ORGANOMETALLICS

Reaction of [P(C6H5)4][Fe2(CO)6(.mu.-CO)(.mu.-CHCH2)] with [RhCl(CO)2]2. Synthesis of a triiron-dirhodium cluster anion containing a CH3C=C=CH2 triiron-dirhodium ligand with an unusual .mu.4-.eta.3 mode of bonding, [P(C6H5)4][Fe3Rh2(CO)10(.mu.-CO)3(.mu.4-.eta.3-CH3C=C=CH2)]

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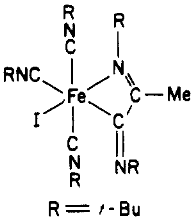
and C(2) deviate significantly from the idealized sp^2 value of 120°: Pd–C(1)–N(1) = 133.1 (5)°, Pd–C(1)–C(9) = 112.7 (4)°, N(1)–C(1)–C(9) = 114.3 (5)°, C(1)–N(1)–C(2) = 127.7 (5)°, and N(1)–C(2)–C(3) = 127.2 (5)°. The ligand appears therefore to be bent in such a way as to shorten the Pd···N(2) intramolecular distance (2.977 (4) Å) and to increase the Pd···C(3) separation (3.357 (6) Å). The coplanarity of the 2-pyridyl ring with the C(2)–N(1)–C(1)–C(9)–N(2) system and the distorted angles at C(1), N(1), and C(2) do not result from the crystal packing shown in Figure 4. No significant intermolecular contacts are observed: the shorter ones are between the two centrosymmetrically related 2-pyridyl rings with C(10)···N(2)′ and C(10)···C(10)′ of 3.481 (6) and 3.411 (7) Å, respectively, which would increase by a further rotation of the rings around the C(1)–C(9) bond. Although some intramolecular steric requirements may be important, the above structural features are probably the result of a repulsion between the Pd center and the closely lying H(3) atom (Pd···H(3) = 2.68 Å) and of a weak bonding interaction with the N(2) 2-pyridyl atom. A four-membered metallacycle with a C,N-chelated 1,2-bis(imino)propyl ligand was proposed for the iron(II) complex:[29]



The structure of *trans*-[PdCl{C(2-C_5H_4N)=NC_6H_4OMe-*p*}$(PMe_2Ph)_2$] accounts also for some features of its ^1H NMR spectrum. Thus, the downfield shift of the C_6H_4OMe-*p* ortho protons clearly results from their close proximity to the d^8 metal center. On the other hand, the restricted rotation of the α-diimino moiety around the Pd–C(1) bond is mainly due to sterical hindrance with adjacent PMe_2Ph ligands, even though some contribution of electronic factors, such as a certain degree of d – π* back donation in the Pd–C(1) bond and a weak Pd···N(2) interaction, cannot be ruled out.

Acknowledgment. We are grateful to Ministero della Pubblica Istruzione for financial support (Research Fund 40%) and to Mrs. M. Magnabosco for technical assistance.

Registry No. **4** (R_1 = H), 42910-70-3; **5** (R_1 = *trans*-PdCl$(PEt_3)_2$), 101652-38-4; **5** (R_1 = H), 101652-39-5; *trans*-[PdCl(2-C_5H_5N)$(PMe_2Ph)_2$]Cl, 101652-34-0; *trans*-[PdCl(2-C_5H_5N)$(PEt_3)_2$]Cl, 101652-35-1; *cis*-[$PdCl_2$(2-C_5H_5N)(PPh_3)], 88227-30-9; *p*-$MeOC_6H_4NC$, 10349-38-9; MeNC, 593-75-9; *trans*-[PdCl{C(2-C_5H_4N)=NC_6H_4OMe-*p*}$(PMe_2Ph)_2$], 101670-83-1; *trans*-[PdCl{C(2-C_5H_4N)=NMe}$(PMe_2Ph)_2$], 101670-84-2; *trans*-[PdCl{C(2-C_5H_4N)=NC_6H_4OMe-*p*}$(PEt_3)_2$], 101652-36-2; *trans*-[PdCl{C(2-C_5H_4N)=NMe}$(PEt_3)_2$], 101652-37-3; C_5H_4N-2-CH=NMe, 40468-83-5.

