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The Structure of the Ether Solvate of *cis*-μ-[Benzyl(phenyl)vinylidene]-μ-carbonyl-bis[carbonyl(η^5-cyclopentadienyl)iron(0)](*Fe–Fe*), *cis*-$Fe_2(CO)_2(\eta^5$-$C_5H_5)_2(\mu$-$CO)$-[μ-$C{=}C(C_6H_5)CH_2C_6H_5].C_4H_{10}O$

By M. B. Hossain, D. J. Hanlon, D. F. Marten and Dick van der Helm

Department of Chemistry, University of Oklahoma, Norman, Oklahoma 73019, *USA*

and E. V. Dehmlow

Fakultät für Chemie, Universität Bielefeld, Bielefeld, West Germany

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Abstract

$C_{28}H_{22}Fe_2O_3.C_4H_{10}O$ crystallizes in the triclinic space group $P\bar{1}$ with $a = 12{\cdot}792\,(7)$, $b = 15{\cdot}514\,(5)$, $c = 7{\cdot}021\,(16)$ Å, $\alpha = 93{\cdot}37\,(6)$, $\beta = 80{\cdot}93\,(9)$, $\gamma = 96{\cdot}12\,(3)^\circ$, $V = 1366{\cdot}8$ Å^3 [at 138 (2) K], $M_r = 592{\cdot}3$, $Z = 2$, $D_c = 1{\cdot}439$ Mg m^{-3}, $\mu = 1{\cdot}11$ mm^{-1} (Mo $K\alpha$). The final R value is 0·072 for all 5613 reflections. The two cyclopentadienyls are *cis* disposed with respect to the Fe—Fe vector. The Fe—Fe distance of 2·5104 (5) Å is relatively short. The central bridging system is folded along the Fe—Fe vector with a dihedral angle between the two bridging systems of 24·8°. The centroids of the two cyclopentadienyl groups, the two Fe atoms and the two terminal carbonyl groups are coplanar within experimental error. The two cyclopentadienyl rings assume a staggered orientation with respect to each other.

Introduction

μ-[Benzyl(phenyl)vinylidene]-μ-carbonyl-bis[carbonyl-(η^5-cyclopentadienyl)iron(0)](*Fe—Fe*) was prepared by the reaction of bis[dicarbonyl(η^5-cyclopentadienyl)iron] with 1,1-dichloro-*trans*-2,3-diphenylcyclopropane in the presence of 50% NaOH, THF and n-$Bu_4N^+HSO_4^-$ (typical phase-transfer conditions) (Marten, Dehmlow, Hanlon, Hossain & van der Helm, 1981). An initial purple compound (the *trans* isomer) was isolated by column chromatography. The *trans* isomer slowly converted to the red *cis* isomer. This latter compound is the subject of the present investigation.

Dark-red, prismatic needle-shaped crystals of the compound, recrystallized from diethyl ether/hexane, undergo slow decomposition on exposure to air at room temperature, but they are stable at low temperature. All X-ray measurements were carried out by placing the crystal in a stream of cold nitrogen gas [138 (2) K]. Two separate crystals were used to collect the intensity data owing to a problem in the cryogenic setup during the data-collection period. Preliminary X-ray investigation indicated the crystals to be triclinic. Intensity statistics and Patterson analysis confirmed the space group to be $P\bar{1}$.

The cell parameters were obtained from a least-squares fit to $+2\theta$ and -2θ values of 35 reflections, measured at 138 (2) K using Mo $K\alpha_1$ ($\lambda = 0{\cdot}70926$ Å) radiation. Intensities of all unique reflections with $2\theta \leq 53^\circ$ were measured using Mo $K\bar{\alpha}$ radiation reflected from a graphite-crystal monochromator, and applying θ–2θ scan techniques with variable scan speed. The variable scan width was calculated for each reflection from $(A + B\tan\theta)^\circ$, where $A = (0{\cdot}80, 0{\cdot}90)$ and $B = (0{\cdot}20, 0{\cdot}20)$ for the two data crystals. A horizontal aperture with width $(2{\cdot}50 + 0{\cdot}86\tan\theta)$ mm and a height of 6 mm was placed at a distance of 173 mm from the crystal. The maximum scan time for a reflection was 50 s. Intensities of three reflections were monitored every 7200 s of X-ray exposure for both crystals. The monitor intensities varied a maximum of 2% for the first crystal and 6% for the second. The data from the two crystals were brought to a common scale with the help of the three monitor reflections. Lorentz and polarization corrections were applied to the scaled data, but no absorption correction was made. The crystal dimensions were $0{\cdot}22 \times 0{\cdot}11 \times 0{\cdot}09$ and $0{\cdot}24 \times 0{\cdot}11 \times 0{\cdot}10$ mm. The ratio of the maximum and minimum transmission for the two crystals was 1·13. Each structure amplitude was assigned an experimental weight $w_F = 1/\sigma_F^2$, where σ_F was obtained from counting statistics (Ealick & van der Helm, 1975). In all, 5613 reflections were recorded, out of which 4282 had $I > 2\sigma(I)$.

