mercurial $Hg\{Co[P(OCH_3)_3]_4\}_2$. Ostensibly,[23] because no substantial quantity of $\{Co[P(OCH_3)_3]_4^+\}$ was present in this reduction system, no detectable quantity of the dimer $Co_2[P(OCH_3)_3]_8$ was formed.

Aside from the complexity of the trimethyl phosphite based reduction system, the really remarkable features of this chemistry are the isolation of the monomeric and dimeric forms of $Co[P(OCH_3)_3]_4$ and the demonstration of the noninterconvertibility of these two complexes and their relatively low reactivity, as discussed below.

The radical $Co[P(OCH_3)_3]_4$ displayed no detectable degree of dimerization in solution or in the solid state at 25–100 °C nor did the radical abstract hydrogen atoms at 20–70 °C from organic solvents such as tetrahydrofuran or toluene to form the exceedingly stable $HCo[P(OCH_3)_3]_4$ complex. Complementarily, the dimer, $Co_2[P(OCH_3)_3]_8$, gave no evidence of dissociation to the monomer in solution at temperatures below ~70 °C or of hydrogen atom abstraction from solvent at 20–70 °C. Both monomer and dimer in toluene solution very slowly formed $HCo[P(OCH_3)_3]_4$ at 90 °C. Neither the monomer nor the dimer detectably reacted with hydrogen at 20–50 °C (reaction time of 1 week) but they did slowly at 90 °C to form $HCo[P(OCH_3)_3]_4$. Under photolysis at 0 °C, however, both reacted with hydrogen to form $HCo[P(OCH_3)_3]_4$. Photolysis converted both the monomer and the dimer in toluene solution at 0 °C to $Co[P(O)(OCH_3)_2][P(OCH_3)_3]_4$. Clearly, this chemistry establishes that the dimer is not formed from the monomer, that the dimer does not thermally dissociate readily to form the monomer, and that the radical monomer does not readily abstract hydrogen atoms from organic solvents. The radical monomer did react slowly with mercury and with sodium amalgam to form $Hg\{Co[P(OCH_3)_3]_4\}_2$ and $Na^+\{Co[P(OCH_3)_3]_4^-\}$, respectively.[24]

Much of the chemistry established for the zerovalent phosphite complexes is in sharp contrast to the zerovalent cobalt carbonyl chemistry where the dimer is the only isolable form under normal conditions. Presumably, the stability of $Co[P(OCH_3)_3]_4$ resides in a relative resistance to phosphite dissociation and a large steric barrier to direct dimerization to $Co_2[P(OCH_3)_3]_8$. Carbonyl analogues contrast sharply in their dimerization proclivities. For example, the bimolecular rate constants[25] for recombination of $M(CO)_5$ radicals at 22 °C are 9.5×10^8 and 3.7×10^9 M^{-1} s^{-1} for $Mn_2(CO)_{10}$ and $Re_2(CO)_{10}$, respectively. We are now in the process of examining the thermodynamic and kinetic (reaction mechanistic) character of basic reactions such as ligand substitution in the monomeric $Co[P(OCH_3)_3]_4$ complex and in the dimer.

Acknowledgment. We thank John McCracken, Department of Chemistry, University of California, Berkeley, for obtaining the ESR spectrum of $Co[P(OCH_3)_3]_4$ and Professor Paul Ellis, NSF Facility in NMR Spectroscopy, University of South Carolina, for providing a high-field (160 MHz) $^{31}P\{^1H\}$ NMR spectrum of $\{Co[P(OCH_3)_3]_5^+\}\{Co[P(OCH_3)_3]_4Na[(CH_3O)_3P]_4Co^-\}$. Microanalyses were performed by Vazken H. Tashinian at the UCB Department of Chemistry Microanalytical Laboratory. The earliest electron spin resonance studies were performed by Dr. Kirill Zamaraev. We gratefully acknowledge the National Science Foundation for support of this research, for a Postdoctoral Fellow (J.R.B.), and for a departmental equipment grant to the Department of Chemistry for the purchase of a 180-MHz NMR spectrometer. E.L.M. is indebted to the Miller Institute for a grant in the form of a Miller Professorship.

Supplementary Material Available: Synthesis details for the amalgam reduction reaction (1 page). Ordering information is given on any current masthead page.

