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The Crystal and Molecular Structure of Biferrocenyl

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Crystals of biferrocenyl, $C_5H_5.Fe.C_5H_4.C_5H_4.Fe.C_5H_5$, are monoclinic, with $a=10{\cdot}35$, $b=7{\cdot}87$, $c=12{\cdot}63$ Å, $\beta=131{\cdot}72°$, The space group is $P2_1/c$ and there are two molecules in the unit cell, so that a molecular centre of symmetry is required. The intensities of 1111 reflexions were measured with a scintillation counter, Mo $K\alpha$ radiation being used. The structure was determined by Patterson and Fourier methods, and refined by least squares and differential syntheses. The final R value for 896 observed reflexions is 0·16.

Each iron atom is sandwiched between two five-membered rings which are planar, approximately parallel, separated by 3·32 Å, and oriented about midway between the fully eclipsed and fully staggered conformations. The two five-membered rings which are bonded together are coplanar, and the molecular configuration is *trans*. The mean bond distances are Fe–C = 2·04 Å, C–C (in cyclopentadienyl rings) = 1·40 Å, C–C (between rings) = 1·48 Å. There are however deviations from fivefold symmetry which are possibly significant; these deviations are the same in both crystallographically independent rings, and are similar to those observed (but considered not significant) in ferrocene. All the intermolecular separations correspond to normal van der Waals interactions.

Introduction

The crystal structure of biferrocenyl (Fig. 1), prepared by Rausch (1960), has been investigated to determine the molecular structure, and in particular whether the pairs of cyclopentadienyl rings are staggered as in other ferrocenyl derivatives (Trotter, 1963).

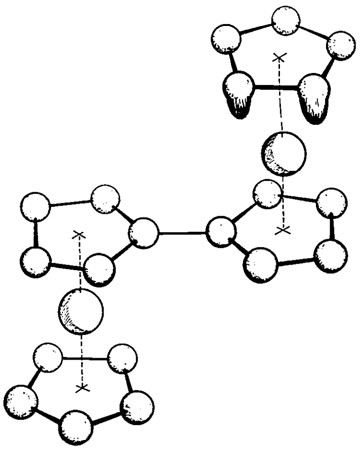

Fig. 1. Biferrocenyl.

Experimental

Crystals of biferrocenyl are dark orange plates elongated along b with (100) developed. The density was measured by flotation in aqueous potassium iodide, and the unit-cell dimensions and space group were determined from various rotation, Weissenberg and precession photographs, and on the G.E. Spectrogoniometer.

Crystal data (λ, Cu $K\alpha=1{\cdot}5418$; λ, Mo $K\alpha=0{\cdot}7107$ Å)

Biferrocenyl,

$C_{20}H_{18}Fe_2$; M.W. 370·0; m.p. 239–240 °C.

Monoclinic,

$a=10{\cdot}35\pm0{\cdot}02$, $b=7{\cdot}87\pm0{\cdot}02$, $c=12{\cdot}63\pm0{\cdot}02$ Å; $\beta=131{\cdot}72\pm0{\cdot}05°$.

Volume of the unit cell: 768 Å^3.

$D_m=1{\cdot}61$ g.cm^{-3}.

D_x (with $Z=2$) = 1·60 g.cm^{-3}.

Absorption coefficients for X-rays:

$\lambda=1{\cdot}5418$ Å, $\mu=162$ cm^{-1}.

$\lambda=0{\cdot}7107$ Å, $\mu=19{\cdot}2$ cm^{-1}.

$F(000)$ = 380.

Absent reflexions: $h0l$ when l is odd, $0k0$ when k is odd. Space group is $P2_1/c$ (C_{2h}^5).

The intensities of the reflexions were measured on a General Electric XRD 5 Spectrogoniometer with Single Crystal Orienter, using a scintillation counter and Mo $K\alpha$ radiation, an approximately monochromatic beam being obtained by use of a zirconium filter and a pulse height analyser. The moving crystal–moving counter technique (Furnas, 1957) was used. All the 1111 reflexions in the range $0<2\theta\leq49°$ (corresponding to a minimum interplanar spacing $d=0{\cdot}86$ Å) were examined and 896 had an intensity above background, 81% of the total number of reflexions in the range. All the intensities were corrected for background, which was found to be approximately a function of θ only. All the crystals were rather thin plates; the one used in recording the intensities was mounted with b parallel to the φ axis of the goniostat, and had cross-section 0·30 × 0·03 mm. No absorption corrections were applied, but absorption is not negligible even for Mo $K\alpha$ radiation, and errors of up to about 19% in structure amplitude are possible for a few reflexions as a result of absorption (for most planes it is much less than this). Lorentz and polarization corrections were made and the structure amplitudes derived.

Structure analysis

Since there are two molecules in the unit cell a molecular centre of symmetry is required, so that the molecule has a *trans* configuration. The iron atom position was determined from the three axial Patterson projections, and a three-dimensional Fourier series was summed, with signs of the structure amplitudes derived from the iron contributions only. On the resulting electron-density map all the iron and carbon atoms were well-resolved. Structure factors were calculated by use of the Fe and C scattering factors of *International Tables for X-ray Crystallography* (1962), with $B = 4{\cdot}5$ Å^2 for all the atoms; the discrepancy index R was 29·8% for the observed reflexions.

Table 1. *Progress of analysis*

Coordinates from	R
Patterson projections–Fe only	—
1st 3-D Fourier synthesis	0·298
1st differential synthesis	0·188
1st least-squares	0·170
2nd least-squares	0·165
2nd differential syntheses	0·163
3rd differential syntheses	—

Refinement of the positional and isotropic thermal parameters proceeded (Table 1) by computation of a differential synthesis with observed structure amplitudes and calculated signs; R was reduced to 18·8%. Two cycles of least squares, with a program previously described (Camerman & Trotter, 1964), reduced R successively to 17·0% and 16·5%. The function minimized was $\Sigma w(F_o - F_c)^2$, with $\sqrt{w} = F_o/30$ when $F_o < 30$ and $\sqrt{w} = 30/F_o$ when $F_o \geq 30$. Refinement was then continued with a cycle of observed and calculated differential syntheses (Ahmed & Cruickshank, 1953). New positional parameters were obtained (corrected for series termination by the 'backshift' method (Booth, 1946)), and in addition an examination of the peak electron densities and curvatures suggested significantly smaller thermal factors for most of the atoms (the B values from the second least squares and from second differential syntheses are included in Table 4).

Application of these various shifts reduced R to 16·3% (Table 1). A third cycle of differential syntheses was then computed, and the observed and calculated peak electron densities and curvatures are listed in Table 2. These suggest further temperature factor reductions for most of the atoms, but it is felt that some of the differences between the observed and calculated densities and curvatures might be a result of absorption errors, and that the least-squares B values are probably more reliable. The variation of thermal parameters is actually about the same in both sets listed in Table 4, but the least-squares values are generally higher. The third differential cycle indicated no changes in the Fe positional parameters and only small changes in those of the C atoms, the mean shifts in the x, y, and z directions being 0·010, 0·008, and 0·009 Å respectively. Since these changes would produce negligible differences in calculated structure factors, the final values were not computed, but those at the penultimate stage ($R = 16{\cdot}3\%$) are listed together with the measured values in Table 3. In view of the possible absorption errors the agreement is satisfactory.

