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THE MOLECULAR STRUCTURE OF A PRODUCT OF THE REACTION OF BICYCLO [6.2.0] DECA-2,4,6-TRIENE WITH DIIRON NONACARBONYL*

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SUMMARY

The reaction of bicyclo[6.2.0]deca-2,4,6-triene with diiron nonacarbonyl affords at least four products, one of which is a yellow substance with the empirical formula $C_{10}H_{12}Fe_2(CO)_6$. The structure of this substance has been determined by X-ray crystallography. The organic portion consists of a 1,3-cyclohexadiene ring with a CH_2 group at the 5 position and an allyl group at the 6 position. One $Fe(CO)_3$ group is bound to the 1,3-diene part of the organic group and another $Fe(CO)_3$ group is bound to the CH_2 group by a C–Fe σ bond and to the allyl group functioning as a *trihapto* allyl ligand. The essential crystallographic data are as follows: The space group is $P\bar{1}$ with $a=7.140(3)$, $b=10.029(6)$, $c=13.875(5)$ Å, $\alpha=110.09(4)°$, $\beta=118.50(3)°$, $\gamma=77.65(4)°$, $V=818.5(7)$ Å^3 and $Z=2$. The structure was solved by Patterson heavy atom methods. With all atoms located the conventional R value for 1210 observed reflections measured with a Syntex $P\bar{1}$ diffractometer is 0.044.

INTRODUCTION

The reaction of bicyclo[6.2.0]deca-2,4,6-triene, I, with diiron nonacarbonyl has been shown to produce at least four products[1,***], as shown in Scheme 1. Products II and III can reasonably be expected[1,3] and their structures have been firmly established by spectroscopic[1,4] and X-ray crystallographic investigations[5,6]. The structures of compounds IV (an isomer of II) and V could not be inferred from indirect (*i.e.*, chemical and spectroscopic) evidence, although some very tentative suggestions were put forward for the structure of IV.

We have now established, conclusively, the structure of IV by X-ray crystallography. The structure found was entirely unanticipated, and raises interesting questions regarding its mode of formation.

EXPERIMENTAL

Collection of X-ray data. Compound IV was prepared as a yellow micro-

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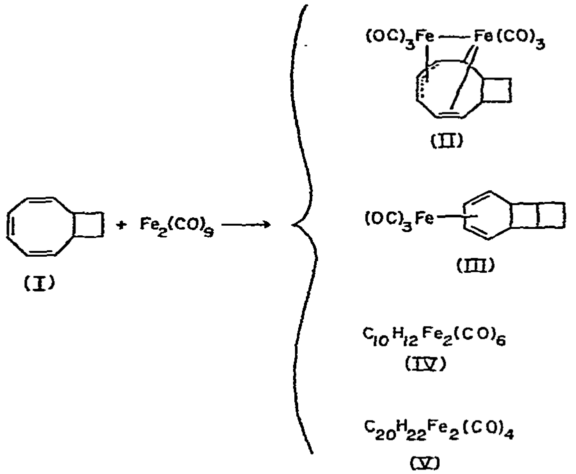
[***] Cotton and Troup have subsequently isolated three more products[2].

SCHEME 1



crystalline solid by Deganello using a procedure previously described[1]. However, duplication of the synthesis gave much smaller yields than the original preparation. Numerous attempts at recrystallization employing a large variety of solvents yielded only twinned crystals. Examination of these crystals on the diffractometer showed doubly-peaked ω scans for intense reflections and a unit cell volume requiring eight formula units per asymmetric unit. A small fraction of the crystals obtained from recrystallization using carbon disulfide were found to be single; the crystal morphology of the single crystals was the same as that of the twinned crystals. A platelet measuring approximately 0.24 × 0.17 × 0.05 mm was selected for data collection and was mounted in a glass capillary to protect it from slow air decomposition.

Preliminary precession and diffractometer work revealed the crystal to be triclinic and a Delauney reduction did not indicate higher symmetry. The space group $P\bar{1}$ was assumed; this choice was later confirmed by the successful solution and refinement of the structure.

