of course, entirely consistent with previous studies showing metal–carbon bond contractions in other systems (such as acyls,[19] aryls,[20,21] or perfluoroaryls[20,22–24]) which are in conjugation with a metal atom.

Carbon–carbon bond lengths within the *trans*-buta-1,3-diene fragment are: C(6)–C(7) = 1.340 ± 0.007 Å, C(7)–C(7′) = 1.450 ± 0.011 Å, C(7′)–C(6′) = 1.340 ± 0.007 Å (1.36, 1.47, 1.36 Å from Davis's study[1b]). These distances are each in good agreement with the bond distances in *trans*-buta-1,3-diene itself—1.337 ± 0.005, 1.483 ± 0.010, 1.337 ± 0.005 Å, as determined from electron diffraction studies.[25]

Finally, it should be noted that possible reasons for the C_4H_4 moiety exhibiting a *single* proton magnetic resonance peak at τ 3.76 have been discussed in another publication.[1a]

The Crystal Structure

Figure 4 shows the packing of molecules in the crystal as viewed down *b*. The closest intermolecular contacts (of each type) are: hydrogen···hydrogen, 2.48 Å; oxygen···hydrogen, 2.73 Å; carbon···hydrogen, 2.83 Å; oxygen···carbon, 3.25 Å; oxygen···oxygen, 3.41 Å; and carbon···carbon, 3.50 Å.

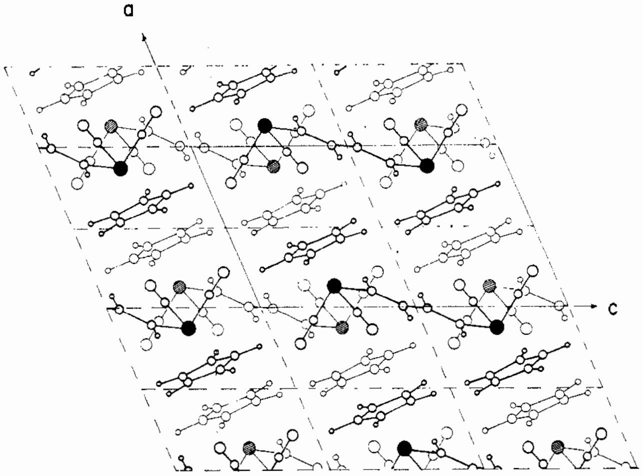

Figure 4.—Packing of molecules within the crystal, viewed down *b*.

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The Molecular Structure of Diazulenetetrairon Decacarbonyl in Crystalline $(C_{10}H_8)_2Fe_4(CO)_{10}\cdot C_2H_4Cl_2$

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The reaction of azulene with an iron carbonyl is known to give rise to azulenediiron pentacarbonyl and a compound of high molecular weight, previously formulated as $(C_{10}H_8)_2Fe_5(CO)_{13}$. It is now shown that this latter complex is actually $(C_{10}H_8)_2Fe_4(CO)_{10}$ and that it crystallizes from a 1,2-dichloroethane–hexane mixture as $(C_{10}H_8)_2Fe_4(CO)_{10}\cdot C_2H_4Cl_2$. Unit cell data are: $a = 17.296 \pm 0.012$ Å, $b = 15.541 \pm 0.011$ Å, $c = 12.915 \pm 0.009$ Å, $\beta = 114.53 \pm 0.02°$, $Z = 4$, space group C2/c (C_{2h}^6; no. 15). Observed and calculated densities are 1.818 ± 0.015 and 1.805 g cm^{-3}, respectively. A single-crystal X-ray diffraction study has been completed using data to $\sin\theta = 0.40$ (Mo Kα radiation) measured with a scintillation counter. All nonhydrogen atoms have been located, the final discrepancy index being $R_F = 6.22\%$ for the 2040 independent, nonzero reflections. The crystal is composed of discrete molecular units of $(C_{10}H_8)_2Fe_4(CO)_{10}$ along with 1,2-dichloroethane of crystallization (which is disordered). The polynuclear azuleneiron–carbonyl complex has crystallographically imposed C_2 symmetry and is composed of a 4-*endo*,4′-*endo*-diazulene ligand whose five-membered rings are bridged by an $Fe_2(CO)_4$ moiety, while $Fe(CO)_3$ groups are linked to the 1,3-diene systems remaining in each of the seven-membered rings.

Introduction

The present paper is the sixth in our series on transition metal complexes of azulene and follows detailed reports on the crystal structures of $C_{10}H_8Fe_2(CO)_5$,[2] $[C_{10}H_8Mo(CO)_3CH_3]_2$,[3] $(i\text{-}C_3H_7)(CH_3)_2C_{10}H_5Mo_2(CO)_6$,[4] $C_{10}H_8Mn_2(CO)_6$,[5] and $(C_{10}H_8)_2Fe$.[6]

The reaction of azulene with an iron carbonyl (*i.e.*,

[1] Research Fellow of the Alfred P. Sloan Foundation, 1968–1970.

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$Fe(CO)_5$,[7,8] $Fe_2(CO)_9$,[9] or $Fe_3(CO)_{12}$[7,8]) has been shown to yield azulenediiron pentacarbonyl and a compound of high molecular weight which was tentatively formulated as $(C_{10}H_8)_2Fe_5(CO)_{13}$. We have investigated the crystal structure of this latter complex in the expectation that it might provide a further example of the "ligand-to-cluster" bonding discovered in 4,6,8-trimethylazulenetetraruthenium enneacarbonyl.[10,11] It is found however (*vide infra*) that the polynuclear iron complex does not contain a cluster[12] of iron atoms and that its correct formulation is $(C_{10}H_8)_2Fe_4(CO)_{10}$. An account of this work at an intermediate stage of refinement has appeared previously.[13]

Experimental Section

The complex was prepared from 5.05 g of $Fe(CO)_5$ and 0.260 g of azulene, following the procedure of Burton, Pratt, and Wilkinson.[8] The slow cooling of a solution of the complex in a 60:40 1,2-dichloroethane–hexane mixture yielded beautiful multifaceted deep brown crystals, which were used in the subsequent crystallographic analysis. A mass spectrometric study of the complex showed the parent-ion peak at m/e 760. The accidental mass relationship $^{56}Fe \equiv 2(^{12}C^{16}O)$ prevented the unequivocal chemical identification of the molecule but led to the partial formula $(C_{10}H_8)_2Fe_n(CO)_{18-2n}$ (where n is probably 3, 4, or 5).

Unit Cell and Space Group

Optical examination and the observed reciprocal lattice symmetry of C_{2h} (2/m) indicated that the crystals belonged to the monoclinic system. A careful survey of 0*kl*, 1*kl*, 2*kl*, *h*0*l*, *h*1*l*, *h*2*l*, *hk*0, *hk*1, and *hk*2 precession photographs (all taken with Mo Kα radiation) revealed the systematic absences *hkl* for $h + k = 2n + 1$ and *h*0*l* for $l = 2n + 1$, compatible with space groups Cc (no. 9) or C2/c (no. 15).

Unit cell dimensions, obtained by a least-squares analysis of calibrated (a_{NaCl} = 5.640 Å) precession photographs taken with Mo Kα radiation (λ 0.7107 Å) at 23 ± 2°, are $a = 17.296 \pm 0.012$ Å, $b = 15.541 \pm 0.011$ Å, $c = 12.915 \pm 0.009$ Å, and $\beta = 114.53 \pm 0.02°$. The unit cell volume is 3160 Å^3. The measured density ($\rho_{obsd} = 1.818 \pm 0.015$ g cm^{-3}, by flotation in aqueous zinc iodide solution) was not in good agreement with that calculated for $(C_{10}H_8)_2Fe_n(CO)_{18-2n}$ ($\rho = 1.596$ g cm^{-3} for $M = 760$, $Z = 4$) but was found, at a later date, to be in excellent agreement with the density calculated for four units of $(C_{10}H_8)_2Fe_4(CO)_{10} \cdot C_2H_4Cl_2$ per unit cell ($\rho_{calcd} = 1.805$ g cm^{-3}). If the space group is the noncentrosymmetric Cc, there are no symmetry restrictions either on the iron complex or on the solvent molecule, but if the space group is the centrosymmetric C2/c, each species is required (in the absence of disorder) to possess either a twofold rotation axis or a center of symmetry. The successful refinement of the crystallographic analysis (*vide infra*) shows the true space group to be C2/c (C_{2h}^6).

Collection and Reduction of X-Ray Diffraction Data

Two multifaceted crystals, each of which was close to spherical in shape, were used in the course of data collection. Crystal A was mounted along its 100 axis, while crystal B was mounted on its 010 axis.

Intensity data were collected using zirconium-filtered molybdenum radiation (Mo Kα, $\bar{\lambda}$ 0.7107 Å), a 0.01°-incrementing Buerger automated diffractometer, and the customary "stationary-background, ω-scan, stationary-background" counting sequence. A careful examination of Weissenberg photographs led to the choice of scan angle as $\omega = [1.5 + (0.8/L)]°$, where $1/L$ is the Lorentz factor; initial and final backgrounds (B_1, B_2) were counted for one-fourth the time taken for the associated ω scan, and $I(hkl)$ (the intensity of the reflection *hkl*) was calculated as $I(hkl) = C(hkl) - 2[B_1(hkl) + B_2(hkl)]$, where $C(hkl)$ is the count recorded during the ω scan. Standard deviations assigned to reflections were: $I(hkl) > 1225$, $\sigma\{I(hkl)\} = 0.1[I(hkl)]$; $I(hkl) \leqslant 1225$, $\sigma\{I(hkl)\} = 3.5[I(hkl)]^{1/2}$. [Experience has shown that this proportional weighting scheme is more reasonable than simple counting statistics, probably as a result of the errors intrinsic in the ω-scan technique when using filtered, rather than monochromated, radiation.]

