

Photochemical Generation of $[\text{Rh}(\text{dmgH})_2\text{PPh}_3]_2$ from $[\text{CpW}(\text{CO})_3]_2$ and $\text{XRh}(\text{dmgH})_2\text{PPh}_3$ ($\text{X} = \text{Cl}, \text{Br}$) with Intermediate 17-Electron Tungsten and Rhodium Radicals

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Received September 3, 1992

Laser flash photolysis of $[\text{CpW}(\text{CO})_3]_2$ in a CH_3CN solution containing $\text{XRh}(\text{dmgH})_2\text{PPh}_3$ (X is Cl, Br) leads to $\text{CpW}(\text{CO})_3\text{X}$ and $[\text{Rh}(\text{dmgH})_2\text{PPh}_3]_2$. The reaction proceeds by way of two different 17-electron organometallic radical intermediates. The W-W bond of $[\text{CpW}(\text{CO})_3]_2$ cleaves in the laser flash to give $\text{CpW}(\text{CO})_3$ radicals. The tungsten radicals abstract a halogen atom from the halorhodium complex, $k_{\text{Cl}} = (9.2 \pm 0.4) \times 10^7$ and $k_{\text{Br}} = (1.57 \pm 0.05) \times 10^8 \text{ L mol}^{-1} \text{ s}^{-1}$, forming the 17-electron radical $\text{Rh}(\text{dmgH})_2\text{PPh}_3$ that is observed spectroscopically at 580 nm. The $\text{Rh}(\text{dmgH})_2\text{PPh}_3$ radicals subsequently dimerize, $k = 2.1 \times 10^8 \text{ L mol}^{-1} \text{ s}^{-1}$, to yield $[\text{Rh}(\text{dmgH})_2\text{PPh}_3]_2$, observed spectroscopically at 452 nm.

Introduction

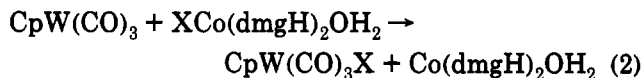
Organometallic radicals are now recognized as intermediates in substitution and redox mechanisms.¹ Mechanistic proposals have been based mostly on the results of long time scale photochemical experiments. They have revealed that 17-electron radicals are highly labile to ligand substitution.¹⁻⁷ Many are good electron donors, especially when coordinated by Lewis bases.⁸⁻¹³ Because most such radicals couple at or near the diffusion-controlled rate,¹⁴⁻¹⁹ information about their reactivity has usually been derived indirectly.

In this work we are concerned principally with the 17-electron radical $\text{CpW}(\text{CO})_3$. Like similar radicals, this molecule abstracts halogen atoms from organic halides. The identification of the independently known halotungsten product indicates that this occurs by atom abstraction, eq 1.



The studies of this reaction, either by photostationary or time-resolved methods, do not directly verify that R^\cdot

is formed, nor do they show how it reacts. The same limitation applies to studies of halocobalt(III) complexes, such as the bis(dimethylglyoximate) complex shown in eq 2.



In an effort to describe halogen abstraction processes more fully, we have now used halorhodium(III) complexes as substrates. Its advantages for this purpose are that the mononuclear rhodium(II) complex $\text{Rh}(\text{dmgH})_2\text{PPh}_3$ (a) is unstable as such,^{20,21} (b) has a distinctive absorption spectrum, and (c) forms a dimer that has a known rate of buildup and a characteristic visible spectrum of sufficient intensity for direct monitoring.^{20,21} This system is the subject addressed here. In this work, we present a time-resolved study of a photochemical reaction in which $\text{Rh}(\text{dmgH})_2\text{PPh}_3$ is a short-lived intermediate whose buildup and loss can be followed directly. This leads to a dimeric rhodium(II) product that can also be monitored directly.

