There is then either something very unusual about platinum dimethylglyoxime or a large error in the reported angle. This can only be resolved by a more complete study of the structure of platinum dimethylglyoxime which has been studied[5] only in projections.

A nearly complete neutron diffraction study[10] of the nickel complex indicates a single equilibrium position

(10) E. O. Schlemper, S. J. LaPlaca, and W. C. Hamilton, unpublished work.

for the hydrogen atom in the hydrogen bond. This would be less likely for the platinum complex with the longer O- - -O distance.

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Crystal and Molecular Structure of Ferrocenedicarboxylic Acid

By GUS J. PALENIK

Ferrocenedicarboxylic acid crystallizes as orange-red laths in the monoclinic space group $P2_1/c$ with $a = 8.403 \pm 0.010$ Å, $b = 8.910 \pm 0.010$ Å, $c = 14.192 \pm 0.018$ Å, $\beta = 90.41 \pm 0.08°$, and four molecules per unit cell. The structure was refined by least-square methods with anisotropic thermal parameters to a final R of 6.2% for the 1618 observed reflections measured using an automatic diffractometer with molybdenum radiation. Two molecules of ferrocenedicarboxylic acid form hydrogen-bonded dimers. The average C–C distance in the cyclopentadienide rings of 1.424 ± 0.013 Å is normal. The two cyclopentadienide rings are twisted by an average of $1° 38'$, making the molecule one of the most nearly eclipsed ferrocene derivatives.

Introduction

Ferrocene and many of its derivatives are characterized by weak intermolecular forces in the crystalline state. Crystal structure studies carried out at room temperature frequently find high thermal motion which reduces the accuracy of the measured bond distances and angles. The hydrogen bonding of the two carboxyl groups in ferrocenedicarboxylic acid would give rise to strong intermolecular forces and might limit the thermal motion of the molecule. Thus, the possibility of obtaining precise distances for the ferrocene molecule was one reason for the crystal structure determination of ferrocenedicarboxylic acid.

The relative orientation of the two cyclopentadienide rings in ferrocene derivatives appears to be dependent on the substituents on the rings. Trotter and Macdonald,[1] Churchill and Mason,[2] and Churchill and Wormald[3] have discussed the relationship between orientation and intermolecular forces. The conformation of ferrocenedicarboxylic acid is important since strong intermolecular forces will be present because of the hydrogen bonding. The question of the effect of the hydrogen bonding on the orientation of the cyclopentadienides provided the second reason for the investigation of this compound.

Experimental Section

Ferrocenedicarboxylic acid was purchased from Alfa Inorganics. Crystals were grown by cooling slowly a solution of the acid in hot glacial acetic acid. The crystals were orange-red laths which were found to be elongated along b. Weissenberg photographs taken about [010] revealed only the systematic extinctions $h0l$ absent when l is odd.

One small, well-developed lath was cleaved with a razor blade to give a small crystal ($0.11 \times 0.18 \times 0.05$ mm parallel to a, b, and c, respectively) which was mounted on a glass fiber. The fiber and crystal were dipped into liquid nitrogen and then mounted on a General Electric single-crystal orienter. The long dimension of the crystal (the b axis) was parallel to the ϕ axis of the orienter. The unit cell dimensions were determined using a narrow beam of molybdenum radiation ($\lambda(\alpha_1)$ 0.70926 and $\lambda(\alpha_2)$ 0.71354). The average of these measurements were: $a = 8.403 \pm 0.010$ Å, $b = 8.910 \pm 0.010$ Å, $c = 14.192 \pm 0.018$ Å, $\beta = 90.41 \pm 0.08°$. The density calculated for four molecules per unit cell is 1.713 g/cm³, while the density determined by flotation in a carbon tetrachloride–methylene iodide solution is 1.715 g/cm³. A survey of the $0k0$ reflections with the diffractometer revealed the additional systematic absence, $0k0$ absent if k is odd. The most probable space group was therefore $P2_1/c (C_{2h}^5)$.

