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REACTIONS OF ETHYNYLFERROCENE AND 3,3,3-TRIFLUOROPROPYNE WITH $(PPh_3)_2Ni(CO)_2$, $Co_2(CO)_8$ AND *trans*-$(PPh_3)_2IrCl(CO)$*

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Summary

Ethynylferrocene (FcC≡CH, Fc = ferrocene) oligomerized to linear dimer FcCH=CHC≡CFc (V), linear trimer FcCH-CHC(=CHFc)C≡CFc and 1,2,4-triferrocenylbenzene in the presence of $(PPh_3)_2Ni(CO)_2$ and its polymer-anchored analogs. The rate of oligomerization decreased as the crosslink density of the anchored catalysts increased, but product distribution remained the same as when the soluble catalyst was used. 3,3,3-Trifluoropropyne did not oligomerize at temperatures up to 150°. Ethynylferrocene was converted to μ-ferrocenylacetylenehexacarbonyldicobalt (VIII) upon reaction with one equivalent of $Co_2(CO)_8$. Reaction of ethynylferrocene with *trans*-$(PPh_3)_2IrCl(CO)$ gave V, VI and six-coordinate $CO(Cl)(FcC{\equiv}C)H(PPh_3)_2Ir^{III}$ (IX). Compound IX exhibited a $\Delta\nu(CO)$ of 42 cm^{-1} relative to Vaska's complex, just below the "easily reversible" category of Vaska, and it could not be isolated in pure form due to facile reductive elimination of ethynylferrocene. 3,3,3-Trifluoropropyne underwent oxidative addition with *trans*-$(PPh_3)_2IrCl(CO)$ to give six-coordinate $CO(Cl)(CF_3C{\equiv}C)H(PPh_3)_2Ir^{III}$ but no polymer was formed. Isolated X exhibited a $\Delta\nu(CO)$ of 85 cm^{-1}, placing it in the "stable-reversible" category.

Introduction

The cyclotrimerization of acetylenes to aromatic compounds over $(PPh_3)_2$-$Ni(CO)_2$, discovered by Reppe [1], was extensively investigated by Meriwether et al. [2—5]. In general, alkylacetylenes gave linear oligomers with only low yields of aromatics. Arylacetylenes and acetylenic alcohols gave linear or aromatic oligomers as the major product, depending on reaction conditions, and acetylenic ethers, ketones and esters gave almost entirely aromatic cyclotrimerization products in >85%. The unsymmetrical 1,2,4-substitution pattern predominated. Thus,

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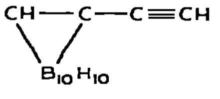
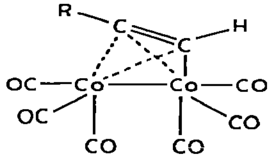
electron-withdrawing groups promoted cyclotrimerization. Even the bulky carboranyl compound I, which is an electron-deficient alkyne, cyclotrimerized in 85% yield [6]. Attempts to effect the cyclotrimerization of the very electron deficient 3,3,3-trifluoropropyne have not been reported. The bulky, electron rich, ethynylferrocene was converted to 1,2,4-triferrocenylbenzene [7,8] in 64% yield upon reaction with $Co_2(CO)_8$ in refluxing dioxane [7]. These reactions are of interest since they represent extreme electronic effects versus Meriwether's order of reactivity.

Acetylenes generally react with $Co_2(CO)_8$ by displacing two bridging carbonyl groups to give a μ-alkynehexacarbonyldicobalt (II) [6,9]. However, with *trans*-$(PPh_3)_2IrCl(CO)$, monosubstituted acetylenes give six-coordinate hydridoacetylides (III) via oxidative addition [10]. An exception was I which gave the very unusual 2/1 adduct IV, where R represents the carborane cage. These results suggested the reaction of ethynylferrocene with both $Co_2(CO)_8$ and *trans*-$(PPh_3)_2$-$IrCl(CO)$ should be undertaken. Furthermore, the reaction of $CF_3C{\equiv}CH$ with Vaska's complex should be studied. Previously, the reaction of $CF_3C{\equiv}CH$ with $Co_2(CO)_8$ was shown to give a mixture of 1,2,4- and 1,3,5-tris(trifluoromethyl)-benzenes as well as the bridged complexes $Co_2(CO)_6(CF_3C_2H)$, $Co_3(CO)_9C_3H_2F_3$, and $Co_2(CO)_4(CF_3C_2H)_3$ [11].



