426 nm, $\epsilon = 5.18 \times 10^4$ M^{-1} cm^{-1}; (**1**)Cr^{V}(O)(X), λ_{max} = 410 nm, $\epsilon = 3.39 \times 10^4$ M^{-1} cm^{-1}.

In the reaction of (**1**)Cr^{V}(O)(X) with 3-cyclohexene-1-carboxylic acid and 2-cyclopentene-1-acetic acid, a decrease in the λ_{max} of the Cr^{V}–oxo species is observed, which is accompanied by an increase and then a decrease in the absorbance of the Cr^{IV}–oxo species and a continuing increase in absorbance of the Cr^{III} species. These observations are in accord with previous studies of alkene epoxidation in an aprotic solvent (CH_2Cl_2) with various chromium(V)–oxo porphyrins (see discussion of kinetics in ref 1k). Reactions of (**1**)Cr^{V}(O)(X) with alkenes were monitored at the Soret band of this species (λ_{max} = 410 nm). The decrease in A_{410} is biphasic (Figure 2). The first phase (~70% decrease in A_{410}) is due to disappearance of (**1**)Cr^{V}(O)(X) and appearance of (**1**)Cr^{IV}(O) [i.e., (**1**)Cr^{V}(O)(X) epoxidizes alkene, in the rate-determining step, and is converted to (**1**)Cr^{III}(H_2O)$_2$, which immediately reacts with remaining (**1**)Cr^{V}(O)(X) to provide 2 × (**1**)Cr^{IV}(O)]. The second phase corresponds to the disappearance of (**1**)Cr^{IV}(O) and appearance of (**1**)Cr^{III}(H_2O)$_2$. The pseudo-first-order rate constants (k_{obsd}) for epoxidation were calculated from the change in A_{410} with time during the first phase of reaction. The second-order rate constant, k_2 (=k_{obsd}/[alkene]), for reaction of (**1**)Cr^{V}(O)(X) (1.5×10^{-5} M) with 2-cyclopentene-1-acetic acid at 1.17×10^{-2} M was calculated to be 0.16 M^{-1} s^{-1}. The value of k_2 at [2-cyclopentene-1-acetic acid] = 4.61×10^{-3} M and [(**1**)Cr^{V}(O)(X)] = 1.7×10^{-4} M was determined to be 0.19 M^{-1} s^{-1}. In the case of [3-cyclohexene-1-carboxylic acid] = 8.56×10^{-3} M and [(**1**)Cr^{III}(O)(X)] = 1.7×10^{-4} M, k_2 = 2.0 M^{-1} s^{-1}.

The percentage yield of epoxide products were determined from the kinetic runs at [(**1**)Cr^{V}(O)(X)] = 1.7×10^{-4} M. When corrected for recovery, the yields of epoxide products based upon [(**1**)Cr^{V}(O)(X)]$_i$ were 21% for methyl 3,4-epoxycyclopentane-1-acetate and 53% for methyl 3,4-epoxycyclohexane-1-carboxylate. We were unable to determine if an endothermic comproportionation of 2 × (**1**)Cr^{IV}(O) could provide (**1**)Cr^{III}(H_2O)$_2$ + (**1**)-Cr^{V}(O)(X), with the latter being trapped by alkene. If such comproportionation were inoperative, the yields of epoxide would be 100% for methyl 3,4-epoxycyclohexane-1-carboxylate and 42% for methyl 3,4-epoxycyclopentane-1-acetate.