The intensity measurements were made with a scintillation counter using a wide beam of molybdenum radiation (takeoff angle of 3.7°). A linear amplifier–pulse height selector combination together with a zirconium filter was employed to eliminate the majority of the noncharacteristic radiation. The diffractometer was automated by the Datex Corp. and was controlled by a prepunched paper tape. The stationary counter-stationary crystal technique was employed in measuring 4348 reflections with $2\theta \leq 60.0°$. A 20-sec count was made for each reflection. Four standard reflections were counted periodically with 46 measurements of each standard being made during the course of the intensity measurements. The root-mean-square deviation for each of the four reflections was about 2% with no apparent trend, indicating good crystal and instrument stability. Reflections of the type $h0l$ with $l = 2n + 1$, required by the space group to be absent, were also measured. Those absent reflections which were not on a radiation streak were used to derive

(1) J. Trotter and A. C. MacDonald, *Acta Cryst.*, **21**, 359 (1966).
(2) M. R. Churchill and R. Mason, *Advan. Organometal. Chem.*, **5**, 93 (1967).
(3) M. R. Churchill and J. Wormald, *Inorg. Chem.*, **8**, 716 (1969).

an empirical background curve. Of the 3069 unique reflections with $2\theta \leq 60°$ only the 1618 reflections whose intensities were greater than 1.2 times the background count were considered as observed reflections and were used in the structure analysis. No correction for the $\alpha_1-\alpha_2$ splitting was made since the doublet could be measured under the experimental conditions employed. The reduction of these data to a set of observed amplitudes on an arbitrary scale was carried out in the usual manner.

Determination and Refinement of the Structure

The position of the iron atom was deduced from a three-dimensional Patterson function with the origin peak removed. The lighter atoms were located in subsequent difference and observed Fourier syntheses. The position parameters and an individual isotropic thermal parameter for each atom were refined by full-matrix least-squares methods; three cycles reduced the R value to 9.6%. The weighting scheme used in this and subsequent least-squares cycles was $\sqrt{w} = 1.0$ if $F_o \leq 30.0$ and $\sqrt{w} = 30.0/F_o$ if $30.0 < F_o$, where F_o is on the scale given in Table IV. Anisotropic thermal parameters were introduced at this point for all of the atoms. The full matrix was approximated by 3×3 blocks for position parameters and 6×6 blocks for thermal parameters. After five cycles the value of R was reduced to 6.8%.

A difference Fourier synthesis was computed to locate the hydrogen atoms. The hydrogen atoms on the cyclopentadienide rings were located easily but the two hydrogen atoms in the carboxyl groups could not be determined with any degree of certainty. The eight hydrogen atoms on the two rings were included in the structure factor calculation (using an isotropic B of 5.0), but their parameters were not refined.

Five least-squares cycles were computed using the block approximation and the residual R decreased to 6.2%. The shifts in all the parameters after the last cycle were small and a tabulation of the shifts revealed no apparent trend; therefore, the refinement was terminated. The final positional parameters for the 17 nonhydrogen atoms are given in Table I and their final thermal parameters are presented in Table II. A final difference Fourier synthesis was computed without including the hydrogen atom contributions and a new set of hydrogen atom positions was obtained. The final positions of the eight ring hydrogen atoms are given in Table III. The final set of calculated structure factors was computed using the parameters in Tables I–III and is presented in Table IV.

In all the above calculations the scattering factors for Fe, O, and H were from the "International Tables for X-Ray Crystallography."[4] For the carbon atoms the graphite curve given by McWeeny[5] was used.

Discussion

Two molecules of ferrocenedicarboxylic acid related by a center of symmetry form a hydrogen-bonded dimer. The formation of these dimers appears to influence strongly many features of the molecular

(4) "International Tables for X-Ray Crystallography," Vol. III, The Kynoch Press, Birmingham, England, 1962.
(5) R. McWeeny, *Acta Cryst.*, **4**, 513 (1951).