(I)



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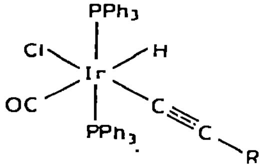

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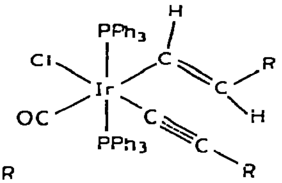


(IV)

Continuing our previous studies of ethyl propiolate cyclooligomerization by polymer-anchored $(PPh_3)_2Ni(CO)_2$ to 1,2,4- and 1,3,5-benzene derivatives and tetrasubstituted cyclooctatetraenes [12], this anchored catalyst was used in the current work. The preparation and swelling of these anchored catalysts was reported elsewhere [12].

Results and discussion

Benzene or THF solutions of ethynylferrocene reacted with both homogeneous and polymer-anchored $(PPh_3)_2Ni(CO)_2$ at 80°. Thin layer chromatography (silica and alumina) indicated a mixture of three products plus a tar. These were separated by dry chromatography (silica gel, benzene/petr. ether, 1/4). As shown in eqn. 1 the linear dimer 1,4-diferrocenylbutyn-3-ene (V), the acyclic trimer VI and 1,2,4-triferrocenylbenzene were produced. In every case trimer VI was the major product (yields are summarized in Table 1). High pressure analytical liquid chromatography (isooctane/$CHCl_3$, Corasil I) confirmed that the fractions V and VI were pure compounds. These results place ethynylferrocene between alkylacetylenes and phenylacetylene with respect to Meriwether's order of reactivity [2]. That is, the product distribution resembled that observed with alkylacetylenes while the overall conversion resembled that obtained with phenylacetylene.

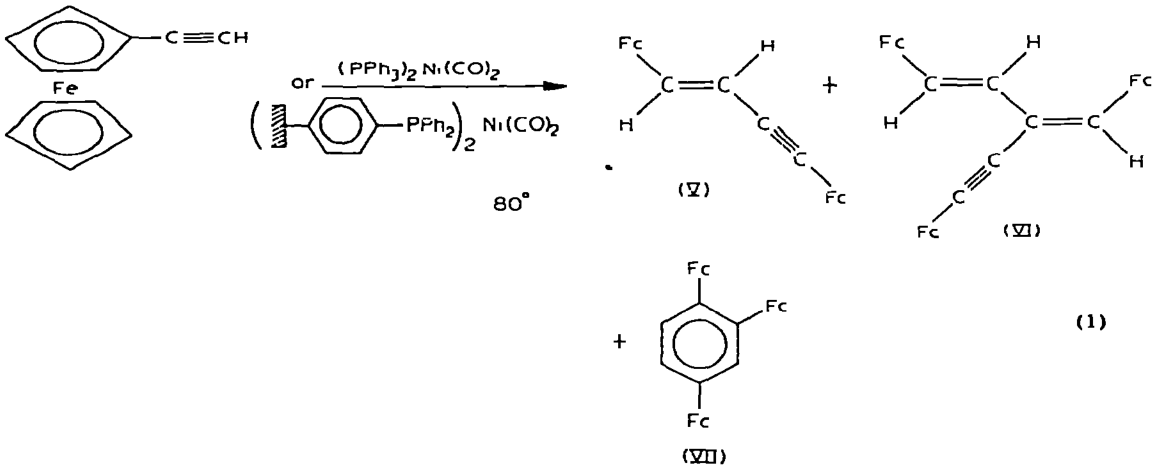
The product distributions using the bound or soluble catalyst were similar. However, the yields differed. That the catalyst bound to the 1% divinylbenzene-

