largest shifts in position in the final cycle of refinement were 0·20 and 0·70 of a corresponding

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* Lists of structure factors and anisotropic thermal parameters, and a tabulation of atomic displacements from the plane of the butadiene residue have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33849 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

e.s.d. for nonhydrogen and H atoms respectively. The largest peak of a final difference electron density map was 0·3 e Å^{-3}; from a previous map the H atoms of this structure exhibited peaks of 0·4–0·8 e Å^{-3}.

Final atomic positional parameters with e.s.d.'s, as obtained from the least-squares inverse matrix, are presented in Table 2.

Discussion. A view of the molecular structure of the dialcohol precursor of the cyclohexyl compound is shown in Fig. 1. The crystal structure consists of discrete molecules of $[(CO)_3Fe(C_4H_5)CHOHC_2H_4]_2$. There are no unusual intermolecular contacts and, quite surprisingly, there is no hydrogen bonding between hydroxyl groups – a feature which is commonly observed in crystal structures of this kind. The molecules possess rigorous C_i symmetry; each Fe atom is coordinated with three CO ligands and the four C atoms of a butadiene residue in the usual manner. The hydrocarbon chain of the ligand is fully extended (see Fig. 1). The bond lengths and bond angles (Tables 3 and 4), which are particularly well determined, agree with values found in other related structures (Riley & Davis, 1976).

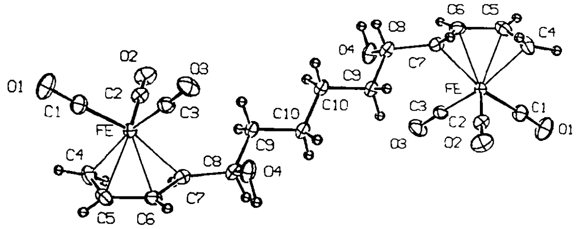

Fig. 1. A view of a single molecule of $[(CO)_3Fe(C_4H_5)CHOHC_2H_4]_2$, illustrating the atom-numbering scheme. Nonhydrogen atoms are shown as ellipsoids of 30% probability, and H atoms as spheres of radius 0·1 Å.

Table 2. *Fractional coordinates and, for* H, *isotropic thermal parameters*

See Fig. 1 for identity of the atoms. Numbers in parentheses are the estimated standard deviations in the units of the last significant digits for the corresponding parameter. H atoms are numbered in accordance with the C or O atoms to which they are bonded.

	x	y	z
Fe	0·19497 (1)	0·22025 (4)	0·13256 (2)
O(1)	0·0690 (1)	0·0650 (3)	−0·0760 (1)
O(2)	0·1398 (1)	−0·0535 (2)	0·2977 (1)
O(3)	0·3504 (1)	−0·0200 (2)	0·0738 (1)
O(4)	0·4509 (1)	0·3319 (3)	0·2508 (1)
C(1)	0·1171 (1)	0·1278 (3)	0·0055 (1)
C(2)	0·1616 (1)	0·0475 (3)	0·2321 (1)
C(3)	0·2921 (1)	0·0763 (3)	0·0997 (1)
C(4)	0·0958 (1)	0·4645 (3)	0·1491 (2)
C(5)	0·1667 (1)	0·5456 (3)	0·1003 (1)
C(6)	0·2649 (1)	0·5174 (3)	0·1586 (1)
C(7)	0·2860 (1)	0·4102 (3)	0·2617 (1)
C(8)	0·3884 (1)	0·3514 (3)	0·3235 (1)
C(9)	0·3954 (1)	0·1398 (3)	0·3886 (1)
C(10)	0·4930 (1)	0·1119 (3)	0·4730 (1)

