Crystal and Molecular Structure of Tricarbonyl-π-[1,1,1-tricarbonyl-2,3-dimethoxy-5-(diphenylmethyl)ferracyclopentadiene]iron(*Fe–Fe*), a Product from the Reaction between Diphenyldiazomethane and Tricarbonyl-π-[1,1,1-tricarbonyl-2,5-dimethoxyferracyclopentadiene]iron(*Fe–Fe*)

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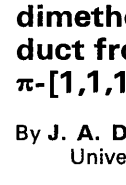
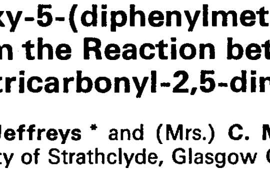
The crystal and molecular structure of the title compound (2) have been determined by *X*-ray diffraction methods from photographic data. Crystals are monoclinic prisms, space group $P2_1/c$, with $Z = 4$ in a cell with a = 14·75, b = 8·95, c = 19·58 Å, β = 108·2°. The structure was solved by Patterson and Fourier methods and refined by least-squares techniques to R 0·125 for 1547 reflections. Both iron atoms have distorted octahedral co-ordination. The Fe–C–O systems are all slightly bent. The three C–C bond lengths in the five-membered ring are all 1·40 ± 0·01 Å, the iron atom lying 0·18 Å out of the plane through the other four atoms on the side away from the other iron atom. Fe–Fe is 2·54 Å.

IRRADIATION of a mixture of diphenyldiazomethane and tricarbonyl-π-[1,1,1-tricarbonyl-2,5-dimethoxyferracyclopentadiene]iron (1a) produces a complex mixture of compounds. The crystal structure determination of one of these, tricarbonyl-π-[1,1,1-tricarbonyl-2,3-dimethoxy-5-(diphenylmethyl)ferracyclopentadiene]iron, (2), is described here. The results have been summarised in an earlier publication.[1]



(1)

	R¹	R²
a;	H	Me
b;	Me	H



(2)

EXPERIMENTAL

The compound separated from light petroleum as prisms, m.p. 148 °C, elongated along b with (100) prominent.

Crystal Data.—$C_{25}H_{18}Fe_2O_8$, M = 558, Monoclinic, a = 14·75, b = 8·95, c = 19·58 Å, β = 108·2°, U = 2455 Å, D_m = 1·495 ± 0·005 (by flotation), Z = 4, D_c = 1·509, $F(000)$ = 1136. Space group $P2_1/c$ (No. 14) from systematic absences. Cu-K_α radiation, λ = 1·542 Å; μ(Cu-K_α) 105·8 cm^{-1}. Crystal dimensions, 0·1 × 0·55 × 0·3 mm.

Crystallographic Measurements.—The symmetry and cell dimensions were obtained from rotation and Weissenberg photographs about b with Cu-K_α radiation, and from precession photographs with Mo-K_α (λ = 0·7107 Å) radiation. The intensity data for the layers $h0$—$7l$ were collected as equi-inclination multiple-film Weissenberg photographs and estimated visually; accidentally absent reflections were included at one third of the locally observable minimum.[2] The intensities were corrected for time of exposure, polarisation and Lorentz factors, but not for absorption, and 1582 independent structure factors were evaluated of which 250 were unobserved.

Structure Determination.—A three-dimensional Patterson map yielded co-ordinates for the two iron atoms, and successive rounds of structure-factor calculations and Fourier syntheses revealed the remaining atom positions. Whenever structure factors were calculated with isotropic temperature parameters, the data were rescaled so that for each layer $\Sigma K|F_o| = \Sigma|F_c|$, where K is a scaling factor. Initially, the summations were based on observed F-values. Later, a program was written (see Appendix) providing automatic refinement for a difference synthesis. When all the atoms had been located, three rounds of block-diagonal least-squares refinement of co-ordinates and isotropic temperature factors (but not layer scale-factors) gave R 0·151, but the largest values of $|F_c|$ were systematically greater than the corresponding $|F_o|$ values. After three

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[1] M. M. Bagga, G. Ferguson, J. A. D. Jeffreys, Miss C. M. Mansell, P. L. Pauson, I. C. Robertson, and J. G. Sime, *Chem. Comm.*, 1970, 672.