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[23] We infer that dimers are formed only by electron transfer between $\{Co[P(OR)_3]_4^+\}$ and $\{Co[P(OR)_3]_4^-\}$. The only established synthesis of $Co_2[P(OCH_3)_3]_8$ is from $K^+\{Co[P(OCH_3)_3]_4^-\}$ and $(CH_3)_3GeCl$.[8] We assume that in this reaction, electron transfer yields the cationic $\{Co[P(OCH_3)_3]_4^+\}$ species, which then reacts with $\{Co[P(OCH_3)_3]_4^-\}$ to form the dimer. Consistent with this proposal, the germanium-containing product of the reaction was $Ge_2(CH_3)_6$.

[24] Explicably, the monomer was oxidized by silver ion to give $Co[P(OCH_3)_3]_5^+$. The monomer also reacted rapidly with oxygen. An oxygen complex, deep purple, formed at −78 °C. Thermal reactivity of the complex was high, as evidenced by the irreversible color changes to dark green at −10 °C and to blue at 15 °C. Addition of 2 more equivalents of oxygen at 20 °C discharged the blue color within minutes, and brown solids separated.

[25] Wegman, R. W.; Olsen, R. J.; Gard, D. R.; Faulkner, L. R.; Brown, T. L. *J. Am. Chem. Soc.* **1981**, *103*, 6089.

Stereochemical Consequence of the Interaction of Alkylaluminums with Titanacyclobutanes and Its Relationship to the Olefin Metathesis Reaction

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Lewis acids are necessary cocatalysts in many olefin metathesis[1,2] and Ziegler–Natta polymerization[3,4] systems. Many of these catalyst systems are highly stereospecific but become less so on increasing the concentration of the Lewis acid cocatalyst.[2] We describe here the stereochemical result of the interaction of a Lewis acid, dimethylaluminum chloride, with a bis(η^5-cyclopentadienyl)titanacyclobutane, a well-defined olefin metathesis catalyst.[5]

Compound I[5] (Chart I) is smoothly converted to II[6] and 3,3-dimethyl-1-butene (Scheme I) with dimethylaluminum chloride. The reactions were followed by ^1H or ^2H NMR spectroscopy.[18] The reaction proceeds with second-order kinetics, first order in I and Me_2AlCl ($k_2 = (5.8 \pm 1.7) \times 10^{-3}$ M^{-1} s^{-1} at 294 K). A plot of ln (k/T) vs. $1/T$ yields $\Delta G^{\ddagger}{}_{294} = 20 \pm 1$ kcal/mol^{-1}, and $\Delta S^{\ddagger} = -38 \pm 1$ eu. A small secondary isotope effect was observed ($k_H/k_D = 1.18 \pm 0.2$) in the reaction of I-d_2[7] and Me_2AlCl, producing 3,3-dimethyl-1-butene-*1,1*-d_2 and II-d_2 in the ratio 1.40 ± 0.03:1. Scheme I shows a reasonable mechanism for this reaction and, therefore, the cocatalyzed route for metathesis.[8]

As part of a study of the stereochemistry of the metathesis reaction catalyzed by *trans*-I-d_1, a surprising result was observed. During reaction of *trans*-I-d_1 with Me_2AlCl, ^2H NMR spectroscopy at 77 MHz indicated that the stereochemistry of the α-carbon of the metallacycle was rapidly (only a few seconds are required) and completely scrambled, giving equal amounts of *cis*- and *trans*-I-d_1 *before* cleavage to II and II-d_1. We have since found that *trans*-I-d_1 is rapidly and catalytically isomerized by less than 0.1 equiv of Me_2AlCl at room temperature.

Initially, we considered that a possible mechanism for the isomerization involved a reversible β-H abstraction by Me_2AlCl to produce a titanocene allyl cation (Scheme II). Rapid rotation of a C–C bond via an η^3–η^1 allyl rearrangement in the cation

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[1] For leading references, see: Katz, T. J.; Lee, S. J.; Nair, M.; Savage, E. B. *J. Am. Chem. Soc.* **1980**, *102*, 7940.

[2] Gunther, P.; Pampus, G.; Haas, F.; Marwede, G.; Nützel, K.; Oberkirch, W.; Schön, N.; Witte, J. *Angew. Makromol. Chem.* **1970**, *14*, 87.