The unit cell dimensions at 22° and the orientation matrix for data collection were determined by least-squares refinement of the setting angles for fifteen carefully centered reflections. These dimensions and other crystal data are given in Table 1. The reduced cell parameters are: $a = 10.029$, $b = 12.205$ $c = 7.140$ Å, $\alpha = 92.44°$, $\beta = 102.35°$, and $\gamma = 105.39°$. These are obtained from the cell parameters used in data collection by means of the following transformation matrix:

$$\begin{pmatrix} 0 & -1 & 0 \\ -1 & 0 & -1 \\ 1 & 0 & 0 \end{pmatrix}$$

A width at half-height of 0.20° was obtained from ω scans of the crystal. The data were collected using a Syntex $P\bar{1}$ diffractometer and Mo-$K\alpha$ radiation. The incident beam was monochromatized with a graphite crystal. The operation of the diffractometer has been described previously[5]. Intensity measurements were made at 22° using the θ–2θ scan technique and a scan rate that varied from 2.0 to 24.0°/min

TABLE 1

CRYSTAL DATA FOR $(C_{10}H_{12})Fe_2(CO)_6$

$C_{16}H_{12}Fe_2O_6$	Triclinic
Mol. wt. 411.97	Space group $P\bar{1}$
$a=7.140(3)$ Å	$F(000)=416$
$b=10.029(6)$	Mo-$K\alpha$ radiation
$c=13.875(5)$	$\mu=19.0\ cm^{-1}$
$\alpha=110.09(4)°$	$Z=2$
$\beta=118.50(3)$	$\rho_{calc}=1.67\ g\cdot cm^{-3}$
$\gamma=77.65(4)$	$\rho_{obs}=1.65(2)\ g\cdot cm^{-3}$ (by flotation)
$V=818.5(7)$ Å^3	

depending on the intensity of the reflection. Unique data with 2θ(Mo-$K\alpha$) values up to 45° were collected with a scan range from 2θ(Mo-$K\alpha_1$) $-0.8°$ to 2θ(Mo-$K\alpha_2$) $+0.8°$. Three standard reflections, measured every 100 reflections, showed no significant change in intensity throughout the data collection. After carefully measuring the distances between the crystal faces (forms $\{01\bar{1}\}$, $\{101\}$, $\{1\bar{1}\bar{1}\}$) a numerical absorption correction was applied to the data and yielded transmission factors varying from 0.710 to 0.915. Lorentz and polarization corrections were made; no extinction correction was deemed necessary. Of the 1893 reflections collected, only the 1210 reflections having $F_o^2 > 3\,\sigma(F_o^2)$ were used in refinement of the structure.

Solution and refinement of the structure. The iron atoms were located using a Patterson function. Two cycles of least-squares refinement of their coordinates gave the following agreement factors: $R_1=(\Sigma|\,|F_o|-|F_c|\,|)/\Sigma|F_o|=$ 0.33 and $R_2=(\Sigma w(|F_o|-|F_c|)^2)/\Sigma w|F_o|^2)^{1/2}=0.37$, where $|F_o|$ and $|F_c|$ are the observed and calculated structure factor amplitudes and w is the weighting factor, given by $4|F_o|^2/\sigma^2$. Here σ is the e.s.d. of $|F_o|^2$. Scattering factors were taken from the International Tables[7]. Anomalous dispersion effects were included in the calculated structure factors for iron, using $\Delta f'$ and $\Delta f''$ values given by Cromer and Liberman[8].

A difference electron density map then revealed the positions of all carbon and oxygen atoms, and two cycles of least-squares refinement with isotropic thermal parameters lead to agreement indices of $R_1=0.105$ and $R_2=0.113$. One cycle of refinement with anisotropic temperature parameters gave $R_1=0.047$ and $R_2=0.052$.

A difference electron density map now showed the positions of nine hydrogen atoms. The positions expected for the other three were calculated and refinement was resumed, assigning a fixed isotropic temperature parameter of 4.0 Å^2 to each hydrogen atom.