TABLE 1

OLIGOMERIZATION OF ETHYNYLFERROCENE CATALYZED BY SOLUBLE AND POLYMER ANCHORED $(PPh_3)_2Ni(CO)_2$[a]

Catalyst	mmol catalyst[b]	Overall yield	V % yield	VI % yield	VII % yield	Recovered ethynyl-ferrocene
$(PPh_3)_2Ni(CO)_2$	0.088	81.5	9.8	44.4	9.5	18.5
Catalyst anchored on 2% divinylbenzene crosslinked polystyrene	0.155	44.6	5.9	27.8	6.2	55.4
recycled	0.155	43.2	8.3	21.6	8.9	56.8
Catalyst anchored on 1% divinylbenzene	0.179	100.0	18.1	55.8	15.4	0
recycled	0.179	23.4	3.1	13.7	3.8	76.6

[a] Reactions run for 24 h at 80°. Yields are mole percents based on ethynylferrocene (1.2 g, 6.2 mmol used in each run), and are averaged for three runs. Tar formation accounted for the difference in overall yield and the combined yield of isolated products. [b] Actual mmols of nickel sites available to reactants.



(1)

crosslinked polymer gave higher product yields in 24 h at 80° was a manifestation of the greater catalyst concentration used. Actually, the rates using this bound catalyst and the soluble catalyst were approximately equal. Thus, diffusion into the resin beads (when a 1% crosslinked resin was used) does not appear to be significantly retarding the rate. However, diffusion was a major factor when the resin was 2% crosslinked. As can be seen in Table 1, the yields after 24 h at 80° were substantially lower. The maximum molar turnover of each of these catalysts was between 900 and 1000, after which the rate dropped precipitously (see Table 1).

Linear dimer V was characterized by analysis, its mass spectral parent ion, *m/e* 420, the presence of a carbon—carbon triple bond absorption at 2160 cm^{-1}, and its NMR spectrum which exhibited *J* 16 Hz for the *trans* olefinic proton coupling. Trimer VI exhibited a parent ion at *m/e* 630, a carbon—carbon triple bond absorption at 2160 cm^{-1} and its 270 MHz proton NMR spectrum showed a *J* 16

TABLE 2

ANALYTICAL RESULTS OF THE DEACTIVATED POLYMER ANCHORED CATALYSTS USED IN ETHYNYLFERROCENE OLIGOMERIZATION

Polymer	P (%)	Ni (%)	Fe (%)	Mole ratio Ni/Fe
2 % crosslinked	1.83	1.64	1.58	0.98
1% crosslinked sample A	2.16	1.78	1.75	0.97
1% crosslinked sample B	2.22	1.50	1.43	1.00
2% crosslinked[a]	—	1.42	0.36	3.75

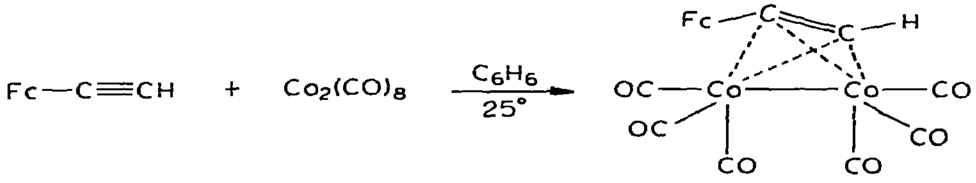
[a] Analyzed after reacting with a 400 mol excess of ethynylferrocene. This catalyst still exhibited CO bands at 1997 and 1943 cm^{-1}, though their intensity was decreased relative to their intensities before use.

Hz coupling (the *trans* olefinic protons) and a lone olefinic proton with an allylic coupling of less than 0.5 Hz. While the (*E*,*Z*) isomer VI was the assigned structure, the (*E*,*E*) isomer cannot be ruled out. 1,2,4-Triferrocenylbenzene also exhibited a parent ion at *m*/*e* 630 and its IR and NMR spectra and m.p. were identical to those of authentic samples [13,14].

