

Figure 2. Seven-coordinate monocapped-trigonal-prismatic environment of the manganese atom in $\text{Mn}(\text{BF}_4)_2$. The Mn, F(1), F(2), and F(3) atoms all lie on a mirror plane.

independent Mn–F distances of 2.10 (1) and 2.13 (1) Å found in MnF_2 , where the metal atom adopts a slightly distorted octahedral environment.¹⁹ The distances in $\text{Mn}(\text{BF}_4)_2$ are clearly longer, presumably the result of the higher coordination number.

The metal atom is situated within the trigonal prism component of its coordination polyhedron [defined by F(1), F(3), F(4'), F(5), and F(5')] and lies 0.442 Å from the plane through the atoms defining the capped face [F(4), F(4'), F(5), and F(5')]. This face is precisely planar, a consequence of the crystallographic symmetry, and the other two rectangular faces are essentially planar, the maximum deviation from planarity being 0.018 Å; the distance of the Mn atom from these faces is 1.020 Å. The capping atom is F(2) with the Mn–F(2) bond almost exactly normal to the face, the angle between the normal and the bond being 1.5°. The two independent distances between F(2) and the atoms of the capped face are 2.741 (6) and 2.745 (7) Å. The closest approach to the two uncapped rectangular faces is made by F(6^{VII}) and F(6^{VIII}), but the very long distance of 3.433 (4) Å formed by these atoms to the manganese atom indicates that there is no significant bonding between them and that the Mn atom is truly seven-co-

ordinate. This is supported by a comparison of the F...F distances around the coordination polyhedra. Those between the symmetry-related atoms of the capped face are 3.192 (5) and 3.176 (5) Å. These are very much larger than the remaining F...F distances, which all lie in the range 2.655 (5) – 2.800 (5) Å, indicating that neither of the two remaining rectangular faces is capped. These distances also indicate that the introduction of the seventh ligand expands the capped face by folding the other two rectangular faces about the hinge formed by F(1) and F(3).

BF_4 Tetrahedral Units. The relationship of the two crystallographically independent BF_4 tetrahedral with respect to the MnF_7 unit is shown in Figure 1. The BF_4 groups adopt different bridging arrangements. Each of the fluorine atoms of that group which has B(2) as its central atom also participates in the coordination polyhedron of a different Mn atom and hence may be considered in a formal sense to be quadruply bridging and described as $(\text{BF}_4)_{4/4}$. Only three of the fluorine atoms of the tetrahedral unit that has B(1) as its central atom coordinate a Mn atom; the fourth fluorine atom F(6), as has already been noted, does not. This BF_4 unit is therefore triply bridging and can be described as $(\text{BF}_4)_{3/3}$. Thus, the overall structure can be written as $\text{Mn}(\text{BF}_4)_{4/4}(\text{BF}_4)_{3/3}$.

As can be seen from Table III, the B–F bond lengths, which lie between 1.341 (10) and 1.408 (10) Å, are comparable with those found, for example, for $\text{Ca}(\text{BF}_4)_2$ ²⁰ [1.384 (3)–1.393 (3) Å] and KBF_4 ²¹ [1.378 (5)–1.391 (3) Å]. As expected, the shortest bond is B1–F(6) since F(6) does not participate in any further bonding. The bond angles around the B atoms are close to the ideal tetrahedral value.

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Registry No. $\text{Mn}(\text{BF}_4)_2$, 30744-82-2; BF_3 , 7637-07-2; HF, 7664-39-3; Mn, 7439-96-5.

Supplementary Material Available: Listings of thermal parameters and deviations from least-squares mean planes and a figure that depicts the atomic arrangement in the unit cell (3 pages); a listing of observed and calculated structure factors (3 pages). Ordering information is given on any current masthead page.

