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Structure of a New Polymorph of *cis*-$[(\mu\text{-}CH_2)(\mu\text{-}CO)\{Fe(\eta^5\text{-}C_5H_5)CO\}_2]$

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Abstract. μ-Carbonyl-μ-methylene-bis[carbonyl(η^5-cyclopentadienyl)iron](*Fe—Fe*), $[\{Fe(CO)(\eta^5\text{-}C_5H_5\}_2(\mu\text{-}CO)(\mu\text{-}CH_2)]$, $M_r = 339.9$, triclinic, $P\bar{1}$, $a = 6.618\,(2)$, $b = 9.0321\,(10)$, $c = 11.750\,(2)$ Å, $\alpha = 101.68\,(1)$, $\beta = 99.00\,(2)$, $\gamma = 103.52\,(2)°$, $V = 653.2\,(5)$ Å^3, $Z = 2$, $D_x = 1.728$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 22.2$ cm^{-1}, $F(000) = 344$, $T = 292$ K, $R = 0.028$ for 1804 observed reflections. The structure reported herein is a different polymorph from the earlier reported monoclinic form [Korswagen, Alt, Speth & Ziegler (1981). *Angew. Chem. Int. Ed. Engl.* **20**, 1049–1051]. The molecular structure of the triclinic form agrees well with that of the monoclinic form, but more precise structural parameters are reported herein, and the bridging methylene H atoms are located. The Fe—Fe bond distance of 2.5196 (6) Å is regarded as a weak bond. The Fe—CH_2 bond distances of 1.965 (4) and 1.967 (3) Å are longer than the Fe—CO bond distances of 1.900 (3) and 1.913 (3) Å corresponding to the bridging carbonyl ligand. The Fe—CH_2—Fe bond angle of 79.7 (1)° is more acute than the Fe—CO—Fe bond angle of 82.7 (1)°. As predicted from molecular orbital calculations [Jemmis, Pinhas & Hoffmann (1980). *J. Am. Chem. Soc.* **102**, 2576–2585], the two bridging units are puckered along the Fe—Fe bond with a fold angle of 162.6 (4)°. The C—H bond lengths are 0.95 (3) and 1.00 (4) Å and the H—C—H bond angle is 110 (3)°. The molecular structure of *cis*-$[(\mu\text{-}CH_2)(\mu\text{-}CO)\{Fe(\eta^5\text{-}C_5H_5)CO\}_2]$ is comparable to the molecular structures of other bridging methylene metal dimers. Also, no statistically significant differences are found in the C—O bond distances of the terminal carbonyl ligands when the methylene bridge is replaced by a carbonyl bridge as in *cis*-$(\mu\text{-}CO)_2[Fe(\eta^5\text{-}C_5H_5)CO]_2$ [Bryan, Greene, Newlands & Field (1970). *J. Chem. Soc. A*, pp. 3068–3074], although the bridging methylene ligand is commonly regarded as a better donor and acceptor than the bridging carbonyl ligand.

Introduction. In assessing the charge density at the bridging C atom in bridging methylene metal dimers with solid-state deuterium NMR spectroscopy (Altbach, Hiyama, Gerson & Butler, 1987) and solid-state ^{13}C NMR spectroscopy (Kim, Altbach & Butler, 1991), we have prepared *cis*-$[(\mu\text{-}CH_2)(\mu\text{-}CO)\{Fe(\eta^5\text{-}C_5H_5)CO\}_2]$, (I). The crystal structure of (I) has been previously reported in brief by Korswagen, Alt, Speth & Ziegler (1981), but little information is presented in the literature. Having obtained a crystal with a different morphology from the previous reported structure we decided to determine the crystal structure of (I). The structure

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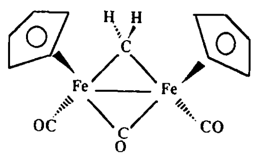
of (I) is compared to the structure of other bridging methylene metal dimers focusing on the *M*—CH_2—*M* bridging unit. Also, the structure of (I) is compared to other doubly bridged metal dimers, including the doubly bridging carbonyl dimer *cis*-$[(\mu\text{-CO})_2\{Fe(\eta^5\text{-}C_5H_5)CO\}_2]$, (II) (Bryan, Greene, Newlands & Field, 1970).



(I)

Experimental. Complex (I) was prepared following the method of Korswagen *et al.* (1981). The *cis* and *trans* isomers obtained from the synthesis were separated by low-temperature liquid chromatography. Single crystals of (I) were grown from an ether/pentane solution at 196 K.

