We account for the finding of generally longer bonds as the number of phosphine groups in the molecule increases and the absence of any structural *trans* effect in the imino complexes, as well as the finding of structural *trans* effect in both nitrido complexes. In addition, the failure to isolate a triphenylphosphine analog of $ReNCl_2(P(C_2H_5)_2C_6H_5)_3$ may be rationalized. Incorporation of three bulky triphenylphosphine groups into the molecule sets up such steric strain that one of these groups is expelled and a five-coordinate species $ReNCl_2(P(C_6H_5)_3)_2$ is obtained. Both the five-coordinate and the six-coordinate nitrido compounds have been prepared with diphenylethylphosphine, and in view of the above results a structural study of the six-coordinate compound should be extremely interesting as an example of an overcrowded molecule. We can now also rationalize the apparent insensitivity of the Re–N bond length in the imino complexes to change of the substituent on the nitrogen atom. The Re–N bond is already highly strained so that small changes in the bond strength are dissipated in increasing or relieving the strain in the other ligands.

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The Crystal and Molecular Structure of Racemic Bis(π-azulene)iron

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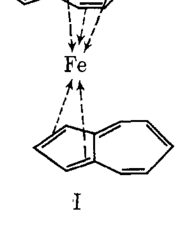
Bis(π-azulene)iron, $(C_{10}H_8)_2Fe$, crystallizes in the centrosymmetric monoclinic space group $P2_1/c$ (C_{2h}^5 no. 14) with $a = 7.523$ (5) Å, $b = 12.071$ (4) Å, $c = 15.419$ (16) Å, $\beta = 98.26$ (4)°, $Z = 4$. Observed and calculated densities are 1.45 ± 0.03 and 1.496 g cm^{-3}, respectively. A single-crystal X-ray diffraction study has been completed. Data to $\sin \theta = 0.38$ (Mo Kα radiation) were collected with a 0.01°-incrementing Buerger automated diffractometer, and the structure was solved using conventional Patterson, Fourier, and least-squares refinement techniques. All atoms have been located, the final discrepancy index being $R_F = 7.95\%$ for the 1162 independent nonzero reflections. The crystal consists of distinct molecular units of $(C_{10}H_8)_2Fe$, separated by normal van der Waals distances. The molecule is found to be a highly substituted ferrocene in which two azulene systems have dimerized *via ortho-para* (*i.e.*, 4-*endo*,6′-*endo*) coupling.

Introduction

Our recent series of structural studies on the azulene–metal–carbonyl complexes $C_{10}H_8Fe_2(CO)_5$,[1] $C_{10}H_8Mo_2(CO)_6$,[2] $[(i\text{-}C_3H_7)(CH_3)_2C_{10}H_5]Mo_2(CO)_6$,[2a,3] $C_{10}H_8Mn_2(CO)_6$,[4] $[C_{10}H_8Mo(CO)_3CH_3]_2$,[5] $[(CH_3)_3C_{10}H_5]Ru_4(CO)_9$,[6] and $(C_{10}H_8)_2Fe_4(CO)_{10}$[7] has indicated that a dominant feature of the azulene-to-metal bonding in these complexes is the utilization of the five-membered ring in a π-cyclopentadienyl→metal linkage. The only other presently known class of azulene–metal complexes has as its sole representative[8] the compound bis(π-azulene)iron.[9] On the basis of its proton magnetic resonance spectrum and hydrogenation studies (the complex takes up 5 mol of hydrogen), this species has been represented as a complex stabilized by metal–diene and metal–triene bonding (as in I).[9]



I

We have therefore undertaken an X-ray structural analysis of bis(π-azulene)iron, with a view to determining unequivocally its molecular structure. A brief report of this work has appeared previously.[10]

Experimental Section

Bis(π-azulene)iron was prepared *via* the "tris(isopropyl)iron" method,[11] following the procedure of Fischer and Müller.[9] Anhydrous $FeCl_3$ (1.3 g) and azulene (2 g) in dry diethyl ether (35 ml) were treated at −50° under N_2 with isopropylmagnesium bromide,

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[1] M. R. Churchill, *Chem. Commun.*, 450 (1966); *Inorg. Chem.*, **6**, 190 (1967).

[2] (a) M. R. Churchill and P. H. Bird, *Chem. Commun.*, 746 (1967); (b) J. S. McKechnie and I. C. Paul, *ibid.*, 747 (1967).

[3]. M. R. Churchill and P. H. Bird, *Inorg. Chem.*, **7**, 1545 (1968).

[4] P. H. Bird and M. R. Churchill, *Chem. Commun.*, 145 (1968); M. R. Churchill and P. H. Bird, *Inorg. Chem.*, **7**, 1793 (1968).

[5] P. H. Bird and M. R. Churchill, *Chem Commun.*, 705 (1967); *Inorg. Chem.*, **7**, 349 (1968).

[6] M. R. Churchill and P. H. Bird, *J. Am. Chem. Soc*,. **90**, 800 (1968).

[7] M. R. Churchill and P. H. Bird, *ibid.*, **90**, 3241 (1968).

[8] There are, however, two known azulenium complexes of chromium—$Cr(C_{10}H_9)_2$[9] and π-$C_5H_5Cr(C_{10}H_9)$ [E. O. Fischer and S. Breitschaft, *Ber.*, **96**, 2451 (1963)]—and a number of azulenium complexes of iron, including $Fe(C_{10}H_9)_2$ [G. R. Knox and P. L. Pauson, *J. Chem. Soc.*, 4610 (1961)].

[9] E. O. Fischer and J. Müller, *J. Organometal. Chem.* (Amsterdam), **1**, 464 (1964).

[10] M. R. Churchill and J. Wormald, *Chem. Commun.*, 1033 (1968).

[11] E. O. Fischer and J. Müller, *Z. Naturforsch.*, **17b**, 776 (1962).

previously prepared from isopropyl bromide (16 ml) and magnesium turnings (4 g) in dry diethyl ether (45 ml). The solution was allowed to warm to room temperature and was irradiated with a sunlamp for 15 hr. Gas evolution was observed and a red-brown solution was obtained. Solvolysis of excess Grignard reagent at −50° with 100 ml of 50:50 methanol–diethyl ether was followed by filtration, evaporation, and redissolution in benzene. Chromatography on a 30-cm column of Woelm grade III alumina, with hexane as eluent, yielded the four bands described by Fischer and Müller:[9] yellow, blue, pale orange, and bright orange (in that order). The last band was collected, concentrated, and rechromatographed. Evaporation and slow cooling of the solution in a refrigerator yielded *ca.* 100 mg of red-brown crystals (5% yield based on $FeCl_3$). The identity of the product as $C_{20}H_{16}Fe$ was confirmed by a qualitative mass spectral examination, which showed strong peaks at *m/e* 312, 184, 128, and 56, consistent with $C_{20}H_{16}Fe^+$, $C_{10}H_8Fe^+$, $C_{10}H_8^+$, and Fe^+, respectively.

Crystals suitable for an X-ray crystallographic analysis were obtained by the slow cooling of a saturated solution of the complex in pentane. The red-brown crystals thus obtained are air stable and are not X-ray sensitive.

Unit Cell and Space Group

Optical examination and the observed reciprocal lattice symmetry (C_{2h}; 2/m) indicated that the crystals belonged to the monoclinic system. Unit cell dimensions, obtained by a least-squares analysis of high-angle precession data, taken with Mo Kα radiation ($\bar{\lambda}$ 0.7107 Å) at 24 ± 2° and calibrated with sodium chloride (a_{NaCl} = 5.640 Å) are: a = 7.523 (5) Å, b = 12.071 (4) Å, c = 15.419 (16) Å, β = 98.26 (4)°. The unit cell volume is 1386 Å^3.

A survey of 0*kl*, 1*kl*, *h*0*l*, *h*1*l*, *hk*0, and *hk*1 precession photographs revealed the systematic absences *h*0*l* for $l = 2n + 1$ and 0*k*0 for $k = 2n + 1$, compatible only with space group P2_1/c (C_{2h}^5; no. 14).[12] The observed density (ρ_{obsd} = 1.45 ± 0.03 g cm^{-3}, by flotation in aqueous zinc iodide solution) is consistent with four molecules per unit cell (ρ_{calcd} = 1.496 g cm^{-3} for M = 312.18, Z = 4). The space group imposes no specific symmetry on the molecule.

Collection and Reduction of the X-Ray Diffraction Data

The crystal used during the crystallographic analysis was a well-formed parallelepiped with large and distinct (100), (011), and (01$\bar{1}$) faces. Its dimensions were 0.16 × 0.40 × 0.38 mm (referring sequentially to the 100, 011, 01$\bar{1}$ directions). It was mounted initially along its *b* axis and was later remounted along its *a* direction. [Damage caused in transferring the crystal from a *b* to an *a* mounting created a rather irregular new face and reduced the volume of the crystal, *vide infra*.]

