Table 6. *Powder diffraction data of quinhydrone*

Triclinic		Monoclinic	
d	Intensity	*d*	Intensity
5·44 Å	*s*	5·34 Å	*s*
3·77	*w*	3·98	*w*
3·12	*m*	3·58	*m*
3·06	*s*	3·22	*m*
2·78	*m*	3·08	*s*
2·66	*m*	2·87	*w*
2·54	*m*	2·72	*w*
2·32	*w*	2·59	*m*
2·16	*m*	2·42	*m*
2·01	*w*	2·30	*m*
1·90	*w*	2·15	*w*
1·82	*w*	2·09	*w*
1·76	*w*	1·80	*w*
1·75	*w*	1·69	*w*
1·66	*w*	1·59	*m*
1·60	*m*		
1·51	*w*		
1·16	*w*		

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The Crystal Structure of α-Keto-1,1′-trimethyleneferrocene*

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The crystal structure of α-keto-1,1′-trimethyleneferrocene has been determined by three-dimensional X-ray diffraction analysis. This compound, $C_{13}H_{12}OFe$, crystallizes in the centrosymmetric monoclinic space group $P2_1/a$. The unit cell contains four molecules and has the dimensions

$$a = 22{\cdot}981 \pm 0{\cdot}002,\ b = 7{\cdot}381 \pm 0{\cdot}001,\ c = 5{\cdot}833 \pm 0{\cdot}001\ \text{Å};\ \beta = 93{\cdot}38 \pm 0{\cdot}02°.$$

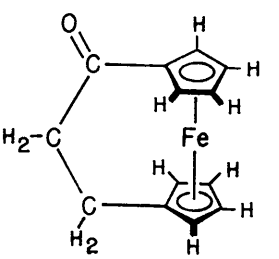
A trial structure was obtained from the locations of the iron–iron vectors in the (001) Patterson projection and was refined in both two and three dimensions by the method of least squares. The final reliability factor, *R*, was 0·067.

The dihedral angle between the best planes through the two nearly planar cyclopentadienyl rings is about 8·8°. The average ring carbon–carbon bond distance is 1·424 ± 0·010 Å and the average iron–carbon bond distance is 2·039 ± 0·006 Å. The principal thermal motion is a vibration of the entire molecule in a direction roughly perpendicular to the *ab* plane.

Introduction

The stable bridged 'sandwich' compound α-keto-1,1′-trimethyleneferrocene, $C_{13}H_{12}OFe$ (I), may be prepared by the procedure of Rinehart & Curby (1957).

(I)



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The determination of the crystal structure of this compound was prompted by its unusual geometry and more specifically by recent studies (Hill & Richards, 1961; Richards & Hill, 1959) on the reactivities of α-metallocenyl carbonium ions.

These X-ray results are of significance in the interpretation of the solvolysis rate of α-acetoxy-1,1′-trimethyleneferrocene which is depressed by a surprisingly small degree (*i.e.* by a factor of 291) when compared with an analogue in which the α-carbon atom can be coplanar with the cyclopentadienyl ring to which it is attached, *e.g.* α-acetoxyethylferrocene. The reasons for this small rate difference will be discussed in detail in a paper on the mechanistic interpretation of these solvolytic processes.

Experimental

α-Keto-1,1′-trimethyleneferrocene crystallizes from n-heptane in the form of red-orange monoclinic needles melting at 144·0–144·5°. From a crystal mounted to rotate about the needle axis, *c*, rotation and equi-inclination Weissenberg photographs were obtained for layers 0 through 3. The absence on these photographs of reflections $0k0$ with k odd and $h0l$ with h odd is indicative of the centrosymmetric space group $P2_1/a$.

The unit-cell dimensions were obtained from Straumanis-type, zero-layer Weissenberg photographs taken at room temperature with nickel-filtered copper radiation using crystals mounted about the *b* and *c* axes. The measured Bragg angles for 189 $h0l$ and $hk0$ reflections were used in a least-squares calculation of the unit-cell dimensions; the resulting values are

$$a = 22{\cdot}981 \pm 0{\cdot}002,\ b = 7{\cdot}381 \pm 0{\cdot}001,$$
$$c = 5{\cdot}833 \pm 0{\cdot}001\ \text{Å};\ \beta = 93{\cdot}38 \pm 0{\cdot}02°,$$
$$[\lambda(\text{Cu } K\alpha_1) = 1{\cdot}54051;\ \lambda(\text{Cu } K\alpha_2) = 1{\cdot}54433\ \text{Å}]$$

where the uncertainties listed are about ten times the calculated standard deviations. Absorption and eccentricity parameters were included in the least-squares calculation but were found to be negligible.

The density measured by flotation in mixed solvents was found to be 1·60 g.cm^{-3}; the density calculated on the basis of four molecules of $C_{13}H_{12}OFe$ (M.W. 240·1) in the unit cell is 1·61 g.cm^{-3}.

In order to minimize absorption errors a small acicular crystal, approximately 0·1 mm. in diameter and with roughly hexagonal cross section, was chosen for the *c*-axis intensity photographs. Reflections $hk0$ to $hk5$, inclusive, were recorded using the multiple-film, equi-inclination Weissenberg technique. (α-Keto-1,1′-trimethyleneferrocene is quite stable to X-rays, there being no apparent change in the crystal after exposure to radiation for over 250 hours. However, all X-ray photographs taken from crystals of this compound exhibit a high background due to fluorescent scattering by the iron atoms.) Some difficulty was experienced in cutting a crystal in order to obtain satisfactory diffraction photographs about the *b* axis. The long, slender needles fractured unevenly; after several dozen unsuccessful attempts, a rather unsatisfactory fragment was mounted. From this crystal the reflections $h0l$ to $h2l$, inclusive, were recorded as before. Intensities were estimated by visual comparison with a graduated scale prepared from the crystal mounted to rotate about the *c* axis. After correction for Lorentz and polarization effects, the F^2 values from the several photographs were put on the same arbitrary scale using a weighted least-squares procedure. Because of a number of serious discrepancies in intensities of high-angle reflections measured about the two axes (and, in retrospect, apparently caused by misorientation of the crystal mounted along **b**), the *b*-axis data were used only to scale the *c*-axis data, the latter being used in all subsequent calculations.

Determination and refinement of the structure

The x and y coordinates of the iron atom were readily determined from iron–iron vector peaks in the (001) Patterson projection. Using these coordinates, the contributions of the iron atom to about 100 of the largest $hk0$ structure factors were calculated. For about 25 of these reflections the iron atom contributions were sufficiently large to determine with certainty the signs of the structure factors. A (001) electron density map prepared from these phased structure factors clearly showed the positions of the iron atom, the non–carbonyl carbon atoms of the bridge and one carbon atom in each ring. The atoms of the carbonyl group and the rest of the ring atoms were poorly resolved owing to overlaps in projection. However, from consideration of the geometry of the molecule it was possible to assign trial coordinates to all atoms.

