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IRON CARBONYL COMPLEXES CONTAINING AN AZOMETHYNE MOIETY

I. SYNTHESIS AND X-RAY STRUCTURE OF A NOVEL σ,π-ARENE-BRIDGED COMPLEX, μ-(o-$C_6H_4CH_2NC_6H_5$)$Fe_3(CO)_8$

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Summary

N-(Benzylidene)aniline (BZA) reacts with $Fe_3(CO)_{12}$ yielding μ-(o-$C_6H_4CH_2NC_6H_5$)$Fe_2(CO)_6$ (I), μ-(o-$C_6H_4CH_2NC_6H_5$)$Fe_3(CO)_8$ (II) and various products from the reduction and carbonylation of BZA and its fragments. II was characterized by X-ray crystallography and proved to be the first complex prepared with a $\sigma,\pi(\eta^1,\eta^6)$-bridging arene ligand.

Introduction

Iron carbonyls form a wide variety of complexes with nitrogen-containing ligands [1], and are able to *ortho*-metallate the *N*-substituted aromatic rings of those ligands [2–8]. This reaction usually yields binuclear complexes, e.g. Schiff base diiron complexes [2,3]. However, no trinuclear complexes have been prepared by this route.

In the present paper we report the first example of such a triiron complex, obtained from the simplest aromatic Schiff base: *N*-(benzylidene)aniline (BZA).

Results and discussion

Treatment of BZA with $Fe_3(CO)_{12}$ produced, besides complex I (previously prepared from $Fe_2(CO)_9$ [2,3]), a novel trinuclear complex, II (eq. 1).

Besides iron complexes, some products of the reduction and carbonylation of BZA and its fragments (viz., $PhNH_2$, $PhCH_2OH$, $PhCH_2NHPh$, PhNHNHPh and phthalimidine) were detected in the reaction mixture by gas-liquid chromatography

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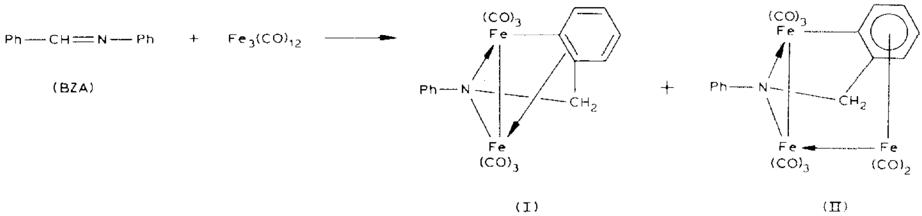


(1)

and mass spectroscopy. The presence of reduction products indicates that iron carbonyl hydrides are probably the intermediates of this reaction. As Andrews and Kaesz have found [9,10], such iron carbonyl hydrides may form in the reaction between iron carbonyls and organic nitriles, if the system contains a source of hydrogen. In contrast to reported data [11], we observed no promoting effect from tungsten carbonyl on this reaction.

Compound II is a brown crystalline substance, rather stable in the solid state and decomposing slowly in solution. It is poorly soluble in n-heptane and moderately soluble in benzene, chloroform and alcohols. A thermogravimetric study of II demonstrated that its thermal decomposition begins at 50°C without melting and proceeds in three stages: (1) slightly endothermic elimination of two CO groups at 50–140°C; (2) strongly endothermic loss of six CO groups and a half equivalent of Fe (the latter probably in the form of volatile $Fe(CO)_5$) per molecule of II, and a small part of organic ligand at 140–172°C; (3) complete elimination of the organic part of II, without any thermal effect at 172–250°C. The mass of the non-volatile