TABLE I
THE FINAL POSITIONAL PARAMETERS ($\times 10^4$) AND THEIR ESTIMATED STANDARD DEVIATIONS

Atom	$x\ (\sigma_x)$	$y\ (\sigma_y)$	$z\ (\sigma_z)$
C(01)	3879 (8)	1142 (8)	0656 (5)
C(02)	2459 (8)	0451 (9)	0291 (5)
C(03)	1956 (10)	−0602 (9)	0980 (6)
C(04)	2983 (10)	−0537 (10)	1755 (6)
C(05)	4208 (8)	0526 (10)	1571 (5)
C(06)	4796 (8)	2311 (8)	0184 (5)
O(01)	6034 (6)	2811 (7)	0605 (4)
O(02)	4352 (6)	2762 (7)	−0620 (3)
C(11)	1655 (8)	3769 (8)	1638 (4)
C(12)	0242 (9)	3051 (9)	1308 (6)
C(13)	−0249 (9)	1980 (9)	1997 (6)
C(14)	0854 (9)	2070 (9)	2751 (5)
C(15)	2041 (9)	3155 (9)	2547 (5)
C(16)	2564 (8)	4898 (8)	1134 (5)
O(11)	3823 (7)	5400 (7)	1528 (4)
O(12)	2148 (6)	5291 (7)	0320 (4)
Fe	2013 (1)	1531 (1)	1526 (1)

TABLE II
FINAL THERMAL PARAMETERS ($\times 10^4$) WITH THEIR ESTIMATED STANDARD DEVIATIONS IN PARENTHESES[a]

Atom	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
C(01)	115 (9)	120 (11)	43 (3)	27 (15)	20 (9)	−14 (9)
C(02)	132 (10)	121 (11)	52 (4)	−14 (17)	24 (10)	−25 (11)
C(03)	180 (13)	103 (11)	84 (6)	−29 (20)	104 (14)	−47 (13)
C(04)	183 (13)	127 (12)	61 (4)	78 (21)	70 (12)	40 (12)
C(05)	133 (10)	176 (14)	46 (4)	107 (20)	44 (10)	36 (12)
C(06)	120 (10)	105 (10)	46 (3)	22 (15)	39 (9)	−25 (10)
O(01)	129 (7)	189 (9)	57 (3)	−44 (14)	5 (7)	2 (9)
O(02)	164 (8)	168 (9)	42 (2)	−5 (14)	6 (7)	13 (8)
C(11)	149 (11)	112 (11)	37 (3)	41 (16)	2 (9)	0 (9)
C(12)	128 (10)	127 (12)	66 (4)	7 (17)	22 (11)	10 (12)
C(13)	148 (11)	118 (11)	72 (5)	47 (18)	62 (12)	24 (12)
C(14)	152 (11)	144 (12)	53 (4)	36 (19)	73 (11)	0 (11)
C(15)	183 (13)	137 (13)	40 (3)	25 (19)	51 (10)	−24 (10)
C(16)	113 (9)	108 (10)	46 (3)	15 (16)	3 (9)	10 (10)
O(01)	201 (10)	170 (9)	48 (3)	−99 (16)	−25 (8)	23 (8)
O(02)	149 (8)	177 (10)	58 (3)	−9 (14)	−35 (8)	69 (9)
Fe	111 (1)	107 (1)	38 (0.3)	13 (3)	24 (1)	2 (2)

[a] The temperature factor is of the form $\exp[-(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + \beta_{12}hk + \beta_{13}hl + \beta_{23}kl)]$.

TABLE III
PROBABLE HYDROGEN POSITIONS[a]

Atom	x	y	z	Dist, Å
H(02) (C(02))	189	058	−035	1.00
H(03) (C(03))	091	−106	093	0.87
H(04) (C(04))	297	−125	242	1.13
H(05) (C(05))	528	092	195	1.10
H(12) (C(12))	−030	304	067	0.97
H(13) (C(13))	−124	141	160	1.08
H(14) (C(14))	080	158	334	0.85
H(15) (C(15))	303	336	298	1.02

[a] The hydrogen atom is given followed by the atom to which it is bonded (in parentheses), the position parameters ($\times 10^3$), and the corresponding C–H distance.

geometry and crystal packing. Figure 1 illustrates the hydrogen-bonded dimers and their packing in the unit cell. The intramolecular distances and angles in the dimer unit are illustrated in Figure 2.

The two carboxyl groups and the attached ring carbon atoms are planar (see Table VI) but are twisted slightly with respect to the corresponding cyclopentadienide (Cp) ring. The angle between the plane of C(06)–O(01)–O(02) and its Cp ring is 0° 50′ while the corresponding angle for C(16)–O(11)–O(12) is 4° 40′.