Table 2. *Peak electron densities* (e.Å^{-3}) *and curvatures* (e.Å^{-5}) *from third differential syntheses*

	ϱ		$-\partial^2\varrho/\partial x^2$		$-\partial^2\varrho/\partial y^2$		$-\partial^2\varrho/\partial z^2$	
Atom	Obs.	Calc.	Obs.	Calc.	Obs.	Calc.	Obs.	Calc.
C(1)	5·0	4·7	20·2	17·4	38·4	35·4	20·4	20·6
C(2)	5·1	4·8	34·0	25·7	34·3	35·5	24·0	20·7
C(3)	5·6	5·3	40·1	30·2	26·9	28·9	31·0	27·8
C(4)	5·7	5·5	48·8	44·4	30·8	30·2	34·1	32·4
C(5)	6·1	5·9	37·8	36·1	42·8	42·0	32·2	31·7
C(6)	6·1	6·4	33·5	37·9	40·3	47·0	30·5	30·5
C(7)	7·2	7·0	50·1	46·7	55·9	51·6	43·0	42·3
C(8)	6·6	6·3	58·5	52·1	41·8	36·8	41·8	36·4
C(9)	6·0	5·7	44·1	40·8	31·2	30·9	37·0	32·6
C(10)	5·5	5·1	33·2	29·0	28·0	28·0	29·8	24·1
Fe(11)	44·2	45·8	346·8	343·5	323·9	358·3	292·7	299·5

A final three-dimensional Fourier series was summed, and superimposed sections of the resulting

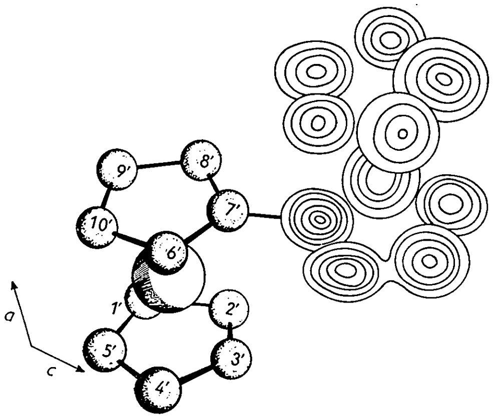

Fig. 2. Superimposed sections of the three-dimensional electron-density distribution, taken through the atomic centres parallel to (010). Contours start at 2 e.Å^{-2} and are at intervals of 1 e.Å^{-2}; the iron atom is omitted. A drawing of half a molecule, showing the numbering used, is also given.

Table 3. *Measured and calculated structure factors*

Unobserved reflexions, for which F_o is listed as 0·0, have threshold values in the range 5–10