Intensity data (Mo Kα radiation, $\bar{\lambda}$ 0.7107 Å) were collected with a 0.01°-incrementing Supper-Pace Buerger automated diffractometer, using the stationary-background, ω-scan, stationary-background counting sequence that has been described in detail in a previous publication.[13] Experimental details specific to the present structural investigation follow. (i) The X-ray generator was stabilized at 49.5 kV/19.6 mA. (ii) Owing to the relatively large mosaic spread of the diffraction data, the angle scanned (ω) was chosen as $\omega = [3.0 + (1.0/L)]°$, where 1/L is the Lorentz factor. (iii) The speed of the ω scan was 2°/min. (iv) Initial and final backgrounds (B_1 and B_2) were each measured for one-fourth the time of the main scan (C counts). (v) Within each level, a carefully preselected check reflection was remeasured after each batch of 20 reflections had been collected. (No significant, *i.e.*, $\geqslant 3(\text{count})^{1/2}$, variations from the mean were detected. This confirms the stability of the electronics and the continued alignment of the crystal.) (vi) $I(hkl)$, the intensity of the reflection *hkl*, was calculated as $I(hkl) = C(hkl) - 2[B_1(hkl) + B_2(hkl)]$.

Using equiinclination Weissenberg geometry, data for the quadrants *hKl* and *hK*$\bar{l}$ (K = 0–12) were collected from the *b*-mounted crystal, and data for the quadrants *Hkl* and *Hk*$\bar{l}$ (H = 0–5) were collected from the *a*-mounted crystal. [The first set of data is complete to sin θ = 0.38, save for the few reflections in the range $0 < \theta < 4°$ which are shielded from the counter by a lead backstop. Long-exposure Weissenberg photographs had shown previously that there were few observable data beyond sin θ = 0.38.]

Data were assigned standard deviations according to the following scheme {where $\delta(hkl) = 3[C(hkl) + B_1(hkl) + B_2(hkl)]^{1/2}$—*i.e.*, the maximum probable error based solely on counting statistics}: $I(hkl) \geqslant 1225$, $\sigma(hkl) = 0.1[I(hkl)]$; $1225 > I(hkl) \geqslant \delta(hkl)$, $\sigma(hkl) = 3.5[I(hkl)]^{1/2}$; $I(hkl) < \delta(hkl)$, reflection rejected.

Of the 1834 reflections obtained from the *b*-mounted crystal, 700 were rejected; of 1482 reflections obtained from the *a*-mounted crystal, 581 were rejected. Intensities were corrected for Lorentz and polarization effects $[(Lp)^{-1} = 2\cos^2\bar{\mu}\sin\Upsilon/(1 + \cos^2 2\theta)]$[14] and an absorption correction was applied[15] (μ = 10.76 cm^{-1} for Mo Kα radiation); calculated transmission coefficients ranged from 0.766 to 0.848 for the *b*-mounted crystal (volume 0.0119 mm^3) and from 0.772 to 0.851 for the *a*-mounted crystal (volume 0.0072 mm^3). The 19 zones of data were merged to a common scale by a least-squares analysis of common reflections.[16] The resulting 1162 independent nonzero reflections were used in a Wilson plot,[17] from which the approximate absolute scale and the over-all isotropic thermal parameter ($\bar{B}$ = 1.52 Å^2) were obtained.

Elucidation and Refinement of the Structure

A three-dimensional Patterson synthesis,[18] which

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(12) "International Tables for X-Ray Crystallography," Vol. I, The Kynoch Press, Birmingham, England, 1965, p 99.

(13) M. R. Churchill and J. P. Fennessey, *Inorg. Chem.*, **7**, 1123 (1968).

(14) $\bar{\mu}$ is the equiinclination angle, Υ is the vertical Weissenberg coordinate (in degrees), and θ is the Bragg angle.

(15) Using the general absorption program GNABS, by C. W. Burnham.

(16) A. D. Rae, *Acta Cryst.*, **19**, 683 (1965).

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

(18) All crystallographic routines (including Fourier syntheses, structure factor calculations, least-squares refinement routines, and distance angle plane calculations) were performed using the CRYRM system—an integrated set of crystallographic routines for the IBM 7094, written by Professor R. E. Marsh and his coworkers at the California Institute of Technology.