Reflections were rejected from the subsequent analysis on two bases: (1) if the backgrounds were grossly asymmetric—*i.e.*, if B_1/B_2 or $B_2/B_1 \geqslant 3.0$—since this is usually symptomatic of overlap of adjacent reflections; (2) if the intensities were not significantly different from zero—*i.e.*, if $I(hkl) < 3[C(hkl) + B_1(hkl) + B_2(hkl)]^{1/2}$. All other experimental precautions were identical with those described in detail in a previous publication.[14]

A total of 2560 reflections in the quadrants *Hkl* and *Hkl* (H = 0–17) were collected from crystal A. [This represents a single set of data to $\sin\theta = 0.40$, complete save for the few reflections with $\theta \leqslant 4°$ which are shielded from the counter by a lead backstop.] A total of 1153 reflections in the quadrants *hKl* and *hKl* (K = 0–5) was collected from crystal B in order to correlate the a-axis data. All data were corrected for Lorentz and polarization effects, and absorption corrections were applied.[15] [With $\mu = 21.3$ cm^{-1}, transmission coefficients varied from 0.727 to 0.756 for crystal A (volume 0.0113 mm^3) and from 0.633 to 0.802 for crystal B (volume 0.0097 mm^3).[16]] The 24 zones of data were merged to a common scale[17] using a least-squares procedure which minimizes a sum of residuals linear in

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(12) The term "cluster" is here used to define a situation where three or more metal atoms are mutually linked.

(13) M. R. Churchill and P. H. Bird, *J. Am. Chem. Soc.*, **90**, 3241 (1968).

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(15) Using a locally modified version of GNABS—a general absorption program for the IBM 7094, by C. W. Burnham.

(16) These values are for the correct formulation of the crystals as $(C_{10}H_8)_2Fe_4(CO)_{10} \cdot C_2H_4Cl_2$. Since the analysis was initiated under the erroneous assumption that the crystals were the nonsolvated species $(C_{10}H_8)_2Fe_4(CO)_{10}$, the originally applied absorption coefficient and subsequent interzonal scaling were incorrect. All data processing was redone at an intermediate stage in the refinement.

(17) Reflections with h and k both even and with h and k both odd were treated separately in the scaling process, since the C-centering operation results in there being no cross terms between these two groups of reflections. The two groups were initially assumed to be on the same scale; later refinement using two scale factors showed this assumption to be true.

the logarithm of the individual scale factors.[18] The resulting 2040 independent nonzero reflections were placed on an approximately absolute scale by means of a Wilson plot.[19]

Elucidation and Refinement of the Crystal Structure

The phase problem was circumvented by means of the three-dimensional Patterson synthesis,[20] $P(U, V, W)$, which had been sharpened such that the average intensity was no longer θ dependent, and which had the origin peak removed. A search of the Harker line 0, V, $^1/_2$ and the Harker section U, 0, W quickly indicated that there were only two crystallographically independent iron atoms within the unit cell. This strongly suggested that the space group was C2/c and (in conjunction with mass spectral data) indicated that the complex had the molecular formula $(C_{10}H_8)_2Fe_4(CO)_{10}$. [It should be emphasized that confirmation that the space group is truly C2/c comes from a consideration of thermal parameters and geometry for the atoms in the $(C_{10}H_8)_2Fe_4(CO)_{10}$ molecule at the end of refinement; *vide infra.*] The positions obtained for the iron atoms [Fe(1) at $x_1 = -0.008$, $y_1 = +0.162$, $z_1 = -0.158$; Fe(2) at $x_2 = -0.214$, $y_2 = +0.405$, $z_2 = -0.222$] were such that it was immediately apparent that a symmetry-related iron atom, Fe(1′) at $x = -x_1$, $y = y_1$, $z = -^1/_2 - z_1$, was only ~2.5 Å from Fe(1) and must therefore be part of the same molecule. The molecule was thus required to possess crystallographically imposed C_2 symmetry.

A three-dimensional difference-Fourier synthesis, phased only by the iron atoms ($R_F = 43.8\%$, $R_{wF^2} = 35.2\%$),[21] revealed the positions of all carbon and oxygen atoms within the $(C_{10}H_8)_2Fe_4(CO)_{10}$ molecule. A structure factor calculation based on these positions showed significantly reduced discrepancy indices ($R_F = 33.5\%$, $R_{wF^2} = 34.0\%$), but three cycles of refinement of positional and individual isotropic thermal parameters converged to the unexpectedly high values of $R_F = 20.5\%$ and $R_{wF^2} = 20.6\%$. A second difference-Fourier map revealed some extremely strong features symmetrically disposed about $-^1/_2$, 0.16, $-^1/_4$, revealing that the crystal lattice was acting as host to some solvent of crystallization. A survey of all materials to which the crystal had been exposed (*i.e.*, $Fe(CO)_5$, azulene, $C_{10}H_8Fe_2(CO)_5$, ligroin (bp 100–120°), hexane, 1,2-dichloroethane, and air) showed that the uninvited guest could only be 1,2-dichloroethane. [The difference-Fourier map so far showed only the two chlorine atoms—about 5 Å apart and related by a twofold axis.]

With chlorine atoms now included, four cycles of refinement of positional and individual thermal parameters resulted in convergence at $R_F = 12.3\%$ and $R_{wF^2} = 9.6\%$. Now that the stoichiometry of the crystals had finally been established, it became necessary to repeat a number of steps of the data-reduction sequence. The absorption coefficient was correctly recalculated and the newly treated data were rescaled.[16] Positions for all hydrogen atoms of the $(C_{10}H_8)_2Fe_4(CO)_{10}$ molecule were now calculated[22] and positional and anisotropic thermal parameters for all nonhydrogen atoms (except the carbon atoms of the 1,2-dichloroethane molecule, which had yet to be detected) were allowed to refine. Four cycles led to the lowering of the discrepancy indices to $R_F = 7.27\%$ and $R_{wF^2} = 2.22\%$.

A difference-Fourier map in the vicinity of the 1,2-dichloroethane molecule showed two symmetry-related sets of three weak (but distinct) peaks in positions where the carbon atoms were expected. This was taken as evidence that the 1,2-dichloroethane molecules within the crystal lattice were disordered. The three positions for carbon atoms were given equal weight (*i.e.*, an occupancy factor of 0.333 for each position) and were included in the refinement. Positions, occupancy factors, and isotropic thermal parameters for each "part-carbon" were allowed to refine along with positional and anisotropic thermal parameters for all other nonhydrogen atoms. Four cycles of full-matrix least-squares refinement resulted in convergence at $R_F = 6.22\%$ and $R_{wF^2} = 1.60\%$. [The average error in an observation of unit weight was 1.53, indicating a slight underestimation in the standard deviations assigned to the intensity data.] A final difference-Fourier synthesis showed no significant features, thus confirming the essential correctness of the least-squares refinement procedure.

Throughout the analysis, scattering factors for *neutral* iron, chlorine, oxygen, carbon, and hydrogen were used.[23] The values for neutral iron and chlorine were corrected to allow for dispersion ($\Delta f'(\mathrm{Fe}) = +0.4\ e^-$, $\Delta f''(\mathrm{Fe}) = +1.0\ e^-$, $\Delta f'(\mathrm{Cl}) = +0.1\ e^-$, $\Delta f''(\mathrm{Cl}) = +0.2\ e^-$). The residual minimized during the least-squares refinement procedure was $\Sigma w(|F_o|^2 - |F_c|^2)^2$ where $w = [(\mathrm{Lp})^{-1} \cdot (T^*)^{-1}\sigma\{I(hkl)\}]^{-2}$ (T^* is the transmission factor for the appropriate reflection). Observed and calculated structure factors are displayed in Table I. Atomic positions are given in Table II. Thermal parameters are collected in Table III; their associated atomic vibration ellipsoids are defined in Table IV.

The Molecular Stereochemistry

The numbering of atoms within the $(C_{10}H_8)_2Fe_4(CO)_{10}$ molecule is shown clearly in Figure 1, which depicts the molecule viewed along the b direction. Figure 2 shows the molecule viewed along a direction perpendicular to b and also shows the 68% probability envelopes for the atomic vibration ellipsoids. Intramolecular distances and their estimated standard deviations (esd's) are collected in Table V; bond angles (with esd's) are shown in Table VI.

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(19) A. J. C. Wilson, *Nature*, **150**, 152 (1942).

(20) All crystallographic computations were performed using CRYM—an integrated sequence of crystallographic routines for the IBM 7094, compiled under the direction of Professor R. E. Marsh at the California Institute of Technology.

(21) $R_F = \Sigma||F_o| - |F_c||/\Sigma|F_o|$; $R_{wF^2} = \Sigma w(|F_o|^2 - |F_c|^2)^2/\Sigma w|F_o|^4$.

(22) Hydrogen atom positions were calculated assuming $d(\mathrm{C{-}H}) = 1.080$ Å and the appropriate trigonal or tetrahedral geometry. Hydrogen atoms were included in the structure factor calculations, with $B = 4.0$ Å^2. Neither the positions nor the thermal parameters of the hydrogens were allowed to refine. The positions were, however, redefined with respect to newly refined carbon atom positions at the end of each batch of refinement.