[2] W. C. Hamilton, *Acta Cryst.*, 1955, **8**, 185.

TABLE 1

Convergence of the refinement

Least-squares round no.	R (overall)	R'
(9)	0·1253	0·0238 *
(10)	0·1246	0·0235

Mean values of $w\Delta^2$ in round (10).

Range of $\lvert F_o\rvert$	0–10	10–20	20–30	30–40	40–50	50–60	60–70	70–80	80–90	90–100
$\langle w\Delta^2\rangle$	16·43	15·83	16·61	20·82	24·17	14·50	22·43	15·05	9·13	13·16
No. of values	291	363	338	230	127	97	57	27	13	4

Final structure-factor calculation with output from round (10)

Layer k =	0	1	2	3	4	5	6	7	Overall
R	0·120	0·120	0·124	0·122	0·110	0·120	0·149	0·192	0·125
No. of data	166	223	223	244	233	183	183	161	1547

* $R' = \Sigma w\Delta^2/\Sigma w(aF_o{}^2)$.

further such rounds, using a correction for the real part of the anomalous dispersion for iron, and with anisotropic temperature factors for these atoms, R was 0·134; however, the discrepancy between $\lvert F_o\rvert$ and $\lvert F_c\rvert$ persisted. This was assumed to be due to secondary extinction, and all reflections with $\lvert F_c\rvert > 100{\cdot}0$ were removed from the file,[3] leaving 1547. Structure factors were recalculated using atomic co-ordinates and isotropic temperature factors from the latest least-squares round (for the iron atoms, the mean values of $U_{11} + U_{22} + U_{33}$ was used), and the layers rescaled. After four more rounds of least-squares refinement, as already described, refinement had converged at R 0·125. The weighting scheme used in the refinement was $\sqrt{w} = 1$ if $\lvert F_o\rvert \leqslant F_o{}^*$; $\sqrt{w} = F_o{}^*/\lvert F_o\rvert$ if $F_o\rvert > F_o{}^*$. The value of $F_o{}^*$ was adjusted during the refinement, and during the last four rounds $F_o{}^*$ was 40·0. In the structure-factor calculations the atomic scattering factors were taken from ref. 4. Observed and calculated structure factors are listed in Supplementary Publication No. SUP 20400 (8 pp., 1 microfiche).†

RESULTS AND DISCUSSION

Table 1 shows the final weighting scheme analysis and the convergence of the refinement. Table 2 gives the final co-ordinates of the atoms, their temperature factors, and standard deviations derived from the final least-squares matrix. Figure 1a shows one molecule projected on the *ac* plane. The numbering system used is shown in Figure 1b. Table 3 lists interatomic distances, valency angles, and some non-bonded interactions. Table 4 gives the mean values for the lengths and angles for selected types of bond, together with the means of some previously measured values, and the mean estimated standard deviations for bond lengths and angles. Our values for bond lengths and angles are similar to those already determined, and all the values for the bond lengths averaged in this table lie within 1·5σ of the appropriate mean.

The Fe(1)–Fe(2) separation, 2·54 Å, lies within the usual range for a bonded distance.[5] Formal bond angles at each iron atom are listed in Table 3. If the links between Fe(1) and the π-electrons of the diene system C(30)—(33) are considered as equivalent to bonding to two pseudo-atoms, M(1), and M(2), re-

TABLE 2

Final co-ordinates and isotropic temperature parameters with standard deviations in parentheses