Supplementary Material Available: Listings of anisotropic thermal parameters, least-squares planes, and observed and calculated structure factors (17 pages). Ordering information is given on any current masthead page.

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(29) Bassett, J. M.; Green, M.; Howard, J. A. K.; Stone, F. G. A. *J. Chem. Soc., Dalton Trans.* 1980, 1779.

Reaction of $[P(C_6H_5)_4][Fe_2(CO)_6(\mu\text{-}CO)(\mu\text{-}CHCH_2)]$ with $[RhCl(CO)_2]_2$. Synthesis of a Triiron–Dirhodium Cluster Anion Containing a $CH_3C=C=CH_2$ Triiron–Dirhodium Ligand with an Unusual $\mu_4\text{-}\eta^3$ Mode of Bonding, $[P(C_6H_5)_4][Fe_3Rh_2(CO)_{10}(\mu\text{-}CO)_3(\mu_4\text{-}\eta^3\text{-}CH_3C=C=CH_2)]$

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$[P(C_6H_5)_4][Fe_2(CO)_6(\mu\text{-}CO)(\mu\text{-}CHCH_2)]$ reacts with $[RhCl(CO)_2]_2$ in the presence of $TlBF_4$ at room temperature to give a 17% yield of $[P(C_6H_5)_4][Fe_3Rh_2(CO)_{10}(\mu\text{-}CO)_3(\mu\text{-}\eta^3\text{-}CH_3C=C=CH_2)]$ (1) whose structure has been determined by X-ray diffraction. 1 is monoclinic of space group $P2_1/n$ with a = 10.076 (2) Å, b = 30.326 (4) Å, c = 13.824 (3) Å, β = 104.16 (2)°, and Z = 4. The structure has been solved and refined to R and R_w values of 0.038 and 0.044, respectively, using 4018 reflections. The anionic part of 1 consists of a square-based pyramid of metals, with a rhodium atom at the top of the pyramid. The 3-methyl-3-allenyl ligand results from the coupling of an ethylidyne and vinylidene ligand derived from the initial vinyl ligands. The allenyl ligand shows an unusual $\mu_4\text{-}\eta^3$ mode of bonding to the $RhFe_3$ unit. Under hydrogen pressure (10 atm) and at 60 °C, 1 slowly transforms 1-octene into 25% octane and 75% mixture of 2- and 3-octene.

In a recent publication[1] we have shown that the mixed iron–rhodium cluster $HFe_3Rh(CO)_{11}[\mu_4\text{-}\eta^2\text{-}C=CH(C_6H_5)]$, synthesized by the reaction of the $[HFe_3(CO)_9(\mu_3\text{-}\eta^2\text{-}C=CHC_6H_5)]^-$ anion with $[RhCl(CO)_2]_2$, was catalytic precursor for the isomerization of alkenes under hydrogen pressure. The cluster was destroyed during the catalysis, but the results suggest that the active species was a mixed rhodium–iron system. At the end of the reaction the only isolable compound was $H_3Fe_3(CO)_9(\mu_3\text{-}CCH_2C_6H_5)$, but this cluster was not an active catalyst for isomerization of alkenes under the same conditions. All these observations

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(1) Attali, S.; Mathieu, R. *J. Organomet. Chem.* 1985, *291*, 205–211.

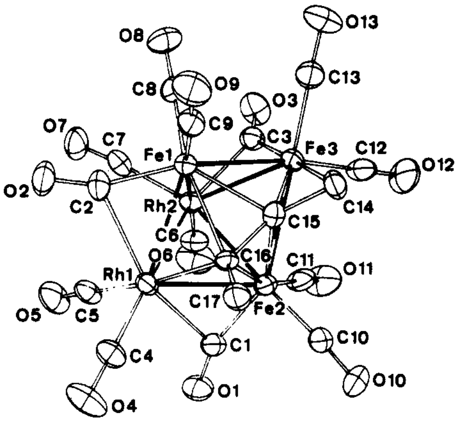

Figure 1. Structure of the anionic part of complex 1 showing the atomic numbering scheme with the ellipsoids at the 35% probability level