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TABLE I

OBSERVED AND CALCULATED STRUCTURE FACTOR AMPLITUDES (IN ELECTRONS × 10.00) for $(C_{10}H_8)_2Fe_4(CO)_{10}\cdot C_2H_4Cl_2$

K	L	FO	FC
****************			
H = -19			
1	4	638	677
1	5	210	194
1	6	395	397
1	8	237	188
3	6	618	558
3	7	124	19
****************			
H = -18			
0	4	187	139
0	6	675	556
0	8	824	776
0	10	1140	1070
2	1	95	87
2	2	258	231
2	3	855	818
2	4	372	363
2	5	1089	1076
2	6	220	180
2	7	486	490
2	8	96	241
2	9	98	124
4	2	95	62
4	3	116	43
4	5	222	74
4	6	443	381
4	7	422	398
4	8	1046	965
4	9	800	817
****************			
H = -17			
1	1	140	180
1	2	196	163
1	3	826	802
1	4	363	378
1	5	551	506
1	6	124	38
1	8	182	138
1	9	536	511
1	10	281	194
1	11	690	672
3	1	349	338
3	2	265	240
3	3	262	223
3	4	826	770
3	5	207	141
3	6	209	127
3	7	350	329
3	8	435	405
3	9	654	606
3	10	697	675
3	11	429	391
5	3	520	523
5	4	321	294
5	5	678	679
5	7	229	192
5	8	278	253
5	9	676	644
5	10	672	677
****************			
H = -16			
0	2	992	892
0	4	1198	1201
0	6	363	321
0	8	482	455
0	10	193	167
0	12	196	168
2	1	653	654
2	4	171	52
2	5	142	144
2	7	871	858
2	9	1013	1006
2	10	583	590
2	11	447	441
2	12	116	64
4	1	89	42
4	2	379	354
4	3	481	482
4	4	821	807
4	5	816	820
4	6	881	886
4	7	444	426
4	9	128	69
4	10	197	131
4	11	285	276
6	1	61	37
6	2	571	577
6	3	123	141
6	4	219	195
6	5	466	467
6	6	183	173
6	7	378	364
6	8	869	825
6	9	263	270
6	10	861	842
8	1	210	194
8	3	916	919
8	4	331	351
8	5	835	829
8	7	321	325
8	8	189	201
8	9	116	145
****************			
H = -15			
1	1	187	212
1	2	125	28
1	3	854	822
1	4	239	6
1	5	350	363
1	6	353	357
1	8	752	710
1	9	219	168
1	10	887	842
1	11	180	119
1	12	415	392
1	13	538	569
3	1	153	96
3	2	227	238
3	3	402	366
3	4	365	367
3	5	824	790
3	7	532	514
3	8	112	12
3	9	462	456
3	10	790	775
3	11	458	455
3	12	377	357
5	1	69	62
5	2	110	135
5	3	483	465
5	4	928	936
5	5	720	679
5	6	306	554
5	8	522	521
5	9	642	644
5	10	172	150
5	11	442	477
5	12	506	496
7	1	324	295
7	2	245	244
7	3	174	186
7	4	106	110
7	5	299	312
7	6	107	66
7	7	579	540
7	8	87	146
7	9	895	852
7	11	755	718
9	1	216	196
9	2	444	422
9	3	161	119
9	4	737	725
9	5	91	102
9	6	470	435
9	7	259	246
9	8	206	149
9	9	470	476
11	4	89	105
11	5	291	272
11	6	414	381
****************			
H = -14			
0	2	257	296
0	4	966	887
0	8	679	605
0	10	703	678
0	12	305	261
2	2	394	386
2	3	706	690
2	4	435	445
2	5	1064	1015
2	7	358	332
2	9	472	475
2	10	377	333
2	11	570	577
2	12	246	231
2	13	121	60
4	1	756	742
4	2	714	648
4	3	100	67
4	5	466	416
4	6	558	527
4	7	81	96
4	9	384	351
4	10	477	469
4	11	507	529
4	12	193	178
4	13	189	94
6	1	194	191
6	2	617	646
6	3	131	151
6	4	1365	1393
6	5	175	165
6	6	686	647
6	7	358	172
6	8	218	200
6	10	508	524
6	12	218	209
8	1	825	809
8	2	150	174
8	3	178	116
8	4	353	345
8	5	208	164
8	6	284	259
8	7	123	128
8	8	119	170
8	9	710	671
8	10	130	185
8	11	163	204
10	1	131	93
10	2	713	709
10	3	728	725
10	4	650	661
10	5	585	576
10	6	437	473
10	7	108	65
10	8	327	364
10	9	263	237
12	3	117	112
12	4	128	135
12	5	147	158
****************			
H = -13			
1	1	487	497
1	2	143	109
1	3	167	173
1	4	967	971
1	5	331	335
1	6	1030	850
1	7	321	318
1	8	562	531
1	9	863	788
1	11	534	454
1	12	430	361
3	1	464	431
3	2	661	634
3	3	826	823
3	4	874	953
3	5	599	549
3	6	709	721
3	7	543	506
3	8	280	270
3	9	578	558
3	10	920	859
3	11	747	679
3	12	158	76
3	13	395	359
5	1	568	528
5	2	580	554
5	3	111	67
5	4	250	223
5	5	477	536
5	6	855	817
5	7	323	306
5	8	291	317
5	9	207	152
5	10	768	751
5	11	479	399
5	12	539	528
5	13	192	174
7	1	120	132
7	2	70	22
7	3	543	514
7	4	257	287
7	5	947	930
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14	5	326	348
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14	8	214	174
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16	5	473	439
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3	7	921	856
3	9	885	815
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15	7	390	379
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17	3	324	339
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8	3	1102	986
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8	6	111	97
8	7	640	569
8	8	473	303
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8	10	273	255
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10	6	843	763
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12	3	639	599
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12	9	129	74
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14	7	421	395
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16	2	723	695
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16	5	552	501
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3	12	545	567
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5	4	1880	1741
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5	6	845	854
5	7	434	371
5	8	691	684
5	9	169	126
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5	11	445	476
5	12	342	364
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7	2	952	977
7	3	1102	1030
7	4	780	746
7	5	1871	1853
7	6	331	331
7	7	1095	1064
7	8	73	119
7	10	521	503
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9	2	1544	1570
9	3	467	449
9	4	290	302
9	5	775	729
9	6	561	580
9	7	653	618
9	8	77	17
9	9	271	189
9	10	821	798
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11	2	400	358
11	3	456	444
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11	8	429	462
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13	3	561	455
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13	6	445	333
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15	6	672	700
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17	2	164	90
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2	6	590	556
2	7	908	931
2	9	183	144
2	10	220	278
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4	5	1329	1324
4	6	286	304
4	7	506	517
4	8	127	117
4	9	306	285
4	10	186	165
4	11	618	666
4	12	634	654
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6	1	1053	963
6	2	970	961
6	3	305	265
6	4	829	772
6	5	444	381
6	6	618	635
6	7	163	195
6	8	208	240
6	9	227	193
6	10	314	270
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8	1	395	370
8	2	627	681
8	3	1044	985
8	5	1415	1402
8	6	614	550
8	7	752	763
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10	5	362	359
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10	7	182	234
10	8	140	201
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12	2	86	42
12	3	680	687
12	5	620	619
12	6	522	557
12	7	194	195
12	8	160	135
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14	3	209	268
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16	4	598	571
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1	11	523	496
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3	2	1392	1312
3	3	1636	1466
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3	6	740	661
3	7	1204	1116
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3	9	772	768
3	10	679	626
3	11	150	181
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5	4	2014	1948
5	5	1004	958
5	6	1746	1707
5	7	530	492
5	8	758	672
5	9	157	146
5	10	166	141
5	11	609	588
7	0	325	301
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7	2	263	233
7	3	1228	1116
7	4	1252	1120
7	5	511	417
7	6	1436	1298
7	7	183	178
7	9	576	487
7	10	384	351
7	11	634	570
9	0	1461	1273
9	1	785	780
9	2	240	157
9	3	1286	1143
9	4	1469	1331
9	5	685	655
9	6	1384	1222
9	7	143	95
9	8	245	265
9	9	427	344
9	10	604	521
11	0	781	737
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11	2	907	836
11	3	363	286
11	4	1058	906
11	5	586	554
11	6	547	494
11	8	582	533
11	9	411	404
13	0	1058	976
13	1	185	161
13	2	606	583
13	3	568	539
13	4	717	708
13	5	890	812
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13	7	930	862
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15	4	182	128
15	5	296	222
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2	7	176	70
2	9	603	571
2	10	569	589
2	11	921	945
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4	2	972	699
4	3	119	77
4	4	594	453
4	5	684	711
4	6	622	651
4	7	746	769
4	8	641	627
4	9	256	236
4	10	98	138
4	11	297	272
6	0	654	557
6	2	738	719
6	3	158	174
6	4	903	900
6	6	347	357
6	7	387	344
6	8	166	16
6	9	523	497
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8	0	710	708
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8	2	450	479
8	4	589	569
8	5	1019	990
8	7	795	750
8	8	174	160
8	9	105	107
8	10	169	180
10	0	227	244
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10	5	302	299
10	6	511	548
10	7	174	161
10	8	360	399
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12	0	654	571
12	1	714	729
12	2	326	329
12	3	284	279
12	4	579	513
12	5	574	557
12	6	836	794
12	7	230	265
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14	1	425	421
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14	5	301	169
14	6	142	131
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16	1	544	531
16	2	175	176
16	3	154	106
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1	9	623	646
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3	2	151	66
3	3	722	713
3	4	1053	966
3	5	1188	1132
3	6	817	807
3	7	797	762
3	8	618	669
3	9	490	460
3	10	497	494
3	11	183	178
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5	1	1417	1322
5	2	870	820
5	3	411	371
5	4	922	962
5	5	948	937
5	6	1073	1049
5	7	295	290
5	8	466	493
5	9	302	291
5	10	304	279
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7	1	156	146
7	2	813	756
7	3	1469	1455
7	5	1364	1357
7	6	805	842
7	7	759	759
7	9	159	164
7	10	263	299
9	0	1660	1703
9	1	663	626
9	2	978	925
9	3	529	539
9	4	284	297
9	5	518	506
9	6	376	336
9	7	526	537
9	8	311	292
9	9	108	103
11	0	490	451
11	1	606	597
11	2	567	563
11	3	273	292
11	4	256	205
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11	6	451	478
11	7	687	702
11	8	412	411
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13	2	133	171
13	3	554	564
13	4	81	5
13	5	446	416
13	6	137	125
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15	4	702	688
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0	8	667	688
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2	5	1522	1552
2	6	872	857
2	7	1153	1166
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2	9	342	362
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4	1	933	906
4	2	674	672
4	3	440	426
4	5	184	159
4	6	160	118
4	7	189	205
4	8	611	605
4	9	202	212
4	10	734	750
6	0	1554	1489
6	2	682	701
6	3	507	445
6	4	1259	1231
6	5	608	541
6	6	981	884
6	7	560	594
6	8	435	444
6	9	119	117
8	0	600	500
8	1	1258	1245
8	2	106	91
8	3	654	660
8	4	399	388
8	5	417	383
8	7	270	238
8	8	390	385
8	9	712	708
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10	1	798	760
10	2	312	300
10	3	224	246
10	4	338	330
10	5	435	442
10	6	1027	1041
10	7	440	419
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12	2	361	305
12	4	263	240
12	6	276	238
14	0	78	23
14	1	568	621
14	2	162	126
14	4	147	190
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16	1	577	576
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1	3	130	41
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1	5	737	736
1	6	371	404
1	7	183	171
1	8	77	15
1	9	133	183
1	10	328	328
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3	1	571	587
3	2	543	524
3	3	965	950
3	4	625	622
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3	6	426	440
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3	8	350	352
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5	2	349	351
5	3	354	292
5	4	779	771
5	5	430	429
5	6	277	273
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5	8	359	352
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7	2	222	249
7	3	357	382
7	4	613	571
7	6	744	726
7	7	354	268
9	0	145	177
9	1	499	451
9	2	470	489
9	3	583	552
9	4	162	101
9	5	341	350
9	6	843	856
9	8	107	140
11	0	540	536
11	1	562	559
11	2	519	492
11	3	140	82
11	4	307	318
11	5	275	274
11	6	299	244
13	0	622	637
13	1	318	330
13	2	223	223
13	3	325	268
13	4	132	135
13	5	388	415
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15	1	103	80
15	2	647	631
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2	5	225	201
2	6	332	248
2	9	406	416
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4	3	461	449
4	4	489	467
4	5	1241	1231
4	6	519	519
4	7	638	652
4	8	483	456
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6	3	248	242
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6	7	240	221
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8	2	556	494
8	4	444	444
8	5	835	812
8	6	291	258
8	7	881	851
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10	2	511	449
10	3	293	288
10	4	387	420
10	5	348	318
10	6	83	40
12	0	521	545
12	1	425	428
12	2	143	19
12	3	291	285
12	4	362	381
12	5	609	607
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14	1	966	967
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14	3	301	257
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1	6	145	139
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1	8	132	74
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3	1	1148	1093
3	2	461	460
3	3	590	538
3	4	231	207
3	5	215	190
3	6	434	396
3	7	212	236
3	8	298	300
5	0	1237	1205
5	1	1243	1243
5	2	1225	1239
5	3	699	665
5	4	189	147
5	5	212	203
5	6	178	10
5	7	685	672
5	8	279	305
7	1	183	190
7	2	851	849
7	3	118	46
7	4	126	90
7	5	748	728
7	6	510	476
7	7	432	393
9	0	1481	1449
9	1	414	446
9	2	774	724
9	4	144	159
9	5	114	110
9	6	591	575
11	0	724	669
11	1	640	614
11	4	280	274
11	5	479	450
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13	1	446	418
13	2	528	481
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15	0	236	251
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2	2	119	32
2	3	489	487
2	4	853	860
2	5	913	915
2	6	348	337
2	7	809	833
2	8	348	351
4	0	905	848
4	0	911	848
4	3	810	816
4	4	744	758
4	5	117	89
4	6	185	63
4	7	112	52
6	1	94	141
6	4	578	570
6	5	468	464
6	6	996	1002
6	7	471	459
8	1	1342	1323
8	2	207	208
8	3	407	376
8	4	508	533
8	5	151	159
8	6	289	291
10	0	532	370
10	1	250	241
10	2	191	209
10	3	166	150
10	4	650	640
10	5	561	528
12	0	786	770
12	1	814	789
12	2	269	219
12	3	532	528
14	0	214	195
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1	2	1128	1125
1	3	187	166
1	4	354	330
1	5	775	763
1	6	621	606
1	7	276	254
3	0	1251	1212
3	1	133	146
3	2	1114	1090
3	3	278	263
3	4	103	48
3	5	140	168
3	6	629	593
3	7	84	7
5	0	520	515
5	1	1049	1052
5	2	113	80
5	3	327	280
5	4	192	183
5	5	413	362
5	6	270	263
7	0	668	649
7	1	817	809
7	2	501	464
7	3	397	347
7	4	172	171
7	5	458	450
7	6	593	563
9	0	668	670
9	1	604	668
9	2	408	432
9	4	332	276
9	5	210	207
11	0	344	73
11	1	943	932
13	0	318	304
13	1	145	35
****************			
H = 12			
0	0	603	231
0	2	267	268
0	4	1066	1084
0	6	902	910
2	0	404	409
2	1	1782	1763
2	2	837	847
2	3	934	917
2	5	200	229
4	0	394	422
4	2	459	467
4	3	344	335
4	4	435	456
4	5	624	626
4	6	594	614
6	0	1178	1126
6	1	920	899
6	2	1021	1007
6	3	415	376
6	4	342	324
6	5	81	10
8	0	213	227
8	1	264	270
8	2	326	353
8	3	180	203
8	4	286	240
10	0	1179	1125
10	1	377	441
10	2	724	741
10	3	153	212
12	0	380	253
****************			
H = 13			
1	0	630	606
1	1	788	775
1	2	580	584
1	4	121	54
1	5	492	493
3	0	753	715
3	1	454	453
3	2	235	254
3	3	490	482
3	4	455	421
3	5	392	379
5	0	176	115
5	1	390	377
5	2	168	38
5	3	174	127
5	4	349	361
5	5	422	394
7	0	900	898
7	1	287	304
7	2	382	359
7	3	460	448
7	4	282	255
9	0	563	535
9	1	200	190
9	2	476	400
11	1	475	502
****************			
H = 14			
0	0	1563	1450
0	2	1393	1354
0	4	124	83
2	2	215	235
2	3	411	393
2	4	196	191
4	0	804	790
4	1	841	839
4	2	492	492
4	3	380	349
4	4	301	278
6	2	249	266
6	3	312	290
8	1	1072	1052
8	2	181	155
10	0	136	111
****************			
H = 15			
1	0	217	25
1	1	604	582
1	2	185	153
1	3	233	262
3	0	234	177
3	1	304	290
3	2	306	313
3	3	493	454
5	0	287	248
5	1	114	143
5	2	503	522
7	0	242	207
7	1	322	333
9	0	377	385
****************			
H = 16			
0	2	252	216
2	0	338	315
2	1	802	824
2	2	199	116
4	0	170	122
4	1	188	210
6	0	730	735
6	1	249	223
****************			
H = 17			
1	0	524	516
1	1	393	395
3	0	421	344