TABLE IV

Observed and Calculated Structure Factors for $(H_4C_5COOH)_2Fe^a$

H= 0,K= 0		
2	-47	-52
4	750	-733
6	717	695
8	178	-81
10	727	-720
12	233	193
14	174	137
16	185	-153
18	-33	29
H= 1,K= 0		
-18	163	-159
-16	-32	-35
-14	222	218
-12	155	-161
-10	-37	50
-8	1167	1147
-6	367	-349
-4	654	-634
-2	971	932
0	141	-74
2	2671	-2676
4	426	382
6	623	601
8	560	-553
10	54	88
12	103	88
14	258	-263
16	101	-104
18	109	101
H= 2,K= 0		
-18	-34	-48
-16	182	170
-14	81	71
-12	266	-266
-10	226	227
-8	-32	25
-6	548	-529
-4	769	760
-2	566	552
0	827	-824
2	238	-222
4	101	33
6	376	-374
8	445	-446
10	366	358
12	61	-21
14	-35	29
16	134	130
18	-34	58
H= 3,K= 0		
-18	60	56
-16	165	163
-14	74	-82
-12	85	-91
-10	262	272
-8	358	-358
-6	604	-573
-4	-34	-15
-2	762	-789
0	687	-668
2	614	614
4	270	274
6	94	-117
8	-35	30
10	282	299
12	212	-205
14	-34	-9
16	136	108
18	-34	-29
H= 4,K= 0		
-18	83	90
-16	115	-104
-14	315	-309
-12	107	111
-10	-34	19
-8	209	-208
-6	429	444
-4	553	549
-2	609	-605
0	352	327
2	688	676
4	434	-441
6	-33	-3
8	623	629
10	89	86
12	176	-177
14	132	124
16	54	7
18	155	-134
H= 5,K= 0		
-18	-34	-17
-16	85	-85
-14	147	155
-12	222	229
-10	295	-303
-8	70	85
-6	144	160
-4	391	-406
-2	-32	3
0	1045	1039
2	-32	4
4	510	-511
6	306	326
8	81	-99
10	556	-527
12	-35	0
14	100	95
16	109	-79
18	-33	0
H= 6,K= 0		
-16	-34	30
-14	108	95
-12	149	-154
-10	-35	-5
-8	364	364
-6	87	-42
-4	192	-198
-2	305	322
0	234	-94
2	598	-607
4	-36	35
6	324	323
8	220	-228
10	-35	20
12	240	199
14	86	-66
16	-34	-61
H= 7,K= 0		
-16	111	102
-14	50	34
-12	138	-134
-10	132	126
-8	-35	-6
-6	221	-220
-4	61	-56
-2	201	194
0	308	-265
2	118	-90
4	134	136
6	75	-87
8	173	-189
10	136	134
12	68	24
14	110	-62
H= 8,K= 0		
-14	74	-67
-12	102	-96
-10	145	139
-8	123	-128
-6	158	-143
-4	121	116
-2	-35	42
0	182	-149
2	194	213
4	112	121
6	-34	-64
8	108	102
10	108	106
12	51	-67
14	-34	-9
H= 9,K= 0		
-12	93	69
-10	76	63
-8	94	-89
-6	117	134
-4	167	156
-2	169	-160
0	67	27
2	102	90
4	134	-136
6	-32	-24
8	121	120
10	64	-65
12	-34	-58
H=10,K= 0		
-10	105	-89
-8	-34	17
-4	86	-104
-2	67	-56
0	190	176
2	-31	-35
4	-31	-52
6	76	78
8	-34	-17
10	114	-100
H=11,K= 0		
-6	-34	-43
-4	56	-54
-2	78	66
0	-34	11
2	119	-113
4	-34	3
6	-34	28
H= 0,K= 1		
0	-45	0
1	281	313
2	1125	-1095
3	452	-429
4	385	369
5	148	131
6	268	275
7	147	111
8	628	-614
9	240	-195
10	87	66
11	-36	21
12	161	155
13	147	153
14	130	-119
15	-34	-11
16	101	-90
17	77	-61
18	81	85
19	-34	21
H= 1,K= 1		
-19	-34	-7
-18	-33	-42
-17	72	-54
-16	131	122
-15	131	117
-14	141	-136
-13	-35	8
-12	236	-238
-11	219	-225
-10	293	298
-9	-34	-7
-8	-32	-11
-7	229	236
-6	635	-638
-5	355	-355