induced us to attempt the synthesis of a mixed trinuclear iron–rhodium cluster in which the metallic framework would be maintained by an alkylidyne ligand, this ligand being stable under our catalytic experimental conditions. Taking into account our observation of the facile conversion of a vinyl group into an ethylidyne ligand in polynuclear iron complexes,[2] we thought that the action of $[RhCl(CO)_2]_2$ on $[P(C_6H_5)_4][Fe_2(CO)_6(\mu\text{-}CO)(\mu\text{-}CHCH_2)]$ could be a convenient way to synthesize the mixed-cluster $Fe_2Rh(CO)_{10}(CCH_3)$. Actually, as is often the case in the preparation of mixed-metal clusters,[3] the reaction was not so straightforward. Here we report that it led unexpectedly to a mixed pentanuclear clusters anion which was fully identified by single-crystal X-ray diffraction as $[P(C_6H_5)_4][Fe_3Rh_2(CO)_{10}(\mu\text{-}CO)_3(\mu_4\text{-}CH_3C{=}C{=}CH_2)]$.

Results

The reaction of $[P(C_6H_5)_4][Fe_2(CO)_6(\mu\text{-}CO)(\mu\text{-}CHCH_2)]$ with half an equimolecular amount of $[RhCl(CO)_2]_2$ in acetone in the presence of thallium tetrafluoroborate led upon stirring at room temperature to a brown solution. After evaporation of the solvent, extraction with diethyl ether gave a green-brown solution, leaving a dark residue. The infrared spectrum of the latter in the ν(CO) strecthing region showed only broad, weak, and ill-defined absorption bands and was not further studied. Crystallization of the ether extract from methanol led to the formation of the green complex 1. The infrared spectrum of 1 in the carbonyl stretching region consisted of five bands in the terminal carbonyl region at 2055 (m), 2005 (s), 1990 (s), 1970 (sh), and 1925 (m) cm^{-1} and two weak ill-defined broad bands centered at 1855 and 1805 cm^{-1} attributed to bridging carbonyl groups. The ^1H NMR spectrum gave evidence for the presence of the tetraphenylphosphonium cation and showed two singlets in the relative ratio of 20/2/3. Proton-coupled ^{13}C resonance confirmed the presence of CH_2 and CH_3 groups.

So the reaction was more complex than expected, as confirmed by the chemical analysis which was with a formulation with an Fe_3Rh_2 core for compound 1.

As it was difficult to ascertain the formulation on the basis only of spectroscopic data, the precise nature of 1 was established by X-ray crystallography.

X-ray Structure of 1. An Ortep plot of the anionic part of 1 is shown in Figure 1, and bond distances and angles of interest are gathered in Table I.

Table I. Selected Bond Lengths (Å) and Angles (deg) and Their Estimated Standard Deviations for 1