The $(C_{10}H_8)_2Fe_4(CO)_{10}$ molecule has precise (*i.e.*, crystallographically required) C_2 symmetry and consists of a 4-*endo*,4′-*endo*-diazulene ligand in which the two five-membered rings function as substituted π-cyclopentadienyl systems and are linked by an $Fe_2(CO)_4$ bridge, while the *cis*-1,3-diene systems remaining in the *seven*-membered rings each are linked to an $Fe(CO)_3$ group.

Each of the iron atoms within the molecule attains the appropriate noble gas configuration (*i.e.*, that of krypton). Fe(1) may be regarded as a d^7 Fe^+ ion that obtains six electrons from a π-cyclopentadienyl anion, two electrons from a terminal carbonyl ligand, one electron from each of two bridging carbonyls, and one electron from an iron–iron bond. Fe(2) may be considered as a d^8 Fe^0 ion that obtains two electrons from each of three terminal carbonyl ligands and four electrons from a 1,3-diene system. The complex is thus expected to be diamagnetic—not paramagnetic as previously reported.[7,8]

The bridging $Fe_2(CO)_4$ group—which is composed of an iron–iron bond, two bridging carbonyl ligands, and two terminal carbonyls—is analogous to that found in $[\pi\text{-}C_5H_5Fe(CO)_2]_2$.[24] The iron–iron bond lengths (Fe(1)–Fe(1′) = 2.508 ± 0.003 Å in the present complex and 2.49 ± 0.02 Å in $[\pi\text{-}C_5H_5Fe(CO)_2]_2$[24]) are identical within the limits of experimental error. However, the over-all conformations of the $Fe_2(CO)_4$ groups in the

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[24] O. S. Mills, *Acta Cryst.*, **11**, 620 (1958).

