

**THE STEREOCHEMISTRY OF
 (5-*exo*-ACYLCYCLOHEXA-1,3-DIENE)TRICARBONYLIRON DERIVATIVES
 AND THE CRYSTAL STRUCTURE OF
 (5-*exo*-CYANOCYCLOHEXA-1,3-DIENE)TRICARBONYLIRON**

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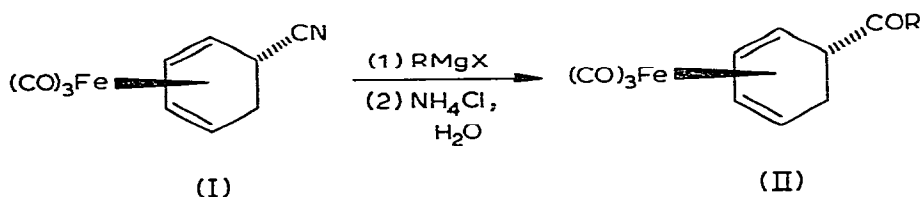
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Summary

(5-Cyanocyclohexa-1,3-diene)tricarbonyliron reacts with Grignard reagents to yield a series of (5-acylcyclohexa-1,3-diene)tricarbonyliron complexes. The cyano derivative crystallises in space group $Pna2_1$, with $Z = 8$, and cell dimensions of a 28.235(19), b 6.498(7), c 11.114(10) Å. The structure was solved by Patterson and Fourier methods, and final residuals were $R = 0.070$ and $R_w = 0.057$ on 803 observed intensity data. The molecules are monomeric and the cyano ligand adopts the *exo* stereochemistry.

Introduction

As part of our studies on dieneiron complexes, a stereospecific synthesis of (5-*exo*-acetocyclohexa-1,3-diene)tricarbonyliron (IIa) [1] was desirable. It was achieved by reaction of the Grignard methylmagnesium iodide with (5-*exo*-cyanocyclohexa-1,3-diene)tricarbonyliron (I) (eq. 1). This method offers a con-



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TABLE 1
COMPOUNDS AND YIELDS PREPARED BY EQ. 1

Compound II	Grignard reagent	Yield (%)
R = Me	MeMgI	75
R = Et	EtMgI	60
R = n-Pr	n-PrMgBr	60
R = n-Bu	n-BuMgI	50
R = Ph	PhMgBr	55

venient route to a variety of 5-*exo*-acyl compounds as illustrated in Table 1.

The stereospecificity of the reaction depended on I being correctly assigned as the *exo* derivative. Although this stereochemistry has always been assumed for I, it has never been unambiguously confirmed. We therefore undertook a single-crystal X-ray structure determination of I.

Experimental

(5-*exo*-Cyanocyclohexa-1,3-diene)tricarbonyliron (I) was prepared by the published procedure [2].

The preparation of the (5-*exo*-acyl)tricarbonyliron compounds II is exemplified for (5-*exo*-acetyl)cyclohexa-1,3-diene)tricarbonyliron.

1.45 g of methyl iodide in ether (10 ml) was added to 0.25 g of magnesium turnings under nitrogen. Reaction proceeded smoothly, and to the resultant solution was added 0.5 g of (5-*exo*-cyanocyclohexa-1,3-diene)tricarbonyliron in ether (20 ml). Reaction was complete within 5 minutes (TLC), and treatment of the solution with aqueous ammonium chloride followed by ether work-up gave (5-*exo*-acetyl)cyclohexa-1,3-diene)tricarbonyliron (0.4 g, 75%). ¹³C NMR δ(CDCl₃) (ppm) 211.0 (Fe(CO)₃), 208.0 (—C=O), 86.7 (C(3)), 84.1 (C(2)) 60.6 (C(4)), 58.6 (C(1)), 51.0 (C(5)), 28.1 (CH₃), 27.5 (C(6)); ¹H NMR δ(CDCl₃) (ppm) 5.36 (2H, m, H(2,3)), 3.05 (3H, m, H(1,4,5)), 2.07 (3H, s, CH₃), 1.89 (2H, m, H(6,6,1)); ν_{max} (cyclohexane) 2048, 1975 cm⁻¹ C=O; 1717 cm⁻¹; M⁺ 262.