The positions of the two Fe atoms were obtained from a three-dimensional Patterson map. The positions of the C and O atoms, the diethyl ether molecule of

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Table 1. *Positional parameters and* U_{eq} *values* (Å^2) *of non-hydrogen atoms*

Standard deviations for the last digit are given in parentheses. $U_{eq} = (1/6\pi^2)\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j$.

	x ($\times 10^4$)	y ($\times 10^5$)	z ($\times 10^4$)	U_{eq} ($\times 10^4$)
Fe(1)	429·8 (3)	16802 (2)	1794·2 (7)	121 (2)
Fe(2)	−494·7 (3)	30144 (2)	1608·6 (7)	137 (2)
O(1)	1837 (2)	16466 (11)	−1860 (3)	245 (12)
O(2)	447 (2)	36087 (11)	−2160 (3)	294 (13)
O(3)	−1315 (1)	14765 (10)	−493 (3)	234 (12)
C(1)	1274 (2)	16673 (15)	−424 (5)	173 (16)
C(2)	72 (2)	33652 (15)	−653 (5)	184 (16)
C(3)	−731 (2)	18564 (15)	473 (4)	157 (15)
C(4)	929 (2)	29007 (14)	2106 (4)	126 (14)
C(5)	1792 (2)	33923 (15)	2482 (4)	133 (14)
C(6)	2747 (2)	30058 (14)	2931 (4)	144 (15)
C(7)	3032 (2)	31248 (16)	4779 (5)	230 (17)
C(8)	3888 (2)	27447 (19)	5256 (5)	273 (18)
C(9)	4480 (2)	22351 (18)	3901 (5)	265 (18)
C(10)	4216 (2)	21201 (16)	2058 (5)	242 (17)
C(11)	3363 (2)	25069 (15)	1553 (5)	182 (16)
C(12)	1855 (2)	43845 (15)	2655 (5)	180 (16)
C(13)	2762 (2)	48351 (15)	1296 (5)	173 (15)
C(14)	3363 (2)	55741 (15)	1921 (5)	206 (17)
C(15)	4160 (2)	60065 (15)	650 (5)	254 (18)
C(16)	4374 (2)	57099 (16)	−1246 (5)	255 (18)
C(17)	3788 (2)	49746 (16)	−1888 (5)	229 (17)
C(18)	2987 (2)	45386 (15)	−618 (5)	188 (16)
C(19)	882 (2)	4841 (15)	2492 (5)	247 (18)
C(20)	−239 (2)	4250 (15)	2682 (5)	254 (18)
C(21)	−590 (2)	10496 (16)	4099 (5)	256 (18)
C(22)	309 (2)	14960 (16)	4797 (5)	208 (16)
C(23)	1219 (2)	11442 (15)	3810 (5)	220 (17)
C(24)	−1178 (2)	41716 (16)	2451 (5)	271 (18)
C(25)	−827 (2)	38651 (16)	4071 (5)	237 (17)
C(26)	−1385 (2)	30486 (16)	4465 (5)	240 (17)
C(27)	−2083 (2)	28470 (16)	3085 (5)	244 (17)
C(28)	−1961 (2)	35362 (18)	1832 (5)	270 (18)
C(29)	6510 (2)	639 (19)	1759 (6)	352 (21)
C(30)	6539 (2)	−150 (18)	3885 (5)	313 (20)
O(31)	6513 (2)	8331 (11)	4803 (3)	259 (12)
C(32)	6545 (2)	8173 (19)	6822 (5)	316 (19)
C(33)	6369 (2)	17070 (22)	7712 (6)	380 (22)