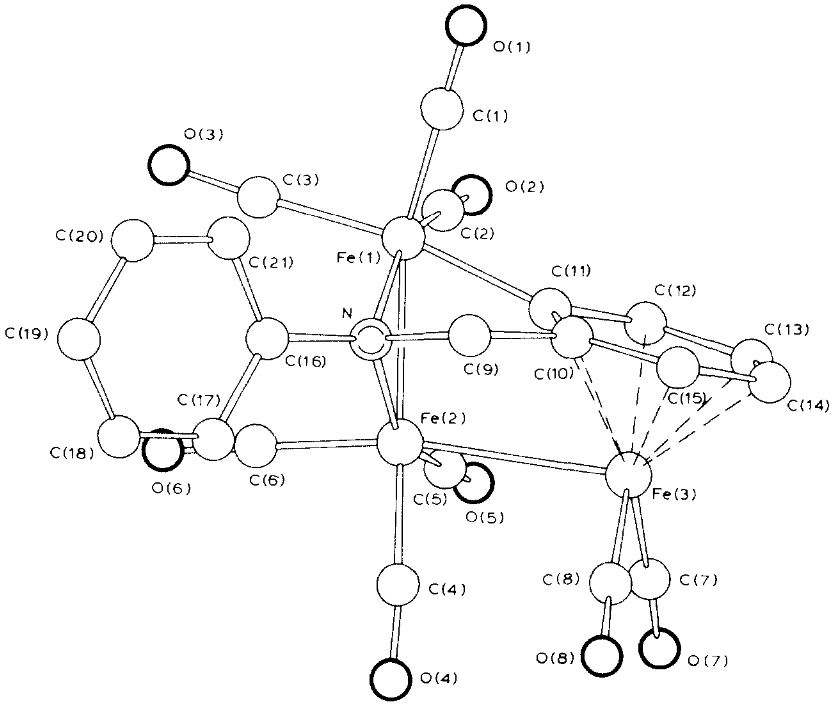


Fig. 1. Molecular structure of the complex II (hydrogen atoms are omitted).

residue corresponds to 2.5 equivalents of iron per molecule of II.

The molecular structure of II, determined by X-ray crystallography, is shown in Fig. 1. Bond lengths and angles are listed in Tables 1 and 2. The molecule contains a non-linear Fe(1)Fe(2)Fe(3) framework. All eight carbonyl groups are terminal, two are bonded to Fe(3) and three each to Fe(1) and Fe(2). The organic ligand bridges Fe(1) and Fe(2) by the μ_2 nitrogen atom (which thus achieves a tetrahedral environment) and bridges Fe(1) and Fe(3) by the C(10)···C(15) arene ring.

The Fe(1)–Fe(2) distance (2.452(1) Å) is somewhat longer than strong Fe–Fe bonds (2.37–2.42 Å) in diiron complexes bridged by two sp^3 hybridized nitrogen atoms [12]. The essentially equal Fe–μ-N distances (mean 2.010(6) Å) in II fall within the range observed in such complexes (1.96–2.02 Å), while the Fe(1)NFe(2) angle (75.2(2)°) is slightly larger than FeNFe angles (72.5–74.4°) in those complexes. The non-bridged Fe(2)–Fe(3) distance (2.822(1) Å) is ca. 0.4 Å longer than the Fe(1)–Fe(2) one, but is comparable with the non-bridged Fe–Fe bond length (2.787(2) Å) in $[(Ph_3P)_2N^+]_2[Fe_2(CO)_8{}^{2-}]\cdot 2CH_3CN$ [13] and with the Fe–Fe distance in $Fe_2(CO)_6\{\mu\text{-}P(CF_3)_2\}_2$. In the latter complex the existence of an Fe–Fe bond is confirmed by MO calculations and by its diamagnetism [14]. Thus, in II the Fe(2)–Fe(3) interaction can be regarded as a metal–metal bond.

The most unusual feature of II is the bridging function of the C(10)···C(15) aromatic ring. It is σ-bonded to Fe(1) via C(11) and is coordinated in a η^6-mode to Fe(3). To our knowledge, II is the fourth example of η^6-areneiron complexes studied structurally, the previously studied ones being $[Fe(\eta^6\text{-}C_6H_5CH_2CH_2CH_2\text{-}\eta^5\text{-}C_5H_4)][PF_6]$ [15], $(\eta^5\text{-}C_5H_5)Fe(\eta^6\text{-fluorenyl})$ [16], $(\eta^6\text{-}C_6H_5Me)Fe(\eta^5\text{-}2,3\text{-}Me_2\text{-}2,3\text{-}C_2B_9H_9)$ [17], and the first complex with a η^1,η^6-bridging arene. Previously, such a σ,π-bridging arrangement of aromatic ring had been reported when cyclopentadienyl acted as such a ligand [18].

The Fe(1)···Fe(3) distance (3.967(1) Å) is too long for covalent interaction.

