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Organometallic Complexes. VII.* The Structure of the Iron Carbonyl Phenylacetylene Complex, $Fe_2(CO)_6(C_6H_5C_2H)_3$

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$Fe_2(CO)_6(C_6H_5C_2H)_3$ crystallizes with four molecules per unit cell in space group $P2_1/n$ with

$$a = 11{\cdot}963 \pm 0{\cdot}003,\ b = 20{\cdot}442 \pm 0{\cdot}005,\ c = 10{\cdot}326 \pm 0{\cdot}003\ \text{Å};\ \beta = 93°\ 24' \pm 5'.$$

The structure has been determined from visually estimated three-dimensional intensity data obtained using Co $K\alpha$ radiation. The iron positions were found from a sharpened Patterson synthesis. Twenty-six of the light atoms were located by use of a minimum function and the remainder from two successive Fourier syntheses. Least-squares refinement led to a final R value of 15%.

The three phenylacetylene residues and one carbonyl group form a seven-membered carbon chain with phenyl groups at the 1, 3 and 6 positions and the carbonyl group in the 5 position. This chain is σ-bonded from the 1, 4 and 7 positions to one iron atom to form two fused five-membered rings with an iron and a carbon atom in common. This iron atom is also bonded to three carbonyl groups and is octahedrally coordinated. The second iron atom is linked to the remaining two carbonyl groups, to the three-electron donor system C_1–C_2–C_3 and to C_6–C_7.

Bond lengths are: Fe–Fe, 2·501 ± 0·003 (e.s.d.); Fe–C (carbonyl) 1·775 ± 0·012; Fe–C(σ) 2·006, 2·097, 2·088; Fe–C (three-centre) 1·96–2·23 Å. All other bond lengths are normal.

Introduction

The reaction between iron dodecacarbonyl and phenylacetylene (Hübel & Braye, 1959) gives a number of organometallic compounds of which $Fe_2(CO)_6(C_6H_5C_2H)_3$ is of particular interest. Although very stable at ordinary temperatures, it decomposes at its melting point to give 1,3,5-triphenylbenzene. In boiling benzene it is converted to two isomers of triphenyltropone iron tricarbonyl, $Fe(CO)_4(C_6H_5C_2H)_3$. Recent work (Braye & Hübel, unpublished) has shown that it is also possible to obtain triphenyltropone from $Fe_2(CO)_6(C_6H_5C_2H)_3$ by treatment with triphenylphosphine. In view of these interesting reactions it seemed worth while to determine the structure of this complex.

Experimental

Crystals of the complex were obtained from a petroleum–ether/benzene solution as described by Hübel & Braye. They are thin black monoclinic plates lying on (010) and bounded by general forms.

Cell dimensions were determined by Farquhar & Lipson's (1946) method from oscillation photographs taken with Co $K\alpha$ radiation

$$(\lambda\alpha_1 = 1{\cdot}78890,\ \lambda\alpha_2 = 1{\cdot}79279\ \text{Å})$$

and are

$$a = 11{\cdot}963 \pm 0{\cdot}003,\ b = 20{\cdot}442 \pm 0{\cdot}005,$$
$$c = 10{\cdot}326 \pm 0{\cdot}003\ \text{Å},\ \beta = 93°\ 24' \pm 5'.$$

Systematic absences ($h0l$ for $h+l$ odd and $0k0$ for k odd) were determined from Weissenberg photographs about the b and c axes. The space group is thus $P2_1/n$. This orientation was chosen to make the a and c axes as nearly orthogonal as possible. The observed density is 1·54 ± 0·02 g.cm.$^{-3}$ and that calculated for $Z = 4$ is 1·544 g.cm.$^{-3}$.

Multiple-film equi-inclination Weissenberg photographs were taken about the a ($h = 0 \rightarrow 4$), b ($k = 0$ and 1) and c ($l = 0 \rightarrow 6$) axes using crystals 0·18 × 0·13 × 0·10 mm., 0·11 × 0·08 × 0·08 mm., and 0·12 × 0·09 × 0·07 mm., respectively, the shortest dimension being in the b direction. In this way 3415 of the 3685 reflections accessible to Co $K\alpha$ radiation were recorded. Of these 2631 were strong enough to be observed. Intensities were estimated by visual comparison with a set of standard spots prepared from the same crystal. No attempt was made to correct for absorption or for variation in spot shape due to the irregular shapes of the crystals. Allowance was made for the extension and compression of spots on the upper-level Weissenberg photographs following Phillips (1954). Corrections for Lorentz and polarization factors were made by direct computation. Wilson's (1942) method was used to place the intensities on an absolute scale. This method also gave a mean temperature factor of 3·0 Å^2.

Determination of the structure

While three-dimensional intensity data were being collected, attempts were made to interpret the c- and a-axis projections. Sharpened Patterson functions were calculated for both these projections. In the

* Part VI. Krüerke, U., Hoogzand, C., & Hübel, W. (1961). *Chem. Ber.* **94**, 2817.

c-axis Patterson projection several more or less consistent sets of possible iron–iron vectors were found. All but one were eliminated by the use of a Buerger (1951) minimum function. A *c*-axis electron-density projection was calculated using signs from the iron-atom contributions alone but could not be interpreted in terms of any conceivable structure. It was impossible unambiguously to determine the *z* coordinates of the iron atoms from the *a*-axis Patterson projection, probably because these coordinates do not differ greatly and are both close to 0·75.

The three-dimensional intensity data were sharpened and then modified by the factor

$$(\sin\theta/\lambda)^4 \exp\{-25{\cdot}64\ (\sin\theta/\lambda)^2\}$$

(Shoemaker *et al.*, 1953) which has a maximum at $\sin\theta=0{\cdot}5$ and a very small value at $\sin\theta=1$. From these modified data a three-dimensional Patterson function was calculated using the *M*-card method* (V. Schomaker, unpublished). The positions of the iron atoms were easily found from the Patterson function. The vectors between one iron atom and the three atoms related to it by the cell symmetry were used as the basic translations in the computation of a minimum function of rank 4. From the minimum function twenty-six light atoms were located in chemically reasonable positions. A retrospective examination showed clearly three more atoms in positions that were *a priori* unreasonable.

These twenty-eight atoms were used to calculate structure factors on an IBM 650 computer. The scattering factors used were taken from Berghuis *et al.* (1955) for C and O, from Freeman & Wood (1959) for Fe and, in the last stages of the work, from McWeeny (1951) for H. The values for Fe were reduced by a dispersion correction of 3·89 electron units calculated from the table given by James (1950). The discrepancy factor R was 0·41. A three-dimensional Fourier synthesis showed eight more atoms and a further structure-factor calculation ($R=0{\cdot}38$) and Fourier synthesis indicated the positions of the two remaining atoms. A third calculation of structure factors ($R=0{\cdot}28$) and Fourier synthesis confirmed these positions. The structure was refined by six applications of the method of least squares using the programme NYXR2 which is a development of NYXR1 (Friedlander, Love & Sayre, 1955) for the IBM 704 computer. The F_o were weighted simply according to the multiplicity with the exception that the non-observed data for which F_o was taken as $(\frac{1}{2}F^2_{\text{min.}})^{\frac{1}{2}}$ were given 20% of this weight. Refinement of an isotropic temperature factor for each atom was introduced in the third cycle. The progress of the refinement is shown in Table 1 where

$$R=\Sigma(K|F_o|-|F_c|)/\Sigma K|F_o|$$
$$\text{and}\quad R'=\Sigma w(K|F_o|-|F_c|)^2.$$

* The master cards were kindly supplied by Dr V. Schomaker.

Table 1. *Progress of the least-squares refinement*

	R	R' 1st weighting	R' 2nd weighting
Parameters from 3rd Fourier synthesis	0·29	—	—
1st least-squares cycle	0·232	120516	—
2nd cycle	0·193	86053	—
3rd cycle, temperature factor refinement introduced	0·189	81815	—
4th cycle	0·178	72472	—
5th cycle	0·172	67961	—
6th cycle	0·169	66271	—
6th cycle, non-observed reflections omitted	0·151	64669	313650
7th cycle, hydrogen atoms included	0·145	—	299420
8th cycle	0·149	—	267106
9th cycle	0·152	—	261728

A three-dimensional difference synthesis was calculated using the structure factors calculated during the sixth refinement cycle for reflections of $\sin\theta/\lambda \leq 0{\cdot}3$ Å^{-1}. There were more or less well-defined positive regions of height ca. 0·4 e.Å^{-3} in the difference synthesis corresponding to the fifteen phenyl hydrogen atoms. In view of the broadness of some of these peaks these hydrogen atoms were placed on the diagonals of the benzene rings so that C–H = 1·05 Å.

The three remaining hydrogen atoms showed up as relatively sharp peaks from which their positions could be determined. There were about five other peaks of similar size which could not be accounted for by hydrogen atoms and in some cases might be due to anisotropic thermal vibration of phenyl carbon atoms. There were also negative regions (−0·7 e.Å^{-3}) centred on the iron atoms; these are discussed below.