-4	64	-90
-3	-46	90
-2	907	859
-1	359	325
0	1040	-1071
1	368	-358
2	-48	63
3	282	-261
4	662	630
5	168	169
6	110	-127
7	62	-67
8	284	-289
9	-34	-30
10	445	439
11	177	167
12	85	-82
13	67	63
14	63	-81
15	99	-91
16	81	83
17	-31	-9
18	-33	35
19	-34	33
H= 2,K= 1		
-19	53	-51
-18	75	78
-17	-31	19
-16	81	78
-15	103	103
-14	152	-153
-13	148	-157
-12	146	148
-11	-35	19
-10	378	377
-9	253	267
-8	438	-429
-7	296	-294
-6	-32	-43
-5	398	-395
-4	401	410
-3	358	341
-2	199	247
-1	86	-114
0	617	-618
1	253	-250
2	737	715
3	362	350
4	-35	5
5	400	356
6	498	-482
7	-32	-60
8	148	163
9	-35	43
10	115	113
11	99	91
13	110	-112
14	-35	52
15	-34	-73
16	94	88
17	86	68
18	75	-77
19	-34	20
H= 3,K= 1		
-19	-34	4
-17	90	85
-16	97	-95
-15	71	-66
-14	171	-165
-13	145	-147
-12	179	167
-11	167	159
-10	123	-132
-9	-37	-55
-8	373	-382
-7	388	-395
-6	475	496
-5	573	579
-4	227	223
-3	439	440
-2	416	-438
-1	293	-303
0	175	162
1	-35	-30
2	-35	84
3	247	253
4	653	-644
5	242	-246
6	-32	13
7	149	-140
8	438	447
9	104	122
10	176	-179
11	-34	-19
12	-35	-10
13	96	-101
14	182	179
15	85	78
16	-30	-30
17	-33	31
18	68	-82
19	66	-58
H= 4,K= 1		
-18	-34	-36
-17	-34	-6
-16	54	-21
-15	159	-143
-14	150	141
-13	109	120
-12	176	179
-11	105	116
-10	172	-185
-9	185	-185
-8	81	95
-7	144	136
-6	270	280
-5	497	489
-4	526	-541
-3	435	-446
-2	-32	16
-1	170	-180
0	683	671
1	242	258
2	306	-311
3	-32	-5
4	-32	8
5	139	-141
6	428	421
7	85	108
8	100	-110
9	55	-51
10	359	-354
11	149	-136
12	119	129
13	-35	27
14	-33	18
15	61	44
16	115	-92
17	50	-55
18	-34	27
H= 5,K= 1		
-18	90	-80
-17	70	-72
-16	-33	26
-15	-31	32
-14	-32	49
-13	149	159
-12	221	-226
-11	112	-122
-10	-35	-22
-9	-34	8
-8	186	196
-7	227	233
-6	-37	-74
-5	164	-154
-4	310	-306
-3	285	-293
-2	425	431
-1	140	137
0	187	-191
1	-32	34
2	640	-625
3	184	-198
4	248	256
5	-36	-16
6	122	131
7	-36	-23
8	234	-223
9	87	-79
10	116	113
11	60	44
12	186	176
13	79	75
14	79	-70
15	67	-28
16	-34	-30
17	-34	-3
H= 6,K= 1		
-17	-34	-24
-16	56	77
-15	65	79
-14	-31	-10
-13	53	-42
-12	139	-142
-11	83	-95
-10	262	269
-9	80	86
-8	85	-79
-7	-34	-45
-6	190	-202
-5	191	-201
-4	110	126
-3	-37	46
-2	349	356
-1	112	123
0	205	-209
1	-37	-41
2	-37	76
3	125	-136
4	347	342
5	81	83
6	158	-169
7	-34	30
8	162	-158
9	-35	40
10	115	123
11	60	65
12	-33	-38
13	-31	17
14	66	-67
15	-34	-24
16	-34	38
17	-34	13
H= 7,K= 1		
-16	-34	-17
-15	-34	19
-14	80	-78
-13	-32	-38
-12	-30	19
-11	-32	6
-10	189	197
-9	-34	55
-8	234	-243
-7	113	-109
-6	-35	-53
-5	90	-90
-4	151	161
-3	154	146
-2	174	-188
-1	-34	25
0	225	-208
1	135	-129
2	137	133
3	85	81
4	69	75
5	191	205
6	122	-150
7	-35	-23
8	74	101
9	-34	-10
10	49	66