Conclusions

The second-order rate constants and epoxide product yields for reaction of oxo(5,10,15,20-tetrakis(2,6-dimethyl-3-sulfonatophenyl)porphinato)chromium(V) ((**1**)Cr^{V}(O)(X)) with 3-cyclohexene-1-carboxylic acid and 2-cyclopentene-1-acetic acid have been determined in aqueous solution (30 °C). Recently, water-soluble manganese porphyrins have been employed to catalyze the epoxidation of 2,5-dihydrofuran using hypochlorite as oxidant in aqueous solution.[4] In this catalytic system, the yields of epoxide were 60 and 70%. We now present the results of the stoichiometric epoxidation of alkenes by a hypervalent metal–oxo porphyrin in aqueous solution. In CH_2Cl_2 (30 °C), the second-order rate constant for reaction of cyclohexene with oxo(5,10,15,20-tetrakis(2,4,6-trimethylphenyl)porphinato)chromium(V) ((Me$_{12}$TPP)Cr^{V}(O)(X)) was determined as 5.19×10^{-3} M^{-1} s^{-1}. This value, when compared to that for the second-order reaction of (**1**)Cr^{V}(O)(X) with 3-cyclohexene-1-carboxylic acid (2.0 M^{-1} s^{-1}), shows that these reactions are favored in water. In water, the rate constant for reaction of (**1**)Cr^{V}(O)(X) with 3-cyclohexene-1-carboxylic acid is about 12-fold greater than that in the reaction with 2-cyclopentene-1-acetic acid. In CH_2Cl_2, the second-order rate constant for reaction of oxo(5,10,15,20-tetrakis(2,6-dibromophenyl)porphinatochromium(V) with cyclohexene is ~22-fold greater than the rate constant for reaction with cyclopentene. Therefore, the change in solvent has little effect on the ratio of rate constants.

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Contribution from the Department of Chemistry,
Purdue University, West Lafayette, Indiana 47907

Crystal Structure of [Ru(OEP–*N*-C_6H_5)(C_6H_5)](BF_4). An Organometallic Porphyrin Complex Containing an *N*-C_6H_5 Group with an Agostic Hydrogen

Jeffery W. Seyler, Phillip E. Fanwick, and Charles R. Leidner*

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The so-called "suicide adducts"[1] of cytochrome P_{450} result from the Fe-to-N migration of an alkyl or aryl group within the high-valent oxo–alkene or oxo–arene intermediate.[2] Analogous N-substituted porphyrin complexes[3-5] can be prepared by chemical and electrochemical means from synthetic metalloporphyrins. While Fe complexes have the most obvious biological relevance, information about the structural, spectroscopic, and chemical properties of related N-substituted complexes is of interest. Relatively few[4,5] N-substituted porphyrin complexes have been characterized by X-ray crystallography. As a part of our study[6] of the redox and reaction chemistry of Ru(OEP)(C_6H_5)$_n$ complexes (OEP is octaethylporphyrin), we have observed that oxidation of Ru(OEP)(C_6H_5)$_2$ leads to Ru-to-N migration of a C_6H_5 group. We report herein the crystal structure of [Ru(OEP–*N*-C_6H_5)(C_6H_5)](BF_4), which contains a Ru–C_6H_5 group and a *N*-C_6H_5 group with an agostic hydrogen.[7]

Experimental Section

Materials and Methods. All manipulations were performed in a Vacuum Atmospheres inert-atmosphere drybox with an O_2 level below 2 ppm (Model A0-316-C oxygen analyzer). Solvents were dried by distillation over Na/benzophenone (toluene) or P_2O_5 (CH_2Cl_2). AgBF_4 (Aldrich) was used as received. Ru(OEP)(C_6H_5)$_2$ was prepared according to the method of James and Dolphin.[8]

Synthesis of [Ru(OEP–*N*-C_6H_5)(C_6H_5)](BF_4). In a typical preparation 8 mg (0.041 mmol) of AgBF_4 was added to a well-stirred solution of 20 mg (0.025 mmol) of Ru(OEP)(C_6H_5)$_2$ in 10 mL of toluene. The resulting brown suspension was stirred for 1 h and then filtered to yield

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Table I. Crystallographic Data for [Ru(OEP-*N*-C_6H_5)(C_6H_5)](BF_4)