H	K	L	F OBS	F CALC
1	0	0	48.9	44.9
2	0	0	79.8	-70.1
3	0	0	40.3	-36.5
4	0	0	66.5	57.2
5	0	0	52.4	47.2
6	0	0	18.5	-8.3
7	0	0	32.2	-28.0
8	0	0	14.6	-10.5
-9	0	2	16.3	-15.4
-8	0	2	6.4	-3.8
-7	0	2	27.0	28.0
-6	0	2	44.6	35.8
-5	0	2	48.1	-40.1
-4	0	2	65.7	-50.1
-3	0	2	5.1	4.7
-2	0	2	136.5	122.1
-1	0	2	61.8	56.4
0	0	2	184.1	-156.5
1	0	2	56.6	-52.5
2	0	2	56.2	51.5
3	0	2	76.4	73.3
4	0	2	22.3	-19.3
5	0	2	29.2	-25.1
6	0	2	9.9	-7.5
7	0	2	24.5	17.7
-10	0	4	0.0	-3.3
-9	0	4	15.4	14.1
-8	0	4	21.0	20.2
-7	0	4	13.7	-14.9
-6	0	4	54.9	-48.3
-5	0	4	9.0	10.1
-4	0	4	71.2	60.7
-3	0	4	18.9	18.6
-2	0	4	81.1	-76.1
-1	0	4	42.9	-35.9
0	0	4	35.6	32.0
1	0	4	57.5	50.6
2	0	4	11.2	-11.8
3	0	4	50.6	-44.9
4	0	4	9.4	-4.6
5	0	4	12.9	12.0
6	0	4	13.3	8.5
-11	0	6	14.2	14.4
-10	0	6	18.5	19.6
-9	0	6	7.3	-5.2
-8	0	6	17.2	-15.4
-7	0	6	7.3	7.9
-6	0	6	72.5	64.7
-5	0	6	33.5	28.6
-4	0	6	76.8	-69.6
-3	0	6	55.4	-42.1
-2	0	6	24.0	25.6
-1	0	6	24.5	17.8
0	0	6	14.2	-22.3
1	0	6	46.9	-44.2
2	0	6	21.5	-17.3
3	0	6	13.3	11.7
4	0	6	10.7	6.8
-11	0	8	10.3	-10.3
-10	0	8	25.7	-23.9
-9	0	8	6.4	8.4
-8	0	8	33.0	27.2
-7	0	8	19.7	18.5
-6	0	8	59.2	-55.5
-5	0	8	41.6	-37.5
-4	0	8	44.6	38.6
-3	0	8	60.1	52.9
-2	0	8	10.7	-12.5
-1	0	8	42.1	-34.6
0	0	8	7.3	-1.7
1	0	8	19.7	17.8
2	0	8	15.0	12.9
3	0	8	6.0	-7.5
-12	0	10	22.3	-15.5
-11	0	10	0.0	2.1
-10	0	10	26.2	21.3
-9	0	10	0.0	3.6
-8	0	10	35.6	-31.5
-7	0	10	33.0	-30.3
-6	0	10	31.8	29.0
-5	0	10	37.3	30.0
-4	0	10	6.9	-6.5
-3	0	10	53.2	-45.7
-2	0	10	11.6	-5.3
-1	0	10	24.0	21.5
0	0	10	13.7	11.8
1	0	10	7.7	-7.7
1	1	0	139.0	-128.8
2	1	0	10.3	2.5
3	1	0	41.6	40.5
4	1	0	41.6	41.0
5	1	0	33.5	-30.3
6	1	0	25.7	-23.6
7	1	0	0.0	0.6
8	1	0	27.5	20.8
-9	1	1	0.0	-3.6
-8	1	1	6.9	-8.4
-7	1	1	5.1	4.2
-6	1	1	25.3	22.2
-5	1	1	9.9	10.0
-4	1	1	20.6	-22.9
-3	1	1	4.7	3.0
-2	1	1	96.1	87.2
-1	1	1	51.9	61.8
0	1	1	23.2	29.0
1	1	1	15.0	-13.7
2	1	1	16.7	-21.7
3	1	1	19.7	18.7
4	1	1	27.5	26.2
5	1	1	0.0	-0.5
6	1	1	15.4	-15.1
7	1	1	0.0	-2.7
8	1	1	0.0	2.4
-9	1	2	0.0	-3.2
-8	1	2	22.3	21.3
-7	1	2	18.9	19.4
-6	1	2	33.0	-30.6
-5	1	2	61.4	-54.9
-4	1	2	14.6	11.4
-3	1	2	18.5	22.8
-2	1	2	30.9	32.0
-1	1	2	120.1	-128.3
0	1	2	43.3	-45.1
1	1	2	47.2	47.8
2	1	2	56.2	51.1
3	1	2	29.6	-29.4
4	1	2	33.7	-33.7
5	1	2	11.6	10.4
6	1	2	21.5	18.6
7	1	2	15.4	9.1
-10	1	3	0.0	-2.8
-9	1	3	0.0	1.0
-8	1	3	15.4	13.4
-7	1	3	0.0	1.6
-6	1	3	21.5	-19.4
-5	1	3	18.0	-18.4
-4	1	3	34.8	32.8
-3	1	3	10.7	18.9
-2	1	3	27.9	-29.7
-1	1	3	36.0	-39.8
0	1	3	12.0	-14.4
1	1	3	30.0	30.8
2	1	3	22.3	22.9
3	1	3	0.0	-4.1
4	1	3	17.2	-17.2
5	1	3	0.0	1.3
6	1	3	6.9	8.1
7	1	3	0.0	0.8
-10	1	4	10.3	12.5
-9	1	4	15.9	15.8
-8	1	4	11.6	-11.8
-7	1	4	34.3	-30.0
-6	1	4	21.9	18.7
-5	1	4	71.2	65.5
-4	1	4	12.4	13.3
-3	1	4	38.6	-40.4
-2	1	4	26.2	-27.8
-1	1	4	39.5	44.0
0	1	4	26.2	30.3
1	1	4	17.2	-16.5
2	1	4	59.2	-55.9
3	1	4	5.6	2.0
4	1	4	23.2	13.8
5	1	4	5.6	2.1
6	1	4	15.9	-13.0
-11	1	5	0.0	-1.9
-10	1	5	11.6	9.6
-9	1	5	11.6	11.2
-8	1	5	6.9	-3.2
-7	1	5	8.6	-8.5
-6	1	5	15.4	16.6
-5	1	5	32.6	32.4
-4	1	5	7.3	4.4
-3	1	5	11.2	-13.4
-2	1	5	37.3	-31.1
-1	1	5	7.7	8.7
0	1	5	12.4	12.8
1	1	5	17.2	-14.9
2	1	5	24.9	-27.5
3	1	5	11.2	-9.7
4	1	5	3.9	4.5
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-11	1	6	7.3	8.5
-10	1	6	10.3	-10.5
-9	1	6	20.6	-19.7
-8	1	6	4.7	6.6
-7	1	6	35.6	33.4
-6	1	6	17.6	14.8
-5	1	6	58.4	-57.3
-4	1	6	18.0	-21.5
-3	1	6	44.2	44.2
-2	1	6	34.8	31.7
-1	1	6	18.5	-18.4
0	1	6	27.5	-26.7
1	1	6	5.6	1.8
2	1	6	31.1	28.9
3	1	6	12.0	8.1
4	1	6	7.3	-11.7
-11	1	7	12.4	8.4
-10	1	7	0.0	-0.3
-9	1	7	9.0	-8.1
-8	1	7	0.0	3.6
-7	1	7	23.6	22.4
-6	1	7	7.7	3.6
-5	1	7	20.2	-17.0
-4	1	7	18.0	-29.1
-3	1	7	0.0	-7.4
-2	1	7	26.2	21.8
-1	1	7	3.0	-0.9
0	1	7	23.6	-22.1
1	1	7	13.3	-12.8
2	1	7	7.7	7.6
3	1	7	5.6	3.7
4	1	7	0.0	-2.6
-11	1	8	22.3	-19.0
-10	1	8	5.6	6.1
-9	1	8	20.2	18.8
-8	1	8	0.0	3.7
-7	1	8	31.3	-30.2
-6	1	8	27.9	-26.2
-5	1	8	38.6	38.7
-4	1	8	36.0	35.6
-3	1	8	13.3	-12.3
-2	1	8	35.2	-33.9
-1	1	8	10.3	11.6
0	1	8	27.0	23.2
1	1	8	8.6	7.7
2	1	8	12.4	-12.0
3	1	8	9.9	-8.7
-11	1	9	6.0	-6.3
-10	1	9	0.0	0.0
-9	1	9	6.4	8.8
-8	1	9	0.0	-1.3
-7	1	9	19.7	-15.5
-6	1	9	8.2	-9.9
-5	1	9	18.5	6.0
-4	1	9	24.0	20.3
-3	1	9	0.0	4.0
-2	1	9	6.0	-14.7
-1	1	9	11.6	-9.5
0	1	9	11.6	8.4
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-8	1	10	21.9	-20.9
-7	1	10	16.7	19.4
-6	1	10	19.3	17.6
-5	1	10	15.0	-13.3
-4	1	10	36.5	-34.1
-3	1	10	9.0	-6.4
-2	1	10	27.9	24.7
