metrical parameters are unreliable; those that are precisely determined are given in the caption to Figure 1.

Although a detailed discussion of the structure is unwarranted, two of its features merit comment. First, the gross structural features and geometry of 2 are similar to those of the related amido complex $Cp^*_2Hf(H)(NHMe)$ in which a degree of N-to-Hf π bonding is important;[6] at the very least we know that the PHPh ligand of 2 is in an orientation consistent with the molecular geometry required for effective P-to-Hf π overlap.[17] Second, the Hf–P bond length of 2.549 (8) Å indicates multiple-bond character in this linkage. The classic structure of $(C_5H_5)_2Hf(PEt_2)_2$ by Baker et al. gives good values for Hf–P single and double bonds since the geometry of one of the PEt_2 ligands is essentially pyramidal about P (Hf—P, 2.682 (1) Å) and that of the other is trigonal planar about P (Hf=P, 2.488 (1) Å).[18] Thus, the Hf–P bond order in 2 is about 1.5 on the basis of the bond length.

Conclusions

A series of monomeric $Cp^*_2M(X)(PRR')$ (M = Zr, Hf) complexes has been prepared from primary and secondary phosphines and derived lithium phosphide reagents, and the molecular structure of one of these complexes, $Cp^*_2Hf(H)(PHPh)$ (2), was determined by single-crystal X-ray diffraction methods. The structure of 2, when considered with its ^1H NMR data and reactivity characteristics, suggests that the phenylphosphido ligand is a moderately effective π-donor in this complex. Some reactions of these complexes with small molecules like CO, CO_2, H_2, and C_2H_4 have been explored. Particularly interesting results were obtained for the purple complex $Cp^*_2Hf(H)(PPh_2)$ (1), where the steric demands of the two bulky Cp* ligands usually result in facile reductive eliminations of diphenylphosphine; consistent with these reactivity trends, ^1H NMR data suggest that diphenylphosphide is a poor π-donor ligand in 1. Carbon dioxide reacts with 1 to give the product of insertion, with a surprising preference for insertion of CO_2 into the Hf–P bond instead of the Hf–H bond. Initial attempts to utilize halide complexes of primary phosphides as precursors to terminal phosphinidene complexes were not entirely successful, but the dehydrohalogenation of $Cp^*_2Hf(I)(PHPh)$ with $NaN(SiMe_3)_2$ appears to have resulted in the formation of a complex with the empirical formula "$Cp^*_2Hf(PPh)$".

The phosphide complexes reported here complement the array of known complexes of decamethylzirconocene and decamethylhafnocene that contain related amide and alkoxide ligands.

Acknowledgment. Financial support of the National Science Foundation (Grant CHE-8818607) and a fellowship from the Alfred P. Sloan Foundation (1989–1991) is sincerely appreciated by G.L.H. The NMR facilities were supported in part through the University of Chicago Cancer Center Grant (NIH-CA-14599). We thank Prof. R. A. Jones for sharing unpublished data on related Zr systems with us.

Registry No. 1, 121055-41-2; 2, 121055-42-3; 3, 121055-43-4; 4, 121055-44-5; 5, 121055-45-6; 6, 121055-46-7; 7, 121055-47-8; 8, 121055-48-9; 9, 121055-49-0; $Cp^*_2HfH_2$, 81956-87-8; $Cp^*_2HfI_2$, 92786-75-9; $Cp^*_2HfCl_2$, 85959-83-7; $Cp^*_2ZrCl_2$, 54039-38-2; $Cp^*_2Hf(CO)_2$, 76830-38-1; $Cp^*_2Hf(C_4H_8)$, 92786-86-2; $PHPh_2$, 829-85-6; PH_2Ph, 638-21-1; PH_2Cy, 822-68-4; LiPHCy, 51918-33-3; C_2H_4, 74-85-1.

Supplementary Material Available: Table III (atomic coordinates and isotropic thermal parameters), Table IV (bond lengths), Table V (bond angles), and Table VI (anisotropic thermal parameters) (4 pages); a listing of observed and calculated structure factors (8 pages). Ordering information is given on any current masthead page.