solvation and all H atoms were obtained from successive difference Fourier maps. The H atoms were refined isotropically and all other atoms anisotropically. The anomalous-dispersion effect of the Mo radiation by Fe atoms was taken into account. The refinement was discontinued when the maximum parameter shifts were less than $\frac{1}{3}$ of their corresponding standard deviations. The R factor $(=\sum||kF_o|-|F_c||/\sum|kF_o|)$ based on the final parameters (Table 1)* is 0·072 for all 5613 reflections. All refinements were carried out by using a block-diagonal least-squares program (Ahmed, 1966). The Fe, C and O scattering factors were taken from *International Tables for X-ray Crystallography* (1974), and those for H atoms from Stewart, Davidson & Simpson (1965).

Results and discussion

The atom-numbering scheme is shown in a perspective view of the molecule in Fig. 1. Bond lengths are given in Fig. 2, while the corresponding interatomic angles are listed in Table 2. Least-squares-plane parameters are tabulated in Table 3. The geometry of the molecule consists of an Fe—Fe single bond bridged by a carbonyl group and the C=C(Ph)(CH$_2$Ph) ligand. In addition, about each Fe atom there is a cyclopentadienyl ring and a terminal carbonyl ligand in a *cis* configuration. The centroids of the two cyclopentadienyl rings, Cp1 and Cp2, the two Fe atoms, and the terminal carbonyls C(1)O(1) and C(2)O(2) are very nearly coplanar (the r.m.s. deviation from the mean plane is 0·011 Å, Table 3), and their mean plane is approximately perpendicular to the mean planes of the cyclopentadienyl groups (dihedral angles are 90·1 and 88·7°). The four-membered bridging ring Fe(1)Fe(2)C(3)C(4) is not planar, but folded along the Fe—Fe line. The dihedral angle between the bridging systems Fe(1)—C(3)—Fe(2) and Fe(1)—C(4)—Fe(2) is 24·8°. Some of these general features of the molecule can be appreciated from Fig. 3, which gives a view of the molecule down the Fe(1)—Fe(2) line. In addition to the planarity of the two centroids, two Fe atoms, and the two terminal carbonyl groups, and the folding of the

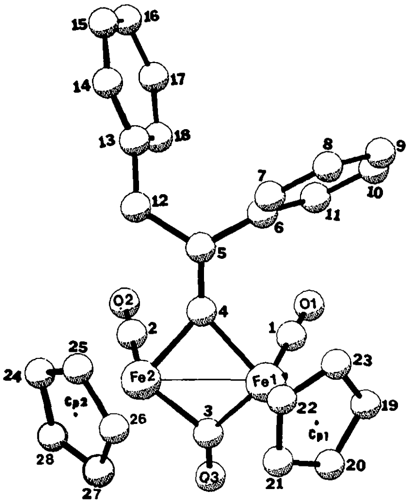

Fig. 1. A perspective view of the molecule of $(\eta^5$-$C_5H_5)_2Fe_2(CO)_3[C{=}C(Ph)CH_2Ph]$ showing atom numbering. C atoms are represented by numerals only. Cp1 and Cp2 are the centroids of the cyclopentadienyl rings.

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 36586 (27 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

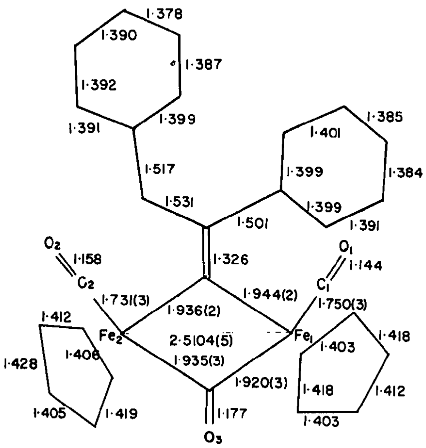

Fig. 2. Bond distances (Å). Standard deviations of the last digit are shown in parentheses. Additional distances are Fe(1)–C(19) 2·108, Fe(1)–C(20) 2·129, Fe(1)–C(21) 2·127, Fe(1)–C(22) 2·123, Fe(1)–C(23) 2·117, Fe(2)–C(24) 2·094, Fe(2)–C(25) 2·129, Fe(2)–C(26) 2·145, Fe(2)–C(27) 2·128 and Fe(2)–C(28) 2·100 Å, all with an estimated standard deviation of 0·003 Å. The estimated standard deviations for the C–C distances are between 0·003 and 0·004 Å.