[3] See: Boor, Jr., J. "Ziegler-Natta Catalysts and Polymerizations"; Academic Press: New York, 1979; pp 118–124 and references therein.

[4] Pino, P.; Mulhaupt, R. *Angew. Chem., Int. Ed. Engl.* **1980**, *19*, 857 and references therein.

[5] Lee, J. B.; Howard, T. R.; Grubbs, R. H. *J. Am. Chem. Soc.* **1980**, *102*, 4515.

[6] Tebbe, F. N.; Parshall, g. W.; Reddy, G. S. *J. Am. Chem. Soc.* **1978**, *100*, 3611.

[7] Compounds I, I-d_2, and *trans*-I-d_1 were prepared as previously described (ref 5) from II, the corresponding olefin, and 4-dimethylaminopyridine (DMAP). I-d_5 was prepared from II-d_2 which had been prepared by reaction of Cp_2TiCl_2 with 2 equiv of $AlMe_3$-d_9. I-2,3-d_2 was prepared by exchange of $Cp_2\overline{TiCH_2CMe_2C}H_2$ with *cis*-3,3-dimethyl-1-butene-*1,2*-d_2.

[8] Tebbe has shown that II will catalyze the metathesis of terminal olefins: Tebbe, F. N.; Parshall, G. W.; Ovenall, D. W. *J. Am. Chem. Soc.* **1979**, *101*, 5074. Klabunde, U.; Tebbe, F. N.; Parshall, G. W.; Harlow, R. L. *J. Mol. Catal.* **1980**, *8*, 37. This metathesis is slow, even at 51 °C. From this work, we infer the existence of the olefin II complex depicted in Scheme I.

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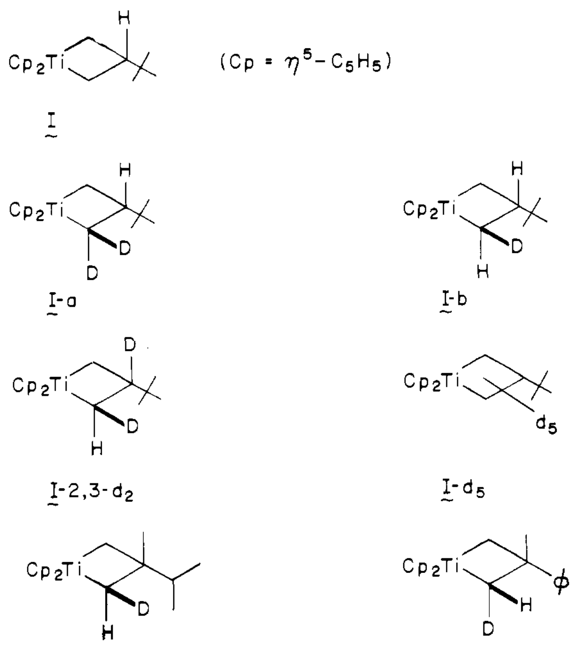
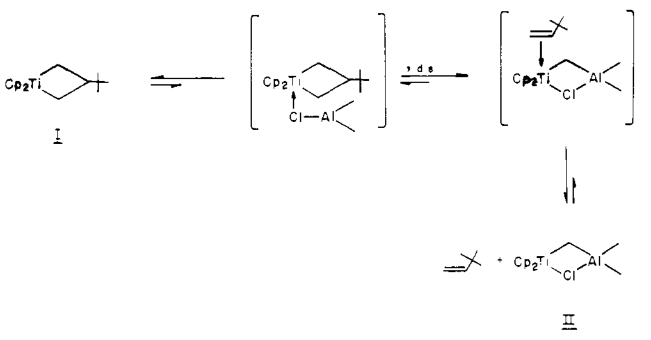
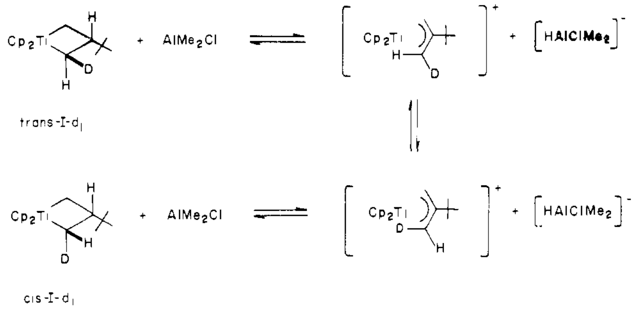
Chart I