Table I

Observed and Calculated Structure Factors (×10) for $(C_{10}H_8)_2Fe$

K	L	FO	FC
H = -8			
0	2	211	232
0	4	153	97
H = -7			
0	6	235	224
0	8	195	215
0	10	164	154
1	0	240	221
1	1	142	113
1	2	145	136
1	4	214	210
1	6	155	157
1	8	121	116
1	9	143	103
1	10	115	112
2	0	149	142
2	2	134	88
2	3	241	232
2	5	135	125
2	6	216	213
2	7	183	146
3	1	199	204
3	5	212	192
3	9	199	221
4	3	256	260
4	7	351	344
5	1	137	124
5	4	119	101
5	5	217	224
6	1	116	76
6	2	137	150
6	3	139	144
H = -6			
0	4	294	313
0	6	133	151
0	8	143	157
0	10	122	88
0	12	129	81
1	0	122	102
1	3	170	171
1	5	119	87
1	6	198	195
1	10	155	142
2	0	236	220
2	4	139	151
2	5	133	89
2	9	138	156
2	12	141	121
3	1	194	196
3	3	294	310
3	5	168	193
3	7	304	301
3	11	127	111
4	1	158	134
4	3	140	142
4	5	284	275
4	7	155	96
4	9	145	165
5	1	162	98
5	3	245	242
5	5	127	156
5	7	218	199
5	10	135	117
6	2	132	87
6	3	137	117
6	4	212	221
6	5	169	164
6	7	145	65
7	2	189	183
7	4	122	70
7	6	220	190
7	7	155	108
8	4	273	247
H = -5			
0	0	121	34
0	2	249	306
0	6	108	119
0	10	180	128
0	14	137	26
1	0	375	360
1	2	244	258
1	4	398	395
1	5	124	88
1	7	122	144
1	8	181	181
1	14	122	121
2	0	321	332
2	1	313	319
2	2	282	310
2	3	231	245
2	5	157	173
2	8	143	138
2	9	109	66
2	10	138	71
2	12	126	130
3	1	216	227
3	3	362	384
3	4	111	72
3	7	244	258
3	10	112	93
3	11	196	228
3	13	203	165
4	0	135	167
4	1	447	447
4	3	160	146
4	4	175	202
4	5	475	479
4	6	124	133
4	7	205	200
4	9	132	122
4	10	139	101
4	13	234	210
5	0	191	166
5	1	134	136
5	3	324	334
5	4	163	49
5	5	167	162
5	10	114	71
5	11	199	216
5	12	154	140
6	2	197	212
6	5	144	41
6	6	206	165
6	8	159	126
6	10	145	126
7	0	138	136
7	3	121	112
7	4	235	244
7	8	132	110
7	9	194	161
8	1	190	197
8	2	238	221
8	3	306	303
8	6	209	190
8	9	156	103
9	0	204	201
9	1	130	68
9	3	119	130
9	4	111	121
9	5	135	142
9	6	198	182
10	0	185	162
10	1	218	200
10	3	165	149
H = -4			
0	2	694	722
0	4	481	496
0	6	382	433
0	8	344	380
0	10	173	186
0	14	237	280
1	0	536	535
1	1	334	311
1	2	393	448
1	3	99	77
1	4	339	354
1	6	220	250
1	7	142	139
1	8	304	371
1	10	242	272
1	14	124	88
2	0	119	116
2	2	252	277
2	3	334	380
2	4	186	197
2	5	226	255
2	6	140	173
2	7	371	454
2	8	127	85
2	10	195	213
2	11	298	306
2	14	129	90
3	0	476	478
3	1	951	997
3	3	182	166
3	5	448	598
3	8	151	161
3	9	324	303
3	11	114	120
3	13	265	274
4	0	442	495
4	1	128	149
4	2	98	118
4	3	564	587
4	4	169	171
4	6	103	112
4	7	281	334
4	9	139	50
4	11	437	457
5	0	237	271
5	1	600	559
5	2	171	165
5	4	346	309
5	5	279	266
5	8	162	150
5	9	304	282
5	10	124	135
5	13	284	274
6	0	388	401
6	1	107	77
6	3	192	182
6	4	156	161
6	5	100	83
6	9	176	194
6	10	171	166
6	11	209	223
6	12	209	226
7	0	326	333
7	1	110	126
7	2	255	267
7	4	228	201
7	6	135	147
7	8	188	168
7	10	154	136
7	12	208	190
8	0	327	315
8	1	144	81
8	2	257	264
8	3	164	159
8	4	186	176
8	5	150	135
8	6	233	259
8	7	142	141
8	8	158	183
8	9	143	137
8	10	194	163
9	0	177	206
9	1	240	231
9	2	168	153
9	3	131	140
9	4	173	167
9	6	123	112
9	8	193	163
9	9	208	202
10	0	156	134
10	1	105	80
10	3	342	324
10	6	153	168
10	7	285	274
11	1	220	234
11	2	103	58
H = -3			
0	2	99	94
0	4	1049	1021
0	6	316	302
0	8	689	609
0	10	213	210
0	12	227	210
0	14	116	127
1	0	140	158
1	1	223	199
1	2	287	305
1	4	124	114
1	5	346	323
1	6	767	746
1	7	325	328
1	8	123	55
1	9	189	179
1	10	516	460
1	13	221	201
1	14	225	217
2	0	515	529
2	1	227	224
2	2	149	155
2	3	186	207
2	4	580	582
2	5	113	127
2	6	121	109
2	7	333	331
2	8	438	432
2	9	241	252
2	10	133	133
2	11	207	239
2	12	136	148
2	13	170	135
3	0	322	226
3	1	443	456
3	2	382	365
3	3	467	486
3	4	347	341
3	5	343	356
3	6	92	67
3	7	631	635
3	8	187	188
3	9	235	234
3	10	136	139
3	11	276	293
3	13	185	164
3	15	126	88
4	0	165	150
4	1	630	632
4	3	524	547
4	5	398	424
4	6	98	113
4	7	417	420
4	9	450	437
4	11	241	240
4	13	247	206
4	15	149	119
5	0	348	330
5	1	533	575
5	2	95	102
5	3	257	240
5	4	227	215
5	5	142	150
5	7	217	213
5	9	180	171
5	10	285	276
5	11	200	220
5	14	121	104
6	0	637	655
6	1	135	169
6	3	126	87
6	4	217	230
6	5	107	130
6	6	119	75
6	7	271	264
6	8	296	300
6	9	282	290
6	12	174	147
7	2	378	372
7	6	281	299
7	8	96	100
7	10	304	269
8	0	342	352
8	2	91	92
8	3	143	135
8	4	301	287
8	6	157	156
8	7	216	207
8	8	215	192
8	12	203	187
9	0	127	146
9	1	185	197
9	2	162	179
9	3	145	189
9	5	151	168
9	6	278	285
9	7	130	122
9	8	106	54
9	9	143	143
9	10	192	188
10	3	185	183
10	5	184	170
10	7	139	131
10	8	126	139
10	9	120	136
11	1	175	173
11	3	183	201
11	5	176	181
11	6	104	51
H = -2			
0	2	766	743
0	4	359	360
0	6	931	886
0	8	155	173
0	10	383	388
0	12	182	189
0	14	346	330
1	1	557	519
1	2	316	309
1	3	285	255
1	4	824	817
1	5	255	252
1	7	191	211
1	8	251	253
1	10	134	122
1	12	322	315
1	13	143	52
1	16	231	217
2	0	1338	1384
2	2	444	381
2	3	107	125
2	4	209	197
2	5	502	523
2	6	188	207
2	7	185	184
2	8	194	204
2	9	269	275
2	10	267	276
2	11	124	117
2	12	184	180
2	13	261	263
2	16	126	67
3	0	343	369
3	1	379	373
3	3	532	527
3	4	280	298
3	5	220	230
3	6	260	262
3	7	398	412
3	9	124	129
3	10	175	172
3	11	199	194
3	15	141	142
4	0	331	319
4	1	463	449
4	2	399	382
4	3	429	411
4	4	230	231
4	5	368	380
4	6	268	264
4	7	220	233
4	9	248	238
4	10	143	131
4	12	146	135
4	14	119	99
5	0	396	387
5	2	139	111
5	3	344	386
5	4	141	117
5	5	175	161
5	7	745	737
5	8	245	293
5	9	225	185
5	10	94	85
5	15	124	77
6	0	107	116
6	1	409	392
6	2	302	324
6	4	239	215
6	5	96	90
6	6	445	430
6	8	165	163
6	9	112	50
6	10	253	253
6	14	125	98
7	0	443	447
7	2	92	85
7	3	160	177
7	4	361	353
7	6	142	140
7	7	109	89
7	8	246	236
7	10	101	82
8	0	284	307
8	2	350	365
8	4	282	271
8	5	216	197
8	6	447	423
8	7	107	100
8	9	157	173
8	10	202	163
9	0	130	129
9	2	98	109
9	3	137	131
9	4	313	319
9	5	133	124
9	7	165	177
10	1	172	154
10	4	100	99
10	5	213	207
10	9	195	162
11	0	95	120
11	2	105	75
11	3	289	303
11	5	174	179
11	6	99	92
11	7	135	108
12	1	101	46
12	5	192	199
H = -1			
0	4	195	189
0	6	271	280
0	10	301	337
0	12	331	345
0	14	366	343
0	16	217	212
1	3	415	400
1	4	895	824
1	5	371	314
1	6	355	360
1	7	171	167
1	8	196	218
1	9	136	105
1	10	155	152
1	11	124	135
1	12	356	382
1	13	184	156
1	14	227	218
1	16	177	130
2	1	449	618
2	2	421	395
2	3	348	315
2	6	210	231
2	10	172	186
2	11	143	170
2	14	309	313
2	15	191	161
3	0	768	774
3	1	537	485
3	2	127	114
3	3	99	86
3	4	94	74
3	5	557	542
3	7	155	161
3	9	199	217
3	11	145	139
3	13	150	133
4	0	72	56
4	1	449	411
4	2	144	138
4	3	1126	1076
4	4	226	234
4	6	129	131
4	7	467	467
4	8	234	248
4	9	135	126
4	10	107	78
4	11	210	193
4	15	193	170
5	1	524	496
5	2	107	101
5	4	440	425
5	5	629	613
5	6	245	247
5	8	237	227
5	9	228	240
5	10	140	141
5	11	123	135
5	13	148	130
6	0	477	490
6	1	222	212
6	2	356	360
6	3	214	210
6	4	380	392
6	5	372	355
6	6	449	470
6	7	423	[illegible]
6	8	298	323
6	9	121	117
6	10	194	217
6	12	115	121
6	14	178	174
7	0	616	625
7	2	371	401
7	3	88	91
7	4	722	677
7	5	122	71
7	6	395	402
7	8	351	326
7	10	135	147
7	12	151	199
8	0	308	289
8	1	124	119
8	2	498	509
8	3	89	102
8	4	473	377
8	5	191	128
8	6	213	214
8	7	211	237
8	9	162	147
8	10	215	215
9	0	246	275
9	1	313	308
9	2	140	54
9	4	378	322
9	5	136	133
9	6	103	98
9	7	128	138
9	8	125	137
9	11	147	110
10	2	225	245
10	3	274	247
10	10	114	19
11	1	378	331
11	2	145	145
11	5	112	108
11	6	153	143
12	3	324	321
12	4	167	174
H = 0			
0	4	1515	1388
0	8	722	691
0	10	260	188
0	12	765	754
0	14	238	184
0	16	219	158
1	3	245	275
1	4	387	329
1	5	117	107
1	6	701	686
1	7	266	178
1	8	239	217
1	9	125	72