The deactivated resin-anchored catalyst exhibited no carbonyl absorption (IR) and analysis confirmed a 1/1 Ni/Fe ratio, suggesting a complex of unknown structure had formed. Heating or photolyzing the deactivated catalyst in benzene in the presence of 400 psi of carbon monoxide failed to regenerate an active species.

In contrast to other electron deficient alkynes, 3,3,3-trifluoropropyne was inert to oligomerization in the presence of $(PPh_3)_2Ni(CO)_2$ or its polymer-bound analogs at 90, 120 and 130°. Even at 150° no reaction took place. Attempts to promote the reaction with phenylacetylene failed to oligomerize $CF_3C{\equiv}CH$, while the phenylacetylene itself was oligomerized quantitatively. This lack of reactivity of $CF_3C{\equiv}CH$ was surprising in view of its ready cyclotrimerization with $Co_2(CO)_8$ [9] and its linear polymerization by Rh [11] and Pt [15] derivatives.

Ethynylferrocene reacted readily with an equivalent of $Co_2(CO)_8$ in benzene at 25° under nitrogen to give a 53% yield of μ-ferrocenylacetylenehexacarbonyldicobalt, isolated by column chromatography as green, air stable crystals (eqn. 2). The mass spectrum of VIII exhibited a parent ion at *m*/*e* 496 consistent with



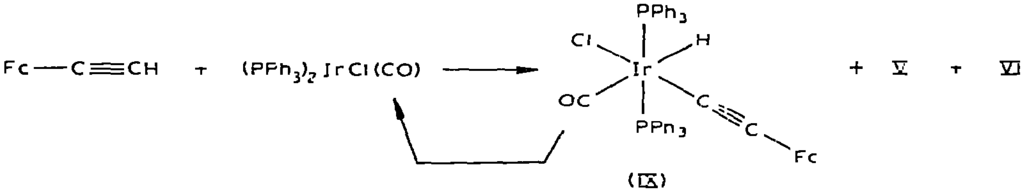
(2)

(VIII)

$[C_{18}H_{10}Co_2FeO_6]^{+}$ and major peaks at *m*/*e* 468, 440, 412, 384, 356 and 328 which represented the successive loss of six carbonyl groups. Carbonyl absorptions at 2045 cm^{-1} and a broad band between 2020 and 1855 cm^{-1} in addition to the bridging "acetylene" absorption at 1765 cm^{-1} were observed. Its NMR spectrum exhibited the acetylenic proton as a singlet at δ 6.27, a multiplet (4H) at δ 4.36 for the substituted Cp protons, and a singlet (5H) at δ 4.15 ppm for the unsubstituted Cp protons. Analysis was in accord with the formulation VIII.

These results differ from the cyclotrimers previously reported to result from the reaction of $Co_2(CO)_8$ with ethynylferrocene [7,8]. However, the previous studies were carried out in different solvents and at temperatures 35° higher than our synthesis of VIII. This suggests VIII was an intermediate in the formation of VII in Rosenblum's [7] studies. Refluxing VIII and excess ethynylferrocene in dioxane gave a 32% (based on VIII) yield of VII, in agreement with the possible intermediacy of VIII in the previous work [7,8].

Treating ethynylferrocene with Vaska's complex in benzene give linear dimer V (5%), trimer VI (26%) and a brown complex with structure IX. While IX was never isolated in pure form, its IR spectrum exhibited a very strong sharp band at



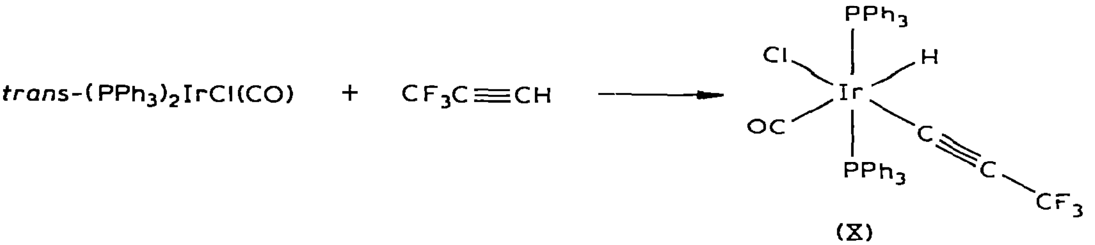
(IX)

