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BRIDGED FERROCENES

III *. THE CRYSTAL AND MOLECULAR STRUCTURE OF 1,1′,2,2′,3,4,4′,5′-TETRAKIS(TRIMETHYLENE)FERROCENE

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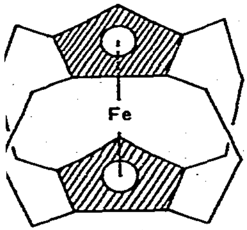
Summary

The structure of 1,1′,2,2′,3,4,4′,5′-tetrakis(trimethylene)ferrocene has been determined by X-ray diffraction. This compound forms prismatic crystals of monoclinic space group $P2_1/c$ and four molecules in the unit cell with a 8.649(4), b 21.623(2), c 9.120(1) Å and β 107.21(3)°. The structure was solved from three dimensional Patterson and Fourier synthesis, and was refined by least squares. The two cyclopentadienyl rings are eclipsed and the dihedral angle between the rings is 11.1°. The iron—carbon distances ranged from 1.997(2) to 2.047(2) Å.

Introduction

The multi-bridged ferrocenes have been receiving attention because of interesting chemical and physical properties as a cage compound containing an iron at the center. The preparation of 1,1′,2,2′,3,3′,4,4′-tetrakis(trimethylene)ferrocene (I) was first described by Schögl and Peterlik [1].



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[*] For part II see ref. 4.

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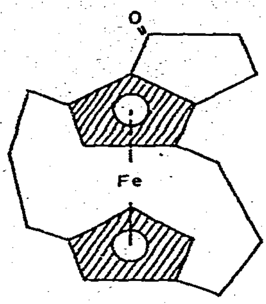
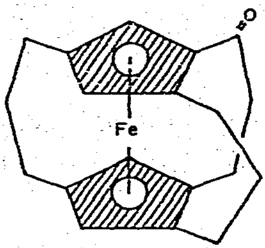
However, Bublitz and Rinehart [2] provided convincing evidence in conflict with this report. Apparently Schlögl and Peterlik had confused ketones II and III, and used II for further synthesis rather than III. Therefore, the compound

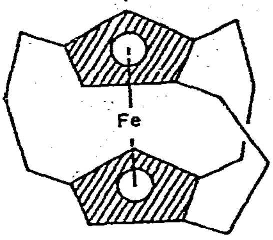


II



III

prepared contained at least one homoannular trimethylene group. Vigo [3] using III as an intermediate reported the successful preparation of I. We had already confirmed [4] the structure of 1,1′,2,2′,4,4′-tris(trimethylene)ferrocene (IV) as previously assigned by Rinehart and co-workers [5] and found interesting features concerning the iron—ring distances and ring planarity in agreement with predictions from Mössbauer experiments [6]. Since compound I would come



IV

under the influence of strain introduced by the bridges even more than IV, we decided to determine its structure and to measure the pertinent bond distances. As it turned out, however, the compound is not I, but has the structure of 1,1′,2,2′,3,4,4′,5′-tetrakis(trimethylene)ferrocene (Fig. 1).

Experimental

1,1′,2,2′,3,4,4′,5′-Tetrakis(trimethylene)ferrocene was prepared by the methods developed by Rinehart et al. [5] and Vigo [3] with only minor modifications. Dark yellow prismatic crystals were obtained by recrystallization from benzene.

A crystal was mounted on a glass fiber, using epoxy cement, such that the a^* axis was approximately parallel to the fiber axis. The precession photographs, using Zr-filtered Mo-K_α radiation (λ 0.7107 Å) indicated lattice symmetry $2/m$. The systematic absences, $h0l (l = 2n + 1)$ and $0k0$ $(k = 2n + 1)$, were consistent with monoclinic space group $P2_1/c$.