Intensity data were collected from a red crystal of dimensions 0.08 × 0.12 × 0.40 mm at 292 K using an Enraf–Nonius CAD-4 diffractometer with graphite-monochromated Mo *Kα* radiation. Compound (I) is stable in air in the solid state, thus no extra precautions were taken for mounting the crystal. Cell dimensions and crystal orientation were determined from diffractometer coordinates of 25 reflections having $11 \leq \theta \leq 12°$. Data collection was by the ω–θ scan technique with scan rates varying from 0.53 to 4.0° min^{-1} so as to yield $I \simeq 50\sigma(I)$ for all significant data. The maximum time spent on a single scan was 120 s. 2295 unique reflections were measured in one hemisphere having $1 < \theta < 25°$, $0 \leq h \leq 7$, $-10 \leq k \leq 10$ and $-14 \leq l \leq 14$. Three standard reflections, 100, 050, 002, were monitored and showed no decrease in intensity. Background, Lorentz and polarization corrections were applied to the data. Absorption corrections were applied using the ψ-scan method. The minimum relative transmission coefficient was 91%. Of the 2295 unique data measured, 1804 had $I > 3\sigma(I)$ and were used in the refinement. The atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV, p. 95). Corrections for anomalous dispersion were made using the coefficients of Cromer (1965). The space group was determined by successful refinement of a centrosymmetric model. The structure was solved by heavy-atom methods. 221 parameters were refined by weighted full-matrix least-squares methods minimizing $\sum w(|F_o| - |F_c|)^2$, where the weights were $w = 4F_o^2/\sigma^2(F_o^2)$, and $\sigma^2(F_o^2) = [S^2(C + R^2B) + (pF_o^2)^2]/(Lp)^2$ where *S* is the scan rate, *C* is the total integrated peak count, *R* is the ratio of scan time to background counting time, *B* is the total background count, Lp is the Lorentz–polarization factor, and *p* was set to 0.02. Calculations were performed using the *SDP* software package (Frenz, 1985). Non-H atoms were refined anisotropically. H atoms were located from difference maps and refined isotropically. A correction factor for secondary extinction was refined to $g = 3.2\,(7) \times 10^{-7}$, where the correction factor $(1 + gI_c)^{-1}$ was applied to F_c. The maximum correction factor was 14.2% for the 101 reflection. Convergence was achieved with $R = 0.028$, $wR = 0.033$, $S = 1.801$, $(\Delta/\sigma)_{max} = 0.08$. The maximum and minimum residuals in a final difference Fourier map were 0.36 and -0.35 e Å^{-3}, respectively. The atomic parameters are listed in Table 1.*

Table 1. *Atomic coordinates and isotropic or equivalent isotropic thermal parameters* (Å^2)

H atoms were refined isotropically.

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

	x	*y*	*z*	B_{eq}/B_{iso}
Fe1	0.33852 (6)	0.33051 (5)	0.23195 (4)	2.712 (9)
Fe2	0.27988 (6)	0.07848 (4)	0.29856 (4)	2.497 (9)
O1	0.1143 (4)	0.4990 (3)	0.3801 (2)	5.16 (7)
O2	0.0674 (4)	0.1692 (3)	0.4866 (2)	6.43 (7)
O3	−0.0732 (3)	0.1035 (3)	0.1303 (2)	4.00 (6)
C1	0.2042 (5)	0.4308 (4)	0.3234 (3)	3.32 (7)
C2	0.1503 (6)	0.1321 (4)	0.4124 (3)	3.67 (7)
C3	0.0988 (4)	0.1515 (3)	0.1925 (3)	2.83 (6)
C4	0.4958 (5)	0.2810 (4)	0.3690 (3)	3.44 (7)
C1*A*	0.3637 (7)	0.2859 (5)	0.0507 (3)	5.4 (1)
C2*A*	0.5617 (6)	0.3157 (4)	0.1226 (4)	5.93 (9)
C3*A*	0.6150 (6)	0.4663 (5)	0.1959 (4)	6.0 (1)
C4*A*	0.4435 (7)	0.5261 (4)	0.1672 (4)	6.2 (1)
C5*A*	0.2938 (6)	0.4166 (4)	0.0790 (3)	5.27 (9)
C1*B*	0.2388 (6)	−0.1174 (4)	0.1555 (3)	4.19 (8)
C2*B*	0.4559 (6)	−0.0472 (4)	0.2011 (4)	4.94 (9)
C3*B*	0.5019 (6)	−0.0544 (4)	0.3190 (4)	5.3 (1)
C4*B*	0.3168 (7)	−0.1254 (4)	0.3464 (3)	4.99 (9)
C5*B*	0.1546 (6)	−0.1667 (4)	0.2450 (4)	4.8 (1)
H4	0.640 (4)	0.282 (3)	0.368 (2)	2.6 (6)
H5	0.486 (6)	0.335 (4)	0.450 (3)	6 (1)

Discussion. A view of the molecule showing the atomic labeling scheme is given in Fig. 1. Bond distances and relevant bond and torsion angles are given in Table 2.

The overall molecular structure of (I) reported herein is substantially the same as that reported by Korswagen *et al.* (1981) in the monoclinic ($P2_1/n$) form. Shown in Fig. 2 is the triclinic unit cell, the dimensions of which are closely related to those of the monoclinic form. Notably, our *c* axis, 11.750 (2) Å, is approximately half the length of the monoclinic symmetry axis, 22.772 (22) Å, while the

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* A complete list of bond angles, lists of H-atom coordinates and thermal parameters, bond distances and angles involving H atoms, anisotropic thermal parameters, results of least-squares-planes calculations, and structure-factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54682 (23 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: ST0504]

other axial lengths differ by less than 1%. Unfortunately, detailed comparison of the two modes of molecular packing is not possible since the coordinates for the monoclinic form are neither published nor available in the Cambridge Structural Database Files (Allen, Kennard & Taylor, 1983). Relative to the earlier report (in which $R = 0.098$), structural parameters have been more accurately determined in this work. Most importantly, in the present work, the bridging methylene H atoms were located and refined.