Refinement converged with no parameter shift exceeding 0.5 times the standard deviation in the parameter, giving final agreement indices of $R_1=0.044$ and $R_2=0.049$. The error in an observation of unit weight was 1.22[*].

No systematic trends were found in the data as a function of $\lambda^{-1}\sin\theta$, $|F_o|$, Miller indices, or reflection number. A final difference Fourier function contained no

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[*] The Table of structure factors has been deposited as NAPS Document No. 02228, with the ASIS National Auxiliary Publication Service, c/o Microfiche Publications, 305E, 46th St., New York, New York 10017. A copy may be secured by citing the document number and by remitting $1.50 for a microfiche or $5.00 for photocopies. Advance payment is required. Make checks or money orders payble to: Microfiche Publications.

TABLE 2

ATOMIC POSITIONAL AND ANISOTROPIC THERMAL PARAMETERS FOR NON-HYDROGEN ATOM

Atom	*x*	*y*	*z*	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Fe(1)	0.1953(2)	0.7757(2)	0.9036(1)	0.0246(5)	0.0136(3)	0.0077(2)	−0.0022(3)	0.0039(3)	0.0031
Fe(2)	−0.1544(2)	0.2443(2)	0.6062(1)	0.0263(6)	0.0132(3)	0.0073(2)	−0.0022(3)	0.0044(3)	0.0030
O(1)	0.249(2)	1.033(1)	0.8686(8)	0.054(4)	0.019(2)	0.015(1)	−0.007(2)	0.005(2)	0.008(
O(2)	0.604(1)	0.617(1)	0.9382(7)	0.028(3)	0.023(2)	0.014(1)	0.004(2)	0.008(1)	0.007(
O(3)	0.264(2)	0.914(1)	1.1408(8)	0.058(4)	0.026(2)	0.012(1)	0.002(2)	0.010(2)	0.005(
O(4)	−0.265(2)	−0.024(1)	0.6010(7)	0.073(5)	0.022(2)	0.010(1)	−0.015(2)	0.006(2)	0.005(
O(5)	0.284(2)	0.140(1)	0.6466(9)	0.035(3)	0.026(2)	0.018(1)	0.007(2)	0.012(2)	0.005(
O(6)	−0.381(1)	0.199(1)	0.3576(7)	0.044(3)	0.030(2)	0.008(1)	−0.008(2)	0.001(1)	0.006(
C(1)	0.053(2)	0.606(1)	0.8933(9)	0.034(4)	0.010(2)	0.006(1)	−0.005(2)	0.006(2)	0.003(
C(2)	−0.100(2)	0.711(1)	0.8498(10)	0.025(4)	0.012(2)	0.009(1)	−0.002(2)	0.007(2)	0.002(1
C(3)	−0.091(2)	0.737(1)	0.7616(11)	0.024(4)	0.013(2)	0.010(1)	−0.002(2)	0.002(2)	0.003(1
C(4)	0.067(2)	0.660(1)	0.7283(10)	0.033(4)	0.016(2)	0.006(1)	−0.007(2)	0.005(2)	0.002(1
C(5)	0.107(2)	0.502(1)	0.7169(9)	0.022(3)	0.015(2)	0.007(1)	−0.006(2)	0.003(2)	0.001(1
C(6)	0.104(2)	0.472(1)	0.8159(9)	0.025(4)	0.016(2)	0.006(1)	−0.003(2)	0.004(2)	0.004(1
C(7)	−0.039(2)	0.356(1)	0.7826(10)	0.039(5)	0.016(2)	0.008(1)	−0.001(3)	0.006(2)	0.005(1
C(8)	−0.256(2)	0.348(1)	0.7309(12)	0.035(5)	0.021(2)	0.012(2)	−0.004(3)	0.008(2)	0.006(2
C(9)	−0.390(3)	0.399(2)	0.6410(15)	0.040(5)	0.022(3)	0.016(2)	0.000(3)	0.016(3)	0.007(2
C(10)	−0.050(2)	0.419(1)	0.5975(10)	0.025(4)	0.014(2)	0.009(1)	−0.001(2)	0.006(2)	0.004(1
C(11)	0.229(2)	0.933(1)	0.8838(11)	0.036(4)	0.013(2)	0.012(1)	−0.001(2)	0.005(2)	0.005(1
C(12)	0.448(2)	0.683(1)	0.9272(9)	0.039(5)	0.019(2)	0.006(1)	−0.002(3)	0.008(2)	0.002(1
C(13)	0.234(2)	0.861(1)	1.0454(12)	0.033(4)	0.017(2)	0.010(1)	−0.001(2)	0.007(2)	0.005(1
C(14)	−0.288(2)	0.083(1)	0.6041(10)	0.048(5)	0.015(2)	0.007(1)	−0.007(3)	0.007(2)	0.002(1
C(15)	0.111(2)	0.176(1)	0.6305(10)	0.040(5)	0.018(2)	0.009(1)	0.002(3)	0.012(2)	0.002(1
C(16)	−0.297(2)	0.214(1)	0.4529(12)	0.028(4)	0.019(2)	0.012(1)	−0.005(2)	0.007(2)	0.005(2