Atom	$10^4x/a$	$10^4y/b$	$10^4z/c$	10^4U_{iso}
Fe(1)	2125(1)	3391(3)	282(1)	*
Fe(2)	3096(1)	1003(3)	430(1)	*
O(3)	889(9)	5624(16)	583(7)	988(46)
O(4)	2323(14)	4483(24)	−996(11)	1616(74)
O(5)	578(10)	1446(17)	−507(8)	1121(57)
O(6)	4836(10)	−274(17)	1401(8)	1059(48)
O(7)	3168(10)	290(17)	−1010(8)	1085(49)
O(8)	1920(11)	−1708(18)	233(8)	1166(54)
O(9)	4318(8)	3616(13)	273(6)	694(35)
O(10)	3923(8)	5225(14)	1209(6)	793(38)
C(11)	1376(13)	4723(21)	477(10)	707(56)
C(12)	2257(17)	4255(28)	−440(13)	1136(80)
C(13)	1216(13)	2175(23)	−221(11)	823(62)
C(14)	4132(13)	227(22)	1018(10)	764(58)
C(15)	3175(13)	593(22)	−423(10)	787(59)
C(16)	2329(13)	−583(21)	320(10)	746(58)
C(17)	3291(12)	178(20)	2478(9)	656(53)
C(18)	3771(15)	−832(24)	3059(11)	910(67)
C(19)	3305(14)	−2064(24)	3126(11)	862(63)
C(20)	2395(14)	−2411(24)	2702(11)	899(66)
C(21)	1912(14)	−1381(22)	2121(11)	825(62)
C(22)	2412(11)	−86(19)	2025(9)	599(50)
C(23)	1582(13)	2684(21)	2405(10)	746(57)
C(24)	960(15)	3694(24)	2644(11)	927(68)
C(25)	21(16)	3888(26)	2163(12)	1014(73)
C(26)	−321(16)	3202(27)	1529(13)	1088(77)
C(27)	277(13)	2189(21)	1307(10)	761(58)
C(28)	1243(12)	2024(19)	1749(9)	636(51)
C(29)	1858(12)	1003(20)	1464(9)	664(53)
C(30)	2541(11)	1870(18)	1124(8)	527(46)
C(31)	2883(12)	3254(20)	1418(9)	660(53)
C(32)	3476(12)	3853(18)	1057(9)	589(49)
C(33)	3665(12)	2911(19)	538(9)	609(50)
C(34)	4675(14)	2815(24)	−250(11)	920(67)
C(35)	3724(14)	6137(22)	1787(11)	803(62)

* An anisotropic temperature factor was used in the form: $T = \exp[-2\pi^2(U_{11}h^2a^{*2} + U_{22}k^2b^{*2} + U_{33}l^2c^{*2} + 2U_{12}hka^*b^* + 2U_{13}hla^*c^* + 2U_{23}klb^*c^*)]$.

Final values ($\times 10^4$) for the components of the tensor:

Atom	U_{11}	U_{22}	U_{33}
Fe(1)	592(19)	665(19)	580(20)
Fe(2)	566(19)	579(18)	564(20)

Atom	$2U_{12}$	$2U_{13}$	$2U_{23}$
Fe(1)	175(30)	530(31)	225(30)
Fe(2)	−32(30)	470(31)	−104(29)

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† For details see Notice to Authors No. 7 in *J. Chem. Soc.* (*A*), 1970, Issue No. 20 (items less than 10 pp. are sent as full size copies).

[3] B. G. Brandt and A. C. Skapski, *Acta Cryst.*, 1968, **A24**, 699.

[4] 'International Tables for *X*-Ray Crystallography,' vol. III, Kynoch Press, Birmingham, 1962.

[5] M. I. Bruce, *Organometallic Chem. Rev.*, *B*, 1970, **6**, 937, and references therein.