The molecular and crystal structure of (CO)₃FeC₆H₇CN (I)

Crystal data: C₁₀H₇NO₃Fe, mol. wt. 245.02, orthorhombic, *a* 28.235(19), *b* 6.498(7), *c* 11.114(10) Å, *U* 2039.1 Å³, *D_c* 1.60 g cm⁻³, *Z* 8, *F*(000) 991.9. Space group *Pna*2₁, from systematic absences (*Ok*l: *k* + *l* = 2*n* + 1, *h*0l: *h* = 2*n* + 1) and structure solution. Graphite monochromated Mo-K_α radiation, λ 0.71069 Å, μ(Mo-K_α) 13.85 cm⁻¹. Intensity data was recorded from a crystal of dimensions 0.46 × 0.03 × 0.05 mm.

Pale yellow needles of I were obtained by slow crystallisation from ether solution. The crystals were mounted in Lindemann capillaries, and the intensity data recorded on a Nonius CAD4 four-circle diffractometer, by the ω/2θ scan technique. Of the 1901 independent reflections measured within the range 1.5 < θ < 25.0° only 803 were significant (*I* > σ(*I*)). Standard deviations were given by σ(*I*) = (I_m + 4(BGL + BGR) + (0.03I)²)^{1/2}, where I_m is the nett measured

intensity, BGL and BGR the left and right hand backgrounds, respectively, and $I = I_m - 2(\text{BGL} + \text{BGR})$. Lorentz polarisation and numerical absorption corrections were applied. The crystal was bounded by the (010), (100), (101), (001), ($\bar{1}01$), ($\bar{1}00$), ($10\bar{1}$), ($00\bar{1}$), ($\bar{1}0\bar{1}$) and ($0\bar{1}0$) faces.

The structure was solved by a combination of Patterson and Fourier techniques. The calculated density indicated the presence of eight molecules in the unit cell. This was interpreted to mean that there was one iron atom in the asymmetric unit if the space group was *Pnam* (a non-standard setting of *Pnma*), or two if the compound crystallised in the non-centrosymmetric space group *Pna2₁*. The Patterson map yielded a straightforward solution in terms of two iron atoms, while the only solution for one heavy atom placed it on a special position ($x, y, 1/4$). This would result in a situation which would not be consistent with the expected molecular structure. So the solution was attempted in space group *Pna2₁*. A Fourier difference map computed on the iron atom coordinates revealed the positions of all the non-hydrogen atoms in the two independent molecules. The residual, [$R = \sum ||F_o| - |F_c|| / \sum F_o$], with all these atoms included was 0.109. The hydrogen atoms in the cyclohexadiene moieties were placed in geometrically acceptable positions and constrained to lie 1.08 Å from the relevant carbons. The hydrogens in each ring system were assigned a common

TABLE 2

ATOM COORDINATES ($\times 10^4$) AND ISOTROPIC TEMPERATURE FACTORS ($\text{Å}^2 \times 10^3$)

	x/a	y/b	z/c	U
Fe(1)	2035(1)	10044(4)	0	
C(11)	2312(10)	11504(46)	8943(25)	23(7)
O(11)	2444(9)	12639(34)	8121(22)	70(7)
C(12)	2275(18)	11644(75)	11227(41)	96(18)
O(12)	2528(9)	12195(38)	11915(24)	76(8)
C(13)	2424(6)	7963(26)	10161(32)	45(5)
O(13)	2659(4)	6519(20)	9896(26)	64(5)
C(101)	1348(11)	10358(54)	9511(24)	47(11)
C(102)	1367(10)	10416(47)	10780(24)	30(8)
C(103)	1541(10)	8888(43)	11193(27)	33(8)
C(104)	1483(11)	6499(46)	10708(26)	40(9)
C(105)	1395(11)	6570(47)	9312(26)	40(9)
C(106)	1577(9)	8470(39)	8800(26)	31(8)
C(10)	989(8)	5899(35)	11249(21)	54(6)
N(1)	610(7)	5458(33)	11631(20)	74(6)
Fe(2)	4001(1)	6025(4)	10053(10)	
C(21)	3704(9)	7621(40)	8945(24)	24(7)
O(21)	3565(8)	8704(34)	8235(22)	63(6)
C(22)	3625(6)	3817(29)	10162(34)	49(6)
O(22)	3388(5)	2454(23)	9834(21)	69(5)
C(23)	3767(14)	7442(69)	11257(41)	91(16)
O(23)	3485(9)	8270(35)	11896(23)	74(8)
C(201)	4664(10)	6227(49)	10843(26)	53(9)
C(202)	4683(9)	6558(44)	9543(20)	36(7)
C(203)	4492(12)	4553(55)	8722(34)	76(14)
C(204)	4606(11)	2361(54)	9495(23)	62(11)
C(205)	4639(10)	2598(43)	10871(22)	35(7)
C(206)	4489(9)	4686(40)	11108(24)	27(7)
C(20)	5074(10)	11905(39)	8926(26)	73(8)
N(2)	5423(7)	1471(33)	8489(21)	73(6)