-1	1	10	0.0	1.8
0	1	10	12.4	-11.4
1	1	10	9.9	-8.6
-11	1	11	0.0	2.8
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-6	1	11	6.4	3.4
-4	1	11	18.9	-15.6
-3	1	11	5.6	-4.8
-2	1	11	5.6	7.1
-1	1	11	4.3	6.4
0	2	0	40.8	-42.9
1	2	0	41.2	39.7
2	2	0	17.6	22.5
3	2	0	25.7	22.0
4	2	0	19.7	-20.3
5	2	0	13.7	-16.4
6	2	0	8.6	11.6
7	2	0	20.2	15.8
8	2	0	0.0	-0.2
-9	2	1	8.6	-8.2
-7	2	1	19.7	19.1
-6	2	1	11.2	10.4
-5	2	1	24.9	-20.5
-4	2	1	40.8	-38.7
-3	2	1	13.3	12.4
-2	2	1	8.2	0.3
-1	2	1	32.6	-46.9
0	2	1	37.3	-41.2
1	2	1	32.2	-35.6
2	2	1	14.6	17.2
3	2	1	15.0	15.0
4	2	1	13.3	-13.5
5	2	1	20.6	-18.3
6	2	1	0.0	8.9
7	2	1	18.5	17.0
8	2	1	0.0	3.0
-9	2	2	17.6	16.1
-8	2	2	11.6	11.5
-7	2	2	18.0	-17.3
-6	2	2	24.0	-24.2
-5	2	2	39.9	34.1
-4	2	2	38.6	38.3
-3	2	2	22.3	17.0
-2	2	2	4.7	-8.3
-1	2	2	25.3	-27.9
0	2	2	42.1	48.3
1	2	2	30.0	24.9
2	2	2	10.7	-11.0
3	2	2	36.9	-34.9
4	2	2	16.7	16.3
5	2	2	15.0	11.3
6	2	2	0.0	-1.3
7	2	2	6.0	-12.7
-10	2	3	0.0	-2.9
-9	2	3	15.9	15.3
-8	2	3	9.9	8.8
-7	2	3	20.2	-16.9
-6	2	3	28.7	-26.1
-5	2	3	11.2	10.8
-4	2	3	40.8	41.6
-3	2	3	20.2	-16.9
-2	2	3	28.3	-30.9
-1	2	3	15.4	-15.4
0	2	3	38.6	-40.6
1	2	3	11.2	14.4
2	2	3	25.3	-23.1
3	2	3	25.7	-26.1
4	2	3	6.9	1.2
5	2	3	18.9	16.0
6	2	3	5.6	-2.0
-10	2	4	6.0	8.6
-9	2	4	11.6	-12.0
-8	2	4	17.2	-16.4
-7	2	4	20.6	20.8
-6	2	4	54.5	49.8
-5	2	4	12.0	-8.8
-4	2	4	38.2	-37.0
-3	2	4	0.0	-6.6
-2	2	4	37.8	38.1
-1	2	4	33.0	32.8
0	2	4	19.3	-18.0
1	2	4	45.1	-44.2
2	2	4	0.0	0.0
3	2	4	30.5	28.9
4	2	4	8.2	-6.1
5	2	4	15.4	-14.6
6	2	4	13.7	-6.7
-10	2	5	15.9	13.1
-9	2	5	11.6	-8.6
-8	2	5	13.3	-13.1
-7	2	5	15.0	12.0
-6	2	5	44.2	45.4
-5	2	5	8.6	7.8
-4	2	5	19.3	-22.9
-3	2	5	20.6	-23.6
-2	2	5	21.6	23.3
-1	2	5	25.3	30.5
0	2	5	26.2	-30.1
1	2	5	28.7	-30.2
2	2	5	6.9	-2.0
3	2	5	21.5	18.8
4	2	5	0.0	-0.0
5	2	5	14.6	-10.9
-11	2	6	0.0	-5.5
-10	2	6	9.0	-10.5
-9	2	6	15.0	12.4
-8	2	6	23.6	21.6
-7	2	6	6.0	2.8
-6	2	6	36.9	-37.3
-5	2	6	9.4	-9.0
-4	2	6	33.9	36.2
-3	2	6	15.4	18.4
-2	2	6	27.5	-30.1
-1	2	6	32.6	-34.2
0	2	6	4.3	1.0
1	2	6	26.6	25.0
2	2	6	0.0	1.8
3	2	6	23.2	-21.2
4	2	6	11.2	-7.4
-11	2	7	10.3	-8.5
-10	2	7	19.7	-17.3
-9	2	7	3.9	-3.7
-8	2	7	17.2	18.6
-7	2	7	0.0	1.6
-6	2	7	27.0	-28.6
-5	2	7	11.6	-14.0
-4	2	7	18.9	12.2
-3	2	7	38.2	41.0
-2	2	7	4.3	1.2
-1	2	7	16.7	-16.2
0	2	7	6.9	5.4
1	2	7	26.6	25.6
2	2	7	9.4	9.2
3	2	7	13.7	-9.6
-11	2	8	4.3	5.0
-10	2	8	17.2	16.0
-9	2	8	0.0	-3.8
-8	2	8	13.3	-12.8
-7	2	8	18.9	-15.8
-6	2	8	14.2	16.4
-5	2	8	10.7	10.3
-4	2	8	15.4	-15.9
-3	2	8	28.3	-32.0
-2	2	8	7.3	3.4
-1	2	8	19.3	17.4
0	2	8	0.0	-3.6
1	2	8	15.9	-16.0
2	2	8	0.0	-7.8
-11	2	9	0.0	0.4
-10	2	9	11.2	12.1
-9	2	9	6.4	6.0
-8	2	9	18.5	-17.3
-7	2	9	10.7	-11.5
-6	2	9	6.9	7.4
-5	2	9	13.3	15.9
-4	2	9	9.0	-5.6
-3	2	9	21.5	-21.7
-2	2	9	9.4	-5.0
-1	2	9	11.2	10.7
0	2	9	3.9	7.1
1	2	9	11.6	-10.2
-11	2	10	0.0	-0.3
-10	2	10	15.9	-14.8
-9	2	10	7.7	-8.2
-8	2	10	6.4	6.1
-7	2	10	12.4	10.8
-6	2	10	7.7	-10.5
-5	2	10	18.5	-14.9
-4	2	10	12.4	-9.3
-3	2	10	16.7	17.5
-2	2	10	6.4	-0.3
-1	2	10	10.7	-11.9
0	2	10	6.0	-4.5
-11	2	11	0.0	1.8
-10	2	11	12.4	-11.5
-9	2	11	7.7	-8.4
-8	2	11	0.0	3.6
-7	2	11	8.2	8.6
-6	2	11	9.9	-5.7
-5	2	11	15.0	-16.6
-4	2	11	0.0	-0.9
-3	2	11	8.5	9.3
-2	2	11	0.0	4.9
-1	2	11	4.3	-6.7
1	3	0	27.0	28.8
2	3	0	5.1	4.4
3	3	0	6.9	-8.1
4	3	0	27.9	-24.0
5	3	0	0.0	3.1
6	3	0	10.7	9.2
7	3	0	5.6	3.7
8	3	0	7.3	-7.0
-8	3	1	19.3	18.2
-7	3	1	12.4	11.0
-6	3	1	17.6	-16.3
-5	3	1	29.6	-27.3
-4	3	1	17.2	14.2
-3	3	1	63.1	57.7
-2	3	1	5.6	-10.1
-1	3	1	27.9	-39.8
0	3	1	12.4	-13.5
1	3	1	42.5	44.9
2	3	1	28.3	25.3
3	3	1	25.3	-24.9
4	3	1	24.0	-21.3
5	3	1	10.3	8.7
6	3	1	24.0	19.5
7	3	1	0.0	-3.2
-9	3	2	0.0	2.2
-8	3	2	14.2	-11.5
-7	3	2	6.4	-3.0
-6	3	2	18.5	17.2
-5	3	2	10.3	10.1
-4	3	2	49.3	-40.6
-3	3	2	13.7	-19.7
-2	3	2	9.9	-14.5
-1	3	2	18.5	24.0
0	3	2	8.6	4.9
1	3	2	24.0	-25.9
2	3	2	27.9	-25.7
3	3	2	8.2	10.1
4	3	2	17.2	17.2
5	3	2	6.4	-1.9
6	3	2	3.9	-4.5
7	3	2	7.3	-2.9
-9	3	3	9.0	8.8
-8	3	3	17.2	-15.3
-7	3	3	12.4	-10.8
-6	3	3	21.5	20.0
-5	3	3	78.5	68.0
-4	3	3	15.4	16.9
-3	3	3	34.3	-38.9
-2	3	3	7.3	-6.5
-1	3	3	57.5	64.8
0	3	3	36.5	39.9
1	3	3	36.0	-35.7
2	3	3	31.3	-30.2
3	3	3	4.7	-3.2
4	3	3	22.3	17.7
5	3	3	0.0	-3.2
6	3	3	26.6	-17.3
-10	3	4	0.0	-5.7
-9	3	4	0.0	-3.8
-8	3	4	15.9	14.6
-7	3	4	19.3	18.1
-6	3	4	15.0	-12.3
-5	3	4	27.9	-28.2
-4	3	4	25.3	25.8
-3	3	4	24.9	30.2
-2	3	4	19.4	17.3
-1	3	4	21.9	-29.7
0	3	4	29.2	-28.8
1	3	4	5.1	-0.7
2	3	4	22.3	24.1