TABLE II

ATOMIC COORDINATES (WITH ESD'S)[a] FOR $(C_{10}H_8)_2Fe_4(CO)_{10}\cdot C_2H_4Cl_2$

Atom	*x*	*y*	*z*
(a) Atoms in $(C_{10}H_8)_2Fe_4(CO)_{10}$ Molecule[b,c]			
Fe(1)	−0.00660 (5)	0.16074 (6)	−0.15691 (7)
Fe(2)	−0.21298 (5)	0.40304 (7)	−0.22236 (8)
O(1)	−0.1632 (3)	0.1235 (3)	−0.3584 (4)
O(2)	−0.0131 (4)	−0.0227 (4)	−0.1220 (5)
O(3)	−0.3509 (4)	0.3801 (5)	−0.1494 (7)
O(4)	−0.3006 (4)	0.5483 (5)	−0.3702 (6)
O(5)	−0.0933 (5)	0.4907 (7)	−0.0209 (7)
C(1)	−0.0474 (4)	0.1965 (4)	−0.0307 (5)
C(2)	0.0424 (3)	0.2014 (4)	0.0131 (5)
C(3)	0.0617 (4)	0.2646 (4)	−0.0526 (5)
C(4)	−0.0278 (3)	0.3737 (4)	−0.2151 (5)
C(5)	−0.1197 (4)	0.3981 (4)	−0.2908 (5)
C(6)	−0.1939 (4)	0.3462 (5)	−0.3527 (5)
C(7)	−0.2218 (4)	0.2854 (5)	−0.2991 (6)
C(8)	−0.1761 (4)	0.2723 (5)	−0.1789 (5)
C(9)	−0.0840 (4)	0.2567 (4)	−0.1191 (5)
C(10)	−0.0168 (3)	0.2980 (4)	−0.1368 (5)
C(11)	−0.0907 (4)	0.1399 (4)	−0.3087 (5)
C(12)	−0.0093 (4)	0.0491 (5)	−0.1336 (5)
C(13)	−0.2985 (5)	0.3879 (5)	−0.1804 (7)
C(14)	−0.2652 (5)	0.4929 (6)	−0.3100 (7)
C(15)	−0.1404 (5)	0.4571 (7)	−0.0980 (7)
H(1)	−0.0820	0.1517	−0.0017
H(2)	0.0877	0.1642	0.0827
H(3)	0.1243	0.2858	−0.0400
H(4)	−0.0023	0.4238	−0.1516
H(5)	−0.1313	0.4668	−0.3007
H(6)	−0.2285	0.3561	−0.4431
H(7)	−0.2775	0.2490	−0.3468
H(8)	−0.2120	0.2745	−0.1276
(b) Atoms in the $C_2H_4Cl_2$ Molecule[d]			
Cl	−0.36678 (24)	0.15978 (29)	−0.17257 (69)
C(16A)	−0.4514	0.1380	−0.2777
C(16B)	−0.4776	0.1468	−0.1921
C(16C)	−0.4755	0.2012	−0.2271

[a] Esd's (estimated standard deviations), shown in parentheses, are right-adjusted to the last digit of the preceding number. [b] Hydrogen atoms are in calculated positions (see footnote 22). [c] The listed atomic positions define the asymmetric unit of the $(C_{10}H_8)_2Fe_4(CO)_{10}$ molecule. The other half of the molecule is defined by the transformation: $x' = -x, y' = y, z' = -1/2 - z$. [d] Only the asymmetric unit of the disordered $C_2H_4Cl_2$ molecule is given. The other half of the molecule is defined by the transformation: $x^* = -1 - x, y^* = y, z^* = -1/2 - z$.

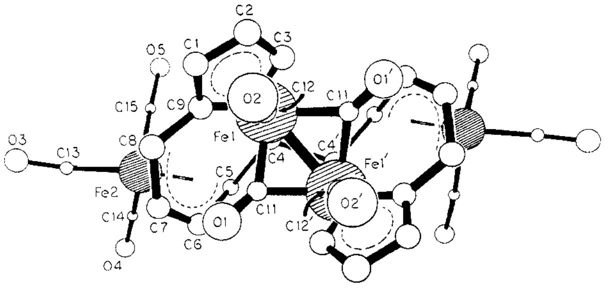

Figure 1.—The numbering of atoms within the $(C_{10}H_8)_2Fe_4(CO)_{10}$ molecule. (The molecule is viewed along *b*.)

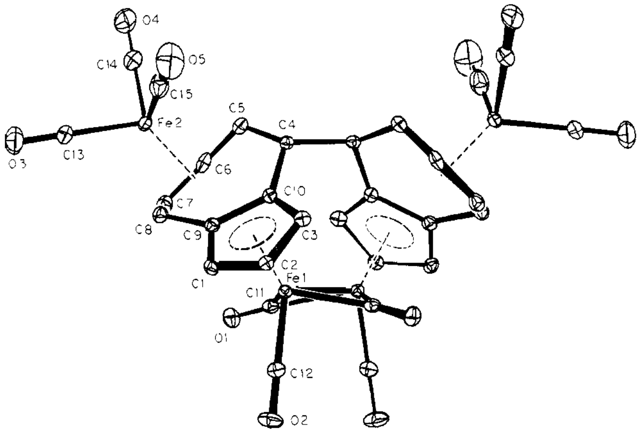

Figure 2.—The $(C_{10}H_8)_2Fe_4(CO)_{10}$ molecule viewed from a direction in the *ac* plane 10° from $+a$, toward $+c$. (The *b* direction is vertical.) The 68% probability envelopes of the atomic vibration ellipsoids are shown. [The diagram was drawn using OTLIPS—an IBM 1620/CALCOMP program written by P. H. B.]

two complexes are distinctly different. In $[\pi\text{-}C_5H_5Fe(CO)_2]_2$ the $Fe(CO)_2Fe$ bridge is planar (having crystallographically imposed C_i symmetry)[24] whereas in $(C_{10}H_8)_2Fe_4(CO)_{10}$ the $Fe_2(CO)_4$ group has C_2 symmetry and is nonplanar, the angle between the planes of the two carbonyl bridges (planes e and f of Table VII) being 154° 40′. [This nonplanarity is illustrated clearly in Figure 2.]

The Diazulene Ligand.—The 4,4′-diazulene ligand possesses a crystallographic twofold axis and is accurately described as a 4-*endo*,4′-*endo*-diazulene system (it being understood that the prefix *endo* describes the geometry relative to the $Fe_2(CO)_4$ moiety).

The two (symmetry-related) five-membered rings behave as π-cyclopentadienyl ligands in bonding to Fe(1) and Fe(1′). The π-cyclopentadienyl system defined by C(1)–C(2)–C(3)–C(10)–C(9) has a root-mean-square (rms) deviation of 0.013 Å from the least-squares plane: $-0.280X + 0.707Y + 0.649Z = 2.122$ [see Table VII]. Individual carbon–carbon bond lengths range from 1.407 ± 0.009 to 1.439 ± 0.008 Å and average 1.423 Å; internal angles range from 106.8 ± 0.5 to 109.7 ± 0.6°, averaging 108.0°—the ideal value for a regular, planar pentagon. Each of the observed bond distances and bond angles is within 3σ of the mean value; the five-membered ring thus has D_{5h} symmetry within the limits of experimental error. Iron–carbon distances around the substituted π-cyclopentadienyl system range from 2.096 ± 0.006 to 2.190 ± 0.006 Å. This spread is sufficiently large ($\Delta/\sigma = 15.5$) as to indicate a real asymmetry in the iron–π-cyclopentadienyl bonding, probably a result of strain in the molecule caused by simultaneous requirements for iron–π-cyclopentadienyl, iron–iron, and carbon–carbon [C(4)–C(4′)] bonding. It may be noted that Fe(1) lies 1.757 Å below the plane of the π-cyclopentadienyl ring.

The seven-membered ring is severely distorted from planarity owing to a combination of (i) the tetrahedral nature of C(4) and (ii) the usual "bend" across the ends of a coordinated *cis*-1,3-diene system (*vide infra*). Individual displacements of atoms from the least-squares plane through the five-membered ring are: C(4), +0.154 Å; C(5), +0.178 Å; C(6), −0.598 Å; C(7),

TABLE III

ANISOTROPIC THERMAL PARAMETERS[a] FOR $(C_{10}H_8)_2Fe_4(CO)_{10}\cdot C_2H_4Cl_2$

Atom	$10^4\beta_{11}$	$10^4\beta_{22}$	$10^4\beta_{33}$	$10^4\beta_{12}$	$10^4\beta_{13}$	$10^4\beta_{23}$
Fe(1)	15.8 (0.4)	16.2 (0.5)	28.2 (0.7)	0.0 (0.6)	14.8 (0.7)	3.3 (0.8)
Fe(2)	19.8 (0.4)	23.9 (0.5)	47.8 (0.8)	7.1 (0.6)	30.6 (0.8)	−3.6 (0.9)
O(1)	20 (2)	39 (3)	47 (3)	−14 (4)	13 (4)	−15 (5)
O(2)	66 (3)	22 (3)	76 (5)	−6 (5)	62 (6)	18 (6)
O(3)	58 (3)	75 (5)	156 (8)	25 (6)	138 (9)	43 (10)
O(4)	50 (3)	60 (4)	171 (8)	57 (6)	98 (8)	105 (10)
O(5)	68 (4)	131 (7)	154 (8)	−80 (8)	75 (9)	−189 (12)
C(1)	28 (3)	24 (3)	36 (4)	−7 (5)	37 (6)	−1 (6)
C(2)	19 (2)	21 (3)	32 (4)	0 (4)	11 (5)	−6 (6)
C(3)	23 (2)	24 (3)	26 (4)	−4 (4)	10 (5)	−10 (9)
C(4)	18 (2)	16 (3)	34 (4)	0 (4)	15 (5)	−2 (6)
C(5)	24 (3)	26 (3)	43 (5)	11 (5)	27 (6)	2 (6)
C(6)	19 (2)	39 (4)	38 (5)	20 (5)	18 (5)	−8 (7)
C(7)	18 (3)	35 (4)	60 (5)	2 (5)	25 (6)	−16 (8)
C(8)	20 (2)	26 (3)	54 (5)	3 (5)	24 (6)	−3 (7)
C(9)	24 (2)	20 (3)	36 (4)	−1 (4)	29 (5)	−5 (6)
C(10)	20 (2)	22 (3)	25 (4)	−2 (4)	20 (5)	−6 (6)
C(11)	26 (3)	19 (3)	36 (4)	−1 (5)	26 (6)	−4 (6)
C(12)	34 (3)	23 (4)	36 (5)	4 (5)	31 (6)	2 (7)
C(13)	35 (3)	34 (4)	81 (7)	11 (6)	68 (8)	3 (8)
C(14)	34 (3)	37 (4)	96 (7)	15 (6)	57 (8)	10 (10)
C(15)	36 (4)	65 (6)	81 (7)	−12 (7)	52 (8)	−58 (11)
Cl	52.7 (1.7)	80.3 (2.7)	523.4 (12.3)	3.1 (3.2)	121.0 (7.0)	24.1 (8.9)
C(16A)	B = 9.6 Å^2; occupancy = 0.26					
C(16B)	B = 10.3 Å^2; occupancy = 0.39					
C(16C)	B = 5.6 Å^2; occupancy = 0.34					

[a] The anisotropic thermal parameter (T) is defined as: $T = \exp[-\beta_{11}h^2 - \beta_{22}k^2 - \beta_{33}l^2 - \beta_{12}hk - \beta_{13}hl - \beta_{23}kl]$.

