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SYNTHESIS OF 1,3-BIS(DICARBONYL-η-CYCLOPENTADIENYLIRON)PROP-2-EN-1-ONE AND ITS INTERACTION WITH NONACARBONYLDIIRON. CRYSTAL STRUCTURE OF OCTACARBONYL-(η-CYCLOPENTADIENYL)-μ_3-ETHYLIDYNE-*triangulo*-TRIIRON

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Summary

Reaction of *trans*-β-chloroacryloyl chloride with FpNa (Fp = η-$C_5H_5Fe(CO)_2$) yielded the complex FpCH=CHCOFp, which reacted with $Fe_2(CO)_9$ to give trinuclear complexes IIa—IIc. The structure of IIc was established by an X-ray study.

Introduction

β-Substituted σ-vinyl derivatives of dicarbonyl-η-cyclopentadienyliron FpCH=CHCOX (X = Alk, Ar [1—3]; X = OR, OH [4]; X = NR_2 [5]) appear rather interesting from the synthetic point of view. Insertion of an organometallic β-substituent into the reactive unsaturated molecules of these compounds extends possibilities of their application in synthesis. For example, starting with FpCH=CHCOX we have developed a synthetic route to binuclear complexes with σ,π-bridging ligands.

In the present paper we report the preparation of *trans*-FpCH=CHCOFp (I) and its reactions with $Fe_2(CO)_9$ and discuss some peculiarities of the chemical behaviour of I arising from the presence of two Fp-substituents.

Results and discussion

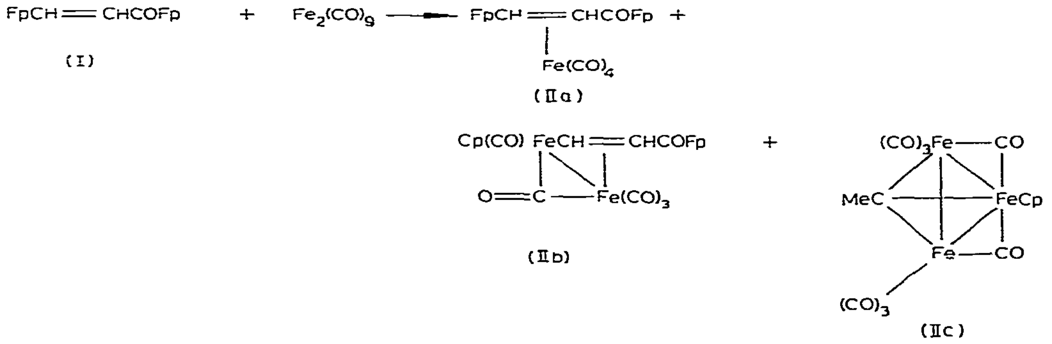
The reaction of *trans*-β-chloroacryloyl chloride with FpNa in THF:

$$\mathrm{ClCH{=}CHCOCl} + 2\ \mathrm{FpNa} \rightarrow \underset{\text{(I)}}{\mathrm{FpCH{=}CHCOFp}} + 2\ \mathrm{NaCl}$$

produces I in 40% yield. I is an orange crystalline substance stable in the solid state but slowly decomposing in solution. It was characterized by spectroscopy (see the Experimental section) as a σ-acryloyliron derivative β-substituted by the second σ-bonded dicarbonyl-η-cyclopentadienyliron moiety.

It is to be noted that an attempt to substitute only one chlorine atom in ClCH=CHCOCl by adding only one equivalent of FpNa was unsuccessful, yielding a mixture of FpCl and I. The rate of halogen substitution (both at acyl and olefinic carbon atoms) proved too high, obviously due to a high nucleophility of the Fp anion [6].

We began investigating the reactivity of I by adding it to $Fe_2(CO)_9$, in order to obtain trinuclear carbonyliron complexes with σ,π-bridging olefinic ligands. From the reaction mixture obtained by heating I with $Fe_2(CO)_9$ in benzene at 35—40°C we isolated three carbonyliron complexes (IIa, b, c), separated by repeated thin layer chromatography on silica gel. The yields of IIa, IIb and IIc are 45.15 and 2%, respectively.



The orange crystalline IIa is a π-olefinic tetracarbonyliron complex with two dicarbonyl-η-cyclopentadienyliron moieties attached to the olefinic ligand. The IR spectrum of IIa as compared to that of I shows a shift of the ν(C=C) stretching band to lower wavenumber by ca. 100 cm^{-1}, but practically no shift of the acyl group band. In the ^1H NMR spectrum of IIa the olefin protons exhibit an expected high field shift, viz. δ(CH) 3.27 and 1.28 ppm, $J = 8$ Hz.