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Synthesis and Structure of the $[Rh_2(dmpe)_4(\mu\text{-dmpe})]^{2+}$ Salt of the Naked Cyclopentadienyl Anion: A Comparison of the Reactivity of $[(\eta\text{-Indenyl})Rh(\eta\text{-}C_2H_4)_2]$ and $[(\eta\text{-}C_5H_5)Rh(\eta\text{-}C_2H_4)_2]$

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Excess $Me_2PCH_2CH_2PMe_2$ (dmpe) displaces all π-bound ligands from $[(\eta\text{-}C_9H_7)Rh(\eta\text{-}C_2H_4)_2]$ (1) much faster than from $[(\eta\text{-}C_5H_5)Rh(\eta\text{-}C_2H_4)_2]$ (3) as shown by a competition experiment. Direct reaction of 3 with a large excess of dmpe yields the salt $[Rh_2(dmpe)_4(\mu\text{-dmpe})]^{2+}[C_5H_5^-]_2$ (4), which has been characterized by X-ray crystallography. A square-pyramidal geometry at each rhodium center was found with the bridging dmpe bonded weakly in the apical site. The C_5H_5 anions deviate only slightly from D_{5h} symmetry. Crystal data for 4: triclinic, $P\bar{1}$; $a = 9.405$ (1), $b = 10.259$ (2), $c = 14.951$ (3) Å; $\alpha = 111.38$ (1), $\beta = 99.55$ (1), $\gamma = 93.41$ (1)°; $U = 1313.4$ (4) Å^3; $Z = 1$; $R = 0.028$, $R_w = 0.032$.

Introduction

The question of ring slippage in transition-metal cyclopentadienyl and indenyl complexes has attracted much recent attention[1] in light of the enhanced reactivity of indenyl complexes compared with their η^5-C_5H_5 analogues.[2-5] During the course of our investigations[6] of

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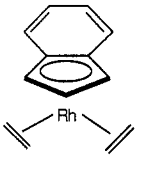
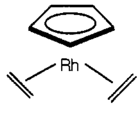
Table I. Bond Lengths (Å) for Compound 4

Rh–P(1)	2.3084 (7)	Rh–P(2)	2.2931 (8)
Rh–P(3)	2.2891 (8)	Rh–P(4)	2.3037 (8)
Rh–P(5)	2.4100 (7)	P(1)–C(1)	1.860 (4)
P(1)–C(3)	1.830 (4)	P(1)–C(4)	1.834 (4)
P(2)–C(2)	1.831 (4)	P(2)–C(5)	1.823 (4)
P(2)–C(6)	1.819 (3)	P(3)–C(7)	1.848 (4)
P(3)–C(9)	1.818 (4)	P(3)–C(10)	1.830 (4)
P(4)–C(8)	1.837 (4)	P(4)–C(11)	1.821 (3)
P(4)–C(12)	1.825 (4)	P(5)–C(13)	1.851 (4)
P(4)–C(14)	1.825 (4)	P(5)–C(15)	1.830 (4)
C(1)–C(2)	1.524 (5)	C(7)–C(8)	1.516 (5)
C(13)–C(13′)	1.534 (4)	C(16)–C(17)	1.380 (6)
C(17)–C(18)	1.389 (6)	C(18)–C(19)	1.391 (6)
C(19)–C(20)	1.392 (6)	C(20)–C(16)	1.383 (6)

indenyl ring slippage in $[(\eta\text{-}C_9H_7)RhL_2]$ complexes, we observed[7] displacement of all π-bound ligands when $[(\eta\text{-}C_9H_7)Rh(\eta\text{-}C_2H_4)_2]$ (1)[3,4] was treated with 2 molar equiv of $Me_2PCH_2CH_2PMe_2$ (dmpe). The resulting salt $[Rh(dmpe)_2]^+[C_9H_7]^-$ (2) was structurally characterized[7] and found to contain no close cation–anion contacts. It was of interest to carry out the analogous reaction using $[(\eta\text{-}C_5H_5)Rh(\eta\text{-}C_2H_4)_2]$ (3)[8] and to compare the relative rates of ligand displacement in 1 and 3.