Table 2. *Interatomic angles* (°)

Standard deviations for last digit are in parentheses.

Fe(2)–Fe(1)–C(1)	101·8 (1)	C(5)–C(6)–C(7)	120·2 (2)
Fe(1)–Fe(2)–C(2)	101·5 (1)	C(5)–C(6)–C(11)	121·8 (2)
Fe(2)–Fe(1)–C(3)	49·6 (1)	C(7)–C(6)–C(11)	118·1 (2)
Fe(1)–Fe(2)–C(3)	49·1 (1)	C(6)–C(7)–C(8)	121·3 (3)
Fe(2)–Fe(1)–C(4)	49·5 (1)	C(7)–C(8)–C(9)	120·4 (3)
Fe(1)–Fe(2)–C(4)	49·8 (1)	C(8)–C(9)–C(10)	118·9 (3)
C(1)–Fe(1)–C(3)	89·4 (1)	C(9)–C(10)–C(11)	121·2 (3)
C(2)–Fe(2)–C(3)	88·8 (1)	C(10)–C(11)–C(6)	120·0 (3)
C(1)–Fe(1)–C(4)	87·5 (1)	C(5)–C(12)–C(13)	114·1 (2)
C(2)–Fe(2)–C(4)	87·7 (1)	C(12)–C(13)–C(14)	120·8 (2)
C(3)–Fe(1)–C(4)	96·1 (1)	C(12)–C(13)–C(18)	120·5 (2)
C(3)–Fe(2)–C(4)	95·9 (1)	C(14)–C(13)–C(18)	118·7 (3)
*Cp1–Fe(1)–Fe(2)	132·1 (1)	C(13)–C(14)–C(15)	120·6 (3)
*Cp2–Fe(2)–Fe(1)	132·5 (1)	C(14)–C(15)–C(16)	120·3 (3)
Cp1–Fe(1)–C(1)	126·1 (1)	C(15)–C(16)–C(17)	119·8 (3)
Cp2–Fe(2)–C(2)	125·9 (1)	C(16)–C(17)–C(18)	120·1 (3)
Cp1–Fe(1)–C(3)	122·5 (1)	C(17)–C(18)–C(13)	120·5 (3)
Cp2–Fe(2)–C(3)	122·1 (1)	C(20)–C(19)–C(23)	108·4 (3)
Cp1–Fe(1)–C(4)	124·9 (1)	C(19)–C(20)–C(21)	107·5 (3)
Cp2–Fe(2)–C(4)	125·9 (1)	C(20)–C(21)–C(22)	108·5 (3)
Fe(1)–C(1)–O(1)	178·5 (2)	C(21)–C(22)–C(23)	107·9 (3)
Fe(2)–C(2)–O(2)	179·3 (3)	C(22)–C(23)–C(19)	107·7 (3)
Fe(1)–C(3)–Fe(2)	81·2 (1)	C(25)–C(24)–C(28)	108·4 (3)
Fe(1)–C(3)–O(3)	140·9 (2)	C(24)–C(25)–C(26)	107·7 (3)
Fe(2)–C(3)–O(3)	137·9 (2)	C(25)–C(26)–C(27)	108·3 (3)
Fe(1)–C(4)–Fe(2)	80·6 (1)	C(26)–C(27)–C(28)	108·4 (3)
Fe(1)–C(4)–C(5)	139·4 (2)	C(27)–C(28)–C(24)	107·3 (3)
Fe(2)–C(4)–C(5)	139·9 (2)	C(29)–C(30)–O(31)	108·3 (3)
C(4)–C(5)–C(6)	121·7 (2)	C(30)–O(31)–C(32)	111·9 (2)
C(4)–C(5)–C(12)	122·9 (2)	O(31)–C(32)–C(33)	108·3 (3)
C(6)–C(5)–C(12)	115·2 (2)		

* Cp1 and Cp2 are the centroids of the cyclopentadienyl rings.