The two-dimensional structure was refined rapidly using a block-diagonal least-squares program written for the Burroughs 220 computer. This program minimizes the quantity $\Sigma w(F_o^2 - F_c^2)^2$. Atomic form factors for carbon and oxygen were an average of the values of Berghuis, Haanappel, Potters, Loopstra, MacGillavry & Veenendaal (1955) and Hoerni & Ibers (1954) and for iron were taken from Thomas & Umeda (1957). Each observation was weighted according to the function $\sqrt{w} = 1/f_C$, where f_C is the form factor of carbon at the calculated value of $\sin\theta/\lambda$. (We have found this weighting function frequently useful for rapid convergence in the initial stages of least-squares refinement.) After several least-squares cycles using individual isotropic temperature factors, the R index, based on observed reflections only, was 0·085.

From packing considerations, approximate z coordinates were calculated for all atoms. The first cycle of three-dimensional structure-factor calculations and least-squares refinement was run using isotropic temperature factors with $B = 3{\cdot}0$ Å^2 for all atoms; the observations were weighted as before. The R index was 0·50 and there were large negative z shifts indicated for

Table 1. *Final heavy atom parameters and their calculated standard deviations*

All values have been multiplied by 10^4. The temperature factors are expressed in the form $T=\exp(-B_{11}h^2-B_{22}k^2-B_{33}l^2-B_{12}hk-B_{13}hl-B_{23}kl)$.

	x	y	z	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Fe	1515(·4)	1259(1)	1913(1)	12(·2)	75(2)	211(4)	−4(1)	−3(1)	4(3)
O	955(3)	4880(7)	6090(9)	26(2)	188(13)	274(21)	34(7)	26(9)	−83(22)
C(1)	1584(3)	3702(8)	3418(12)	12(1)	88(11)	264(25)	−13(6)	−12(9)	−52(21)
C(2)	1925(3)	2429(9)	4727(11)	14(2)	115(12)	262(26)	−9(6)	−32(10)	−13(24)
C(3)	2350(3)	1701(10)	3344(12)	12(2)	157(15)	293(28)	8(6)	−40(10)	−8(26)
C(4)	2278(3)	2544(9)	1141(14)	12(2)	115(13)	404(33)	−15(7)	10(11)	9(29)
C(5)	1794(3)	3767(8)	1161(12)	15(2)	85(11)	264(25)	−23(6)	−11(10)	55(22)
C(6)	634(3)	985(8)	1599(13)	12(2)	101(13)	344(29)	−22(6)	−4(11)	−37(24)
C(7)	892(3)	−438(9)	3029(12)	17(2)	120(13)	243(25)	−28(7)	10(10)	15(24)
C(8)	1285(3)	−1426(8)	1736(14)	14(2)	56(11)	446(33)	−9(6)	23(12)	3(24)
C(9)	1275(4)	−637(9)	−508(13)	22(2)	116(13)	297(29)	−15(8)	−26(12)	−125(27)
C(10)	873(3)	815(8)	−580(12)	14(2)	102(12)	244(26)	−6(6)	−28(10)	−52(22)
C(11)	1014(3)	4421(8)	4123(12)	15(2)	78(11)	256(25)	−3(6)	18(10)	−49(22)
C(12)	501(3)	4343(9)	2391(12)	14(2)	108(12)	275(26)	22(6)	1(10)	−47(24)
C(13)	224(3)	2460(9)	2396(13)	11(2)	148(14)	351(31)	−6(7)	8(11)	−68(29)

many atoms; accordingly, the molecule was moved as a unit by −0·05 in z and another structure-factor least-squares calculation was performed. R for this calculation dropped to 0·37 and the shifts in the z coordinates, although still mostly negative, were considerably smaller. Several more least-squares cycles, in which the individual isotropic temperature factors were permitted to shift, saw R decrease rapidly to 0·12. At this point anisotropic temperature parameters were introduced for the iron atom and the weighting function was changed to that suggested by Hughes (1941). After two more least-squares cycles R had dropped to 0·092. Contributions of the hydrogen atoms were then included in the structure factors, based on assumed C–H bond distances of 1·0 Å and isotropic temperature factors equal to those of the heavy atoms to which they are attached. The parameters of the hydrogen atoms were not refined. Anisotropic temperature factors for the oxygen atom and carbon atoms were introduced, and R dropped to 0·080 after four more least-squares cycles.

The weighting scheme was then changed to

$$\sigma(F^2)=1/\sqrt{w}=KF_o^2 \text{ for } F_o\geq 3\cdot 0$$
$$=3KF_o \text{ for } F_o<3\cdot 0,$$

which we believe adequately represents the uncertainties in F_o^2. After five more cycles of least-squares, R was 0·067 and no parameter shift was larger than one-third of its standard deviation; the refinement was considered complete.

A total of 1466 reflections were included in the final structure-factor calculation, of which 1196 contributed to the least-squares sums (and to the R index). Of the latter, 14 are 'less than' reflections with calculated structure factors larger than their estimated threshold values. Table 1 lists the final heavy-atom parameters and their standard deviations; Table 2 gives the assumed parameters of hydrogen atoms. The standard deviations in the heavy-atom parameters were calculated from the diagonal terms of the inverse block-diagonal matrices. The final observed and calculated structure factors are given in Table 3.

The observed structure factors and their calculated signs were used in the calculation of a three-dimensional electron density map, shown in Fig. 1. A three-dimensional difference map, shown in Fig. 2, was also prepared using ΔF's derived from structure factors calculated from only the heavy atoms, but with the signs of the F_o's being those of the final F_c's. Electron

Table 2. *Assumed hydrogen atom parameters*

The temperature factors are expressed in the form $T=\exp(-B\sin^2\theta/\lambda^2)$.

	Bonded to	x	y	z	B
H(14)	C(2)	0·187	0·211	0·641	3·5
H(15)	C(3)	0·267	0·075	0·374	4·4
H(16)	C(4)	0·252	0·228	−0·020	4·1
H(17)	C(5)	0·164	0·453	0·019	3·2
H(18)	C(7)	0·080	−0·063	0·465	3·9
H(19)	C(8)	0·153	−0·246	0·228	3·7
H(20)	C(9)	0·153	−0·109	−0·183	3·7
H(21)	C(10)	0·073	0·162	−0·192	2·7
H(22)	C(12)	0·066	0·455	0·076	3·8
H(23)	C(12)	0·023	0·531	0·264	3·8
H(24)	C(13)	−0·015	0·245	0·140	4·4
H(25)	C(13)	0·013	0·219	0·403	4·4

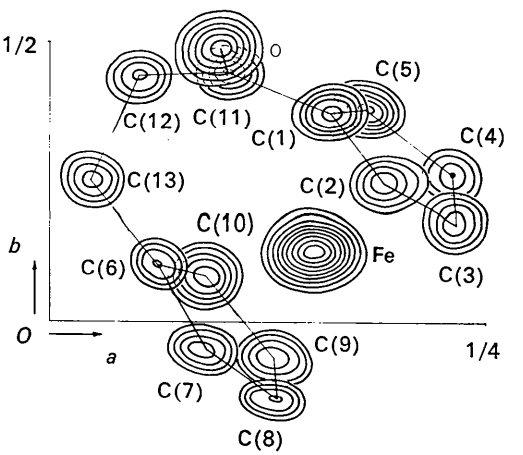

Fig. 1. The final composite three-dimensional electron density map projected onto (001). The contours are drawn at 4, 5, 6,...e.Å^{-3}, except for the iron atom, where the contours are at 5, 10, 15,...e.Å^{-3}.