Scheme I



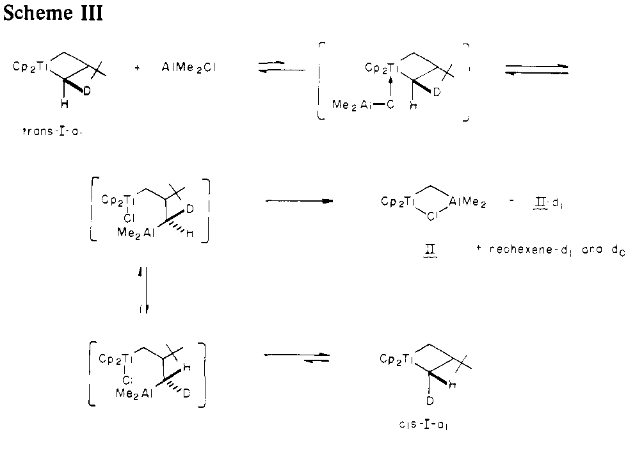
Scheme II



followed by closure to titanacyclobutane[9] would result in isomerization.

This possibility was further investigated by synthesizing III and IV,[10] which lack β-hydrogens. Reaction of III-d_1 or IV-d_1 with Me_2AlCl below 0 °C in toluene results in rapid formation of II and II-d_1. The deuterated olefin (2,3-dimethyl-1-butene or α-methylstyrene) produced is 1:1 *E*/*Z*. This experiment is complicated due to the isomerization of III-d_1 or IV-d_1 at 0 °C in the absence of Me_2AlCl due to nonstereospecific olefin interchange. This olefin interchange does not, however, compete with cleavage or isomerization by Me_2AlCl as shown by the following experiment. When IV-d_0 was allowed to react with 1 equiv of Me_2AlCl in the presence of a large excess of (Z)-α-methylstyrene-d_1, no II-d_1 was observed by ^2H NMR spectroscopy. Since Me_2AlCl-induced isomerization is observed for III-d_1 and IV-d_1, it is unlikely that the β-hydrogen is involved in isomerization of I.

Scheme III



To determine if an intermolecular process was involved in the observed scrambling, we performed the following crossover experiments. When I-*2,3*-d_2 was reacted with excess Me_2AlCl (50 °C, toluene, 1 h), GC/MS analysis of the volatiles indicated that only 3,3-dimethyl-1-butene-d_1 and -d_2 were produced. Similarly, when equimolar amounts of I and I-d_5 were reacted with Me_2AlCl, only 3,3-dimethyl-1-butene-d_0 and -d_3 were produced. In neither instance were intermolecular crossover products detected. It is important to note that the olefins are configurationally stable under these conditions in the absence of titanacyclobutanes and that there is no α to β hydrogen scrambling in either I-d_2 or *trans*-I-d_1.

The above evidence leads us to believe that the isomerization involves a process that only scrambles the α-positions of the titanacyclobutane and that can ultimately lead to C–C bond cleavage to give II. We propose that the first step of the isomerization involves a rapid, reversible transmetalation at the Ti–C bond (Scheme III). Such rapid transmetalations have precedent in both Ti[11] and Zr[12] chemistry. The chloride on aluminum is important, since $AlMe_3$ isomerizes I-b only very slowy, whereas $AlCl_3$ catalyzes the isomerization of I-c.[19] Following transmetalation, the intermediate can undergo carbon–carbon bond cleavage to give II or rapid, reversible inversion at the carbon adjacent to aluminum followed by closure back to titanacyclobutane.

Inversion at the α-carbon atom in Li, Mg, and Zn alkyls[13] via an S_E2 reaction has been demonstrated to be fast on the NMR time scale. Aluminum alkyls also undergo α-carbon inversion on the NMR time scale,[14] but the mechanism is not understood in similar detail. Trace amounts of diethyl ether slow the inversion of aluminum alkyls dramatically.[14] Similarly, reaction of I-*2,3*-d_2 with a solution containing 1.3 equiv of $Me_2AlCl{\cdot}Et_2O$ resulted in only 10% isomerization of I-2,3-d_2 after 2 h.[15] The similarities of the titanacyclobutane isomerization and alkylaluminum inversion suggests that both processes proceed through identical pathways.