(3)

1997 cm^{-1} with a shoulder at 1993 cm^{-1} (this corresponding to Vaska's complex). The 810 cm^{-1} band of ferrocene's cyclopentadienyl rings was seen in the spectrum. Thus two species, Vaska's complex and a six-coordinate Ir^{III} complex, were present. The six-coordinate Ir^{III} complexes are known [16,17] to have carbonyl bands shifted from 10 to 115 cm^{-1} to higher frequencies as a result of a higher charge on Ir which diminishes back-donation from Ir to the antibonding CO orbitals.

Attempts to recrystallize IX failed due to its continued equilibration to $(PPh_3)_2IrCl(CO)$ and ethynylferrocene. After each attempt, the intensity of the 1997 and 810 cm^{-1} bands decreased rapidly. High pressure liquid chromatography gave back only starting materials. The formation and dissociation of IX parallels the results of Collman [16] and Vaska [17]. Vaska classified these oxidative addition products into three categories: "easily reversible" (showing high frequency $\Delta\nu(CO)$ values, 48 to 67 cm^{-1}), "stable-irreversible" (exhibiting $\Delta\nu(CO)$ values, 68 to 100 cm^{-1}), and "stable-reversible" (with $\Delta\nu(CO)$ values, 100 to 115 cm^{-1}). Ethynylferrocene adduct IX exhibited a $\Delta\nu(CO)$ value of 42 cm^{-1}, just below the lower limit of the "easily reversible" category. Thus, IX easily underwent reductive elimination during purification attempts.

Reaction of 3,3,3-trifluoropropyne with Vaska's complex had not been reported, thus it was carried out in the hope of obtaining a 1/2 complex similar to Hawthorne's complex IV. However, when an excess of the alkyne was used, it



(X)

(4

reacted to give the stable six coordinate hydridoacetylide X (eqn. 4) in 83% yield. No polymer was formed. Complex X was a tan powder exhibiting IR bands at 2135 cm^{-1} for the acetylenic triple bond, a sharp band at 2060 cm^{-1} assigned to the Ir—H stretch and a very strong 2040 cm^{-1} band typical of a six-coordinate iridium-bound carbonyl. A parent ion at *m/e* 873 corresponds to $[C_{40}H_{31}ClF_3IrOP_2]^+$ and strong peaks at *m/e* 845 and 751 from successive loss of a carbonyl and a trifluoropropyne moiety. Analysis was satisfactory. The $\Delta\nu$(CO) shift of 85 cm^{-1} relative to Vaska's complex places X in the "stable-reversible" category.

Experimental

Benzene, THF and toluene were dried over CaH_2 for at least 24 h and then distilled under nitrogen. Similar care was taken to dry all solvents. Nitrogen and carbon monoxide were obtained commercially (>99% purity) and used as received. Organometallic complexes were obtained from Strem or Pressure Chemical Co. GLC separations were done on a Varian Model 90-P gas chromatograph using Carbowax 20-M or SE-30 (15% on 100-120 NAW Chromasorb P, 6′ × ¼″). The IR, ^{1}H NMR, UV, and mass spectra were obtained on a Beckman Ir-33, Perkin—Elmer R20B or Varian HA-100, Cary 14, and a Perkin—Elmer—Hitachi RMU-6M, respectively. The preparation of polymer-bound $(PPh_3)_2Ni(CO)_2$ has been described [10] and commercial (Strem Chemicals) $(PPh_3)_2Ni(CO)_2$ was used. The preparation and purification of ethynylferrocene was previously given [7,8]. The method of Finnegan and Norris [18] was used to prepare $CF_3C{\equiv}CH$.