[a] Numbers in parentheses are estimated standard deviations in the least significant digits in this and all other tabl The anisotropic temperature parameters are of the form $\exp[-(\beta_{11}h^2+\beta_{22}k^2+\beta_{33}l^2+2\beta_{12}hk+2\beta_{13}hl+2\beta_{23}kl)$

TABLE 3

ATOMIC POSITIONAL PARAMETERS FOR HYDROGEN ATOMS

Atom[a]	*x*	*y*	*z*
H(1)	0.07(1)	0.590(8)	0.971(8)
H(2)	−0.19(1)	0.790(9)	0.900(7)
H(3)	−0.17(1)	0.816(9)	0.727(7)
H(4)	0.09(2)	0.680(10)	0.679(8)
H(5)	0.24(1)	0.485(9)	0.717(7)
H(6)	0.22(1)	0.444(10)	0.853(8)
H(7)	0.04(1)	0.301(9)	0.830(8)
H(8)	−0.34(1)	0.287(9)	0.744(7)
H(9,1)	−0.52(2)	0.356(9)	0.610(8)
H(9,2)	−0.36(2)	0.460(10)	0.624(8)
H(10,1)	−0.17(1)	0.484(9)	0.570(8)
H(10,2)	0.04(1)	0.382(8)	0.548(7)

[a] Numbering of hydrogen atoms refers to the carbons to which they are attached. The second numbers distinguish between the two attached to C(9) and C(10).

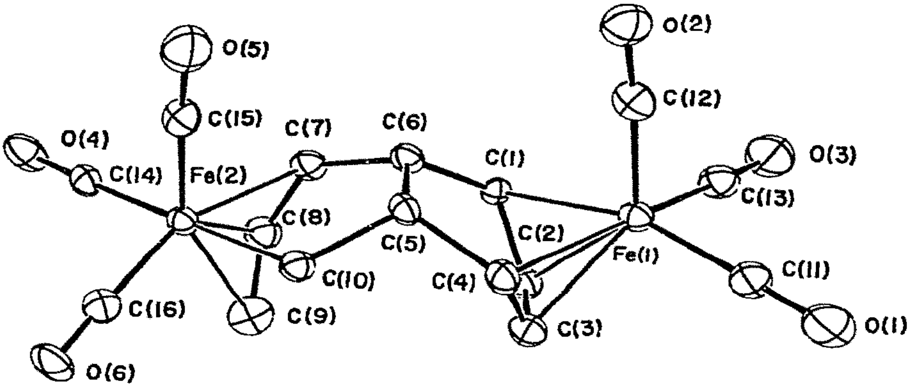


Fig. 1. A view of the structure emphasizing the geometrical similarity of the two chemically dissimilar halves of the molecule.