Table 2. *Bond distances* (Å) *and relevant bond and torsion angles* (°)

Fe1—Fe2	2.5196 (6)	C1—O1	1.143 (4)
Fe1—C1	1.746 (4)	C2—O2	1.136 (5)
Fe1—C3	1.900 (3)	C3—O3	1.182 (3)
Fe1—C4	1.965 (4)		
Fe1—C1*A*	2.126 (4)	C4—H4	0.95 (3)
Fe1—C2*A*	2.114 (4)	C4—H5	1.00 (4)
Fe1—C3*A*	2.106 (4)		
Fe1—C4*A*	2.078 (4)	C1*A*—C2*A*	1.378 (5)
Fe1—C5*A*	2.104 (4)	C2*A*—C3*A*	1.387 (5)
Fe1—Cp*A*(av.)*	2.106 (4)	C3*A*—C4*A*	1.391 (7)
Fe1—Cp*A*(cg)†	1.751	C4*A*—C5*A*	1.356 (5)
		C5*A*—C1*A*	1.364 (6)
Fe2—C2	1.746 (4)		
Fe2—C3	1.913 (3)	C1*B*—C2*B*	1.396 (5)
Fe2—C4	1.967 (3)	C2*B*—C3*B*	1.389 (6)
Fe2—C1*B*	2.111 (3)	C3*B*—C4*B*	1.362 (6)
Fe2—C2*B*	2.113 (4)	C4*B*—C5*B*	1.395 (5)
Fe2—C3*B*	2.119 (4)	C5*B*—C1*B*	1.367 (6)
Fe2—C4*B*	2.086 (4)		
Fe2—C5*B*	2.098 (3)		
Fe2—Cp*B*(av.)*	2.105 (4)		
Fe2—Cp*B*(cg)†	1.747		
C1—Fe1—C4	91.0 (2)	C2*A*—C1*A*—C5*A*	107.5 (3)
C1—Fe1—Fe2	99.7 (1)	C1*A*—C2*A*—C3*A*	108.8 (4)
		C2*A*—C3*A*—C4*A*	105.9 (3)
C2—Fe2—Fe1	97.7 (1)	C3*A*—C4*A*—C5*A*	108.9 (4)
C2—Fe2—C4	86.5 (1)	C4*A*—C5*A*—C1*A*	108.8 (4)
Fe1—C1—O1	177.5 (3)	C2*B*—C1*B*—C5*B*	107.2 (3)
Fe2—C2—O2	179.0 (3)	C1*B*—C2*B*—C3*B*	108.2 (3)
		C2*B*—C3*B*—C4*B*	107.7 (3)
Fe1—C3—Fe2	82.7 (1)	C3*B*—C4*B*—C5*B*	108.3 (4)
Fe1—C3—O3	139.6 (3)	C4*B*—C5*B*—C1*B*	108.5 (3)
Fe2—C3—O3	137.6 (3)		
		Cp*A*(cg)—Fe1—Fe2	135.9
Fe1—C4—Fe2	79.7 (1)	Fe1—Fe2—Cp*B*(cg)	135.5
Fe1—C4—H4	116 (2)		
Fe1—C4—H5	118 (2)		
Fe2—C4—H4	117 (2)		
Fe2—C4—H5	115 (2)		
H4—C4—H5	110 (3)		
C1*A*—Fe1—Fe2—C1*B*	−5.8 (2)	C4*A*—Fe1—Fe2—C4*B*	11.3 (7)
C2*A*—Fe1—Fe2—C2*B*	−5.7 (2)	C5*A*—Fe1—Fe2—C5*B*	−2.9 (3)
C3*A*—Fe1—Fe2—C3*B*	−4.8 (2)	C1—Fe1—Fe2—C2	−4.3 (2)

* Average distance from Fe to the cyclopentadienyl ring C atoms.

† Distance from Fe to the centroid of the cyclopentadienyl ring.

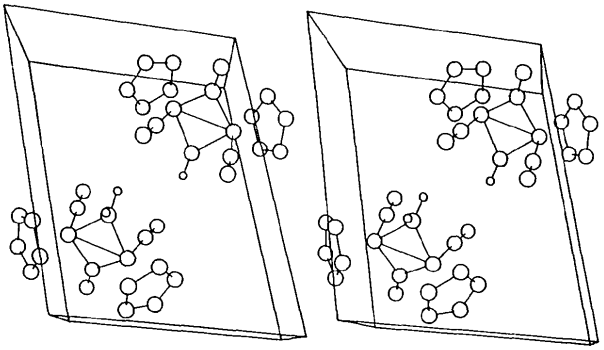

Fig. 2. Stereoview of the unit cell, viewed approximately down the *a* axis with *b* horizontal. The origin is in the lower left background.