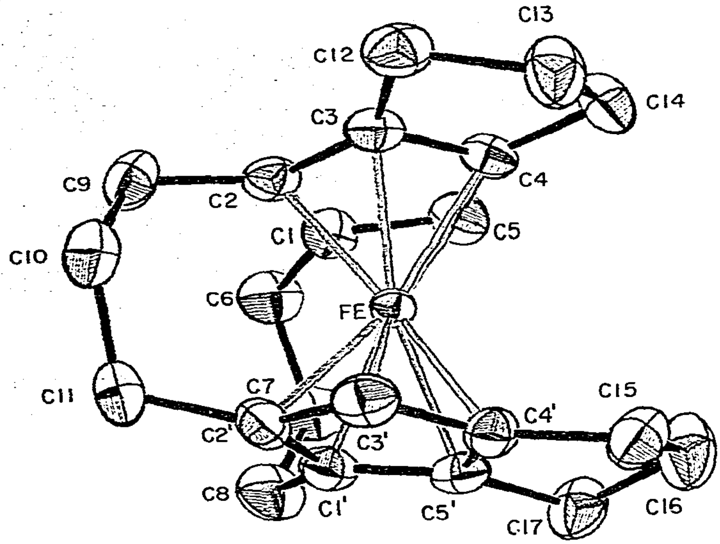


Fig. 1. Molecular structure of 1,1′, 2,2′,3,4,4′,5′-tetrakis(trimethylene)ferrocene with the atoms represented by their 50% probability ellipsoids for thermal motion.

Unit cell parameters and the orientation matrix were determined on a CAD-4 diffractometer using Nb-filtered Mo-K_α radiation. Twenty-five reflections whose θ values ranged from 13.9 to 24.1° were automatically centered and used in the least-squares refinement of lattice parameters and the orientation matrix. Unit cell parameters obtained are shown in Table 1.

(Continued on p. 104)

TABLE 1

CRYSTALLOGRAPHIC DATA

Molecular formula	$C_{22}H_{26}Fe$
Molecular weight	346.297
Space group	$P2_1/c$
a	8.649(4) Å
b	21.623(2) Å
c	9.120(1) Å
β	107.21(3)°
V	162 Å^3
Z	4
Calculated density	1.412 g cm^{-3}
Crystal dimensions (nm)	$d(100) = 0.346$, $d(\bar{1}00) = 0.346$
	$d(010) = 0.131$, $d(0\bar{1}0) = 0.123$
	$d(0.\overline{11}) = 0.160$, $d(0\bar{1}1) = 0.197$
	$d(\overline{011}) = 0.262$, $d(011) = 0.209$
Data collection	
two theta range	$0 < 2\theta \leqslant 60°$
number of reflections	10972 (hemisphere)
	3768 (unique and $F_o^2 \geqslant 3\sigma_c(F_o^2)$)
Data reduction and refinement	
scattering factors	Doyle and Turner [9],
	Stewart, Davidson and Simpson [11]
anomalous dispersion	Cromer and Liberman [10]
linear abs. coeff.	9.48 cm^{-1}

TABLE 2

FINAL ATOMIC POSITIONAL AND THERMAL PARAMETERS[a]