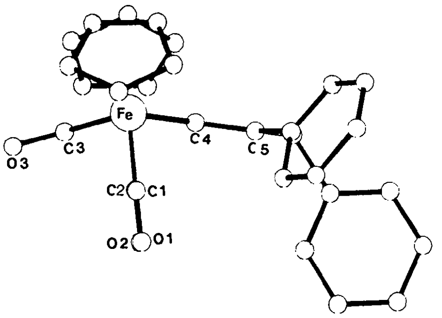

Fig. 3. A view of the molecule projected down the Fe–Fe vector. The view shows the nearly complete staggering of the cyclopentadienyl rings and the folding of the bridging ligands.

Table 3. *Least-squares planes and the atomic deviations from the planes in* Å

The equations of the planes are expressed as $m_1x + m_2y + m_3z = d$, where x,y,z are fractional coordinates. Atoms marked * were not included in the least-squares calculations. E.s.d.'s on atomic deviations and dihedral angles are 0·001–0·003 Å and 0·1° respectively.

Plane 1

$$9\cdot3557x + 7\cdot1028y + 3\cdot5435z = 2\cdot2262$$

Fe(1)	+0·005	C(1)	−0·001	C(2)	+0·000	Cp1	−0·010
Fe(2)	+0·022	O(1)	+0·003	O(2)	−0·011	Cp2	−0·008

Plane 2: cyclopentadienyl ring 1

$$1\cdot4194x - 11\cdot0231y + 5\cdot1989z = 0\cdot8900$$

C(19)	−0·003	C(21)	−0·000	C(23)	+0·003
C(20)	+0·002	C(22)	−0·002	Fe(1)*	1·748

Plane 3: cyclopentadienyl ring 2

$$8\cdot7060x - 7\cdot5359y - 3\cdot3043z = -4\cdot9781$$

C(24)	−0·001	C(26)	+0·000	C(28)	+0·001
C(25)	+0·000	C(27)	−0·001	Fe(2)*	1·744

Plane 4: phenyl ring 1

$$6\cdot2542x + 11\cdot9174y - 1\cdot6642z = 4\cdot8225$$

C(6)	−0·010	C(8)	+0·006	C(10)	−0·002
C(7)	+0·003	C(9)	−0·006	C(11)	+0·010

Plane 5: phenyl ring 2

$$9\cdot9009x - 10\cdot2857y + 2\cdot7308z = -1\cdot8816$$

C(13)	−0·003	C(15)	−0·000	C(17)	−0·000
C(14)	+0·002	C(16)	−0·001	C(18)	+0·002

Plane 6: central bridging system

$$4\cdot4864x + 2\cdot2756y - 6\cdot0254z = -0\cdot3484$$

Fe(1)	−0·157	C(3)	+0·158
Fe(2)	−0·157	C(4)	+0·156

Dihedral angles between planes

Plane 1/plane 2	90·1°
Plane 1/plane 3	88·7

bridging system, the diagram shows the almost complete staggered orientation of the two *cis* cyclopentadienyl rings.

The Fe–C distances to the cyclopentadienyl ring vary from 2·108 to 2·129 (3) Å in one ring, and from 2·094 (3) to 2·145 (3) Å in the other. Each Fe atom is situated symmetrically about the cyclopentadienyl ring. Fe(1)–Cp1 (the centroid of the cyclopentadienyl ring) is 1·748 Å and Fe(2)–Cp2 is 1·745 Å, whereas the deviations of the two Fe atoms from the mean planes of the two cyclopentadienyl rings are 1·748 and 1·744 Å respectively (Table 3). The Fe to terminal carbonyl

Table 4. *Comparison of geometric parameters of some related diiron bridged complexes*

(i) Description of compounds and their references

	Molecule	Bridging system	Reference
(*a*)	*cis*-$[(\eta^5\text{-}C_5H_5)Fe(CO)_2]_2$	2(CO)	Bryan *et al.* (1970)
(*b*)	*trans*-$[(\eta^5\text{-}C_5H_5)Fe(CO)_2]_2$	2(CO)	Mitschler *et al.* (1978)
(*c*)	*cis*-$Fe_2(CO)_3(\eta^5\text{-}C_5H_5)_2[C{=}C(CN)_2]$	(CO),[CC(CN)$_2$]	Kirchner & Ibers (1974)
(*d*)	*cis*-$(\eta^5\text{-}C_5H_5)_2Fe_2(CO)_3(C{=}NPh)$	(CO),(CNPh)	Joshi *et al.* (1965)
(*e*)	*trans*-$[(\eta^5\text{-}C_5H_5)_2Fe_2(CO)_2](C{=}NPh)_2$	2(CNPh)	Howell *et al.* (1977)
(*f*)	*cis*-$(\eta^5\text{-}C_5H_5)_2Fe_2(CO)_3[C{=}C(Ph)CH_2Ph]$	(CO),[C=C(Ph)CH$_2$Ph]	Present work