Bond Lengths			
Rh(1)–Rh(2)	2.7337 (9)	Rh(2)–C(7)	1.897 (9)
Rh(1)–Fe(1)	2.672 (1)	Fe(1)–C(8)	1.741 (7)
Rh(1)–Fe(2)	2.649 (1)	Fe(1)–C(9)	1.751 (8)
Rh(2)–Fe(1)	2.679 (1)	Fe(2)–C(10)	1.753 (9)
Rh(2)–Fe(2)	2.690 (1)	Fe(2)–C(11)	1.740 (11)
Rh(2)–Fe(3)	2.684 (1)	Fe(3)–C(12)	1.756 (9)
Fe(1)–Fe(3)	2.683 (2)	Fe(3)–C(13)	1.785 (7)
Fe(2)–Fe(3)	2.716 (1)	Rh(1)–C(16)	2.100 (7)
Rh(1)–C(1)	2.379 (8)	Fe(1)–C(15)	2.079 (7)
Fe(1)–C(1)	1.831 (9)	Fe(1)–C(16)	2.159 (6)
Rh(1)–C(2)	2.311 (8)	Fe(2)–C(15)	2.078 (7)
Fe(2)–C(2)	1.807 (8)	Fe(2)–C(16)	2.146 (7)
Rh(2)–C(3)	2.065 (8)	Fe(3)–C(14)	2.084 (9)
Fe(3)–C(3)	1.908 (8)	Fe(3)–C(15)	1.919 (7)
Rh(1)–C(4)	1.901 (8)	C(14)–C(15)	1.412 (11)
Rh(1)–C(5)	1.878 (9)	C(15)–C(16)	1.402 (9)
Rh(2)–C(6)	1.907 (9)	C–O	1.124–1.171 (12)[a]
Bond Angles			
Fe(1)–Rh(1)–Fe(2)	77.39 (4)	Fe(1)–C(1)–O(1)	158.7 (6)
Rh(1)–Fe(2)–Fe(3)	101.02 (4)	Rh(1)–C(2)–O(2)	122.9 (6)
Fe(1)–Fe(3)–Fe(2)	76.07 (4)	Fe(2)–C(2)–O(2)	158.1 (7)
Fe(3)–Fe(1)–Rh(1)	101.29 (4)	Rh(2)–C(3)–O(3)	130.9 (7)
Rh(1)–Rh(2)–Fe(1)	59.15 (3)	Fe(3)–C(3)–O(3)	144.2 (7)
Rh(1)–Rh(2)–Fe(2)	58.47 (3)	Rh(1)–C(16)–C(17)	119.2 (5)
Fe(2)–Rh(2)–Fe(3)	60.71 (3)	C(15)–C(16)–C(17)	117.4 (6)
Fe(1)–Rh(2)–Fe(3)	60.04 (4)	C(14)–C(15)–C(16)	141.1 (7)
Rh(1)–C(1)–O(1)	123.7 (5)		

[a] Maximum esd.

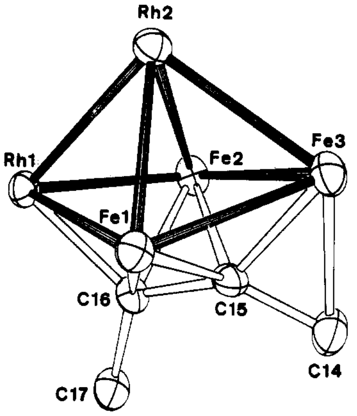

Figure 2. Core geometry of the anionic part of complex 1 showing the mode of bonding of the allenyl ligand

The exact formulation of 1 is $[P(C_6H_5)_4][Fe_3Rh_2(CO)_{10}(\mu\text{-}CO)_3(\mu_4\text{-}\eta^3\text{-}CH_3C{=}C{=}CH_2)]$. The anionic part consists of a square-based pyramid of metals with a rhodium atom (Rh(2)) at the top of the pyramid. The $RhFe_3$ square base is not strictly planar. Fe(1) and Fe(2) are located below the mean plane at distances of 0.1835 (9) and 0.244 (1) Å, respectively, while Rh(1) and Fe(3) are over this plane at 0.0662 (6) and 0.228 (1) Å, respectively. There are no special features about the metal–metal bond distances which are in the normal range.[4] Each metal is surrounded by two terminal carbonyl groups, and three bridging carbonyl groups span the Rh(1)–Fe(1), Rh(1)–Fe(2), and Rh(2)–Fe(3) metal–metal bonds. These bridging carbonyl groups are of two types: one, C(3)–O(3), bridges nearly symmetrically the Rh(2)–Fe(3) bond and the two others, C(1)–O(1) and C(2)–O(2), are of the semibridging type[5] and are more tightly bonded to iron metals

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(3) Gladfelter, W. L.; Geoffroy, G. L. *Adv. Organomet. Chem.* **1980**, *18*, 207–273.

(4) Breen, M. J.; Geoffroy, G. L. *Organometallics* **1982**, *1*, 1473–1443 and references therein.

(5) Curtis, M. D.; Han, K. R.; Butler, W. M. *Inorg, Chem.* **1980**, *19*, 2096–2101.

centers than to rhodium atoms (Fe(1)–C(1) = 1.831 (9) Å, Rh(1)–C(1) = 2.379 (8) Å, Fe(2)–C(2) = 1.807 (8) Å, Rh(1)–C(2) = 2.311 (8) Å).