	x	y	z	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Fe	0.13214(3)	0.12565(1)	0.25398(3)	0.00620(4)	0.00096(1)	0.00588(4)	−0.00005(1)	0.00151(3)	0.00003(1)
C(1)	0.2751(3)	0.1976(1)	0.2489(3)	0.0085(3)	0.0012(1)	0.0098(3)	−0.0007(1)	0.0014(2)	0.0002(1)
C(2)	0.2318(2)	0.1654(1)	0.1035(2)	0.0083(3)	0.0013(1)	0.0084(3)	−0.0002(1)	0.0031(2)	0.0006(1)
C(3)	0.0589(2)	0.1673(1)	0.0457(2)	0.0084(3)	0.0013(1)	0.0071(3)	0.0000(1)	0.0018(2)	0.0006(1)
C(4)	−0.0030(2)	0.1999(1)	0.1511(2)	0.0085(3)	0.0011(1)	0.0090(3)	0.0004(1)	0.0017(2)	0.0002(1)
C(5)	0.1280(3)	0.2192(1)	0.2768(2)	0.0114(4)	0.0011(1)	0.0097(3)	0.0000(1)	0.0018(3)	−0.0006(1)
C(1′)	0.2871(2)	0.0775(1)	0.4236(2)	0.0080(3)	0.0014(1)	0.0081(3)	0.0001(1)	0.0009(2)	0.0009(1)
C(2′)	0.2488(3)	0.0448(1)	0.2786(2)	0.0091(3)	0.0011(1)	0.0097(3)	0.0005(1)	0.0030(3)	0.0004(1)
C(3′)	0.0769(3)	0.0343(1)	0.2276(2)	0.0103(3)	0.0011(1)	0.0087(3)	−0.0004(1)	0.0027(3)	−0.0001(1)
C(4′)	0.0103(2)	0.0603(1)	0.3389(2)	0.0081(3)	0.0013(1)	0.0078(3)	−0.0004(1)	0.0022(2)	0.0004(1)
C(5′)	0.1385(3)	0.0864(1)	0.4579(2)	0.0097(3)	0.0014(1)	0.0062(2)	0.0000(1)	0.0019(2)	0.0005(1)
C(6)	0.4411(3)	0.2037(1)	0.3617(3)	0.0088(4)	0.0019(1)	0.0132(4)	−0.0014(1)	0.0006(3)	−0.0001(1)
C(7)	0.4588(3)	0.1735(1)	0.5185(3)	0.0095(4)	0.0025(1)	0.0104(4)	−0.0014(1)	−0.0011(3)	−0.0006(1)
C(8)	0.4490(3)	0.1034(1)	0.5144(3)	0.0083(4)	0.0024(1)	0.0109(4)	0.0003(1)	−0.0010(3)	0.0008(1)
C(9)	0.3433(3)	0.1307(1)	0.0340(3)	0.0099(4)	0.0023(1)	0.0110(3)	0.0000(1)	0.0054(3)	0.0005(1)
C(10)	0.3215(3)	0.0606(1)	0.0285(3)	0.0120(4)	0.0023(1)	0.0122(4)	0.0009(1)	0.0063(3)	−0.0006(1)
C(11)	0.3611(3)	0.0290(1)	0.1859(3)	0.0114(4)	0.0017(1)	0.0145(4)	0.0014(1)	0.0054(3)	0.0001(1)
C(12)	−0.0766(3)	0.1474(1)	−0.0921(2)	0.0111(4)	0.0019(1)	0.0070(3)	−0.0001(1)	0.0010(3)	0.0004(1)
C(13)	−0.2254(3)	0.1534(1)	−0.0319(3)	0.0084(4)	0.0024(1)	0.0105(3)	0.0000(1)	0.0001(3)	0.0000(1)
C(14)	−0.1844(3)	0.2040(1)	0.0931(3)	0.0091(4)	0.0018(1)	0.0121(4)	0.0013(1)	0.0021(3)	0.0003(1)
C(15)	−0.1488(3)	0.0675(1)	0.3714(3)	0.0091(4)	0.0021(1)	0.0102(3)	−0.0008(1)	0.0040(3)	0.0006(1)
C(16)	−0.1082(3)	0.1168(2)	0.5001(3)	0.0122(4)	0.0036(1)	0.0104(4)	0.0004(2)	0.0052(3)	−0.0010(1)
C(17)	0.0749(3)	0.1124(1)	0.5818(3)	0.0126(4)	0.0028(1)	0.0069(3)	−0.0002(2)	0.0032(3)	−0.0002(1)