(ii) Comparison of average interatomic distances (Å) and angles (°)

Molecule	Fe—Fe	Fe—C_B bridge	Fe—C_t terminal	Fe—C cpd	Fe—Cp	Fe—C_B—Fe	C_B—Fe—C_B	Fold angle
a	2·531 (2)	1·92 (1)	1·75 (2)	2·11 (2)	1·745 (5)	82·6 (2)	96·0 (3)	16°
b	2·5389 (3)	1·9242 (8)	1·7611 (8)	2·1272 (8)	1·7511 (8)	82·56 (3)	97·44 (3)	0
*c**	2·511 (4)	1·94 (3)	1·79 (2)	2·13 (2)	1·74 (2)	82 (2)	96 (1)	18
d	2·530	1·91				84		14
e	2·530	1·93				81·7	98·2	0
f	2·5104 (5)	1·934 (3)	1·741 (3)	2·120 (3)	1·747 (3)	80·9 (1)	96·0 (1)	25

(iii) Torsion angles (°)

	*c**	*a*	*f*		*c**	*a*	*f*
C(1)—Fe(1)—Fe(2)—C(2)	3·2 (9)	5·6 (10)	0·7 (1)	Cp1—Fe(1)—Fe(2)—C(2)	−178·2 (9)	177·5 (5)	−178·8 (2)
C(1)—Fe(1)—Fe(2)—Cp2	−179·3 (8)	173·9 (5)	178·2 (2)	Cp1—Fe(1)—Fe(2)—Cp2	1·8 (8)	3·0 (5)	−1·4 (2)

* Average values for two independent molecules.

distances of 1·750 (3) and 1·731 (3) Å are systematically shorter than the corresponding distances in the bridging carbonyl; Fe(1)—C(3) is 1·920 (3) and Fe(2)—C(3) is 1·935 (3) Å. In general, the coordination geometry in the present structure has the familiar symmetrical conformation observed in a variety of diiron bridged complexes, where the bridge carbonyl ligands are replaced by heavier groups. However, there are some notable trends which warrant a description.

In Table 4 some of these geometrical parameters of the present structure are given in comparison with five other related structures. The most striking difference in the present structure is the fold angle about the Fe—Fe vector of 24·8°, which is on the average about 7–10° larger than that found in most of the known bridged diiron complexes. The present structure differs from $[(\eta^5\text{-}C_5H_5)Fe(CO)_2]_2$ (fold angle, 16°) (Bryan, Greene, Newlands & Field, 1970) in having a CO and a C=C(Ph)CH$_2$Ph bridging system instead of two symmetric carbonyl bridging ligands. If the bulkier ligand in the bridging system is responsible for the greater fold in the present structure, the structure of $(\eta^5\text{-}C_5H_5)_2Fe_2(CO)_3(C{=}NPh)$ (Joshi, Mills, Pauson, Shaw & Stubbs, 1965) which has a phenyl isocyanide bridging ligand and a folding angle of only 14° does not justify such an assumption, neither does the structure of $Fe_2(CO)_3(\eta^5\text{-}C_5H_5)_2[C{=}C(CN)_2]$ (Kirchner & Ibers, 1974), where a bridging carbonyl is replaced by a dicyanovinylidene ligand. Some of the larger fold angles observed are 25·3° in *cis*-(azulene)$_2$Fe$_4$(CO)$_{10}$ (Churchill & Bird, 1969), where cyclopentadienyls are replaced by azulene rings, 22·6° in *cis*-$(\eta^5\text{-}C_5H_5)_2Fe_2(CO)_3(SO_2)$ (Churchill & Kalra, 1973), 22·1° in $(\eta^5\text{-}C_5H_5)_2Fe_2(CO)_2(CNCH_3)_2$ (Cotton & Frenz, 1974), where both carbonyls are replaced by methyl isocyanide ligands, and 22·4° in $(CH_3)_2Si[C_5H_4Fe(CO)_2]_2$ in which the cyclopentadienyl rings are linked by a $(CH_3)_2Si$ group (Wegner, Uski, Kiester, Dabestani & Day, 1977).