−0.642 Å; C(8), +0.090 Å (where a minus sign indicates displacement toward Fe(1) and a plus sign shows displacement away from this atom).

The C(sp^2)–C(sp^3) bonds, C(10)–C(4) = 1.510 ± 0.008 Å and C(5)–C(4) = 1.529 ± 0.009 Å, are each in agreement with the recognized single-bond distance of 1.510 ± 0.005 Å,[25] and the C(8)–C(9) bond (which links the unsaturated π-cyclopentadienyl and *cis*-1,3-diene systems) is 1.473 ± 0.009 Å in length—*i.e.*, indistinguishable from a C(sp^2)–C(sp^2) linkage of unit bond order, for which the recognized value is 1.465 ± 0.005 Å.[25] It would seem, therefore, that there is virtually no conjugation between the π-cyclopentadienyl and *cis*-1,3-diene systems. [A similar conclusion has been reached for the π-cyclopentadienyl and π-pentadienyl systems of $C_{10}H_8Mn_2(CO)_6$,[5] which are linked by two C(sp^2)–C(sp^2) bonds each of length 1.476 ± 0.013 Å.]

The atoms of the *cis*-1,3-diene system—C(5), C(6), C(7), C(8)—have an rms deviation of only 0.002 Å from the least-squares plane: $-0.708X + 0.685Y + 0.170Z = 4.021$. Carbon–carbon distances within this system [C(5)–C(6) = 1.444 ± 0.009 Å, C(6)–C(7) = 1.371 ± 0.009 Å, C(7)–C(8) = 1.434 ± 0.009 Å] are appreciably different from those within a noncoordinated 1,3-diene system [an electron diffraction study of *trans*-buta-1,3-diene reveals bond lengths of 1.337 ± 0.005, 1.483 ± 0.010, and 1.337 ± 0.005 Å, respectively[26]] and are similar to those found in previously examined diene–iron tricarbonyl species[27–41] [see Table VIII]. As may clearly be seen in Figure 2, the seven-membered ring distorts about the C(5)···C(8) axis such that C(4) and C(9) are bent away from the *cis*-1,3-diene system so as to be further distant from the coordinated $Fe(CO)_3$ group. This phenomenon is observed in all complexes in which a *cis*-1,3-diene system is bonded to a transition metal.[42]

Individual iron–carbon bond distances for the terminal carbon atoms [Fe(2)–C(5) = 2.138 ± 0.006 and Fe(2)–C(8) = 2.134 ± 0.006 Å] are considerably greater than the distances for the central carbon atoms of the *cis*-1,3-diene system [Fe(2)–C(6) = 2.045 ± 0.006 and Fe(2)–C(7) = 2.055 ± 0.007 Å].

With the exception of the interior angles of the *cis*-1,3-diene system [C(5)–C(6)–C(7) = 121.5 ± 0.6° and C(6)–C(7)–C(8) = 119.5 ± 0.6°], angles within the seven-membered rings are each significantly (*i.e.*, $>3\sigma$) larger than the appropriate ideal sp^2 or sp^3 value. Thus the value of 115.4 ± 0.5° for C(10)–C(4)–C(5) is some 12σ greater than the ideal tetrahedral angle of 109° 28′,

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TABLE IV

VIBRATION ELLIPSOIDS FOR ATOMS IN THE $(C_{10}H_8)_2Fe_4(CO)_{10}\cdot C_2H_4Cl_2$ MOLECULE[a,b]

Atom	B_{max}, Å^2	B_{med}, Å^2	B_{min}, Å^2
Fe(1)	1.79	1.56	1.43
Fe(2)	2.71	2.50	1.52
O(1)	3.99	3.20	1.62
O(2)	6.63	4.30	1.81
O(3)	10.11	6.27	3.10
O(4)	11.88	5.24	2.50
O(5)	17.79	7.51	2.90
C(1)	2.96	2.18	1.44
C(2)	2.48	1.99	1.52
C(3)	2.69	2.41	1.28
C(4)	2.24	1.72	1.55
C(5)	2.97	2.36	1.83
C(6)	4.44	2.07	1.43
C(7)	4.15	2.78	1.76
C(8)	3.16	2.49	1.90
C(9)	2.42	2.03	1.69
C(10)	2.17	1.99	1.31
C(11)	2.65	2.06	1.74
C(12)	3.46	2.22	1.90
C(13)	4.78	3.46	2.16
C(14)	7.17	3.45	2.96
Cl	31.80	7.72	4.97

[a] The magnitudes of the major, median, and minor axes of the atomic vibration ellipsoids are defined in terms of the isotropic thermal parameter, B. This is related to the root-mean-square displacement, $(\overline{U^2})^{1/2}$, by: $(\overline{U^2})^{1/2} = [B/8\pi^2]^{1/2}$. [b] The 68% probability envelopes for these ellipsoids are depicted in Figure 2.

and the remaining (sp^2) angles which range from 125.0 ± 0.5 to 131.7 ± 0.6° are each significantly (10–20σ) greater than the ideal trigonal angle of 120.0°. This systematic pattern of increased bond angles presumably results from the angular requirements of the seven-membered ring system. [The internal angle for a regular, planar heptagon is 128.6°, while angles within the planar seven-membered rings of azulene-1,3-dipropionic acid[43] and the azulene–1,3,5-trinitrobenzene molecular complex[44] are known to vary from 125.9 to 130.2°.]

Finally, it should be noted that the bridging C(4)–C(4′) bond length of 1.567 ± 0.012 Å, like the corresponding value of 1.561 ± 0.018 Å for $[C_{10}H_8Mo(CO)_3CH_3]_2$,[3] is slightly (*i.e.*, about 2σ) greater than the ideal C(sp^3)–C(sp^3) distance [which is estimated as 1.537 ± 0.005 Å from a variety of organic molecules or as 1.5445 ± 0.0001 Å from diamond].[45]

The Carbonyl Ligands.—Within the $Fe_2(CO)_4$ moiety, terminal carbonyl ligands are attached to Fe(1) and Fe(1′) by iron–carbon bonds 1.764 ± 0.007 Å in length. The carbonyl bridges [Fe(1)–C(11)O(1)–Fe(1′) and Fe(1)–C(11′)O(1′)–Fe(1′)] are symmetric within the limits of experimental error, individual bond lengths being Fe(1)–C(11) = Fe(1′)–C(11′) = 1.923 ± 0.006 Å, Fe(1)–C(11′) = Fe(1′)–C(11) = 1.938 ± 0.006 Å, and C(11)–O(1) = C(11′)–O(1′) = 1.175 ± 0.008 Å. Each of these distances is in good agreement with values ob-

TABLE V

INTERATOMIC DISTANCES WITHIN THE $(C_{10}H_8)_2Fe_4(CO)_{10}\cdot C_2H_4Cl_2$ MOLECULE, WITH ESTIMATED STANDARD DEVIATIONS[a]