TABLE 3

Selected valency parameters

(*a*) Bond lengths (Å)

Fe(1)–Fe(2)	2·54
Fe(1)–C(11)	1·75
Fe(1)–C(12)	1·68
Fe(1)–C(13)	1·77
Fe(1)–C(30)	2·08
Fe(1)–C(31)	2·16
Fe(1)–C(32)	2·13
Fe(1)–C(33)	2·21
Fe(2)–C(14)	1·74
Fe(2)–C(15)	1·75
Fe(2)–C(16)	1·79
Fe(2)–C(30)	1·95
Fe(2)–C(33)	1·89
C(11)–O(3)	1·14
C(12)–O(4)	1·19
C(13)–O(5)	1·14
C(14)–O(6)	1·16
C(15)–O(7)	1·18
C(16)–O(8)	1·16
C(17)–C(18)	1·45
C(17)–C(22)	1·34
C(18)–C(19)	1·33
C(19)–C1(20)	1·38
C(20)–C(21)	1·46
C(21)–C(22)	1·42

C(23)–C(24)	1·46
C(23)–C(28)	1·36
C(24)–C(25)	1·42
C(25)–C(26)	1·33
C(26)–C(27)	1·42
C(27)–C(28)	1·42
C(22)–C(29)	1·51
C(28)–C(29)	1·51
C(29)–C(30)	1·57
C(30)–C(31)	1·39
C(31)–C(32)	1·39
C(32)–C(33)	1·41
C(32)–O(10)	1·38
O(10)–C(35)	1·50
C(33)–O(9)	1·38
O(9)–C(34)	1·48

M(1) M(2) are the mid-points of bonds C(30)–C(31), and C(32)–C(33), respectively

Fe(1)–M(1)	2·00
Fe(1)–M(2)	2·05

(*b*) Valency angles (°)

Around Fe(1)

Fe(2)–Fe(1)–C(11)	157
Fe(2)–Fe(1)–C(12)	106
Fe(2)–Fe(1)–C(13)	81
Fe(2)–Fe(1)–C(30)	49
Fe(2)–Fe(1)–C(31)	75
Fe(2)–Fe(1)–C(32)	73
Fe(2)–Fe(1)–C(33)	46
C(11)–Fe(1)–C(12)	97
C(11)–Fe(1)–C(13)	97
C(11)–Fe(1)–C(30)	109
C(11)–Fe(1)–C(31)	88
C(11)–Fe(1)–C(32)	103
C(11)–Fe(1)–C(33)	139
C(12)–Fe(1)–C(13)	95
C(12)–Fe(1)–C(30)	153
C(12)–Fe(1)–C(31)	137
C(12)–Fe(1)–C(32)	100
C(12)–Fe(1)–C(33)	84
C(13)–Fe(1)–C(30)	91
C(13)–Fe(1)–C(31)	127
C(13)–Fe(1)–C(32)	153
C(13)–Fe(1)–C(33)	124
C(30)–Fe(1)–C(31)	38
C(30)–Fe(1)–C(32)	65
C(30)–Fe(1)–C(33)	70
C(31)–Fe(1)–C(32)	38
C(31)–Fe(1)–C(33)	66
C(32)–Fe(1)–C(33)	38
Fe(2)–Fe(1)–M(1)	57
Fe(2)–Fe(1)–M(2)	58
C(11)–Fe(1)–M(1)	99
C(11)–Fe(1)–M(2)	122
C(12)–Fe(1)–M(1)	149
C(12)–Fe(1)–M(2)	92
C(13)–Fe(1)–M(1)	110
C(13)–Fe(1)–M(2)	140

Around Fe(2)

Fe(1)–Fe(2)–C(14)	139
Fe(1)–Fe(2)–C(15)	106
Fe(1)–Fe(2)–C(16)	110
Fe(1)–Fe(2)–C(30)	53
Fe(1)–Fe(2)–C(33)	58
C(14)–Fe(2)–C(15)	104
C(14)–Fe(2)–C(16)	99
C(14)–Fe(2)–C(30)	100
C(14)–Fe(2)–C(33)	91
C(15)–Fe(2)–C(16)	87
C(15)–Fe(2)–C(30)	156
C(15)–Fe(2)–C(33)	98
C(16)–Fe(2)–C(30)	91
C(16)–Fe(2)–C(33)	168
C(30)–Fe(2)–C(33)	80

Carbonyl groups

Fe(1)–C(11)–O(3)	178
Fe(1)–C(12)–O(4)	171
Fe(1)–C(13)–O(5)	173
Fe(2)–C(14)–O(6)	178
Fe(2)–C(15)–O(7)	176
Fe(2)–C(16)–O(8)	172