1	10	577	506
1	11	185	190
1	12	141	18
1	13	133	33
1	14	362	391
1	15	124	81
2	2	60	24
2	3	1158	1085
2	4	459	434
2	5	244	265
2	7	523	538
2	8	209	335
2	9	255	238
2	11	199	193
2	12	306	317
2	13	184	201
2	14	135	96
2	16	134	126
3	1	959	874
3	2	182	150
3	3	1185	1119
3	4	221	187
3	6	141	117
3	8	133	117
3	9	416	421
3	11	348	374
3	12	272	288
3	13	174	191
3	14	163	184
3	15	211	212
4	1	330	302
4	2	285	249
4	3	748	712
4	4	409	251
4	5	576	540
4	6	114	119
4	7	411	404
4	9	408	388
4	11	226	223
4	13	250	242
5	1	473	460
5	2	370	383
5	3	636	619
5	5	423	404
5	6	529	496
5	7	409	384
5	9	328	328
5	10	369	375
5	11	307	288
5	13	188	174
5	14	238	244
5	15	148	115
6	0	469	476
6	1	255	243
6	2	168	146
6	3	308	305
6	4	598	633
6	5	426	402
6	6	124	81
6	7	242	212
6	8	363	359
6	9	175	181
6	11	256	216
6	12	214	224
6	13	147	168
7	1	104	49
7	2	732	727
7	5	155	92
7	6	617	586
7	7	241	225
7	8	105	8
7	9	179	211
7	10	569	558
7	12	139	97
8	0	745	764
8	1	180	183
8	3	98	58
8	4	521	525
8	5	130	56
8	8	422	428
8	9	127	111
8	11	128	52
8	12	352	349
9	1	176	185
9	2	384	431
9	3	173	73
9	5	239	216
9	6	133	122
9	7	305	347
9	8	136	130
9	9	157	108
9	10	232	235
10	1	337	349
10	2	86	70
10	3	117	96
10	5	151	24
10	6	112	113
10	7	162	160
10	8	121	66
11	1	330	322
11	2	78	39
11	3	287	277
11	4	90	99
11	5	98	109
11	7	122	120
12	1	301	307
12	3	94	49
12	4	243	255
H = 1			
0	4	431	388
0	6	1290	1185
0	8	240	258
0	10	681	735
0	12	167	69
1	3	487	431
1	4	444	435
1	6	514	481
1	7	444	429
1	8	619	599
1	9	176	83
1	10	389	452
1	11	172	138
1	12	340	326
1	14	137	107
2	3	654	585
2	4	128	133
2	5	200	214
2	6	540	523
2	7	117	60
2	9	456	473
2	10	274	274
2	11	152	132
2	12	223	233
2	13	173	183
3	0	845	774
3	1	269	257
3	2	518	497
3	3	633	580
3	5	163	135
3	6	125	117
3	7	724	725
3	8	209	202
3	9	195	189
3	11	294	299
3	13	132	125
3	14	132	137
3	15	115	148
4	1	606	618
4	3	547	511
4	4	444	395
4	5	337	331
4	6	121	108
4	8	302	310
4	9	530	571
4	10	146	144
4	11	150	162
4	13	386	379
5	1	161	162
5	2	290	284
5	3	409	391
5	5	191	190
5	6	237	217
5	7	358	334
5	9	171	174
5	10	146	175
5	11	312	311
5	13	183	182
5	14	127	116
6	0	489	490
6	2	226	235
6	4	462	465
6	5	244	264
6	6	451	459
6	7	181	151
6	8	262	274
6	10	306	358
6	11	141	124
6	12	246	229
6	13	120	33
6	14	217	244
7	0	642	625
7	1	312	303
7	2	132	136
7	4	275	297
7	6	151	145
7	7	112	123
7	8	287	316
7	10	179	201
7	11	128	123
7	12	328	307
8	0	329	289
8	1	169	199
8	2	282	285
8	4	181	175
8	5	164	178
8	6	214	204
8	7	272	244
8	8	278	279
8	10	326	356
9	0	258	275
9	1	73	82
9	2	94	62
9	3	218	228
9	4	132	133
9	5	122	104
9	6	200	204
9	7	160	169
9	8	285	292
9	9	140	92
9	10	125	120
9	11	173	169
10	0	78	29
10	1	218	217
10	5	166	167
10	9	219	206
11	1	114	105
11	2	163	163
11	3	146	145
11	4	114	97
11	6	103	93
11	7	250	258
12	0	151	174
12	1	266	288
12	2	136	129
12	3	91	74
12	4	111	100
12	5	216	192
H = 2			
0	4	199	200
0	6	708	650
0	10	473	446
1	1	141	138
1	3	293	272
1	4	649	595
1	5	272	274
1	6	458	461
1	7	135	124
1	8	671	664
1	9	104	109
1	10	276	299
1	13	119	27
2	0	1435	1384
2	1	282	261
2	2	517	481
2	3	217	217
2	4	92	102
2	5	229	204
2	6	219	231
2	7	481	490
2	9	97	76
2	10	192	190
2	14	135	143
2	15	129	103
3	0	378	369
3	1	505	480
3	2	95	83
3	4	94	93
3	5	490	489
3	7	267	266
3	8	195	203
3	9	453	451
3	11	184	166
3	13	295	302
4	0	326	319
4	1	108	119
4	2	117	97
4	3	366	368
4	5	281	253
4	6	111	106
4	7	440	428
4	10	158	124
4	11	183	193
5	0	395	387
5	1	424	456
5	2	115	113
5	3	188	200
5	4	446	437
5	5	522	504
5	6	113	100
5	7	108	34
5	8	197	189
5	10	147	126
5	12	290	297
5	13	224	210
6	0	136	116
6	1	242	259
6	2	173	178
6	3	97	100
6	5	123	124
6	6	150	159
6	7	142	85
6	9	118	84
6	10	150	157
7	0	442	440
7	1	129	115
7	2	163	181
7	3	158	158
7	4	311	322
7	5	100	61
7	6	138	133
7	7	116	114
7	8	202	204
8	0	292	307
8	1	209	204
8	2	215	206
8	3	290	286
8	4	163	148
8	5	246	222
8	6	383	390
8	8	189	178
8	10	187	209
9	0	112	129
9	3	112	93
9	4	110	103
9	5	174	174
9	8	236	204
9	9	151	135
10	1	110	107
10	3	320	310
10	4	118	114
10	6	124	92
10	7	281	287
11	0	150	120
11	5	149	165
12	2	109	113
12	3	182	188
10	7	290	287
H = 3			
0	0	296	313
0	2	276	264
0	4	942	936
0	6	109	72
0	8	435	477
0	14	127	135
1	0	165	158
1	2	381	383
1	3	161	173
1	4	136	122
1	6	470	475
1	7	184	189
1	8	136	128
1	10	240	251
1	14	210	226
2	0	550	529
2	1	345	307
2	2	91	69
2	3	482	486
2	4	633	644
2	5	373	360
2	6	262	254
2	7	176	177
2	8	377	388
2	9	171	153
2	10	116	118
2	11	156	180
2	12	224	212
2	13	140	120
3	0	340	226
3	1	634	634
3	2	247	254
3	3	492	473
3	5	433	451
3	7	426	418
3	8	176	198
3	11	202	198
3	13	206	207
4	0	146	150
4	1	820	823
4	2	116	127
4	3	772	757
4	4	111	97
4	5	433	423
4	6	113	114
4	7	459	481
4	9	265	268
4	11	299	291
4	13	261	248
5	0	337	330
5	1	401	371
5	2	378	380
5	3	422	467
5	5	241	238
5	6	120	133
5	7	112	103
5	9	176	168
5	10	139	128
5	11	129	101
6	0	630	655
6	3	406	387
6	4	396	421
6	5	220	237
6	8	98	76
6	10	148	145
6	11	119	122
7	2	443	450
7	6	355	366
7	8	116	36
7	9	117	32
7	10	200	180
8	0	337	352
8	1	119	116
8	3	100	76
8	4	317	334
8	7	136	130
8	8	167	159
9	0	146	146
9	1	158	165
9	2	386	401
9	3	310	318
9	6	373	381
9	9	161	159
10	3	210	208
10	4	122	121
10	5	165	163
10	7	229	197
11	1	163	175
11	3	203	201
11	4	106	101
11	5	231	234
H = 4			
0	0	253	195
0	2	439	434
0	4	547	493
0	6	445	447
0	8	131	122
0	10	341	315
1	0	566	535
1	1	223	209
1	2	470	439
1	3	285	273
1	4	396	387
1	6	304	305
1	10	290	279
1	12	284	279
2	0	99	116
2	1	631	651
2	2	480	479
2	4	144	152
2	5	300	339
2	6	209	234
2	7	121	126
2	9	211	253
3	0	488	478
3	2	185	201
3	3	818	829
3	4	115	102
3	6	142	153
3	7	463	536
3	9	144	151
3	10	209	240
3	11	320	359
3	12	174	228
4	0	463	495
4	1	929	886
4	3	266	258
4	4	140	95
4	5	410	410
4	7	166	130
4	9	448	437
5	0	281	271
5	2	293	288
5	3	577	531
5	4	215	207
5	6	166	137
5	7	385	367
5	8	184	163
5	9	133	102
5	11	289	290
6	0	416	401
6	1	407	385
6	2	302	299
6	3	196	196
6	4	177	177
6	6	161	161
6	7	122	58
6	8	201	192
6	9	259	223
6	10	242	215
7	0	343	333
7	1	146	145
7	2	234	220
7	4	266	239
7	5	120	77
7	6	164	154
7	8	184	185
8	0	349	315
8	2	329	325
8	4	311	315
8	5	121	[illegible]
8	6	351	345
8	7	129	86
9	0	217	206
9	3	272	283
9	4	228	219
9	7	147	130
10	0	129	134
10	1	265	259
10	2	163	158
10	5	299	272
H = 5			
0	2	346	365
0	8	132	75
0	10	247	237
1	0	384	360
1	1	112	119
1	2	218	258
1	4	312	317
1	6	174	200
1	7	122	132
1	8	187	202
1	9	120	126
1	10	149	119
1	11	134	154
2	0	313	332
2	1	289	276
2	3	276	219
2	5	116	89
2	9	192	186
2	10	187	203
3	1	352	359
3	2	149	141
3	3	136	119
3	5	328	338
3	7	168	125
3	9	373	378
4	1	139	147
4	2	192	189
4	3	230	247
4	4	122	37
4	5	112	142
4	6	130	139
4	7	440	459
5	0	193	166
5	1	278	200
5	7	157	170
5	8	199	195
5	9	388	374
6	1	112	122
6	5	277	256
6	7	157	108
7	0	130	135
7	3	155	123
7	4	244	199
7	5	114	55
8	2	178	136
8	5	115	106
8	6	210	175
9	0	218	201
9	3	117	115
9	4	144	162
10	0	170	162
H = 6			
0	0	413	417
0	4	352	345
0	6	143	74
0	8	325	323
1	0	132	102
1	2	299	268
1	6	216	190
1	9	176	167
2	0	199	220
2	2	121	45
2	3	232	218
2	4	139	83
2	7	251	256
2	8	171	145
3	3	110	173
3	5	190	186
3	7	138	129
4	2	159	164
4	3	218	218
4	5	170	187
4	7	253	235
5	5	200	203
5	6	149	149
5	7	191	155
7	2	126	93
8	0	136	115
H = 7			
0	0	417	390
0	2	377	374
0	4	133	17
1	0	240	221
1	2	214	195
1	3	131	147
1	4	156	155
2	0	190	142
2	2	162	160
2	4	137	137
3	0	146	43
3	3	374	364
4	1	215	183
5	0	147	125