11	54	26
12	86	-92
13	-32	-46
14	-34	2
15	-34	-24
H= 8,K= 1		
-14	-34	-22
-13	-34	-45
-12	76	88
-11	-32	12
-10	56	-38
-9	-32	-0
-8	114	-122
-7	100	-108
-6	213	208
-5	151	153
-4	171	178
-3	194	193
-2	125	-125
-1	154	-143
0	182	177
1	-35	3
2	-35	20
3	184	184
4	155	-150
5	76	-87
6	-34	-55
7	95	-111
8	103	97
9	-32	40
10	77	-56
11	-32	-14
12	-34	-9
13	-34	-37
14	64	65
H= 9,K= 1		
-12	-34	39
-11	-34	57
-10	75	-91
-9	-32	-36
-8	-31	36
-7	-31	50
-6	-32	1
-5	173	157
-4	176	-169
-3	141	-130
-2	124	-113
-1	128	-131
0	130	124
1	137	141
2	109	-109
3	-33	-42
4	-33	20
5	125	-130
6	135	124
7	56	49
8	-31	-24
9	-32	7
10	75	-80
11	-34	-43
12	-34	27
H=10,K= 1		
-10	-34	-0
-9	-34	-25
-8	88	91
-7	50	64
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-7	-35	-27
-6	-35	28
-5	-35	-30
-4	202	207
-3	182	-186
-2	189	-188
-1	-34	-2
0	180	-188
1	93	101
2	210	217
3	60	-57
4	-35	7
5	65	-81
6	99	-110
7	136	147
8	82	90
9	-34	-29
10	-33	31
11	-32	-39
12	86	-81
13	102	90
14	-34	26
15	-34	18
H= 7,K= 4		
-14	-34	10
-13	-34	46
-12	65	64
-11	-31	-64
-10	105	-106
-9	-32	31
-8	99	-107
-7	70	92
-6	171	179
-5	133	-130
-4	-35	-25
-3	-35	-14
-2	179	-186
-1	178	169
0	134	136
1	-35	-32
2	-35	59
3	92	-89
4	269	-255
5	186	186
6	-35	-54
7	51	69
8	62	77
9	73	-79
10	55	-64
11	-32	34
12	-33	1
13	-34	46
H= 8,K= 4		
-13	-34	-47
-12	-34	-34
-11	-34	-36
-10	52	-55
-9	77	61
-8	94	93
-7	-31	-47
-6	-32	28
-5	72	-65
-4	188	-196
-3	198	200
-2	-34	18
-1	-34	50
0	151	142
1	133	-143
2	85	-92
3	-34	47
4	-34	-1
5	54	59
6	188	180
7	126	-122
8	-31	27
9	-32	-17
10	-33	-43
11	-34	51
12	-34	46
13	52	-23
H= 9,K= 4		
-10	-34	37
-9	-34	48
-8	50	42
-7	76	-72
-6	86	-75
-5	-31	29
-4	-30	1
-3	91	72
-2	127	134
-1	114	-104
0	-31	-13
1	-31	-40
2	81	-89
3	113	112
4	-31	61
5	-31	-29
6	-32	14
7	-33	-42
8	64	-78
9	-34	31
10	-34	-0
11	-34	-17
H=10,K= 4		
-8	-34	-14
-7	-34	-46
-6	-34	-32
-5	67	71
-4	64	68
-3	-34	-11
-2	-33	18
-1	85	-85
0	81	-70
1	51	20
2	-33	6
3	-34	3
4	65	60
5	67	-70
6	-34	-32
7	-34	19
8	-34	-20
H=11,K= 4		
-3	-34	-46
-2	50	-61
-1	49	20
0	-34	-22
1	49	57
2	78	52
3	-34	-17
H= 0,K= 5		
0	-32	-0
1	363	-335
2	758	721
3	67	-78
4	347	-349
5	238	234
6	204	-211
7	95	106
8	355	344
9	-34	-4
10	145	-141
11	53	70
12	236	-222
13	-34	8
14	101	106
15	-31	-41
16	-33	14
17	-34	-40
18	93	-82
H= 1,K= 5		
-18	-34	31
-17	-34	-9
-16	113	-111
-15	72	66
-14	-32	3
-13	-34	-12
-12	155	157
-11	-35	-47
-9	189	-189
-8	116	-123
-7	-37	-11
-6	403	396
-5	-35	-55
-4	318	-327
-3	-32	-53
-2	419	-390
-1	-32	67
0	746	739
1	158	160
2	-32	-62
3	-32	-2
4	484	-500
5	-35	32
6	303	300
7	224	-237
8	103	125
9	116	-112
10	234	-255
11	-35	79
12	112	121