Table 3. *Observed and calculated structure factors*

The three columns within each group contain the values of h, $10F_o$ and $10F_c$. Observed values indicated by an asterisk (*) were of doubtful validity and were omitted from the least-squares calculations and from the R index.

h00		
0	* ----	4966
4	* 1089	-1286
6	195	184
8	144	137
10	569	-547
12	486	452
14	259	264
16	455	-471
18	93	-92
20	270	288
22	56	-26
24	254	-244
26	163	159
28	144	-134

h01		
4	201	166
6	276	274
8	750	-724
10	630	-570
12	673	609
14	149	-136
16	88	-84
18	248	240
20	137	-131
22	294	-287
24	166	156
26	76	83
28	124	-117
2	551	-660
8	345	330
8	886	977

h02		
2	* 716	-600
4	520	465
6	447	-392
8	504	-526
10	658	742
12	208	195
14	323	-327
16	245	221
18	114	85
20	255	-266
22	< 63	21
24	137	157
26	46	44
4	101	-93
6	* 337	-540
8	231	264

h03		
2	572	561
4	* 586	496
6	754	-757
8	341	301
10	448	448
12	114	-124
14	119	-112
16	230	231
18	149	-145
20	172	-192
22	150	149
24	151	143
4	91	82

h04		
2	557	626
4	132	-117
6	148	-141
8	344	340
10	48	-56
12	313	-334
14	77	78
16	210	207
18	271	-289
20	95	87
22	109	108

h05		
2	184	182
4	399	-386
6	82	77
8	145	156
10	* 209	-269
12	126	-114
14	219	258
16	181	-174
18	< 36	6
20	144	169
4	166	-205

h10		
1	334	394
2	* 986	-1214
3	538	-593
4	540	574
5	234	-232
6	458	-474
7	385	470
8	417	-428
9	158	-166
10	140	131
11	370	-388
12	360	352
13	512	513
14	492	-476
15	221	196
16	50	-41
17	268	-263
18	206	200
19	< 55	-23
20	< 57	-15
21	57	34
22	286	-294
23	122	-148
24	148	171
25	86	85
26	85	87
27	49	67
28	134	-130
29	90	-92

h11		
0	* 1037	-1286
1	171	159
2	204	207
3	618	-625
4	171	-116
5	266	-207
6	571	-537
7	378	358
8	172	-163
9	122	-121
10	722	658
11	309	303
12	< 73	13
13	181	185
14	357	-367
15	398	-410
16	293	279
17	130	-122
18	< 95	49
19	226	255
20	195	-203
21	98	-103
22	95	107
23	< 112	-70
24	175	194
25	162	158
26	73	-79
27	< 79	-43
1	* 1002	1175
2	285	324
3	130	-147
4	82	56
5	* 782	-948
6	448	-490
7	90	-31
8	< 54	4
9	292	318

h12		
0	320	-333
1	271	-262
2	348	350
3	422	410
4	110	92
5	492	473
6	354	-357
7	464	-507
8	543	545
9	279	-261
10	339	343
11	272	282
12	393	392
13	97	-94
14	83	70
15	96	-94
16	188	187
17	254	266
18	147	-162
19	67	-56
20	65	17
21	296	-288
22	140	140
23	187	184
24	< 67	12
25	142	139
26	77	-89
27	88	-94
1	150	-117
2	233	-211
3	399	420
4	162	172
5	374	-363
6	176	-172
7	58	32
8	379	-414
9	481	480

h13		
0	75	68
1	27	- 4
2	510	500
3	480	449
4	313	-280
5	325	-300
6	50	94
7	407	-383
8	299	308
9	328	330
10	295	-273
11	102	85
12	108	-92
13	228	-203
14	138	140
15	128	124
16	48	-11
17	157	161
18	186	-211
19	234	-258
20	116	114
21	55	3
22	53	63
23	174	178
24	79	-73
25	59	-56
1	488	-511
2	179	-182
3	292	277
4	127	124
5	345	329
6	149	161
7	319	-328
8	212	-211
9	212	198

h14		
0	268	302
1	366	393
2	60	22
3	27	11
4	266	-270
5	418	-434
6	206	192
7	155	154
8	166	-130
9	272	256
10	172	-168
11	94	-93
12	< 43	28
13	70	-69
14	179	196
15	110	128
16	278	-254
17	93	-91
18	< 52	-28
19	70	-76
20	178	181
21	116	108
22	89	-81
23	< 32	-18
1	205	-219
2	27	-28
3	339	-316
4	180	-179
5	284	268
6	227	213
7	99	-89
8	74	61
9	166	-152

h15		
0	67	69
1	128	146
2	201	-238
3	73	-69
4	40	-10
5	159	155
6	132	138
7	176	188
8	178	-188
9	156	-164
10	71	-81
11	140	-168
12	230	241
13	99	97
14	50	-55
15	61	55
16	137	-155
17	55	-66
18	176	199
19	< 32	26
1	77	-84
2	84	92
3	300	-346
4	61	-68
5	123	117
6	138	-135
7	301	308
8	181	191
9	217	-230
10	116	-122
11	134	-129

h20		
0	212	218
1	270	-298
2	555	596
3	148	137
4	794	-818
5	153	157
6	545	-561
7	137	-154
8	132	-117
9	359	-377
10	154	157
11	581	608
12	124	128
13	122	122
14	< 47	-40
15	528	-533
16	63	-82
17	65	102
18	< 55	44
19	222	219
20	114	125
21	282	-300
22	< 68	54
23	< 66	-25
24	72	100
25	291	277
26	80	-77
27	84	-85

h21		
0	49	-49
1	251	-279
2	< 49	-32
3	100	24
4	209	-189
5	54	-36
6	276	287
7	440	-474
8	186	-174
9	314	323
10	172	177
11	601	631
12	< 79	- 53
13	539	580
14	198	175
15	299	-290
16	182	182
17	387	413
18	< 95	9
19	< 98	-35
20	< 98	-48
21	244	-266
22	< 114	-26
23	354	318
24	< 103	- 4
25	< 95	40
26	< 85	9
27	162	-180
1	< 49	2
2	160	-154
3	761	824
4	227	241
5	208	219
6	286	296
7	424	-480
8	152	-152
9	240	262
10	326	-340
11	103	75
12	142	-118

h22		
0	77	64
1	69	68
2	162	145
3	401	396
4	167	-131
5	349	-363
6	51	-55
7	310	-309
8	208	200
9	601	671
10	108	104
11	108	-101
12	< 55	- 1
13	409	-402
14	< 61	25
15	251	260
16	65	-66
17	166	178
18	< 67	-21
19	361	-380
20	92	-95
21	155	161
22	85	-74
23	238	244
24	< 65	30
25	198	-209
26	57	-45
1	628	-692
2	59	63
3	369	370
4	38	-27
5	546	550
6	89	86
7	519	-534
8	266	263
9	< 46	2
10	115	-100
11	463	481

