fraction points that are postulated on the basis of several properties.[12] Knowledge of the partial molar volume, $\bar{V}$, of the hydroxide ion in the solvent mixtures used would be valuable.[13] But a much better basis for analysis and interpretation of ΔV^* would be possible if $\bar{V}$ were also available for the iron(II) complex cations.

Data cannot be acquired with sufficient accuracy to permit quantitative correlation with solvent properties and solvent structure as they change with composition. However, it is evident that the magnitude of ΔV^* variation is greatest when alcohol cosolvent causes water structure change and breakdown for the lowest values of x_2 (among the alcohols studied) where these changes occur; that is, when *tert*-butyl alcohol is used as a solvent. The smaller effects seen in aqueous methanol reflect findings that the solvent structure reorganization positions are less well defined with the binary system.

Acknowledgment. We are grateful to the National Science Foundation (Grant No. CHE-7908399) for providing funds for the purchase of the Cary 219 spectrophotometer and to the Royal Society for a grant-in-aid toward the purchase of the Unicam SP 8-100 spectrophotometer. C.D.H. acknowledges, with thanks, provision of laboratory facilities and high-pressure equipment at the University of Leicester. We thank Dr. Paul R. Jones for nomenclature advice.

Registry No. Fe(hxsb)$^{2+}$, 88686-51-5; OH$^-$, 14280-30-9.

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Contribution from the School of Chemical Sciences
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Synthesis and Crystal Structure of $[Et_4N]_2[Re_7C(CO)_{21}Pd(C_9H_9)]$ (C_9H_9 = η^3-1-Phenylallyl). An Example of a Non-"Slipped" Palladium(II) Capping Moiety

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Received January 28, 1988

In a previous paper[1] we noted the similarity of the cluster $[Re_7C(CO)_{21}]^{3-}$ to cyclopentadienide, $[C_5H_5]^-$, and the dicarbollide ion, $[C_2B_9H_{11}]^{2-}$. We described the synthesis and characterization of a number of compounds of the general formula $[Re_7C(CO)_{21}ML_n]^{2-}$, where ML_n is a platinum-metal-based fragment. Variable-temperature ^{13}C NMR studies showed that these complexes adopt a (1,4)-bicapped octahedral geometry[2] in solution; a crystal structure of $[PPN]_2[Re_7C(CO)_{21}Pt(\eta^3\text{-2-methylallyl})]$ agreed with this assignment. Unfortunately, a detailed analysis of this structure was not possible due to disorder. In this note we describe the preparaton and full crystallographic characterization of the mixed-metal cluster $[Re_7C(CO)_{21}Pd(\eta^3\text{-1-phenylallyl})]^{2-}$. (The phenylallyl ligand will hereafter be referred to as C_9H_9.)

When a late-transition-metal-based moiety caps a carborane, the resulting cluster often exhibits a "slip" distortion, where the metal cap asymmetrically bridges the carborane face. The most thoroughly studied of these metallocarborane complexes are those of $[C_2B_9H_{11}]^{2-}$. In these compounds, the degree of slippage is defined as Δ, the distance between the perpendicular from the capping metal to the mean pentagonal C_2B_3 plane and the pentagon centroid.[3] This quantity depends on the capping metal and the ligands on the metal; for example, the fragment $Rh(PPh_3)_2$ symmetrically caps the carborane, whereas $Pd(PMe_3)_2$, $Pd(C_2H_4(NMe_2)_2)$, and $Au(S_2CNEt_2)$ display more open structures.[4-6] The electronic factors responsible for the slip distortion have been elucidated in a theoretical study by Mingos.[3] Similar distortions have been observed in cyclopentadienyl complexes of late transition metals. We were therefore interested in determining whether the slip distortion observed in many metallocarboranes and cyclopentadienyl compounds is also seen in our mixed-metal clusters.