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Notes

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Estimation of the Rh–Rh Bond Dissociation Energy in the (Octaethylporphyrinato)rhodium(II) Dimer by ¹H NMR Line Broadening

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The octaethylporphyrin rhodium dimer [(OEP)Rh]₂ has an interesting range of organometallic reactivity that includes reaction with H₂, reaction with C–H bonds in alkylaromatics^{1,2} and aldehydes and ketones,³ and addition to C=C⁴ and CO⁵ multiple

bonds. The reactivity patterns of [(OEP)Rh]₂ in hydrocarbon media suggest the importance of the metallo radical, (OEP)Rh^{II•}, and Halpern has demonstrated that the kinetics for addition of [(OEP)Rh]₂ to styrene can be described by a metallo radical chain mechanism.⁴ Thermodynamic and reactivity studies for the (OEP)Rh system have suggested that the (OEP)Rh–C bonds must be relatively strong (~50–60 kcal/mol) and that the Rh–H bond energy is in the expected range (~60 kcal/mol).⁶ Evaluation of absolute Rh–H and Rh–C bond energies for the (OEP)Rh system depends upon having a reliable value for the Rh–Rh bond energy in the dimer [(OEP)Rh]₂. This paper reports on an estimation of the Rh–Rh bond dissociation energy in [(OEP)Rh]₂ from ¹H NMR line broadening studies and the use of this value in deriving the (Rh–H)BDE in (OEP)Rh–H and the (Rh–C)BDE in (OEP)Rh–CHO.

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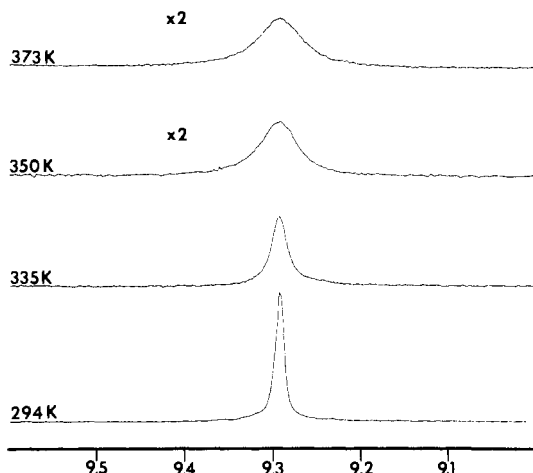


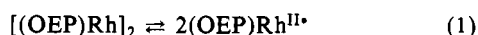
Figure 1. Line broadening of the porphyrin methyne hydrogen peak with increasing temperature as seen in the 200-MHz ^1H NMR spectrum of a $(4.8 \pm 0.1) \times 10^{-3}$ M solution of $[(\text{OEP})\text{Rh}]_2$ in benzene- d_6 . Temperatures are measured by an internal ethylene glycol capillary to within ± 0.4 K.

Experimental Section

$[(\text{OEP})\text{Rh}]_2$ was prepared by literature methods.² The sample must be free from $(\text{OEP})\text{Rh}-\text{H}$, which could contribute to the line broadening by exchange with $[(\text{OEP})\text{Rh}]_2$. In order to rule out trace concentrations of $(\text{OEP})\text{RhH}$, experiments were conducted in three sets, each using $[(\text{OEP})\text{Rh}]_2$ from a different preparation. Stock solutions of $[(\text{OEP})\text{Rh}]_2$ in benzene- d_6 were prepared under argon atmosphere in a Vacuum Atmospheres inert-atmosphere box. The benzene- d_6 used was predried over CaH_2 or Na/benzophenone and freshly distilled prior to making up the solutions. Approximately 0.40 mL of solution was syringed into an NMR tube containing a 0.70 mm o.d. capillary tube filled with ethylene glycol as a temperature reference and equipped with a stopcock for use on a high-vacuum line. The argon was removed by three freeze-pump-thaw cycles, and the tubes were sealed. The six samples that were studied contained concentrations of $[(\text{OEP})\text{Rh}]_2$ that varied from 1×10^{-4} to 5×10^{-3} M and were measured over the range 290–360 K. Proton NMR spectra were recorded on Bruker WP 250, WM200SY, and AF200SY spectrometers equipped with a Bruker VT-1000 temperature controller. Typical spectral parameters used were selected to give a digital resolution of 0.365 Hz. The internal temperature in the sample was measured within ± 0.4 K by using the separation of the methylene and hydroxyl protons of ethylene glycol. No measurable concentration or frequency dependence of the methyne porphyrin ^1H NMR line width was observed. The full resonance line width at half-maximum amplitude ($\nu_{1/2}$) is related to T_2 by the relationship $T_2^{-1} = \pi\nu_{1/2}$. The contribution of chemical exchange to the observed T_2^{-1} is $T_2^{-1}(\text{EX}) = T_2^{-1}(\text{obsd}) - T_2^{-1}(\text{nat})$. The natural line width ($\nu_{1/2}(\text{nat}) = 1.83$ Hz) was determined from the observed line width at 250 K, where the contribution from exchange is negligible. The slope of the temperature dependence ($\Delta \ln T_2^{-1}(\text{EX})$ vs $\Delta(1/T)$) gives the enthalpy of activation for the process responsible for the line broadening. In the case of $[(\text{OEP})\text{Rh}]_2$, line broadening that occurs at elevated temperatures is ascribed to formation of a metallo radical: $(\text{OEP})\text{Rh}-\text{Rh}(\text{OEP}) \rightleftharpoons 2(\text{OEP})\text{Rh}^{\cdot}$. The forward activation energy (ΔH_f^*) for homolysis of $[(\text{OEP})\text{Rh}]_2$ in benzene is 18.5 ± 0.8 kcal/mol. Similar results were obtained in cyclohexane ($\Delta H_f^* = 19.5 \pm 1.0$ kcal/mol).