The dark green complex IIb, similar to the previously reported binuclear complex with a σ,π-bridging olefin [7], was characterized by elemental analysis and IR spectroscopy (see the Experimental section). IIb is unstable, especially in solution.

The formation of complex IIc from I and $Fe_2(CO)_9$ under mild conditions was rather surprising. Its structure was established by a single crystal X-ray analysis. Recently, similar iron complexes were isolated from the reactions of methylacetylene with $Fe_3(CO)_{12}$ [8] and of the σ-propargyl complex $FpCH_2C{\equiv}CH$ with $Fe_2(CO)_9$ [9], but up to now the possibility of their formation from σ-alkenyl derivatives of dicarbonyl-η-cyclopentadienyliron was quite unknown.

From the data available at present we cannot deduce which one of the two iron atoms of I is involved in forming IIc; but in any case the formation of the ethylidyne ligand requires a deep rearrangement of the bridging acryloyl moiety, —CH=CHCO—.

Molecular structure of IIc

The molecular geometry of IIc is shown in Fig. 1 and bond lengths and angles are listed in Table 1. The molecule involves a tetrahedral heteroatomic cluster Fe_3C as a core. All of its iron atoms satisfy the EAN rule under the assumption that they form two-electron covalent bonds with each other. IIc resembles the recently studied $(\eta^5\text{-}C_5H_2Me_2C_2H_3)Fe_3(CO)_8(\mu_3\text{-}CEt)$ (III) [8] and $(\eta^5\text{-}C_5H_5)Fe_3(CO)_8(\mu_3\text{-}CCH{=}CH_2)$ (IV) [9], differing from III by the absence of substituents in the Cp ring and from both III and IV by the substituent at the μ_3-carbon atom C(9). The molecule IIc has an approximate non-crystallographic mirror plane through Fe(1), C(9), C(10) and C(11) atoms and the midpoint of the Fe(2)—Fe(3) bond.

The carbonyl-bridged Fe(1)—Fe(2) and Fe(1)—Fe(3) bonds are significantly shorter (average 2.506(4) Å) than the non-bridged Fe(2)—Fe(3) bond (2.594(1) Å). All Fe—Fe distances in IIc are close to the corresponding values in III and IV and are comparable to the doubled covalent radius of iron 2.48 Å [10].

Carbonyl bridges in IIc are asymmetric, the bonds of C(1) and C(2) with Fe(1) (average 1.891(5) Å) being 0.13 Å shorter than their bonds with both Fe(2) and Fe(3) (average 2.023(5) Å). In III the corresponding difference is 0.14 Å and in IV 0.16 Å. This may be ascribed to different oxidation states of the iron atoms, being formally +2 for Fe(1) and +1 for Fe(2) and Fe(3). It is noteworthy that of the three Fe—C bonds involving C(9) the shortest is also the one with Fe(1).

As is usual in M_3C clusters the geometry of the triply bridging C(9) atom is a result of a compromise between the opposing trends of C(9) to acquire ideal sp^3 angles (109.5°) and to form Fe—C(sp^3) bonds of a normal length of 2.098 Å [11]. Actually, both values mentioned (average FeC(9)Fe angle 82.3(9)°, average Fe—C(9) distance 1.927(14) Å) are far from ideal but close to those in III and IV.

The planar Cp ring in IIc forms a dihedral angle of 74.7° with the plane through the three iron atoms (72.7° in III, 73.8° in IV). The C(9) atom is

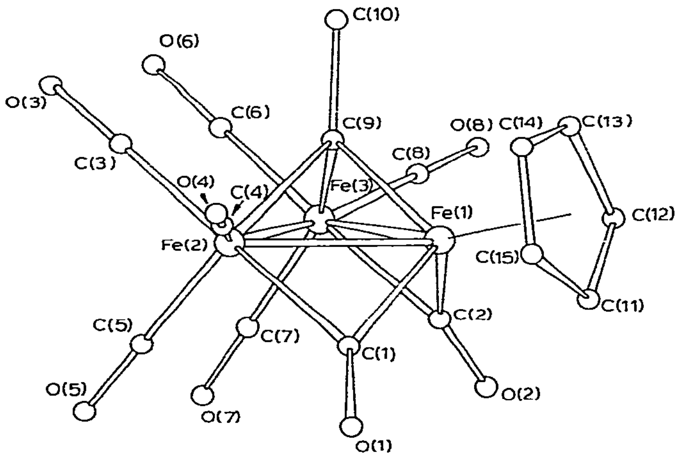


Fig. 1. Molecular structure of complex IIc. Hydrogen atoms are omitted.