TABLE 4

BOND LENGTHS (Å)

Fe(1)–C(1)	2.10(1)	C(1)–C(2)	1.41(1)
Fe(1)–C(2)	2.04(1)	C(1)–C(6)	1.50(2)
Fe(1)–C(3)	2.04(1)	C(2)–C(3)	1.37(2)
Fe(1)–C(4)	2.12(1)	C(3)–C(4)	1.40(2)
Fe(1)–C(11)	1.77(1)	C(4)–C(5)	1.52(2)
Fe(1)–C(12)	1.78(2)	C(5)–C(6)	1.52(2)
Fe(1)–C(13)	1.76(2)	C(5)–C(10)	1.54(1)
		C(6)–C(7)	1.52(2)
Fe(2)–C(7)	2.12(1)	C(7)–C(8)	1.37(2)
Fe(2)–C(8)	2.06(1)	C(8)–C(9)	1.35(2)
Fe(2)–C(9)	2.14(2)		
Fe(2)–C(10)	2.10(1)	C(1)–H(1)	1.08(9)
Fe(2)–C(14)	1.80(1)	C(2)–H(2)	1.18(9)
Fe(2)–C(15)	1.78(2)	C(3)–H(3)	0.99(8)
Fe(2)–C(16)	1.81(1)	C(4)–H(4)	0.88(8)
		C(5)–H(5)	0.93(9)
C(11)–O(1)	1.14(1)	C(6)–H(6)	0.78(9)
C(12)–O(2)	1.14(1)	C(7)–H(7)	0.90(9)
C(13)–O(3)	1.17(1)	C(8)–H(8)	1.07(8)
C(14)–O(4)	1.14(1)	C(9)–H(9,1)	0.94(8)
C(15)–O(5)	1.14(1)	C(9)–H(9,2)	0.81(8)
C(16)–O(6)	1.13(1)	C(10)–H(10,1)	0.99(9)
		C(10)–H(10,2)	1.08(8)

peaks greater than 0.33 $e\text{Å}^{-3}$, whereas hydrogen atoms were located on a previous map at 0.38 $e\text{Å}^{-3}$. The atomic coordinates, anisotropic thermal parameters and estimated standard deviations for non-hydrogen atoms are given in Table 2. The hydrogen atom coordinates and estimated standard deviations are given in Table 3.

RESULTS

The structure is shown in Figs. 1 and 2. Tables 4 and 5 list the bond lengths

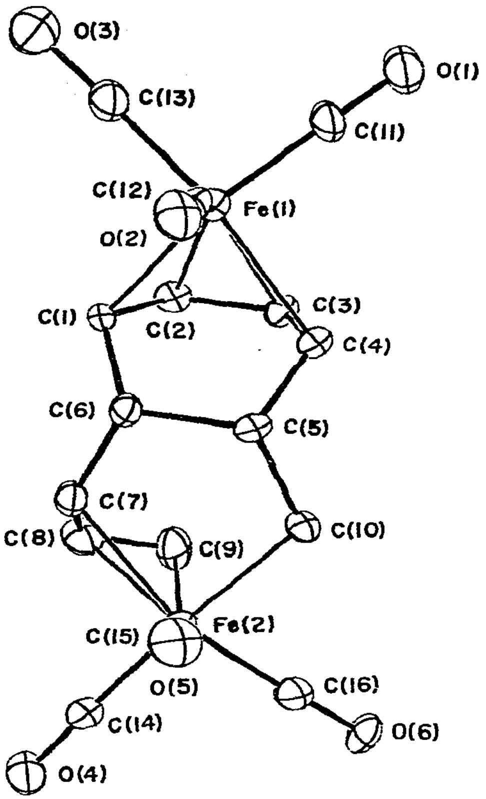


Fig. 2. Another view of the structure from a direction nearly perpendicular to that in Fig. 1.

and bond angles, respectively, with their estimated standard deviations. Table 6 gives the equations for best (least-squares) planes through several sets of atoms, the deviations of individual atoms from these planes, and the dihedral angles between pairs of planes.

The structure consists of discrete molecules, with no abnormal intermolecular contacts. The $C_{10}H_{12}$ component of the structure can be described as a 1,3-cyclohexadiene ring with a CH_2 group attached to the 5 position and an allyl group attached to the 6 position. The crystallographic numbering scheme was made identical to this chemical numbering scheme. One $Fe(CO)_3$ group is bonded to the 1,3-butadiene moiety, while the other is attached to the allyl group in the usual *trihapto* fashion and to the CH_2 group by a C–Fe single bond.