These features are consistent with the broad and ill-defined absorption bands observed in the infrared spectrum of 1 and centered at 1855 and 1805 cm^{-1}. The hydrocarbon part of the anion is a 3-methyl-3-allenyl ligand which is the result of the coupling of an ethylidyne and a vinylidene fragment probably derived from the rearrangement of the initial vinyl ligand. This allenyl ligand is interacting with four metal centers (Figure 2) which is, to our knowledge, the first case of this mode of bonding in polynuclear complexes, a more common mode of bonding being the interaction with three metal centers.[6,7] It can be described as σ-bonded by the C(16) carbon atom to Rh(1), η^2-bonded to Fe(3) by the C(14)–C(15) double bond, and bonded further by the unusual μ_2-η^2 mode of bonding of the C(15)–C(16) double bond with the Fe(1) and Fe(2) metal centers. The C(15) and C(16) atoms are nearly equidistant from Fe(1) and Fe(2), respectively, (Fe(1)–C(15) = 2.079 (7) Å, Fe(2)–C(15) = 2.078 (7) Å, Fe(1)–C(16) = 2.159 (6) Å, Fe(2)–C(16) = 2.146 (7) Å), and as it has been generally observed for the allenyl ligands μ_3-η^3 bonded,[8–11] the central carbon C(15) of the allene lies closer to the metal than the terminal carbon atoms (Fe(3)–C(15) = 1.919 (7) Å, Fe(3)–C(14) = 2.084 (9) Å). Unexpectedly, this mode of bonding of the 3-methyl-3-allenyl ligand has no obvious consequences on bond lengths and bond angles in the C(14)–C(15)–C(16) unit. Indeed, the C(14)–C(15) and C(15)–C(16) bond lengths are nearly the same and have the same order of magnitude as in other cases of allene ligands bonded to di- or trinuclear complexes.[6–11] Moreover, the bonding angle at the central atom C(15) (141.1 (7)°) compares well with the value found for the 1-methyl-3-ethyl-3-allenyl ligand bonded in a μ_3-η^3 mode to a $Ru_3(CO)_9$ cluster unit (142.3 (6)°).[6] This is perhaps due to the fact that the description of the bonding mode in terms of localized σ and π contributions is an oversimplification for an allenyl ligand.[12]

Finally the Rh(1), Rh(2), Fe(3), C(15), and C(16) atoms are nearly planar, and C(17) and C(14) deviate from this plane by 0.014 (7) and 0.024 (8) Å, respectively. Furthermore, the Fe(1) and Fe(2) atoms are quasi-symmetrically located above and below this plane (1.6604 (9) and 1.666 (1) Å, respectively). Taking into account these observations, the best description for the structure of this 74-valence-electron pentanuclear cluster is, according to Wade's theory,[13] a closo pentagonal bipyramid.

In summary, the X-ray structure of 1 confirms what was suggested by the spectroscopic data: the reaction between the dinuclear anionic iron complex and the chloro rhodium dicarbonyl group is much more complex than expected. Nevertheless the initially sought Fe_2RhCCH_3 fragment can be recognized in 1 (Fe(1)Fe(2)Rh(1)C(16)C(17)) and is perhaps the first step of the reaction, but it is difficult to consider a pathway for the formation of 1. This reaction provides a further example of carbon–carbon bond formation between carbyne and vinylidene fragments. However, in the two other known cases of dinuclear cationic complexes of ruthenium[14] or iron[15] containing carbyne ligands, coupling occurs between the methylene carbon of the vinlyidene and the carbyne carbon atom.

Catalytic Experiments. As our initial aim was to synthesize a mixed rhodium–iron cluster in order to check its catalytic activity toward the isomerization of alkenes, we have studied the activity of 1 toward the isomerization of 1-octene, even though the rhodium–iron frame was not stabilized by a carbyne ligand.

Actually, compared to $HFe_3Rh(CO)_{11}(\mu_4\text{-}C{=}C(C_6H_5)H)$,[1] 1 was far less active, as under 10 atm of hydrogen it was necessary to heat at 60 °C to observe some activity. After 24 h, 25% hydrogenation and 75% isomerization of 1-octene into 60% 2-octenes and 40% 3-octenes were observed. Nevertheless, at the end of the reaction, the infrared spectrum of the clear solution only gave evidence of the presence of 1 which was recovered in 60% yield by crystallization after the experiment. This stability of 1 was confirmed by the fact that after one run of catalysis the solution showed the same activity when more 1-octene was added.