The refinement was completed by three more least-squares cycles in which hydrogen contributions were included and the non-observed reflections were omitted. The weighting system was changed to $w=500\ p/F^2$ (where p is the multiplicity) with a minimum value of 1 and a maximum of 25. This change caused R to increase slightly (Table 1) owing to the change in scale factor. The effect of the hydrogen contributions to the structure factors is negligible and the improvement in R' during the last three refinement cycles was due mainly to the change in the weighting system. Examination of the values of observed and calculated structure factors from the ninth cycle showed that in most cases where $|F_o|-|F_c|$ was large this quantity was negative, suggesting a systematic error in scale or temperature factors. This was borne out by the negative regions round the iron atoms in the difference Fourier synthesis. It has recently been pointed out (Geller & Durand, 1960) that the programme NYXR2 has a theoretical fault in that it applies the scale factor correction to the observed data which should be kept constant in any least-squares treatment. They too found that this programme gave low values of the temperature factor.

Table 2. *Comparison of observed and calculated structure factors*

N signifies a negative value

K	$\|F_o\|$	F_c
00 K 00		
02	101	112N
04	44	43N
06	75	82N
08	26	25
10	76	89
12	38	39
14	61	72N
16	14	8N
18	7	5
20	15	8N
22	8	3N
00 K 01		
01	22	17
02	164	199N
03	80	84
04	66	66
05	21	19
06	78	82
07	26	25N
08	35	34
09	48	48
10	14	11N
11	4	2N
12	68	82N
14	15	11
15	9	8
16	41	51
17	13	11N
18	23	21N
19	3	2N
20	11	11N
00 K 02		
00	120	125N
01	5	N
02	70	71
03	62	70N
04	38	38
05	18	12
06	53	53N
07	24	20N
08	41	41N
09	14	11
10	13	11N
11	21	19N
12	26	25
13	19	19
14	60	73
15	20	17N
16	6	7N
17	16	13N
18	31	32N
19	18	17
20	14	8
21	17	20N
22	11	11
00 K 03		
01	85	87
02	103	113
03	63	72N
04	47	49
05	54	56
06	20	17N
07	5	4
08	34	35N
09	57	56N
10	19	13
11	71	78
12	38	39
13	5	2N
14	16	13
15	24	24N
16	42	39N
17	6	9
18	5	7N
19	15	10N
20	19	15
00 K 04		
00	95	96N
01	7	3
02	53	51
03	41	41
04	49	50N
05	5	1N
06	42	42N
07	36	38N
08	37	37
09	32	28
10	45	47
11	51	51
12	10	7N
13	46	56N
14	26	26N
15	40	45
17	10	8N
18	20	15
19	2	6N
20	3	7
21	10	14
00 K 05		
01	26	25N
02	55	55N
03	8	4N
04	23	27N
05	66	70N
06	21	21
07	17	12
08	29	28
09	33	38
10	23	22N
11	39	42N
12	28	23N
13	21	9
14	17	13
15	17	13
16	7	2N
18	20	19
19	20	20
20	13	14N
00 K 06		
00	9	7
01	42	39
02	53	56N
03	27	27
04	30	30
05	7	7N
06	51	58
07	32	35
08	32	32N
09	43	40N
10	34	31N
11	23	18N
12	26	25
13	17	17
15	15	11
17	9	7
19	4	5N
00 K 07		
01	22	24N
02	13	14N
03	19	17
04	24	22
05	21	21
06	25	26N
07	6	10
08	25	25N
09	11	9N
10	31	36
11	25	24N
12	2	3
13	4	3N
14	13	12N
16	9	10
00 K 08		
00	39	42N
01	7	5N
02	30	30
03	25	27
04	20	21N
05	4	3
06	16	20N
07	22	20N
08	18	17
09	35	33N
10	11	4N
11	9	4N
12	17	17N
13	4	7
14	2	2N
15	5	6N
00 K 09		
01	4	3N
02	28	27
03	22	23N
04	31	30N
05	16	13
06	14	11
09	6	5N
10	22	19N
11	4	6
12	8	7N
13	14	17N
00 K 10		
00	16	12N
01	20	22N
02	5	5
03	10	5N
04	3	1N
05	25	22
06	9	6
07	5	2N
08	12	11N
09	8	9
10	3	2N
00 K 11		
01	16	18N
02	5	6
03	14	15
04	10	11
05	6	4
01 K 00		
01	110	124
02	29	30
03	83	88N
04	29	29N
05	35	33N
06	40	39
07	36	34N
08	50	52
09	107	111
10	46	44N
11	5	2
12	18	17N
13	36	34N
15	40	44N
17	22	23
18	28	31
20	24	23N
21	18	17N
22	13	15N
01 K 01		
00	56	58
01	121	148N
02	28	26
03	57	61N
04	77	77N
05	52	54
06	10	11
07	36	39
08	75	81N
09	20	18
10	18	14
11	37	40N
12	31	34
13	26	31N
15	29	30
16	12	11
17	22	18
18	13	12N
19	29	31N
20	8	10N
01 K 02		
01	90	105N
02	112	130
03	41	45
04	27	26
05	30	31N
06	61	62N
07	19	13N
08	15	13N
09	15	11N
10	69	66N
11	27	23N
13	40	41
15	47	50
16	5	1N
17	52	55N
18	13	7N
19	8	7N
20	6	5N
21	13	13
01 K 03		
00	101	118
01	54	57N
02	21	23
03	16	14N
04	14	7N
05	70	71N
06	19	15N
08	13	11N
09	23	19N
10	21	18
11	23	18
12	20	19
13	30	32
14	23	18N
15	51	45N
16	24	24N
17	17	13N
18	10	11
19	24	23
20	17	17N
01 K 04		
01	45	47
03	46	54N
04	31	36
05	14	15
06	11	6
07	22	18N
08	47	50N
09	10	8N
10	19	20
11	20	20
12	8	6N
13	22	18N
14	16	16N
15	19	17N
16	28	29
17	12	11
18	20	21N
19	15	12
20	12	12
21	8	12N
01 K 05		
00	43	50N
01	33	30
02	29	31N
03	61	79N
04	29	27
05	24	24
06	69	73N
07	17	13N
08	44	48
09	24	27
11	42	39N
12	30	36N
13	14	10
14	29	25
15	5	6N
17	25	23
19	7	9N
20	11	12
01 K 06		
01	16	17N
02	7	9
03	14	14
04	32	35N
05	46	53
06	32	34
07	18	21N
08	26	27
09	20	17N
10	17	10N
11	15	12N
12	6	6
13	18	11
14	17	16
15	15	13N
16	5	6N
17	20	15
18	13	12
19	6	9N
01 K 07		
00	6	6
01	25	28
02	10	13N
03	31	37
05	14	12
06	46	60
07	17	22N
08	22	27N
09	12	18
10	18	21N
11	30	34
12	6	5
13	3	3N
16	4	5
01 K 08		
01	15	17N
02	14	12
03	19	20
05	28	34N
06	24	27
08	12	16N
09	21	22
10	6	10
11	15	17N
12	8	13
13	20	25N
14	4	4N
15	8	8
01 K 09		
00	32	35
02	17	16N
06	27	1N
08	15	
09	7	17N
10	5	11
12	3	1N
01 K 10		
01	7	8N
02	17	20N
03	12	14N
04	12	13
05	22	28
06	8	8
07	9	10
09	15	20N
01 K 11		
00	23	29N
04	14	21
02 K 00		
00	118	139
01	43	42
02	107	119N
03	12	11
04	34	31N
05	44	48N
06	52	43
07	106	111
08	18	16
09	28	25N
11	31	30N
12	19	18N
13	29	30
14	23	24N
15	21	25N
16	8	10
17	30	25
19	19	19
20	11	14
21	16	18N
22	10	12N
02 K 01		
01	49	55N
02	122	127N
03	59	62N
04	67	68
05	50	51N
06	19	14
07	19	15
08	24	25
09	51	53N
10	21	16
11	53	53
12	26	29N
14	6	3
15	18	19N
16	56	58
17	31	31
18	5	2
19	20	20N
20	14	9N
02 K 02		
00	95	120N
01	101	121N
02	14	12
03	28	30
04	33	33
05	20	14N
06	38	40N
07	13	8N
08	12	10N
09	15	13
10	32	33N
11	24	20
12	15	10N
13	23	18N
14	41	40
15	19	11
16	5	2
18	22	20N
20	20	16
21	13	11
02 K 03		
01	58	65
02	81	93
03	28	28
04	24	23N
05	25	18
06	8	7N
07	15	13N
08	12	13
09	10	9
10	42	39N
11	7	5N
12	43	46
13	8	9N
16	42	39N
17	11	8N
18	22	20
19	18	17N
20	19	17
02 K 04		
00	95	104
01	42	45N
02	11	5
03	13	10N
04	32	34N
05	7	7N
06	28	28
07	18	17N
08	17	12N
09	23	22
10	29	30
13	33	33N
14	49	48N
15	37	36
17	15	17N
18	28	29
20	8	9N
02 K 05		
01	12	6
02	72	88N
03	24	25
04	45	46
05	42	45N
06	17	7N
07	21	16N
09	11	10
11	17	11N
12	53	56N
13	28	25
14	17	15
15	20	15
16	10	7N
17	21	18N
18	8	5
19	15	15
02 K 06		
00	47	56N
01	21	19
02	15	11
03	23	23N
04	40	50
05	29	33N
06	11	7
07	46	51
08	12	6
09	7	6N
10	25	21N
11	18	13N
13	15	11
14	33	32
15	23	21N
16	12	7N
17	11	7
18	10	8N
02 K 07		
01	11	12
02	28	30
03	7	9N
04	24	26
05	26	29
06	26	34N
07	8	10
08	6	9N
09	30	30N
10	23	21
11	12	15
12	30	26
13	11	11
14	13	14N
15	7	6N
16	5	7N
17	6	11
02 K 08		
00	15	2N
01	15	15N
02	14	13
03	19	13
04	23	21N
05	6	6
06	23	22N
07	22	26N
08	29	33
10	17	15
11	24	22
12	8	11N
13	9	10N
02 K 09		
02	9	7
04	35	37N
06	23	25
08	18	18
09	30	37
10	24	27N
11	16	17N
12	5	6
02 K 10		
01	6	4
03	14	13N
04	4	4
05	11	9
06	26	33
07	5	8
08	21	27N
02 K 11		
01	5	7N
03	3	4
03 K 00		
01	66	70
02	35	38N
03	86	102
04	132	154N
05	15	14
06	9	7N
07	12	8
08	79	82
09	91	95
10	55	53N
11	44	44N
12	6	4
13	11	7N
15	29	25N
16	14	12N
17	13	6N
18	19	16
19	26	27
20	7	5N
21	10	7N
22	5	6N
03 K 01		
00	28	29
01	124	136
02	20	22N
03	148	163N
04	24	17
05	39	41
06	49	51
07	61	60
08	48	45N
09	29	24
10	27	19N
11	29	32N
12	46	51
13	9	9N
14	29	31N
15	12	12N
16	30	25
17	28	25
18	6	5
19	7	3
20	15	14N
21	5	5N
03 K 02		
01	35	36N
02	49	56N
03	22	16N
04	4	1N
05	16	10
06	23	14
07	30	24
08	5	5N
09	24	24N
10	69	74
11	25	23N
12	24	23N
13	39	43
14	33	33N
15	21	16
16	35	29
17	15	12N
18	20	13N
19	12	11N
20	6	4
21	19	21
03 K 03		
00	43	45N
01	75	85
02	87	102
03	49	50
04	18	16N
06	22	16
07	8	5
08	20	14
09	18	15N
10	22	19
11	40	39
12	35	35N
13	42	37
14	9	5
15	35	31N
17	23	19N
19	26	24
20	10	10
21	4	4
03 K 04		
01	77	83
02	9	5N
03	74	78N
04	46	47N
05	39	36N
06	72	76
08	28	24
09	11	5
10	12	11N
11	57	66
12	5	2
13	50	50N
14	4	6N
17	11	11
18	12	8
03 K 05		
00	27	29N
01	82	98N
02	26	20N
03	20	14N
05	40	44
06	13	6N
07	20	18N
08	20	18N
09	19	17
10	5	6N
11	47	45N
12	22	21
13	16	13N
15	26	22
16	8	5
17	21	21
18	6	8N
19	20	25N
03 K 06		
01	34	38N
02	23	21
03	53	61
05	6	7
06	32	29N
07	38	34N
08	21	20
09	17	11N
10	17	11N
11	10	9N
13	30	26
14	6	3
15	16	15
16	21	15N
17	19	16N
03 K 07		
00	39	40
01	32	36
02	23	23N
03	26	28
04	11	10N
05	27	28N
07	17	16N
08	7	7N
09	20	15N
10	17	12N
11	16	10
12	26	22
13	16	15
14	9	8N
15	17	15N
03 K 08		
01	22	20
03	22	22N
04	16	17
05	39	37N
06	30	25N
07	4	3N
08	33	30N
09	7	5
10	18	18
12	6	6N
13	3	1N
14	9	8N
03 K 09		
00	13	8
01	15	13N
02	9	7N
03	9	9N
06	28	27N
07	17	18
08	16	16
10	5	6
11	15	12N
12	13	17N
03 K 10		
04	4	6N
05	11	10
06	16	15
07	4	2
04 K 00		
00	130	149
01	128	145N
02	36	40
03	108	118N
04	14	13
05	26	19
06	91	90N
07	70	75
08	63	67
09	24	20
10	19	20
11	44	44N
12	24	24N
13	25	23N
14	15	14
15	22	18N
16	42	35N
17	5	6
18	24	18N
20	12	16
21	15	17N
04 K 01		
01	75	87N
02	22	19N
03	34	33
04	91	102N
05	44	48
06	38	40
07	16	15
08	25	23
09	78	79N
10	11	9
11	20	17
12	44	45N
13	25	25
14	12	13
15	24	19N
16	22	21N
17	12	12
18	19	16
19	10	9N
20	15	12N
04 K 02		
00	44	44N
01	41	43N
02	48	47N
03	33	31
04	17	14
05	24	20N
06	15	8N
07	42	42N
08	33	30N
09	21	17
10	20	15N
11	45	49
12	11	13