1



3

Results and Discussion

A competition experiment was carried out to obtain relative rate data for the reactions of 1 and 3 with dmpe. Thus, an equimolar mixture of 1 and 3 in tetrahydrofuran-d_8, containing a small amount of *p*-dioxane as an integration standard, was treated with 2 molar equiv of dmpe and its ^1H NMR spectrum recorded. Only resonances due to 3, *p*-dioxane, and traces of dmpe were observed. No resonance due to any indenyl complexes were found, and upon examination of the sample, a voluminous yellow precipitate was observed. Isolation of the precipitate and characterization by ^1H and ^{31}P NMR spectroscopy (using acetonitrile-d_3) demonstrated it to be 2. Thus, *complete displacement of all ligands in* 1 *occurred prior to any significant reaction of dmpe with* 3.

Having observed again the extremely large rate enhancement for the indenyl complex, recently dubbed "the indenyl effect",[2] it still remained to determine whether dmpe would displace both ethylene and cyclopentadienide from 3. Reaction of 3 with an excess of dmpe in THF resulted in slow precipitation of yellow crystals of 4 in 82% yield. The $^{31}P\{^1H\}$ NMR spectrum of 4 in acetonitrile-d_3 displayed a doublet at δ 36.65 ($J_{Rh\text{-}P}$ = 120.6 Hz) for $[Rh(dmpe)_2]^+$ and a singlet at δ −39.67 of $^1/_4$ the integrated intensity for uncoordinated dmpe. The $^{13}C\{^1H\}$ NMR spectrum displayed a triplet at δ 105.28 ($J_{C\text{-}D}$ = 23.5 Hz) for the cyclopentadienide carbon atoms, indicating that exchange of the ring H's for solvent D's had occurred and that the cyclopentadienide was no longer coordinated to Rh. Analogous behavior was found for uncoordinated cyclopentadienide in the crystallographically characterized salt $[Re(NO)(CH_3)(PMe_3)_4]^+[C_5H_5]^-$ (5).[9] Resonances for both coordinated and uncoordinated dmpe were also observed in both the ^{13}C and ^1H NMR spectra. If samples of 4 in acetonitrile-d_3 were prepared and recorded rapidly at −30 °C, a singlet at δ 5.45 could be observed for the cyclopentadienide protons; however, upon warming, this signal disappeared as the exchange of H for solvent D occurred. In pyridine-d_5 solution, a singlet at δ 6.91 was observed for the $[C_5H_5]^-$ protons. We were intrigued by the occurrence of resonances for uncoordinated dmpe in all the NMR spectra and thus undertook a single-crystal X-ray structure determination of 4. An ORTEP diagram of 4 is presented in Figure 1, and bond distances and angles are given in Tables I and II.

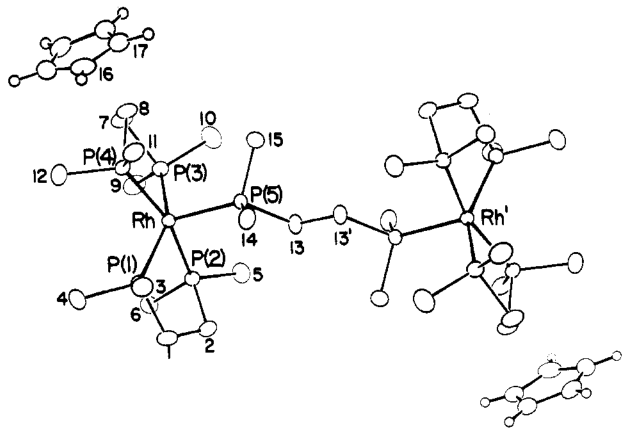


Figure 1. ORTEP view of 4 showing crystallographic numbering. Hydrogen atoms, except on $C_5H_5^-$, have been omitted for clarity.