$RuF_4N_4C_{48}BH_{54}$	fw 874.87
a = 10.923 (1) Å	space group $P\bar{1}$ (No. 2)
b = 13.638 (2) Å	T = 20 °C
c = 16.344 (2) Å	λ = 0.71073 Å
α = 73.827 (8)°	ρ_{calc} = 1.350 g cm^{-3}
β = 75.032 (9)°	μ = 4.10 cm^{-1}
γ = 69.26 (1)°	transm coeff = 1.000–0.717
V = 2152.1 (5) Å^3	$R(F_o)$ = 0.041
Z = 2	$R_w(F_o)$ = 0.051

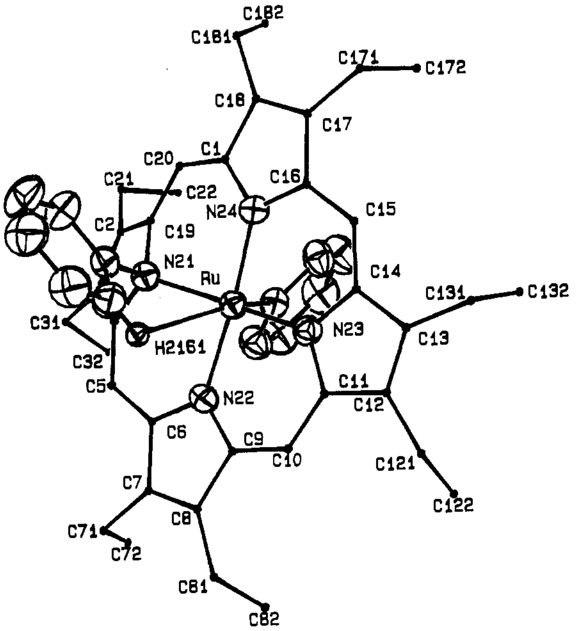

Figure 1. ORTEP view of [Ru(OEP-*N*-C_6H_5)(C_6H_5)](BF_4), emphasizing the central coordination sphere.

18 mg (80%) of brown product. The ^1H NMR spectrum[6] verifies the structure of the paramagnetic complex. Single crystals for X-ray crystallographic study were prepared by dissolving the solid in a minimun of CH_2Cl_2 and carefully adding toluene to the surface of the CH_2Cl_2. After 24 h the red-brown plates were removed from the solution and allowed to dry.

X-ray Crystallography. A red, diamond-shaped crystal with approximate crystal dimensions of 0.6 × 0.3 × 0.05 mm was mounted on a glass fiber in a random orientation. Preliminary examination and data collection were performed on an Enraf-Nonius CAD4 diffractometer. Cell constants and an orientation matrix for data collection were obtained from least-squares refinement by using setting angles of 25 reflections in the range of $16 < \theta < 20°$. Information concerning conditions for crystallographic data collection and structure refinement are listed in Table I. Lorentz and polarization corrections and an empirical absorption correction[9] were applied. The structure was solved by using the Patterson heavy-atom method, which revealed the position of the Ru atom. The remaining atoms were located by using DIRDIF, and in succeeding difference Fourier synthesis. H atoms were located and added to the structure factor calculations; their positions were not refined, except that of the agostic H. The highest peak in the final difference Fourier map was 0.83 e/Å^3.

Results

Our studies[6] of the redox and reaction chemistry of Ru(OEP)(C_6H_5)$_n$ complexes revealed that electrochemical or chemical oxidation of Ru(OEP)(C_6H_5)$_2$ results in the Ru-to-N migration of one of the C_6H_5 groups. Electrochemical and spectroscopic methods provided some information about this novel complex; however, we sought more detailed structural information. [Ru(OEP-*N*-C_6H_5)(C_6H_5)](BF_4), prepared[6] by oxidation of Ru(OEP)(C_6H_5)$_2$[8] in toluene with $AgBF_4$, was crystallized from CH_2Cl_2/toluene to yield red-brown plates. A suitable crystal was selected and characterized by X-ray crystallography. Initial refinements of the structure revealed the expected N-bound and Ru-bound C_6H_5 groups; however, the orientation of the *N*-C_6H_5 group was such that one *o*-H projected onto the Ru center. Refining the position of this H resulted in the structure shown in Figure 1. Fractional coordinates and thermal parameters are listed in Table II; selected bond distances and angles are given in Table III.