The five carbons of the phenylene ring, C(11) to C(15), are coplanar within 0.005

TABLE 1

BOND LENGTHS (Å)

Bond		Bond		Bond	
Fe(1)–Fe(2)	2.452(1)	Fe(3)–C(11)	2.238(6)	N–C(16)	1.452(7)
Fe(2)–Fe(3)	2.822(1)	Fe(3)–C(12)	2.159(6)	C(9)–C(10)	1.481(9)
Fe(1)–C(1)	1.798(8)	Fe(3)–C(13)	2.135(7)	C(10)–C(11)	1.441(9)
Fe(1)–C(2)	1.795(7)	Fe(3)–C(14)	2.114(7)	C(10)–C(15)	1.408(9)
Fe(1)–C(3)	1.804(6)	Fe(3)–C(15)	2.118(6)	C(11)–C(12)	1.434(9)
Fe(1)–C(11)	1.969(6)	Fe(3)–Bz [a]	1.609	C(12)–C(13)	1.420(9)
Fe(1)–N	2.004(4)	C(1)–O(1)	1.131(9)	C(13)–C(14)	1.397(9)
Fe(2)–C(4)	1.793(7)	C(2)–O(2)	1.137(8)	C(14)–C(15)	1.416(9)
Fe(2)–C(5)	1.783(7)	C(3)–O(3)	1.146(8)	C(16)–C(17)	1.390(9)
Fe(2)–C(6)	1.775(7)	C(4)–O(4)	1.150(9)	C(16)–C(21)	1.388(9)
Fe(2)···C(11)	2.427(6)	C(5)–O(5)	1.143(9)	C(17)–C(18)	1.387(9)
Fe(2)–N	2.016(5)	C(6)–O(6)	1.141(8)	C(18)–C(19)	1.36(1)
Fe(3)–C(7)	1.762(7)	C(7)–O(7)	1.153(9)	C(19)–C(20)	1.38(1)
Fe(3)–C(8)	1.765(7)	C(8)–O(8)	1.143(9)	C(20)–C(21)	1.397(9)
Fe(3)–C(10)	2.102(6)	N–C(9)	1.507(8)	C–H	0.93–1.10, av. 1.05

[a] Bz is the centroid of the C(10)···C(15) benzene ring.

TABLE 2

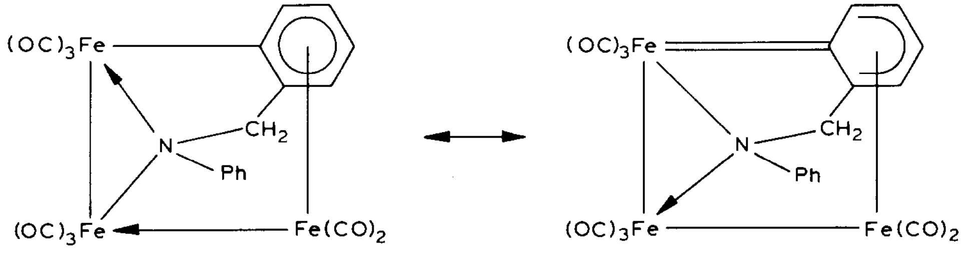
BOND ANGLES (°)