TABLE 1

BOND LENGTHS (Å) AND ANGLES (°) [a]

Bond		Bond		Bond	
Fe(1)—Fe(2)	2.502(1)	Fe(3)—C(9)	1.927(5)	C(9)—C(10)	1.490(7)
Fe(1)—Fe(3)	2.510(1)	Fe(1)—C(11)	2.094(5)	C(11)—C(12)	1.417(8)
Fe(2)—Fe(3)	2.594(1)	Fe(1)—C(12)	2.099(5)	C(12)—C(13)	1.388(8)
Fe(1)—C(1)	1.887(5)	Fe(1)—C(13)	2.117(5)	C(13)—C(14)	1.411(7)
Fe(2)—C(1)	2.027(5)	Fe(1)—C(14)	2.117(6)	C(14)—C(15)	1.399(8)
Fe(1)—C(2)	1.894(5)	Fe(1)—C(15)	2.086(5)	C(15)—C(11)	1.408(8)
Fe(3)—C(2)	2.019(5)	Fe(1)—CP	1.730	C(10)—H(10)	0.92(5)
Fe(2)—C(3)	1.787(5)	C(1)—O(1)	1.158(6)	C(10)—H′(10)	1.00(5)
Fe(2)—C(4)	1.788(6)	C(2)—O(2)	1.156(6)	C(10)—H″(10)	1.03(5)
Fe(2)—C(5)	1.811(5)	C(3)—O(3)	1.138(6)	C(11)—H(11)	0.90(5)
Fe(3)—C(6)	1.798(5)	C(4)—O(4)	1.141(7)	C(12)—H(12)	0.99(5)
Fe(3)—C(7)	1.826(5)	C(5)—O(5)	1.139(6)	C(13)—H(13)	1.01(6)
Fe(3)—C(8)	1.790(6)	C(6)—O(6)	1.137(7)	C(14)—H(14)	0.88(5)
Fe(1)—C(9)	1.903(5)	C(7)—O(7)	1.136(7)	C(15)—H(15)	0.97(5)
Fe(2)—C(9)	1.951(5)	C(8)—O(8)	1.137(7)		