Table II. Fractional Coordinates and Isotropic Thermal Parameters for [Ru(OEP-*N*-C_6H_5)(C_6H_5)](BF_4)

atom	*x*	*y*	*z*	*B*, Å^2
Ru	0.15855 (3)	0.34696 (3)	0.22099 (2)	2.608 (7)
F(1)	0.1595 (4)	0.1488 (3)	0.7612 (3)	11.9 (1)
F(2)	0.0048 (5)	0.1891 (4)	0.6830 (4)	16.3 (2)
F(3)	0.0067 (6)	0.0760 (5)	0.8019 (4)	16.9 (2)
F(4)	0.1380 (7)	0.0372 (5)	0.7005 (4)	21.1 (2)
N(21)	0.0784 (3)	0.2237 (3)	0.3078 (2)	3.00 (8)
N(22)	0.1677 (3)	0.3974 (3)	0.3256 (2)	3.16 (8)
N(23)	0.1945 (3)	0.4786 (2)	0.1429 (2)	2.76 (8)
N(24)	0.1208 (3)	0.3083 (2)	0.1204 (2)	2.81 (8)
C(1)	0.0957 (4)	0.2153 (3)	0.1219 (3)	2.9 (1)
C(2)	0.1550 (4)	0.0382 (3)	0.3384 (3)	3.7 (1)
C(3)	0.1505 (4)	0.0726 (3)	0.4121 (3)	3.8 (1)
C(4)	0.1140 (4)	0.1833 (3)	0.3942 (3)	3.4 (1)
C(5)	0.1241 (4)	0.2493 (3)	0.4416 (3)	3.5 (1)
C(6)	0.1483 (4)	0.3466 (3)	0.4113 (3)	3.3 (1)
C(7)	0.1647 (4)	0.4096 (3)	0.4644 (3)	3.6 (1)
C(8)	0.1873 (4)	0.4985 (3)	0.4112 (3)	3.6 (1)
C(9)	0.1903 (4)	0.4903 (3)	0.3240 (3)	3.2 (1)
C(10)	0.2134 (4)	0.5642 (3)	0.2498 (3)	3.2 (1)
C(11)	0.2180 (4)	0.5587 (3)	0.1663 (3)	3.0 (1)
C(12)	0.2460 (4)	0.6373 (3)	0.0898 (3)	3.2 (1)
C(13)	0.2401 (4)	0.6037 (3)	0.0208 (3)	3.2 (1)
C(14)	0.2042 (4)	0.5061 (3)	0.0539 (3)	2.9 (1)
C(15)	0.1745 (4)	0.4518 (3)	0.0057 (3)	3.1 (1)
C(16)	0.1341 (4)	0.3608 (3)	0.0356 (3)	2.8 (1)
C(17)	0.1088 (4)	0.3029 (3)	−0.0171 (3)	3.1 (1)
C(18)	0.0840 (4)	0.2142 (3)	0.0354 (3)	3.0 (1)
C(19)	0.1132 (4)	0.1273 (3)	0.2743 (3)	3.3 (1)
C(20)	0.1000 (4)	0.1306 (3)	0.1912 (3)	3.4 (1)
C(21)	0.2094 (6)	−0.0757 (4)	0.3257 (4)	5.3 (1)
C(22)	0.3537 (8)	−0.1017 (5)	0.2788 (5)	8.7 (2)
C(31)	0.1929 (6)	0.0025 (4)	0.4955 (3)	5.4 (1)
C(32)	0.3393 (6)	−0.0199 (6)	0.4945 (4)	8.5 (2)
C(71)	0.1668 (5)	0.3715 (4)	0.5598 (3)	4.6 (1)
C(72)	0.3046 (6)	0.2997 (5)	0.5770 (4)	6.5 (2)
C(81)	0.2047 (5)	0.5917 (4)	0.4339 (3)	4.3 (1)
C(82)	0.3480 (5)	0.5884 (4)	0.4213 (4)	6.3 (2)
C(121)	0.2769 (5)	0.7350 (3)	0.0904 (3)	4.2 (1)
C(122)	0.4178 (5)	0.7130 (4)	0.1046 (4)	5.4 (1)
C(131)	0.2708 (4)	0.6530 (4)	−0.0732 (3)	3.9 (1)
C(132)	0.4138 (5)	0.5990 (5)	−0.1134 (4)	5.5 (2)
C(171)	0.1202 (4)	0.3351 (4)	−0.1136 (3)	3.8 (1)
C(172)	0.2633 (6)	0.2989 (5)	−0.1609 (4)	5.8 (2)
C(181)	0.0551 (4)	0.1265 (3)	0.0133 (3)	4.0 (1)
C(182)	0.1803 (6)	0.0355 (4)	−0.0064 (4)	5.8 (2)
C(211)	−0.0685 (4)	0.2794 (3)	0.3149 (3)	3.2 (1)
C(212)	−0.1542 (5)	0.2233 (4)	0.3188 (4)	4.7 (1)
C(213)	−0.2888 (5)	0.2768 (4)	0.3246 (4)	5.8 (2)
C(214)	−0.3390 (5)	0.3834 (5)	0.3268 (4)	5.8 (2)
C(215)	−0.2532 (5)	0.4379 (4)	0.3226 (4)	5.1 (1)
C(216)	−0.1177 (5)	0.3860 (4)	0.3171 (3)	4.2 (1)
C(1001)	0.3426 (4)	0.2445 (3)	0.2080 (3)	3.0 (1)
C(1002)	0.4052 (4)	0.2125 (4)	0.1297 (3)	4.1 (1)
C(1003)	0.5278 (5)	0.1337 (4)	0.1230 (3)	4.9 (1)
C(1004)	0.5900 (5)	0.0861 (4)	0.1935 (4)	5.1 (1)
C(1005)	0.5293 (5)	0.1166 (4)	0.2707 (3)	4.9 (1)
C(1006)	0.4074 (4)	0.1966 (4)	0.2780 (3)	4.1 (1)
B	0.0801 (6)	0.1181 (5)	0.7342 (4)	5.1 (2)
H(2161)	−0.060 (4)	0.422 (3)	0.315 (3)	2 (1)[a]