Experimental Section

Chloro(triphenylphosphine)bis(dimethylglyoximate)-rhodium(III), $\text{ClRh}(\text{dmgH})_2\text{PPh}_3$, (= "chlororhodoxime") and its bromo analog were prepared by the literature method.²² The rhodium dimer $[\text{Rh}(\text{dmgH})_2\text{PPh}_3]_2$ is known independently from the reduction of the chloro complex by sodium borohydride followed by the addition of hydrochloric acid.^{22,23} The stable tungsten dimer, $[\text{CpW}(\text{CO})_3]_2$, was prepared by the literature method.²⁴

Solutions of $[\text{CpW}(\text{CO})_3]_2$ in acetonitrile were prepared in the dark, purged with argon, and kept under anaerobic conditions. The concentration of the stock solution was determined spectrophotometrically at one of its maxima, 356 nm ($\epsilon = 2.1 \times 10^4 \text{ L mol}^{-1} \text{ cm}^{-1}$) or 484 nm ($2.5 \times 10^3 \text{ L mol}^{-1} \text{ cm}^{-1}$). The desired quantity of the rhodium halide complex was then added to this solution in a fluorescence cuvette with 1-cm optical path lengths.

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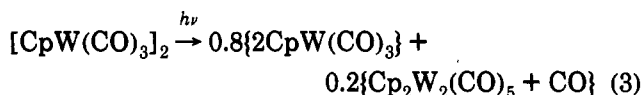
The reaction was initiated by a 0.6- μ s flash of a Phasar flashlamp-pumped dye laser containing LD 490, LD 473, or Coumarin 460 dye. This flash photodissociates the tungsten dimer. After this point all of the reactions are thermal ones. There is no evidence for photocatalysis, which we found in a few of the other reaction systems where $CpW(CO)_3$ was involved.²⁵ The fate of the transients was monitored by a probe beam that impinges on the reaction cuvette at right angles. The flash photolysis apparatus was based on a design in the literature.²⁶ Its details have been described earlier.^{27,28}

Different monitoring wavelengths were used in the various experiments, so as to follow separate species individually. The absorbance changes depend on the species being monitored and on the extent to which competing reactions occur under the given conditions.

Results and Discussion

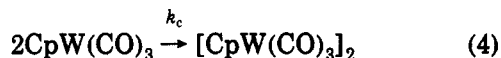
Tungsten radicals abstract halogen atoms from organic and inorganic substrates.^{20,25,29-32} When $CpW(CO)_3$ reacts with a halocobalt(III) complex, the Co(II) product is stable. Also most cobalt(II) complexes are not easily detected during the flash photolysis experiment, leaving unanswered the question as to the stage at which they are formed. However, many Rh(II) complexes, such as $Rh(dmgh)_2PPh_3$, are not stable as monomers. In the absence of other substrates, $Rh(dmgh)_2PPh_3$ dimerizes rapidly.^{20,21}

Photolysis of $[CpW(CO)_3]_2$ at 490 nm in CH_3CN leads to concurrent homolytic cleavage of the W-W bond and CO loss,^{29,30} eq 3. The approximate 4:1 ratio of the



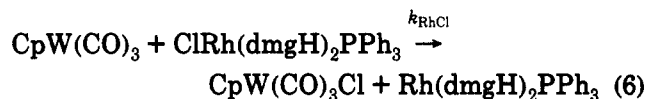
photoproducts is characteristic of irradiation at longer wavelengths (460-490 nm), whereas CO loss predominates when the major band of the dimer at 356 nm is irradiated. The laser excitation in the experiments reported here was therefore confined to the longer wavelengths. In the absence of a reducible substrate (including O_2 , that was rigorously excluded), both tungsten photoproducts revert to the starting dimer. The $CpW(CO)_3$ radicals dimerize rapidly, whereas $Cp_2W_2(CO)_5$ recombines^{16,19} with CO more slowly but, judging from the absorbances, completely. Large absorbance changes at 356 nm are associated with cleavage and reconstitution of the W-W bond, but not with CO capture. Each reaction is kinetically well separated in time, since the dimerization occurs at nearly the diffusion-controlled limit. Thus all of the chemistry of the tungsten radicals, dimerization as well as the other reactions examined in this work, are largely complete before the $Cp_2W_2(CO)_5$ -CO recombination sets in.