The large twist for the C(16) carboxyl group appears to be related to crystal packing forces and is discussed below. Both carboxyl groups are bent slightly out of the plane of the Cp ring toward each other. The average separation of the carboxyl groups is 3.242 Å, which is smaller than the C–C cross-ring distance of 3.291 ± 0.010 Å. Apparently, without the restraining force of the iron atom, the carboxyl groups attempt to achieve van der Waals contacts within the ring as well as without.

TABLE IV (*Continued*)

[Structure factor table data — columns of h, k, l, $10F_o$, and $10F_c$ values]

ᵃ The values of h and k are given above each group with the values of l, $10F_o$, and $10F_c$ given in that order. A negative F_o indicates an unobserved reflection.

The plane of either carboxyl group is displaced from the plane of the carboxyl group to which it is hydrogen bonded. The planarity (or lack of) of carboxylic acid dimers has been discussed recently by Donohue.[6] In the present structure the two planes are twisted by 2° 33′ from coplanarity, a reasonably small angle.

The four C–O distances in the carboxyl groups are not significantly different from each other or the average value of 1.268 ± 0.010 Å. The equality of these bond lengths is surprising since in most carboxylic acids (see ref 6 for examples) the two C–O bonds are definitely different. The equality of the C–O bond lengths together with the lack of evidence for the hydroxyl hydrogen atoms suggests that the molecules may be dis-

(6) J. Donohue, *Acta Cryst.*, **B24**, 1558 (1968).

Figure 1.—A view down the *a* axis illustrating the atom numbering and the packing of the dimer in the unit cell.

Figure 2.—The distances and angles in the dimer unit.

ordered. Because of the dimer formation, the intermolecular forces between molecules are only the weak van der Waals forces and so the existence of disorder is not unlikely. The original structural work on ferrocene[7] itself may have suffered from a similar disorder in which the staggered conformation may be the result of disordering two eclipsed molecules. The recent electron diffraction results[8] which were interpreted in terms of an eclipsed molecule support this hypothesis. In addition, the extensive studies of a similar problem in azulene[9] and phenylazulene[10] have indicated the unreliability of the Fourier synthesis.

The Cp rings are both planar (see Table V) but are 1° 17' from being parallel. Although the average C–C distance of 1.424 ± 0.013 Å is in good agreement with the average value of 1.419 Å cited by Wheatley,[11] there appears to be a trend in the distances within the rings. In both rings the shortest bond distance is the one furthest away from the dimer center. The shortening may therefore be related to thermal motion of the molecule.

The thermal parameters were converted to mean-square displacements and are given in Table VI. The largest displacements for C(03) and C(04) are approximately perpendicular to the ring normal (angles of 82 and 79°, respectively), in agreement with the

(7) J. D. Dunitz, L. E. Orgel, and A. Rich, *Acta Cryst.*, **9**, 373 (1956).

(8) A. Haaland and J. E. Nilsson, *Chem. Commun.*, 88 (1968).

(9) J. M. Robertson, H. M. M. Shearer, G. A. Sim, and D. G. Watson, *Acta Cryst.*, **15**, 1 (1962).

(10) J. Donohue and B. D. Sharma, *Nature*, **198**, 878 (1963).

(11) P. J. Wheatley in "Perspectives in Structural Chemistry," Vol. I, J. D. Dunitz and J. A. Ibers, Ed., John Wiley & Sons, Inc., New York, N. Y., 1967, p 9.