Angle		Angle		Angle	
Fe(2)Fe(1)Fe(3)	62.34(3)	C(3)Fe(2)C(9)	86.5(2)	Fe(2)C(4)O(4)	178.4(5)
Fe(2)Fe(1)C(1)	52.8(2)	C(4)Fe(2)C(5)	106.8(2)	Fe(2)C(5)O(5)	178.7(5)
Fe(2)Fe(1)C(2)	96.1(2)	C(4)Fe(2)C(9)	110.5(2)	Fe(3)C(6)O(6)	177.9(5)
Fe(2)Fe(1)C(9)	50.34(14)	C(5)Fe(2)C(9)	142.7(2)	Fe(3)C(7)O(7)	177.4(5)
Fe(2)Fe(1)Cp	143.6	Fe(1)Fe(3)Fe(2)	58.69(3)	Fe(3)C(8)O(8)	179.2(5)
Fe(3)Fe(1)C(1)	92.4(2)	Fe(1)Fe(3)C(2)	47.9(2)	Fe(1)C(9)Fe(2)	81.0(2)
Fe(3)Fe(1)C(2)	52.3(2)	Fe(1)Fe(3)C(6)	135.2(2)	Fe(1)C(9)Fe(3)	81.9(2)
Fe(3)Fe(1)C(9)	49.48(14)	Fe(1)Fe(3)C(7)	121.4(2)	Fe(2)C(9)Fe(3)	84.0(2)
Fe(3)Fe(1)Cp	146.9	Fe(1)Fe(3)C(8)	103.4(2)	Fe(1)C(9)C(10)	130.6(4)
C(1)Fe(1)C(2)	82.7(2)	Fe(1)Fe(3)C(9)	48.64(14)	Fe(2)C(9)C(10)	131.4(4)
C(1)Fe(1)C(9)	103.0(2)	Fe(2)Fe(3)C(2)	90.3(2)	Fe(3)C(9)C(10)	129.6(4)
C(1)Fe(1)Cp	119.5	Fe(2)Fe(3)C(6)	94.7(2)	C(12)C(11)C(15)	107.6(5)
C(2)Fe(1)C(9)	101.7(2)	Fe(2)Fe(3)C(7)	96.6(2)	C(11)C(12)C(13)	107.7(5)
C(2)Fe(1)Cp	119.2	Fe(2)Fe(3)C(8)	157.5(2)	C(12)C(13)C(14)	108.1(6)
C(9)Fe(1)Cp	122.7	Fe(2)Fe(3)C(9)	48.40(14)	C(13)C(14)C(15)	107.6(5)
Fe(1)Fe(2)Fe(3)	58.97(3)	C(2)Fe(3)C(6)	175.0(2)	C(11)C(15)C(14)	109.0(5)
Fe(1)Fe(2)C(1)	47.85(14)	C(2)Fe(3)C(7)	84.8(2)	C(9)C(10)H(10)	114(3)
Fe(1)Fe(2)C(3)	134.9(2)	C(2)Fe(3)C(8)	85.3(2)	C(9)C(10)H′(10)	112(3)
Fe(1)Fe(2)C(4)	99.7(2)	C(2)Fe(3)C(9)	96.5(2)	C(9)C(10)H″(10)	112(3)
Fe(1)Fe(2)C(5)	123.3(2)	C(6)Fe(3)C(7)	94.9(2)	H(10)C(10)H′(10)	103(4)
Fe(1)Fe(2)C(9)	48.68(14)	C(6)Fe(3)C(8)	90.0(2)	H(10)C(10)H″(10)	107(4)
Fe(3)Fe(2)C(1)	86.9(2)	C(6)Fe(3)C(9)	86.6(2)	H′(10)C(10)H″(10)	108(4)
Fe(3)Fe(2)C(3)	96.9(2)	C(7)Fe(3)C(8)	105.0(2)	C(12)C(11)H(11)	122(3)
Fe(3)Fe(2)C(4)	155.7(2)	C(7)Fe(3)C(9)	144.8(2)	C(15)C(11)H(11)	130(3)
Fe(3)Fe(2)C(5)	95.5(2)	C(8)Fe(3)C(9)	110.1(2)	C(11)C(12)H(12)	122(3)
Fe(3)Fe(2)C(9)	47.64(14)	Fe(1)C(1)Fe(2)	79.4(2)	C(13)C(12)H(12)	130(3)
C(1)Fe(2)C(3)	176.2(2)	Fe(1)C(1)O(1)	143.0(4)	C(12)C(13)H(13)	121(3)
C(1)Fe(2)C(4)	85.8(2)	Fe(2)C(1)O(1)	137.5(4)	C(14)C(13)H(13)	131(3)
C(1)Fe(2)C(5)	84.9(2)	Fe(1)C(2)Fe(3)	79.7(2)	C(13)C(14)H(14)	127(3)
C(1)Fe(2)C(9)	96.5(2)	Fe(1)C(2)O(2)	142.1(4)	C(15)C(14)H(14)	125(3)
C(3)Fe(2)C(4)	90.9(2)	Fe(3)C(2)O(2)	138.1(4)	C(11)C(15)H(15)	128(3)
C(3)Fe(2)C(5)	94.3(2)	Fe(2)C(3)O(3)	178.0(5)	C(14)C(15)H(15)	123(3)

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[a] Cp is the centroid of the cyclopentadienyl ligand.

displaced by 1.25 Å from the latter plane, while the C(9)—C(10) bond is almost normal to it (the angle 88.5°). The mean planes through C(1)O(1) and C(2)O(2) bridging carbonyls and the iron atoms to which they are bonded are tilted off the Fe_3 plane in the opposite (in respect to C(9)) directions by 61.8 and 55.1°, respectively.

Experimental

trans-β-Chloroacryloyl chloride was prepared by the reported method [12]. Complexes I and II were prepared and purified under argon.

IR and ^1H NMR spectra were recorded with a UR-20 spectrometer and Hitachi-Perkin-Elmer R-20 instrument (60 MHz), respectively.

The X-ray experiment was carried out with a Syntex $P2_1$ computer-controlled four-circle diffractometer at —120°C using graphite-monochromated Mo-K_α radiation. All calculations were performed with an Eclipse S/200 mini-computer using EXTL programs modified in our laboratory by R.G. Gerr and A.I. Yanovskii.

Crystal data: $C_{15}H_8Fe_3O_8$, $M = 483.8$, monoclinic, $a = 8.895(4)$, $b = 12.093(4)$, $c = 15.830(15)$ Å, $\alpha = 103.68(6)°$, $V = 1655(4)$ Å^3, $Z = 4$, $D_c = 1.94$ g cm^{-3}, space group $P2_1/c$.