	x	y	z	U (Å^2)
H(O4)	0·481 (2)	0·468 (4)	0·252 (2)	0·072 (8)
H(4,1)	0·102 (1)	0·487 (3)	0·227 (1)	0·034 (4)
H(4,2)	0·035 (1)	0·459 (3)	0·104 (2)	0·055 (6)
H(5)	0·154 (1)	0·621 (3)	0·027 (2)	0·047 (5)
H(6)	0·317 (1)	0·548 (3)	0·124 (1)	0·034 (5)
H(7)	0·245 (1)	0·446 (3)	0·307 (1)	0·024 (4)
H(8)	0·408 (1)	0·467 (3)	0·374 (1)	0·027 (4)
H(9,1)	0·382 (1)	0·013 (3)	0·332 (1)	0·024 (4)
H(9,2)	0·348 (1)	0·148 (3)	0·425 (1)	0·034 (5)
H(10,1)	0·543 (1)	0·146 (3)	0·436 (2)	0·044 (5)
H(10,2)	0·499 (1)	0·229 (3)	0·526 (1)	0·024 (4)

Table 3. *Bond lengths* (Å) *for* $[(CO)_3Fe(C_4H_5)CHOHC_2H_4]_2$

See Fig. 1. for identity of the atoms. Numbers in parentheses are the estimated standard deviations in the least significant digits.

Fe–C(1)	1·783 (2)	C(8)–O(4)	1·443 (2)
Fe–C(2)	1·796 (2)	C(9)–C(10)	1·526 (2)
Fe–C(3)	1·784 (2)	C(10)–C(10)′*	1·518 (2)
Fe–C(4)	2·109 (2)	C(4)–H(4,1)	0·96 (2)
Fe–C(5)	2·050 (2)	C(4)–H(4,2)	0·90 (2)
Fe–C(6)	2·060 (2)	C(5)–H(5)	1·00 (2)
Fe–C(7)	2·139 (2)	C(6)–H(6)	0·98 (2)
C(1)–O(1)	1·142 (2)	C(7)–H(7)	0·94 (2)
C(2)–O(2)	1·138 (2)	C(8)–H(8)	0·94 (2)
C(3)–O(3)	1·138 (2)	C(9)–H(9,1)	1·03 (2)
C(4)–C(5)	1·407 (3)	C(9)–H(9,2)	0·91 (2)
C(5)–C(6)	1·417 (3)	C(10)–H(10,1)	0·97 (2)
C(6)–C(7)	1·413 (2)	C(10)–H(10,2)	0·97 (2)
C(7)–C(8)	1·512 (2)	O(4)–H(O4)	0·93 (3)
C(8)–C(9)	1·520 (2)		

* Coordinates of C(10)′ are related to those given in Table 2 for C(10) by the operation $1-x, -y, 1-z$.

Table 4. *Bond angles* (°) *for* $[(CO)_3Fe(C_4H_5)CHOHC_2H_4]_2$

See Fig. 1 for identity of the atoms. Numbers in parentheses are the estimated standard deviations in the least significant digits.

C(1)–Fe–C(2)	102·6 (1)	C(9)–C(10)–C(10)′*	113·7 (1)
C(1)–Fe–C(3)	88·8 (1)	O(4)–C(8)–C(9)	107·8 (1)
C(2)–Fe–C(3)	103·7 (1)	H(4,1)–C(4)–C(5)	120 (1)
Fe–C(1)–O(1)	178·3 (2)	H(4,1)–C(4)–H(4,2)	117 (2)
Fe–C(2)–O(2)	176·8 (2)	H(4,2)–C(4)–C(5)	115 (2)
Fe–C(3)–O(3)	176·2 (2)	C(4)–C(5)–H(5)	126 (1)
C(4)–C(5)–C(6)	117·7 (2)	H(5)–C(5)–C(6)	117 (1)
C(5)–C(6)–C(7)	118·3 (2)	C(5)–C(6)–H(6)	121 (1)
C(6)–C(7)–C(8)	121·8 (1)	H(6)–C(6)–C(7)	120 (1)
C(7)–C(8)–C(9)	113·4 (1)	C(6)–C(7)–H(7)	115 (1)
C(7)–C(8)–O(4)	*111·9 (1)*	*H(7)–C(7)–C(8)*	*115 (1)*
C(8)–C(9)–C(10)	112·8 (1)	C(8)–O(4)–H(O4)	106 (2)

* Coordinates of C(10)′ are related to those of C(10) by the operation $1-x, -y, 1-z$. Angles involving the H atoms of the tetrahedral C atoms are as follows: H–C–C 105 (1) to 112 (1)°; H–C–H 100 (1) to 111 (2)°.