TABLE 3

ANISOTROPIC TEMPERATURE FACTORS ($\text{\AA}^2 \times 10^3$)The temperature factor exponent takes the form: $-2\pi^2(U_{11}h^2a^{*2} + \dots + 2U_{12}hka^*b^*)$

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Fe(1)	38(1)	38(1)	37(1)	9(4)	18(3)	-2(1)
Fe(2)	42(1)	38(2)	46(2)	5(5)	-7(4)	0(1)

isotropic temperature factor. A weighting scheme of the form $1.3935/(\sigma^2(F) + 0.00089F^2)$ was also introduced, and full-matrix least-squares refinement continued, with the two Fe atoms with anisotropic thermal parameters, until convergence was reached. The final residuals were $R = 0.070$, and $R_w = \sum w^{1/2} \Delta / \sum w^{1/2} |F_o| = 0.057$. The highest peak in a final difference electron density synthesis was less than 0.7 e \AA^{-3} . The final atomic coordinates and isotropic temperature factors for the non-hydrogen atoms are given in Table 2, while the anisotropic thermal parameters are listed in Table 3. The relevant hydrogen atom parameters can be found in Table 4.

Complex neutral-atom scattering factors [3] were employed. Calculations were performed with the University of Cambridge IBM 370/165 computer using programs written by G.M.S. and the PLUTO plotting program written by Dr. W.D.S. Motherwell.

Structure factor tables may be obtained from the authors.

Discussion

The structure determination confirms the expected molecular configuration. The cyano ligand coordinates to the cyclohexa-1,3-diene ring, and exhibits *exo*-stereochemistry with respect to the metal. There is no imposed crystallographic symmetry, and the two independent neutral molecules in the asym-

(continued on p. 121)

TABLE 4

HYDROGEN ATOM COORDINATES ($\times 10^4$) AND ANISOTROPIC TEMPERATURE FACTORS ($\text{\AA}^2 \times 10^3$)

	x/a	y/b	z/c	U
H(1E)	1190(11)	11625(54)	9027(24)	107(28)
H(12)	1227(10)	11653(47)	11325(24)	107(28)
H(13)	1774(10)	9110(43)	11953(27)	107(28)
H(14)	1778(11)	5515(46)	10933(26)	107(28)
H(15A)	1018(11)	6466(47)	9156(26)	107(28)
H(15B)	1570(11)	5288(47)	8883(26)	107(28)
H(16)	1817(9)	8580(39)	8046(26)	107(28)
H(21)	4799(10)	7312(49)	11494(26)	41(20)
H(22)	4812(9)	7978(44)	9158(20)	41(20)
H(23)	4325(12)	4661(55)	7850(34)	41(20)
H(24)	4335(11)	1193(54)	9431(23)	41(20)
H(25A)	4998(10)	2378(43)	11183(22)	41(20)
H(25B)	4408(10)	1507(43)	11311(22)	41(20)
H(26)	4164(9)	4827(40)	11616(24)	41(20)