Of 2893 independent reflections in the range of $2° \leqslant 2\theta \leqslant 48°$ measured by the $\theta/2\theta$ scan technique, 2286 with $I \geqslant 2\sigma$ were used in the analysis. The structure was solved by the direct method using the MULTAN program. All non-hydrogen atoms were refined with anisotropic thermal parameters to $R = 0.046$. All hydrogen atoms were then located directly in the difference synthesis and refined isotropically. Finally $R = 0.039$, $R_w = 0.038$ and goodness of fit of 1.74 with the weighting scheme $w^{-1} = \sigma_F^2 + (0.005\ F_0)^2$. Atomic coordinates and thermal parameters are given in Tables 2 and 3.

Preparation of I

2.3 g (1.8 mmol) of *trans*-β-chcloroacryloyl chloride in 50 ml THF was added dropwise to a stirred solution of NaFp (prepared from 6.4 g or 1.8 mmol of Fp_2) in 250 ml THF at —78°C. Then the mixture was allowed to warm to room temperature and THF was removed in vacuo. The residue was chromatographed twice on a column with alumina (eluent: petroleum ether/$CHCl_3$, 2/1) to give 3.4 g (45%) of complex I, m.p. 95—97°C (hexane). Found: C, 49.84; H, 2.97; Fe, 27.18. $C_{17}H_{12}Fe_2O_5$, calcd.: C, 50.04; H, 2.96; Fe, 27.38%. IR: ν(C=O) 1630, ν(C=C) 1495 cm^{-1} (KBr); ν(C≡O) 2032, 1980 cm^{-1} ($CHCl_3$). ^1H NMR: δ(Cp) 4.92 and 4.98, δ(CH) 8.49 and 7.02 ppm, $J(\alpha\beta)$ 16.6 Hz ($CDCl_3$).

Reaction of I with $Fe_2(CO)_9$

A mixture of 1.0 g (2.5 mmol) of I and 1.8 g (5.0 mmol) of $Fe_2(CO)_9$ in 50 ml of benzene was stirred at 35—40°C for 3 hours. Then the solvent was removed in vacuo and the residue chromatographed on a silica gel plate (eluent: $CHCl_3$). From the first brownish red band 0.03 g (ca. 2%) of IIc was isolated as small crystals, m.p. 118—123°C (dec., hexane). IR: ν(C≡O) 2065, 2021, 2014, 1980, 1971 cm^{-1} (cyclohexane); ν(CO, bridging) 1860, 1820 cm^{-1}. The second green band gave 0.2 g (ca. 15%) of IIb, m.p. 150—160°C (dec., petroleum ether).

TABLE 2

ATOMIC COORDINATES ($\times 10^4$, for Fe $\times 10^5$) AND ANISOTROPIC THERMAL PARAMETERS IN THE FORM $T = \exp[-0{,}25(B_{11}h^2a^{*2} + \ldots + 2B_{23}klb^*c^*)]$