	x	y	z	B
H(5) [b]	0.126(4)	0.241(1)	0.370(3)	4.2(6)
H(3′)	0.019(3)	0.014(1)	0.128(3)	3.1(5)
H(5A)	0.466(3)	0.248(1)	0.375(3)	4.0(6)
H(6B)	0.524(3)	0.186(1)	0.318(3)	4.4(6)
H(7A)	0.373(3)	0.190(1)	0.562(3)	2.6(5)
H(7B)	0.559(4)	0.188(2)	0.591(3)	5.2(7)
H(8A)	0.477(4)	0.085(2)	0.616(3)	5.0(7)
H(8B)	0.526(4)	0.082(2)	0.465(4)	4.4(7)
H(9A)	0.326(4)	0.147(1)	−0.069(3)	4.6(7)
H(9B)	0.459(3)	0.138(1)	0.091(3)	3.3(6)
H(10A)	0.392(4)	0.040(1)	−0.027(4)	4.9(7)
H(10B)	0.207(3)	0.049(1)	−0.032(3)	3.7(6)
H(11A)	0.463(4)	0.042(1)	0.243(3)	4.4(7)
H(11B)	0.353(3)	−0.017(1)	0.175(3)	4.0(6)
H(12A)	−0.054(3)	0.105(1)	−0.123(3)	3.1(6)
H(12B)	−0.069(3)	0.174(1)	−0.177(3)	4.0(7)
H(13A)	−0.317(4)	0.162(1)	−0.110(3)	4.5(7)
H(13B)	−0.240(3)	0.117(2)	0.020(3)	3.4(7)
H(14A)	−0.220(3)	0.243(1)	0.051(3)	3.5(6)
H(14B)	−0.244(3)	0.199(1)	0.160(3)	3.3(7)
H(15A)	−0.182(4)	0.030(2)	0.398(4)	4.5(7)
H(15B)	−0.240(3)	0.080(1)	0.278(3)	3.1(6)
H(16A) [c]	−0.165	0.110	0.568	6.0
H(16B) [c]	−0.132	0.156	0.458	6.0
H(17A)	0.098(3)	0.085(1)	0.669(3)	3.3(5)
H(17B)	0.117(3)	0.153(1)	0.616(3)	3.9(6)

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[a] The form of the thermal ellipsoid expression is $\exp[-\beta_{11}h^2 - \beta_{22}k^2 - \beta_{33}l^2 - 2\beta_{12}hk - 2\beta_{13}hl - 2\beta_{23}kl]$. [b] The hydrogen atom numbering scheme corresponds to attached carbon; HXA indicates one of two hydrogen atoms bonded to CX. [c] The parameters of H(16A) and H(16B) were not refined.

Intensity data were collected using θ—2θ scans with X-ray source and filter settings identical to those used for determination of the unit cell parameters. The intensity data were reduced [7] using the usual Lorentz and polarization terms, and the absorption corrections were carried out by means of the gaussian grid method. Computations were performed using standard programs modified locally [7,8]. The agreement factors were defined in the usual way [4] with:

$$w^{-1} = (\sigma_c^2(F_0^2) + (0.03F_0^2)^2 + (0.01(F_0^2 - TF_0^2))^2)/4F_0^2$$

where $\sigma_c^2(F_0^2)$ is the variance from counting statistics and T is a transmission factor for absorption.

The atomic coordinates for the iron atom were deduced from a three-dimensional Patterson synthesis and refined to give the initial residual, $R = 0.415$. The remaining atoms were located by means of difference Fourier calculations and least-squares refinements. A refinement using isotropic temperature factors for all non-hydrogen atoms converged with $R = 0.079$. After two cycles with anisotropic temperature factors for all non-hydrogen atoms, the structure refined to $R = 0.062$ and $R_w = 0.090$. The 26 hydrogen atoms were located as the principal features on a difference Fourier map. In subsequent steps the hydrogen coordinates and isotropic thermal parameters were refined as a separate block. The parameters of two hydrogen atoms bonded to C(16) did not refine well and were fixed at calculated positions with isotropic thermal parameters of 6.0. The positions of all atoms were then iteratively refined with the thermal parameters as a second block. The refinment including all atoms converged to the final values of $R = 0.035$ and $R_w = 0.048$. The total number of variables was 304 and the maximum parameter shift was less than 0.1 σ. The major feature on a final difference Fourier map was a peak of 0.41 e Å^{-3}; this compares to values of approximately 4.5 and 0.5 e Å^{-3} in electron density for averaged carbon and hydrogen atoms, respectively, in this structure. Final atomic parameters are listed in Table 2; a tabulation of the final observed and calculated structure factors was deposited with NAPS. *

Results and discussions

The molecular geometry omitting all hydrogen atoms is illustrated with the atom numbering scheme in Fig. 1. Bond distances and angles with their estimated standard deviations included in parentheses are collected in Table 3.