3	3	4	0.0	-7.3
4	3	4	20.6	-16.7
5	3	4	0.0	-3.3
-10	3	5	12.0	-12.1
-9	3	5	12.4	-11.1
-8	3	5	14.2	13.4
-7	3	5	31.8	30.6
-6	3	5	0.0	-2.4
-5	3	5	48.5	-49.9
-4	3	5	11.6	-13.0
-3	3	5	37.3	38.6
-2	3	5	40.9	45.2
-1	3	5	39.9	-42.1
0	3	5	33.5	-33.6
1	3	5	16.3	12.3
2	3	5	31.3	27.6
3	3	5	7.7	5.2
4	3	5	20.2	-16.1
5	3	5	11.6	-6.3
-10	3	6	6.0	6.8
-9	3	6	8.6	7.8
-8	3	6	8.2	-5.9
-7	3	6	18.9	-19.4
-6	3	6	8.2	-3.1
-5	3	6	24.9	29.7
-4	3	6	14.2	6.6
-3	3	6	21.0	-21.5
-2	3	6	26.6	-26.8
-1	3	6	11.2	11.6
0	3	6	24.0	22.5
1	3	6	11.6	9.2
2	3	6	17.2	-15.7
3	3	6	0.0	-3.4
4	3	6	0.0	7.9
-11	3	7	7.7	-9.5
-10	3	7	0.0	5.8
-9	3	7	17.6	16.9
-8	3	7	0.0	0.0
-7	3	7	33.9	-34.0
-6	3	7	4.7	-6.5
-5	3	7	25.7	26.0
-4	3	7	19.7	20.4
-3	3	7	23.6	-26.2
-2	3	7	38.8	-38.0
-1	3	7	11.6	10.1
0	3	7	22.7	21.1
1	3	7	0.0	1.1
2	3	7	24.5	-21.6
3	3	7	0.0	-6.8
-11	3	8	0.0	4.0
-10	3	8	9.0	-7.7
-9	3	8	7.7	-9.0
-8	3	8	8.2	-2.5
-7	3	8	9.0	10.9
-6	3	8	9.4	9.3
-5	3	8	4.7	-10.1
-4	3	8	18.5	-15.4
-3	3	8	4.7	5.8
-2	3	8	24.5	24.7
-1	3	8	0.0	2.8
0	3	8	9.4	-8.8
1	3	8	9.0	-6.8
2	3	8	6.0	6.7
-11	3	9	12.0	12.6
-10	3	9	0.0	4.9
-9	3	9	18.5	-18.3
-8	3	9	14.2	-13.1
-7	3	9	9.4	15.5
-6	3	9	8.6	9.0
-5	3	9	19.7	-21.3
-4	3	9	30.9	-32.2
-3	3	9	3.4	0.4
-2	3	9	7.3	11.2
-1	3	9	3.9	-0.7
0	3	9	17.2	-16.8
1	3	9	9.4	-8.0
-11	3	10	7.3	-6.8
-10	3	10	0.0	3.3
-9	3	10	3.9	5.5
-8	3	10	3.9	1.5
-7	3	10	10.3	-11.3
-6	3	10	11.6	-8.9
-5	3	10	9.0	-2.9
-4	3	10	12.9	10.1
-3	3	10	0.0	2.0
-2	3	10	12.6	-12.6
-1	3	10	0.0	-3.6
0	3	10	0.0	4.0
-11	3	11	13.3	-12.7
-10	3	11	8.2	-9.2
-9	3	11	11.6	11.4
-8	3	11	16.3	14.2
-7	3	11	7.7	-6.3
-6	3	11	19.7	-19.5
-5	3	11	6.9	4.2
-4	3	11	11.2	17.1
-3	3	11	0.0	3.2
-2	3	11	6.0	-9.1
-1	3	11	4.3	-6.7
0	4	0	12.9	12.1
1	4	0	9.9	-12.1
2	4	0	9.4	-8.9
3	4	0	9.4	-7.1
4	4	0	0.0	1.1
5	4	0	6.0	2.8
6	4	0	6.4	5.5
7	4	0	0.0	-0.0
8	4	0	0.0	-0.3
-8	4	1	0.0	2.6
-7	4	1	17.6	-18.6
-6	4	1	14.6	-12.1
-5	4	1	24.9	22.7
-4	4	1	52.4	49.4
-3	4	1	22.3	-22.2
-2	4	1	56.6	-66.0
-1	4	1	0.0	2.0
0	4	1	52.4	67.5
1	4	1	25.3	31.9
2	4	1	44.2	-46.7
3	4	1	37.3	-36.2
4	4	1	12.9	13.0
5	4	1	30.9	26.2
6	4	1	6.9	-1.6
7	4	1	21.9	-16.5
-9	4	2	0.0	-1.2
-8	4	2	0.0	3.5
-7	4	2	7.7	4.7
-6	4	2	6.9	2.2
-5	4	2	6.4	-0.0
-4	4	2	11.2	11.6
-3	4	2	25.7	25.7
-2	4	2	6.4	12.0
-1	4	2	7.7	14.4
0	4	2	6.9	-7.8
1	4	2	8.2	10.2
2	4	2	10.3	9.4
3	4	2	4.7	1.6
4	4	2	12.4	-10.2
5	4	2	8.6	-5.1
6	4	2	4.3	-4.6
-9	4	3	14.2	-14.3
-8	4	3	5.6	-6.1
-7	4	3	13.7	15.1
-6	4	3	28.7	27.7
-5	4	3	9.0	-7.7
-4	4	3	55.4	-58.8
-3	4	3	0.0	-5.3
-2	4	3	42.1	58.0
-1	4	3	26.2	33.7
0	4	3	46.8	-53.7
1	4	3	31.8	-34.9
2	4	3	6.4	12.3
3	4	3	29.2	28.6
4	4	3	8.6	4.3
5	4	3	12.0	-19.0
6	4	3	10.7	-6.2
-9	4	4	0.0	5.4
-8	4	4	0.0	5.4
-7	4	4	10.7	9.1
-6	4	4	3.4	1.4
-5	4	4	15.0	15.5
-4	4	4	12.4	10.2
-3	4	4	0.0	-1.8
-2	4	4	3.9	2.0
-1	4	4	4.3	-0.6
0	4	4	10.7	6.3
1	4	4	4.7	-4.5
2	4	4	6.9	-5.1
3	4	4	0.0	-8.9
4	4	4	0.0	3.0
5	4	4	8.6	-0.0
-10	4	5	6.0	-6.8
-9	4	5	13.3	13.6
-8	4	5	12.0	13.1
-7	4	5	8.2	-9.5
-6	4	5	38.2	-39.3
-5	4	5	8.2	-1.4
-4	4	5	36.0	39.2
-3	4	5	21.5	25.4
-2	4	5	21.9	-42.8
-1	4	5	39.0	-42.0
0	4	5	23.2	24.2
1	4	5	28.7	26.9
2	4	5	6.4	3.2
3	4	5	21.5	-19.6
4	4	5	10.3	-6.7
-10	4	6	0.0	1.2
-9	4	6	0.0	-2.3
-8	4	6	5.1	-4.6
-7	4	6	15.0	-12.1
-6	4	6	0.0	5.5
-5	4	6	3.4	-0.5
-4	4	6	4.3	-2.6
-3	4	6	12.0	-7.1
-2	4	6	11.2	4.5
-1	4	6	5.6	3.1
0	4	6	0.0	4.0
1	4	6	7.3	2.1
2	4	6	0.0	-2.1
3	4	6	8.2	3.4
-10	4	7	13.3	11.0
-9	4	7	3.9	-6.7
-8	4	7	25.7	-25.0
-7	4	7	0.0	-0.9
-6	4	7	22.7	35.8
-5	4	7	6.9	8.6
-4	4	7	27.5	-29.5
-3	4	7	33.9	-35.4
-2	4	7	36.0	19.7
-1	4	7	27.5	28.2
0	4	7	0.0	-1.8
1	4	7	22.3	-21.7
2	4	7	9.4	-7.4
3	4	7	15.0	11.1
-10	4	8	0.0	-0.1
-9	4	8	0.0	1.4
-8	4	8	0.0	1.4
-7	4	8	7.3	5.0
-6	4	8	6.0	-7.1
-5	4	8	12.4	-11.1
-4	4	8	13.7	-10.4
-3	4	8	5.1	1.1
-2	4	8	9.0	-9.5
-1	4	8	9.0	-7.4
0	4	8	12.0	-8.3
1	4	8	4.3	-3.3
2	4	8	0.0	0.5
-10	4	9	15.9	-15.1
-9	4	9	5.6	-4.2
-8	4	9	24.9	24.6
-7	4	9	14.6	11.5
-6	4	9	24.5	-24.9
-5	4	9	23.2	-23.5
-4	4	9	15.0	16.3
-3	4	9	26.2	27.7
-2	4	9	7.7	-1.7
-1	4	9	16.7	-17.4
0	4	9	9.4	-8.5
-10	4	10	0.0	0.7
-9	4	10	0.0	-3.5
-8	4	10	0.0	-4.8
-7	4	10	3.9	-4.3
-6	4	10	0.0	-2.1
-5	4	10	7.7	-1.4
-4	4	10	5.6	-0.5
-3	4	10	5.6	-6.5
-2	4	10	0.0	-0.7
-1	4	10	0.0	0.3
1	5	0	9.4	8.3
2	5	0	12.0	11.1
3	5	0	6.4	-6.4
4	5	0	12.9	-12.6
5	5	0	0.0	0.4
6	5	0	0.0	5.1
7	5	0	6.0	-2.3
-8	5	1	11.6	-12.1
-7	5	1	0.0	-4.3
-6	5	1	19.3	17.6
-5	5	1	25.3	24.4
-4	5	1	17.6	-17.9
-3	5	1	39.5	-40.2