Even though these observations do not conclusively demonstrate that 1 is the actual catalyst, this constitutes a second example for a mixed iron–rhodium system in which the known hydrogenation activity of rhodium[16] is lowered by iron, favoring isomerization over hydrogenation reactions.

Experimental Section

All reactions were performed under nitrogen atmosphere. Infrared spectra were recorded with a Perkin-Elmer 225 spectrometer in dichloromethane solution. ^1H NMR spectra were obtained with Bruker WH90 and ^{13}C NMR spectra with Bruker WH250 instrument. Elemental analyses were performed in our laboratory and at the Centre de Microanalyse of the CNRS.

Preparation of $[P(C_6H_5)_4][Fe_3Rh_2(CO)_{10}(\mu\text{-}CO)_3\text{-}\mu_4\text{-}\eta^3\text{-}CH_3C{=}C{=}CH)]$ To $[P(C_6H_5)_4][Fe_2(CO)_6(\mu\text{-}CO)(\mu\text{-}CHCH_2)]^2$ (1 g) dissolved in acetone were added $[RhCl(CO)_2]_2$ (0.288 g) and $TlBF_4$ (0.432 g). The solution was stirred for 4 h. It was then filtered and evaporated to dryness. Extraction of the residue with diethyl ether gave a green solution, leaving a brown precipitate which was discarded. Evaporation of ether and crystallization of the residue from methanol gave 0.145 g of 1 (17% yield on rhodium base).

Anal. Calcd for $C_{41}H_{25}Fe_3O_{13}PRh_2$: C, 43.53; H, 2.21; Fe, 14.86; Rh, 18.23. Found: C, 43.46; H, 2.17; Fe, 15.26; Rh, 18.40. ^1H NMR ($(CD_3)_2CO$): 8.05, 7.95 ($P(C_6H_5)_4$), 4.37 (CH_2), 2.73 (CH_3) ppm. ^{13}C NMR (CD_2Cl_2), except $P(C_6H_5)_4$ resonances): 242.9 ppm (d, J_{Rh-C} = 35.1 Hz), 217.4, 213.8, 212.8 197.1 (d, J_{Rh-C} = 68.2 Hz), 193.2 (d, J_{Rh-C} = 61.0 Hz), 182.3 (d, J_{Rh-C} = 82.6 Hz), 132.2, 48.8 (t, J_{CH} = 165.2 Hz), 40.9 (q, J_{CH} = 129.3 Hz).

Catalytic Experiments. All experiments have been carried out in a 100-mL stainless-steel autoclave fitted with a glass vessel inside. Chromatography analyses of the catalysis reaction were performed on an Intersmat IGC 120F apparatus using a 4-m Carbowax column operating at 60 °C.

1 (50 mg) was dissolved in dichloromethane (15 mL) and 1-octene (2 mL). The solution was pressurized under 10 atm of hydrogen and stirred at 60 °C.

X-ray Structure Analysis of 1. Collection and Reduction of X-ray Data. Crystals belong to the monoclinic system of space

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Table II. Summary of Crystal and Intensity Collection Data

compd	$[Fe_3Rh_2(CO)_{13}H_2CCCCH_3][P(C_6H_5)_4]$
formula	$C_{41}Fe_3H_{25}O_{13}PRh_2$
formula wt	1129.35
a, Å	10.076 (2)
b, Å	30.326 (4)
c, Å	13.824 (3)
β, deg	104.16 (2)
V, Å^3	4096 (2)
Z	4
F(000)	2232
D_{calcd}, g cm^{-3}	1.831
cryst system	monoclinic
space group	C_{2h}^5—$P2_1/n$
cryst size, mm	0.35 × 0.35 × 0.15
radiatn	Mo Kα from graphite monochromator (λ = 0.71073 Å)
linear abs coeff, cm^{-1}	19.17
temp, °C	20
receiving aperture, mm	4.0 × 4.0
takeoff angle, deg	4
scan mode	θ–2θ
scan range, deg	0.90 + 0.35 tan θ
2θ limits, deg	47