[a] *B* value for atom refined isotropically. *B* values for anisotropically refined atoms are given in the form of the isotropic equivalent thermal parameter defined as $(4/3)[a^2\beta(1,1) + b^2\beta(2,2) + c^2\beta(3,3) + ab(\cos\gamma)\beta(1,2) + ac(\cos\beta)\beta(1,3) + bc(\cos\alpha)\beta(2,3)]$.

Discussion

Several salient structural features of [Ru(OEP-*N*-C_6H_5)(C_6H_5)]$^+$ are evident in Figure 1. First, the proximity of H(2161) and Ru (2.52 Å), which is less than the van der Waals

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Table III. Selected Bond Distances (Å) and Angles (deg) for $[Ru(OEP\text{-}N\text{-}C_6H_5)(C_6H_5)](BF_4)$

	Distances		
Ru–N(21)	2.163 (3)	Ru–N(23)	1.993 (3)
Ru–N(22)	2.049 (3)	Ru–N(24)	2.035 (3)
Ru–C(1001)	1.999 (4)	Ru–H(2161)	2.52 (4)
N(23)–C(11)	1.374 (5)	N(23)–C(14)	1.382 (5)
N(21)–C(4)	1.467 (5)	N(21)–C(19)	1.453 (5)
N(21)–C(211)	1.501 (5)	C(216)–H(2161)	0.91 (5)
	Angles		
N(21)–Ru–N(22)	89.6 (1)	N(21)–Ru–N(23)	168.5 (1)
N(21)–Ru–N(24)	88.6 (1)	N(21)–Ru–C(1001)	91.9 (1)
N(21)–Ru–H(2161)	67 (1)	N(22)–Ru–N(23)	90.0 (1)
N(22)–Ru–N(24)	171.1 (1)	N(22)–Ru–C(1001)	97.1 (1)
N(22)–Ru–H(2161)	64 (1)	N(23)–Ru–N(24)	90.2 (1)
N(23)–Ru–C(1001)	99.5 (1)	N(23)–Ru–H(2161)	103 (1)
N(24)–Ru–C(1001)	91.1 (1)	N(24)–Ru–H(2161)	108 (1)
C(1001)–Ru–H(2161)	150 (1)	Ru–N(21)–C(211)	101.4 (2)
Ru–H(2161)–C(216)	110 (3)	C(211)–C(216)–H(2161)	119 (3)
		C(215)–C(216)–H(2161)	121 (3)