DISCUSSION

The actual structure as found in the crystal is quite consistent with all of the previously reported[1] spectral data, although it was not possible to deduce the correct structure from such data. It is interesting that the incorrect structure VI suggested by Cotton and Deganello as the most probable of those then considered on the spectral evidence does in fact contain exactly the same set of major structural components as the correct one IV (a butadiene-$Fe(CO)_3$ group, an allyl-$Fe(CO)_3$ group and a C–Fe σ bond) but assembled in a different way. In actuality the original ligand system I has rearranged and undergone a C–C bond cleavage so as to form a structure basically different from anything previously envisioned.

TABLE 5

SELECTED BOND ANGLES (Deg.)

C(1)–Fe(1)–C(2)	39.8(4)	C(7)–Fe(2)–C(8)	38.2(5)
C(2)–Fe(1)–C(3)	39.2(4)	C(8)–Fe(2)–C(9)	37.3(5)
C(3)–Fe(1)–C(4)	39.2(5)	C(9)–Fe(2)–C(10)	83.3(5)
		C(7)–Fe(2)–C(10)	83.5(4)
C(11)–Fe(1)–C(12)	101.7(6)	C(14)–Fe(2)–C(15)	93.3(6)
C(11)–Fe(1)–C(13)	92.9(6)	C(15)–Fe(2)–C(16)	104.3(5)
C(12)–Fe(1)–C(13)	100.8(6)	C(14–Fe(2)–C(16)	94.6(5)
Fe(1)–C(11)–O(1)	178.6(12)	Fe(2)–C(14)–O(4)	175.7(12)
Fe(1)–C(12)–O(2)	176.3(11)	Fe(2)–C(15)–O(5)	175.8(12)
Fe(1)–C(13)–O(3)	177.5(12)	Fe(2)–C(16)–O(6)	177.0(10)
C(1)–C(2)–C(3)	113.8(11)	C(5)–C(6)–C(7)	115.2(9)
C(2)–C(3)–C(4)	117.4(11)	C(6)–C(7)–C(8)	132.7(12)
C(3)–C(4)–C(5)	119.4(10)	C(7)–C(8)–C(9)	123.9(14)
C(4)–C(5)–C(6)	110.0(10)	C(10)–C(5)–C(6)	114.8(8)
C(5)–C(6)–C(1)	111.3(10)	C(1)–C(6)–C(7)	110.6(9)
C(6)–C(1)–C(2)	121.7(10)	C(4)–C(5)–C(10)	109.6(9)

TABLE 6

LEAST-SQUARES PLANES AND DIHEDRAL ANGLES

Plane	*Atoms defining plane*	*Equation of mean plane*[a]
I	C(1)–C(2)–C(3)–C(4)	$3.698x+6.439y+2.431z=6.2685$
II	C(1)–C(4)–C(5)–C(6)	$5.968x+1.574y+0.963z=2.1351$
III	C(5)–C(6)–C(7)–C(10)	$-4.858x+5.721y+2.093z=3.8761$
IV	C(7)–C(8)–C(9)	$-1.737x+6.757y+6.359z=7.4468$
V	Fe(2)–C(15)–C(16)	$2.934x+9.758y-4.653z=0.8900$

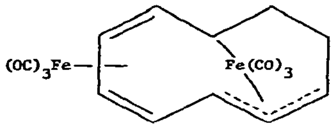
Displacement of atoms from mean plane (Å)

Plane I	*Plane II*	*Plane III*
C(1) −0.003	C(1) 0.008	C(5) −0.026
C(2) 0.006	C(4) 0.008	C(6) 0.027
C(3) −0.006	C(5) 0.013	C(7) −0.014
C(4) 0.003	C(6) 0.013	C(10) 0.014

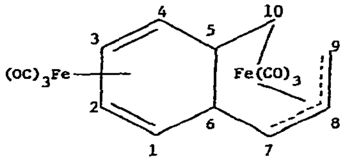
Dihedral angles between planes (Deg.)