The dimerization process, eq 4, has been characterized previously.²⁵ The rate constant, defined as in eq 5, is $k_c = 6.2 \times 10^9 \text{ L mol}^{-1} \text{ s}^{-1}$ at 23 °C in acetonitrile.



$$-\frac{1}{2} \frac{d[CpW(CO)_3]}{dt} = \frac{d[CpW(CO)_3]_2}{dt} = k_c [CpW(CO)_3]^2 \quad (5)$$

Reaction with Halorhodium(III) Complexes. In the presence of $ClRh(dmgh)_2PPh_3$, the tungsten radical is diverted from dimerization by its reaction with the rhodium complex. As we shall show, this is a halogen atom abstraction reaction, that occurs as in eq 6.



When both reactions 4 and 6 occur simultaneously, the reaction will follow the rate law given in eq 7. The existence of concurrent reactions leads to a reasonably complicated

$$-\frac{d[CpW(CO)_3]}{dt} = 2k_c [CpW(CO)_3]^2 + k_\psi [CpW(CO)_3] \quad (7)$$

kinetic analysis that can be simplified somewhat by working with $[halorhodium] \gg [CpW(CO)_3]$. With that, one can substitute $k_\psi = k_{RX}[XRh(dmgh)_2PPh_3]_{av}$, from which the solution in eq 8 follows.

$$[CpW(CO)_3]_t = \frac{k_\psi [CpW(CO)_3]_0 \exp(-k_\psi t)}{k_\psi + 2k_c [CpW(CO)_3]_0 \{1 - \exp(-k_\psi t)\}} \quad (8)$$

It is clear from the chemical and algebraic equations that, with halorhodium complex added, less of the tungsten dimer will be recovered. Also, the reactions will be completed sooner since the radical monomer then disappears by an additional pathway. We can thus say that the tungsten monomer-dimer reaction serves as a *kinetic probe* for the reactions of the rhodium complexes. The design of this experiment, through the selection of the halorhodium(III) concentration, relies upon the dimerization reaction being of sufficient importance to give an appreciable absorbance signal change. Yet its concentration range selected must be such that the reaction of the tungsten radical with the rhodium complex also contributes substantially to the rate so that k_ψ can be obtained to good accuracy. Since the data are obtained as absorbance values proportional to the concentration of $[CpW(CO)_3]_2$, this equation is recast to read

$$Abs_t = Abs_0 + \frac{\epsilon_D}{2} \left\{ \frac{\left[\frac{2k_c [W]_0}{k_\psi} + 1 \right] [1 - e^{-k_\psi t}]}{\frac{2k_c [W]_0}{k_\psi} [1 - e^{-k_\psi t}] + 1} - \frac{k_\psi}{2k_c} \ln \left\{ \frac{2k_c [W]_0}{k_\psi} [1 - e^{-k_\psi t}] \right\} + 1 \right\} \quad (9)$$

where Abs refers to the absorbance readings at the 356-

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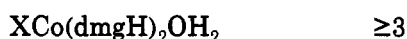
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nm maximum of the tungsten dimer where $\epsilon_D = 2.1 \times 10^4$ L mol⁻¹ cm⁻¹ and $[W]_0$ refers to the initial concentration of the photolytically created radical $CpW(CO)_3$. All of the other species have molar absorptivities at 356 nm that are so small in comparison that they can be neglected. In the treatment of the data k_c was fixed at the independently determined value of 6.2×10^9 L mol⁻¹ cm⁻¹. In principle, then, k_ψ is the only unknown in eq 9 and its value can be found by least-squares fitting. Actually, the small irregularities in the intensity of the laser from one flash to the next lead to some uncertainty in $[W]_0$. For that reason, in fitting the data to eq 9, we treated both k_ψ and $[W]_0$ as unknowns. The value of $[W]_0$ resulting from each calculation was always about what was expected, given the starting concentration on the dimer and the laser parameters. A second test of the validity of the data analysis is that plots of k_ψ were linear in $[XRh(dmgH)_2PPh_3]$.