Internal angles in the benzene rings

C(17)	123
C(18)	117
C(19)	124
C(20)	118
C(21)	118
C(22)	122
C(23)	119
C(24)	116
C(25)	125
C(26)	119
C(27)	119
C(28)	122

TABLE 3 (*Continued*)

(*c*) Other angles

C(17)–C(22)–C(29)	124	C(31)–C(32)–C(33)	116
C(21)–C(22)–C(29)	116	C(32)–C(33)–Fe(2)	116
C(23)–C(28)–C(29)	122	C(31)–C(32)–O(10)	124
C(27)–C(28)–C(29)	116	C(33)–C(32)–O(10)	120
C(22)–C(29)–C(28)	112	C(32)–O(10)–C(35)	117
C(22)–C(29)–C(30)	111	C(32)–C(33)–O(9)	108
C(28)–C(29)–C(30)	113	Fe(2)–C(33)–O(9)	135
C(29)–C(30)–Fe(2)	124	C(33)–O(9)–C(34)	119
C(29)–C(30)–C(31)	117		
Fe(2)–C(30)–C(31)	118	C(19)–C(22)–C(29)	175
C(30)–C(31)–C(32)	110	C(25)–C(28)–C(29)	176

(*d*) Intermolecular contacts <3·6 Å

O(3) ··· O(8^{I})	3·02
O(3) ··· O(3^{III})	3·10
O(6) ··· O(7^{II})	3·27
C(35) ··· C(19^{I})	3·29
C(35) ··· C(20^{I})	3·31
C(19) ··· O(2^{V})	3·34
O(3) ··· C(11^{III})	3·35

C(11) ··· O(8^{I})	3·36
O(3) ··· O(5^{III})	3·37
O(4) ··· C(26^{III})	3·39
C(18) ··· O(4^{V})	3·39
O(10) ··· C(18^{VII})	3·39
O(6) ··· C(34^{II})	3·43
O(9) ··· C(34^{IV})	3·53
C(20) ··· O(4^{V})	3·55
O(3) ··· C(13^{III})	3·56
C(25) ··· C(21^{VI})	3·56

The Roman numerals as superscripts refer to the following equivalent positions:

I	$x, 1 + y, z$	V	$x, \frac{1}{2} - y, \frac{1}{2} + z$
II	$1 - x, \bar{y}, \bar{z}$	VI	$x, \frac{1}{2} + y, \frac{1}{2} - z$
III	$\bar{x}, 1 - y, \bar{z}$	VII	$1 - x, \frac{1}{2} + y, \frac{1}{2} - z$
IV	$1 - x, 1 - y, \bar{z}$		

spectively, at the mid-points of the bonds C(30)–C(31), and C(32)–C(33), then each iron atom has distorted octahedral co-ordination. A program [6] was written to compute (r,θ) values for the stereographic projection of bond vectors around a central atom, taking each vector in turn as the polar axis. Figure 2 shows stereographic projections of the bond vectors about Fe(1) and Fe(2). The pair of views was selected to show that the departures from octahedral co-ordination are similar for the two iron atoms; but, as the sites occupied by the Fe–Fe vector in the two diagrams are different, the

TABLE 4

Means of selected valency parameters. Values in parentheses are the means of previously published values [a]

Bond lengths (Å)		
C(ar)–C(ar)		1·40 (1·39)
C(sp^2)–C(sp^2)		1·40 (1·42) [b–f]
C(sp^3)–C(unsat.)		1·53 (1·51)
Fe–C(carbonyl)		1·74 (1·77) [b–d,g]
Fe(2)–C(alkyl); σ		1·92 (1·97) [b–d]
Fe(1)–C(unsat.); π		2·15 (2·11) [b,c]
C–O(carbonyl)		1·16 (1·15)
Bond angle at carbonyl carbon		175°
Mean estimated standard deviations, over all values		
Bond lengths, (Å)	Fe–C	0·020
	C–O	0·026
	C–C	0·028
Bond angles	(*a*) Fe–C–O	2·7°
	(*b*) All other types	2·4°

[a] Taken from *Chem. Soc. Special Publ.*, No. 18, 1965, unless otherwise stated. [b] Ref. 7. [c] Ref. 8*a*. [d] Ref. 8*b*. [e] Ref. 9. [f] Ref. 10. [g] Ref. 11.