h23		
0	276	228
1	488	507
2	219	198
3	32	-38
4	89	-85
5	424	-458
6	36	-12
7	387	394
8	190	-164
9	141	140
10	128	106
11	323	-326
12	45	-50
13	94	91
14	< 48	-25
15	249	249
16	61	-61
17	276	-295
18	98	-83
19	89	-91
20	65	-60
21	338	314
22	50	38
23	111	-102
24	40	27
1	229	-210
2	206	-180
3	552	-588
4	176	-193
5	469	500
6	62	-52
7	81	-43
8	200	177
9	348	-356
10	185	188
11	405	444
12	53	64

h24		
0	73	-89
1	165	188
2	67	-49
3	366	-364
4	107	87
5	208	183
6	56	-45
7	233	231
8	< 40	2
9	315	-320
10	85	-87
11	54	-36
12	89	-84
13	251	271
14	64	52
15	100	-92
16	63	-47
17	267	-255
18	101	89
19	237	206
20	< 43	-10
21	69	56
22	45	36
1	364	351
2	79	-63
3	349	-372
4	70	57
5	166	-146
6	< 34	6
7	317	310
8	36	18
9	367	-350
10	155	-162
11	83	-65

h25		
0	89	80
1	144	-156
2	< 30	- 8
3	49	-46
4	< 32	-10
5	214	253
6	47	-43
7	216	-220
8	< 36	8
9	166	-188
10	< 36	20
11	213	195
12	44	50
13	116	115
14	49	-48
15	198	-213
16	71	78
17	< 36	40
18	54	42
19	100	117
1	289	314
2	80	-68
3	81	74
4	< 30	12
5	312	-331
6	63	-54
7	99	102
8	41	-32
9	106	112
10	50	48
11	211	-258
12	57	-58
13	63	53

h30		
1	527	-542
2	583	-630
3	401	420
4	57	-30
5	168	176
6	239	246
7	286	-293
8	444	-444
9	67	58
10	470	454
11	296	301
12	371	390
13	311	-306
14	216	-232
15	54	51
16	< 55	- 5
17	172	160
18	357	372
19	139	-144
20	126	-124
21	156	-154
22	169	-186
23	206	204
24	58	73
25	< 52	-10
26	47	45
27	50	-53

h31		
0	446	-477
1	347	358
2	< 62	-29
3	124	127
4	549	570
5	320	-353
6	569	-579
7	171	-170
8	< 73	- 9
9	709	738
10	892	849
11	82	-83
12	85	-80
13	395	-387
14	220	-226
15	114	103
16	< 95	- 3
17	< 95	59
18	< 98	-48
19	194	-185
20	234	-237
21	197	178
22	< 110	35
23	103	93
24	135	125
25	197	-165
1	361	-374
2	333	354
3	62	59
4	401	397
5	273	274
6	230	-244
7	325	-343
8	289	270
9	261	-266
10	230	256
11	395	418
12	144	-116
13	85	60
14	189	-157

h32		
0	268	-278
1	493	499
2	321	334
3	128	94
4	103	-83
5	325	-320
6	325	-329
7	304	309
8	481	460
9	< 55	10
10	199	198
11	444	-436
12	374	-390
13	108	112
14	206	-204
15	243	238
16	< 65	9
17	162	-159
18	65	-86
19	< 79	-56
20	< 77	-61
21	204	189
22	212	205
23	139	-138
24	< 57	-38
1	97	-96
2	308	-306
3	547	-675
4	549	560
5	438	428
6	202	-213
7	298	288
8	209	-222
9	320	-328
10	288	296
11	69	-66
12	258	256
13	217	252

h33		
0	134	124
1	81	68
2	172	176
3	393	-392
4	112	-86
5	97	91
6	122	-128
7	265	241
8	207	189
9	315	-316
10	231	-215
11	< 47	-18
12	198	-188
13	202	195
14	216	213
15	< 50	-34
16	< 50	-36
17	154	-151
18	117	-115
19	219	202
20	173	170
21	< 50	-38
22	133	118
23	130	-112
1	437	440
2	328	-358
3	273	-270
4	< 36	36
5	234	-205
6	181	177
7	438	432
8	338	-340
9	181	-165
10	117	-113
11	268	-265
12	270	269
13	233	228

h34		
0	290	306
1	212	-216
2	119	87
3	102	-94
4	207	-218
5	200	201
6	65	27
7	199	-206
8	< 43	4
9	54	-32
10	221	-225
11	190	206
12	313	295
13	55	45
14	133	129
15	204	-204
16	169	-167
17	91	85
18	38	35
19	87	86
20	164	154
21	110	-102
1	280	266
2	98	-85
3	215	185
4	220	-210
5	438	-430
6	55	31
7	64	51
8	< 42	3
9	278	272
10	121	-104
11	175	-158
12	< 45	26
13	55	-55

h35		
0	112	98
1	199	-228
2	219	-236
3	239	257
4	50	52
5	67	67
6	134	152
7	120	-130
8	136	-152
9	44	43
10	< 44	6
11	51	65
12	210	221
13	93	-97
14	46	-58
15	< 38	4
16	86	-93
17	95	109
1	181	-179
2	186	205
3	190	205
4	93	-93
5	69	54
6	< 34	5
7	154	-161
8	58	15
9	132	147
10	44	-36
11	36	-16
12	51	-42
13	180	-102
14	146	146

h40		
0	571	-582
1	259	231
2	276	265
3	152	138
4	719	675
5	< 44	62
6	800	-811
7	121	-110
8	< 47	-63
9	< 49	41
10	715	731
11	< 50	50
12	203	-197
13	< 54	54
14	47	-34
15	< 56	-59
16	196	196
17	< 57	-69
18	< 56	-32
19	< 55	-65
20	265	-243
21	< 63	10
22	120	124
23	< 55	64
24	50	59
25	< 44	14
26	142	-139

h41		
0	428	-451
1	< 73	- 1
2	652	700
3	73	-79
4	< 76	-47
5	179	-171
6	528	-531
7	< 79	0
8	605	608
9	223	201
10	174	-137
11	< 90	11
12	475	-491
13	93	-90
14	135	121
15	192	-184
16	98	86
17	< 98	10
18	192	-164
19	< 116	74
20	< 114	-41
21	122	103
22	181	187
23	211	219
24	633	-714
25	181	174
26	667	741
27	108	105
1	280	263
2	244	-232
3	363	-397
4	167	-142
5	149	130
6	< 88	13
7	328	340
8	160	124
9	211	-194

h42		
0	454	471
1	123	-113
2	53	53
3	< 53	25
4	560	-570
5	96	-92
6	173	152
7	< 57	- 1
8	413	420
9	61	-60
10	430	-422
11	110	131
12	78	-91
13	< 65	-44
14	162	149
15	< 67	-33
16	< 65	- 2
17	< 65	-13
18	89	-86
19	87	85
20	276	242
21	< 67	7
22	< 61	33
23	< 55	3
24	136	-144
1	104	- 87
2	549	-577
3	105	97
4	141	-129
5	237	208
6	435	456
7	57	50
8	336	-350
9	156	-147
10	242	-243
11	< 61	- 9
12	237	249
13	< 65	45
14	244	232