The molecule consists of two tilted, eclipsed cyclopentadienyl rings bridged by by two trimethylene chains, and two non-eclipsed homoannular trimethylene chains. The equations of the least squares planes of the cyclopentadienyl rings and the deviations of atoms from these planes are given in Table 4. There appears to be no deformation from planarity in the cyclopentadienyl rings. The angle between the least squares planes of the cyclopentadienyl rings is 11.1°. This

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* The table of structure factors has been deposited as NAPS Document No. 03265 (23 pages). Order from ASIS/NAPS, c/o Microfiche Publications, P.O. Box 3513, Grand Central Station, New York, N.Y. 10017. A copy may be secured by citing the document number, remitting $ 5.00 for photocopies or $3.00 for microfiche. Advance payment is required. Make checks payable to Microfiche Publications.

TABLE 3

INTERATOMIC BOND DISTANCES AND ANGLES

(a) BOND DISTANCES

Atoms	Distances (Å)	Atoms	Distances (Å)	Atoms	Distances (Å)
Fe—C1	1.997(2)	C5—C1	1.446(3)	C5—H5	0.97(3)
Fe—C2	2.016(2)	C1′—C2′	1.450(3)	C3′—H3′	1.00(2)
Fe—C3	2.027(2)	C2′—C3′	1.438(3)	C6—H6A	0.97(3)
Fe—C4	2.044(2)	C3′—C4′	1.422(3)	C6—H6B	1.00(3)
Fe—C5	2.034(2)	C4′—C5′	1.420(3)	C7—H7A	0.99(2)
Fe—C1′	2.011(2)	C5′—C1′	1.423(3)	C7—H7B	0.97(3)
Fe—C2′	1.997(2)	C1—C6	1.504(3)	C8—H8A	0.97(3)
Fe—C3′	2.030(2)	C6—C7	1.537(4)	C8—H8B	1.02(3)
Fe—C4′	2.047(2)	C7—C8	1.520(4)	C9—H9A	0.96(3)
Fe—C5′	2.031(2)	C8—C1′	1.507(4)	C9—H9B	0.99(3)
Fe—C6	3.066(3)	C2—C9	1.502(4)	C10—H10A	1.00(3)
Fe—C7	3.292(3)	C9—C10	1.527(4)	C10—H10B	1.01(3)
Fe—C8	3.087(3)	C10—C11	1.534(4)	C11—H11A	0.92(3)
Fe—C9	3.089(2)	C11—C2′	1.504(3)	C11—H11B	0.99(3)
Fe—C10	3.298(2)	C3—C12	1.505(3)	C12—H12A	0.99(3)
Fe—C11	3.066(2)	C12—C13	1.547(4)	C12—H12B	0.98(3)
Fe—C12	3.174(2)	C13—C14	1.544(4)	C13—H13A	0.92(3)
Fe—C13	3.457(3)	C14—C4	1.502(3)	C13—H13B	0.95(3)
Fe—C14	3.185(3)	C4′—C15	1.499(3)	C14—H14A	0.93(3)
Fe—C15	3.189(3)	C15—C16	1.547(4)	C14—H14B	0.91(3)
Fe—C16	3.484(2)	C16—C17	1.540(4)	C15—H15A	1.01(4)
Fe—C17	3.183(3)	C17—C5′	1.504(3)	C15—H15B	1.07(3)
C1—C2	1.447(3)	C1—C1′	3.032(3)	C16—H16A	0.91
C2—C3	1.432(3)	C2—C2′	3.038(3)	C16—H16B	0.93
C3—C4	1.419(3)	C3—C3′	3.301(3)	C17—H17A	0.97(3)
C4—C5	1.415(3)	C4—C4′	3.457(3)	C17—H17B	0.96(3)
		C5—C5′	3.300(3)		