Results and Discussion

$(\text{OEP})\text{Rh}^{\cdot}$ occurs predominantly as a diamagnetic Rh–Rh-bonded dimer in hydrocarbon solvents. The macrocyclic porphyrin ligands restrict the approach of the two $(\text{OEP})\text{Rh}^{\cdot}$ fragments, which results in a relatively weak Rh–Rh bond. The ^1H NMR spectrum of $[(\text{OEP})\text{Rh}]_2$ at room temperature is sharp, but as the temperature is raised, all of the ^1H NMR peaks of the porphyrin ligand broaden. Temperature dependence of the porphyrin methyne hydrogen line width is illustrated in Figure 1. The observed ^1H NMR line broadening is ascribed to exchange of the diamagnetic dimer, $[(\text{OEP})\text{Rh}]_2$ ($S = 0$), with the paramagnetic monomer, $(\text{OEP})\text{Rh}^{\cdot}$ ($S = 1/2$) (eq 1). Absence of an observed



line width dependence on the concentration of $[(\text{OEP})\text{Rh}]_2$ sup-

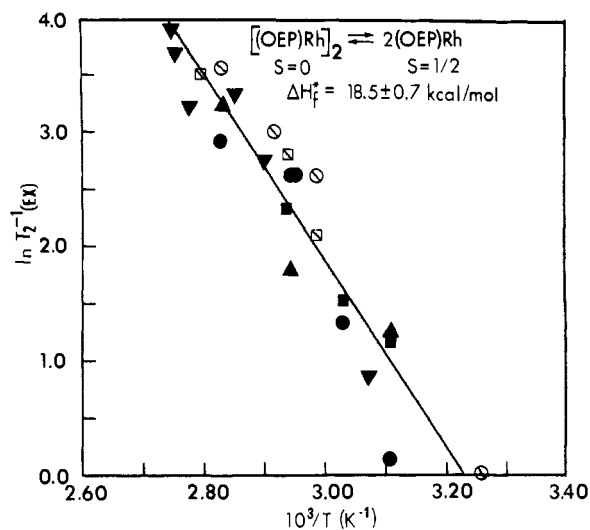


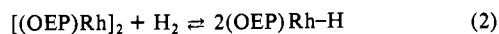
Figure 2. Arrhenius plot showing $T_2^{-1}(\text{EX})$ vs $10^3/T$ for a series of $[(\text{OEP})\text{Rh}]_2$ solutions in C_6D_6 ($T_2^{-1}(\text{EX}) \propto k_f$). $[(\text{OEP})\text{Rh}]_2$: $(4.8 \pm 0.1) \times 10^{-3}$ M, \circ ; $(3.0 \pm 0.5) \times 10^{-3}$ M, \square ; $(4.1 \pm 0.1) \times 10^{-3}$ M, ∇ ; $(1.7 \pm 0.1) \times 10^{-3}$ M, \blacktriangle ; $(3.5 \pm 0.1) \times 10^{-4}$ M, \blacksquare ; $(1.7 \pm 0.1) \times 10^{-3}$ M, \bullet .