Atoms	Distance, Å	Atoms	Distance, Å
(a) Iron–Iron Bond Length			
Fe(1)–Fe(1′)	2.508 (3)		
(b) Iron–Diazulene Distances			
Fe(1)–C(1)	2.101 (6)	Fe(2)···C(4)	3.198 (6)
Fe(1)–C(2)	2.096 (6)	Fe(2)–C(5)	2.138 (6)
Fe(1)–C(3)	2.121 (6)	Fe(2)–C(6)	2.045 (6)
Fe(1)–C(10)	2.166 (6)	Fe(2)–C(7)	2.055 (7)
Fe(1)–C(9)	2.190 (6)	Fe(2)–C(8)	2.134 (6)
Fe(1)···C(4)	3.379 (6)	Fe(2)···C(9)	3.075 (6)
Fe(1)...C(8)	3.315 (6)	Fe(2)···C(10)	3.503 (6)
(c) Bond Lengths within Diazulene Ligand			
C(1)–C(2)	1.417 (9)	C(10)–C(4)	1.510 (8)
C(2)–C(3)	1.425 (8)	C(4)–C(5)	1.529 (9)
C(3)–C(10)	1.439 (8)	C(5)–C(6)	1.444 (9)
C(10)–C(9)	1.427 (8)	C(6)–C(7)	1.371 (9)
C(9)–C(1)	1.407 (9)	C(7)–C(8)	1.434 (9)
C(4)–C(4′)	1.567 (12)	C(8)–C(9)	1.473 (9)
(d) Iron–Carbonyl Distances			
Fe(1)–C(11)	1.923 (6)	Fe(2)–C(13)	1.788 (8)
Fe(1)–C(11′)	1.938 (6)	Fe(2)–C(14)	1.788 (9)
Fe(1)–C(12)	1.764 (7)	Fe(2)–C(15)	1.786 (9)
Fe(1)···O(1)	2.931 (5)	Fe(2)···O(3)	2.926 (8)
Fe(1)···O(1′)	2.919 (5)	Fe(2)···O(4)	2.939 (7)
Fe(1)···O(2)	2.894 (6)	Fe(2)···O(5)	2.906 (9)
(e) Distances within Carbonyl Ligands			
C(11)–O(1)	1.175 (8)	C(13)–O(3)	1.138 (11)
C(12)–O(2)	1.130 (9)	C(14)–O(4)	1.151 (11)
		C(15)–O(5)	1.120 (13)
(f) Diazulene-to-Carbonyl Contacts (to 3.5 Å)			
C(1)···C(11)	3.463 (9)	C(7)···C(13)	2.883 (11)
C(1)···C(12)	2.858 (9)	C(7)···C(14)	3.301 (11)
C(2)···C(12)	2.929 (9)	C(7)···O(1)	2.930 (8)
C(2)···C(11′)	3.226 (9)	C(8)···C(11)	3.359 (8)
C(3)···C(11′)	2.822 (9)	C(8)···C(13)	2.771 (10)
C(3)···O(1′)	3.300 (7)	C(8)···C(15)	3.031 (11)
C(4)···C(15)	3.195 (11)	C(8)···O(1)	3.346 (8)
C(5)···C(14)	2.836 (11)	C(9)···C(11)	3.011 (9)
C(5)···C(15)	2.811 (11)	C(9)···C(15)	3.309 (11)
C(6)···C(13)	3.462 (10)	C(9)···O(1)	3.492 (8)
C(6)···C(14)	2.754 (11)	C(10)···C(11)	3.201 (8)
C(6)···C(15)	3.481 (11)	C(10)···C(15)	3.438 (11)
C(7)···C(11)	3.241 (9)	C(10)···C(11′)	3.329 (8)
(g) Carbon···Carbon Contacts between the Halves of the 4,4′-Diazulene Ligand (to 3.5 Å)			
C(3)···C(4′)	3.279 (9)	C(5)···C(3′)	3.330 (10)
C(3)···C(5′)	3.330 (10)	C(5)···C(4′)	2.549 (10)
C(3)···C(6′)	3.265 (10)	C(5)···C(10′)	3.273 (10)
C(4)···C(3′)	3.279 (9)	C(6)···C(3′)	3.265 (10)
C(4)–C(4′)	1.567 (12)	C(10)···C(4′)	2.619 (10)
C(4)···C(5′)	2.549 (10)	C(10)···C(5′)	3.273 (10)
C(4)···C(10′)	2.619 (10)	C(10)···C(10′)	3.210 (12)
(h) Distances within 1,2-Dichloroethane Molecules			
Molecule 1		Molecule 2	
Cl–C(16A)	1.57	Cl–C(16B)	1.84
C(16A)–C(16C*)	1.62	C(16B)–C(16B*)	1.37
C(16C*)–Cl*	1.83	C(16B*)–Cl*	1.83

[a] Estimated standard deviations include errors in unit cell dimensions.

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(43) H. L. Ammon and M. Sundaralingam, *J. Am. Chem. Soc.*, **88**, 4794 (1966).

(44) A. W. Hanson, *Acta Cryst.*, **19**, 19 (1965).

(45) Reference 25, p S14s.

TABLE VI

ANGLES WITHIN THE $(C_{10}H_8)_2Fe_4(CO)_{10}\cdot C_2H_4Cl_2$ SYSTEM

Atoms	Angle, deg	Atoms	Angle, deg
(a) Angles around Fe(1)			
C(1)–Fe(1)–C(2)	39.5 (2)	Fe(1′)–Fe(1)–C(11′)	49.2 (2)
C(2)–Fe(1)–C(3)	39.5 (2)	Fe(1′)–Fe(1)–C(12)	100.6 (2)
C(3)–Fe(1)–C(10)	39.2 (2)	Fe(1′)–Fe(1)···O(2)	100.0 (1)
C(10)–Fe(1)–C(9)	38.2 (2)	C(11)–Fe(1)–C(11′)	95.7 (3)
C(9)–Fe(1)–C(1)	38.2 (2)	C(11)–Fe(1)–C(12)	86.9 (2)
Fe(1′)–Fe(1)–C(11)	49.7 (2)	C(11′)–Fe(1)–C(12)	87.8 (2)
(b) Angles around Fe(2)			
C(5)–Fe(2)–C(6)	40.3 (3)	C(13)–Fe(2)–C(15)	98.6 (3)
C(6)–Fe(2)–C(7)	39.1 (3)	C(14)–Fe(2)–C(15)	100.6 (3)
C(7)–Fe(2)–C(8)	40.0 (3)	O(3)···Fe(2)···O(4)	91.9 (2)
C(8)–Fe(2)–C(4)	70.7 (3)	O(3)···Fe(2)···O(5)	98.1 (2)
C(13)–Fe(2)–C(14)	91.8 (3)	O(4)···Fe(2)···O(5)	101.8 (2)
(c) Angles within Diazulene Ligand			
C(9)–C(1)–C(2)	109.7 (6)	C(5)–C(4)–C(4′)	110.9 (6)
C(1)–C(2)–C(3)	106.8 (5)	C(10)–C(4)–C(5)	115.4 (5)
C(2)–C(3)–C(10)	108.5 (5)	C(4)–C(5)–C(6)	131.7 (6)
C(3)–C(10)–C(9)	107.0 (5)	C(5)–C(6)–C(7)	121.5 (6)
C(10)–C(9)–C(1)	107.9 (6)	C(6)–C(7)–C(8)	119.5 (6)
C(3)–C(10)–C(4)	127.1 (5)	C(7)–C(8)–C(9)	125.5 (6)
C(1)–C(9)–C(8)	124.0 (5)	C(8)–C(9)–C(10)	128.1 (6)
C(10)–C(4)–C(4′)	116.7 (6)	C(9)–C(10)–C(4)	125.0 (5)
(d) Angles in Carbonyl Ligands			
Fe(1)–C(11)–O(1)	141.0 (5)	Fe(2)–C(13)–O(3)	177.1 (8)
Fe(1′)–C(11)–O(1)	138.0 (6)	Fe(2)–C(14)–O(4)	177.0 (8)
Fe(1)–C(11)–Fe(1′)	81.0 (3)	Fe(2)–C(15)–O(5)	178.3 (9)
Fe(1)–C(12)–O(2)	177.8 (8)		
(e) Angles within Dichloroethane Molecules			
Molecule 1		Molecule 2	
Cl–C(16A)–C(16C*)	107	Cl–C(16B)–C(16B*)	104
C(16A)–C(16C*)–Cl*	118	C(16B)–C(16B*)–Cl*	104

TABLE VII

IMPORTANT LEAST-SQUARES PLANES WITHIN THE $(C_{10}H_8)_2Fe_4(CO)_{10}$ MOLECULE[a,b]

Atom	Dev, Å	Atom	Dev, Å
(a) π-Cyclopentadienyl Plane: $-0.280X + 0.707Y + 0.649Z = 2.122$			
C(1)*	−0.014	O(1)	−3.25
C(2)*	0.005	C(12)	−2.76
C(3)*	0.006	O(2)	−3.42
C(10)*	−0.015	C(13)	1.94
C(9)*	0.018	O(3)	2.39
C(4)	0.154	C(14)	1.75
C(5)	0.178	O(4)	1.98
C(6)	−0.598	C(15)	2.69
C(7)	−0.642	O(5)	3.53
C(8)	0.090	Fe(1)	−1.757
C(11)	−2.96	Fe(2)	1.309
(b) *cis*-1,3-Diene Plane: $-0.708X + 0.685Y + 0.170Z = 4.021$			
C(5)*	0.001	C(14)	2.68
C(6)*	−0.002	C(15)	2.00
C(7)*	0.002	O(3)	3.46
C(8)*	−0.001	O(4)	3.36
Fe(2)	1.592	O(5)	2.23
C(13)	2.72		
(c) $-0.520X + 0.763Y + 0.384Z = 5.969$			
C(13)*	0.000	C(15)*	0.000
C(14)*	0.000		
(d) $-0.506X + 0.774Y + 0.380Z = 6.573$			
O(3)*	0.000	O(5)*	0.000
O(4)*	0.000		
(e) $-0.191X - 0.976Y + 0.107Z = -2.495$			
Fe(1)*	−0.000	C(11)*	0.002
Fe(1′) *	−0.000	O(1)*	−0.001
(f) $0.191X + 0.976Y + 0.107Z = 2.378$			
Fe(1) *	0.000	C(11′) *	−0.002
Fe(1′) *	0.000	O(1′) *	0.001

[a] Planes are defined in Cartesian coordinates (X, Y, Z) which are related to the monoclinic fractional cell coordinates (x, y, z) by the transformation: $X = xa + zc \cos \beta$, $Y = yb$, and $Z = zc \sin \beta$. [b] Planes are calculated using unit weights for all atoms marked with an asterisk.