Concentrations of the tungsten dimer of 20–30 μ M were used for the evaluation of k_{RX} . This usually produces 10–20 μ M of the tungsten radical, since photolysis is rather efficient. This treatment affords these rate constants: $k_{RhCl} = (9.2 \pm 0.4) \times 10^7$ and $k_{RhBr} = (1.57 \pm 0.05) \times 10^8$ L mol⁻¹ s⁻¹ at 23 °C in acetonitrile. Earlier measurements²⁵ of the rate constants for organic halides and for halocobalt(III) complexes have always found the order of reactivity bromide > chloride. In many of those cases, however, particularly the less reactive ones, the reactivity ratio was large, often 10^3 – 10^4 . Although the same order is found for rhodium, it is much less selective. We note, however, that both rhodium halides are highly reactive. That in itself limits the selectivity, since the upper limit on the rate constant is set by the diffusion-controlled limit, ca. 2×10^{10} L mol⁻¹ s⁻¹ in acetonitrile. But there is another consideration for the rhodium complexes as well. This metal is a soft acid center, and as such binds bromide ions more tightly than chloride. Since the transition state features rhodium–halide bond breaking as well as tungsten–halide bond making, there is a degree of compensation in this particular series that is evidently absent for organic halides and halocobalt(III) complexes. More quantitatively, consider these reactivity ratios:



Reaction 6 occurs by chlorine atom abstraction. Transfer of a chlorine from rhodium to tungsten was verified by the appearance of the characteristic ν_{CO} bands of $CpW(CO)_3Cl^{18}$ in the IR spectrum of a photolyzed reaction mixture.

Rhodium Radical. The $Rh(dmgH)_2PPh_3$ produced in reaction 6 is also a 17-electron radical, which is independently known.^{20,21,31} It absorbs in the visible region at 580 nm ($\epsilon = 2 \times 10^3$ L mol⁻¹ cm⁻¹).²⁰ The involvement of this species was explored principally in experiments at higher concentrations of halorhodium complex, so that reaction 6 was complete fairly promptly. This allowed us to monitor the formation and subsequent loss of $Rh(dmgH)_2PPh_3$ at 580 nm, as shown in Figure 1b. The other possible organometallic species, including transient or stable compounds of tungsten, do not absorb significantly in this

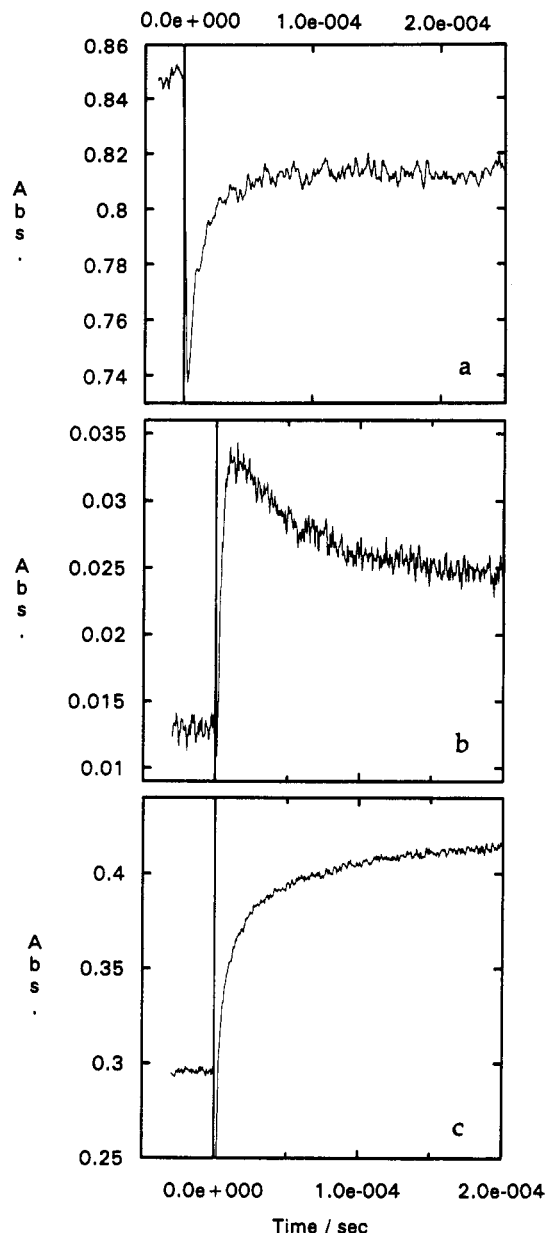
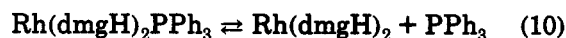