contact distance (ca. 2.9 Å[10]), reveals a bonding interaction between the two atoms. Such agostic hydrogens have been observed in many transition-metal complexes;[7] however, there is only one previous example[4f] of an *N*-aryl or *N*-alkyl porphyrin with a metal–H distance indicative of a metal–H bonding interaction. Second, the C skeleton of the pyrrole containing the $N\text{–}C_6H_5$ group is tilted 38° from the plane of the other three pyrroles; N(21) of this pyrrole is out of the plane of the C skeleton by an additional 0.09 Å. This distortion, common with N-substituted porphyrins,[4,5] results in a Ru–N distance 0.14 Å greater than the average of the other three Ru–N distances (2.026 Å). Third, the Ru-bound C_6H_5 is tilted 11.4° from normal to the porphyrin plane toward the $N\text{-}C_6H_5$ group; the N(21)–Ru–C(1001) bond angle is 91.9°, which is not significantly changed from the (presumed) 90° of the $Ru(OEP)(C_6H_5)_2$ precursor.

These cumulative distortions result in an unusual geometry about the Ru center. The Ru is 0.147 Å out of the porphyrin plane (three pyrroles) toward the "axial" C_6H_5 ligand, while N(21) is 0.126 Å out of the porphyrin plane and slightly farther away from the Ru than the normal, bonding position. These, coupled with the 150° C(1001)–Ru–H(2161) bond angle, result in a severely distorted octahedral geometry about the Ru center.

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Supplementary Material Available: A figure showing a complete ORTEP diagram and listings of crystal data collection parameters, hydrogen atom parameters, anisotropic thermal parameters, and full bond distances and angles (14 pages); a listing of observed and calculated structure factors (33 pages). Ordering information is given on any current masthead page.

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(10) The van der Waals radius for Ru is not available; however, using analogous transition metals as a guide (Bondi, A. *J. Phys. Chem.* **1964**, *68*, 441), we arrive at an estimate of 1.6–1.7 Å. The contact distance is thus ca. 2.9 Å.

Contribution from the Departments of Chemistry,
Yale University, New Haven, Connecticut 06511-8118,
and University of Alberta,
Edmonton, Alberta, Canada T6G 2G2

Synthesis and Structure of $[(\eta\text{-}C_5Me_5)Ir(CO)]_2$

R. G. Ball,[1a] W. A. G. Graham,*,[1a] D. M. Heinekey,*,[1b]
J. K. Hoyano,[1a] A. D. McMaster,[1a] B. M. Mattson,[1b,2]
and S. T. Michel[1b]