Table I. Crystal Data for $[Et_4N]_2[Re_7C(CO)_{21}Pd(C_9H_9)]\cdot CHCl_3$

formula	$C_{48}H_{50}Cl_3N_2O_{21}PdRe_7$
fw	2506.08
cryst dimens, mm	0.2 × 0.3 × 0.4
system	monoclinic
space group	$P2_1$
a, Å	14.406 (5)
b, Å	19.988 (10)
c, Å	11.603 (6)
β, deg	112.08 (3)
V, Å^3	3096 (2)
Z	2
ρ_{calcd}, g/cm^3	2.688
diffractometer	Syntex P2$_1$
monochromator	graphite
scan range, deg	$3.0 < 2\theta < 54.0$
data collected	$\pm h,+k,+l$
λ(K*α*) of Mo radiation, Å	0.71073
T, K	300
no. of obsd reflcns	7611
no. of unique reflcns	7011
R	0.044
R_w	0.048

Table II. Selected Bond Distances (Å) and Angles (deg) for $[Et_4N]_2[Re_7C(CO)_{21}Pd(C_9H_9)]\cdot CHCl_3$

Distances			
Re1–Re2	2.967 (2)	Re1–Re3	2.936 (1)
Re2–Re3	2.959 (2)	Re1–Re4	2.964 (2)
Re2–Re4	2.963 (2)	Re3–Re4	2.953 (2)
Re2–Re5	2.969 (2)	Re3–Re5	3.017 (2)
Re3–Re6	3.009 (2)	Re4–Re6	3.001 (1)
Re5–Re6	3.081 (2)	Re2–Re7	2.968 (2)
Re4–Re7	2.978 (2)	Re5–Re7	3.016 (2)
Re6–Re7	3.067 (2)	Re5–Pd	2.879 (3)
Re6–Pd	2.952 (3)	Re7–Pd	2.786 (3)
Pd–C(1)	2.16 (3)	Pd–C2	2.01 (5)
Pd–C3	2.27 (5)	C1–C2	1.57 (7)
C2–C3	1.11 (7)	av Re–C$_{carbide}$	2.13 (3)
Angles[a]			
Re5–Pd–Re6	63.78 (7)	Re5–Pd–Re7	64.31 (7)
Re6–Pd–Re7	64.53 (6)	C1–C2–C3	128 (5)

[a] All angles within 1.0° of those required for a regular octahedron for Re2 to Re7 and a regular tetrahedron for the Re1 cap.

Results and Discussion

$[Et_4N]_2[Re_7C(CO)_{21}Pd(C_9H_9)]$ is easily prepared from $[Et_4N]_3[Re_7C(CO)_{21}]$ and $[Pd(C_9H_9)Cl]_2$; X-ray-quality crystals were grown from an acetone/chloroform solution. Tables I and II contain crystallographic data and pertinent bond distances and angles for this compound, respectively. Figure 1 shows a view of the entire mixed-metal dianion. The complex crystallizes with one molecule of chloroform of solvation per formula unit. The

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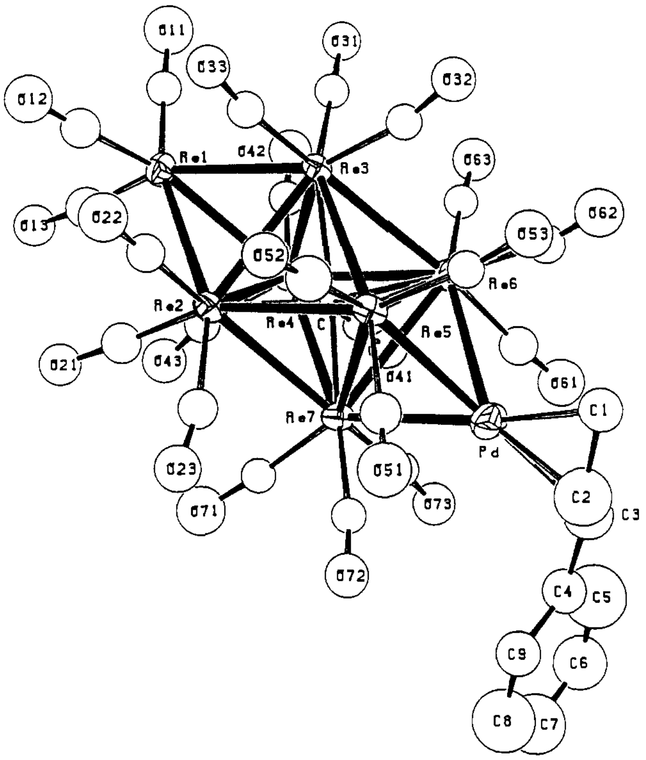


Figure 1. Molecular structure of $[Re_7C(CO)_{21}Pd(C_9H_9)]^{2-}$.