Angle		Angle		Angle	
Fe(2)Fe(1)N	52.6(1)	NFe(2)C(4)	111.6(3)	Fe(2)C(5)O(5)	178.4(6)
Fe(2)Fe(1)C(1)	150.5(2)	NFe(2)C(5)	151.8(3)	Fe(2)C(6)O(6)	178.5(6)
Fe(2)Fe(1)C(2)	101.1(2)	NFe(2)C(6)	92.2(3)	Fe(3)C(7)O(7)	176.8(7)
Fe(2)Fe(1)C(3)	104.9(2)	NFe(2)C(11)	67.8(2)	Fe(3)C(8)O(8)	177.7(7)
Fe(2)Fe(1)C(11)	65.5(2)	C(4)Fe(2)C(5)	95.7(3)	NC(9)C(10)	105.9(5)
NFe(1)C(1)	103.7(3)	C(4)Fe(2)C(6)	95.4(3)	C(9)C(10)C(11)	113.2(5)
NFe(1)C(2)	153.6(3)	C(4)Fe(2)C(11)	127.3(3)	C(9)C(10)C(15)	123.2(6)
NFe(1)C(3)	97.3(3)	C(5)Fe(2)C(6)	92.2(3)	C(11)C(10)C(15)	123.6(6)
NFe(1)C(11)	78.0(2)	C(5)Fe(2)C(11)	90.4(3)	Fe(1)C(11)Fe(2)	66.9(2)
C(1)Fe(1)C(2)	100.1(3)	C(6)Fe(2)C(11)	136.7(3)	Fe(1)C(11)C(10)	113.7(4)
C(1)Fe(1)C(3)	94.7(3)	Fe(2)Fe(3)C(7)	94.8(2)	Fe(1)C(11)C(12)	132.5(5)
C(1)Fe(1)C(11)	94.6(3)	Fe(2)Fe(3)C(8)	96.7(3)	Fe(2)C(11)C(10)	95.0(4)
C(2)Fe(1)C(3)	91.9(3)	C(7)Fe(3)C(8)	86.9(3)	Fe(2)C(11)C(12)	115.2(4)
C(2)Fe(1)C(11)	89.0(3)	Fe(2)Fe(3)Bz	96.8	C(10)C(11)C(12)	113.3(5)
C(3)Fe(1)C(11)	170.3(2)	C(7)Fe(3)Bz	137.5	C(11)C(12)C(13)	123.8(6)
Fe(1)Fe(2)Fe(3)	97.32(4)	C(8)Fe(3)Bz	131.7	C(12)C(13)C(14)	120.0(6)
Fe(1)Fe(2)N	52.2(1)	Fe(1)NFe(2)	75.2(2)	C(13)C(14)C(15)	118.9(6)
Fe(1)Fe(2)C(4)	163.4(2)	Fe(1)NC(9)	109.3(3)	C(10)C(15)C(14)	120.0(6)
Fe(1)Fe(2)C(5)	100.0(2)	Fe(1)NC(16)	124.7(4)	NC(16)C(17)	119.7(5)
Fe(1)Fe(2)C(6)	89.5(2)	Fe(2)NC(9)	112.3(3)	NC(16)C(21)	120.0(5)
Fe(1)Fe(2)C(11)	47.6(1)	Fe(2)NC(16)	123.9(4)	C(17)C(16)C(21)	120.1(6)
Fe(3)Fe(2)N	95.1(1)	C(9)NC(16)	108.0(4)	C(16)C(17)C(18)	118.8(6)
Fe(3)Fe(2)C(4)	79.2(2)	Fe(1)C(1)O(1)	178.2(6)	C(17)C(18)C(19)	122.2(7)
Fe(3)Fe(2)C(5)	82.8(2)	Fe(1)C(2)O(2)	177.2(6)	C(18)C(19)C(20)	119.0(7)
Fe(3)Fe(2)C(6)	172.1(2)	Fe(1)C(3)O(3)	175.9(6)	C(19)C(20)C(21)	120.7(7)
Fe(3)Fe(2)C(11)	49.8(1)	Fe(2)C(4)O(4)	171.3(6)	C(16)C(21)C(20)	119.3(6)

Å, or less than 1σ. The C(10) atom is tilted by 0.06 Å out of their mean plane (referred below as A) towards the exocyclic C(9) atom. The latter is tilted by 0.24 Å out of plane A and only by 0.06 Å out of the plane through C(10), C(11) and C(15). The arene ring is folded along the C(11)···C(15) direction by 5°, i.e. adopts a boat conformation to release the steric strain of the $\overline{\mathrm{Fe(1)C(11)C(10)C(9)N}}$ cycle. This strain manifests itself in the decrease of Fe(1)C(11)C(10) and C(9)C(10)C(11) angles to 113.7 and 113.2°, respectively, compared with the normal value of 120°. The Fe(3) atom is located at 1.62 Å from the plane A (in the same direction as C(9) and C(10)), while the Fe(1) atom is 0.28 Å from this plane in the opposite direction. A rather short intramolecular Fe(2)···C(11) distance of 2.427(6) Å is imposed by the overall molecular geometry and does not indicate a bonding interaction, as the environment of C(11) by Fe(1), C(10) and C(12) remains planar (the sum of bond angles is 359.5°).