h43		
0	359	390
1	68	59
2	114	-118
3	44	31
4	228	-223
5	< 45	8
6	298	300
7	126	-120
8	47	-34
9	97	88
10	308	-308
11	110	98
12	314	314
13	50	16
14	131	132
15	59	-58
16	208	-195
17	< 57	- 5
18	108	93
19	< 51	34
20	158	162
21	< 42	-18
22	131	-134
1	138	-128
2	105	87
3	61	-52
4	498	-554
5	83	-70
6	141	140
7	85	74
8	173	179
9	47	50
10	246	-260
11	128	101
12	130	-124
13	< 50	18
14	228	229

h44		
0	73	52
1	42	29
2	340	-353
3	60	62
4	123	124
5	< 43	- 4
6	298	312
7	< 45	-22
8	347	-301
9	< 45	22
10	< 45	28
11	< 45	24
12	279	263
13	< 52	22
14	201	-191
15	50	-55
16	48	-56
17	45	- 9
18	156	156
19	36	-37
20	43	-50
1	< 42	13
2	324	342
3	< 42	4
4	67	-51
5	43	-39
6	106	-107
7	172	-152
8	132	132
9	45	-36
10	< 45	-27
11	64	50
12	287	-247
13	175	145

h45		
0	195	-224
1	68	63
2	72	-65
3	51	55
4	223	225
5	51	-46
6	174	-169
7	63	-59
8	153	-151
9	50	-49
10	246	245
11	67	60
12	< 39	3
13	< 38	0
14	204	-212
15	< 33	- 7
16	61	77
1	57	68
2	229	261
3	< 36	5
4	95	106
5	< 36	-31
6	198	-195
7	57	-50
8	< 44	26
9	< 44	-31
10	194	197
11	< 42	11
12	73	-66
13	< 41	- 8
14	151	-123

h50		
1	293	-300
2	245	248
3	247	279
4	329	-352
5	220	215
6	261	-286
7	208	-213
8	366	388
9	114	119
10	116	-124
11	104	107
12	336	-324
13	226	-270
14	204	214
15	< 56	-38
16	68	82
17	198	202
18	193	-187
19	155	-130
20	< 59	38
21	< 55	-34
22	94	98
23	118	114
24	118	-125

h51		
0	247	255
1	< 85	2
2	< 85	-36
3	202	186
4	426	-417
5	241	-248
6	167	154
7	< 90	45
8	112	102
9	247	265
10	299	-292
11	118	-108
12	348	351
13	275	-284
14	98	90
15	152	153
16	190	-192
17	< 114	38
18	155	-112
19	105	-82
20	198	193
21	150	136
1	288	-288
2	381	-389
3	85	73
4	172	-181
5	296	293
6	337	333
7	191	-189
8	130	-120
9	181	-138
10	200	-182
11	301	309
12	214	213
13	< 98	8
14	217	217
15	237	-208

Table 3 (*cont.*)