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[6] Program SCXR STER 01: a listing is available from J. A. D. J.

[7] A. A. Hock and O. S. Mills, *Acta Cryst.*, 1961, **14**, 139.

[8] (*a*) G. F. Epstein and L. F. Dahl, *J. Amer. Chem. Soc.*, 1970, **92**, 493; (*b*) p. 502.

[9] O. S. Mills and G. Robinson, *Acta Cryst.*, 1963, **16**, 758.

[10] R. L. Beddoes, P. F. Lindley, and O. S. Mills, *Angew. Chem. Internat. Edn.*, 1970, **9**, 304.

[11] J. P. van Vuuren, R. J. Fletterick, J. Meinwald, and R. E. Hughes, *J. Amer. Chem. Soc.*, 1971, **93**, 4394, and references therein.

distortions of the symmetry along the Fe–Fe bond are different for the two iron atoms.

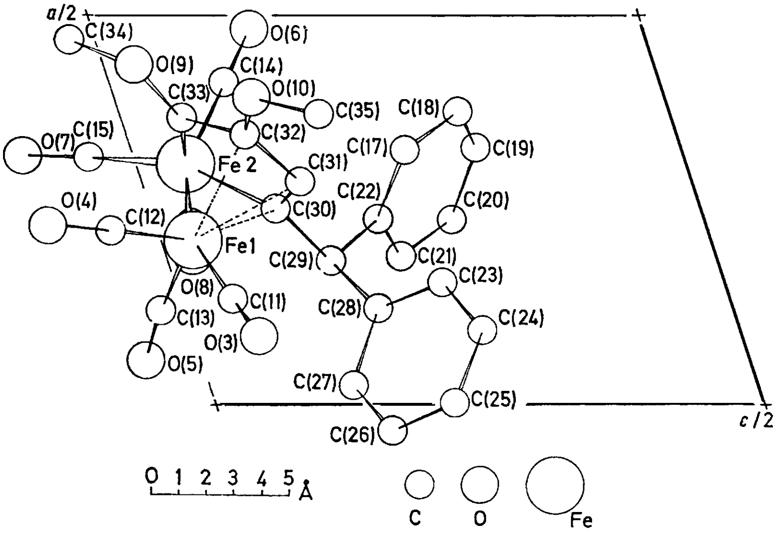

FIGURE 1a The molecule projected on the *ac* plane

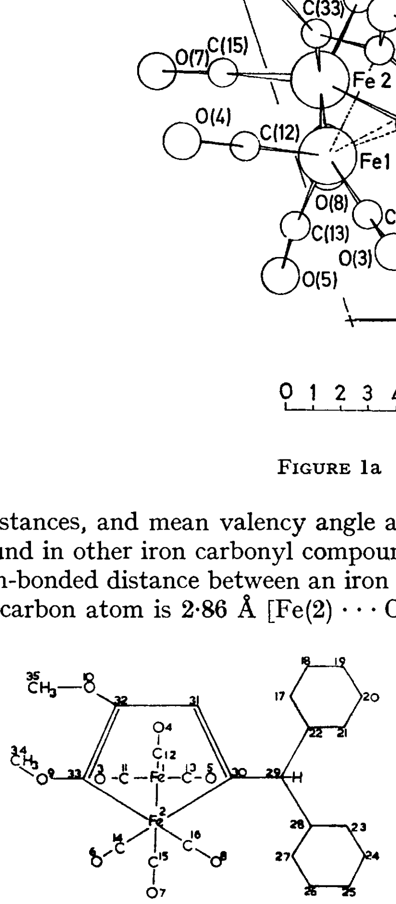

FIGURE 1b

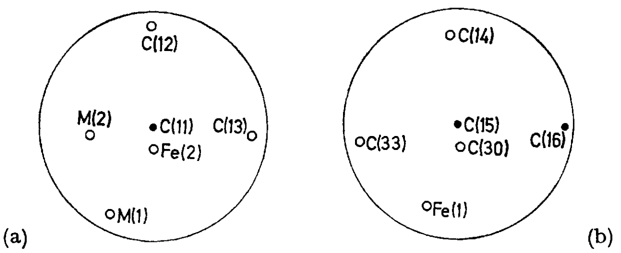

FIGURE 2 Stereographic projections of the bond vectors round (a) Fe(1), and (b) Fe(2)

The bond angles of the Fe–C–O systems are slightly less than 180° and are unexceptional. The mean Fe–C and C–O distances, and mean valency angle are similar to those found in other iron carbonyl compounds. The shortest non-bonded distance between an iron atom and a carbonyl carbon atom is 2·86 Å [Fe(2) · · · C(13)], the bond angle at the carbonyl group being 173°. These values suggest that there is no interaction between one iron atom and a carbonyl group attached to the other iron atom, unlike the case of compound (1b) [ref. 7; *cf.* ref. 8(*a*)].

The three carbon–carbon bonds of the (formal) butadiene system are nearly the same length, showing that the π electrons are delocalised. Such delocalisation has been observed in compound (1b),[7] butadiene-(tricarbonyl)iron,[9] and in more complex compounds containing the latter ligand system.[8b,9,10] The Fe–C σ bonds (mean 1·92 Å) are short; a recent publication [11] quotes a range 1·94—2·10 Å, and the mean value for related compounds (Table 4) is also longer than our value. The bond Fe(1)–C(33) (2·21 Å) is long for this type of bond, but the other three values are unexceptional.