The dimeric unit in (I) contains two Fe atoms bridged by a carbonyl and a methylene group, and two cyclopentadienyl (Cp) and two terminal carbonyl ligands in a *cis* configuration. The metal—metal distance of 2.5196 (6) Å is comparable to those found for related molecules, as shown in Table 3; in general, these metal—metal bonds are regarded as weak single bonds (Bursten & Cayton, 1986, 1987; Mitschler, Rees & Lehmann, 1978). The Fe—CH$_2$—Fe and Fe—CO—Fe bond angles of 79.7 (1) and 82.7 (1)°, respectively, are very acute as compared to the corresponding angles for a tetrahedral or trigonal central C atom. This is a characteristic of bridging metal—metal complexes and is a consequence of restrictions imposed by the metal—metal bond. The Fe—CH$_2$—Fe bond angle is more acute than the Fe—CO—Fe bond angle, and the Fe—CH$_2$ bond lengths of 1.965 (4) and 1.967 (3) Å are longer than the Fe—CO bond lengths (corresponding to the bridging carbonyl unit) of 1.900 (3) and 1.913 (3) Å. This characteristic is also present in cis-[(μ-CO)(μ-CCH$_2$CH$_2$){FeCp(CO)}$_2$] and cis-[(μ-CO)(μ-CHMe){FeCp(CO)}$_2$] (see Table 3). As pointed out for the latter compound (Meyer, Riley & Davis, 1981), this is consistent with a different hybridization for the carbonyl and methylene bridging C atoms. The C atom of a bridging carbonyl ligand is regarded as having an sp^2 hybridization whereas the C atom of the bridging methylene ligand seems to have some sp^3 character based on structural data, IR data and the charge density on C as determined from solid-state deuterium NMR spectroscopy (see below). The data presented in Table 3 for cis-[(μ-CO)(μ-CR){FeCp(CO)}$_2$], where R = S, NPh, C(CN)$_2$ and

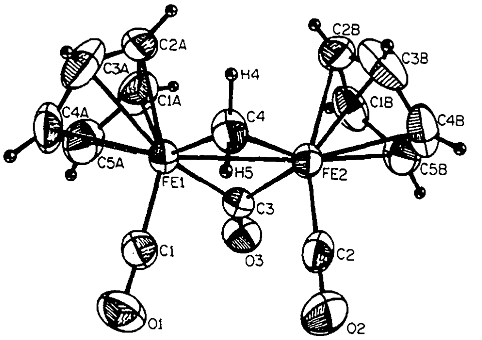

Fig. 1. *ORTEP*II diagram (Johnson, 1976) of cis-[(μ-CH$_2$)(μ-CO){Fe(η^5-C$_5$H$_5$)CO}$_2$] showing the labeling scheme. For clarity the H atoms of the cyclopentadienyl rings are not labeled.

Table 3. *Relevant structural parameters* (Å, °) *for the* Fe_2C_2 *central unit for* Fe *dimers with two bridging ligands*