Table 2 (*cont.*)

K	$\|F_o\|$	F_c
13	54	53N
14	22	17
15	20	13N
16	24	17
17	19	15N
18	18	11N
20	12	12N
21	12	18

04 K 03

01	15	11
02	22	20
03	21	25
04	15	10N
05	62	62N
06	18	15N
07	17	15
08	17	14N
09	61	61
10	12	7
11	18	14N
12	40	40
13	19	18N
14	31	30N
15	11	10
17	31	28N
18	7	8N
19	17	17
20	14	14

04 K 04

00	81	89
01	45	52
02	21	18N
03	16	11N
04	51	52N
05	30	29N
07	51	54
08	5	5
09	5	5N
10	24	23
11	21	24N
12	18	14N
13	14	10
14	20	18N
15	28	23
18	26	21
19	6	9

04 K 05

01	25	24N
02	83	98N
03	31	31N
04	37	35
05	25	25
06	30	29
07	13	8N
08	9	9
09	21	21N
10	12	8N
11	6	5
12	31	27N
13	23	17
14	9	11
16	26	20
17	9	8

04 K 06

00	54	62N
01	34	35
02	43	47
03	20	17
04	26	27
05	25	24
06	17	10N
07	38	33N
08	17	12N
09	15	9N
10	18	14N
11	21	17
13	13	10
14	39	32
15	17	10
16	22	24N
17	5	7

04 K 07

01	12	10
02	63	61
03	11	9
04	8	10N
06	8	5N
08	8	7N
09	6	8N
12	13	11
14	16	16
15	8	8N
16	16	26N

04 K 08

00	23	20
01	13	13N
02	10	7
03	18	16
04	30	31N
05	8	8
06	10	11N
07	19	17N
08	5	5N

K	$\|F_o\|$	F_c
09	18	15N
10	18	18
11	10	9
12	11	11
13	9	11N
14	13	21N

04 K 09

03	7	7
05	20	21N
08	9	10
09	10	12
10	10	9N
11	11	16N

04 K 10

00	17	21N
01	12	12
04	6	4
06	8	9
07	13	20

05 K 00

01	13	6N
02	78	82N
03	75	74
04	32	32
05	51	52N
06	22	22
07	6	4N
08	69	69
09	28	32
10	26	25N
11	29	33N
12	30	37N
13	11	13N
14	27	26
15	24	22
16	24	20
17	22	17N
19	8	7
20	14	14N

05 K 01

00	98	116N
01	46	50
02	18	20N
03	82	99N
04	32	34
05	27	22N
06	15	12
07	82	94
08	43	49N
10	26	27N
11	19	14N
12	42	47
13	20	20
14	19	15
15	26	31N
17	23	20
18	19	18N
19	13	8
20	8	6N

05 K 02

01	25	16
02	38	40
03	82	85N
04	27	33
05	43	45
06	89	96N
08	29	25N
09	43	45N
10	20	19
12	28	30
13	36	35
14	25	19N
15	15	15N
16	8	4
17	20	14
18	13	11N
19	14	12N
20	12	12

05 K 03

00	25	18
01	53	54N
02	26	21
04	36	31
05	25	13N
06	48	43N
07	30	24N
08	48	44
09	12	8
10	27	21
11	32	29
12	49	48N
13	31	25N
15	19	11N

05 K 04

01	30	26
02	25	24
03	31	29N
04	73	72N
05	10	6N
06	42	40

K	$\|F_o\|$	F_c
07	14	10N
09	38	45
10	34	35N
12	12	10
13	26	20N
17	6	2N
18	19	18
19	10	11

05 K 05

00	35	35
01	27	26N
02	41	46N
03	31	29N
04	18	9
05	24	15
06	38	37
08	30	27N
10	29	22N
11	43	41N
12	31	23
13	19	9
14	21	13N
15	18	10
16	27	25

05 K 06

01	40	37N
03	47	47
04	41	37
05	42	38
08	22	16N
09	21	11N
10	45	33
12	16	14N
15	22	21

05 K 07

00	23	29
01	44	52

05 K 09

00	11	3
01	31	37N

06 K 00

00	17	17
01	89	105N
02	16	13
03	27	28N
04	19	16
05	59	62
06	17	12
07	25	30N
08	38	46
09	12	11
10	17	15N
11	23	25N
13	15	10N
14	8	4
15	34	31
17	18	12
18	10	6N
19	11	7N

06 K 01

01	61	73N
02	14	7
03	98	109
04	29	27N
05	15	11
06	29	33
07	23	22N
08	22	21
09	18	15N
10	8	8N
11	31	37N
12	16	15N
13	42	45
14	32	31
15	25	16
19	16	13N

06 K 02

00	26	29N
01	30	32
02	26	22N
03	33	36
04	27	19
05	29	27N
06	48	56
07	9	10N
08	34	35N
09	12	11
10	19	12N
11	36	37
12	22	18
14	10	8N
15	32	26N
16	8	5
18	15	10
19	16	15