Both the A and Fe(1)Fe(2)N planes are almost normal to the plane of the Fe_3-framework (the corresponding dihedral angles are 84.8 and 89.2°). The uncomplexed C(16)···C(21) phenyl ring is planar within 0.003 Å. The nitrogen atom is tilted by 0.11 Å (i.e. the N–C(16) bond is bent by 4.4°) out of the phenyl plane.

The bonding in II can be described as intermediate between the forms **1** and **2**. The contribution of **2** is indicated by the following features: (a) the Fe(3)–C(11) distance of 2.238(6) Å is significantly longer than the other Fe(3)–C(arene) distances (2.102–2.159 Å); (b) the C(10)–C(11) and C(11)–C(12) bonds are the longest ones in the complexed arene ring; (c) the Fe(1)–C(11) distance of 1.969(6) Å falls within the range found for Fe–C(carbene) double bonds (1.88–2.04 Å [19]), though some formally single Fe–C bonds were reported to have the same lengths (i.e. the Fe–C(pyridine) bond of 1.976(6) Å in $C_5H_4N(CO)_6Fe_2SFe_2(CO)_6SC_5H_4N$ [20]).



(1) (2)

In both structures **1** and **2** each iron atom achieves an 18-electron configuration, in accordance with diamagnetism of II.

Experimental

^1H NMR spectra were recorded with a Bruker WH-90 instrument (90 MHz) in $CDCl_3$ with TMS as internal standard, IR spectra with a UR-20 spectrophotometer in CCl_4 and mass spectra with a LKB 2091 instrument (direct injection of the ion source at 50°C, ionization energy of 12 eV and emission current of 25 μA). The thermogravimetric study was carried out with a Paulik-Paulik-Erdey (Hungary) derivatograph under argon, using a heating rate of 3°C min^{-1} and calcined Al_2O_3 as standard.

An X-ray diffraction experiment was carried out with a Syntex $P2_1$ diffractometer at − 120°C, using graphite-monocromated Mo-K_α radiation.

Crystal data: II, $C_{21}H_{11}Fe_3NO_8$, $M = 572.9$, orthorombic, at − 120°C $a = 7.520(1)$, $b = 14.338(3)$, $c = 19.062(4)$ Å, $V = 2055.2(6)$ $Å^3$, $Z = 4$, $d_{calcd.} = 1.85$ g cm^{-3}, space group $P2_12_12_1$.

1943 independent reflections with $I \geqslant 2\sigma$ were measured by the $\theta/2\theta$ scan technique ($2\theta \leqslant 50°$). The structure was solved by direct methods and refined by full matrix least squares with anisotropic thermal parameters for all non-hydrogen atoms to give $R = 0.055$. Further refinement (with inclusion of anomalous dispersion terms for Fe) of the two enantiomeric models converged to $R = 0.0452$, $R_w = 0.0490$ and $R = 0.435$, $R_w = 0.0482$, respectively. All hydrogen atoms were located from a difference Fourier map and included in the calculation of structure factors as fixed contributions (see Table 4). Further refinement (after discarding the strong, extinction-affected 002 reflection) converged to $R = 0.0347$, $R_w = 0.0318$ and $R = 0.0327$, $R_w = 0.0303$ for the two models. Thus, according to the Hamilton test [21], the absolute configuration of II corresponds to the second enantiomeric model with a probability of more than 99.5%. Final positional and thermal atomic parameters are listed in Tables 3 and 4.

TABLE 3. ATOMIC COORDINATES ($\times 10^4$), ANISOTROPIC AND EQUIVALENT ISOTROPIC THERMAL FACTORS[a]