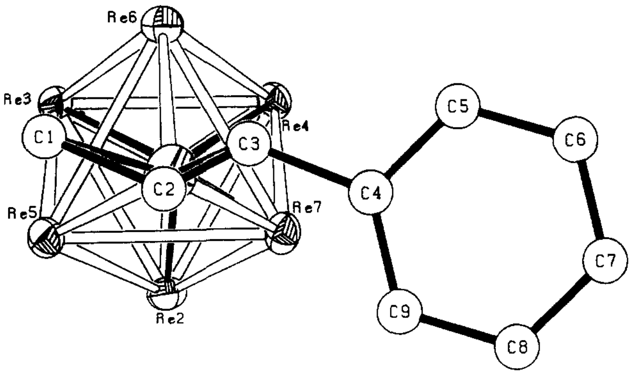


Figure 2. View of $[Re_7C(CO)_{21}Pd(C_9H_9)]^{2-}$ showing the local coordination to the palladium atom. All bonds to carbon, including the μ_6-C, are darkened.

cluster adopts a (1,4)-bicapped octahedral geometry; the Re–Re bond lengths span the range 2.94–3.08 Å, with the longest bonds forming the base of the Pd cap. The Re–Pd bond lengths are 2.786 (3), 2.879 (3), and 2.952 (3) Å (average 2.872 Å). These distances appear to be the first reported for rhenium–palladium bonds. The coordination geometry around the palladium atom is roughly octahedral (see Figure 2).

It is interesting to compare $[Re_7C(CO)_{21}Pd(C_9H_9)]^{2-}$ with its structurally characterized analogue $CpPd(C_9H_9)$.[7,8] In the Cp complex, the palladium atom is quite noticeably slipped; i.e., the metal atom is much closer to three carbon atoms of the Cp ring than to the other two. In this manner the palladium atom distorts toward a 16-electron bis(allyl) complex:

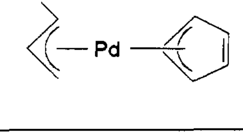
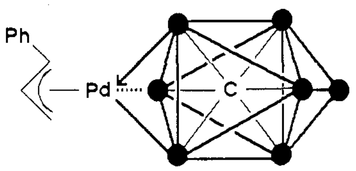


Table III. ^{13}C Chemical Shift Data (δ_C) for the C_9H_9 Groups in $[Re_7C(CO)_{21}Pd(C_9H_9)]^{2-}$ and $[Pd(C_9H_9)Cl]_2$ and for $H_3CCH{=}CH(C_6H_5)$ and $H_2C{=}CHCH_2(C_6H_5)$

	Re_7Pd	$[Pd(C_9H_9)Cl]_2$	$H_3CCH{=}CH(C_6H_5)$	$H_2C{=}CH-CH_2(C_6H_5)$
C1	66.6	64.2	18.3	115.6
C2	105.3	112.6	129.0	128.5
C3[a]	93.4	86.6	131.4	40.3

[a] Bears phenyl group.

A similar distortion in the Re_7Pd complex would move the Pd-(η^3-allyl) moiety toward an edge-bridging configuration:



There is a marked variation in the Re–Pd bond lengths in $[Re_7C(CO)_{21}Pd(C_9H_9)]^{2-}$ but no clear distortion of the Pd cap toward two rhenium atoms and away from the third. The "slip" parameter, Δ, is defined as the distance between the palladium atom and the perpendicular to the Re_3 plane passing through the centroid of the plane. For this complex, $\Delta = 0.13$ Å, a value that for metallocarboranes is considered indicative of an unslipped, closo geometry.[9]

A number of features of the allyl group bear mentioning. First, the allyl C_3 plane forms an angle of 19.9° with the plane of the capped trirhenium face. This angle agrees well with the analogous values of 20.4 and 22.2° observed for the two disordered ring orientations in $CpPd(C_9H_9)$.[7] Second, as observed for other phenylallyl complexes,[7] the carbon atom bearing the phenyl group is significantly further from the palladium atom than the other terminal carbon atom. A measure of this allyl "tilt" is β, defined as the angle between the vector Pd–O (O is the center of mass of the C_3 allyl fragment) and a vector that is parallel to the C1–C3 vector and which passes through O.[10] For $[Re_7C(CO)_{21}Pd(C_9H_9)]^{2-}$, $\beta = 84.2°$. This value indicates a more severe tilting of the phenylallyl group than has previously been observed (for example, $\beta = 88.4°$ for $CpPd(C_9H_9)$[7]), probably due to steric interactions of the phenyl group with the carbonyl ligands on the capped Re_3 face. The angle between the vectors Pd–O and O–C2, defined as τ, is 101.1°, which is typical for allyl compounds. The middle carbon of the allyl group is closer to the palladium atom than either terminal carbon. Third, the C–C bond distances in the allyl ligand appear to differ greatly (1.11 (7), 1.57 (7) Å). However, this difference is exaggerated by the high thermal motion of C2, which likely indicates some unresolved disorder at this site.