TABLE V

LEAST-SQUARES PLANES: THE DEVIATIONS FROM THE PLANE
(Å × 10³) ARE GIVEN FOR THE SPECIFIED ATOM

Atom	Plane I	Plane II	Atom	Plane III	Plane IV
C(01)	004	002	C(11)	003	005
C(02)	−009	−007	C(12)	−005	013
C(03)	011	−013	C(13)	005	084
C(04)	−009	−052	C(14)	−004	096
C(05)	003	−026	C(15)	001	055
C(06)	−025	−006	C(16)	036	−018
O(01)	−011	002	O(11)	067	006
O(02)	−040	002	O(12)	103	007
Fe	−1646	−1665	Fe	1646	1693

| | Direction cosines | | | Distance | |
| | (×10⁴) with respect to | | | from origin, | |
Plane	a	b	c^*	Å	Atoms used to define the plane
I	5618	−7141	−4716	0.708	C(01–C(05)
II	5621	−7044	−4346	0.703	C(01)–C(06)–O(01)–O(02)
III	5595	−7045	−4365	−2.615	C(11)–C(15)
IV	5489	−7311	−4501	−2.648	C(11)–C(16)–O(11)–O(12)

hypothesis of libration effects. However, in the case of C(13) and C(14) the same situation does not apply. Attempts to fit the thermal parameters to various rigid bodies were unsuccessful. Either the motion is more complicated or else systematic errors in the data have been absorbed by the thermal parameters.

TABLE VI

MEAN-SQUARE DISPLACEMENTS

Atom	$(r^2)^{1/2}$ ^{a}			Atom	$(r^2)^{1/2}$ ^{a}		
C(01)	227	220	183	C(11)	241	201	193
C(02)	250	212	204	C(12)	265	225	210
C(03)	334	211	188	C(13)	295	217	198
C(04)	306	209	204	C(14)	277	240	178
C(05)	298	209	180	C(15)	269	242	175
C(06)	240	216	169	C(16)	222	208	196
O(01)	279	241	209	O(11)	303	226	215
C(02)	261	242	205	O(12)	300	236	195
Fe	217	205	178				

^{a} Values times 10³, in ångströms.

The view down the line connecting the two ring centers is given in Figure 3 and illustrates the almost total eclipsing of the two rings. The angle of twist was de-
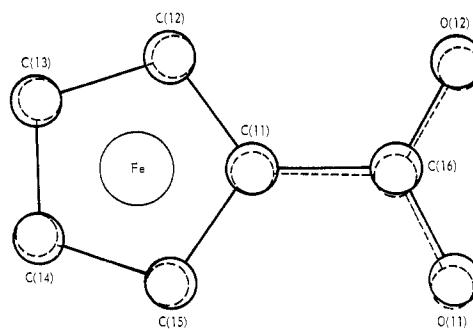

Figure 3.—A view down the ring centers showing the nearly eclipsed configuration.

fined as the dihedral angle between one carbon atom, the two ring centers, and a second carbon atom. The angles varied from 1° 20′ to 1° 53′ with a mean of 1° 38′. The molecule is one of the most nearly eclipsed ferrocene derivatives reported to date. The question of the preferred orientation of the two Cp rings in ferrocene derivatives is largely unsettled. The problem is further complicated by the possible presence of disorder in ferrocene itself. In the dicarboxylic acid the rings are eclipsed with the substituents in the 1,1′ positions. The conformation may result from the hydrogen bonding of two molecules into a dimer unit. The crystal structure of a salt of ferrocenedicarboxylic acid might then have a different conformation. However, all attempts to grow single crystals of several salts have been unsuccessful.

The main forces between dimer molecules appear to be only weak van der Waals forces. In fact the herringbone-type packing illustrated in Figure 1 is very common for closest packed organic molecules. A survey of all of the intermolecular distances less than 3.5 Å did not indicate any abnormally short intermolecular contacts. However, the distance from O(11) to H(05) in position $-x$, $^1/_2 + y$, $^1/_2 - z$ is 2.33 Å, which is approximately equal to the 2.4 Å expected for a van der Waals contact. The O(11) atom is in the carboxyl group which was rotated about 4° more than the other. It is therefore tempting to suggest that the larger twist allows slightly better crystal packing.