Planes	*Planes*	*Planes*
I–II 38.5	II–III 125.5	III–IV 44.3
I–III 87.0	II–IV 83.0	IV–V 50.5
I–IV 46.4		

[a] Equations have the form $Ax+By+Cz=D$ where x, y, and z are fractional triclinic coordinates.



(VI)



(IV)

The infrared spectrum in the CO stretching region consists of six terminal CO stretching bands, having the appearance of being due to two separate Fe(CO)$_3$ groups that are similar, but not equivalent. This is exactly the spectrum that would be expected from the structure. In addition, the proton NMR spectrum can be satisfactorily, though not uniquely assigned. Using the τ values previously reported[1] for CS$_2$ solution and the numbering scheme for hydrogen atoms given in Table 3, the following assignments are very plausible: τ 4.60(2H) (H(2), H(3)); τ 5.61(3H) (H(7), H(8), H(9.1) or H(9,2)); τ 6.38(2H) (H(1), H(4)); τ 6.78(1H) (H(9,1) or H(9,2)); τ 7.15(2H) (H(5), H(6)); τ 8.66(1H) (H(10,1) or H(10,2)) and τ 9.50(1H) (H(10,1) or H(10,2)).

The molecule has approximate *mm* (C_{2v}) symmetry, as can be seen in Fig. 2, with one mirror plane containing Fe(1) and Fe(2) and bisecting the C(5)–C(6) bond, while the other plane contains C(5) and C(6) and bisects a line joining Fe(1) to Fe(2). Of course, no such symmetry actually exists since there is no bond (distance 2.83 Å) between atoms C(9) and C(10), each of which has two hydrogen atoms attached to it. Another violation of *mm* symmetry results from a slight twisting of the allyl group.

The irontricarbonyl–butadiene moiety has the typical shape and dimensions of such groups[9]. The two "outer" C–C bonds of the butadiene appear to be longer (1.399(16)Å and 1.409(14)Å) than the "inner" bond (1.370(15)Å), but the difference of 0.03(2)Å is of doubtful significance.

The C(5)–C(10) distance of 1.542(14)Å is appropriate for a C(sp^3)–C(sp^3) bond, and that from C(6) to C(7), 1.518(15)Å, is slightly shorter as expected for a C(sp^2)–C(sp^3) bond. The C(4)–C(5) and C(1)–C(6) bonds also have lengths of 1.510(16)Å, (av) which are similar to those of comparable bonds in other molecules[6,9].

TABLE 7

A COMPARISON OF BOND DISTANCES (Å) AND ANGLES (Deg.) FOR SEVERAL RELATED COMPLEXES

Compound	*M–C distance for η^3-allyl*			*C–C distances for η^3-allyl*	*C–C–C angle for η^3-allyl*	*M–C distance for σ-carbon*	*Ref.*
	(outer)	*(inter)*	*(outer)*				
$C_{10}H_{12}[Fe(CO)_3]_2$	2.123(12)	2.060(14)	2.144(15)	1.369(16), 1.346(18)	123.9(14)	2.104(11)	[a]
$C_9H_{12}[Fe(CO)_3]_2$	2.19(3)	2.13(3)	2.20(3)	1.48(4), 1.45(4)		2.12(2)	14
$(C_3H_5)Fe(CO)_3I$	2.26	2.09	2.34	1.35, 1.43	131		15
$(C_{13}H_8N_4)Fe(CO)_3$	2.13(4)	2.11(2)	2.09(2)	1.40(4), 1.49(4)	115(2)	2.09(3)	16
$(C_{12}H_{16})Fe_2(CO)_5$	2.117(8)	2.080(7)	2.173(8)	1.42(1), 1.41(1)	127.6(6)		17
	2.163(7)	2.094(8)	2.159(8)	1.42(1), 1.41(1)	116.6(7)		
$(C_{10}H_{12})Fe_2(CO)_6$	2.120(4)	2.042(4)	2.140(4)	1.403(6), 1.399(6)	122.6(4)	2.099(4)	5
$(C_8H_{11}CN)Ru(CO)_3$	2.252(10)	2.210(8)	2.284(8)	1.41(2), 1.41(2)	123.6(10)	2.161(9)	18

[a] This work.