Although the allyl ligand appears to be an $\eta^1{:}\eta^2$ ligand in the solid-state study, solution ^{13}C NMR data support a fully delocalized η^3-allyl structure. Table III compares the chemical shift data for $[Re_7C(CO)_{21}Pd(C_9H_9)]^{2-}$, $[Pd(C_9H_9)Cl]_2$, and the free olefins $H_3CCH{=}CH(C_6H_5)$ and $H_2C{=}CHCH_2(C_6H_5)$.[11] The chemical shift values for the Re_7Pd complex are much closer to those for the allylic dimer $[Pd(C_9H_9)Cl]_2$ than for the clearly sp^3- and sp^2-hybridized carbon atoms of the olefins. In addition, one might expect the $^1J_{C-H}$ values for a localized C_9H_9 group to vary as follows due to the difference in carbon atom hybridization: $^1J_{C(1)-H} < {}^1J_{C(2)-H} \approx {}^1J_{C(3)-H}$. ($^1J_{C-H}$(aliphatic) $\approx$ 120–130 Hz; $^1J_{C-H}$(olefinic) $\approx$ 140–160 Hz.[12]) This is plainly not the case: $^1J_{C(1)-H} = {}^1J_{C(2)-H} = 163$ Hz; $^1J_{C(3)-H} = 142$ Hz.

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Table IV. Refined Atomic Coordinates for $[Re_7Pd(CO)_{21}(C_9H_9)][NC_8H_{20}]_2{\cdot}CHCl_3$