Figure 1. Kinetic traces obtained by laser flash photolysis of $\sim 20 \mu$ M $[CpW(CO)_3]_2$ in the presence of $ClRh(dmgH)_2PPh_3$ and 1 mM PPh_3 in CH_3CN . (a) Partial recovery of $[CpW(CO)_3]_2$ at λ 356 nm, by combination of $CpW(CO)_3$ radicals formed in the flash. $[ClRh(dmgH)_2PPh_3] = 0.11$ mM. (b) Formation and subsequent loss of $Rh(dmgH)_2PPh_3$ at λ 580 nm. $[ClRh(dmgH)_2PPh_3] = 0.75$ mM. (c) Formation of $[Rh(dmgH)_2PPh_3]_2$ at λ 452 nm. $[ClRh(dmgH)_2PPh_3] = 0.75$ mM. In the second and subsequent flashes, the absorbance is partially bleached in the flash due to photolysis of $[Rh(dmgH)_2PPh_3]_2$, as discussed in the text.

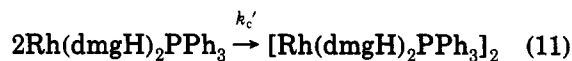
long-wavelength region. The $Rh(dmgH)_2PPh_3$ radical tends to undergo loss of the axial ligand,²⁰ eq 10.



Dissociation was suppressed by the addition of 1 mM PPh_3 to the reaction mixture. This amount of PPh_3 does not affect the rate of reaction 4; i.e., this concentration of PPh_3 is too low to promote disproportionation of the $CpW(CO)_3$ radicals. The data obtained under these circumstances are of inherently lower quality, because the molar absorptivity of the rhodium radical is only about one-tenth that of the tungsten dimer. Indeed, to observe

the rhodium radical, higher concentrations of chlororhodoxime were needed. The data also showed that $\text{Rh}(\text{dmgH})_2\text{PPh}_3$ formed as expected from the earlier determined rate constant.

Rhodium Dimer. The $\text{Rh}(\text{dmgH})_2\text{PPh}_3$ radicals combine to give the strongly absorbing dimer $[\text{Rh}(\text{dmgH})_2\text{PPh}_3]_2$, λ_{max} 452 nm, ϵ 5×10^4 L mol⁻¹ cm⁻¹,²¹ as shown in eq 11 and Figure 1c.



This can be seen at 580 nm, but more precisely at 452 nm. The experimental data used to evaluate k_c' were obtained in experiments with higher rhodium concentrations than those used when k_{RHX} was determined, to avoid overlap in time between the two events. The absorbance-time data were fitted to the rate law for kinetics second order in a single concentration, shown in terms of absorbances in eq 12.

$$\text{Abs}_t = \text{Abs}_\infty + \frac{\text{Abs}_0 - \text{Abs}_\infty}{1 + [\text{Rh}(\text{dmgH})_2\text{PPh}_3]_0 k_c' t} \quad (12)$$

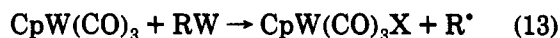
This analysis gave $k_c' = 2.1 \times 10^8$ L mol⁻¹ s⁻¹ at 23 °C in acetonitrile. This value is in excellent agreement with the independently measured rate constant, which is 2.3×10^8 L mol⁻¹ s⁻¹ in 95% ethanol.²⁰ As is typical for coupling reactions between uncharged radicals, the reaction is not strongly dependent on the solvent. The agreement among all of these quantities adds confidence that the rhodium species produced from the tungsten dimer are indeed the same as those known from other sources.