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Transition-metal dimers of the type $[(\eta\text{-}C_5R_5)M(CO)]_2$ (R = H, CH_3; M = Co, Rh, Ir) have received a great deal of attention in recent years in the context of their interesting reactivity and novel electronic structures.[3] For R = H, the cobalt dimer is isolable,[4] but the Rh analogue is unstable above 200 K.[5] Thermolysis of $(\eta\text{-}C_5H_5)Ir(CO)H_2$, which might have given the Ir analogue, instead affords the trimeric species $[(\eta\text{-}C_5H_5)Ir(CO)]_3$.[6] In the pentamethylcyclopentadienyl series (R = CH_3), the cobalt and rhodium complexes are easily prepared and have been extensively studied.[3] The iridium analogue $[(\eta\text{-}C_5Me_5)Ir(CO)]_2$ (**2**) was first reported as a byproduct of alkane activation reactions employing photolysis of $(\eta\text{-}C_5Me_5)Ir(CO)_2$ (**1**).[7] Subsequently, compound **2** was obtained as a minor byproduct in the thermolysis of $(\eta\text{-}C_5Me_5)\overline{Ir[C(p\text{-}ClC_6H_4){=}NOC}{=}O]CO$.[8] It has also been reported that **2** can be obtained in 33% yield from the reaction of $[(\eta\text{-}C_5Me_5)Ir(\mu\text{-}O)]_2$ with carbon monoxide.[9] As part of a study of **2** and its derivatives,[10] we have established a convenient synthetic route to **2** and determined the solid-state structure of **2**. We also report improved procedures for the preparation of $[(\eta\text{-}C_5Me_5)IrCl_2]_2$ and $(\eta\text{-}C_5Me_5)Ir(CO)_2$, which are the immediate precursors to **2**.

Experimental Section

All operations were carried out under an atmosphere of nitrogen by using standard Schlenk techniques. Pentamethylcyclopentadiene was prepared by the method of Bercaw.[11] Solvents were reagent grade and were used as received.

Preparation of $[(\eta\text{-}C_5Me_5)IrCl_2]_2$. To $IrCl_3{\cdot}3H_2O$ (2.0 g, 5.4 mmol) in 50 mL of degassed methanol was added excess pentamethylcyclopentadiene (1.5 mL). The mixture was refluxed for 36 h. After the mixture was cooled to room temperature, the product was isolated by filtration and washed with cold methanol. The supernatant and washings were combined and cooled to 0 °C for 24 h. Total yield: 2.0 g (91%). ^1H NMR (δ, relative to residual solvent): 1.60 ($CDCl_3$) (lit. δ 1.59).[12a]

Preparation of $(\eta\text{-}C_5Me_5)Ir(CO)_2$. $[(\eta\text{-}C_5Me_5)IrCl_2]_2$ (1.0 g, 1.26 mmol) was dissolved in 50 mL of CH_2Cl_2 in a pressure bottle. The bottle was flushed three times with 30 psi of CO and pressurized to 40 psi. After 3 h of stirring, 5 g of zinc (30 mesh) was added. The reactor was pressurized to 40 psi under CO and the mixture stirred for 12 h. Water (50 mL) was added. The mixture was stirred for 1 h and then filtered to remove the zinc. The CH_2Cl_2 layer was separated, and the aqueous layer was extracted with 3 × 20 mL of CH_2Cl_2. The CH_2Cl_2 extracts were combined, dried over $MgSO_4$, filtered, and evaporated to dryness. Sublimation (10^{-3} mmHg, 40 °C) affords $(\eta\text{-}C_5Me_5)Ir(CO)_2$, 0.76 g, 2.0 mmol, 80% yield. ^1H NMR (δ, relative to residual solvent): 2.15 ($CDCl_3$) (lit. δ 2.19).[12a] IR (ν_{CO}, cm^{-1}): 2005, 1930 (CH_2Cl_2); 2020, 1953 (hexane) (lit. 2000, 1925 (solvent not specified)).[12a]

Preparation of $[(\eta\text{-}C_5Me_5)Ir(CO)]_2$ (2). $(\eta\text{-}C_5Me_5)Ir(CO)_2$ (0.250 g, 0.652 mmol) and a boiling chip were placed in a 50-mL pear-shaped flask. Sufficient solvent (1-butanol) was added to give a solvent depth of 2 cm. The flask was outfitted with a reflux condenser and immersed 4.5 cm into an oil bath. The solution was refluxed for 1 week with the bath temperature maintained at 170 °C. After the solution was cooled to room temperature, volatiles were removed by pumping and the residue

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(1) (a) University of Alberta. (b) Yale University.

(2) Permanent address: Department of Chemistry, Creighton University, Omaha, NE.

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