had been sharpened such that the average intensity was invariant with θ, revealed a set of vectors consistent with an iron position at $x = 0.1675$, $y = 0.1825$, and $z = 0.1238$. A three-dimensional "difference-Fourier" synthesis[18] phased only by the iron ($R_F = 41.3\%$)[19] revealed the approximate positions for the 10 carbon atoms of one azulene ligand. A second "difference-Fourier" synthesis[18] phased now by the iron and 10 carbon atoms ($R_F = 34.3\%$) revealed apparent positions for the remaining 10 carbon atoms. Two cycles of refinement[18] of positional and individual isotropic thermal parameters led to a reduction in the discrepancy index (R_F) from 26.7 to 18.6%. It became clear at this stage that two carbon atoms were incorrectly positioned; these atoms were placed in their correct positions (by means of a third "difference-Fourier" synthesis) and two further cycles of isotropic refinement resulted in convergence at $R_F = 14.6\%$ and $R_{wF^2} = 11.1\%$.[19] A difference-Fourier map at this stage showed no evidence for hydrogen atoms and there was a variety of miscellaneous background features having amplitudes up to 0.8 e$^-$ Å^{-3}.

The 20 hydrogen atoms were now placed in calculated positions[20] (with each $B = 4.0$ Å^2). Three cycles of full-matrix refinement[18] of individual positional and anisotropic thermal parameters led to convergence [*i.e.*, (suggested shift/σ) < 0.05] at $R_F = 7.95\%$ and $R_{wF^2} = 3.94\%$. A final attempt was now made to locate the hydrogens—a three-dimensional difference Fourier, phased by all nonhydrogen atoms ($R_F = 9.33\%$, $R_{wF^2} = 5.89\%$), showed peaks (ranging from 0.28 to 0.61 e$^-$ Å^{-3} in height) at each of the calculated hydrogen atom positions. [A final "observed" electron density synthesis showed carbon atom peaks with maxima ranging from 5.0 to 6.9 e Å^{-3}.] Hamilton R-factor ratio tests confirm that both anisotropic thermal parameter refinement and inclusion of the hydrogen atoms result in an improvement that is significant with a level of confidence greater than 99.5%.

Throughout the analysis, the residual $\Sigma w(|F_o|^2 - |F_c|^2)^2$

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

(20) With d(C–H) = 1.080 Å and the appropriate idealized sp^2 or sp^3 geometry.

TABLE II

FINAL POSITIONAL PARAMETERS (WITH ESD'S) FOR $(C_{10}H_8)_2Fe^{a-c}$

Atom	x	y	z
Fe	0.16143 (21)	0.18035 (12)	0.12512 (10)
C(1)	0.2905 (17)	0.0327 (10)	0.1379 (8)
C(2)	0.3685 (20)	0.0982 (12)	0.0780 (8)
C(3)	0.4289 (16)	0.1965 (9)	0.1202 (7)
C(4)	0.4468 (15)	0.2811 (8)	0.2794 (7)
C(5)	0.5257 (16)	0.2328 (11)	0.3661 (8)
C(6)	0.4654 (19)	0.1467 (11)	0.4096 (8)
C(7)	0.3242 (18)	0.0680 (9)	0.3804 (9)
C(8)	0.2567 (17)	0.0416 (9)	0.2990 (9)
C(9)	0.3121 (14)	0.0875 (8)	0.2212 (7)
C(10)	0.3957 (14)	0.1929 (8)	0.2097 (7)
C(1′)	−0.1072 (16)	0.1708 (9)	0.1375 (9)
C(2′)	−0.0832 (18)	0.1826 (11)	0.0483 (10)
C(3′)	0.0098 (16)	0.2832 (10)	0.0356 (8)
C(4′)	0.1616 (17)	0.4332 (8)	0.1403 (9)
C(5′)	0.2661 (16)	0.4485 (8)	0.2137 (8)
C(6′)	0.3033 (15)	0.3736 (9)	0.2922 (8)
C(7′)	0.1431 (17)	0.3324 (9)	0.3271 (8)
C(8′)	0.0019 (16)	0.2797 (8)	0.2794 (8)
C(9′)	−0.0203 (15)	0.2648 (9)	0.1847 (8)
C(10′)	0.0521 (16)	0.3334 (8)	0.1214 (7)
H(1)	0.2332	−0.0457	0.1144
H(2)	0.3756	0.0776	0.0105
H(3)	0.4903	0.2565	0.0819
H(4)	0.5483	0.3262	0.2510
H(5)	0.6466	0.2721	0.3980
H(6)	0.5322	0.1347	0.4758
H(7)	0.2666	0.0256	0.4314
H(8)	0.1510	−0.0196	0.2903
H(1′)	−0.1780	0.0990	0.1565
H(2′)	−0.1321	0.1252	−0.0033
H(3′)	0.0356	0.3048	−0.0294
H(4′)	0.1549	0.4969	0.0908
H(5′)	0.3370	0.5265	0.2195
H(6′)	0.3648	0.4260	0.3448
H(7′)	0.1375	0.3451	0.3960
H(8′)	−0.1010	0.2466	0.3141

[a] Estimated standard deviations, shown in parentheses, are right-adjusted to the least significant digit of the preceding number [b] Positions for hydrogen atoms are calculated as described in footnote 20. [c] A hydrogen atom is numbered similarly to the carbon atom to which it is attached.

was minimized. At the completion of the analysis, the standard error in an observation of unit weight was 2.1.

Scattering factors for neutral carbon and hydrogen[21a] were used; the Thomas–Fermi–Dirac values for neutral iron[21b] were corrected for dispersion ($\Delta f' = +0.4$ e$^-$, $\Delta f'' = +1.0$ e$^-$).[21c] Observed and calculated structure factors are collected in Table I. Final atomic positions are shown in Table II. Thermal parameters are given in Table III; their associated vibration ellipsoids are listed in Table IV and are displayed pictorially[22] in Figure 1.

The Molecular Structure

Interatomic distances, with their estimated standard deviations (esd's), are collected in Table V; bond angles (with esd's) are shown in Table VI. The scheme used for numbering atoms is illustrated in Figure 1 (hydrogen atoms, which are omitted from this diagram for the sake of clarity, are numbered similarly to the carbon atoms to which they are attached).

The over-all molecular structure of bis(π-azulene)iron differs considerably from that predicted on the basis of proton magnetic resonance and hydrogenation studies (*i.e.*, I). The molecule is, in fact, a substituted ferrocene in which two azulene systems have coupled *via ortho-para* (*i.e.*, 4-*endo*,6′-*endo*) carbon–carbon bond formation. Thus the iron atom is directly associated only with the two *five-membered* rings.

The molecule possesses asymmetric centers at C(4) and C(6′) and is not superposable on its mirror image. However, the *c*-glide operation of the space group $P2_1/c$ results in there being equal numbers of molecules of each hand. The crystal thus consists of racemic bis(π-azulene)iron.

Stereochemistry of the Ferrocene Moiety

The π-cyclopentadienyl system defined by C(1)–C(2)–C(3)–C(10)–C(9) has a root-mean-square (rms) deviation of 0.015 Å from the least-squares plane $0.8636X - 0.4268Y + 0.2679Z - 2.0408 = 0$;[23] the system C(1′)–C(2′)–C(3′)–C(10′)–C(9′) has an rms deviation of 0.010 Å from the plane $0.8478X - 0.5040Y + 0.1644Z + 1.6257 = 0$[23] [see Table VII]. Individual carbon–carbon bond distances within these systems range from 1.397 ± 0.018 to 1.452 ± 0.016 Å and average 1.430 Å. [This average carbon–carbon distance is in extremely good agreement with the value of 1.431 ± 0.005 Å from electron diffraction studies on ferrocene[24] and is slightly greater than the average value of 1.419 Å found from X-ray studies on π-cyclopentadienyl compounds.[25] Possibly, this reflects the reduced libration in the present more rigidly held π-cyclopentadienyl systems.] C–C–C bond angles within the two five-membered rings vary from 106.2 ± 1.1 to 110.6 ± 1.3°, averaging 108.0°—the ideal angle for a regular, planar pentagon. None of the observed bond distances or bond angles is significantly different (at the $>2\sigma$ level) from the mean values.

Individual iron–carbon distances vary from 2.018 ± 0.011 to 2.072 ± 0.012 Å, averaging 2.044 Å. [This may be compared with the "best" distance of 2.058 ± 0.002 Å found for ferrocene[24] and with values ranging from

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(21) "International Tables for X-Ray Crystallography," Vol. III, The Kynoch Press, Birmingham, England, 1962: (a) p 202; (b) p 211; (c) p 216.

(22) Atomic vibration ellipsoids have been drawn using OTLIPS, a FORTRAN II program for the IBM 1620/CALCOMP-plotter system, written by Dr. P. H. Bird.

(23) Cartesian coordinates; see Table VII.