Other Kinetic Consequences. The proposed sequence of reactions (4, 6, and 11) is valid only during the first laser flash that impinges on a reaction mixture. The photochemistry is different in subsequent flashes, because the accumulating $[\text{Rh}(\text{dmgH})_2\text{PPh}_3]_2$ is photosensitive in the same spectral region as $[\text{CpW}(\text{CO})_3]_2$. In the second and subsequent laser flashes, the absorbance of the rhodium dimer at 452 nm is partially bleached, then increases with time to a level higher than the preflash absorbance. This phenomenon is caused by the simultaneous photolytic cleavage of both dimers, $[\text{CpW}(\text{CO})_3]_2$ and $[\text{Rh}(\text{dmgH})_2\text{PPh}_3]_2$. That is, the photodissociated rhodium dimer recovers; at the same time its concentration is enhanced by the dimer resulting from the sequence of reactions 6 and 11. Homocoupling of tungsten and rhodium radicals is then possible. There is every reason to expect that cross-coupling would also occur, giving $\text{Cp}(\text{CO})_3\text{W}-\text{Rh}(\text{dmgH})_2\text{PPh}_3$, although evidence for it was neither sought nor obtained. After several flashes, only the photochemistry of $[\text{Rh}(\text{dmgH})_2\text{PPh}_3]_2$ remains, and no absorbance change at 356 nm occurs in a subsequent

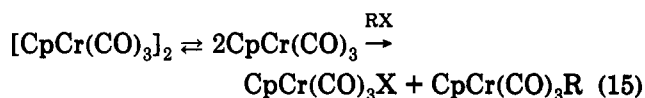
flash. At this stage, $[\text{Rh}(\text{dmgH})_2\text{PPh}_3]_2$ absorbs all of the light whereas $[\text{CpW}(\text{CO})_3]_2$, even though still present in solution, does not absorb enough of the incident light to participate in the photochemistry.

Another interesting feature of this reaction is that it does not yield the mixed dimer $\text{CpW}(\text{CO})_3-\text{Rh}(\text{dmgH})_2\text{PPh}_3$. This may seem surprising since both radicals, $\text{CpW}(\text{CO})_3$ and $\text{Rh}(\text{dmgH})_2\text{PPh}_3$, coexist in solution during reaction 6 and the rate constants for the homocoupling and cross-coupling reactions are expected to be comparable. The combination of the large values of k_6 and $[\text{XRh}(\text{dmgH})_2\text{PPh}_3]$ makes the rate of reaction 6 much larger than the sum of all the rates of radical coupling reactions; i.e., $v_6 \gg v_{11}$. Consequently, all the $\text{CpW}(\text{CO})_3$ is exhausted in reaction 6 well before the radical coupling can take place and the steady-state approximation for $\text{Rh}(\text{dmgH})_2\text{PPh}_3$ does not hold. On the slower time scales associated with radical coupling the only radical left is $\text{Rh}(\text{dmgH})_2\text{PPh}_3$, and the only dimetallic product formed is $[\text{Rh}(\text{dmgH})_2\text{PPh}_3]_2$.

The same delay between the loss of $\text{CpW}(\text{CO})_3$ and the product formation in the reaction of $\text{CpW}(\text{CO})_3$ with alkyl halides²⁵ is responsible for the failure of R^* to couple with $\text{CpW}(\text{CO})_3$ to yield $\text{CpW}(\text{CO})_3\text{R}$. The only radical-derived products are those formed in the self-reactions of R^* , i.e.



In the thermal reactions of $[\text{CpCr}(\text{CO})_3]_2$ with alkyl halides, on the other hand, the generation of the radical intermediates is apparently much slower than the radical trapping reactions, the steady-state approximation holds, and $\text{CpCr}(\text{CO})_3\text{R}$ is produced, eq 15.^{33,34}



Acknowledgment. Professor R. J. Angelici and his research group provided a sample of $[\text{CpW}(\text{CO})_3]_2$. This work was supported by the U. S. Department of Energy, Office of Basic Energy Sciences, Chemical Sciences Division, under Contract W-7405-Eng-82.

OM920536W

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