Table II. Bond Angles (deg) for Compound 4

P(1)–Rh–P(2)	80.79 (2)	P(1)–Rh–P(3)	147.87 (2)
P(1)–Rh–P(4)	93.56 (2)	P(1)–Rh–P(5)	105.90 (2)
P(2)–Rh–P(3)	92.51 (2)	P(2)–Rh–P(4)	162.11 (2)
P(2)–Rh–P(5)	101.05 (2)	P(3)–Rh–P(4)	83.25 (2)
P(3)–Rh–P(5)	106.22 (2)	P(4)–Rh–P(5)	96.82 (2)
Rh–P(1)–C(1)	110.5 (1)	Rh–P(1)–C(3)	121.6 (1)
Rh–P(1)–C(4)	120.5 (1)	C(1)–P(1)–C(3)	102.7 (2)
C(1)–P(1)–C(4)	100.4 (2)	C(3)–P(1)–C(4)	97.8 (2)
Rh–P(2)–C(2)	107.2 (1)	Rh–P(2)–C(5)	122.9 (1)
Rh–P(2)–C(6)	118.6 (1)	C(2)–P(2)–C(5)	103.9 (2)
C(2)–P(2)–C(6)	101.3 (2)	C(5)–P(2)–C(6)	100.2 (2)
Rh–P(3)–C(7)	110.5 (1)	Rh–P(3)–C(9)	122.5 (1)
Rh–P(3)–C(10)	118.8 (2)	C(7)–P(3)–C(9)	100.9 (2)
C(7)–P(3)–C(10)	101.0 (2)	C(9)–P(3)–C(10)	99.8 (2)
Rh–P(4)–C(8)	108.0 (1)	Rh–P(4)–C(11)	121.7 (1)
Rh–P(4)–C(12)	119.8 (1)	C(8)–P(4)–C(11)	103.2 (2)
C(8)–P(4)–C(12)	100.9 (2)	C(11)–P(4)–C(12)	100.4 (2)
Rh–P(5)–C(13)	118.8 (1)	Rh–P(5)–C(14)	117.3 (1)
Rh–P(5)–C(15)	118.0 (1)	C(13)–P(5)–C(14)	100.7 (2)
C(13)–P(5)–C(15)	99.6 (2)	C(14)–P(5)–C(15)	98.9 (2)
P(1)–C(1)–C(2)	111.9 (1)	P(2)–C(2)–C(1)	108.0 (1)
P(3)–C(7)–C(8)	109.4 (1)	P(4)–C(8)–C(7)	108.3 (2)
P(5)–C(13)–C(13′)	118.3 (1)	C(20)–C(16)–C(17)	108.9 (2)
C(16)–C(17)–C(18)	108.2 (2)	C(17)–C(18)–C(19)	107.2 (2)
C(18)–C(19)–C(20)	108.8 (2)	C(19)–C(20)–C(16)	107.0 (3)

In the solid state, 4 consists of discrete cyclopentadienyl anions and the dimeric $[Rh_2(dmpe)_4(\mu\text{-}dmpe)]^{2+}$ dication.

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The dication sits on an inversion center which is located at the center of the C(13)–C(13′) bond of the bridging dmpe ligand. We will consider the geometry of the anion first. The C_5H_5 anion has C–C bond distances ranging from 1.380 (6) to 1.392 (6) Å, the average distance being 1.387 (6) Å. Although the esd's for these C–C distances and C–C–C angles are lower than those previously reported[9] for **5**, the average C–C distance in **5** of 1.399 (8) Å is not significantly different than ours. In **5**, the C–C–C angles show no significant deviation from their average value of 108.0 (5)°, ranging from 107.8 (5) to 108.3 (5)° whereas in **4**, although the average C–C–C angle is still 108.0 (3)°, the range is from 107.0 (3) to 108.9 (2)°. The C_5 ring is planar, the largest deviation from the least-squares plane of C(16)–C(20) being 0.003 (6) Å. Thus, there are only slight deviations from D_{5h} symmetry. The shortest interionic C–C contact in **4** is 3.580 (6) Å between C(18) of the anion and C(11) of the cation, compared with 3.65 Å in **5** and 3.60 Å in **2**. The shortest interionic H–H contact in **4**, 2.53 (7) Å, is between H(17) of the anion and one of the dmpe CH_3 groups, H(9b).