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TABLE III

ANISOTROPIC THERMAL PARAMETERS FOR $(C_{10}H_8)Fe$[a,b]

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	136.3 (4.0)	46.5 (1.2)	29.7 (0.9)	41.7 (3.5)	−23.5 (2.5)	−12.2 (1.7)
C(1)	234 (36)	85 (11)	32 (6)	156 (30)	−76 (22)	−48 (14)
C(2)	372 (43)	127 (13)	20 (7)	299 (35)	−19 (26)	−36 (16)
C(3)	147 (25)	94 (11)	25 (6)	137 (25)	21 (19)	5 (14)
C(4)	135 (26)	60 (8)	30 (6)	−25 (22)	29 (19)	−1 (11)
C(5)	145 (29)	90 (11)	44 (7)	−33 (28)	−45 (23)	−11 (15)
C(6)	281 (38)	100 (13)	24 (6)	102 (34)	−16 (23)	−6 (14)
C(7)	229 (34)	55 (8)	45 (8)	63 (28)	70 (25)	41 (14)
C(8)	253 (34)	48 (8)	41 (7)	−13 (26)	43 (25)	10 (14)
C(9)	92 (24)	50 (8)	31 (6)	29 (21)	−23 (17)	−16 (11)
C(10)	101 (23)	54 (8)	34 (6)	42 (21)	13 (18)	17 (11)
C(1′)	147 (28)	47 (8)	59 (8)	−1 (23)	−46 (22)	−21 (14)
C(2′)	202 (32)	81 (11)	62 (9)	65 (31)	−112 (25)	−6 (17)
C(3′)	190 (30)	87 (11)	39 (7)	112 (27)	−7 (23)	5 (14)
C(4′)	151 (29)	42 (8)	67 (9)	46 (24)	48 (25)	25 (13)
C(5′)	157 (29)	43 (8)	45 (7)	9 (23)	29 (23)	15 (12)
C(6′)	172 (29)	39 (8)	48 (7)	21 (23)	21 (22)	−14 (12)
C(7′)	176 (28)	50 (8)	44 (6)	0 (25)	35 (22)	−3 (13)
C(8′)	147 (27)	48 (8)	47 (7)	30 (23)	55 (22)	18 (12)
C(9′)	138 (26)	52 (8)	42 (7)	66 (23)	−15 (20)	5 (12)
C(10′)	189 (28)	48 (8)	30 (6)	38 (24)	24 (19)	4 (11)

[a] The anisotropic thermal parameter, T, is defined as $T = \exp(-\beta_{11}h^2 - \beta_{22}k^2 - \beta_{33}l^2 - \beta_{12}hk - \beta_{13}hl - \beta_{23}kl)$. [b] Esd's are given in parentheses.

2.01 to 2.10 Å found for ferrocene derivatives[26–50]—see Table VIII.]

The relative orientation of the two π-cyclopentadienyl systems in $(C_{10}H_8)_2Fe$ is pictured in Figure 2, from which it can be seen that the ferrocene moiety adopts an almost perfectly staggered configuration. [The angular rotation from the strictly eclipsed configuration (*i.e.*, ω) is 35.8°.] Since (in the crystalline state) ferrocene adopts a staggered configuration, whereas ruthenocene[51] and osmocene[52] each adopt an eclipsed configuration, it has become generally believed that the minimization of transannular carbon · · · carbon repulsions is the dominating feature in the stereochemistry of bis(π-cyclopentadienyl) derivatives of the first-row transition metals. However, a survey of structural studies[24,26–50] of ferrocene derivatives shows that the picture is nowhere nearly as simple as that generally accepted. Thus, the most recent (and most accurate) electron diffraction studies on ferrocene (in the vapor phase at 140°)[24,29] are found to be consistent with the molecule having an *eclipsed* equilibrium conformation, with a barrier to rotation of 0.9 ± 0.3 kcal/mol. In the solid phase, certain substituted ferrocenes have a strictly staggered conformation (*i.e.*, ω = 36 °—see Table VIII), some are obviously forced by steric requirements to take up a configuration other than the strictly staggered conformation—*viz.*, 1,1′:3,3′-bis(trimethylene)ferrocene, ($\omega = \sim 0°$),[47] $[C_5H_5FeC_5H_4C(CH_3)_2]_2$ (ω = 9–10°),[48] but there are a number of molecules (including $C_5H_5FeB_9C_2H_{11}$[35] and terferrocenyl[36]) in which a ferrocene moiety adopts an eclipsed configuration without there being any obvious intramolecular steric requirement for it to do so. It is apparent, then, that the observed conformation of a ferrocene derivative is probably the result of a balance between *intra*molecular carbon · · · carbon repulsions and *inter*molecular packing

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TABLE IV

DIRECTION COSINES[a] FOR THE ATOMIC VIBRATION ELLIPSOIDS[b,c] IN $(C_{10}H_8)_2Fe$

Atom	B_{max} (dc's major axis)	B_{med} (dc's median axis)	B_{min} (dc's minor axis)
Fe	4.50	2.29	2.00
	(−0.723, −0.463, 0.611)	(−0.066, 0.785, 0.619)	(0.688, −0.412, 0.493)
C(1)	9.69	2.30	1.81
	(−0.703, −0.597, 0.484)	(−0.586, 0.794, 0.244)	(0.403, 0.113, 0.841)
C(2)	12.79	2.63	1.45
	(−0.735, −0.668, 0.222)	(0.627, −0.602, 0.399)	(−0.259, 0.437, 0.890)
C(3)	7.06	2.35	1.64
	(−0.541, −0.840, 0.027)	(−0.015, −0.052, 0.990)	(−0.841, 0.541, 0.136)
C(4)	3.75	2.93	2.53
	(0.507, −0.841, 0.112)	(0.455, 0.445, 0.697)	(−0.732, −0.306, 0.708)
C(5)	5.57	5.37	2.10
	(−0.674, 0.269, 0.777)	(−0.027, −0.939, 0.342)	(0.738, 0.212, 0.528)
C(6)	8.17	4.31	2.09
	(−0.767, −0.633, 0.220)	(−0.622, 0.774, 0.206)	(0.160, −0.022, 0.954)
C(7)	6.42	3.67	2.11
	(0.645, 0.471, 0.503)	(−0.761, 0.322, 0.666)	(0.071, −0.821, 0.550)
C(8)	5.63	3.86	2.60
	(0.975, −0.070, 0.070)	(−0.173, 0.364, 0.930)	(−0.141, −0.929, 0.360)
C(9)	4.15	2.37	1.58
	(−0.515, −0.569, 0.709)	(−0.228, 0.812, 0.564)	(0.827, −0.130, 0.423)
C(10)	3.86	2.97	1.75
	(0.250, 0.744, 0.578)	(−0.557, −0.413, 0.793)	(0.792, −0.526, 0.194)
C(1′)	6.77	2.93	2.26
	(−0.519, −0.172, 0.903)	(0.641, −0.726, 0.154)	(0.565, 0.665, 0.401)
C(2′)	9.26	4.70	2.00
	(−0.687, −0.219, 0.784)	(0.046, 0.938, 0.333)	(0.725, −0.269, 0.523)
C(3′)	6.80	3.94	2.38
	(−0.658, −0.746, 0.193)	(−0.282, 0.368, 0.917)	(0.698, −0.555, 0.523)
C(4′)	6.48	3.50	1.91
	(0.114, 0.242, 0.937)	(−0.886, −0.415, 0.333)	(0.450, −0.877, 0.101)
C(5′)	4.34	3.45	2.35
	(0.087, 0.284, 0.933)	(−0.994, −0.034, 0.242)	(0.061, −0.958, 0.268)
C(6′)	4.65	3.85	2.04
	(−0.324, −0.253, 0.949)	(0.918, 0.146, 0.232)	(−0.226, 0.956, 0.216)
C(7′)	4.23	3.74	2.93
	(0.457, −0.064, 0.812)	(−0.889, −0.065, 0.577)	(−0.028, 0.996, 0.089)
C(8′)	4.93	2.88	2.43
	(0.376, 0.349, 0.796)	(−0.785, −0.373, 0.603)	(0.493, −0.860, 0.061)
C(9′)	4.81	3.75	1.72
	(−0.677, −0.367, 0.729)	(0.297, 0.657, 0.643)	(0.674, −0.659, 0.235)
C(10′)	4.47	2.83	2.49
	(−0.922, −0.371, 0.026)	(−0.201, 0.223, 0.973)	(0.330, −0.901, 0.230)

[a] Direction cosines (dc's) are referred to the monoclinic axes. [b] Atomic vibration ellipsoids are defined in terms of the isotropic thermal parameter, B. The transformation to root-mean-square displacement $(\bar{u}^2)^{1/2}$ is: $(\bar{u}^2)^{1/2} = (B/8\pi^2)^{1/2}$. [c] Atomic vibration ellipsoids are shown pictorially in Figure 1.

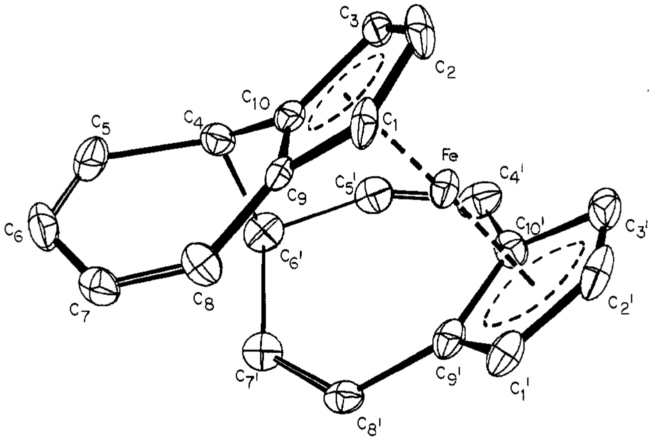

Figure 1.—Numbering of atoms within the $(C_{10}H_8)_2Fe$ molecule. This figure also shows the 68% probability contours of the atomic vibration ellipsoids.[22]

forces. The staggered configuration of bis(π-azulene)iron almost certainly arises to minimize strain in the bridging carbon framework (*vide infra*).