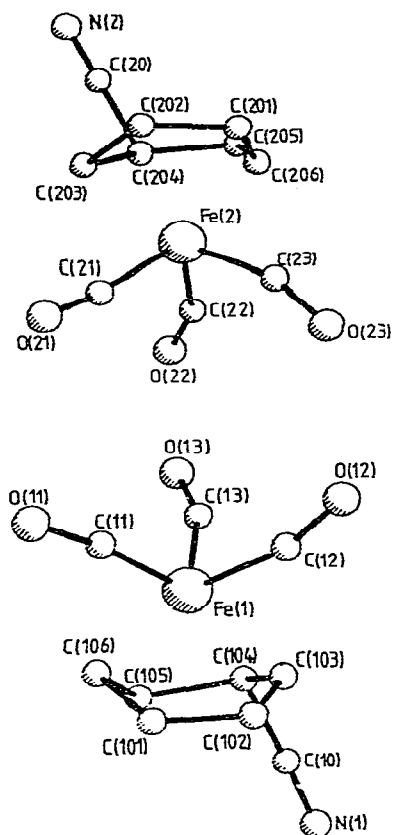


Fig. 1. A projection of the contents of an asymmetric unit, showing the atom numbering scheme adopted (hydrogen atoms have been omitted for clarity, but follow the numbering of the carbons to which they are attached).

TABLE 5

BOND LENGTHS (Å) WITH ESTIMATED STANDARD DEVIATIONS GIVEN IN PARENTHESES

Fe(1)—C(11)	1.70(3)	Fe(2)—C(21)	1.81(3)
Fe(1)—C(12)	1.84(5)	Fe(2)—C(22)	1.79(2)
Fe(1)—C(13)	1.75(2)	Fe(2)—C(23)	1.75(5)
C(11)—O(11)	1.23(4)	C(21)—O(21)	1.13(3)
C(12)—O(12)	1.10(5)	C(22)—O(22)	1.17(2)
C(13)—O(13)	1.19(2)	C(23)—O(23)	1.20(5)
Fe(1)—C(101)	2.03(3)	Fe(2)—C(201)	2.07(3)
Fe(1)—C(102)	2.09(3)	Fe(2)—C(202)	2.04(3)
Fe(1)—C(103)	2.07(3)	Fe(2)—C(203)	2.24(4)
Fe(1)—C(106)	2.12(3)	Fe(2)—C(206)	2.01(3)
C(101)—C(102)	1.41(3)	C(201)—C(202)	1.46(3)
C(102)—C(103)	1.20(4)	C(202)—C(203)	1.68(5)
C(103)—C(104)	1.65(4)	C(203)—C(204)	1.69(5)
C(104)—C(105)	1.57(2)	C(204)—C(205)	1.54(3)
C(105)—C(106)	1.45(4)	C(205)—C(206)	1.45(4)
C(106)—C(101)	1.60(4)	C(205)—C(201)	1.15(4)
C(104)—C(10)	1.57(4)	C(204)—C(20)	1.49(4)
C(10)—N(1)	1.19(3)	C(20)—N(2)	1.13(3)