Recently, Pt and Pd salts of $[C_5H_5]^-$ have been observed;[10] however, no structural data were obtained. In addition, Ibers et al. reported[11] the isolation of $[Ir(dppe)_2]^+[C_5H_5]^-\cdot THF$ (dppe = $Ph_2PCH_2CH_2PPh_2$); although structural data were obtained for this salt, it was not included in the report. The $[C_5(CO_2Me)_5]^-$ anion, stabilized by the electron-withdrawing carbomethoxy groups, has been structurally characterized.[12]

The geometry of the dication is also of interest in that it is the first homoleptic $Rh(phosphine)_5$ complex and the only Rh(dmpe) complex other than **2** and the Rh^{III} salt[13] *trans*-$[Rh(dmpe)_2(CH_2Cl)Cl]Cl$ to have been structurally characterized. In addition, the geometrical preferences of d^8 ML_5 complexes have attracted much attention as there is often a very small energy difference between trigonal-bipyramidal (tbp) and square-planar forms.[14] The neutral complex $[Fe_2(dmpe)_4(\mu\text{-}dmpe)]$ has been reported,[15] and the observed AB_4 pattern in the ^{31}P NMR spectrum was assigned to a fluxional tbp species with the bridging dmpe in an equatorial site and rapid equilibration taking place between axial and equatorial phosphorus atoms of the chelating dmpe ligands. There appear to be very few crystallographically characterized d^8 ML_5 complexes in which all five ligands are phosphorus donors. The homoleptic Ni^{II} complex[16] $[Ni\{P(OCH)_3(CH_2)_3\}_5](ClO_4)_2$ and Co^I complex[17] $[Co\{P(OCH_2)_3CMe\}_5][Co(NO_3)_3NCMe]$ exhibit nearly perfect tbp geometries in the solid-state as do the tetrakis chelate complexes[18a] $[CoP(CH_2CH_2PPh_2)_3P(OMe)_3]BF_4$, $[NiP(CH_2CH_2PPh_2)_3P(OMe)_3](AsF_6)_2$, and $[Ir(PPh_3)(QP)]BPh_4$ [QP = tris(*o*-(diphenylphosphino)phenyl)phosphine].[18b] The tris chelate complex[19] $[CoPhP\{(CH_2)_2PPh_2\}_2\{P(OMe)_3\}_2]BF_4$ and the related monocarbonyl complex[19] $[CoPhP\{(CH_2)_3PPh_2\}_2P(OMe)_3CO]BF_4$ have been structurally characterized; the former complex displays distorted tbp coordination at Co, whereas the latter complex is square-pyramidal. The authors ascribe[19] the different geometries to differences in chelate ring size. It is curious, therefore, that $[Ir(Ph_2PCH_2CH_2PPh_2)_2Me]$ exhibits[11] a highly distorted tbp geometry *with one of the five-membered chelate rings lying in the pseudoequatorial plane.*

It was therefore interesting to find that **4** contains two d^8 Rh centers in only slightly distorted square-pyramidal geometries. The two chelating dmpe ligands occupy the equatorial plane (average Rh–P distance = 2.2986 (8) Å) with the bridging dmpe weakly coordinated at one axial site (Rh–P(5) = 2.4100 (7) Å). The Rh–P(5) distance is 0.1114 (8) Å greater than the average equatorial Rh–P distance consistent with the fact that the bridging dmpe ligand is dissociated from the complex in solution (vide supra).

Applications of the "indenyl effect" to rhodium-catalyzed organic transformations are currently under investigation.[20]

Experimental Section

General Procedures. All manipulations were performed under a nitrogen atmosphere in a glovebox. THF was dried and distilled from Na/benzophenone, and all deuteriated solvents were freeze–pump–thaw degassed and distilled on a high vacuum line. ^1H, $^{13}C\{^1H\}$, and $^{31}P\{^1H\}$ NMR spectra were recorded on a Bruker AC200 or AM250 spectrometer.