A final point worthy of comment is the dihedral angle (ϕ) of 7° 33′ between the two π-cyclopentadienyl rings in the present molecule. This is probably indicative of some intramolecular strain, since other ferrocene derivatives with $\phi > 7°$ are each believed to have distorted from the parallel conformation owing to *intra*molecular interactions. Thus, 2-biphenylylferrocene ($\phi = 8°$)[45] distorts because of steric hindrance between its bulky 2-biphenylyl substituent and the ferrocene framework, whereas the remaining compounds that have been studied—*i.e.*, α-keto-1,1′-trimethyleneferrocene ($\phi = 8.8°$),[46] 1,1′:3,3′-bis(trimethylene)ferrocene ($\phi = 9°$),[47] and $[C_5H_5FeC_5H_4C(CH_3)_2]_2$ ($\phi = 23.2°$)[48]—are each bent from the parallel comformation in order to

Table V

Intramolecular Distances for $(C_{10}H_8)_2Fe$[a]

Atoms	Dist, Å	Atoms	Dist, Å
(a) Distances within the Ferrocene System			
Fe–C(1)	2.025 (18)	Fe–C(1′)	2.060 (12)
Fe–C(2)	2.063 (14)	Fe–C(2′)	2.040 (13)
Fe–C(3)	2.033 (11)	Fe–C(3′)	2.072 (12)
Fe–C(9)	2.062 (10)	Fe–C(9′)	2.027 (11)
Fe–C(10)	2.042 (10)	Fe–C(10′)	2.018 (11)
C(1)–C(2)	1.406 (18)	C(1′)–C(2′)	1.419 (18)
C(2)–C(3)	1.397 (18)	C(2′)–C(3′)	1.428 (18)
C(3)–C(10)	1.436 (15)	C(3′)–C(10′)	1.447 (16)
C(10)–C(9)	1.441 (14)	C(10′)–C(9′)	1.445 (15)
C(9)–C(1)	1.432 (16)	C(9′)–C(1′)	1.452 (16)
(b) Distances within the Seven-Membered Rings			
C(10)–C(4)	1.524 (14)	C(10′)–C(4′)	1.465 (16)
C(4)–C(5)	1.500 (16)	C(4′)–C(5′)	1.296 (16)
C(5)–C(6)	1.350 (18)	C(5′)–C(6′)	1.503 (16)
C(6)–C(7)	1.447 (18)	C(6′)–C(7′)	1.474 (16)
C(7)–C(8)	1.324 (17)	C(7′)–C(8′)	1.360 (16)
C(8)–C(9)	1.436 (15)	C(8′)–C(9′)	1.456 (16)
(c) The 4–6′ Link			
C(4)–C(6′)	1.584 (15)		

[a] Esd's, shown in parentheses, are right-adjusted to the least significant digit in the preceding number.

Table VI

Bond Angles within the $(C_{10}H_8)_2Fe$ Molecule[a]

Atoms	Angle, deg	Atoms	Angle, deg
(a) Within the Ferrocene System			
C(1)–Fe–C(2)	40.2 (0.5)	C(1′)–Fe–C(2′)	40.5 (0.5)
C(2)–Fe–C(3)	39.9 (0.5)	C(2′)–Fe–C(3′)	40.6 (0.5)
C(3)–Fe–C(10)	41.3 (0.4)	C(3′)–Fe–C(10′)	41.4 (0.5)
C(10)–Fe–C(9)	41.1 (0.4)	C(10′)–Fe–C(9′)	41.9 (0.4)
C(9)–Fe–C(1)	41.0 (0.5)	C(9′)–Fe–C(1′)	41.6 (0.5)
C(1)–C(2)–C(3)	108.1 (1.2)	C(1′)–C(2′)–C(3′)	110.6 (1.3)
C(2)–C(3)–C(10)	109.4 (1.0)	C(2′)–C(3′)–C(10′)	106.2 (1.1)
C(3)–C(10)–C(9)	106.5 (0.9)	C(3′)–C(10′)–C(9′)	108.8 (1.0)
C(10)–C(9)–C(1)	107.0 (0.9)	C(10′)–C(9′)–C(1′)	107.2 (1.0)
C(9)–C(1)–C(2)	108.9 (1.1)	C(9′)–C(1′)–C(2′)	107.2 (1.0)
(b) Within the Seven-Membered Rings			
C(9)–C(10)–C(4)	126.9 (0.9)	C(9′)–C(10′)–C(4′)	126.3 (1.0)
C(10)–C(4)–C(5)	112.6 (0.9)	C(10′)–C(4′)–C(5′)	123.0 (1.1)
C(4)–C(5)–C(6)	128.6 (1.1)	C(4′)–C(5′)–C(6′)	129.5 (1.1)
C(5)–C(6)–C(7)	129.4 (1.2)	C(5′)–C(6′)–C(7′)	115.4 (1.0)
C(6)–C(7)–C(8)	128.1 (1.2)	C(6′)–C(7′)–C(8′)	125.1 (1.1)
C(7)–C(8)–C(9)	125.6 (1.1)	C(7′)–C(8′)–C(9′)	124.7 (1.1)
C(8)–C(9)–C(10)	128.8 (1.0)	C(8′)–C(9′)–C(10′)	127.3 (1.0)
(c) In the 4–6′ Link			
C(10)–C(4)–C(6′)	118.2 (0.9)	C(4)–C(6′)–C(5′)	112.0 (0.9)
C(5)–C(4)–C(6′)	110.6 (0.9)	C(4)–C(6′)–C(7′)	114.5 (0.9)

[a] See footnote *a* to Table V.

accommodate the two- and three-membered bridges which link their two π-cyclopentadienyl systems.

The Over-all Stereochemistry of the 4-*endo*,6′-*endo*-Diazulene Ligand

Although the five-membered rings of the diazulene ligand are each planar within experimental error, the seven-membered rings each take up a distinctly nonplanar conformation in order to accommodate the linkage between the sp^3-hybridized carbon atoms C(4) and C(6′). Thus, in the seven-membered ring defined by C(4)–C(5)–C(6)–C(7)–C(8)–C(9)–C(10), the tetrahedral C(4) [and, also, the trigonal C(8)] is approximately coplanar with the five-membered ring, but the remaining atoms (except, of course, C(9) and C(10)) are bent away from the iron atom such that their deviations from the least-squares plane through the five-membered ring are 0.97 Å for C(5), 1.12 Å for C(6), and 0.54 Å for C(7). The other seven-membered ring is even more drastically nonplanar, deviations from the plane of the five-membered ring C(1′)–C(2′)–C(3′)–C(10′)–C(9′) being 0.11 Å for C(4′), 0.73 Å for C(5′), 1.47 Å for the tetrahedral bridging C(6′), 0.72 Å for C(7′), and 0.11 Å for C(8′). In this ring the carbon atoms are each displaced *toward* the iron atom and thus *toward* the other seven-membered ring. It is interesting to note that this second azulene system maintains strict C_s symmetry, as may clearly be seen both from deviations from the plane of the five-membered ring and from Figure 2.

It is apparent that the 4-*endo*,6′-*endo*-diazulene ligand in the present molecule has less delocalization energy (*i.e.*, is less stable) than the 4-*exo*,4′-*exo*-diazulene ligand in $[C_{10}H_8Mo(CO)_3CH_3]_2$[5] and the 4-*endo*,4′-*endo*-diazulene ligand in $(C_{10}H_8)_2Fe_4(CO)_{10}$,[7] since each of these latter two ligands conserves an extra conjugated buta-1,3-diene system. The formation of the 4-*endo*,6′-*endo*-diazulene ligand in bis(π-azulene)iron must occur as a result of steric effects—in fact, *ex post facto* experi-

TABLE VII

LEAST-SQUARES PLANES WITHIN THE $(C_{10}H_8)_2Fe$ MOLECULE[a,b]

Atom	Dist, Å	Atom	Dist, Å
(A) $0.8636X - 0.4268Y + 0.2679Z - 2.0408 = 0$			
C(1)[c]	−0.021 (18)	C(4)	+0.02
C(2)[c]	+0.017 (14)	C(5)	+0.97
C(3)[c]	−0.005 (11)	C(6)	+1.12
C(9)[c]	+0.018 (10)	C(7)	+0.54
C(10)[c]	−0.008 (10)	C(8)	+0.06
		Fe	−1.649
(B) $0.8478X - 0.5040Y + 0.1644Z + 1.6257 = 0$			
C(1′)[c]	−0.010 (12)	C(4′)	+0.11
C(2′)[c]	+0.014 (13)	C(5′)	+0.73
(C3′)[c]	−0.013 (12)	C(6′)	+1.47
C(9′)[c]	+0.002 (11)	C(7′)	+0.72
C(10′)[c]	+0.007 (11)	C(8′)	+0.11
		Fe	+1.637

[a] All planes are expressed in Cartesian coordinates. The transformations are: $X = xa + zc\cos\beta$, $Y = yb$, $Z = zc\sin\beta$. [b] The angle between planes A and B is 7° 33′, the closest interannular approach being C(10) · · · C(9′). [c] A plane is derived using unit weights for these atoms.

mentation with molecular models shows clearly that the 4-*endo*,6′-*endo* linking of two azulene systems yields the only stereochemistry compatible with near-parallelism of the π-cyclopentadienyl systems and with the absence of excessive strain within the diazulene framework. Molecular models also show that the stereochemistry of the bridged diazulene system requires a staggered conformation for the ferrocene moiety (*vide supra*), and it is tempting to speculate that bis(π-azulene)ruthenium (if it can be prepared) would also take up a staggered conformation.

Finally, it may be noted that the uptake of five molecules of hydrogen by bis(π-azulene)iron[9] may be explained by hydrogenation of the four noncoordinated double bonds accompanied by hydrogenolysis of the 4-*endo*-6′-*endo* carbon–carbon bond, yielding the nonbridged ferrocene derivative—bis(4,5,6,7,8-pentahydroazulenyl)iron.