06 K 03

01	52	57

K	$\|F_o\|$	F_c
03	26	18
04	34	33
05	37	36N
06	35	28N
07	24	17
08	35	29N
09	29	26
10	42	38
11	16	10N
13	16	11N
15	21	13N
17	8	5
18	14	12N

06 K 04

00	32	37N
01	23	17N
02	32	28
03	20	16N
04	15	7N
05	29	28N
06	22	14N
07	52	58
08	30	30
09	17	10
11	38	27N
12	44	40N
13	23	15N
14	18	15
15	12	7
16	10	5N

06 K 05

01	27	20N
02	34	30N
03	30	27N
04	28	22N
05	15	10
06	19	12
07	22	16
08	25	21
09	52	44N
10	39	34N
11	27	19
12	25	17
13	19	10
14	25	14N
16	14	9N

06 K 06

00	39	37N
01	12	3N
03	17	10
04	21	14
05	17	9
06	29	24
07	33	30N
08	30	20N
10	18	12N
11	29	22
12	14	6N
14	20	15

06 K 08

00	28	31

07 K 00

02	70	76N
03	15	10N
05	45	58
06	28	31
07	16	17
08	17	11N
09	18	17N
10	13	10
11	13	11N
12	56	68N
13	26	20N
16	17	15

07 K 01

00	75	96N
01	55	66
02	22	22N
03	44	52N
04	46	57
05	25	30
06	14	11N
10	38	39N
14	31	25
17	11	6N
18	17	15N

07 K 02

01	21	16N
02	49	50
03	27	22N
05	28	26
06	27	24N
07	20	12
09	39	46N
12	34	25
15	17	14
16	29	29N

K	$\|F_o\|$	F_c
07	K	03
00	52	53
01	28	23N
03	24	18
04	29	21N
05	39	34
07	35	33N
08	14	10
09	27	21
10	32	29
11	24	19
13	26	19N
14	46	42N

07 K 04

01	15	10N
02	39	43N
03	30	33
04	27	22N
05	29	29N
06	24	18
08	49	41
09	28	21
10	22	14N
11	27	22N
13	17	9
15	14	9
16	20	22
17	10	10N

07 K 05

00	40	43N
01	12	7
02	12	5N
04	12	8
05	28	20N
06	25	20
07	26	22
08	26	18N
10	34	28N
11	22	16N
12	25	18
13	18	15
14	14	6
15	13	11N

07 K 06

01	22	15
03	29	21N
05	20	13
06	25	17N
08	37	22N
09	16	10N
10	48	38
11	28	17

07 K 07

00	12	10

07 K 09

00	9	10
01	15	23N

08 K 00

00	15	6N
01	56	68N
02	14	9N
03	40	46N
04	18	17
05	27	28
06	42	49
07	13	13
08	30	30N
09	16	13
10	21	22N
11	18	16N
12	7	5
13	14	14N

08 K 01

01	62	74N
02	14	11
03	33	32
04	29	27
05	25	22
06	20	8N
07	8	5N
08	26	30N
10	33	29
11	36	33N
12	19	13N
14	11	5N
15	25	21
17	9	8N

08 K 02

00	23	20N
01	34	37
02	21	13N
03	31	28
04	24	17N
05	46	51N
06	11	8N
07	18	16

K	$\|F_o\|$	F_c
09	31	26N
10	24	17
11	14	8
12	18	11N
13	36	31
14	17	7
15	25	23N
16	9	5N
17	15	14N

08 K 03

01	33	32
02	29	23N
03	34	31N
05	18	13
07	11	6N
09	13	10
10	10	3
11	17	7
12	20	15N
15	25	22N
16	10	3

08 K 04

01	45	38N
04	26	11
05	14	11
06	18	15N
08	26	18
09	17	7
10	10	7N
11	10	10N
13	26	24N
14	19	16
15	23	26

08 K 05

01	27	27N
02	28	22
03	42	41
04	35	30N
05	19	8N
06	19	10
07	10	9
09	28	23N
10	23	15N
12	26	22
13	22	15

08 K 06

00	44	38
01	42	39
02	30	17
04	20	13N
05	20	15
06	24	14
07	29	22N
08	23	15N
09	8	5
10	18	12
11	19	13

08 K 07

01	19	20

09 K 00

03	19	17N
04	15	12N
05	50	57
06	13	12
07	21	21
08	21	20
09	42	37N
10	26	29N
11	18	15
12	17	9
13	14	8
14	5	3
16	9	10

09 K 01

00	21	24N
01	12	6
02	14	10N
04	27	32
05	14	13
06	14	7
07	38	42N
09	17	15
10	8	6N
11	36	34
13	13	7N
14	18	14

09 K 02

01	23	21
02	15	14
03	21	16N
04	19	20
05	21	15N
06	19	14N
08	9	9N
09	34	30
11	23	13N
12	14	10

K	$\|F_o\|$	F_c
14	14	11
15	8	6

09 K 03

00	33	38
02	35	26N
03	26	12N
04	28	17
06	28	17
07	18	12
10	9	9
11	33	16N
13	18	10
14	32	28N

09 K 04

01	11	8N
02	35	29N
03	11	9
04	33	29
06	11	7N
07	9	7N
12	23	19N

09 K 05

00	42	46N
02	15	7
03	26	17
10	19	13N
11	11	6N
12	10	8

09 K 06

02	45	38
03	16	10N
04	12	10N
05	25	12
06	16	9N
09	17	7N
10	9	6

09 K 07

00	21	28
01	14	17N

10 K 00

00	11	11
04	13	15
05	7	4N
07	17	10
08	14	12N
09	26	24N
10	20	19N
12	17	17

10 K 01

02	23	29
03	9	5N
04	11	10
05	17	13
06	12	6N
07	8	1N
08	28	29N
09	23	20N
10	31	28
11	14	10

10 K 02

00	32	28N
01	29	23
02	29	24
06	24	14N
07	16	10N
08	30	26
09	15	7
12	29	31N
13	17	15

10 K 03

01	34	29
03	28	15N
06	20	13
07	9	
08	14	10
10	27	28N
12	17	10

10 K 04

00	13	10N
01	32	26N
02	17	14N
06	33	33
08	21	15N
11	11	7N

10 K 05

01	33	31N
02	12	8N
03	31	29

K	$\|F_o\|$	F_c
10	K	06
01	32	26
02	22	13N
03	25	17
05	17	8N
06	12	8
07	10	7N

11 K 00

01	20	18N
02	23	24
03	14	11
04	28	27N
05	7	11
06	13	10
09	15	14N
10	19	22N

11 K 01

01	19	14
02	20	13N
03	14	9
04	9	7N
05	14	12
06	24	15
07	26	24N
08	30	29N
09	9	2
11	17	16
12	7	10

11 K 02

02	16	8
03	14	8N
04	33	29
05	24	18N
06	21	15N
07	10	7
08	19	16N
09	20	14
10	18	16
11	9	8N

11 K 03

00	27	16
03	19	11N
05	19	14N
06	26	22N
07	33	32
08	25	24
09	14	12N
10	9	5

11 K 04

02	25	20N
03	9	6N
05	25	18
06	12	6
08	10	8

11 K 05

00	21	20N
04	19	17

12 K 00

00	33	31N
02	19	14
03	12	10
04	20	21
05	11	8N
06	9	5
07	13	10
08	9	7N
09	8	11N

12 K 01

02	34	34
03	19	13N
05	20	17
06	16	9N
07	8	
08	11	10N

12 K 02

00	18	12
02	6	9
03	15	9
04	22	20N
05	9	5
06	16	13N
07	17	21N

12 K 03

04	18	13N
05	14	9N
06	14	14

Table 2 (*cont.*)