Competition Experiment. A solution of **1** (27.4 mg, 0.1 mmol) and **3** (22.4 mg, 0.1 mmol) in C_6D_6 (1.0 mL) was prepared and transferred to an NMR tube fitted with a septum cap. After 5.0 μL of *p*-dioxane was added (as internal integration standard), a ^1H NMR spectrum was recorded. Careful integration, using long relaxation delays, verified that equimolar quantities of **1** and **3** were present in solution. The NMR tube was returned to the glovebox, and dmpe (34 μL, 0.2 mmol) was added via syringe. A yellow precipitate formed immediately. The mixture was centrifuged, and ^1H, $^{13}C\{^1H\}$, and $^{31}P\{^1H\}$ NMR spectra were recorded demonstrating the presence of **3** in its initial concentration and the absence of **1** in solution. The yellow solid was isolated by filtration and shown to be **2** by ^1H and $^{31}P\{^1H\}$ NMR spectroscopy.

Synthesis of 4. To a solution of **3** (112 mg, 0.5 mmol) in THF (1 mL) was added dmpe (1.67 mL, 10 mmol) in THF (1 mL). An orange-yellow crystalline solid precipitated over the course of ca. 2.5 h, which was collected by filtration. As the first crop of crystals had formed relatively quickly, they were not of sufficient quality for an X-ray structure determination. When the filtrate was allowed to stand for ca. 24 h, a second crop of high quality single crystals deposited, which were again collected by filtration (overall yield 212 mg, 82%).

Atoms in bold refer to coordinated dmpe and those in italics refer to uncoordinated dmpe: $^{31}P\{^1H\}$ NMR (101.26 MHz, CD_3CN) δ 36.7 (d, J_{Rh-P} = 121 Hz, 8 **P**), −39.7 ppm (s, 2 *P*); $^{13}C\{^1H\}$ NMR (62.87 MHz CD_3CN) δ 105.3 (t, J_{C-D} = 23.5 Hz, $C_5D_5^-$), 32.6 (m, $P\mathbf{C}H_2\mathbf{C}H_2P$), 30.1 (s, br, $P\mathit{C}H_2\mathit{C}H_2P$), 20.7 (m, $P(\mathbf{C}H_3)_2$), 18.3 (s, br, $P(\mathit{C}H_3)_2$); ^1H NMR (250.13 MHz, CD_3CN, −30 °C) δ 5.45 (s, 5 H, $C_5H_5^-$), 1.75 (m, 8 H, $PC\mathbf{H}_2C\mathbf{H}_2P$), 1.47 (s, br, 24 H, $P(C\mathbf{H}_3)_2$), 1.33 (s, br, 2 H, $PC\mathit{H}_2C\mathit{H}_2P$), 1.04 (s, br, 6 H, $P(C\mathit{H}_3)_2$); ^1H NMR (250.13 MHz, C_5D_5N) δ 6.91 (s, 5 H, $C_5H_5^-$), 1.67 (m, 8 H, $PC\mathbf{H}_2C\mathbf{H}_2P$), 1.39 (s, br, 24 H, $P(C\mathbf{H}_3)_2$), 1.17 (s, br, 2 H, $PC\mathit{H}_2C\mathit{H}_2P$), 1.00 (s, 6 H, $P(C\mathit{H}_3)_2$).

Crystal Structure Determination for 4: yellow prisms, $Rh_2P_{10}C_{40}H_{90}$; M = 1086.712; triclinic, space group $P\bar{1}$; a = 9.405 (1), b = 10.259 (2), c = 14.951 (3) Å; α = 111.38 (1), β = 99.55 (1), γ = 93.41 (1)°; U = 1313.4 (4) Å^3; Z = 1; D_{calcd} = 1.374 g cm^{-3}; $F(000)$ = 570; T = 294 ± 1 K; λ = 0.71069 Å; μ(Mo Kα) = 9.39 cm^{-1}.