	h52	
0	< 59	55
1	289	296
2	291	-278
3	61	-69
4	< 61	50
5	77	-77
6	327	330
7	202	197
8	144	-123
9	< 65	13
10	80	72
11	229	-245
12	204	198
13	65	72
14	179	-173
15	157	170
16	140	-155
17	181	-164
18	152	144
19	53	50
20	193	196
21	177	163
22	151	-173
$\bar{1}$	175	-170
$\bar{2}$	213	221
$\bar{3}$	321	-343
$\bar{4}$	356	-367
$\bar{5}$	184	176
$\bar{6}$	98	104
$\bar{7}$	199	198
$\bar{8}$	< 63	-14
$\bar{9}$	249	-267
$\bar{10}$	206	-208
$\bar{11}$	81	86
$\bar{12}$	< 67	-51
$\bar{13}$	162	176
$\bar{14}$	228	226
	h53	
0	126	-130
1	48	-17
2	< 58	22
3	306	-292
4	243	234
5	173	175
6	99	91
7	204	196
8	318	-317
9	179	-167
10	131	122
11	99	-91
12	97	106
·13	211	175
14	201	-176
15	131	-107
16	< 53	-14
17	76	-61
18	181	170
19	176	163
20	116	-110
$\bar{1}$	299	312
$\bar{2}$	206	220
$\bar{3}$	108	- 96
$\bar{4}$	90	68
$\bar{5}$	141	-126
$\bar{6}$	154	-146
$\bar{7}$	167	150
$\bar{8}$	99	101
$\bar{9}$	164	-172
$\bar{10}$	61	- 51
$\bar{11}$	233	-216
$\bar{12}$	168	-142
$\bar{13}$	279	288
	h54	
0	64	- 51
1	218	-190
2	101	72
3	< 45	- 22
4	174	152
5	152	130
6	370	-310
7	140	-132
8	159	-154
9	131	-116
10	272	255
11	217	196
12	50	- 43
13	< 48	5
14	111	-109
15	143	-138
16	95	94
17	34	30
18	< 29	- 30
$\bar{1}$	280	244
$\bar{2}$	78	71
$\bar{3}$	188	160
$\bar{4}$	119	91
$\bar{5}$	254	-223
$\bar{6}$	119	-106
$\bar{7}$	< 55	4
$\bar{8}$	< 55	21
$\bar{9}$	157	154
$\bar{10}$	209	190
$\bar{11}$	217	-197
$\bar{12}$	52	17
$\bar{13}$	59	- 62
	h55	
0	157	-144
1	162	-162
2	86	85
3	96	95
4	123	-112
5	< 42	- 6
6	107	-114
7	137	-127
8	158	151
9	93	88
10	51	57
11	63	65
12	140	-136
13′	66	- 74
$\bar{1}$	79	- 84
$\bar{2}$	50	- 51
$\bar{3}$	238	246
$\bar{4}$	180	176
$\bar{5}$	< 42	- 1
$\bar{6}$	< 42	- 17
$\bar{7}$	109	112
$\bar{8}$	138	-148
$\bar{9}$	106	103
$\bar{10}$	40	39
$\bar{11}$	< 38	15
$\bar{12}$	154	-144
$\bar{13}$	157	-145
	h60	
0	205	-212
1	288	310
2	67	- 84
3	289	288
4	< 55	- 52
5	233	-232
6	< 55	32
7	< 57	4
8	80	108
9	175	185
10	< 57	- 38
11	283	-273
12	< 56	21
13	68	- 81
14	< 67	10
15	296	295
16	< 63	2
17	163	-160
18	< 56	- 4
19	171	-178
20	< 49	12
21	178	196
	h61	
0	116	-119
1	314	331
2	< 95	21
3	233	-245
4	118	118
5	214	-189
6	216	239
7	274	286
8	< 98	- 38
9	< 98	- 39
10	< 98	23
11	289	-272
12	< 116	- 14
13	188	176
14	< 112	- 30
15	< 110	72
16	< 105	19
17	271	-244
18	< 93	- 48
19	174	163
$\bar{1}$	133	130
$\bar{2}$	< 95	- 68
$\bar{3}$	212	-235
$\bar{4}$	116	-109
$\bar{5}$	< 95	88
$\bar{6}$	< 95	2
$\bar{7}$	< 95	83
$\bar{8}$	< 98	- 37
$\bar{9}$	376	-372
$\bar{10}$	< 98	54
$\bar{11}$	< 95	- 6
$\bar{12}$	118	-109
$\bar{13}$	396	382
	h62	
0	< 65	- 68
1	80	- 97
2	< 65	22
3	246	-253
4	< 65	41
5	229	237
6	155	164
7	81	68
8	< 65	- 9
9	307	-313
10	< 79	- 37
11	78	60
12	140	-137
13	325	297
14	< 73	19
15	217	-198
16	85	- 89
17	72	- 77
18	124	123
19	270	283
$\bar{1}$	307	314
$\bar{2}$	207	207
$\bar{3}$	81	- 84
$\bar{4}$	132	135
$\bar{5}$	114	-115
$\bar{6}$	115	-120
$\bar{7}$	317	314
$\bar{8}$	< 67	- 75
$\bar{9}$	114	-108
$\bar{10}$	65	- 63
$\bar{11}$	395	-370
$\bar{12}$	120	114
$\bar{13}$	218	222
	h63	
0	86	73
1	314	-320
2	< 50	- 42
3	144	128
4	< 50	33
5	249	218
6	48	-50
7	298	-264
8	170	-141
9	120	- 98
10	< 58	- 10
11	341	311
12	< 53	33
13	52	- 35
14	< 48	- 17
15	239	-216
16	< 42	- 15
17	177	178
$\bar{1}$	93	76
$\bar{2}$	50	- 44
$\bar{3}$	204	211
$\bar{4}$	157	151
$\bar{5}$	237	-261
$\bar{6}$	< 61	2
$\bar{7}$	< 60	42
$\bar{8}$	< 60	14
$\bar{9}$	364	358
$\bar{10}$	< 58	2
$\bar{11}$	222	-216
$\bar{12}$	< 56	36
$\bar{13}$	166	-164
	h64	
0	81	- 78
1	122	-124
2	122	-110
3	270	247
4	< 51	- 35
5	93	- 88
6	51	- 49
7	194	-174
8	79	62
9	228	205
10	< 45	17
11	70	71
12	< 40	0
13	194	-182
14	< 34	12
$\bar{1}$	252	-215
$\bar{2}$	68	54
$\bar{3}$	304	270
$\bar{4}$	68	55
$\bar{5}$	< 43	40
$\bar{6}$	< 42	10
$\bar{7}$	329	-302
$\bar{8}$	50	- 54
$\bar{9}$	151	137
$\bar{10}$	48	- 51
$\bar{11}$	184	169
	h65	
0	38	29
1	167	164
2	53	- 30
3	67	64
4	51	- 46
5	181	-185
6	43	43
7	82	- 66
8	34	26
9	95	114
$\bar{1}$	194	-181
$\bar{2}$	< 38	- 16
$\bar{3}$	< 38	- 26
$\bar{4}$	< 38	- 1
$\bar{5}$	188	178
$\bar{6}$	< 36	- 36
$\bar{7}$	135	-130
$\bar{8}$	< 34	- 28
$\bar{9}$	84	- 97
$\bar{10}$	< 30	- 17
$\bar{11}$	146	164
	h70	
1	80	102
2	338	343
3	149	-170
4	159	-179
5	< 56	9
6	< 55	- 29
7	218	228
8	219	238
9	94	-101
10	92	- 65
11	230	-238
12	124	-148
13	138	137
14	132	138
15	< 54	- 1
16	114	104
17	85	- 88
18	84	- 88
19	107	120
	h71	
0	305	337
1	192	-207
2	118	94
3	203	-219
4	179	-165
5	223	239
6	190	159
7	114	107
8	< 114	1
9	258	-263
10	155	-160
11	< 108	34
12	< 103	23
13	172	162
14	204	213
15	116	-117
16	166	-181
$\bar{1}$	236	244
$\bar{2}$	167	-165
$\bar{3}$	< 118	61
$\bar{4}$	166	-154
$\bar{5}$	166	-142
$\bar{6}$	212	236
$\bar{7}$	116	101
$\bar{8}$	< 114	- 30
$\bar{9}$	145	126
$\bar{10}$	191	-184
$\bar{11}$	187	-177
$\bar{12}$	242	203
	h72	
0	203	188
1	308	-302
2	249	-247
3	111	122
4	< 78	- 2
5	227	215
6	216	184
7	190	-188
•8	134	-139
9	84	- 84
10	140	-142
11	239	231
12	140	143
13	< 61	- 13
14	83	- 80
15	164	-166
16	108	-116
$\bar{1}$	< 78	- 20
$\bar{2}$	< 78	59
$\bar{3}$	257	235
$\bar{4}$	< 78	- 60
$\bar{5}$	196	180
$\bar{6}$	< 77	4
$\bar{7}$	61	- 80
$\bar{8}$	194	189
$\bar{9}$	276	270
$\bar{10}$	150	-162
	h73	
0	97	- 85
1	151	-122
2	213	-221
3	169	164
4	227	220
5	< 54	- 22
6	< 53	- 31
7	59	- 64
8	186	-179
9.	164	163
10	< 47	46
11	< 44	- 5
12	89	87
13	178	-189
$\bar{1}$	215	208
$\bar{2}$	145	131
$\bar{3}$	247	259
$\bar{4}$	< 56	- 36
$\bar{5}$	155	132
$\bar{6}$	< 54	- 55
$\bar{7}$	347	-349
$\bar{8}$	52	55
$\bar{9}$	< 51	- 19
$\bar{10}$	< 48	27
$\bar{11}$	190	200
$\bar{12}$	141	-147
	h74	
0	170	-158
1	182	173
2	56	62
3	< 43	44
4	92	94
5	168	-162
6	126	-118
7	70	66
8	< 36	- 32
9	48	47
10	142	147
$\bar{1}$	124	-117
$\bar{2}$	45	49
$\bar{3}$	89	- 81
$\bar{4}$	71	61
$\bar{5}$	220	198
$\bar{6}$	98	-101
$\bar{7}$	< 42	- 44
$\bar{8}$	< 40	- 10
$\bar{9}$	194	-186
	h80	
0	350	343
1	< 62	58
2	170	-180
3	150	133
4	168	-178
5	< 59	17
6	346	364
7	< 58	- 31
8	< 56	26
9	83	- 85
10	229	-250
11	< 49	- 5
12	85	102
13	< 44	12
14	136	149
	h81	
0	105	101
1	< 103	43
2	315	-324
3	< 103	24
4	219	210
5	< 100	- 10
6	187	179
7	< 95	- 25
8	263	-258
9	< 90	- 14
10	85	- 86
$\bar{1}$	< 103	41
$\bar{2}$	247	229
$\bar{3}$	< 103	- 21
$\bar{4}$	276	-282
$\bar{5}$	< 100	- 65
$\bar{6}$	98	- 83
$\bar{7}$	< 95	14
$\bar{8}$	•308	330
$\bar{9}$	< 90	- 24
$\bar{10}$	88	79
	h82	
0	196	-204
1	< 67	12
2	208	-227
3	< 65	- 14
4	272	272
5	< 63	2
6	61	- 59
7	< 59	- 2
8	174	-175
9	< 55	- 12
10	132	127
11	46	- 47
$\bar{1}$	< 67	3
$\bar{2}$	281	294
$\bar{3}$	< 65	42
$\bar{4}$	106	118
$\bar{5}$	< 63	- 7
$\bar{6}$	256	-274
$\bar{7}$	< 61	- 18
$\bar{8}$	84	- 58
$\bar{9}$	< 57	2
$\bar{10}$	147	176
	h83	
0	255	-276
1	< 44	- 33
2	143	140
3	< 42	8
4	125	130
5	< 40	17
6	194	-217
$\bar{1}$	< 46	- 9
$\bar{2}$	< 46	33
$\bar{3}$	< 42	- 17
$\bar{4}$	154	168
$\bar{5}$	42	42
$\bar{6}$	< 40	- 32
$\bar{7}$	< 38	29
$\bar{8}$	106	120
	h90	
1	88	90
2	131	-144
3	55	- 66
4	184	217
5	< 40	2
6	< 38	32
7	78	84
8	144	-186

density maps in the best planes of the two cyclopentadienyl rings (Table 5) are shown in Fig. 3.