	Fe—Fe	Fe—C—Fe	Fold angle[a]	Fe—C
cis[(μ-CO)(μ-CH$_2$){FeCp(CO)}$_2$][b]	2.5196 (6)	82.7 (1) (CO) 79.7 (1) (CH$_2$)	163	1.900 (3), 1.913 (3) (CO) 1.965 (4), 1.967 (3) (CH$_2$)
cis-[(μ-CO)(μ-CHCH$_3$){FeCp(CO)}$_2$][c]	2.5244 (6)	83.0 (1) (CO) 78.9 (1) (CHCH$_3$)	166	1.905 (1) (CO) 1.987 (1) (CHCH$_3$)
	2.520 (2)	82.9 (2) (CO) 78.8 (1) (CHCH$_3$)	166	1.903 (3) (CO) 1.986 (3) (CHCH$_3$)
cis-[(μ-CO)(μ-CCH$_2$CH$_2$){FeCp(CO)}$_2$][d]	2.503 (7)	81.7 (3) (CO) 80.2 (3) (CCH$_2$CH$_2$)	158	1.914 (10) (CO) 1.943 (8) (CCH$_2$CH$_2$)
cis-[(μ-CO)$_2${FeCp(CO)}$_2$][e]	2.531 (2)	82.8 (3), 82.4 (3)	164	1.918 (7), 1.908 (7) 1.917 (7), 1.925 (7)
trans-[(μ-CO)$_2${FeCp(CO)}$_2$][f]	2.5389 (3)	82.56 (3)	180	1.9279 (8) 1.9204 (8)
cis-[(μ-CO)(μ-CS){FeCp(CO)}$_2$][g]	2.504 (3)	81.6 (4) (CO) 83.0 (3) (CS)	163	1.927 (8), 1.906 (9) (CO) 1.889 (8), 1.889 (8) (CS)
	2.506 (2)	81.8 (4) (CO) 83.5 (4) (CS)	166	1.920 (8), 1.907 (8) (CO) 1.890 (8), 1.875 (8) (CS)
cis-[(μ-CO)(μ-CNPh){FeCp(CO)}$_2$][h]	2.53	84 (CO) 84 (CNPh)	166	1.91 (CO) 1.90 (CNPh)
cis-[(μ-CO){μ-CC(CN)$_2$}{FeCp(CO)}$_2$][i]	2.509 (4)	80 (1) (CO) 85 (1) [CC(CN)$_2$]	162	1.97 (3), 1.91 (3) (CO) 1.83 (2), 1.88 (2) [CC(CN)$_2$]
	2.512 (4)	80.9 (9) (CO) 83.9 (9) [CC(CN)$_2$]	162	1.96 (2), 1.91 (3) (CO) 1.84 (2), 1.92 (2) [CC(CN)$_2$]
cis-[(μ-CO){μ-CC(Ph)(CH$_2$Ph)}{FeCp(CO)}$_2$][j]	2.5104 (5)	81.2 (1) (CO) 80.6 (1) [CC(Ph)(CH$_2$Ph)]	155	1.920 (3), 1.935 (3) (CO) 1.944 (2), 1.936 (2) [CC(Ph)(CH$_2$Ph)]
cis-[(μ-methylcyclopropylidene)(μ-CO){FeCp(CO)}$_2$][k]	2.504 (4)	81.5 (8) (CO) 80.7 (7) (C$_3$H$_6$)	160	1.949 (20), 1.885 (21) (CO) 1.937 (18), 1.930 (19) (C$_3$H$_6$)
cis-[{μ-C(CN)SCH$_3$}(μ-CO){FeCp(CO)}$_2$][l]	2.540 (1)	82.5 (1) (CO) 79.1 (1) [C(CN)SCH$_3$]	166	1.934 (2), 1.917 (2) (CO) 1.989 (2), 1.999 (2) [C(CN)SCH$_3$]
cis-[{μ-C(CN)N(CH$_3$)C(O)SCH$_3$}(μ-CO){FeCp(CO)}$_2$][m]	2.506 (1)	82.3 (1) (CO) 77.9 (1) (CR_2)	163	1.886 (3), 1.924 (3) (CO) 1.995 (3), 1.991 (3) (CR_2)
cis-[(μ-CH$_2$)(μ-CO){FeCp′(CO)}$_2$][n]	2.527 (2)	84.5 (3) (CO) 80.6 (3) (CH$_2$)	162	1.886 (7), 1.874 (7) (CO) 1.950 (8), 1.959 (8) (CH$_2$)
cis-[(μ-C$_3$H$_3$){Fe$_2$(μ-CO)Cp$_2$(CO)$_2$}$_2$]PF$_6$[o]	2.553 (3)	82.3 (5) (CO) 79.8 (4) (CHR)	172	1.91 (1), 1.95 (1) (CO) 1.95 (1), 2.00 (1) (CHR)
	2.555 (3)	83.1 (5) (CO) 79.9 (5) (CHR)	175	1.96 (1), 1.89 (2) (CO) 2.03 (1), 1.95 (1) (CHR)
cis-[(μ-CHCHCO$_2$Et)(μ-CO){FeCp(CO)}$_2$]$_2$[p]	2.533 (1)	82.2 (2) (CO) 79.3 (1) (CHR)	168	1.943 (5), 1.912 (5) (CO) 1.988 (5), 1.984 (4) (CHR)
	2.531 (1)	82.6 (2) (CO) 79.5 (2) (CHR)	165	1.906 (5), 1.928 (5) (CO) 1.983 (4), 1.977 (4) (CHR)
cis-[{μ-CHCNC(CH$_3$)$_3$}(μ-CO){FeCp(CO)}$_2$]PF$_6$[q]	2.534 (1)	81.8 (1) (CO) 79.7 (1) (CHR)	171	1.937 (4), 1.933 (4) (CO) 1.977 (4), 1.978 (4) (CHR)
cis-[(μ-CHCO)(μ-CO){FeCp(CO)}$_2$]PF$_6$[q]	2.548 (1)	81.7 (2) (CO) 79.4 (2) (CHR)	173	1.948 (4) (CO) 1.994 (4) (CHR)
cis-[(μ-CHCOC$_6$C$_5$)(μ-CO){FeCp(CO)}$_2$][r]	2.519 (1)	82.5 (2) (CO) 78.7 (2) (CHR)	161	1.887 (4), 1.934 (5) (CO) 1.970 (5), 2.001 (4) (CHR)
cis-[(μ-CHCO$_2{}^t$Bu)(μ-CO){FeCp(CO)}$_2$][s]	2.513[t]	85.3 (10) (CO) 81.5 (10) (CHR)	162	1.912 (25), 1.794 (23) (CO) 1.862 (24), 1.982 (25) (CHR)
	2.505	81.3 (10) (CO) 79.4 (9) (CHR)	164	1.926 (23), 1.919 (26) (CO) 1.967 (25), 1.995 (22) (CHR)

Notes: (*a*) Dihedral angle formed by the planes defined by the two Fe atoms with the C atom of each bridging ligand. (*b*) Present work. (*c*) This molecule has a mirror plane that passes through the bridging carbonyl bridging methylene ligands (Meyer *et al.*, 1981; Orpen, 1983). (*d*) Hoel, Ansell & Leta (1984). (*e*) Bryan *et al.* (1970). (*f*) This molecule has an inversion center at the middle of the Fe—Fe bond; thus the fold angle is required by symmetry to be 180° (Mitschler *et al.*, 1978; Bryan & Greene, 1970). (*g*) Two independent molecules (Beckman & Jacobson, 1979). (*h*) There is very little information in the literature (Joshi, Mills, Pauson, Shaw & Stubbs, 1965) for this molecule (R = 0.11). (*i*) Two independent molecules (Kirchner & Ibers, 1974). (*j*) Hossain, Hanlon, Marten, van der Helm & Dehmlow (1982). (*k*) Ansell, Leta, Hoel & Habeeb (1986). (*l*) Busetto, Bordoni, Zanotti, Albano & Braga (1988). (*m*) Busetto, Corlucci, Zanotti, Albano & Braga (1990). (*n*) Cp′ = methylcyclopentadienyl (Caballero, Chávez, Göknur, Löchel, Nuber, Pfisterer, Ziegler, Alburquerque, Eguren, & Korswagen, 1989). (*o*) Casey, Marder & Rheingold (1985). (*p*) Casey, Austin & Rheingold (1987). (*q*) Casey, Crocker, Niccolai, Fagan & Konings (1988). (*r*) Casey, Crocker, Vosejpka & Rheingold (1989). (*s*) Two independent molecules (Herrmann, Plank, Bernal & Creswick, 1980). (*t*) Coordinates obtained from Cambridge Structural Database. Standard deviations not available.