Table 5 gives some out-of-plane distances. The atoms of the benzene ring C(17)—(22) are coplanar; those of benzene ring C(23)—(28) are nearly so, but C(29) lies out of the plane of either ring. This distortion is probably due to forces resulting from crystal packing, as ring C(17)—(22) is involved in two carbon–carbon non-bonded distances of 3·30 ± 0·01 Å. This distance is less than the sum of the van der Waals radius of a $>CH_2$ group (2·0 Å) and half the spacing distance in graphite (1·7 Å). The next closest such contact is C(25) · · · $C(21^{IV})$ (3·56 Å). The angle between the planes of the two phenyl residues is 80°. The system C(30)—(33) is nearly planar, but the adjacent atoms, except for O(10) lie outside this plane; Fe(2) lies 0·15 Å out of the plane, on the side remote from Fe(1). This situation also holds in compound (1b), in which the

TABLE 5

Out-of-plane distances (Å)

Plane (1): C(17)—(22)
[C(17) 0·00, C(18) −0·01, C(19) 0·01, C(20) 0·00, C(21) −0·01, C(22) 0·01, C(29) −0·11]
Plane (2): C(23)—(28)
[C(23) −0·01, C(24) −0·01, C(25) 0·01, C(26) 0·01, C(27) −0·03, C(28) 0·03, C(29) 0·07]
Plane (3): C(30)—(33)
[C(30) 0·01, C(31) −0·03, C(32) 0·03, C(33) −0·01, Fe(2) −0·15, C(29) −0·05, O(9) −0·06, O(10) 0·02]

atoms corresponding to C(30)—(33) are accurately coplanar, and the iron atom is 0·18 Å from this plane on the side away from the other iron atom (results calculated from data given in ref. 7); larger departures of the iron atom from the mean plane of a ferracyclopentadiene system have been observed, associated with a longer Fe–C σ bond (0·24 and 1·97,[8b] and 0·31 and 1·98 Å [8a]).