Atom	x	y	z	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}	B_{iso}^{eq} ($\times 10$)
Fe(1)	6258(1)	−767.5(6)	2448.8(4)	20.8(4)	15.6(3)	17.7(3)	0.5(3)	0.0(3)	1.1(3)	18.0(2)
Fe(2)	8397(1)	434.7(6)	2096.0(5)	13.7(4)	20.5(4)	18.6(3)	1.4(3)	−0.3(3)	0.5(3)	17.6(2)
Fe(3)	6925(1)	821.4(7)	765.4(5)	17.2(4)	21.8(4)	18.9(3)	−0.6(4)	−1.4(3)	4.5(3)	19.3(2)
O(1)	2600(7)	−1436(3)	2608(3)	30(3)	32(2)	50(3)	−7(2)	3(2)	8(2)	37(2)
O(2)	7890(8)	−2418(3)	1809(2)	44(3)	24(2)	35(2)	10(2)	4(2)	−5(2)	34(1)
O(3)	7335(8)	−1372(4)	3865(2)	49(3)	46(3)	23(2)	22(3)	1(2)	2(2)	39(2)
O(4)	10299(8)	2176(4)	1791(3)	39(3)	34(3)	47(3)	−16(2)	−1(3)	5(2)	40(2)
O(5)	10852(7)	−715(4)	1278(2)	31(3)	47(3)	31(2)	16(3)	4(2)	−4(2)	36(1)
O(6)	10416(7)	113(4)	3379(3)	25(2)	68(3)	24(2)	5(3)	−4(2)	3(2)	39(2)
O(7)	10347(7)	856(4)	49(3)	25(2)	53(3)	38(3)	−4(3)	7(2)	2(2)	38(2)
O(8)	7275(9)	2840(4)	813(3)	57(4)	24(4)	86(4)	−3(3)	−16(4)	14(3)	56(2)
N	6099(7)	608(3)	2625(2)	19(2)	14(2)	15(2)	3(2)	−2(2)	−1(2)	16(1)
C(1)	4004(10)	−1164(4)	2549(4)	32(4)	12(2)	30(3)	−4(3)	8(3)	7(2)	25(2)
C(2)	7249(10)	−1790(4)	2072(3)	26(3)	23(3)	19(3)	−3(3)	−2(3)	3(2)	23(2)
C(3)	6931(11)	−1105(4)	3321(3)	32(4)	23(3)	21(3)	4(3)	−2(3)	−3(2)	25(2)
C(4)	9433(10)	1531(5)	1904(4)	24(3)	29(3)	31(3)	2(3)	3(3)	−2(3)	28(2)
C(5)	9902(10)	−255(5)	1593(4)	19(3)	30(3)	20(3)	−2(3)	−3(3)	0(3)	23(2)
C(6)	9607(9)	234(5)	2881(4)	16(3)	35(3)	29(3)	8(3)	3(3)	−5(3)	26(2)
C(7)	8984(10)	818(6)	324(3)	31(4)	32(3)	19(3)	−4(3)	−2(3)	0(3)	27(2)
C(8)	7140(11)	2046(5)	809(4)	27(4)	30(3)	44(4)	−3(3)	0(3)	9(3)	34(2)
C(9)	4654(9)	1017(4)	2171(3)	17(3)	12(3)	28(3)	3(2)	−1(3)	4(2)	19(2)
C(10)	4843(8)	567(4)	1476(3)	16(3)	18(3)	18(2)	−1(2)	0(3)	1(2)	18(2)
C(11)	5891(9)	−274(4)	1496(3)	15(3)	23(3)	16(3)	−4(3)	−2(2)	2(2)	18(2)
C(12)	6274(9)	−644(4)	814(3)	25(3)	19(3)	24(3)	−3(3)	0(3)	2(2)	22(2)
C(13)	5629(10)	−250(5)	180(3)	23(3)	31(3)	19(3)	−6(3)	0(3)	1(2)	25(2)
C(14)	4567(9)	549(5)	198(3)	23(3)	29(3)	17(3)	−2(3)	−4(2)	6(2)	23(2)
C(15)	4128(9)	945(5)	856(4)	14(3)	25(3)	34(3)	1(3)	−4(3)	1(3)	24(2)
C(16)	5978(9)	1035(4)	3314(3)	19(3)	19(3)	20(2)	5(2)	0(2)	−1(2)	19(2)
C(17)	7054(10)	1793(4)	3477(3)	27(4)	23(3)	24(3)	−2(3)	−4(3)	−1(2)	25(2)
C(18)	6818(11)	2236(5)	4117(4)	36(4)	21(3)	32(3)	2(3)	−1(3)	−5(2)	30(2)
C(19)	5570(11)	1954(5)	4589(3)	43(4)	25(3)	21(3)	7(3)	0(3)	−7(3)	30(2)
C(20)	4505(11)	1200(5)	4426(3)	36(4)	33(3)	19(3)	6(3)	5(3)	2(3)	29(2)
C(21)	4703(9)	732(5)	3788(3)	23(3)	24(3)	27(3)	3(3)	3(3)	1(3)	24(2)