	x/*a*	*y*/*b*	*z*/*c*
Re1	0.1801 (1)	0.3000	−1.0462 (1)
Re2	0.08336 (8)	0.22556 (9)	−0.9002 (1)
Re3	0.27677 (8)	0.29478 (9)	−0.77398 (9)
Re4	0.26855 (9)	0.17294 (8)	−0.92147 (9)
Re5	0.17730 (9)	0.22102 (8)	−0.6245 (1)
Re6	0.37164 (9)	0.16768 (8)	−0.6437 (1)
Re7	0.17021 (9)	0.09756 (8)	−0.7771 (1)
Pd	0.2589 (2)	0.0937 (1)	−0.5184 (2)
Cl1	0.750 (1)	0.0409 (9)	−0.009 (2)
Cl2	0.551 (1)	0.074 (2)	−0.060 (2)
Cl3	0.598 (1)	0.0096 (8)	−0.247 (1)
O11	0.328 (2)	0.374 (2)	−1.113 (3)
O12	0.042 (2)	0.414 (1)	−1.132 (2)
O13	0.090 (2)	0.248 (1)	−1.320 (2)
O21	−0.040 (2)	0.185 (1)	−1.171 (2)
O22	−0.030 (2)	0.355 (1)	−0.927 (2)
O23	−0.094 (2)	0.157 (1)	−0.858 (2)
O31	0.456 (2)	0.342 (1)	−0.833 (2)
O32	0.393 (2)	0.360 (1)	−0.523 (2)
O33	0.171 (2)	0.426 (1)	−0.825 (2)
O41	0.402 (2)	0.045 (1)	−0.887 (2)
O42	0.427 (2)	0.223 (1)	−1.019 (2)
O43	0.150 (2)	0.103 (1)	−1.171 (2)
O51	0.034 (2)	0.152 (2)	−0.524 (3)
O52	0.033 (2)	0.331 (1)	−0.646 (2)
O53	0.308 (2)	0.271 (1)	−0.362 (2)
O61	0.473 (2)	0.028 (1)	−0.558 (2)
O62	0.490 (2)	0.212 (1)	−0.382 (2)
O63	0.553 (2)	0.200 (1)	−0.703 (2)
O71	0.025 (2)	0.030 (1)	−1.019 (3)
O72	0.019 (2)	0.030 (1)	−0.677 (2)
O73	0.282 (2)	−0.039 (1)	−0.742 (2)
N1	0.203 (2)	0.525 (1)	−0.477 (2)
N2	0.664 (2)	0.330 (1)	−0.012 (2)
C	0.226 (2)	0.188 (1)	−0.774 (2)
C1	0.315 (3)	0.110 (2)	−0.320 (3)
C2	0.271 (4)	0.039 (3)	−0.398 (5)
C3	0.306 (4)	−0.001 (2)	−0.404 (4)
C4	0.264 (2)	−0.068 (2)	−0.463 (3)
C5	0.335 (2)	−0.112 (2)	−0.474 (3)
C6	0.306 (2)	−0.176 (2)	−0.523 (3)
C7	0.206 (2)	−0.196 (2)	−0.560 (3)
C8	0.135 (2)	−0.151 (2)	−0.549 (3)
C9	0.164 (2)	−0.088 (2)	−0.501 (3)
C11	0.264 (2)	0.342 (2)	−1.087 (3)
C12	0.093 (3)	0.370 (2)	−1.096 (3)
C13	0.123 (3)	0.267 (2)	−1.202 (3)
C21	0.014 (3)	0.198 (2)	−1.063 (3)
C22	0.015 (2)	0.304 (2)	−0.917 (3)
C23	−0.021 (3)	0.180 (2)	−0.862 (3)
C31	0.387 (2)	0.321 (1)	−0.815 (3)
C32	0.347 (2)	0.328 (1)	−0.615 (3)
C33	0.206 (2)	0.373 (2)	−0.813 (3)
C41	0.353 (2)	0.090 (1)	−0.890 (2)
C42	0.362 (2)	0.205 (2)	−0.985 (3)
C43	0.187 (3)	0.136 (2)	−1.084 (3)
C51	0.090 (3)	0.173 (2)	−0.562 (3)
C52	0.091 (3)	0.286 (2)	−0.638 (3)
C53	0.250 (2)	0.250 (2)	−0.460 (3)
C61	0.430 (3)	0.080 (2)	−0.589 (3)
C62	0.443 (3)	0.191 (2)	−0.473 (3)
C63	0.482 (2)	0.188 (1)	−0.685 (3)
C71	0.088 (2)	0.052 (1)	−0.922 (3)
C72	0.079 (2)	0.054 (1)	−0.707 (3)
C73	0.241 (3)	0.015 (2)	−0.749 (3)
C81	0.091 (4)	0.548 (3)	−0.549 (4)
C82	0.055 (4)	0.509 (3)	−0.681 (4)
C83	0.269 (3)	0.541 (2)	−0.554 (4)
C84	0.272 (3)	0.618 (2)	−0.582 (4)
C85	0.212 (3)	0.455 (2)	−0.446 (3)
C86	0.144 (3)	0.431 (2)	−0.384 (3)
C87	0.227 (3)	0.564 (2)	−0.357 (4)
C88	0.335 (4)	0.541 (3)	−0.280 (5)
C91	0.559 (3)	0.325 (2)	−0.105 (4)
C92	0.530 (4)	0.366 (3)	−0.213 (4)
C93	0.738 (4)	0.302 (3)	−0.077 (5)
C94	0.710 (6)	0.244 (4)	−0.100 (7)
C95	0.699 (4)	0.393 (2)	0.045 (4)
C96	0.628 (4)	0.429 (3)	0.091 (5)
C97	0.664 (3)	0.282 (2)	0.096 (4)
C98	0.754 (4)	0.275 (3)	0.219 (5)
C100	0.633 (4)	0.015 (3)	−0.086 (5)

Experimental Section

Unless otherwise noted, all operations were carried out under nitrogen with the use of standard Schlenk techniques. Reaction solvents were dried and distilled immediately before use. $[Pd(C_9H_9)Cl]_2$ was synthesized from Na_2PdCl_4 (Engelhard) and cinnamyl chloride (Lancaster) by a literature method.[13] $[Et_4N]_3[Re_7C(CO)_{21}]$ was prepared as described previously.[14] IR spectra of pure compounds were recorded on a Perkin-Elmer Model 1750 Fourier transform spectrophotometer. NMR spectra were recorded on a Nicolet NT-360 instrument. Samples for fast-atom-bombardment mass spectrometry were suspended in a matrix of dithioerythritol/dithiothreitol; spectra were obtained by the staff of the Mass Spectrometry Center of the University of Illinois. Elemental analyses were performed by the Microanalytical Laboratory of the School of Chemical Sciences at the University of Illinois.