K	|Fo|	Fc
12 K 04		
00	4	3
01	6	5
02	9	6N
13 K 00		
01	12	14N
03	12	23
13 K 01		
00	13	12
01	16	24
01N K 01		
00	85	82N
01	186	190N
02	93	94
03	106	107N
04	86	89N
05	28	28
06	150	161N
07	31	30
08	36	36
09	25	19
10	34	37
11	23	26N
12	23	21N
13	13	14N
14	18	14
15	32	38
16	20	23N
17	18	16
18	7	4
20	22	22
01N K 02		
01	47	46N
02	42	42
03	34	33N
04	95	107N
05	26	23N
06	45	46
07	28	29N
08	98	102
09	68	73N
10	64	63N
11	14	10
12	21	18N
13	41	44
14	18	16
15	4	5
16	7	4N
18	16	14
20	5	9N
21	16	16
01N K 03		
00	146	144N
01	36	35
02	38	41
03	66	70
04	50	49
05	46	39
06	91	96
07	82	83N
08	42	43N
09	18	15
10	44	46N
11	54	56
12	35	32
13	26	25
14	6	2N
15	18	12N
16	22	19N
17	19	16N
19	8	7
20	20	19N
21	12	11
01N K 04		
01	89	94
02	46	52
03	49	52
04	23	25
05	139	156N
07	17	18
08	54	60N
09	46	55
10	82	88
12	10	9
13	15	13N
14	26	26N
15	17	17N
16	5	5
17	9	7N
19	17	18
20	10	9
21	5	6
01N K 05		
00	15	12
01	61	62
02	15	14N
03	37	43N
04	39	45N
05	9	7N
06	12	7
07	72	79
08	40	38
09	35	42N
10	24	27
11	58	66N
12	29	22N
13	25	23
14	20	16N
15	23	17N
16	20	13
17	27	27
18	5	6
19	4	6N
20	10	11
01N K 06		
01	10	11N
02	37	53N
03	13	15
04	6	2N
05	58	69
06	19	14
07	9	3N
08	13	14
09	39	40N
10	23	19N
11	24	25
12	13	13N
15	6	6
16	19	14
17	12	10
18	6	2
19	16	20N
01N K 07		
01	15	18N
03	52	63
04	30	33
05	11	11
06	7	6
07	29	38N
09	5	7N
10	18	14N
11	5	7
14	23	24
15	10	7
01N K 08		
01	10	9N
02	32	39
03	5	6
04	17	19N
05	10	11N
06	21	24N
08	6	6
09	5	6
10	35	43N
11	10	12
12	26	29
13	7	6N
15	5	6
16	9	16N
01N K 09		
00	44	51
02	27	20N
03	19	20N
05	11	13
06	7	6
07	6	10N
08	8	6N
09	6	8
10	6	8
11	5	7N
12	4	7
13	5	6N
01N K 10		
02	22	22N
04	25	27
05	10	10
06	9	9
10	8	11
01N K 11		
00	26	30N
01	13	16
02	8	9
03	4	4N
04	15	21
05	8	13N
02N K 01		
01	40	39N
02	3	1
03	4	3N
04	44	40
05	151	161N
06	12	6
07	12	11N
08	18	10
09	51	56
10	60	60N
11	20	18
12	4	7
13	26	24N
14	13	12
15	9	8N
16	6	5
19	32	32
20	9	9N
21	8	6
22	3	4N
02N K 02		
00	157	172N
01	103	104N
02	30	29N
03	40	43N
04	42	40N
05	85	89N
06	55	54
07	96	107
08	37	32N
09	39	37N
10	23	20N
11	45	49N
12	30	31
13	18	17
14	12	11N
15	13	8N
16	3	1N
17	13	16
20	4	2N
21	18	19N
22	6	10
02N K 03		
01	23	20
02	22	20N
03	36	36
04	107	105
05	96	100
06	66	68N
07	17	12N
08	41	37N
09	80	79N
10	34	39
11	16	13
12	11	4N
13	16	11
14	17	17N
15	10	7
16	7	10
17	25	20N
18	7	6
19	23	19N
02N K 04		
00	12	16
01	80	93
02	6	4N
03	69	75
04	51	48
05	52	46N
06	72	76N
07	32	36N
08	79	91
09	17	15N
11	38	44
12	31	33N
13	4	4N
14	35	31N
15	15	11N
17	19	14N
19	9	8
02N K 05		
01	62	71
02	8	7N
03	66	71N
04	37	44N
05	36	42N
06	11	12
07	14	11N
08	21	23
09	30	33
10	46	55N
12	15	10N
13	18	14N
14	5	13
15	10	9N
16	10	8N
17	14	9
18	16	13
19	10	9
20	3	3N
02N K 06		
00	45	56
01	41	55N
02	29	32N
03	42	48N
04	12	9
05	51	58
06	38	35
07	13	14
08	33	38N
09	31	34
11	32	28N
13	37	34N
15	30	25
16	5	4
17	17	14
19	8	9N
02N K 07		
01	42	48N
02	4	5
03	35	41
04	17	19
05	32	34
06	5	4
07	11	15N
08	5	6
10	5	10
11	27	28N
12	11	13
13	22	24
14	8	6
15	20	22
16	4	3
17	18	23N
02N K 08		
00	24	26N
01	11	10N
02	24	27
03	34	38
04	22	26N
05	33	32N
06	18	17N
07	13	16
08	21	18
09	10	7
11	21	18
12	10	7
13	17	20
14	7	4
15	17	22N
02N K 09		
01	13	18
03	25	25N
09	15	12
10	21	24
11	11	16
12	14	17N
13	10	12N
02N K 10		
00	21	20N
01	8	11N
02	6	9
03	24	24N
04	16	16
05	6	5
06	14	14N
07	16	18
08	15	15
09	8	9
10	6	8N
02N K 11		
01	7	7N
02	10	12
03	5	8
04	8	11N
05	7	7
03N K 01		
00	9	7N
01	70	77
02	56	56
03	4	5
04	46	41
05	12	8
06	63	62N
07	66	65
08	78	92
09	20	20N
10	24	23
11	35	38N
12	27	24N
13	50	54
14	5	6
16	18	17N
17	4	4N
18	19	17
19	12	8
20	8	3
21	17	19N
03N K 02		
01	10	12N
02	55	48
03	21	22N
04	38	37
05	89	90
06	7	3N
07	17	17
08	10	9
09	59	61N
10	54	58N
11	49	54
12	32	35N
14	14	12
15	7	4
16	43	40
17	14	11
18	8	5N
20	24	24N
21	5	3N
03N K 03		
00	51	50N
01	16	14N
02	71	64
03	20	18
05	49	47
06	50	51
07	69	74N
08	13	5N
09	30	29
10	13	7
11	25	20
12	7	7N
13	22	16N
14	52	44
15	18	13N
16	34	31N
17	10	8
18	15	9N
19	7	5N
20	6	3N
21	12	11
03N K 04		
01	29	27N
02	89	93
03	42	39
04	47	44N
05	37	33N
06	6	7N
07	21	22
08	29	24N
09	34	35
10	18	18N
11	47	53N
12	44	42
13	16	15
15	7	7
16	14	14N
20	11	12
03N K 05		
00	103	112
01	14	9
02	72	73N
03	22	21N
04	55	60N
05	16	14N
07	19	9
08	23	21N
09	29	25N
10	32	35
11	7	5N
13	4	4
14	30	30N
15	21	15N
16	22	17
17	17	10
18	22	22
19	7	2N
03N K 06		
01	15	11N
02	80	96N
03	17	13N
04	7	5
05	11	8
06	32	37
07	19	20N
09	28	21
10	19	16
11	13	8N
12	29	27N
13	4	3N
15	9	8N
16	24	22
17	11	8
18	9	9N
03N K 07		
00	60	68N
01	13	12
02	12	11
03	25	28N
04	46	56
06	14	15N
07	31	34
08	10	14N
09	16	23
10	34	33N
11	15	9N
12	11	12N
14	36	40
15	8	5
16	12	12N
03N K 08		
01	20	14N
02	36	35
03	14	15N
04	12	10N
05	24	18
06	25	27N
07	20	15
09	33	36N
11	19	17
12	20	22
13	15	15N
14	14	13N
15	9	6N
03N K 09		
00	24	19
02	7	7
03	17	15N
04	25	28N
05	8	10
06	8	6N
07	24	24N
08	14	11
09	16	14
10	6	4
11	8	11
12	3	2
13	13	15N
03N K 10		
01	6	6N
02	18	17N
03	9	10
04	12	11N
05	15	17N
06	16	19
07	15	10
08	14	15
09	10	11
03N K 11		
00	4	5N
01	3	4
02	12	16N
03	10	11N
04	8	12
04N K 01		
01	45	44
02	42	45
03	34	38N
04	50	48N
05	23	22
06	67	66
07	37	32N
08	15	12
09	31	36
10	61	68N
11	18	14N
12	29	21
13	31	33N
14	10	11
15	21	18N
16	27	12
17	30	27
18	22	18
19	9	9
20	11	9N
21	7	6N
04N K 02		
00	36	35
01	19	12
02	57	58N
03	14	10N
04	32	34N
05	59	54
06	26	24
07	15	12N
08	57	61N
09	25	21N
10	13	10N
11	19	13N
12	42	42
13	32	27N
15	25	18
16	13	8N
17	26	23
18	7	6
19	23	20N
20	12	9N
21	4	6N
04N K 03		
01	30	27N
02	34	34
03	37	33
04	34	33
05	28	25
07	24	18N
08	48	44N
09	9	8
10	40	38
11	37	35N
13	11	6
14	35	25
15	52	46
16	10	9N
17	36	32N
19	11	5N
20	8	10N
04N K 04		
00	16	12N
01	80	89
02	4	2N
03	10	9
04	12	13
06	20	17N
07	30	26
08	7	7N
09	20	17N
10	11	10
11	22	21
12	18	15
13	22	17
14	34	32N
15	17	14N
16	28	31
17	18	15N
18	10	9N
19	21	20
04N K 05		
01	30	27
02	33	24N
03	80	82N
04	6	3
05	17	12N
06	21	20N
07	35	32
09	12	4N
10	24	17
11	34	30
12	12	6N
13	18	14N
15	21	12N
17	27	23
18	16	14N
19	5	5
04N K 06		
00	30	29N
01	58	69N
02	22	25
03	6	2N
04	20	18
05	20	13
06	16	11N
07	16	14
08	26	22
09	10	5
10	9	6
11	25	20N
12	24	17N
13	25	19N
14	11	7
15	37	36
04N K 07		
01	23	23N
02	8	7N
03	26	27
04	36	39N
05	31	38
06	18	16
07	6	6
08	40	33
09	14	12N
10	7	7N
13	8	8
14	10	10N
16	7	6N
04N K 08		
01	31	26N
02	10	10N
03	33	27
05	19	24N
06	37	36
07	29	30N
10	23	23N
12	16	15
13	4	4
14	9	6N
15	9	15N
04N K 09		
01	5	6N
02	7	5N
03	22	20
04	9	9
05	22	18N
06	15	17N
07	7	9
08	11	13N
09	18	20
10	22	22
11	8	9N
12	14	15N
04N K 10		
01	22	18
02	8	8
03	27	25N
04	15	12N
06	6	6N
07	18	21
08	16	18
04N K 11		
02	9	11N
05N K 01		
00	29	33
01	36	40
02	13	5
03	15	14N
04	125	132N
05	26	24N
07	21	22
08	22	21N
09	45	54N
10	34	39
11	16	10N
12	18	13
14	33	35N
15	9	8N
18	24	21
19	13	7
20	9	8N
05N K 02		
01	22	21N
02	63	66N
03	36	42N
04	7	4
05	21	18N
06	40	42N
07	31	33N
09	24	23N
10	16	11
11	20	17
12	53	55N
13	9	11N
15	24	15
16	39	39
17	21	12
18	26	24N
19	13	9N
20	12	12N
05N K 03		
00	79	79N
01	39	40N
02	27	28
03	20	18
04	51	53
05	18	9
07	20	20
09	17	7N
10	33	30N
12	36	39N
13	26	13
14	53	52
15	10	7N
16	24	13N
18	26	22N
20	8	7
05N K 04		
01	25	28
02	76	76
04	19	16N
05	21	20N
06	17	17N
07	18	20
09	30	30N
10	14	11
11	23	19
12	30	33
13	27	20
14	20	18
15	35	39N
16	36	39N
18	12	8
19	12	12N
05N K 05		
00	43	51
01	27	25N
02	31	24N
04	16	15N
05	32	27
07	45	45N
08	29	23
09	27	18
10	10	4
12	19	10N
13	21	14N
14	38	33N
16	21	13
18	16	16
05N K 06		
01	16	15N
02	49	55N
03	44	48
05	27	31N
06	32	34
08	27	21
09	30	28
10	22	15N
12	9	4N
14	17	12N
16	16	13
17	17	18N
05N K 07		
00	36	39N
01	33	46
06N K 01		
01	44	47