CC(Ph)(CH$_2$Ph), support this idea. The Fe—C bond distances in Fe—C═S, Fe—C═NPh, Fe—C═C(CN)$_2$ and Fe—C═C(Ph)(CH$_2$Ph) [1.84 (2) to 1.944 (2) Å] are generally shorter than the Fe—C bond distances in Fe—CH$_2$, Fe—CHMe and Fe—CCH$_2$CH$_2$ [1.943 (8) to 1.987 (1) Å] which is consistent with an sp^2 hybridization for the thiocarbonyl, isonitrile and ethylidene bridging ligands. The central Fe$_2$C$_2$ unit in (I) is nonplanar; the dihedral angle formed by the planes defined by the two Fe atoms with the C atom of each bridging ligand is 162.6 (4)°. The two terminal carbonyls are positioned outside of the Fe$_2$C$_2$ unit. Similar dihedral angles are also found in other bridging structures with a *cis* configuration (see Table 3), as anticipated from molecular orbital calculations performed on *cis*-[MCp(CO)$_2$]$_2$ complexes (Jemmis, Pinhas & Hoffmann, 1980). In that molecular orbital description the puckering of the M_2C_2 unit with a *cis* configuration maximizes the interaction between the lobes of the bridging carbonyl and metal orbitals; in the *trans* configuration there is no net gain in the orbital interaction by puckering since the two lobes of the metal orbitals are on different sides of the M_2C_2 unit; thus the unit remains planar {see *trans*-[(μ-CO)$_2${FeCpCO}$_2$] in Table 3}.

The Fe—CH_2—Fe and CH_2 planes are perpendicular to each other within the limits of experimental error [dihedral angle between the planes = 88 (2)°]. This is the preferred conformation according to the molecular orbital description of the bonding in bridging methylene metal dimers (Hofmann, 1979; Bursten & Cayton, 1986, 1987; Calabro, Lichtenberger & Herrmann, 1981; Pinhas, Albright, Hofmann & Hoffmann, 1980), because it places the empty $\pi(CH_2)$ orbital on C (in the plane of the dimetallocyclopropane unit) parallel to the metal—metal bond maximizing its interaction with one of the filled metal-centered π^* orbitals involved in back donation.

The terminal carbonyl groups are not quite eclipsed, as the C1—Fe1—Fe2—C2 torsion angle is −4.30 (16)°. As found in the case of *trans*-$[(\mu\text{-CH}_2)\{\text{RhCp(CO)}\}_2]$ (Takusagawa, Fumagalli, Koetzle & Herrmann, 1981), a small twist between the FeCp(CO) units around the metal—metal bond probably favors a better overlap between the orbitals in the bridging ligands and the orbitals in the $[\text{FeCp(CO)}]_2$ metal fragment.

The C—H bond distances and the H—C—H bond angle found in (I) are 0.95 (3) and 1.00 (4) Å, and 110 (3)°, respectively. The structural parameters found in this work for the CH_2 unit compare well with the values found for the Rh and Os compounds (see Table 4) from neutron diffraction studies. The structural parameters of the CH_2 unit in (I) and in the compounds listed in Table 4 indicate that the C site at the methylene bridge is similar to an aliphatic methylene C atom. Nevertheless, caution has been advised with regard to assigning C atom hybridization based on C—H bond distances and H—C—H bond angles for *M*—CH_2 units since the *M*—CH_2—*M* angle is much smaller than the C—C—C angle for an sp^3 or sp^2 central C atom (Schultz, Williams, Calvert, Shapley & Stucky, 1979). It was also pointed out that the H—C—H bond angle is influenced by a repulsive interaction between the $\pi(CH_2)$ orbital on C and one of the metal–metal π orbitals which is perpendicular to the dimetallocyclopropane plane (Hofmann, 1979). However, IR spectroscopy also points to the aliphatic character of the C—H bond. We have previously assigned the stretching frequencies for the methylene unit to ν(C—H) = 2953, 2899 cm^{-1} (Altbach *et al.*, 1987); note that the C—H stretching frequencies are rather close to a typical aliphatic stretching frequency of 2960 cm^{-1}. In the absence of Fermi resonances and coupling to bending vibrations, the ν(C—H) transition frequency is strongly correlated with the C—H bond distance. This was demonstrated by McKean (1976, 1978) and Long, Morrison, McKean & McQuillan (1984) who developed a linear correlation between C—H bond distances and the so-called isolated C—H stretching frequency, $\nu^{\text{is}}_{\text{CH}}$ (Herzberg, 1945), which is determined from the IR absorption band that corresponds to the C—H stretching frequency for a partially deuteriated C atom, CH^2H. To illustrate the validity of the method for organometallic compounds it is useful to note that, for $[(\mu_3\text{-CH})\{\text{Co(CO)}_3\}_3]$, C—H bond distances of 1.09 (1) and 1.084 (1) Å were obtained from the C—H stretching frequency of 3041 cm^{-1} (Parmeter, Hills & Weinberg, 1986), and from a neutron diffraction study (Leung, Coppens, McMullan & Koetzle, 1981), respectively. Using the quantitative results of McKean (1976, 1978), the C—H bond distance for the bridging methylene site in (I) is estimated to be 1.10 (1) Å. The C—H bond distance of 1.10 (1) Å is comparable to the C—H bond distance of 1.096 (2) Å found for the methylene unit of propane from gas-phase microwave spectroscopy (Pacansky & Dupuis, 1979). Also, it is closer to the value of 1.091 (4) Å found for a terminal CH_3 in $[\text{CpTa(CH}_3)(\text{CH}_2)]$ from a neutron diffraction study (Takusagawa, Koetzle, Sharp & Schrock, 1988), than to the value of 1.081 (5) Å found for the terminal CH_2 in the same compound [the Ta—CH_2 bond is regarded as a double bond (Schrock, 1979)]. Thus, the results of the analysis of the IR and X-ray data support the results of the solid-state deuterium NMR experiments where a zero charge on the bridging methylene C atom of (I) was found when compared to a normal aliphatic C atom such as the bridging C in fluorene (Altbach *et al.*, 1987).