It is interesting to note that in the two *trans* structures reported, *trans*-$[(\eta^5\text{-}C_5H_5)Fe(CO)_2]_2$ (Mitschler, Rees & Lehmann, 1978) and *trans*-$[(\eta^5\text{-}C_5H_5)_2Fe_2(CO)_2](PhNC)_2$ (Howell, Mays, Hunt & Mills, 1977), the central four-membered bridging ring is perfectly planar (fold angle 0°). It is also to be noted that the Fe—Fe distances in the *cis* and *trans* structures remain virtually constant (Table 4). Generally, in most of these diiron bridged compounds, the Fe—Fe distances are similar. However, the Fe—Fe distance of 2·5104 (5) Å in the present structure can be considered significantly shorter than that in the dicarbonyl-bridged complex [Fe—Fe distance of 2·531 (1) Å]. Another significant difference between the present structure and the dicarbonyl analog is in the relative orientation of the two cyclopentadienyl rings. Fig. 3 shows that the two rings assume a completely staggered orientation, whereas in the case of the dicarbonyl-bridged compound, the cyclopentadienyl rings are reported to have a near-eclipsed orientation. A partially staggered orientation of the cyclopentadienyl rings is reported for the dicyanovinylidene-bridged structure, which, incidentally, shows a shortening of the Fe—Fe distance to 2·510 Å as in the present structure. Even shorter Fe—Fe distances are reported for structures with thiocarbonyl bridging: 2·482 (1) Å in $[(\eta^5\text{-}C_5H_5)_2Fe_2(CO)(CS)]_2$ (Dunker, Finer, Clardy & Angelici, 1976) and 2·505 (2) Å in $(\eta^5\text{-}C_5H_5)_2Fe_2(CO)_3(CS)$ (Beckman & Jacobson, 1979). The structure of $(\text{azulene})_2\text{-}Fe_4(CO)_{10}$ (Churchill & Bird, 1969), which has the largest fold angle, 25·3°, also shows a shorter Fe—Fe distance [2·508 (3) Å].

The planarity of the centroids of the two cyclopentadienyl rings, the two Fe atoms and the two terminal carbonyls is somewhat distorted in the dicarbonyl complex compared to the present structure as can be seen from the torsion angles listed at the bottom of Table 4.

The cyclopentadienyl rings appear to be regular with approximate D_{5h} symmetry. C—C distances within the rings have the ranges 1·403 (4)–1·418 (4) and 1·405 (4)–1·428 (4) Å and the bond angles range from 107·3 (3) to 108·5 (3)° in the two rings. The cyclopentadienyl rings are planar with mean atomic deviations of 0·003 and 0·001 Å for the two rings (Table 3).

The two terminal carbonyls are approximately linear, with angles of 178·5 (2) and 179·2 (3)°; however, the two carbonyl groups are not equivalent: the Fe(1)—C(1) distance of 1·750 (3) Å is about 6σ longer than the Fe(2)—C(2) distance [1·731 (3) Å], while the C(1)—O(1) distance [1·144 (4) Å] is about 5σ shorter than the C(2)—O(2) distance [1·158 (3) Å]. Such asymmetry seems to be quite common in similar structures. The C—O distances in the terminal carbonyl groups are considerably shorter than the corresponding C—O distance in the bridging carbonyl [1·177 (3) Å]. The C(4)—C(5) double bond is somewhat shorter than expected, 1·326 (3) Å. The phenyl rings are perfectly planar with normal bond lengths and angles. The solvate ether molecule has a symmetrical, extended chain with normal bond distances and angles. The hydrogen distances in the structure vary from 0·89 (3) to 1·04 (3) Å. There are no unusual short intermolecular contacts.

Note added in proof: Following the acceptance of this paper we became aware of the results of a related structure $[(\eta^5\text{-}C_5H_5)Fe(CO)]_2(\mu\text{-}CO)(\mu\text{-}CHCH_3)$ (Dyke, Knox, Naish & Orpen, 1980; Meyer, Riley & Davis, 1981).

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