TABLE 6

BOND ANGLES (°) WITH ESTIMATED STANDARD DEVIATIONS GIVEN IN PARENTHESES

C(11)—Fe(1)—C(12)	91.6(11)	C(21)—Fe(2)—C(22)	103.4(13)
C(11)—Fe(1)—C(13)	102.3(14)	C(21)—Fe(2)—C(23)	92.5(12)
C(12)—Fe(1)—C(13)	97.4(18)	C(22)—Fe(2)—C(23)	98.4(18)
Fe(1)—C(11)—O(11)	170.2(24)	Fe(2)—C(21)—O(21)	172.8(24)
Fe(1)—C(12)—O(12)	158.7(44)	Fe(2)—C(22)—O(22)	157.9(31)
Fe(1)—C(13)—O(13)	159.4(29)	Fe(2)—C(23)—O(23)	160.2(35)
C(11)—Fe(1)—C(101)	101.5(14)	C(21)—Fe(2)—C(201)	131.9(12)
C(11)—Fe(1)—C(102)	129.6(14)	C(21)—Fe(2)—C(202)	98.6(12)
C(11)—Fe(1)—C(103)	163.1(13)	C(21)—Fe(2)—C(203)	94.7(15)
C(11)—Fe(1)—C(106)	96.6(14)	C(21)—Fe(2)—C(206)	163.9(12)
C(12)—Fe(1)—C(101)	119.6(19)	C(22)—Fe(2)—C(201)	123.9(13)
C(12)—Fe(1)—C(102)	87.7(18)	C(22)—Fe(2)—C(202)	135.7(10)
C(12)—Fe(1)—C(103)	88.8(18)	C(22)—Fe(2)—C(203)	94.0(13)
C(12)—Fe(1)—C(106)	164.0(17)	C(22)—Fe(2)—C(206)	91.1(12)
C(13)—Fe(1)—C(101)	134.9(11)	C(23)—Fe(2)—C(201)	89.0(17)
C(13)—Fe(1)—C(102)	127.8(13)	C(23)—Fe(2)—C(202)	118.6(16)
C(13)—Fe(1)—C(103)	94.4(13)	C(23)—Fe(2)—C(203)	163.8(17)
C(13)—Fe(1)—C(106)	94.3(11)	C(23)—Fe(2)—C(206)	92.3(17)
Fe(1)—C(101)—C(102)	72.5(23)	Fe(2)—C(201)—C(202)	67.9(20)
Fe(1)—C(101)—C(106)	70.6(17)	Fe(2)—C(201)—C(206)	70.5(21)
C(102)—C(101)—C(106)	120.0(36)	C(202)—C(201)—C(206)	113.3(35)
Fe(1)—C(102)—C(101)	67.4(22)	Fe(2)—C(202)—C(201)	70.5(21)
Fe(1)—C(102)—C(103)	72.0(21)	Fe(2)—C(202)—C(203)	73.5(16)
C(101)—C(102)—C(103)	112.0(8)	C(201)—C(202)—C(203)	114.3(30)
Fe(1)—C(103)—C(102)	74.4(21)	Fe(2)—C(203)—C(202)	73.5(16)
Fe(1)—C(103)—C(104)	101.5(17)	Fe(2)—C(203)—C(204)	98.1(20)
C(102)—C(103)—C(104)	127.7(28)	C(202)—C(203)—C(204)	108.4(27)
C(103)—C(104)—C(105)	108.1(23)	C(203)—C(204)—C(205)	115.5(30)
C(103)—C(104)—C(10)	101.4(21)	C(203)—C(204)—C(20)	96.9(24)
C(10)—C(104)—C(105)	104.2(28)	C(20)—C(204)—C(205)	112.7(30)
C(104)—C(105)—C(106)	110.8(30)	C(204)—C(205)—C(206)	104.9(28)
Fe(1)—C(106)—C(101)	64.2(18)	Fe(2)—C(206)—C(201)	76.7(22)
Fe(1)—C(106)—C(105)	112.3(21)	Fe(2)—C(206)—C(205)	120.1(20)
C(105)—C(106)—C(101)	108.4(23)	C(205)—C(206)—C(201)	129.9(28)
C(104)—C(10)—N(1)	178.3(24)	C(204)—C(10)—N(2)	117.0(30)

TABLE 7

HYDROGEN BOND ANGLES (deg.)

H(11)—C(101)—Fe(1)	127.2(10)	C(102)—C(101)—H(11)	119.5(26)
C(106)—C(101)—H(11)	120.4(16)	H(12)—C(102)—Fe(1)	130.3(8)
H(12)—C(102)—C(101)	124.5(26)	C(103)—C(102)—H(12)	123.5(21)
H(13)—C(103)—Fe(1)	92.4(9)	H(13)—C(103)—C(102)	116.0(20)
C(104)—C(103)—H(13)	116.2(16)	H(14)—C(104)—C(103)	113.8(16)
C(105)—C(104)—H(14)	111.6(22)	C(10)—C(104)—H(14)	116.7(14)
H(15A)—C(105)—C(104)	108.2(21)	H(15B)—C(105)—C(104)	109.9(22)
C(106)—C(105)—H(15B)	108.6(17)	C(106)—C(105)—H(15A)	109.9(17)
H(16)—C(106)—C(101)	126.0(16)	H(16)—C(106)—Fe(1)	94.2(9)
H(21)—C(201)—Fe(2)	130.1(9)	H(16)—C(106)—C(105)	125.6(16)
C(206)—C(201)—H(21)	123.1(21)	C(202)—C(201)—H(21)	123.6(22)
H(22)—C(202)—C(201)	122.0(22)	H(22)—C(202)—Fe(2)	125.0(9)
H(23)—C(203)—Fe(2)	107.1(11)	C(203)—C(202)—H(22)	123.7(16)
H(24)—C(204)—C(203)	115.1(17)	H(23)—C(203)—C(202)	125.2(17)
C(20)—C(204)—H(24)	117.3(17)	C(205)—C(204)—H(24)	100.3(22)
H(25B)—C(205)—C(204)	110.3(22)	H(25A)—C(205)—C(204)	111.3(21)
C(206)—C(205)—H(25A)	107.3(16)	C(206)—C(205)—H(25B)	110.9(16)
H(26)—C(206)—Fe(2)	71.7(9)	H(26)—C(206)—C(201)	115.1(21)
H(26)—C(206)—C(205)	115.0(16)		