Synthesis of $[Et_4N]_2[Re_7C(CO)_{21}Pd(C_9H_9)]{\cdot}CHCl_3$. $[Et_4N]_3[Re_7C(CO)_{21}]$ (62.4 mg, 0.0272 mmol) and $[Pd(C_9H_9)Cl]_2$ (30.2 mg, 0.0583 mmol) were dissolved in acetonitrile to give a green solution. After 45 min of stirring at room temperature, the solvent was removed under vacuum; the resulting green oil was dissolved in 10 mL of acetone. To this solution was added 60 mL of chloroform. This mixture was stored at −15 °C for 2 days to give a precipitate of dark green crystals (59.3 mg, 87%). Anal. Calcd for $C_{48}H_{50}N_2Re_7PdO_{21}Cl_3$: C, 23.00; H, 2.01; N, 1.12. Found: C, 23.21; H, 2.14; N, 1.04. IR, ν_{CO}(acetone): 2040 (w), 1989 (vs), 1960 (w), 1931 (w), 1914 (w), 1878 (vw) cm^{-1}. ^1H NMR ($(CD_3)_2CO$, 20 °C): δ 1.38 (tt, 24 H, CH_3CH_2N), 3.47 (q, 16 H, CH_3CH_2N), 3.94 (d, 1 H, J = 11.7 Hz, anti H of $-CH_2$), 4.66 (d, 1 H, J = 6.7 Hz, syn H of $-CH_2$), 5.52 (d, 1 H, J = 11.8 Hz, $-CHPh$), 6.07 (m, 1 H, $-CH-$), 7.3–8.0 (m, 5 H, C_6H_5). ^{13}C NMR ($(CD_3)_2CO$, 20 °C): δ 66.6 (t, 1 C, $^1J_{C-H}$ = 163 Hz, $-CH_2$), 93.4 (d, 1 C, $^1J_{C-H}$ = 142 Hz, $-CHPh$), 105.3 (d, 1 C, $^1J_{C-H}$ = 163 Hz, $-CH-$), 128.5, 128.7, 129.6, 138.5 (2:1:2:1 C; meta, para, ortho, ipso carbons of phenyl ring), 192.3, 195.7, 206.8 (all s, 9:9:3 C, Re–CO). FAB-MS (negative ion): *m*/*z*-(^{187}Re, ^{106}Pd) 2262 (M − Et_4N), 2132 (M − $2Et_4N$), 2015 ($Re_7C(CO)_{21}Pd$), 1909 ($Re_7C(CO)_{21}$), 1909 − 28*x*, *x* = 1–9($Re_7C(CO)_{21}$ − *x*CO's). Large prisms suitable for X-ray studies were obtained by slow evaporation of an acetone/chloroform solution in air.

X-ray Crystallography. Crystal and diffraction data are shown in Table I. The 7011 unique reflections (R_i = 0.021, 4363 observed, $I > 2.58\sigma(I)$) were corrected for anomalous dispersion, absorption (μ = 142.95 cm^{-1}, maximum and minimum numerical transmission factors 0.138 and 0.066), Lorentz, and polarization effects. Correction for extinction was tested but not applied. The structure was solved by direct methods (SHELXS-86), which located the Re and Pd atoms, and subsequent difference Fourier syntheses gave positions for the remaining non-hydrogen atoms, including a chloroform solvate molecule. Refinement was effected with SHELX-76. The H atoms bound to C1, C2, and C3 were never located; the remaining H atoms were included as fixed contributors in idealized positions. The phenyl ring was refined as a rigid idealized group. In the final least-squares cycle the Re, Pd, and Cl atoms were refined with anisotropic thermal coefficients, the remaining non-hydrogen atoms were refined with isotropic coefficients, and a group isotropic thermal parameter was varied for the H atoms. The total number of variables was 372. The final difference Fourier map (range $1.28 > e/Å^3 > -2.43$) located maximum residual electron density in the vicinity of the metal atoms. A final analysis of variance showed no apparent systematic errors.

Acknowledgment. This work was supported by National Science Foundation Grants DMR 86-12860 to the Materials Research Laboratory and CHE 84-07233 to J.R.S.

Supplementary Material Available: Tables of derived atomic coordinates, thermal parameters, and bond lengths and angles for $[Et_4N]_2[Re_7C(CO)_{21}Pd(C_9H_9)]$ (8 pages); a table of observed and calculated structure factors (23 pages). Ordering information is given on any current masthead page.

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