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Table III. Fractional Atomic Coordinates ($\times 10^4$) for the Non-Hydrogen Atoms and U_{eq} for Compound 4

atom	x	y	z	U_{eq},[a] Å^2
Rh	2568.8 (2)	862.1 (2)	2559.9 (2)	25
P(1)	2727.3 (8)	−990.6 (8)	1142.3 (5)	31
P(2)	925.5 (8)	−818.5 (8)	2635.8 (5)	30
P(3)	1323.9 (8)	2658.3 (8)	3325.5 (6)	33
P(4)	3640.8 (8)	2531.8 (8)	2099.9 (6)	32
P(5)	4613.4 (8)	905.2 (8)	3783.0 (5)	29
C(1)	1991 (4)	−2696 (3)	1170 (3)	43
C(2)	1625 (4)	−2513 (3)	2161 (3)	42
C(3)	4476 (4)	−1361 (4)	786 (3)	49
C(4)	1707 (5)	−1120 (5)	−50 (3)	52
C(5)	446 (4)	−762 (4)	3781 (3)	49
C(6)	−875 (3)	−1217 (4)	1856 (3)	42
C(7)	1842 (4)	4247 (4)	3085 (3)	49
C(8)	3405 (4)	4283 (3)	2953 (3)	42
C(9)	−648 (4)	2524 (4)	3042 (4)	54
C(10)	1641 (6)	3389 (5)	4667 (3)	62
C(11)	5576 (4)	2698 (4)	2090 (3)	47
C(12)	2895 (5)	2635 (5)	924 (3)	57
C(13)	4335 (3)	27 (4)	4636 (2)	39
C(14)	6193 (4)	138 (4)	3351 (3)	47
C(15)	5531 (5)	2626 (4)	4669 (3)	47
C(16)	8050 (5)	5154 (4)	1643 (3)	57
C(17)	8135 (4)	5754 (4)	2643 (3)	51
C(18)	6872 (4)	6358 (4)	2810 (3)	52
C(19)	6012 (5)	6112 (4)	1898 (6)	62
C(20)	6745 (5)	5373 (4)	1172 (3)	60

[a] $U_{eq} = (U_{11}U_{22}U_{33})^{1/3}$.

A crystal of dimensions 0.22 × 0.22 × 0.24 mm was thinly coated with epoxy and mounted on a Syntex $P2_1$ diffractometer. Accurate unit cell dimensions were calculated by using 15 reflections well distributed in reciprocal space ($22 < 2\theta < 30°$). Data were collected by the θ–2θ scan method ($2\theta \leq 55°$) using variable scan rates (2.93–29.30° min^{-1}) and a scan width of 0.8° below $K\alpha_1$ to 0.8° above $K\alpha_2$. The intensities of two standard reflections (107, 060) were monitored every 100 measurements. These exhibited only statistical fluctuations. From a total of 6087 unique reflections measured, 4859 with $I \geq 3\sigma(I)$ were considered observed and used in the structure solution and refinement. Data were corrected for Lorentz and polarization effects, but not absorption (μ = 9.39 cm^{-1}, transmission factors = 0.76–0.85). The structure was solved by Patterson and Fourier techniques and refined by full-matrix least-squares. The refinement progressed to an isotropic R of 0.058 and anisotropic R of 0.042 ($R = \sum||F_o| - |F_c||/\sum|F_o|$). At this stage a difference Fourier synthesis yielded all hydrogen atom sites. Inclusion of these atoms with isotropic thermal parameters led to convergence at R = 0.028 with R_w = 0.032 ($R_w = [\sum w(|F_o| - |F_c|)^2/\sum wF_o^2]^{1/2}$). An empirical weighting scheme of the form $w^{-1} = 1.64 - 0.034|F_o| + 0.0006|F_o|^2$ was included in the final cycles of refinement. A final difference Fourier exhibited maximum residuals of 0.5 e Å^{-3} in the vicinity of the rhodium atom. Scattering factors including the effects of anomalous scattering of rhodium were taken from ref 21. For hydrogen, the values of Stewart et al.[22] were used. Programs used have been reported elsewhere.[23] Atomic coordinates and isotropic thermal parameters are listed in Table III.

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Supplementary Material Available: Tables of hydrogen atom positions and isotropic thermal parameters, C–H bond lengths, and anisotropic thermal parameters (3 pages); a listing of structure factor amplitudes (25 pages). Ordering information is given on any current masthead page.

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