Table 3 (*cont.*)

H	K	L	F OBS	F CALC
-2	5	1	10.7	16.4
-1	5	1	33.0	49.1
0	5	1	13.0	13.2
1	5	1	33.5	-39.0
2	5	1	18.5	-23.7
3	5	1	26.6	25.6
4	5	1	32.2	28.0
5	5	1	8.6	-7.6
6	5	1	25.7	-19.8
-8	5	2	0.0	-1.8
-7	5	2	0.0	-2.0
-6	5	2	15.0	13.9
-5	5	2	6.9	8.8
-4	5	2	5.6	0.1
-3	5	2	5.6	-8.9
-2	5	2	10.3	11.8
-1	5	2	16.7	18.0
0	5	2	7.7	7.4
1	5	2	0.0	-2.5
2	5	2	13.7	-15.1
3	5	2	7.7	-1.3
4	5	2	9.4	6.1
5	5	2	0.0	-0.7
6	5	2	7.3	-6.0
-8	5	3	13.7	14.2
-7	5	3	12.9	15.5
-6	5	3	0.0	2.1
-5	5	3	21.5	-22.1
-4	5	3	6.4	11.8
-3	5	3	31.8	43.1
-2	5	3	13.7	18.8
-1	5	3	24.9	-31.8
0	5	3	12.9	-18.0
1	5	3	21.9	23.2
2	5	3	8.6	18.5
3	5	3	6.4	-8.3
4	5	3	26.2	-24.2
5	5	3	0.0	-5.0
-9	5	4	0.0	-2.3
-8	5	4	0.0	4.1
-7	5	4	0.0	1.8
-6	5	4	17.2	-15.4
-5	5	4	10.0	-11.0
-4	5	4	12.0	-11.9
-3	5	4	11.6	14.0
-2	5	4	5.1	-2.2
-1	5	4	16.3	-9.6
0	5	4	13.7	-14.4
1	5	4	0.0	5.3
2	5	4	13.3	13.6
3	5	4	3.9	4.2
4	5	4	0.0	-0.8
-9	5	5	10.3	11.6
-8	5	5	0.0	-4.7
-7	5	5	16.3	-17.3
-6	5	5	0.0	-0.7
-5	5	5	23.2	24.8
-4	5	5	8.2	8.1
-3	5	5	13.7	-18.9
-2	5	5	21.9	-26.2
-1	5	5	21.0	20.3
0	5	5	15.9	18.2
1	5	5	7.3	-7.3
2	5	5	17.2	-16.6
3	5	5	0.0	-1.9
4	5	5	13.7	10.9
-9	5	6	0.0	4.3
-8	5	6	9.9	-8.3
-7	5	6	7.7	-7.0
-6	5	6	0.0	2.7
-5	5	6	6.0	6.5
-4	5	6	0.0	4.4
-3	5	6	13.7	-13.3
-2	5	6	4.7	-9.9
-1	5	6	0.0	-4.9
0	5	6	11.2	13.8
1	5	6	0.0	-4.3
2	5	6	4.3	-7.1
3	5	6	0.0	-3.9
-9	5	7	15.4	-15.8
-8	5	7	0.0	-0.7
-7	5	7	26.2	24.3
-6	5	7	0.0	4.4
-5	5	7	20.2	-21.1
-4	5	7	16.3	-19.0
-3	5	7	8.2	9.3
-2	5	7	14.2	21.4
-1	5	7	8.6	-8.8
0	5	7	18.9	-18.9
1	5	7	8.2	-5.2
2	5	7	7.3	11.3
-10	5	8	0.0	0.4
-9	5	8	0.0	-4.5
-8	5	8	0.0	3.8
-7	5	8	7.7	4.8
-6	5	8	0.0	0.4
-5	5	8	10.3	-10.2
-4	5	8	7.3	-7.4
-3	5	8	3.9	-1.1
-2	5	8	7.7	5.8
-1	5	8	0.0	2.8
0	5	8	10.3	-9.4
1	5	8	0.0	-1.1
-9	5	9	18.0	16.6
-8	5	9	5.6	6.2
-7	5	9	24.9	-23.3
-6	5	9	18.5	-18.5
-5	5	9	9.4	9.0
-4	5	9	11.6	16.3
-3	5	9	6.9	-9.6
-2	5	9	20.6	-20.7
-1	5	9	7.3	-6.3
0	5	9	12.4	10.8
0	6	0	21.5	22.3
1	6	0	13.3	10.3
2	6	0	7.7	-9.5
3	6	0	7.3	-6.3
4	6	0	12.0	9.9
5	6	0	13.3	12.4
6	6	0	6.0	-4.4
-7	6	1	12.0	10.6
-6	6	1	9.9	11.0
-5	6	1	12.9	-9.0
-4	6	1	17.2	-16.6
-3	6	1	15.4	17.2
-2	6	1	20.6	28.2
-1	6	1	6.9	5.0
0	6	1	11.6	-16.5
1	6	1	8.6	-5.9
2	6	1	15.9	16.8
3	6	1	20.2	17.0
4	6	1	12.0	-12.0
5	6	1	21.9	-17.5
6	6	1	6.0	0.5
-7	6	2	0.0	8.7
-6	6	2	8.2	9.1
-5	6	2	12.4	-8.0
-4	6	2	6.4	-7.3
-3	6	2	6.0	6.6
-2	6	2	14.6	22.6
-1	6	2	6.0	-3.3
0	6	2	7.7	-12.7
1	6	2	6.0	-5.4
2	6	2	15.0	12.8
3	6	2	12.4	10.9
4	6	2	9.9	-6.1
5	6	2	0.0	-9.1
-8	6	3	0.0	6.1
-7	6	3	0.0	-5.3
-6	6	3	10.3	-13.4
-5	6	3	0.0	3.6
-4	6	3	15.7	20.4
-3	6	3	0.0	0.0
-2	6	3	12.9	-22.3
-1	6	3	9.9	-13.0
0	6	3	13.3	16.3
1	6	3	12.0	10.3
2	6	3	7.7	-7.6
3	6	3	16.7	-15.0
4	6	3	0.0	2.0
-8	6	4	9.4	10.6
-7	6	4	5.6	-5.8
-6	6	4	0.0	-6.3
-5	6	4	0.0	1.3
-4	6	4	9.4	15.6
-3	6	4	0.0	4.6
-2	6	4	13.7	-17.1
-1	6	4	6.0	-3.8
0	6	4	5.1	7.6
1	6	4	9.9	11.4
2	6	4	13.7	-10.2
3	6	4	10.3	-10.0
4	6	4	8.6	-0.6
-8	6	5	13.3	-13.1
-7	6	5	0.0	-2.0
-6	6	5	10.7	11.1
-5	6	5	0.0	-3.9
-4	6	5	14.6	-16.7
-3	6	5	11.6	-14.5
-2	6	5	9.0	11.3
-1	6	5	14.6	16.4
0	6	5	8.6	-8.5
1	6	5	11.2	-11.0
2	6	5	8.2	2.8
3	6	5	13.7	12.1
-8	6	6	11.2	-10.3
-7	6	6	0.0	3.5
-6	6	6	9.4	10.2
-5	6	6	9.4	11.2
-4	6	6	10.3	-13.3
-3	6	6	9.0	-10.5
-2	6	6	0.0	0.4
-1	6	6	10.3	9.5
0	6	6	0.0	-4.4
1	6	6	14.2	-14.1
2	6	6	0.0	-0.8
-9	6	7	0.0	3.9
-8	6	7	13.3	14.4
-7	6	7	0.0	2.5
-6	6	7	14.2	-15.1
-5	6	7	6.9	-7.1
-4	6	7	0.0	2.9
-3	6	7	13.3	10.7
-2	6	7	0.0	-9.3
-1	6	7	16.7	-17.8
0	6	7	4.3	-2.8
1	6	7	8.6	7.0
-9	6	8	0.0	-0.7
-8	6	8	10.3	8.8
-7	6	8	4.3	4.8
-6	6	8	5.6	-8.4
-5	6	8	9.0	-12.5
-4	6	8	0.0	3.6
-3	6	8	3.9	7.2
-2	6	8	0.0	3.5
-1	6	8	13.3	-11.8
0	6	8	0.0	-2.7
1	7	0	19.7	-20.5
2	7	0	7.7	-6.7
3	7	0	14.2	12.8
4	7	0	9.9	9.6
5	7	0	0.0	-7.3
-6	7	1	6.0	-4.8
-5	7	1	0.0	-5.1
-4	7	1	13.3	12.8
-3	7	1	12.9	14.9
-2	7	1	3.9	1.0
-1	7	1	0.0	-5.6
0	7	1	5.1	2.5
1	7	1	12.4	11.3
2	7	1	13.3	10.6
3	7	1	0.0	-1.5
4	7	1	0.0	-9.0
5	7	1	0.0	2.3
-6	7	2	4.3	-8.6
-5	7	2	9.0	-8.8
-4	7	2	0.0	6.7
-3	7	2	18.0	21.1
-2	7	2	0.0	0.0
-1	7	2	11.2	-21.2
0	7	2	0.0	-3.6
1	7	2	14.2	14.0
2	7	2	10.3	9.7
3	7	2	13.7	-10.9
4	7	2	14.2	-11.7
-7	7	3	0.0	-4.2
-6	7	3	0.0	4.4
-5	7	3	6.9	10.4
-4	7	3	0.0	-6.1
-3	7	3	11.6	-17.3
-2	7	3	0.0	-7.1
-1	7	3	7.7	14.6
0	7	3	8.6	7.4
1	7	3	0.0	-4.3
2	7	3	12.0	-9.7
3	7	3	0.0	0.8
-7	7	4	11.6	-12.5
-6	7	4	0.0	7.6
-5	7	4	6.9	11.7
-4	7	4	0.0	2.4
-3	7	4	21.5	-24.7
-2	7	4	10.3	-7.5
-1	7	4	9.4	12.8
0	7	4	6.9	11.2
1	7	4	10.7	-9.1
2	7	4	13.7	-11.8
3	7	4	9.9	3.8
-7	7	5	7.3	7.5
-6	7	5	0.0	-1.3
-5	7	5	6.9	-10.0
-4	7	5	0.0	-0.7
-3	7	5	5.6	12.5
-2	7	5	6.9	11.4
-1	7	5	12.0	-12.3
0	7	5	15.0	-15.1
1	7	5	4.3	-1.5
2	7	5	6.0	6.1
-7	7	6	12.0	11.1
-6	7	6	0.0	3.1
-5	7	6	12.0	-15.1
-4	7	6	8.2	-12.1
-3	7	6	6.9	12.7
-2	7	6	9.9	9.2
-1	7	6	7.3	-5.0
0	7	6	15.9	-15.3
1	7	6	0.0	2.0
-7	7	7	6.0	-7.8
-6	7	7	0.0	0.4
-5	7	7	4.3	8.1
-4	7	7	0.0	3.3
-3	7	7	0.0	-5.9
-2	7	7	8.2	-13.3
-1	7	7	0.0	-0.0
0	8	0	18.5	-15.3
1	8	0	8.2	1.6
2	8	0	21.0	21.4
3	8	0	12.0	11.6
4	8	0	9.9	-7.1
-4	8	1	0.0	2.0
-3	8	1	0.0	-3.1
-2	8	1	0.0	-2.5
-1	8	1	0.0	-1.3
0	8	1	3.9	1.4
1	8	1	9.0	-3.3
2	8	1	4.3	-0.6
3	8	1	0.0	-4.2
-5	8	2	0.0	8.2
-4	8	2	14.6	15.1
-3	8	2	0.0	-0.1
-2	8	2	16.3	-22.0
-1	8	2	0.0	-4.7
0	8	2	13.3	16.7
1	8	2	13.7	11.8
2	8	2	12.0	-9.3
3	8	2	13.7	-11.6
-5	8	3	0.0	-1.7
-4	8	3	0.0	-4.3
-3	8	3	0.0	-1.6
-2	8	3	0.0	4.8
-1	8	3	0.0	-1.5
0	8	3	0.0	-6.0
1	8	3	0.0	-1.6
2	8	3	0.0	-1.5
-5	8	4	0.0	-1.7
-4	8	4	14.6	-18.5
-3	8	4	0.0	-5.9
-2	8	4	17.6	18.8
-1	8	4	8.2	10.7
	8	4	9.4	-9.1
1	8	4	16.3	-15.3
-5	8	5	0.0	-0.4
-4	8	5	0.0	3.0
-3	8	5	0.0	2.5
-2	8	5	0.0	-5.9
-1	8	5	0.0	-5.0
0	8	5	0.0	1.6
1	9	0	12.7	12.8
-1	9	1	0.0	0.2
0	9	1	0.0	-1.6
-1	9	2	6.0	9.9