(b) BOND ANGLES

Atoms	Angles(°)	Atoms	Angles/(°)
C(1)—C(2)—C(3)	106.4(2)	C(2)—C(3)—C(12)	140.3(2)
C(2)—C(3)—C(4)	109.1(2)	C(4)—C(3)—C(12)	110.6(2)
C(3)—C(4)—C(5)	108.8(2)	C(3)—C(4)—C(14)	110.7(2)
C(4)—C(5)—C(1)	107.4(2)	C(5)—C(4)—C(14)	140.4(2)
C(5)—C(1)—C(2)	108.3(2)	C(3′)—C(4′)—C(15)	140.5(2)
C(1′)—C(2′)—C(3′)	107.9(2)	C(5′)—C(4)—C(15)	111.2(2)
C(2′)—C(3′)—C(4′)	107.8(2)	C(1′)—C(5′)—C(17)	140.4(2)
C(3′)—C(4′)—C(5′)	108.3(2)	C(4′)—C(5′)—C(17)	110.3(2)
C(4′)—C(5′)—C(1′)	109.3(2)	C(1)—C(6)—C(7)	114.3(2)
C(5′)—C(1′)—C(2′)	106.8(2)	C(5)—C(7)—C(8)	114.2(2)
C(2)—C(1)—C(6)	127.2(2)	C(1′)—C(8)—C(7)	114.8(2)
C(5)—C(1)—C(6)	124.3(2)	C(2)—C(9)—C(10)	114.9(2)
C(1)—C(2)—C(9)	127.1(2)	C(9)—C(10)—C(11)	114.9(2)
C(3)—C(2)—C(9)	126.2(2)	C(2′)—C(11)—C(10)	115.0(2)
C(2′)—C(1′)—C(8)	127.3(2)	C(3)—C(12)—C(13)	101.7(2)
C(5′)—C(1′)—C(8)	125.6(2)	C(12)—C(13)—C(14)	106.8(2)
C(1′)—C(2′)—C(11)	127.8(2)	C(4)—C(14)—C(13)	101.9(2)
C(3′)—C(2′)—C(11)	124.1(2)	C(4′)—C(15)—C(16)	101.9(2)
		C(15)—C(16)—C(17)	107.1(2)
		C(5′)—C(17)—C(16)	102.2(2)

value is almost the same as the values given for ferrocene derivatives with one three-carbon bridge: α-keto-1,1′-trimethyleneferrocene [12] (8.8°), α-keto-γ-phenyl-1,1′-trimethyleneferrocene [13] (10°), 2-(α-phenyl-α-hydroxypropyl)-

TABLE 4

LEAST SQUARES PLANES OF CYCLOPENTADIENYL RINGS

Equation[a] $pX + qY + rZ = s$

Coefficient	Ring	
	I	II
p	0.207	0.015
q	0.859	0.872
r	−0.473	−0.488
s	2.992	−0.320

DEVIATIONS OF ATOMS (Å)[b]

	Ring	
	I	II
C(1) or C(1′)	−0.003	−0.000
C(2) or C(2′)	0.002	0.001
C(3) or C(3′)	−0.001	−0.002
C(4) or C(4′)	−0.001	0.002
C(5) or C(5′)	0.002	−0.001
C(12) or C(15)	0.032	−0.023
C(13) or C(16)	−0.404	0.359
C(14) or C(17)	0.022	−0.051

[a] Direction cosines of the plane refer to the orthogonal axis system a, b, c^*. [b] In Ring I, negative deviations are toward the iron atom, and in Ring II, positive deviations are toward the iron atom.