Discussion of the structure

Molecular dimensions

The bond angles and interatomic distances were calculated from the final refined atomic coordinates

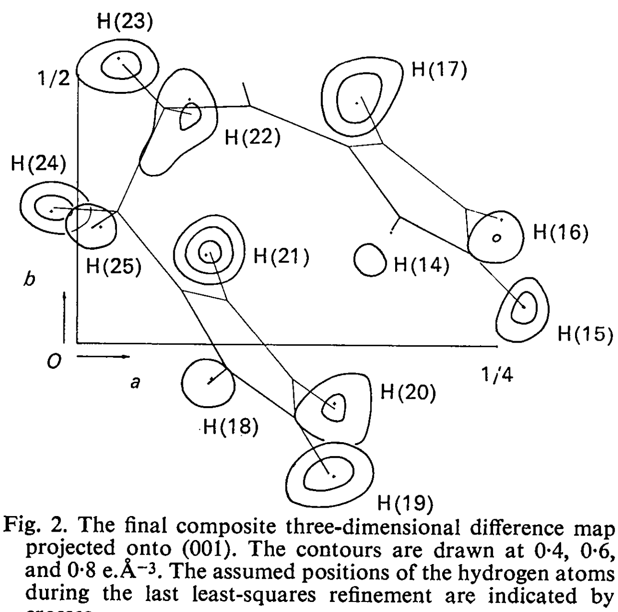

Fig. 2. The final composite three-dimensional difference map projected onto (001). The contours are drawn at 0·4, 0·6, and 0·8 e.Å^{-3}. The assumed positions of the hydrogen atoms during the last least-squares refinement are indicated by crosses.

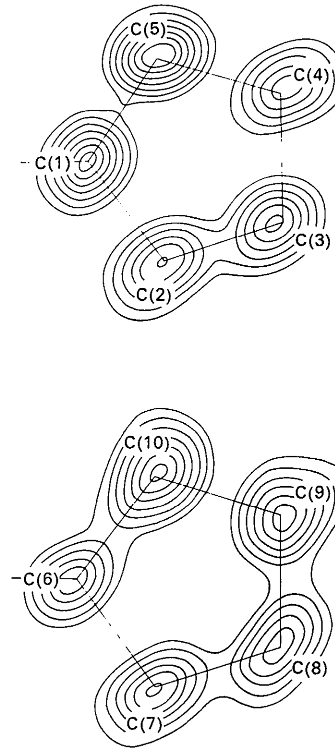

Fig. 3. Electron density maps in the best planes of the cyclopentadienyl rings. The contours are drawn at 2, 3, 4,. . . e.Å^{-3}.

Table 4. *Magnitudes and direction cosines of the principal axes of the thermal ellipsoids*

The direction cosines, q, are taken relative to a, b and c^*. The root mean square deviation, U, is given by $(B/8\pi^2)^{\frac{1}{2}}$.

	Axis i	B_i	U_i	q_{ia}	q_{ib}	q_{ic}^*		Axis i	B_i	U_i	q_{ia}	q_{ib}	q_{ic}^*
Fe	1	2·93	0·192	−0·346	0·061	0·926	C(7)	1	4·19	0·231	0·854	−0·509	0·122
	2	2·39	0·174	0·925	−0·136	0·379		2	3·32	0·205	−0·003	0·182	0·983
	3	1·61	0·143	0·148	0·989	−0·005		3	1·99	0·159	0·517	0·841	−0·139
O	1	6·20	0·279	0·879	0·468	0·099	C(8)	1	6·11	0·278	0·138	−0·003	0·994
	2	4·49	0·239	0·257	−0·602	0·763		2	2·99	0·194	0·975	−0·169	−0·108
	3	2·64	0·183	−0·397	0·647	0·639		3	1·16	0·121	0·168	0·986	−0·016
C(1)	1	3·78	0·219	−0·271	−0·165	0·940	C(9)	1	5·32	0·260	0·801	0·090	−0·567
	2	2·78	0·188	0·846	−0·509	0·179		2	4·26	0·232	0·535	−0·546	0·660
	3	1·53	0·139	0·457	0·844	0·292		3	1·74	0·148	0·264	0·832	0·494
C(2)	1	4·30	0·232	−0·610	0·055	0·772	C(10)	1	4·05	0·226	−0·618	−0·122	0·758
	2	2·72	0·186	−0·437	0·808	−0·407		2	2·70	0·185	−0·637	0·655	−0·424
	3	2·08	0·162	0·659	0·587	0·489		3	1·81	0·152	0·458	0·746	0·497
C(3)	1	4·70	0·243	−0·502	−0·152	0·836	C(11)	1	3·80	0·219	0·483	−0·193	0·868
	2	3·41	0·208	0·035	0·980	0·197		2	2·86	0·190	0·874	0·107	−0·446
	3	1·87	0·154	0·863	−0·128	0·512		3	1·61	0·143	−0·001	0·975	0·220
C(4)	1	5·48	0·264	0·034	0·021	0·999	C(12)	1	3·95	0·224	−0·339	−0·383	0·849
	2	2·98	0·194	−0·684	0·728	−0·012		2	3·26	0·203	0·756	0·431	0·514
	3	1·97	0·158	0·727	0·685	−0·018		3	1·75	0·149	−0·558	0·817	0·127
C(5)	1	4·14	0·230	−0·595	0·358	0·701	C(13)	1	4·96	0·251	0·053	−0·321	0·947
	2	3·02	0·196	0·700	−0·205	0·704		2	3·05	0·197	−0·211	0·922	0·319
	3	1·45	0·135	0·392	0·911	−0·118		3	2·31	0·171	0·974	0·217	0·047
C(6)	1	4·72	0·244	−0·074	−0·105	0·990							
	2	3·09	0·198	0·767	−0·640	0·012							
	3	1·51	0·138	0·635	0·761	0·146							

and are shown in Fig. 4. The standard deviations in the positional parameters (Table 1) are approximately 0·007 Å for the oxygen and carbon atoms and 0·0009 Å for the iron atom. These values correspond to uncertainties of approximately 0·010 Å for the C–C and C–O bonds and 0·007 Å for the Fe–C bonds. The excellent agreement among the ten C–C bond lengths in the rings indicates that these standard deviations are realistic.