[a] In the form $T = \exp[-0.025(B_{11}h^2a^{\star 2} + B_{22}k^2b^{\star 2} + B_{33}l^2c^{\star 2} + 2B_{12}hka^{\star}b^{\star} + 2B_{13}hla^{\star}c^{\star} + 2B_{23}klb^{\star}c^{\star})]$ and $B_{iso}^{eq} = \frac{1}{3}\Sigma_i\Sigma_j B_{ij}a_i^{\star}a_j^{\star}(\vec{a}_i\vec{a}_j)$.

TABLE 4

COORDINATES ($\times 10^3$) OF HYDROGEN ATOMS [a]

Atom	x	y	z	Atom	x	y	z
H(C9)	338	86	243	H(C17)	784	210	305
H′(C9)	474	174	214	H(C18)	740	280	418
H(C12)	712	−117	80	H(C19)	549	237	506
H(C13)	615	−51	−29	H(C20)	355	88	480
H(C14)	415	89	−26	H(C21)	398	16	368
H(C15)	325	155	94				

[a] Isotropic thermal assigned to each H atom is $B = B_{\text{iso}}^{\text{eq}} + 1$, where $B_{\text{iso}}^{\text{eq}}$ is that of the corresponding carbon atom.

Reaction of BZA with $Fe_3(CO)_{12}$

A mixture of 18.1 g (0.1 mol) of BZA and 70.46 g (0.14 mol) of $Fe_3(CO)_{12}$ in 500 ml n-heptane was heated to 80°C and stirred until complete dissolution of $Fe_3(CO)_{12}$ (for 24 hours). The hot mixture was filtered, the precipitate was washed with hot benzene and the washings were combined with the filtrate. The filtrate was rotary evaporated, and the dry residue was dissolved in 300 ml of petroleum ether, b.p. 40–70°C. The solution was kept at −5°C for 16 hours, the crystalline precipitate formed was filtered off, washed with cold petroleum ether and dried, yielding 2.23 g (4%) of II. The sample of II used for elemental analysis was prepared by recrystallization from benzene. Found: C, 44.11; H, 1.94; Fe, 29.07; N, 2.45. $C_{21}H_{11}Fe_3NO_8$, calcd.: C, 44.03; H, 1.94; Fe, 29.25; N, 2.44%. According to thermogravimetry decomposition begins at 50°C. IR spectrum: $\nu(C\equiv O)$ 1963, 1978, 1988, 2000, 2023, 2032, 2062, 2071 cm^{-1}. Mass spectrum, m/e: 573 (M), 545, 517, 489, 461, 433, 405, 377, 349, 321, 293, 269, 237, 209, 183, 182, 181, 93, 92, 78. ^1H NMR spectrum: δ 2.45 (d, 1H, CH_2), 3.88 (d, 1H, CH_2) ppm, AB system, J_{AB} 14.8 Hz; δ 4.235 (d, 1H, C_6H_4), 4.88 (d, 1H, C_6H_4), 5.33 (t, 1H, C_6H_4), 6.71 (t, 1H, C_6H_4) ppm, ABKM system, J_{AK} 5.9 Hz, J_{KM} 5.9 Hz, J_{BM} 5.9 Hz; δ 7.16 ppm (s, 5H, C_6H_5).

After isolation of II the filtrate was rotary evaporated to give 70 ml of concentrated solution, which was cromatographed on an Al_2O_3 column (d = 35 mm, l = 1400 mm). 15.97 g of I was eluted with petroleum ether. Yield 35%, m.p. 80–81°C, cf. 82-82.5°C according to the literature [3]. The mixture of substances not containing iron was subsequental eluted with acetone. After removal of acetone, 10 g of a liquid sample was obtained which was investigated by gas-liquid chromatography and mass spectroscopy and was found to consist of (yield, %): aniline (20), benzyl alcohol (10), phenylbenzylamine (10), phthalimidine (5), hydrazobenzene (3), BZA (2) and non-identified substances (5).

When the reaction was performed under the same conditions in the presence of tungsten carbonyl, no increase in yields of I and II was observed.

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