1,1′-trimethyleneferrocene [14] (10°) and 1,1′-(1″,3″-cyclopentylene)ferrocene [15] (11.0°). The point where the perpendicular from the iron to the least squares plane of the cyclopentadienyl ring passes through the plane is shifted about 0.04 Å from the center of gravity of the ring towards the carbons with the bridges. In tetramethylethyleneferrocene [16], a bridged ferrocene with one

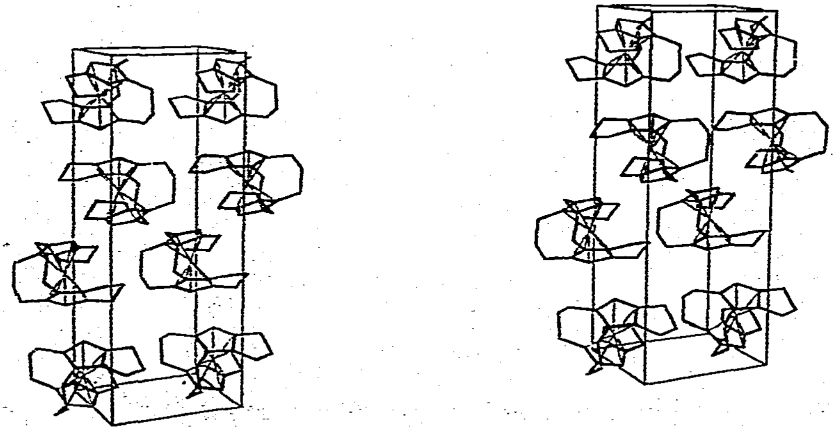

Fig. 2. Stereoscopic drawing showing the packing of 1,1′,2,2′,3,4,4′5′-tetrakis(trimethylene)ferrocene. The front face is the ab plane. The origin is the lower right hand corner of the front face.

TABLE 5

SELECTED SHORT INTERMOLECULAR DISTANCES

From atom at x, y, z	To atom at	Distance
C(5)	C(3) (x, 0.5 — y, 0.5 + y)	3.643(3)
C(12)	C(17) (x, y, z — 1)	3.666(3)
H(5)	C(3) (x, 0.5 — y, 0.5 + y)	2.73(3)
H(5)	C(2) (x, 0.5 — y, 0.5 + y)	2.89(3)
C(5)	H(12B) (x, 0.5 — y, 0.5 + y)	2.97(3)
H(3')	H(10B) (—x, —y, —z)	2.33(4)
H(3')	H(3') (—x, —y, —z)	2.34(5)
H(5)	H(12B) (x, 0.5 — y, 0.5 + y)	2.45(4)

of the largest measured ring tilt, this shift was calculated as 0.09 Å.

The iron to ring-carbon distances (1.997(2) to 2.047(1) Å, mean 2.023 Å) and the iron to ring distance (1.616 Å) are shorter than those measured for ferrocene derivatives with one three-carbon bridge, and about the same as in the dicyclopentyleneferrocene derivatives [17] but significantly longer than those of 1,1',2,2',4,4'-tris(trimethylene)ferrocene (IV) [4].

The geometry of the 1,1',2,2'-trimethylene chains are similar to those in 1,1',2,2',4,4'-tris(trimethylene)ferrocene. Although there exist alternative conformations for the trimethylene bridges, the conformation found seems to be the energetically most stable with the middle carbons of the 1,1',2,2'-bridges pointed away from each other. The middle carbon atoms of the homoannular trimethylene links are about 0.4 Å towards the iron atom from the least squares planes of the cyclopentadienyl rings. Off hand, it seems strange that this is so, and one would expect that these methylene groups would be oriented in the opposite direction, but models show that there are fewer repulsive interactions in the observed orientation. The average distance between the middle carbon atom of the 1,1'-bridge and the 2,2'-bridge and an adjacent carbon atom of 1.530(4) Å is slightly shorter than the corresponding distance of 1.545(2) Å in the homoannular chain. The distances between a cyclopentadienyl and the methylene carbon atom adjacent to it, 1.503(1) Å are quite similar in the two kinds of trimethylene chains.

The molecular packing omitting all hydrogen atoms is illustrated in Fig. 2. None of the intermolecular distances is significantly shorter than the sum of Van der Waals radii. The shortest intermolecular distances of carbon—carbon, carbon—hydrogen, and hydrogen—hydrogen are given in Table 5.

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