TABLE VIII

CARBON–CARBON DISTANCES AROUND THE COORDINATED *cis*-1,3-DIENE SYSTEMS IN SOME (diene)Fe(CO)$_3$ DERIVATIVES

Complex	Bond lengths, Å	Ref
$C_4H_6Fe(CO)_3$	1.46, 1.45, 1.46	27, 28
$C_8H_8Fe(CO)_3$	1.42, 1.42, 1.42	29, 30
$C_8H_8[Fe(CO)_3]_2$	1.43, 1.39, 1.40 1.48, 1.40, 1.44	31, 30
$C_6F_8Fe(CO)_3$	1.397, 1.374, 1.397	32, 33
$[(CF_3)_4C_5O]Fe(CO)_3$	1.416, 1.395, 1.365	34, 35
$C_{16}H_{16}Fe(CO)_3$	1.42, 1.41, 1.46	36, 37
$[C_7H_6O]Fe(CO)_3$	1.442, 1.396, 1.435	38
$[(C_6H_5)_3C_7H_3O]Fe(CO)_3$	1.45, 1.38, 1.49	39
(Vit A aldehyde)Fe(CO)$_3$	1.45, 1.39, 1.49	40
(thebaine)Fe(CO)$_3$	1.52, 1.39, 1.44	41
$(C_{10}H_8)_2Fe_4(CO)_{10}$	1.444, 1.371, 1.434	This work

tained from previous crystallographic studies on polynuclear iron complexes.[46]

Iron–carbon bond lengths within the Fe(CO)$_3$ group range from 1.786 ± 0.009 to 1.788 ± 0.009 Å, averaging 1.787 Å; carbon–oxygen distances range from 1.120 ± 0.013 to 1.151 ± 0.011 Å, the mean value being 1.136 Å. Owing to the lack of cylindrical symmetry *trans* to the Fe(CO)$_3$ group, the three carbonyl ligands are not arranged about Fe(2) according to strict C_{3v} symmetry, angles at the iron atom being C(13)–Fe(2)–C(14) = 91.8 ± 0.3°, C(13)–Fe(2)–C(15) = 98.6 ± 0.3°, and C(14)–Fe(2)–C(15) = 100.6 ± 0.3°. The plane defined by the three carbon atoms of the Fe(CO)$_3$ group is not parallel to the *cis*-1,3-diene system, but, rather, there is an angle of 17° 00′ between these two planes. This is common to all (*cis*-1,3-diene)Fe(CO)$_3$ complexes, the corresponding angle in $C_6F_8Fe(CO)_3$,[33] for example, being 21°.

The 1,2-Dichloroethane of Crystallization.—The 1,2-dichloroethane of crystallization that is found to be present in the crystal is grossly disordered. There are three sites for each carbon atom [*i.e.*, C(16A), C(16B), C(16C) and C(16A*), C(16B*), C(16C*)] but only one site for each of the chlorine atoms—although a consideration of the anisotropic thermal parameters for the chlorine atoms shows a gross elongation in the z direction. A survey of all interatomic carbon–chlorine and carbon–carbon distances leads to the conclusion that there are two possible conformations for 1,2-dichloroethane molecules within the crystal. The molecule defined by Cl–C(16B)–C(16B*)–Cl* has an occupancy of 0.39 while the 1,2-dichloroethane molecules defined by Cl–C(16A)–C(16C*)–Cl* and Cl–C(16C)–C(16A*)–Cl*

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(46) See all references given in Table VII of ref 3.

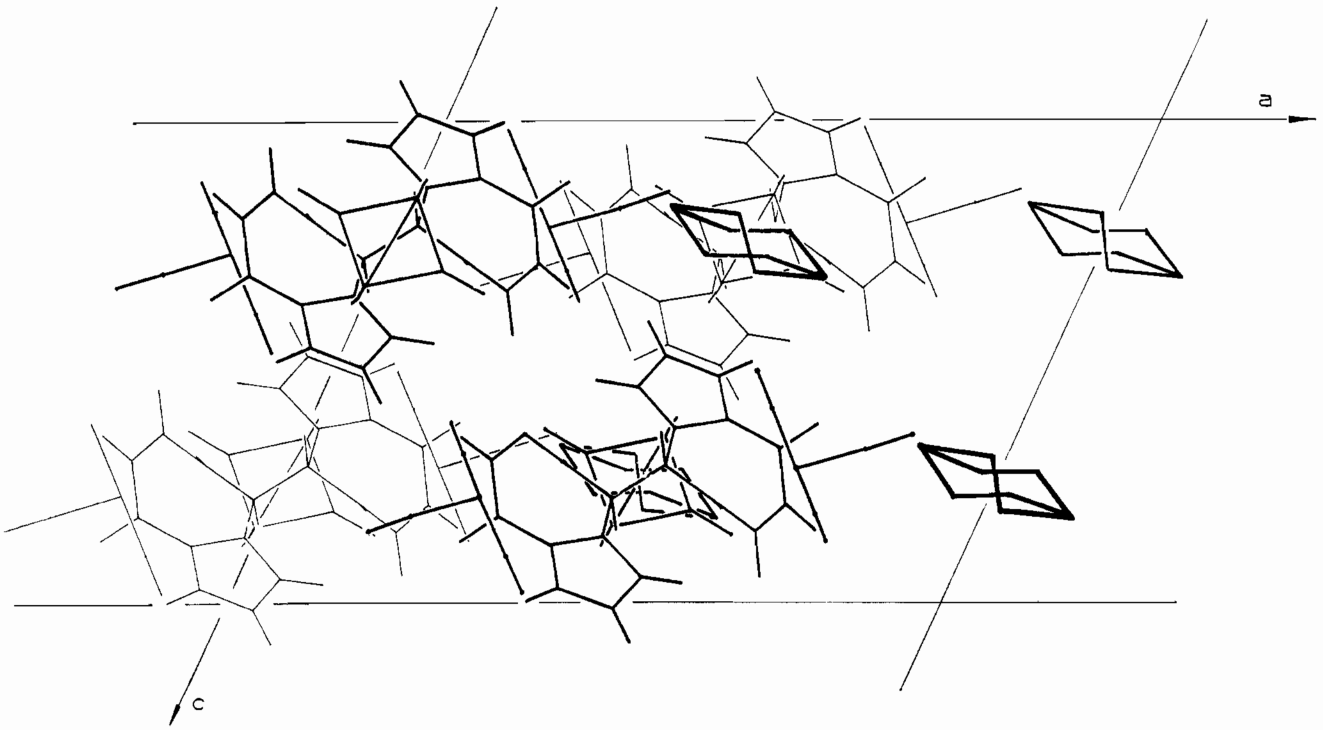


Figure 3.—Packing within the $(C_{10}H_8)_2Fe_4(CO)_{10} \cdot C_2H_4Cl_2$ crystal, viewed down *b*. The diagram is simplified in the following manner: around $x = 0$ only $(C_{10}H_8)_2Fe_4(CO)_{10}$ molecules are shown, around $x = 1$ only the (threefold disordered) $C_2H_4Cl_2$ molecules are shown, but at $x = {}^1/_2$ both are shown.

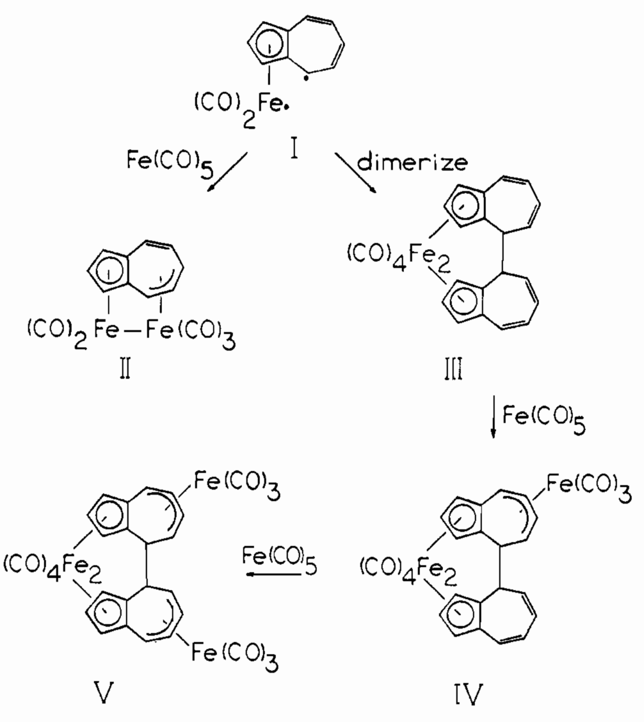
each have an occupancy of 0.30.[47] These latter two molecules do not individually possess a C_2 axis but, taken together, provide the crystallographically required C_2 axis on a statistical basis.

The Crystal Structure.—Figure 3 shows the packing of molecules within the crystal lattice, viewed down *b*. There are no abnormally short intermolecular contacts.

Discussion

In a previous publication[3] we have outlined a number of ways in which azulene might be expected to interact with a transition metal carbonyl. The present structural analysis shows that the scheme must be extended slightly.

The initial reaction of azulene with an iron carbonyl is believed to involve the formation of a *pseudo*-π-cyclopentadienyliron-dicarbonyl species (I). This either may interact with a further iron carbonyl residue to form the known[2] compound $C_{10}H_8Fe_2(CO)_5$ (II) or may dimerize to form a complex of stoichiometry $[(C_{10}H_8Fe(CO)_2]_2$ (II)—a molecule which King[48] has reported to be isolable from the reaction of azulene and iron pentacarbonyl "under slightly different conditions" [from those reported by Burton, *et al.*[8]]. The present compound $(C_{10}H_8)_2Fe_4(CO)_{10}$ (V) may be seen to be derived by utilizing each of the *cis*-1,3-diene systems of III in bonding to an $Fe(CO)_3$ group. The scheme also indicates that a complex of stoichiometry $(C_{10}H_8)_2Fe_3(CO)_7$ (IV) should be isolable from the reaction of azulene with an iron carbonyl. This species, however, has not yet been reported.



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[47] In refining occupancy factors, those for C(16A), C(16B), and C(16C) were allowed to refine independently. The net result [see Table III] was for C(16A) and C(16C) to end up with unequal occupancies: 0.26 and 0.34, respectively. These must, of course, be equal and are given as 0.30 in the text.

[48] R. B. King, *J. Am. Chem. Soc.*, **88**, 2075 (1966).