Table 2 (*cont.*)

K	$\|F_o\|$	F_c
02	30	31
03	65	66N
04	12	3N
05	16	10
06	7	6N
07	26	29
08	18	18N
09	17	15N
10	17	18
11	47	59
13	45	47N
14	15	13
15	10	
16	17	15N
17	25	25
19	12	8
06N K 02		
00	20	22N
01	52	62N
02	11	6
03	60	69N
04	20	18N
05	44	47
07	12	2
08	37	44
09	31	33
11	17	13N
12	22	18N
13	34	30N
14	23	11
16	22	15
17	23	19
19	19	22N
06N K 03		
01	18	13N
02	17	12N
03	35	36
04	11	12N
05	45	43
06	15	7
07	11	7
08	42	41
10	52	49N
12	25	25
13	21	18
14	27	16
15	29	20
17	42	40N
06N K 04		
00	14	12
01	12	10
02	22	22N
03	30	27
04	52	52
05	27	28N
06	39	41
07	23	26N
08	36	36N
09	12	12
10	12	9N
11	23	21
12	24	23
13	27	22
14	42	41N
15	27	22N
16	22	22
17	17	14N

K	$\|F_o\|$	F_c
06N K 05		
01	23	22
02	25	24N
03	24	20N
04	38	35
05	37	40N
06	32	32N
07	16	11N
08	31	25N
09	45	44
10	19	15
11	15	12N
12	17	12N
14	26	19N
15	28	22N
16	17	14
17	9	6
06N K 06		
00	12	6N
02	8	10
03	19	14N
05	29	26N
06	35	36N
07	37	37
08	24	20
09	10	6N
11	19	17N
12	22	11N
13	9	1
15	7	8
16	10	14N
06N K 10		
00	17	25
07N K 01		
00	56	62
01	24	17N
02	17	12N
04	32	34N
05	37	39
06	33	26
07	20	21N
09	28	28
10	19	17
11	28	33
12	25	24N
13	17	11N
14	26	25N
15	7	4N
16	13	10
18	17	18
07N K 02		
01	34	36N
02	39	48N
04	22	16
06	24	18
08	9	9
09	46	54
11	36	31N
12	45	48N
13	22	14N
14	34	30
16	26	18
17	8	9

K	$\|F_o\|$	F_c
07N K 03		
00	27	22N
01	13	7
02	16	12N
03	25	19N
04	42	44
05	12	12N
06	35	37
07	46	47
08	35	34N
11	27	20N
12	20	13N
13	31	24
14	36	32
16	19	11N
17	12	13N
07N K 04		
01	22	23N
02	16	13
04	30	27
05	20	17
06	23	18N
07	26	25
08	28	19N
09	36	34N
10	32	28
11	14	11
13	23	17
15	19	17N
17	9	8
07N K 05		
00	22	18N
02	22	14
03	24	17
04	26	24N
05	19	13N
06	47	35N
07	42	41N
08	36	28
10	10	7
11	24	15
13	24	13N
16	11	8N
07N K 06		
01	15	15
04	38	31N
05	31	24N
06	25	22
08	25	19
09	30	22
10	27	26N
12	25	13
13	11	7N
14	16	13N
07N K 07		
00	11	17
07N K 09		
00	14	20

K	$\|F_o\|$	F_c
08N K 01		
01	19	12
02	22	23N
03	30	32N
04	38	42
05	20	18N
06	28	31N
07	23	25
08	33	42N
09	16	16
10	23	28
13	33	40N
15	12	9N
17	13	10
08N K 02		
00	28	27N
01	18	10
02	17	13
03	40	45N
04	16	13N
05	13	5
06	41	44N
07	26	21
08	39	28
09	30	23N
11	40	41N
12	27	23N
16	8	4N
08N K 03		
01	25	22N
03	24	15N
04	31	27N
05	52	56
06	34	36
07	16	11N
09	26	22N
10	34	27N
11	27	17N
13	30	20
08N K 04		
00	15	11N
01	19	16N
02	20	16N
03	28	27
04	31	29
05	24	21
07	42	38N
08	24	19N
10	21	18N
11	22	23
12	29	24
14	21	17N
08N K 05		
01	11	7N
02	44	39
03	35	21
04	25	11N
05	47	42N
06	21	12N
08	18	10N
09	22	15
10	28	13
12	9	9

K	$\|F_o\|$	F_c
14	16	11N
15	13	13
08N K 06		
00	39	32
01	28	19
03	16	11N
04	33	27N
05	13	10N
07	31	29
08	27	16
10	22	14
13	11	8
14	17	20N
08N K 08		
00	7	12N
01	17	11N
09N K 01		
00	13	4N
01	8	2
02	29	32
03	23	23
04	20	24N
05	17	14
06	15	12N
07	37	42N
08	22	21
09	21	19N
10	13	8
11	22	18
12	13	9N
09N K 02		
03	27	26
04	27	27N
05	45	46N
06	19	14
08	12	6
09	24	19
10	25	24N
11	24	17
12	24	17N
13	14	11N
14	16	12
16	8	5N
09N K 03		
00	39	44
01	32	29N
02	31	26N
03	22	16N
04	15	13
06	30	31
07	25	23
08	25	26N
10	9	3N
11	17	8N
12	22	14
15	16	16
09N K 04		
01	21	13N

K	$\|F_o\|$	F_c
03	47	40
04	39	34
05	7	10
06	28	25N
07	9	7N
09	22	19N
10	12	7
12	14	11
13	22	24
14	7	6N
09N K 05		
00	23	15N
01	39	29
02	27	16
03	31	20
04	19	9N
05	34	24N
06	17	11N
07	25	17N
08	24	19
11	17	12
12	17	13N
13	16	12
09N K 06		
01	49	43
02	16	12N
03	47	41N
11	18	14
09N K 07		
00	19	31N
10N K 01		
01	22	23N
03	31	36
04	28	25
05	27	24N
06	9	6N
09	22	16
10	15	13
11	20	18N
12	11	10
13	10	8
10N K 02		
00	15	14
01	40	35
02	12	8
03	32	32N
04	33	35N
05	15	8N
07	19	12
09	21	14
10	9	8
12	13	12
13	21	18
14	10	8N
10N K 03		
01	28	20
02	24	9N
03	31	20N

K	$\|F_o\|$	F_c
05	21	14
06	33	29
10N K 04		
00	45	46N
01	36	32N
02	12	7
04	39	36
10	11	8N
11	17	14
10N K 05		
01	18	14
02	46	38
03	16	11N
04	22	12N
06	28	25N
08	8	6N
10	19	14N
10N K 06		
00	32	31
01	12	2N
02	20	12N
04	16	12N
10	10	13
11N K 01		
00	10	5N
05	22	20N
06	21	17N
07	18	10
08	8	3
10	5	7N
11	17	18
11N K 02		
02	21	18
03	22	16N
04	23	19N
07	12	2N
09	13	7
10	11	8N
11N K 03		
00	31	22
01	35	31N
02	29	20N
03	24	19N
05	23	15
09	12	10
11N K 04		
01	16	10N
03	18	12
04	14	8N
07	11	11N
11N K 05		
01	21	15

K	$\|F_o\|$	F_c
03	16	13
05	19	17N
06	17	16
08	11	9N
09	8	10N
11N K 06		
01	9	10
02	18	18N
03	16	16N
04	21	23
06	12	10N
12N K 01		
02	27	28
03	18	15
04	21	16N
06	11	6N
08	7	6N
09	10	6
12N K 02		
00	25	16
04	21	18N
05	12	9N
06	15	14
08	9	4
12N K 03		
02	19	13N
05	16	3
12N K 04		
00	10	11N
01	9	5
04	17	20
06	10	6N
12N K 05		
03	10	9N
13N K 01		
00	13	12
01	19	30
02	13	14N
03	7	6
13N K 02		
01	11	9
03	10	15N

Using the structure factors from the ninth least-squares cycle, a two-by-two matrix was calculated on the IBM 602A machine for the refinement of scale and overall temperature factors with the F_o fixed. This resulted in a change of 3·5% in the scale factor in the opposite sense (reducing the F_c) to that calculated in the ninth 704 cycle, which reduced the F_o by 1·5% or, with the 'fudge' factor of 0·5 built into the programme, by 0·75%. The increase in the overall temperature factor was 0·09 Å^2. The effect of these changes on R and $R'/\Sigma wF_o^2$ was very small. Final values of observed and calculated structure factors are given in Table 2 and final atomic parameters together with standard deviations calculated from the least-squares matrix in Table 3.