The Cp rings are planar; the maximum deviations from the planes defined by the rings, denoted from now on as Cp*A* and Cp*B*, are 0.004 (4) and 0.009 (4) Å for atoms C4*A* and C4*B*, respectively. The rings are in an almost eclipsed conformation; the C_{CpA}—Fe1—Fe2—C_{CpB} dihedral angles vary from −2.9 (3) to 11.3 (7)°. The ring C—C bond lengths vary between 1.356 (5) and 1.391 (7) Å in Cp*A*, and between 1.362 (6) and 1.396 (5) Å in Cp*B*; the maximum deviation of any individual C—C bond length from the average values of 1.375 Å for Cp*A* and 1.382 Å for Cp*B* never exceeds 4σ. The variation in the C—C bond length is widely encountered in crystal structures of organometallic compounds with Cp ligands. Even though the difference in C—C bond distance may arise from a differential interaction between the Cp ligand and the metal orbitals (Takusagawa *et al.*, 1981; Mitschler *et al.*, 1978; Albright, Hofmann & Hoffmann, 1977), in most cases it is a consequence of thermal motions (Fitzpatrick, Le Page & Butler, 1981; Fitzpatrick, Le Page, Sedman & Butler, 1981). Solid-state proton and deuterium NMR spin-lattice relaxation-time experiments have shown that Cp rings are rapidly rotating along their fivefold axes at room temperature (Altbach, Hiyama, Wittebort & Butler, 1990; Gilson, Gomez, Butler &

Table 4. *Structural parameters* (Å, °) *for some bridging methylene metal compounds*

	M—*M*	*M*—C	*M*—C—*M*	C—H	H—C—H	Dihedral angle[a]
trans-$[(\mu\text{-CH}_2)\{\text{MnCp(CO)}_2\}_2]$[b]	2.7996 (11)	2.026 (2)	87.4 (1)	0.93 (2)	109 (2)	80.4
cis-$[(\mu\text{-CH}_2)(\mu\text{-CO})\{\text{FeCp(CO)}\}_2]$[c]	2.5196 (6)	1.965 (4) 1.967 (3)	79.7 (1)	0.95 (3) 1.00 (4)	110 (3)	88 (2)
trans-$[(\mu\text{-CH}_2)\{\text{Co(MeCp)(CO)}\}_2]$[d]	2.497 (1)	1.925 (2) 1.920 (2)	81.0 (1)	1.03 (2) 0.86 (2)	108 (2)	85.6 (10)
trans-$[(\mu\text{-CH}_2)\{\text{RhCp(CO)}\}_2]$[e]	2.662 (1)	2.048 (1) 2.051 (1)	81.0 (1)	1.095 (2) 1.094 (2)	110.4 (1)	84.1 (6)
$[(\mu\text{-H})_2(\mu\text{-CH}_2)\text{Os}_3(\text{CO})_{10}]$[f]	2.824 (3)	2.151 (5) 2.510 (6)	82.1 (2)	1.090 (11) 1.091 (10)	106.0 (8)	88.8 (4)
cis-$[(\mu\text{-CH}_2)(\mu\text{-CO})\{\text{FeCp}'(\text{CO})\}_2]$[g]	2.527 (2)	1.950 (8) 1.959 (8)	80.6 (3)	N.A.		
$[(\mu\text{-CH}_2)(\mu\text{-CO})\{\text{Cp(CO)FeMn(CO)}_4\}]$[h]	2.6178 (10), 2.6127 (9)		N.A			
$[(\mu\text{-CH}_2)(\mu\text{-CO})_2\{\text{Fe(CO)}_3\}_2]$[i]	2.504 (1)		N.A.			