group $P2_1/n$. A black square-based parallelepiped was sealed on a glass fiber and mounted on an Enraf-Nonius CAD 4 diffractometer. Cell constants were obtained from a least-squares fit of the setting angles of 25 reflections. A summary of crystal and intensity collection data is given in Table II. A total of 6049 independent reflections were recorded at a 2θ(Mo) maximum of 47° by procedures described elsewhere.[17] Intensity standards, recorded periodically, showed only random, statistical fluctuations. Data reduction was then performed,[18] and absorption corrections[19] were made on 4018 reflections having $F_o^2 > 4\sigma(F_o^2)$ (μ = 19.2 cm^{-1}, calculated transmission range 0.54–0.76).

Structure Solution and Refinement. The structure was solved[20] by the heavy atom method. Successive difference Fourier maps and least-squares refinement cycles revealed the positions of all nonhydrogen atoms.

All non-hydrogen atoms were refined anisotropically, except phenyl rings were refined as isotropic rigid groups (C–C = 1.385 Å). Hydrogen atoms were located on a difference Fourier map. They were included in calculations in constrained geometry (C–H = 0.97 Å) with the exception of those bonded to C(14). All hydrogen atoms were given a fixed isotropic temperature factor U_H = 0.07 Å^2.

The atomic scattering factors used were those proposed by Cromer and Waber[21] with anomalous dispersion effects.[22] Scattering factors for the hydrogen atoms were taken from Stewart et al.[23]

The final full-matrix least-squares refinement coverged to $R = \sum||F_o| - |F_c||/\sum|F_o| = 0.038$ and $R_w = [\sum w(|F_o| - |F_c|)^2/\sum w|F_o|^2]^{1/2} = 0.044$, the weighting scheme used in the minimization of the function $\sum w(|F_o| - |F_c|)^2$ being defined as $w[\sigma^2(F_o) + 0.012F_o^2]^{-1}$. The error in an observation of unit weight was $S = [\sum w(|F_o| - |F_c|)^2/(n - m)]^{1/2} = 2.6$ with n = 4018 observations and m = 379 variables. An analysis of variance showed no unusual trends. In the last cycle of refinement the shifts for all parameters were less than 0.1σ, except those for hydrogens bonded to C(14) ($\sim\sigma$). A final difference Fourier map showed a residual electron density of 0.5 e/Å^3. The final fractional atomic coordinates are listed in Table III.