TABLE 8

THE LEAST-SQUARES PLANES ARE GIVEN BY THE EQUATION $px + qy + rz = s$, WHERE x , y AND z ARE THE ATOMIC FRACTIONAL COORDINATES. INDIVIDUAL ATOMIC DEVIATIONS FROM THE PLANE ARE GIVEN IN PARENTHESES (Å)

Plane	Number	p	q	r	s
C(101)—C(102)—C(104)—C(106) [C(101) -0.02, C(102) 0.02, C(103) -0.01, C(106) 0.01]	1	25.067	2.988	-0.228	6.274
C(103)—C(104)—C(105)—C(106) [C(103) -0.07, C(104) 0.12, C(105) -0.14, C(106) 0.08]	2	27.489	-1.484	0.044	3.036
C(201)—C(202)—C(203)—C(206) [C(201) 0.03, C(202) -0.02, C(203) 0.01, C(206) -0.02]	3	25.790	-2.645	0.067	10.428
C(203)—C(204)—C(205)—C(206) [C(203) 0.03, C(204) -0.04, C(205) 0.05, C(206) -0.03]	4	27.313	1.639	-0.279	12.747

Angles between normals to planes ($^{\circ}$)

2	3	4	Plane
40.6	51.4	12.8	1
	10.8	27.9	2
		38.7	3

metric unit have similar geometries, as can be seen in Fig. 1, a view down the b axis. The bond lengths and interbond angles for the non-hydrogen atoms are listed in Tables 5 and 6 respectively, while bond angles involving hydrogen atoms are given in Table 7, and details of the least-squares planes are given in Table 8.

The iron atoms are formally four-coordinate, the fourth site being occupied by the butadiene segment of the cyclohexadiene ring. In both molecules the butadiene moiety is planar and the carbon atoms may be considered as sp^2 hybridised. The Fe—C(butadiene) distances are similar (mean 2.09 Å), and lie within the range of values reported in a number of structures where iron is bonded to a butadiene unit [4–9]. The ring bond lengths and angles are not significantly different from those found in other (cyclohexa-1,3-diene)tricarbonyliron derivatives [10,11], and the dihedral angles between the C(101), C(102), C(103), C(106), and C(103), C(104), C(105), C(106), and the C(201), C(202), C(203), C(206) and C(203), C(204), C(205), C(206) sets, at 40.6° and 38.7°, are close to the values of 40.0° and 38.5° reported in $[\text{Ph}_3\text{P}(\text{C}_6\text{H}_7)\text{Fe}(\text{CO})_3][\text{BF}_4]$ [10] and $\text{C}_{10}\text{H}_{12}\text{Fe}_2(\text{CO})_{16}$ [12], respectively.

The cyano ligands are approximately linear, and the C(ring)—C (mean 1.53 Å) and C—N (mean 1.16 Å) distances suggest that there is little electron donation from the C—N bond to the ring system, so that it may be considered as a C—N triple bond.

The geometries of the $\text{Fe}(\text{CO})_3$ moieties are similar to those found in related compounds [4–12], allowing for the high standard deviations in the bond parameters caused by the scarcity of intensity data. There is considerable distortion from the idealised C_{3v} symmetry.

There are no abnormally short intramolecular non-bonded contacts with the molecule in the *exo* configuration. Neither are there any significant intermolecular short approaches, the molecules being separated by typical Van der Waals' distances.

Conclusion

The *exo*-orientation of the cyano group in (5-*exo*-cyanocyclohexa-1,3-diene)tricarbonyliron (I) has been confirmed by the X-ray analysis. This allows the *exo* assignment to be given to the 5-acyl substituents of the (5-*exo*-acylcyclohexa-1,3-diene)tricarbonyliron derivatives prepared from I.

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