Notes: (*a*) Dihedral angle between the methylene plane and the M_2C plane. (*b*) Structural parameters obtained from single-crystal X-ray diffraction data at 130 K (Clemente, Biagini, Rees & Herrmann, 1982). (*c*) Present work. (*d*) Theopold & Bergman (1983). (*e*) Structural parameters obtained from a neutron diffraction study at 15 K (Takusagawa *et al.*, 1981). (*f*) Structural parameters obtained from a neutron diffraction study (Schultz *et al.*, 1979). (*g*) Cp′ = methylcyclopentadienyl, H-atom positions assigned (Caballero *et al.* 1989). (*h*) Disordered methylene position (Gadol & Davis, 1982). (*i*) Disordered methylene position (Meyer *et al.*, 1981).

Fitzpatrick, 1983). For instance, the results of a solid-state deuterium NMR spin-lattice relaxation-time study on *trans*-$[(\mu\text{-CO})_2\{\text{FeCp(CO)}\}_2]$ (Altbach *et al.*, 1990) show that the jump rate between neighboring sites of the Cp ring is $1.9\,(4) \times 10^{11}\ \text{s}^{-1}$ at 280 K. The presence of thermal motions in (I) is also reflected in the large values of the thermal parameters for the C atoms of the rings. The angles between the Fe1—Cp*A*(cg) and Fe2—Cp*B*(cg) vector [Cp*A*(cg) and Cp*B*(cg) refer to the centroids of the Cp*A* and Cp*B* rings] and the Fe—Fe bond vector are 135.9 and 135.5°, respectively. It has been discussed previously, in connection with the crystal structure determination of (II), that this tilt angle is not a consequence of H···H interactions between the two rings since in *trans*-$[(\mu\text{-CO})_2\{\text{FeCp(CO)}\}_2]$ the angle is 139.1° where there are no H···H interactions (Bryan & Greene, 1970; Mitschler *et al.*, 1978). Furthermore, there are only two Cp···Cp contacts in (I) (Cp···Cp denotes contacts between C atoms belonging to different Cp rings) that are less than 4 Å. These correspond to the Cp···Cp distances of 3.527 (6) and 3.901 (6) Å for C2*A*···C2*B* and C1*A*···C2*B*, respectively. Thus, the degree of tilting must be imposed by electronic demands (Albright *et al.*, 1977).

The crystal structure of (I) is comparable in most features to other bridging methylene metal (Mn, Fe, Rh and Co) dimers (Herrmann, 1982; also refer to Table 4). The *M*—CH$_2$ bond lengths in (I), 1.965 (4) and 1.967 (3) Å, are in the range of bond distances found in other bridging methylene metal dimers. The *M*—CH$_2$—*M* bond angle of 79.7 (1)° in (I) is more acute than in the other dimers listed in Table 4 probably because of constraints imposed by the second bridging ligand in (I), instead of constraints imposed by the metal—metal bond, since in *trans*-$[(\mu\text{-CH}_2)\{\text{Co(MeCp)CO}\}_2]$, the metal—metal bond length is shorter than in (I) but the *M*—CH$_2$—*M* bond angle is larger. This idea is supported by the fact that the first two Fe dimers listed in Table 3, which have a second bridging ligand, have *M*—C*RR*′—*M* bond angles of 78.9 (1) and 80.2 (3)°, respectively; also in the triply bridged Ru dimer, $[(\mu\text{-CH}_2)_3\{\text{Ru(PMe}_3)_3\}_2]$, the *M*—CH$_2$—*M* angle is 78.0° (Andersen, Jones, Wilkinson, Hursthouse & Abdul Malik, 1977), despite the fact that the Ru—Ru bond length of 2.650 (1) Å is longer than those of most of the compounds listed in Table 4.

The molecular structures of (I) and (II) are very similar. No notable differences are seen in the structural features of the Fe$_2$C$_2$ central unit; the Fe—Fe and Fe—CO bond lengths are the same to within 0.01 Å, and both the Fe—CO—Fe bond angles and the fold angle between the two planes of the central unit are also the same within the limits of experimental errors (see Table 3). Even though the bridging methylene ligand is described as a better acceptor than the bridging carbonyl based on molecular orbital calculations (Bursten & Cayton, 1986, 1987; Hofmann, 1979; Shaik, Hoffmann, Fisel & Summerville, 1980), no statistically significant difference in C—O bond lengths for the terminal carbonyls is found; the C—O bond lengths of the terminal carbonyls are 1.143 (4) and 1.136 (5) Å in (I), and 1.147 (10) and 1.159 (9) Å in (II).

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Structure of 1,10-Diaminodecane Tetrachlorozincate

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Abstract. [NH$_3$(CH$_2$)$_{10}$NH$_3$][ZnCl$_4$], $M_r = 381.51$, triclinic, $P\bar{1}$, $a = 7.296\,(1)$, $b = 10.110\,(3)$, $c = 12.814\,(4)$ Å, $\alpha = 90.84\,(2)$, $\beta = 101.17\,(2)$, $\gamma = 92.52\,(2)°$, $V = 926.13$ Å^3, $Z = 2$, $D_x = 1.37$ Mg m^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 1.925$ mm^{-1}, $F(000) = 396$, $T = 298$ K, final $R = 0.070$ for 1237 unique reflections [$I > 3\sigma(I)$]. The structure is characterized by layers of inorganic ions sandwiched between layers formed by the paraffinic chains.

Introduction. The alkyldiammonium tetrahalometallates of general formula [NH$_3$(CH$_2$)$_n$NH$_3$]-