Atom	x	y	z	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Fe(1)	18964(8)	71954(4)	12185(4)	1,56(3)	1,37(3)	1,23(3)	−0,13(2)	0,40(2)	−0,06(2)
Fe(2)	18794(8)	90152(6)	19687(4)	1,65(3)	1,56(3)	1.30(3)	−0,06(2)	0,62(2)	−0,14(2)
Fe(3)	43251(8)	83318(6)	15001(4)	1,35(3)	1,72(3)	1,69(3)	−0,06(2)	0,52(2)	−0,04(3)
O(1)	1369(5)	6997(3)	2950(2)	4,8(2)	2,8(2)	1,5(2)	−0,6(2)	1,6(2)	0,3(1)
O(2)	4602(4)	6021(3)	2174(2)	2,6(2)	2,0(2)	2,9(2)	0,4(1)	−0,4(1)	0,5(1)
O(3)	2026(4)	11330(3)	1460(3)	3,3(2)	1,7(2)	4,3(2)	−0,2(1)	0,2(2)	0,2(2)
O(4)	−1451(4)	9219(3)	1832(3)	1.9(2)	3,9(2)	3,8(2)	0,0(1)	1,2(2)	−0,1(2)
O(5)	3464(5)	9412(4)	3801(2)	4,2(2)	5,0(2)	1,5(2)	−1,1(2)	0,8(2)	−0,7(2)
O(6)	5147(4)	10516(3)	938(3)	3.7(2)	2,5(2)	3,1(2)	−0,8(2)	1,7(2)	0,3(2)
O(7)	6446(4)	8472(3)	3247(3)	3,3(2)	2,5(2)	2,6(2)	−0,1(2)	−0,5(2)	−0,1(2)
O(8)	6142(5)	7311(4)	390(3)	3.8(2)	5,3(3)	4,9(2)	0,9(2)	2,7(2)	−0,8(2)
C(1)	1627(6)	7440(4)	2351(3)	2,1(2)	2,1(2)	1,7(2)	0,5(2)	0,6(2)	0,1(2)
C(2)	3907(6)	6769(4)	1832(3)	2,2(2)	2,1(2)	1,6(2)	−0,3(2)	0,5(2)	−0,3(2)
C(3)	1997(5)	10429(4)	1663(3)	1,7(2)	2,1(2)	2,0(2)	0,0(2)	0,2(2)	−0,2(2)
C(4)	−150(6)	9152(4)	1893(3)	2,5(3)	1,9(2)	1,7(2)	−0,2(2)	0,9(2)	−0,2(2)
C(5)	2848(6)	9247(4)	3096(3)	2,4(2)	2,5(2)	1,8(2)	−0,5(2)	1,1(2)	−0,3(2)
C(6)	4837(6)	9676(5)	1172(3)	1.7(2)	2,8(3)	2,0(2)	−0,2(2)	0,8(2)	−0,4(2)
C(7)	5636(6)	8391(4)	2577(4)	1.7(2)	1.9(2)	2,6(3)	0,0(2)	0,5(2)	−0,1(2)
C(8)	5428(6)	7701(5)	820(4)	2,0(2)	2,8(3)	3,0(3)	0,1(2)	1,1(2)	−0,4(2)
C(9)	2256(5)	8643(4)	838(3)	1,6(2)	1,7(2)	1,5(2)	0,1(2)	0,6(2)	−0,1(2)
C(10)	1729(6)	9176(5)	−31(3)	2,3(2)	2,1(2)	1,5(2)	−0,3(2)	0,5(2)	0,4(2)
C(11)	1073(6)	5567(4)	1118(4)	2,6(2)	1,5(2)	2,1(2)	−0,7(2)	0,1(2)	0,1(2)
C(12)	1864(6)	5772(4)	456(3)	2,4(2)	1,6(2)	2,2(2)	−0,3(2)	0,3(2)	−0,9(2)
C(13)	1075(6)	6629(4)	−74(3)	3,1(3)	2,0(2)	1,4(2)	−0,8(2)	0,3(2)	−0,6(2)
C(14)	−181(6)	6952(4)	259(4)	2,0(2)	1,7(2)	2,5(2)	−0,4(2)	−0,4(2)	−0,4(2)
C(15)	−163(6)	6298(4)	997(4)	2,0(2)	2,2(2)	2,5(2)	−0,9(2)	0,7(2)	−0,3(2)

TABLE 3

COORDINATES (X10^3) AND B_{iso} (Å^2) OF HYDROGEN ATOMS

Atom	x	y	z	B_{iso}
H(10)	196(5)	992(4)	—3(3)	2(1)
H'(10)	226(6)	886(4)	—47(3)	2(1)
H''(10)	55(6)	910(4)	—27(3)	3(1)
H(11)	145(5)	510(4)	156(3)	2(1)
H(12)	280(6)	536(4)	41(3)	2(1)
H(13)	144(6)	691(4)	—59(4)	3(1)
H(14)	—85(5)	748(4)	6(3)	1(1)
H(15)	—88(5)	641(4)	137(3)	2(1)

Found: C, 43.39; H, 2.30; Fe, 30.56. $C_{20}H_{12}Fe_3O_8$, calcd.: C, 43.84; H, 2.21; Fe, 30.58%. IR: ν(C≡O) 2060, 2035, 1990, 1965; ν(CO, bridging) 1805; ν(C=C) 1350; ν(C=O) 1630 cm^{-1}. The third orange band gave 0.66 g (ca. 45%) of IIa, m.p. 129—134°C (dec., benzene/hexane, 1/3 mixture). Found: C, 35.64; H, 1.16; Fe, 33.75. $C_{30}H_{12}Fe_6O_{18}$, calcd.: C, 36.19; H, 1.22; Fe, 33.66%. IR: ν(C=O) 1625, ν(C=C) 1400 cm^{-1} (KBr); ν(C≡O) 2065, 2031, 2015, 1990, 1961 cm^{-1} (cyclohexane).

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