An examination of the distribution (which is non-Gaussian) of the eighteen C–C bonds in the phenyl groups suggests a standard deviation of their bond lengths of 0·029 Å compared with a mean value of 0·019 Å calculated from the standard deviations of atomic positions. The most pessimistic view based on the absolute equality of these eighteen bonds, when the variance would be due entirely to random errors, leads to the hypothesis that all standard deviation values in Tables 3, 4, and 5 should be multiplied by 1·5. While there is no scientific justification for this, as the method of calculating standard deviations by the inversion of the least-squares matrix is the most pessimistic known, it may be pointed out that such an increase would in no way affect the discussion that follows.

Discussion of the structure

The molecule, which is asymmetric, exists in two enantiomorphic forms and the substance crystallizes as the racemate. Bond lengths and bond angles are listed in Tables 4 and 5. Fig. 1 shows the molecule which has two distinct parts. The first is centred on Fe_1, which is octahedrally coordinated to three carbonyl groups and to the three atoms C_1, C_4 and C_7 of the

Table 3. *Final atomic parameters with their standard deviations*

	x/a	y/b	z/b	B (Å²)	$\sigma(x)$ (Å²)	$\sigma(y)$ (Å²)	$\sigma(z)$ (Å²)	$\sigma(B)$ (Å²)
Fe_1	0·5410	0·3893	0·7315	2·96	0·002	0·002	0·002	0·04
Fe_2	0·3858	0·3243	0·8202	2·81	0·002	0·002	0·002	0·04
O_1	0·6923	0·2314	0·8467	4·17	0·009	0·008	0·008	0·16
O_2	0·2594	0·4159	0·9705	6·52	0·013	0·011	0·012	0·24
O_3	0·2590	0·2180	0·9307	6·17	0·012	0·011	0·011	0·23
O_4	0·4994	0·5236	0·8205	6·44	0·012	0·011	0·011	0·24
O_5	0·5693	0·4118	0·4536	6·29	0·012	0·011	0·011	0·24
O_6	0·7786	0·3967	0·8070	6·28	0·012	0·011	0·011	0·24
C_1	0·3781	0·3728	0·6545	3·28	0·013	0·010	0·011	0·19
C_2	0·3647	0·3066	0·6192	3·23	0·012	0·010	0·010	0·19
C_3	0·4507	0·2630	0·6662	3·08	0·012	0·010	0·010	0·18
C_4	0·5663	0·2897	0·6942	2·79	0·011	0·010	0·010	0·17
C_5	0·6069	0·2647	0·8242	2·71	0·011	0·009	0·010	0·17
C_6	0·5408	0·2897	0·9292	3·14	0·012	0·010	0·010	0·19
C_7	0·5221	0·3578	0·9130	3·52	0·013	0·011	0·011	0·20
C_8	0·3083	0·3811	0·9056	4·11	0·014	0·012	0·013	0·23
C_9	0·3083	0·2608	0·8860	4·38	0·015	0·012	0·013	0·24
C_{10}	0·5150	0·4722	0·7820	4·08	0·014	0·012	0·012	0·23
C_{11}	0·5586	0·4046	0·5627	4·10	0·014	0·012	0·013	0·23
C_{12}	0·6848	0·3958	0·7784	4·58	0·015	0·013	0·013	0·26
C_{13}	0·3086	0·4233	0·5852	3·61	0·013	0·011	0·011	0·21
C_{14}	0·2572	0·4754	0·6480	4·91	0·016	0·014	0·014	0·27
C_{15}	0·1872	0·5171	0·5775	5·74	0·018	0·015	0·016	0·31
C_{16}	0·1703	0·5088	0·4477	5·77	0·018	0·016	0·016	0·31
C_{17}	0·2154	0·4582	0·3818	6·08	0·018	0·016	0·016	0·33
C_{18}	0·2844	0·4148	0·4513	4·14	0·014	0·012	0·012	0·23
C_{19}	0·4305	0·1909	0·6515	2·97	0·012	0·010	0·010	0·18
C_{20}	0·3221	0·1653	0·6391	4·22	0·015	0·012	0·012	0·24
C_{21}	0·3105	0·0972	0·6120	4·46	0·015	0·013	0·013	0·25
C_{22}	0·3997	0·0576	0·6053	4·88	0·016	0·014	0·014	0·26
C_{23}	0·5074	0·0830	0·6202	4·54	0·015	0·013	0·013	0·25
C_{24}	0·5248	0·1477	0·6445	3·85	0·013	0·012	0·012	0·21
C_{25}	0·5323	0·2538	0·0519	3·09	0·012	0·010	0·011	0·19
C_{26}	0·5050	0·2867	0·1668	4·26	0·014	0·012	0·013	0·24
C_{27}	0·4970	0·2530	0·2793	5·54	0·018	0·014	0·016	0·30
C_{28}	0·5144	0·1876	0·2892	5·04	0·016	0·014	0·014	0·28
C_{29}	0·5404	0·1531	0·1768	4·69	0·015	0·013	0·014	0·26
C_{30}	0·5518	0·1885	0·0600	4·37	0·015	0·012	0·013	0·24

Table 4. *Interatomic distances*

Bond	Length	Standard deviation
Fe_1–Fe_2	2·501 Å	0·003 Å
Fe_1–C_{10}	1·806	0·012
Fe_1–C_{11}	1·796	0·013
Fe_1–C_{12}	1·764	0·016
Fe_2–C_8	1·756	0·013
Fe_2–C_9	1·754	0·013
Fe_1–C_1	2·088	0·013
Fe_1–C_4	2·097	0·010
Fe_1–C_7	2·006	0·011
Fe_2–C_1	1·975	0·011
Fe_2–C_2	2·108	0·011
Fe_2–C_3	2·203	0·011
Fe_2–C_6	2·227	0·012
Fe_2–C_7	1·965	0·013
C_8–O_2	1·159	0·017
C_9–O_3	1·167	0·017
C_{10}–O_4	1·142	0·016
C_{11}–O_5	1·151	0·017
C_{12}–O_6	1·144	0·020
C_1–C_2	1·407	0·015
C_2–C_3	1·425	0·016
C_3–C_4	1·499	0·016
C_4–C_5	1·490	0·014
C_5–O_1	1·238	0·014
C_5–C_6	1·471	0·014
C_6–C_7	1·419	0·015
C_1–C_{13}	1·483	0·017
C_{13}–C_{14}	1·408	0·018
C_{14}–C_{15}	1·373	0·022
C_{15}–C_{16}	1·354	0·023
C_{16}–C_{17}	1·367	0·023
C_{17}–C_{18}	1·382	0·021
C_{18}–C_{13}	1·406	0·017
C_3–C_{19}	1·499	0·014
C_{19}–C_{20}	1·398	0·019
C_{20}–C_{21}	1·424	0·018
C_{21}–C_{22}	1·344	0·021
C_{22}–C_{23}	1·389	0·022
C_{23}–C_{24}	1·359	0·018
C_{24}–C_{19}	1·438	0·017
C_6–C_{25}	1·473	0·015
C_{25}–C_{26}	1·419	0·016
C_{26}–C_{27}	1·359	0·020
C_{27}–C_{28}	1·356	0·020
C_{28}–C_{29}	1·408	0·019
C_{29}–C_{30}	1·420	0·019
C_{30}–C_{25}	1·357	0·016
Non-bonded approaches		
Fe_2–C_4	2·682	0·011
Fe_2–C_5	2·910	0·011
C_1–C_7	3·105	0·017
C_2–C_6	3·743	0·016
C_3–C_5	2·407	0·016