Reaction of ethynylferrocene with bis(triphenylphosphine)nickel dicarbonyl

Ethynylferrocene (1.30 g, 6.2 mmol), bis(triphenylphosphine)nickel dicarbonyl (0.050 g, 0.07 mmol) and 20 ml of dry, nitrogen-saturated benzene were heated at reflux under nitrogen for 24 h. At this point, TLC (on silica, 20% benzene in 30—60° pet. ether) indicated the presence of three new products and unreacted ethynylferrocene. Separation by normal column chromatography was unsuccessful. Separation by "dry column" [19] chromatography (25 mm × 5′, silica) was successful. Yields were 81% overall (based on ethynylferrocene). The product consisted of a dimer V (9.8%), trimer VI (44.4%) and 1,2,4-triferrocenylbenzene (9.5%). Dimer V was recrystallized from CH_2Cl_2 and pet. ether: m.p. 227—229°; mass spec. (70 eV) *m/e* 420, 356, 300, 299, 234, 210; IR (KBr) 3060 m, 2170 w, 1615 m, 1100 s, 1000 s, 950 s, 822 s, 810 s cm^{-1}; NMR ($CDCl_3$) δ 6.71 (d, 1H, *J* 16 Hz, olefinic proton), δ 5.83 (d, 1 H, *J* 16 Hz, olefinic proton) and δ 4.14—4.40 ppm (complex, 18 H, cyclopentadienyl protons). Found: C, 68.18; H, 4.91; Fe, 25.00. $C_{24}H_{20}Fe_2$ calcd.: C, 68.61; H, 4.80; Fe 26.59%.

Trimer VI was recrystallized from $CHCl_3$/MeOH; m.p. 180—182°; mass spec. (70 eV) *m/e* 630, 564, 445, 389, 324, 316, 265; IR (KBr) 3060 m, 2160 w, 1610 m, 1100 s, 1000 s, 810 s cm^{-1}; 270 MHz NMR ($CDCl_3$) δ 4.01—4.35 (complex pattern, 27 H, cyclopentadienyl protons), δ 6.59 (d, 1 H, *J* 16 Hz, olefinic proton), δ 6.24 (s, 1 H, small allylic coupling less than 0.5 Hz, olefinic proton), δ 5.90 (d, 1 H, *J* 16 Hz, olefinic proton). Found: C, 68.61; H, 4.81; Fe, 26.59. $C_{36}H_{30}Fe_3$ calcd.: C, 68.61; H, 4.80; Fe, 26.59%.

The 1,2,4-triferrocenylbenzene was recrystallized from methylene chloride/pet. ether; m.p. 245—247° (lit. [13] 247—250°); mass spec. (70 eV) *m/e* 630, 420, 117, 115; IR (KBr) 3060 m, 1610 w, 1100 s, 1000 s, 810 s, 750 m cm^{-1};

NMR ($CDCl_3$) δ 7.26—7.96 (complex, 3 H, aromatic CH) and δ 4.06—4.78 (complex, 27 H, cyclopentadienyl protons) identical to that reported [13]. Found: C, 68.14; H, 5.02; Fe, 26.33. $C_{36}H_{30}Fe_3$ calcd.: C, 68.61; H, 4.80; Fe, 26.59%.

Reactions with the polymer-bound catalyst (0.50 g, 0.179 mmol Ni) were conducted in the same manner. Bound catalysts were recycled by filtration under nitrogen after each reaction. The polymeric catalyst then was washed with benzene, dried in vacuum, and carried through the same reaction procedure again.