Table 4. *Final positional parameters (fractional) with standard deviations* (Å), *temperature factors and standard deviations* (Å²), *and deviations from plane* 1 (Δ_1) *and from plane* 2 (Δ_2).

							2nd L.S.		2nd D.S.		
Atom	x	y	z	$\sigma(x)$	$\sigma(y)$	$\sigma(z)$	B	$\sigma(B)$	B	Δ_1 (Å)	Δ_2 (Å)
C(1)	0·2991	0·4406	0·2698	0·033	0·025	0·038	7·67	0·93	7·0	+0·030	—
(2)	0·2287	0·4068	0·1265	0·019	0·028	0·032	6·34	0·76	6·0	−0·019	—
(3)	0·3349	0·2800	0·1445	0·016	0·036	0·025	6·17	0·76	6·0	−0·002	—
(4)	0·4590	0·2349	0·2870	0·013	0·031	0·023	5·80	0·75	5·5	+0·021	—
(5)	0·4351	0·3234	0·3648	0·017	0·022	0·024	5·79	0·61	5·5	−0·032	—
(6)	0·1770	−0·0591	0·2007	0·020	0·024	0·025	4·25	0·56	4·4	—	−0·002
(7)	0·0377	0·0146	0·0735	0·013	0·017	0·018	4·28	0·53	4·0	—	+0·011
(8)	−0·0394	0·1347	0·1050	0·011	0·023	0·018	4·93	0·67	4·7	—	−0·007
(9)	0·0616	0·1305	0·2527	0·015	0·031	0·021	5·08	0·73	4·7	—	+0·005
(10)	0·1913	0·0088	0·3058	0·020	0·034	0·026	7·08	0·81	6·0	—	−0·003
Fe(11)	0·2177	0·1913	0·2146	0·0019	0·0030	0·0026	3·06	0·05	3·3	−1·663	+1·635

electron-density distribution taken through the atomic centres are shown in Fig. 2.

Coordinates and molecular dimensions

The positional and thermal parameters are given in Table 4, x, y, and z being coordinates from the third differential syntheses referred to the monoclinic crystal axes and expressed as fractions of the unit cell edges. Also included in Table 4 are the standard deviations of the parameters; those of the atomic positional coordinates were calculated from Cruickshank's (1949) formulae, and the thermal parameter standard deviations were calculated from the least-squares residuals of the second least-squares cycle.

The bond distances and valency angles in the molecule are given in Table 5. The mean standard deviations are 0·02 Å for Fe–C bonds, 0·03 Å for C–C bonds (0·04 Å for C(7)–C(7′)), and 2° for C–C–C angles.

The best plane through the ten carbon atoms of the two five-membered rings which are bonded together has equation

$$0{\cdot}687X'+0{\cdot}716Y+0{\cdot}122Z'=0 \quad \text{(Plane 1)},$$

where X', Y, Z' are coordinates in Å referred to orthogonal axes a, b and c^*. The plane through the other cyclopentadienyl ring has equation

Table 5. *Bond distances and valency angles*

	Fe–C(1)	2·07 Å		C(1)–C(2)–C(3)	104°
	Fe–C(2)	2·07		C(2)–C(3)–C(4)	110
	Fe–C(3)	2·04		C(3)–C(4)–C(5)	111
	Fe–C(4)	2·04		C(4)–C(5)–C(1)	106
	Fe–C(5)	2·02		C(5)–C(1)–C(2)	109
	Fe–C(6)	2·00		C(6)–C(7)–C(8)	107
	Fe–C(7)	2·04		C(7)–C(8)–C(9)	107
	Fe–C(8)	2·07		C(8)–C(9)–C(10)	106
	Fe–C(9)	2·03		C(9)–C(10)–C(6)	111
	Fe–C(10)	1·97		C(10)–C(6)–C(7)	109
Mean	Fe–C	$2{\cdot}03_5$	Mean	C–C–C	108
	C(1)–C(2)	1·46 Å		C(6)–C(7)–C(7′)	132°
	C(2)–C(3)	1·39		C(8)–C(7)–C(7′)	121
	C(3)–C(4)	1·39			
	C(4)–C(5)	1·36		C(1)–Fe–C(6)	169
	C(5)–C(1)	1·43		C(2)–Fe–C(10)	171
	C(6)–C(7)	1·38		C(3)–Fe–C(9)	169
	C(7)–C(8)	1·45		C(4)–Fe–C(8)	169
	C(8)–C(9)	1·41		C(5)–Fe–C(7)	167
	C(9)–C(10)	1·40			
	C(10–C(6)	1·35			
Mean	C–C	1·40			
	C(7)–C(7′)	1·48			

$$0{\cdot}712X' + 0{\cdot}685Y + 0{\cdot}152Z' = 3{\cdot}324 \quad \text{(Plane 2)}.$$

These planes are nearly parallel (the angle between their normals is 2·8°), and the mean distance between them is 3·32 Å. The deviations of the atoms from the planes are included in Table 4.