ports assigning the line-broadening process to the formation of the metallo radical, $(\text{OEP})\text{Rh}^{\cdot}$. The contribution of the exchange reaction to the line width at half-height ($\Delta\nu_{1/2}$), $T_2^{-1} = \pi\Delta\nu_{1/2}$, is given by the general expression $T_2^{-1}(\text{EX}) = \pi A^{-1}[(A\tau_p/2)^2][1 + (A\tau_p/2)^2]^{-1}$, where τ_d and τ_p are the lifetimes of the nuclear spin states in the diamagnetic and paramagnetic species and A is the electron-nucleus coupling constant for the nucleus being observed.^{7–9} When $(A\tau_p/2)^2 \gg 1$, then $T_2^{-1}(\text{EX}) = \tau_d^{-1}$ and the forward rate constant (k_f) is given by $\tau_d^{-1} = k_f$ for the exchange reaction. Temperature dependence of k_f yields the activation enthalpy (ΔH_f^*) for the forward process. This type of experiment has been previously used in evaluating k_f and ΔH_f^* for the dissociation of an organic dimer into radicals.¹⁰ In the specific case of $(\text{OEP})\text{Rh}^{\cdot}$ where the values of A and τ_p are unknown, only the activation energy can be confidently determined.

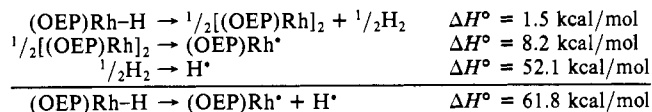
The methyne porphyrin protons of $[(\text{OEP})\text{Rh}]_2$ in C_6D_6 occur as well-separated singlet ($\delta = 9.28$) and provide a convenient peak for observation. Line widths of the methyne protons were measured on six independent samples in the temperature range 290–380 K (Figure 1). Temperature dependence of the exchange rate ($T_2^{-1}(\text{EX}) = k_f(\text{const})$) derived from line broadening of the porphyrin methyne hydrogens results in an activation energy (ΔH_f^*) of 18.5 ± 0.8 kcal/mol for the homolytic dissociation of $[(\text{OEP})\text{Rh}]_2$ in benzene (eq 1) (Figure 2). The ΔH_f^* of 18.5 kcal/mol for $[(\text{OEP})\text{Rh}]_2$ can be compared with the $\Delta H_f^* = 20.6$ kcal/mol obtained by Espenson for the homolytic dissociation of the analogous Rh–Rh-bonded complex $[\text{Rh}(\text{DMG})_2(\text{Ph}_3\text{P})_2]$ by kinetic trapping of the Rh^{\cdot} species.¹¹ The forward activation energy (ΔH_f^*) corresponds to an upper limit for the desired Rh–Rh bond dissociation energy ($\Delta H_1^\circ = \Delta H_f^* - \Delta H_r^*$). Radical recombination reactions related to the reverse of reaction 1 are usually diffusion controlled and have small activation energies ($\Delta H_r^* \sim 2$ kcal/mol).^{12,13} Using an estimate of 2 kcal/mol for ΔH_r^* results in an estimated ΔH_1° of 16.5 kcal/mol in benzene. Line-broadening studies of $[(\text{OEP})\text{Rh}]_2$ in cyclohexane yield similar results ($\Delta H_f^* = 19.5 \pm 1$ kcal/mol; $\Delta H_1^\circ \sim 17.5$ kcal/mol).

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Availability of a value for the (Rh-Rh)BDE permits extraction of the (Rh-H)BDE from thermodynamic data for reaction 2 ($\Delta H_2^\circ = -3.0 \pm 1$ kcal/mol; $\Delta S_2^\circ = 0.8 \pm 2$ eu).¹⁴ Evaluation