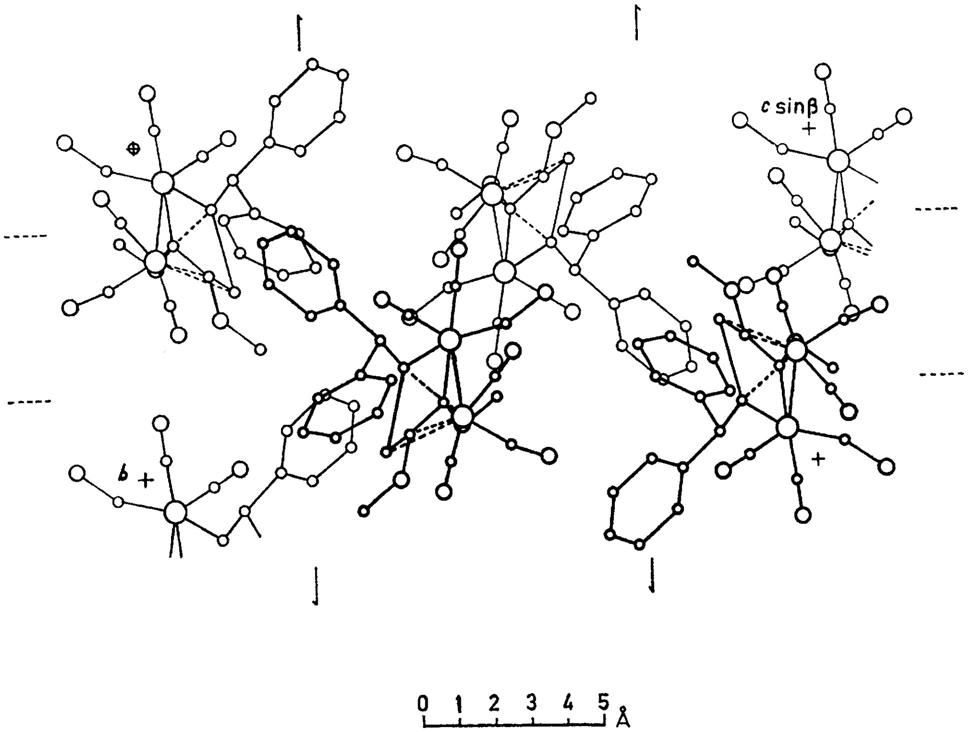

FIGURE 3 The packing of the molecules, seen down *a*. Heavy lining indicates proximity to the observer

Figure 3 shows the arrangement of the molecules seen down the *a*-axis. These are packed with the carbonyl groups of one molecule facing the carbonyl groups of neighbouring molecules. With the exception of the close approaches mentioned above, the contacts between different molecules are of the order of the sums of the van der Waals radii of the atoms concerned.

APPENDIX

Difference Fourier Synthesis with Automatic Refinement.—If x_0 is the postulated site of an atom, then the correction to the x co-ordinate is approximately proportional to (slope of electron density at $x = x_0$) over (curvature of the electron density at the centre of the atom).[12] If ρ_{-1}, ρ_0, ρ_{+1} are the electron-densities at successive grid points, with ρ_0 at the grid point nearest x_0, then adequate correction was obtained by making the correction equal to [$(\rho_{+1} - \rho_{-1})$ multiplied by grid spacing in Å/10 Z_0 multiplied by length of all cell edge in Å], where Z_0 is the atomic number of the atom concerned, and, for the temperature parameter, empirically, the correction is equal to $-\rho_0/50$. Trials of this program with the compound described in this paper have shown that when R is *ca.* 0·20, R was reduced by *ca.* 0·02 in each round of structure-factor and Fourier calculations, refinement converging at R *ca.* 0·15. Similar results were obtained with the isomeric compound described in a preliminary communication.[1]

Calculations were performed on the ICL 1905 computer at the University of Strathclyde. We thank Professor Pauson for suggesting the problem, and Mrs. C. Metters (née Tuladhar) for the drawings.

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[12] G. H. Stout and L. H. Jensen, '*X*-Ray Structure Determination,' Collier–Macmillan, London, 1968, pp. 376—381, and references therein.