Table 5. *Bond angles*

	Angle	Standard deviation
C_1–Fe_1–C_4	85·1°	0·4
C_1–Fe_1–C_7	98·6	0·5
C_4–Fe_1–C_7	83·4	0·4
C_1–Fe_1–C_{10}	95·1	0·5
C_1–Fe_1–C_{11}	79·6	0·5
C_1–Fe_1–C_{12}	171·8	0·6
C_4–Fe_1–C_{10}	173·3	0·5
C_4–Fe_1–C_{11}	88·0	0·5
C_4–Fe_1–C_{12}	88·5	0·5
C_7–Fe_1–C_{10}	90·0	0·5
C_7–Fe_1–C_{11}	171·4	0·6
C_7–Fe_1–C_{12}	86·0	0·6
C_{10}–Fe_1–C_{11}	98·6	0·6
C_{10}–Fe_1–C_{12}	91·9	0·6
C_{11}–Fe_1–C_{12}	94·8	0·6
C_8–Fe_2–C_9	89·3	0·6
C_1–Fe_2–C_8	96·0	0·5
C_2–Fe_2–C_8	124·9	0·5
C_3–Fe_2–C_8	163·4	0·5
C_6–Fe_2–C_8	113·9	0·5
C_7–Fe_2–C_8	88·4	0·6
C_1–Fe_2–C_9	135·0	0·6
C_2–Fe_2–C_9	102·7	0·5
C_3–Fe_2–C_9	94·3	0·5
C_6–Fe_2–C_9	90·7	0·5
C_7–Fe_2–C_9	120·8	0·6
C_1–Fe_2–C_7	104·0	0·5
Fe_1–C_{10}–O_4	176·0	1·2
Fe_1–C_{11}–O_5	176·8	1·2
Fe_1–C_{12}–O_6	176·4	1·3
Fe_2–C_8–O_2	174·4	1·2
Fe_2–C_9–O_3	180·0	1·2
Fe_1–C_1–Fe_2	76·0	0·4
Fe_1–C_7–Fe_2	78·0	0·4
Fe_1–Fe_2–C_1	54·1	0·3
Fe_1–Fe_2–C_7	51·7	0·3
Fe_2–Fe_1–C_1	50·0	0·3
Fe_2–Fe_1–C_7	50·3	0·3
Fe_1–C_1–C_2	110·1	0·8
C_1–C_2–C_3	116·4	1·0
C_2–C_3–C_4	118·5	0·9
C_3–C_4–Fe_1	104·3	0·7
Fe_1–C_4–C_5	102·2	0·7
C_3–C_4–C_5	107·2°	0·9
C_4–C_5–O_1	125·4	1·0
O_1–C_5–C_6	121·7	1·0
C_4–C_5–C_6	112·6	0·9
C_5–C_6–C_7	109·0	0·9
C_6–C_7–Fe_1	113·5	0·8
Fe_2–C_1–C_2	75·0	0·7
Fe_2–C_2–C_1	64·8	0·6
Fe_2–C_2–C_3	74·3	0·6
Fe_2–C_3–C_2	67·1	0·6
Fe_2–C_6–C_7	60·5	0·6
Fe_2–C_7–C_6	80·6	0·7
Fe_1–C_1–C_{13}	123·7	0·8
C_2–C_1–C_{13}	119·5	1·0
C_1–C_{13}–C_{14}	123·6	1·1
C_1–C_{13}–C_{18}	117·8	1·1
C_{13}–C_{14}–C_{15}	119·6	1·3
C_{14}–C_{15}–C_{16}	119·9	1·5
C_{15}–C_{16}–C_{17}	123·2	1·6
C_{16}–C_{17}–C_{18}	117·9	1·5
C_{17}–C_{18}–C_{13}	120·9	1·2
C_{18}–C_{13}–C_{14}	118·4	1·1
C_2–C_3–C_{19}	118·2	0·9
C_4–C_3–C_{19}	121·3	0·9
C_3–C_{19}–C_{20}	121·4	1·0
C_3–C_{19}–C_{24}	119·2	1·0
C_{19}–C_{20}–C_{21}	117·6	1·2
C_{20}–C_{21}–C_{22}	122·0	1·3
C_{21}–C_{22}–C_{23}	120·2	1·3
C_{22}–C_{23}–C_{24}	121·0	1·3
C_{23}–C_{24}–C_{19}	119·7	1·1
C_{24}–C_{19}–C_{20}	119·4	1·0
C_5–C_6–C_{25}	121·9	0·9
C_7–C_6–C_{25}	124·9	1·0
C_6–C_{25}–C_{26}	121·0	1·0
C_6–C_{25}–C_{30}	121·5	1·0
C_{25}–C_{26}–C_{27}	120·4	1·2
C_{26}–C_{27}–C_{28}	123·1	1·4
C_{27}–C_{28}–C_{29}	118·2	1·4
C_{28}–C_{29}–C_{30}	118·8	1·3
C_{29}–C_{30}–C_{25}	121·9	1·2
C_{30}–C_{25}–C_{26}	117·5	1·1

organic system. The second consists of Fe_2 with two carbonyl groups perpendicular to one another. The organic system forms with Fe_1 two five-membered rings having the bond Fe_1–C_4 in common. It is shown in detail in Fig. 2 in which the organic bond lengths are also indicated.

The lengths of the Fe–C and C–O bonds in the iron carbonyl groups are all normal. The bonds Fe_2–C_1 and Fe_2–C_7 are shorter (1·97 Å as against 2·12 Å) than the corresponding bonds in the but-2-yne complex of iron carbonyl hydride, $Fe_2C_{12}O_8H_8$ (Hock & Mills, 1961). A possible explanation is that the bonds Fe_2–C_1, Fe_2–C_7, Fe_1–C_1 and Fe_1–C_7 must be either distorted or shortened in order that the iron–iron distance shall be normal. The angle C_1–Fe_1–C_7 is substantially greater than the octahedral angle, the π-bonds Fe_2–C_1 and Fe_2–C_7 are shorter than those in $Fe_2C_{12}O_8H_8$ and Fe_1–C_7 is shorter than the other Fe–C σ-bonds. A reduction in the length of Fe_1–C_1 may be prevented by steric interaction between the phenyl group attached to C_1 and the carbonyl group C_{10}–O_4. No explanation can be offered for the fact that the iron–carbon σ-bonds are significantly ($4\frac{1}{2}$ standard deviations for Fe_1–C_7 and 9 standard deviations for Fe_1–C_1 and Fe_1–C_4) longer than in $Fe_2C_{12}O_8H_8$.

C_3–C_4 and C_4–C_5 do not differ substantially from single bonds while C_5–C_6 is slightly shorter. The three bonds C_1–C_2, C_2–C_3 and C_6–C_7 are all much shorter and of the same order as those found in $Fe_2C_{12}O_8H_8$ and in ferrocene (Dunitz, Orgel & Rich, 1956) where there are π-bonds involving iron. The shortest intermolecular contacts are between phenyl and carbonyl groups and are all more than 3·5 Å.

These results suggest that the two parts of the molecule are linked by a π-bond between C_6–C_7 and Fe_2, by a three-electron donor system from C_1–C_2–C_3 to Fe_2 and by an iron–iron bond. The system C_1–C_2–C_3 resembles the allyl group in allylpalladium chloride (Dehm & Chien, 1960) and the group CHMe–CH–CH_2 in butadiene cobalt hydrocarbonyl (Moore, Jonassen,

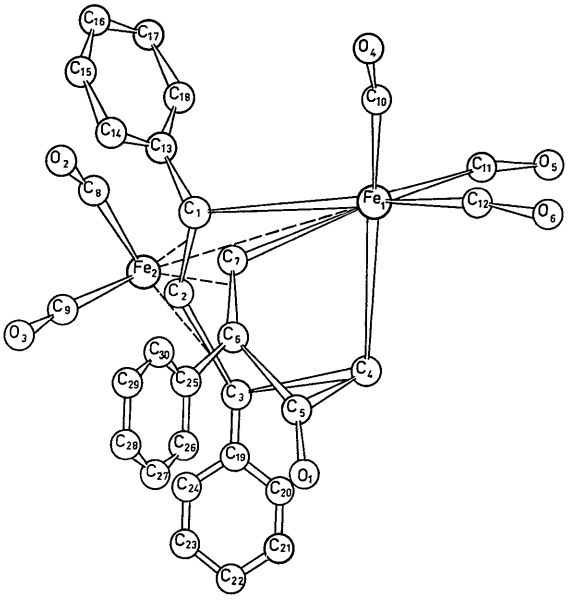

Fig. 1. $Fe_2(CO)_6(C_6H_5C_2H)_3$.

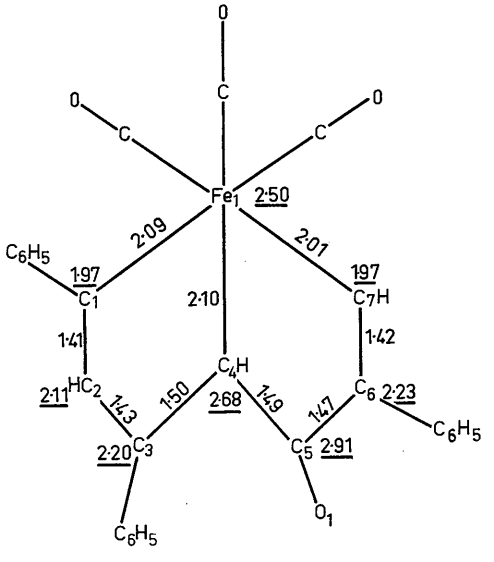

Fig. 2. Bond lengths in $Fe_2(CO)_6(C_6H_5C_2H)_3$. Values underlined are distances from Fe_2.

Joyner & Bertrand, 1960) in which nuclear magnetic resonance measurements have shown the allyl carbon–carbon bonds to be equivalent.

The same system exists in cyclopentenylcyclopentadienylnickel(II) (Fischer & Werner, 1961; Jones, Parshall, Pratt & Wilkinson, 1961; Shaw & Sheppard, 1961) where the cyclopentenyl ring corresponds to the ring C_1–C_2–C_3–C_4–Fe_1 in the present structure.

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