As stated above, the isolation of only one cycloalkane-containing stereoisomer [*e.g.* $(CO)_3Fe(C_4H_5)CH(CH_2)_3CH(C_4H_5)Fe(CO)_3$] may be due to the formation of only one of two possible isomers of diketone (**1**) of the previously mentioned reaction scheme. Determination of the analogous *trans* stereochemistry for dialcohol (**3**) [which was studied rather than diketone (**1**) (or **2**) of the scheme given elsewhere (Sapienza *et al.*, 1976) because of ease of crystal growth] by single-crystal methods supports this suggestion.

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Lithium Hydrogen Phthalate Monohydrate

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Abstract. $LiHC_8O_4H_4.H_2O$, $C_8H_5LiO_4.H_2O$, triclinic, $P\bar{1}$, $a = 11{\cdot}932\ (2)$, $b = 8{\cdot}874\ (2)$, $c = 7{\cdot}943\ (2)$ Å, $\alpha = 97{\cdot}13\ (5)$, $\beta = 95{\cdot}88\ (5)$, $\gamma = 93{\cdot}12\ (5)°$, $Z = 4$, $D_x = 1{\cdot}524$ Mg m^{-3}. The structure has been solved by direct methods and refined to $R = 0{\cdot}032$ for 3446 independent reflexions. The Li ions are coordinated by distorted O tetrahedra. In the two symmetrically independent hydrogen *o*-phthalate ions very short intramolecular hydrogen bonds between the two adjacent carboxylic groups are formed with O—O distances 2·400 (2) and 2·390 (2) Å.

Introduction. Recent structure analyses of the acid lithium phthalates $LiHC_8O_4H_4.2H_2O$ (Gonschorek & Küppers, 1975) and $LiHC_8O_4H_4.CH_3OH$ (Adiwidjaja & Küppers, 1978) revealed intramolecular hydrogen bonds with extremely short O—O distances. The aim of the present investigation is to check whether similarly short hydrogen bonds occur in the monohydrate. In the course of growth experiments on large single crystals of orthorhombic lithium hydrogen phthalate dihydrate, a triclinic crystal species appeared by chance during one experiment which was found to be the monohydrate. The predominating faces of these crystals as grown from aqueous solution are {110}, {001}, and $\{1\bar{1}0\}$. Additionally, small faces of type {100} and $\{\bar{1}11\}$ occur. Perfect cleavage is observed parallel to (110). The optical character is negative and the acute bisectrix is nearly perpendicular to (110).

X-ray measurements were carried out by an automatic Philips PW 1100 four-circle diffractometer using Mo $K\alpha$ radiation. Lattice parameters were determined by a least-squares treatment of 48 reflexions. Using a specimen of approximately spherical shape with a mean diameter of 0·4 mm intensities of all 7884 reflexions in the range $2 < \theta < 28°$ were collected. 848 of these were not centred or had negative intensities. Averaging the Friedel pairs yielded 3629 unique reflexions with an internal consistency index $R = 0{\cdot}039$ (Sheldrick, 1976). No correction for absorption seemed necessary ($\mu = 0{\cdot}118$ mm^{-1}).

The approximate location of the C and O atoms of the phthalate molecules was found by direct methods using *MULTAN* (Germain, Main & Woolfson, 1971). Further calculations were carried out using the *SHELX* 76 system (Sheldrick, 1976). The approximate positions of the remaining atoms were determined using a difference Fourier map. Full-matrix least-squares refinement with anisotropic temperature factors for the non-hydrogen atoms resulted in a weighted R value of 0·032 (non-weighted $R = 0{\cdot}039$). Squared reciprocal standard deviations were used as weights. Atomic scattering factors including correction for anomalous dispersion were taken from *International Tables for X-*