Crystal Packing

Figure 3 shows the packing of molecules in the crystal

TABLE VIII

A SUMMARY OF RESULTS FROM X-RAY DIFFRACTION AND ELECTRON DIFFRACTION (ed) STUDIES ON FERROCENE DERIVATIVES[a]

Complex	Fe-C (av), Å	φ[b]	ω[c]	Technique	Ref	Comment
$(C_5H_5)_2Fe$	2.045	0[d]	36[d]	X-Ray	26	
$(C_5H_5)_2Fe$	2.03	0	. . .	ed	27	Rings freely rotating at 400°
$(C_5H_5)_2Fe$	2.06	0	. . .	ed	28	
$(C_5H_5)_2Fe$	2.058	0	0	ed	24	Eclipsed equilibrium conformation at 140°
$(C_5H_5)_2Fe$	. . .	0	0	ed	29	Eclipsed equilibrium conformation at 140°; barrier to rotation estimated as 0.9 ± 0.3 kcal/mol
$(C_5H_5)_2Fe \cdot C_2(CN)_4$	. . .	0[d]	36[d]	X-Ray	30	
$(ClSO_2C_5H_4)_2Fe$	2.07	0[d]	36[d]	X-Ray	31	
$(HO_2C \cdot C_5H_4)_2Fe$	2.043	∼0	1.6	X-Ray	32	
$(C_5H_5FeC_5H_4)_2CO$	2.05	∼0	5	X-Ray	33	
$(CH_3COC_5H_4)_2Fe$	. . .	∼0	4.8	X-Ray	34	
Terferrocenyl	. . .	∼0 0[d]	∼0 ∼36[d]	X-Ray	36	Central ferrocene residue is staggered; terminal ferrocenes are each eclipsed
$(C_5H_5FeC_5H_4)_2$	2.035	1.2[a]	16	X-Ray	37	
α-Keto-1,5-tetramethyleneferrocene	2.053	1.5	8.9	X-Ray	38	
$(C_2H_5C_5H_4FeC_5H_4)_2$	2.07	2.3	3.6	X-Ray	39	
1,12-Dimethyl[1.1]ferrocenophane	2.05	2.7	22.4	X-Ray	40	
$(C_5H_5FeC_5H_4)_2$	2.04	2.8[a]	17	X-Ray	41	
$(C_9H_7)_2Fe$	2.10	3	∼36	X-Ray	42	
$(C_5H_5)Fe(B_9C_2H_{11})$	. . .	3.3[e]	3.3[e]	X-Ray	35	
$CH_3C_5H_4FeC_5H_3(CH_3)CO_2^-$	2.06	3.3	9.8[e]	X-Ray	43	Quinidine salt of (−) isomer
$(ClC_5H_4FeC_5H_4)_2$	2.065	4	5.2	X-Ray	44	
$(C_{10}H_8)_2Fe$	2.044	7.5	35.8	X-Ray	This work	
$C_5H_5FeC_5H_4(C_6H_4C_6H_5)$	2.05	8	7.2[e]	X-Ray	45	2-Biphenylyl deriv
α-Keto-1,1′-trimethyleneferrocene	2.039	8.8	11.8	X-Ray	46	
Bis(trimethylene)ferrocene	2.01	9	∼0	X-Ray	47	1,1′:3,3′ isomer
$\{C_5H_5FeC_5H_4C(CH_3)_2-\}_2$	2.04	23.2	9–10	X-Ray	48	
$C_5H_5FeC_5H_4COC_5H_4RuC_5H_5$	. . .	. . .	∼18	X-Ray	49	
$(C_6H_5COC_5H_4)_2Fe$	2.05	. . .	. . .	X-Ray	50	

[a] The table is ordered in terms of increasing φ. For this reason the two independent structural studies of biferrocenyl (which yield slightly differing values of φ) occur at different positions in the table. [b] φ is the dihedral angle (in degrees) between the two five-membered rings. [c] ω is the relative rotational orientation (in degrees) of the two rings. Thus we have ω = 0° for the eclipsed configuration and ω = 36° for the perfectly staggered configuration. [d] Required by the symmetry of the space group. [e] Calculated by the present authors from data available in the appropriate paper.

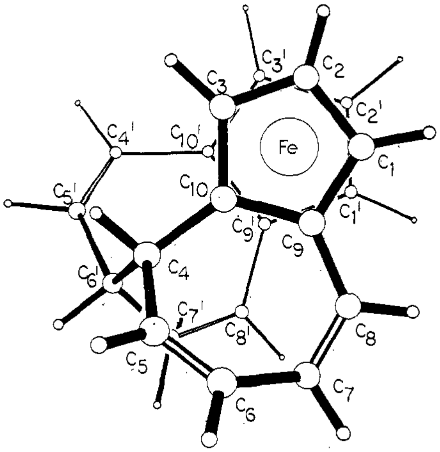

Figure 2.—The $(C_{10}H_8)_2Fe$ molecule projected onto the plane $0.8557X - 0.4654Y + 0.2161Z - 0.2075 = 0$, showing the staggered conformation of the ferrocene system.

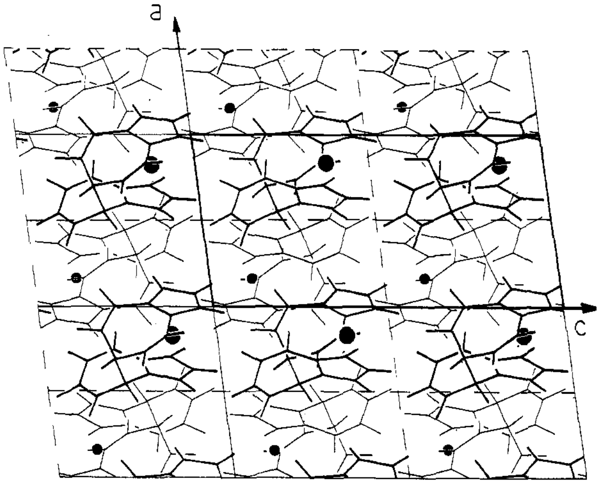

Figure 3.—Packing of molecules in the $(C_{10}H_8)_2Fe$ crystal, viewed down *b*.

Table IX

Intermolecular Contacts (to 3.1 Å) within the $(C_{10}H_8)_2Fe$ Crystal

Atoms	Trans[a]	Dist, Å
C(1)···H(6′)	III	2.87
C(1)···H(2′)	X	2.94
C(2)···H(6′)	III	3.01
C(5)···H(5′)	III	3.07
C(5)···H(8′)	XII	3.03
C(6)···H(5′)	III	3.02
C(6)···H(3)	IX	2.88
C(9)···H(5′)	III	2.77
C(10)···H(5′)	III	2.94
C(2′)···H(7′)	II	3.08
C(2′)···H(5)	VI	2.91
C(2′)···H(1)	X	3.08
C(3′)···H(7′)	II	2.92
C(4′)···H(8)	XIV	2.78
C(5′)···H(1′)	XIV	2.85
C(6′)···H(1′)	XIV	3.02
C(7′)···H(3′)	IX	2.97
C(8′)···H(3′)	IX	3.10
C(8′)···H(8)	V	2.83
C(9′)···H(8)	XIV	2.83
C(10′)···H(8)	XIV	2.82
H(1)···H(4)	III	2.90
H(1)···H(5)	III	2.40
H(1)···H(6′)	III	3.02
H(1)···H(2′)	X	2.02
H(1)···H(7′)	XIII	3.07
H(1)···H(8′)	XIII	2.97
H(2)···H(6′)	II	2.54
H(2)···H(2)	VIII	2.70
H(2)···H(2′)	X	3.05
H(4)···H(8)	V	3.07
H(4)···H(8′)	XII	2.84
H(5)···H(2′)	XI	2.43
H(5)···H(8′)	XII	2.47
H(6)···H(7)	I	2.73
H(6)···H(3)	IX	2.16
H(7)···H(3′)	IX	2.81
H(7)···H(4′)	IX	2.72
H(1′)···H(5′)	XIII	2.55
H(1′)···H(6′)	XIII	2.51
H(3′)···H(4′)	IX	2.88
H(7′)···H(2)	II	2.51
H(7′)···H(2′)	II	2.75
H(7′)···H(3)	II	2.33

[a] Transformations are: (I) $-x + 1, -y, -z + 1$; (II) $x, -y + 1/2, z + 1/2$; (III) $-x + 1, y + 1/2, -z + 1/2$; (IV) $-x, -y + 1, -z$; (V) $-x + 1, y - 1/2, -z + 1/2$; (VI) $x + 1, -y + 1/2, z + 1/2$; (VII) $x + 1, y, z$; (VIII) $-x + 1, -y, -z$; (IX) $x, -y + 1/2, z - 1/2$; (X) $-x, -y, -z$; (XI) $x - 1, -y + 1/2, z - 1/2$; (XII) $x - 1, y, z$; (XIII) $-x, y + 1/2, -z + 1/2$; (XIV) $-x, y - 1/2, -z + 1/2$.

projected onto the 010 plane. Table IX contains quantitative information concerning intermolecular contacts. It may be noted that closest contacts are 2.02 Å for hydrogen···hydrogen and 2.77 Å for carbon···hydrogen interactions.

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