Temperature parameters

The magnitudes and orientations of the thermal ellipsoids for each of the heavy atoms were calculated by the method of Rollett & Davies (1955) and are listed in Table 4. The anisotropies in the thermal motions are sizable. The major and minor axes of the ellipsoids for all atoms except the oxygen atom and C(7) are oriented approximately along the c and a axes, respectively, suggesting a relatively large lattice vibration. (Since only c-axis data were used in the refinement of the structure, the thermal anisotropy may be, in part at least, an artifact arising from systematic errors in the data.) Superimposed on this vibration are the motions of the individual atoms; the libration of the ring carbon atoms may be clearly seen in Fig. 3.

Molecular geometry

The least-squares planes (Schomaker, Waser, Marsh & Bergman, 1959) through each of the cyclopentadienyl rings and through the carbonyl group are given in Table 5. The ring atoms do not deviate significantly from coplanarity. The three-carbon bridge causes tilting of the two cyclopentadienyl rings so they are no longer parallel as in the parent compound, ferrocene, but without causing noticeable ring bond distortions. It also appears that there is little strain in the α-ketotrimethylene bridge, since the bond lengths and angles are quite close to the expected values. The dihedral angle between the cyclopentadienyl rings, defined here as the acute angle formed by the normals to the best planes, is 8·8°.

Table 5. *Best planes through the rings and the carbonyl group*

The values q_i are the direction cosines of the normal to the best plane with respect to the orthogonal axes a, b, and c^*. Atoms indicated by an asterisk (*) were not included in the calculation of the plane.

Direction cosines	Atom	Deviation
q_a = 0·613	C(1)	−0·005 Å
q_b = 0·731	C(2)	−0·001
q_c^* = 0·300	C(3)	0·006
	C(4)	−0·010
	C(5)	0·009
	*C(11)	0·301
	*Fe	1·640
	*Origin	4·748
q_a = 0·722	C(6)	−0·006
q_b = 0·622	C(7)	0·003
q_c^* = 0·303	C(8)	0·001
	C(9)	−0·004
	C(10)	0·006
	*C(13)	−0·123
	*Fe	−1·640
	*Origin	1·741
q_a = 0·246	C(1)	−0·012
q_b = 0·929	C(11)	0·039
q_c^* = −0·276	C(12)	−0·012
	O	−0·012
	*C(13)	−1·462
	*Origin	−2·870

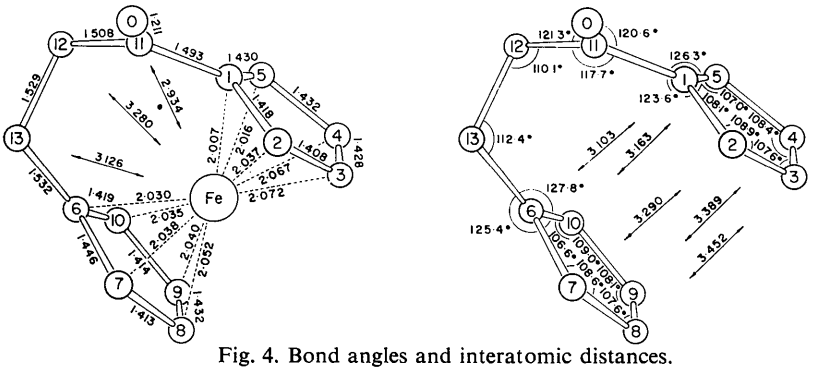

Fig. 4. Bond angles and interatomic distances.

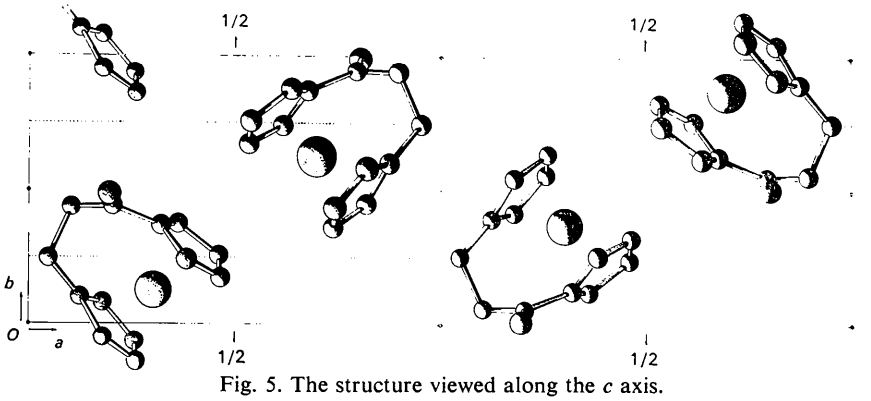

Fig. 5. The structure viewed along the *c* axis.

In the crystal structure of ferrocene as determined by Dunitz, Orgel & Rich (1956) the iron atoms lie at centers of symmetry and the rings are therefore staggered by exactly 36°. In our bridged ketone the rings are more nearly eclipsed, being staggered by only 11·8°. (The degree of staggering is defined here as the angle between the lines from the centroid of each ring to the carbon atom attached to the bridge after the lines have been projected onto an 'average' plane. The 'average' plane is taken so that its normal bisects the acute angle formed by the normals to the best planes through the two rings.) The average ring C–C bond length in ferrocene is 1·403 ± 0·020 Å and the average Fe–C bond is 2·045 ± 0·010 Å; for the bridged ketone these bonds are 1·424 ± 0·010 Å and 2·039 ± 0·006 Å, respectively. Thus the two determinations agree well within the estimated standard deviations.

Table 6. *Intermolecular distances less than* 3·7 Å

The atoms in the first column belong to the reference molecule at *x*, *y*, *z*.

From atom	To atom	In molecule at	Distance
C(11)	C(8)	$x, 1+y, z$	3·439
O	C(13)	$\bar{x}, 1-y, 1-z$	3·501
O	C(5)	$x, y, 1+z$	3·532
O	C(12)	$\bar{x}, 1-y, 1-z$	3·559
O	C(10)	$x, y, 1+z$	3·585
C(10)	C(13)	$\bar{x}, \bar{y}, \bar{z}$	3·606
C(4)	C(9)	$\frac{1}{2}-x, \frac{1}{2}+y, \bar{z}$	3·624
C(12)	C(8)	$x, 1+y, z$	3·637
C(12)	C(12)	$\bar{x}, 1-y, \bar{z}$	3·642
C(6)	C(6)	$\bar{x}, \bar{y}, \bar{z}$	3·668

Molecular packing

The molecular packing as viewed along the *c* axis is shown in Fig. 5 and the shortest intermolecular distances are listed in Table 6. The H–H and H-heavy atom distances are not included since the coordinates of the hydrogen atoms are only assumed.

We wish to thank Dr E. A. Hill, who supplied the sample of α-keto-1,1′-trimethyleneferrocene used in this study. One of us (N. D. J.) is grateful to the Division of General Medical Sciences, United States Public Health Service for a Predoctoral Fellowship during the tenure of which this investigation was carried out.

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