thermodynamic cycle I (Rh-H) BDE

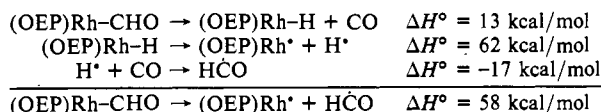


of thermodynamic cycle I results in a (Rh-H)BDE of approximately 62 kcal/mol. Calorimetric methods were used by Drago to obtain an average Rh-H bond energy of 57.6 kcal/mol in $\text{Rh}(\text{Cl})(\text{P}(4\text{-tolyl})_3)_3(\text{H})_2$,¹⁵ and the $\nu_{\text{Rh-H}}$ stretching frequencies for (OEP)Rh-H (2220 cm^{-1}) and $\text{Rh}(\text{Cl})(\text{P}(4\text{-tolyl})_3)_3(\text{H})_2$ (2034 cm^{-1}) qualitatively correlate with the Rh-H bond energies. A (Rh-H)BDE of 62 kcal/mol for (OEP)Rh-H is clearly in the range expected for a second-row transition-metal hydride,¹⁶ and judging from the unusually high Rh-H stretching frequency for (OEP)Rh-H, the (Rh-H)BDE of 62 kcal/mol is probably at the high end of the range of Rh-H bond energies.

The (Rh-H)BDE of 62 kcal/mol can be subsequently used in deriving a (Rh-C)BDE in (OEP)Rh-CHO from thermodynamic data for reaction 3 ($\Delta H_3^\circ = -13 \pm 1$ kcal/mol; $\Delta S_3^\circ = -29 \pm 3$ eu)⁶ and the formyl radical.^{17,18} The relatively large (Rh-



thermodynamic cycle II (Rh-C)BDE



C)BDE (~ 58 kcal/mol) and small difference in Rh-H and Rh-C bond energies (~ 4 kcal/mol) provides an operational explanation for the range of unusual organometallic chemistry associated with rhodium porphyrins, which is not observed for cobalt porphyrins.¹⁻⁶ A (Rh-C)BDE of 58 kcal/mol in (OEP)Rh-CHO is approximately twice the Co-C bond energy (Co-C ~ 28 kcal/mol) recently reported for a (OEP)Co-alkyl complex.¹³ Rh-C bonds are expected to be stronger than Co-C bonds due to improved overlap of the 4d orbitals, but differences of the magnitude observed for the (OEP)M complexes must be predominantly of steric origin.¹⁹

NMR line broadening is potentially a useful method for estimating M-M bond energies in the range 8-25 kcal/mol when NMR experiments in the temperature range 170-470 K are feasible. $[(\text{OEP})\text{Rh}]_2$ is an ideal case for use of this method because bond homolysis is the only pathway for a chemical exchange process to contribute to the line width. The primary criteria for using this method to estimate M-M bond energies are that bond homolysis occur at a rate of 2-100 events/s in an accessible temperature range and that a magnetic nucleus in the compound be coupled significantly with the unpaired electron on the paramagnetic monomer. Complexes that have M-M single bonds where the ligands have large steric requirements are potentially good candidates for application of this method.

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Registry No. $[(\text{OEP})\text{Rh}]_2$, 63439-10-1.

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Cyclic Voltammetric Study of Organoactinide Compounds of Uranium(IV) and Neptunium(IV). Ligand Effects on the M(IV)/M(III) Couple

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The physical and chemical properties of organoactinides vary greatly with the nature of the ligands about the actinide center.² The effects of ligand environment on the bonding and chemical behavior of organoactinide systems have been studied by a number of techniques, including magnetic susceptibility,³ Mössbauer spectroscopy,⁴ photoelectron spectroscopy,⁵ and solution calorimetry.⁶

Electrochemical methods have provided a considerable amount of information on the nature of the ligand-metal interaction in transition-metal complexes.^{7,8} This tool has been utilized less frequently in organoactinide systems.⁹⁻¹¹ Herein, we report the results of a cyclic voltammetric study of a series of uranium(IV) and neptunium(IV) organometallics. Our objective is to gain insight into the sensitivity of the M(IV)/M(III) couple to ligand environment.

Experimental Section

Synthetic Methods. The organometallics were prepared and handled in Schlenk glassware on a dual-manifold Schlenk line or interfaced to a high-vacuum system. Solid transfers were accomplished under nitrogen in a Vacuum Atmospheres Corp. inert-atmosphere box. Solvents were purified and dried as previously described.^{6b}

The compounds Cp_4U^{12} ($\text{Cp} = \eta^5\text{-C}_5\text{H}_5$), Cp_3UCl ,¹³ Cp^*UCl_2 ($\text{Cp}^* = \eta^5\text{-C}_5\text{Me}_5$), Cp_4Np ,¹⁵ Cp_3NpCl ,¹⁶ and Cp^*NpCl_2 ¹¹ were prepared by literature procedures.

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