Table III. Fractional Atomic Coordinates with Esd's in Parentheses

atom	*x/a*	*v/b*	*z/c*
Rh(1)	0.21253 (5)	0.32725 (2)	0.53302 (4)
Rh(2)	0.41922 (5)	0.38685 (2)	0.59584 (4)
Fe(1)	0.45455 (9)	0.30160 (3)	0.64641 (7)
Fe(2)	0.3819 (1)	0.34553 (4)	0.41902 (7)
Fe(3)	0.62740 (9)	0.34155 (4)	0.55448 (7)
C(1)	0.3031 (8)	0.3001 (3)	0.6974 (6)
O(1)	0.2392 (6)	0.2944 (2)	0.7533 (4)
C(2)	0.2070 (8)	0.3649 (3)	0.3867 (5)
O(2)	0.1091 (6)	0.3823 (2)	0.3403 (4)
C(3)	0.6279 (8)	0.3965 (3)	0.6218 (6)
O(3)	0.6973 (6)	0.4256 (2)	0.6605 (4)
C(4)	0.0569 (8)	0.2910 (3)	0.4832 (6)
O(4)	−0.0409 (6)	0.2723 (2)	0.4530 (5)
C(5)	0.1106 (7)	0.3716 (3)	0.5764 (6)
O(5)	0.0464 (6)	0.3979 (2)	0.6023 (5)
C(6)	0.3652 (8)	0.4430 (3)	0.5371 (6)
O(6)	0.3383 (7)	0.4770 (3)	0.5084 (6)
C(7)	0.3826 (7)	0.4010 (3)	0.7204 (6)
O(7)	0.3525 (6)	0.4101 (2)	0.7915 (4)
C(8)	0.5631 (8)	0.3248 (3)	0.7517 (5)
O(8)	0.6338 (6)	0.3399 (2)	0.8231 (4)
C(9)	0.5087 (7)	0.2482 (3)	0.6855 (5)
O(9)	0.5436 (6)	0.2138 (2)	0.7169 (4)
C(10)	0.3664 (7)	0.3192 (4)	0.3039 (6)
O(10)	0.3481 (6)	0.3022 (3)	0.2273 (4)
C(11)	0.4532 (9)	0.3927 (4)	0.3807 (6)
O(11)	0.4927 (7)	0.4245 (3)	0.3505 (5)
C(12)	0.7222 (8)	0.3631 (3)	0.4744 (6)
O(12)	0.7839 (7)	0.3771 (3)	0.4225 (5)
C(13)	0.7782 (8)	0.3251 (3)	0.6446 (6)
O(13)	0.8745 (6)	0.3133 (2)	0.6994 (5)
C(14)	0.6181 (7)	0.2791 (3)	0.4903 (6)
H1(C14)	0.659 (7)	0.254 (1)	0.532 (3)
H2(C14)	0.644 (7)	0.271 (2)	0.429 (2)
C(15)	0.4949 (7)	0.2962 (2)	0.5064 (5)
C(16)	0.3565 (6)	0.2852 (2)	0.4937 (4)
C(17)	0.3106 (7)	0.2403 (3)	0.4495 (5)
H1(C17)	0.2134 (7)	0.2368 (3)	0.4439 (5)
H2(C17)	0.3607 (7)	0.2176 (3)	0.4930 (5)
H3(C17)	0.3288 (7)	0.2377 (3)	0.3840 (5)
P	0.3275 (2)	0.07310 (7)	0.5683 (1)
C(18)	0.2261 (4)	0.1172 (2)	0.5978 (3)
C(19)	0.1053 (4)	0.1301 (2)	0.5321 (3)
C(20)	0.0387 (4)	0.1680 (2)	0.5507 (3)
C(21)	0.0929 (4)	0.1930 (2)	0.6350 (3)
C(22)	0.2137 (4)	0.1801 (2)	0.7007 (3)
C(23)	0.2803 (4)	0.1422 (2)	0.6821 (3)
C(24)	0.2249 (4)	0.0321 (2)	0.4901 (3)
C(25)	0.2193 (4)	−0.0098 (2)	0.5286 (3)
C(26)	0.1463 (4)	−0.0428 (2)	0.4693 (3)
C(27)	0.0790 (4)	−0.0338 (2)	0.3713 (3)
C(28)	0.0846 (4)	0.0081 (2)	0.3327 (3)
C(29)	0.1576 (4)	0.0411 (2)	0.3921 (3)
C(30)	0.4150 (4)	0.0476 (2)	0.6817 (3)
C(31)	0.5480 (4)	0.0327 (2)	0.6936 (3)
C(32)	0.6114 (4)	0.0093 (2)	0.7783 (3)
C(33)	0.5418 (4)	0.0008 (2)	0.8512 (3)
C(34)	0.4087 (4)	0.0156 (2)	0.8393 (3)
C(35)	0.3454 (4)	0.0390 (2)	0.7545 (3)
C(36)	0.4488 (5)	0.0956 (2)	0.5066 (3)
C(37)	0.4549 (5)	0.0828 (2)	0.4116 (3)
C(38)	0.5446 (5)	0.1035 (2)	0.3649 (3)
C(39)	0.6283 (5)	0.1371 (2)	0.4130 (3)
C(40)	0.6222 (5)	0.1500 (2)	0.5080 (3)
C(41)	0.5324 (5)	0.1292 (2)	0.5548 (3)

Registry No. 1, 101916-63-6; $[(P(C_6H_5)_4[Fe_2(Co)_6(\mu\text{-}CO)(\mu\text{-}CHCH_2)]$, 83544-41-6; $[RhCl(CO)_2]_2$, 14523-22-9; 1-octene, 111-66-0; 2-octene, 111-67-1; 3-octene, 592-98-3; octane, 111-65-9.

Supplementary Material Available: Tables of thermal parameters, hydrogen parameters, structure factors, and least-squares plane equations (23 pages). Ordering information is given on any current masthead page.

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