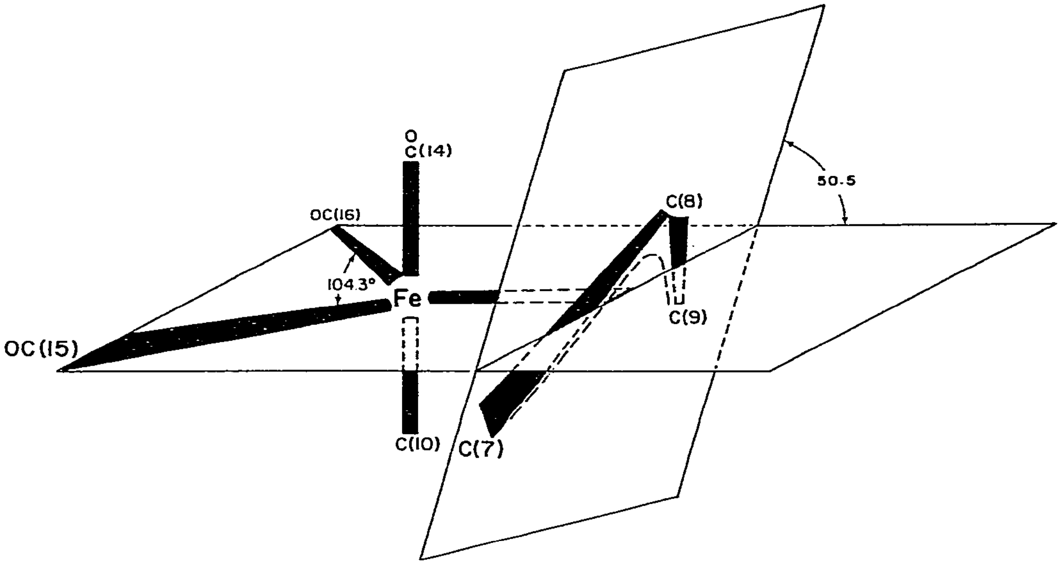


Fig. 3. A diagram of the coordination of Fe(2) in such a way as to show how it may be regarded as distorted octahedral or distorted trigonal bipyramidal.

The (η^3-allyl)–Fe(2) system has normal dimensions for such a moiety, as shown by the comparisons in Table 7. The Fe(2)–C(10) σ bond has a length which is similar to those for other Fe–C σ bonds, as also indicated by data in Table 7. The coordination geometry about Fe(2) can be described as approximately octahedral, if the η^3-allyl group is assumed to occupy two *cis* positions or trigonal bipyramidal if the η^3-allyl is considered unidentate as shown in Fig. 3. Since the "bite" of the η^3-allyl group is small, the opposite C(15)–Fe(2)–C(16) angle is appreciably larger than 90° (*viz.*, 104.3°), but other C–Fe–C angles are between 90 and 95°. The plane of the η^3-allyl group has the acute angle of 50.5° with respect to the plane of the iron, C(15) and C(16).

The binding of an $Fe(CO)_3$ or $Ru(CO)_3$ group to an organic moiety by one σ bond to a carbon atom and one η^3-allyl bond is not without precedent. The structures of $(C_8H_{11}CN)Ru(CO)_3$ and $C_9H_{12}Fe_2(CO)_6$ (see Table 7) provide clear examples that have been documented crystallographically. There are numerous other compounds that are chemically related to or analogous to this structure in which a similar bonding arrangement may be assumed to occur on the basis of chemical and spectral evidence[10–13].

The unexpected, and unusual, nature of this molecule creates interest in possible mechanisms for its formation. However, in view of the extremely small yield on the repetition of this preparation, this compound could possibly have arisen from any of a multitude of sources, and mechanistic speculations seem unjustified at present.

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