Table 6. *Shorter intermolecular distances*

All crystallographically independent distances ≤ 4 Å between molecule 1 at *x*, *y*, *z* and neighbouring molecules are listed

Atom (molecule 1)	to Atom	in Molecule	*d*
1	4	9	3·77 Å
1	6	2	4·00
1	9	5	3·85
2	2	3	3·80
2	4	9	3·67
3	4	9	3·92
4	6	9	4·00
8	10	5	3·84
9	2	6	3·80
9	3	6	3·77
9	6	5	3·77
9	10	5	3·67
10	2	6	3·85
10	3	6	3·82

Molecule				
1	at	x,	y,	z
2	at	x,	$1+y$,	z
3	at	$-x$,	$1-y$,	$-z$
5	at	$-x$,	$\frac{1}{2}+y$,	$\frac{1}{2}-z$
6	at	x,	$\frac{1}{2}-y$,	$\frac{1}{2}+z$
9	at	$1-x$,	$\frac{1}{2}+y$,	$\frac{1}{2}-z$

All the shorter intermolecular contacts are listed in Table 6, and the packing of the molecules is shown in Fig. 3.

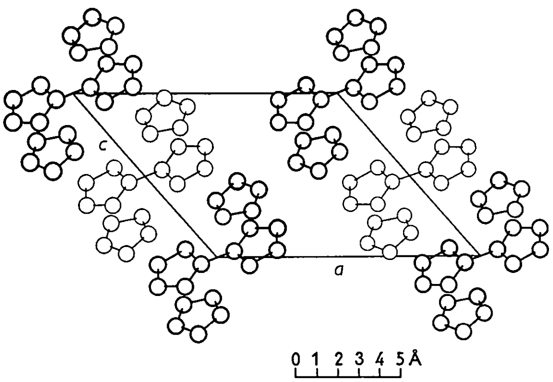

Fig. 3. Projection of the structure along [010].

Discussion

The analysis has shown that the molecule has a *trans* configuration (Figs. 1 and 2), the atoms of the bonded five-membered rings all lie in one plane, and each iron atom is sandwiched between two rings which are planar within experimental error, approximately parallel (angle between the planes is 2·8°) and separated by 3·32 Å.

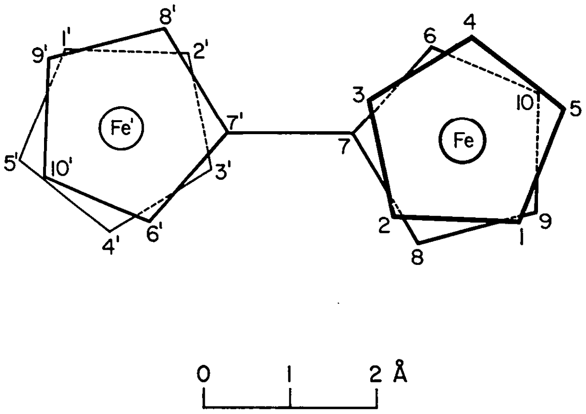

Fig. 4. View of the molecule along the normal to the planes of the rings. Heavier lines are nearer the viewer.

The primary object of the investigation was the determination of the configuration of the cyclopentadienyl rings; this is illustrated in Figs. 1 and 2, and more clearly in Fig. 4, which shows a view of the molecule along the normal to the planes of the rings. The configuration is neither fully eclipsed, as in ruthenocene (Hardgrove & Templeton, 1959), nor fully staggered, as in ferrocene (Dunitz, Orgel & Rich, 1956), but somewhere between these extremes. It is best described in terms of the rotation of one ring from the fully eclipsed position as given by the angles between the projections of the Fe–C bonds on the mean plane of a cyclopentadienyl ring:

C(1)–Fe–C(9) = 10·1°
C(2)–Fe–C(8) = 18·5
C(3)–Fe–C(7) = 21·1
C(4)–Fe–C(6) = 22·1
C(5)–Fe–C(10) = 14·3

The variation in these values is related to the deviations from pentagonal symmetry, described below, but the mean value of 17° indicates that the configuration is about midway between the eclipsed (0°) and fully staggered (36°) positions.

The different configurations in ferrocene and ruthenocene can hardly be related to any differences in bonding orbitals of the metal atoms. It is possible that intramolecular steric effects in ferrocene, where the rings are separated by only 3·32 Å, as in biferrocenyl, would make the eclipsed configuration less stable than the staggered position, while in ruthenocene these effects would be smaller (separation of rings 3·68 Å) and the eclipsed configuration would be possible. This does not indicate why the eclipsed configuration is preferred in ruthenocene, and it seems likely that intermolecular interactions are also involved (Wilkinson & Cotton, 1959). The importance of intermolecular forces is supported by the fact that the configuration in biferrocenyl, where the distances within the molecule are similar to those in ferrocene, has now been shown to be intermediate between ferrocene and ruthenocene.

The mean bond distances in biferrocenyl are Fe–C = $2{\cdot}03_5 \pm 0{\cdot}01$ Å, C–C (cyclopentadienyl rings) = $1{\cdot}40 \pm 0{\cdot}01$ Å, C–C (between rings) = $1{\cdot}48 \pm 0{\cdot}04$ Å. However, detailed examination of the individual distances (Table 5) indicates that there are deviations from pentagonal symmetry in each ring. The shortest bonds in each ring (1·36 and 1·35 Å) differ from the longest bonds (1·46 and 1·45 Å) by 2·3 standard deviations, and hence the differences are significant. The other three bonds in each ring (1·38–1·42 Å) are about the usual aromatic length. The fact that the variations are almost exactly the same in both crystallographically independent rings is a further indication that the differences are real, and that the iron atom is therefore not bonded equally to all five carbon atoms in each ring. Further support for this conclusion is provided by the fact that exactly the same type of variation is present in ferrocene (Dunitz, Orgel & Rich, 1956), although the authors considered that the deviations from strict D_{5d} symmetry were of doubtful significance.

All theories of bonding in π-cyclopentadienyl compounds assume that the metal atom is bonded equally to all five carbon atoms in each ring. Although the results for ferrocene and biferrocenyl suggest that this is not so, it is just possible that the observed variations in bond distance are the result of systematic errors, such as extinction or absorption (as suggested by Dunitz, Orgel & Rich (1956) for ferrocene). Investigations of other molecules of this type are therefore desirable before any speculations about bonding should be made.

The C(7)–C(7′) bond joining the five-membered rings ($1{\cdot}48 \pm 0{\cdot}04$ Å) corresponds to an sp^2–sp^2 single bond (Dewar & Schmeising, 1959).

The bond angles show deviations from the angle in a regular pentagon; the mean angle in the cyclopentadienyl rings is 108°. The C(6)–C(7)–C(7′) (132°) and C(8)–C(7)–C(7′) (121°) angles differ significantly, and this difference is probably associated with the bond length variations described above.

All the intermolecular contacts (Table 6 and Fig. 3) correspond to normal van der Waals interactions.

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