Reaction of ethynylferrocene with dicobalt octacarbonyl

A solution of $Co_2(CO)_8$ (2.10 g, 6.15 mmol) was treated with ethynylferrocene (1.3 g, 6.2 mmol) in dry benzene (60 ml, nitrogen saturated), which had been filtered to remove insoluble oxides. Gas evolution began immediately. Under nitrogen flow, the solution was stirred at 25° for 24 h. The solvent was evaporated and the residue chromatographed on 1" × 12" column of silica. Elution of a dark green band with nitrogen-saturated pet. ether (30—60°), and recrystallization from dry, nitrogen-saturated methanol gave dark green air-stable crystals, 1.636 g, 52.5% yield, m.p. 64—66°: NMR ($CDCl_3$) δ 6.27 (s, 1 H, vinyl proton), δ 4.15 (s, 5 H, unsubstituted cyclopentadienyl protons) and δ 4.36 (m, 4 H, substituted cyclopentadienyl protons); IR (KBr) 3060 m, 2045 vs, 2020—1855 vs (br), 1765 w, 1408 m, 1388 m, 1192 m, 1100 s, 1031 s, 1021 s, 1000 m, 850 s, 820 s, 810 s, 763 m, 705 s cm^{-1}; mass spec. (60 eV) *m/e* 496, 468, 440, 412, 384, 356, 328, 210. Found: C, 43.42; H, 2.08; Co, 23.62; Fe, 11.36; O, 19.51. $C_{18}H_{10}Co_2FeO_6$ calcd.: C, 43.44; H, 2.03; Co, 24.04; Fe, 11.22; O, 19.29%.

Reaction of ethynylferrocene with trans-chlorocarbonylbis(triphenylphosphine)iridium

trans-Chlorocarbonylbis(triphenylphosphine)iridium (1.0 g, 1.28 mmol), ethynylferrocene (1.3 g, 6.2 mmol) and dry benzene (60 ml, nitrogen-saturated) were heated at 80° under N_2 for 24 h with stirring. TLC showed two ethynylferrocene oligomers but only a long smear for a possible organometallic complex. The solution was evaporated and chromatographed on a silica column (1" × 12"). Benzene/pet. ether (1/9) eluted unreacted ethynylferrocene (0.544 g, 41.8%). Benzene/pet. ether (1/1) eluted dimer V (0.063 g, 4.9%, parent ion *m/e* 420) and linear trimer VI (0.344 g, 26.4%, parent ion *m/e* 630). Benzene eluted 0.683 g of a brown compound containing both iridium and ferrocene. Recrystallization attempts were unsuccessful. Precipitation from a THF solution with methanol (all under nitrogen) gave a light brown powder, IR (KBr) 2000 vs, 1991 m, 1955 s, 810 s cm^{-1}; whose analysis showed 1.61% Fe and 16.06% Ir. Upon repeated precipitation, a yellow powder was obtained; IR (KBr) 2000 m, 1911 w, 1955 vs, 810 vw cm^{-1}. Found: C, 56.80; H, 4.00; Cl, 4.66; Fe 0.20; Ir, 19.42; P, 7.69. $(Ph_3P)_2IrCl(CO)$ calcd.: C, 56.95; H, 3.88; Cl, 4.54; Ir, 24.64; P, 7.94%.

An analytical liquid chromatography spectrum of the yellow powder showed one major peak corresponding to Vaska's complex and one minor peak of less than 1% relative area.

Reaction of 3,3,3-trifluoropropyne with trans-chlorocarbonylbis(triphenylphosphine)iridium

trans-Chlorocarbonylbis(triphenylphosphine)iridium (0.50 g, 0.64 mmol), dry benzene (40 ml, nitrogen saturated), and 3,3,3-trifluoropropyne (3.0 g, 31.9 mmol) were heated in a thick-walled tube at 60° for 24 h after thorough degasing. Excess 3,3,3-trifluoropropyne was recovered by cooling and venting into a cold trap. Evaporation left an oily residue. Trituration with diethyl ether gave 0.457 g (83%) of a tan powder, m.p. 211—213° (dec.); IR (KBr) 3045 m, 2135 s, 2060 s, 2040 vs, 1610 m, 1490 s, 1442 s, 1260 s(br), 1110 s(br), 1000 m, 752 s, 700 s cm^{-1}; mass spec. (50 eV) *m/e* 873, 845, 751, 634, 450, 262; ^{1}H NMR ($CDCl_3$) δ 7.38—7.63 (complex, aromatic protons). Found: C, 54.49; H, 3.47; Cl, 3.95; F, 7.36; Ir, 21.93; P, 6.66. $C_{40}H_{31}ClF_3IrOP_2$ calcd.: C, 54.92; H, 3.58; Cl, 4.06; F, 6.52; Ir, 22.80; P, 7.09%.

Acknowledgements

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