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(9) This would be similar to the synthesis of bis(cyclopentadienyl)tungstenacyclobutanes: Adam, G. J. A.; Davies, S. G.; Ford, K. A.; Ephritikkine, M.; Green, M. L. H. *J. Mol. Catal.* **1980**, *8*, 15.

(10) These metallacycles must be synthesized and handled below 0 °C. Otherwise, the dimer $(Cp_2TiCH_2)_2$ is isolated: Ott, K. C.; Grubbs, R. H. *J. Am. Chem. Soc.* **1981**, *103*, 5922. This paper describes the synthesis and characterization of III and IV.

(11) Reaction of $Cp_2Ti(Me)Cl$ with $AlMe_3$-d_9 results in rapid equilibration with $Cp_2Ti(CD_3)Cl$ and $AlMe_3$-d_6: Lee, J. B.; Ott, K. C.; Grubbs, R. H., unpublished results.

(12) Carr, D. B.; Schwartz, J. *J. Am. Chem. Soc.* **1979**, *101*, 3521. Yoshida, T.; Negishi, E. *Ibid.* **1981**, *103*, 4985.

(13) Matteson, D. S. "Organometallic Reaction Mechanisms of the Nontransition Elements"; Academic Press: New York, 1974; pp 66–71. Witanowski, M.; Roberts, J. D. *J. Am. Chem. Soc.* **1966**, *88*, 737.

(14) Fraenkel, G.; Dix, D. T.; Carlson, M. *Tetrahedron Lett.* **1968**, 579. Eisch, J. J. *J. Organomet. Chem. Rev. Annu. Surv.* **1968**, *4B*, 314.

(15) Even trace amounts of diethyl ether remaining from recrystallization of I-*2,3*-d_2 is sufficient to retard the isomerization.

Katz has suggested that the general lack of stereospecificity in Lewis acid cocatalyzed metathesis systems is due to a reversible Lewis acid induced cleavage of transient metallacyclobutanes to a 3-metallapropyl cation, which can then undergo C–C bond rotation, ultimately resulting in scrambling of stereochemistry.[16,17] The similarity of Katz's suggestion to our proposal is apparent. The tendency of a given metathesis catalyst system to show high stereospecificity in the presence of Lewis acids may depend on steric and electronic factors that can determine the susceptibility of a transient metallacyclobutane to transmetalation by the Lewis acid cocatalyst.

Acknowledgment. We gratefully acknowledge the support of the National Science Foundation (Grant CHE 8016528), the Department of Energy, and the use of the Southern California Regional NMR Facility (NSF Grant 7916324A1).

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(16) Katz, T. J.; Hersh, W. H. *Tetrahedron Lett.* **1977**, 585.

(17) Not all aluminum alkyl cocatalyzed metathesis systems show such a dependence of stereospecificity on the Lewis acid concentration. For example, the stereochemistry of ring-opening metathesis polymerization of cyclopentene with $MoCl_5/AlEt_3$ is unaffected by raising the $AlEt_3$ concentration: Dall'Asta, G.; Motroni, G. *Angew. Makromol. Chem.* **1971**, *16/17*, 51.

(18) All reactions described herein were followed by NMR spectroscopy with either a Bruker WM–500 (500.13 MHz ^1H, 76.76 MHz ^2H) or a JEOL FX-90Q (89.60 MHz ^1H) spectrometer.

(19) Reaction of *trans*-I-d_1 with 1 equiv of $AlMe_3$ resulted in less than 5% isomerization in 20 min at room temperature. Reaction of *trans*-I-d_1 with $AlCl_3$ resulted in decomposition of the metallacycle, but in the initial stages of reaction, the metallacycle was observed to be isomerized. Reaction of *trans*-I-d_1 with diphenylacetylene resulted in no isomerization of the metallacycle, and *trans*-3,3-dimethyl-1-butene-.-d_1 is observed as the only deuterated olefin. The half-life for thermal isomerization of *trans*-I-d_1 is 2 h at 50 °C: Lee, J. B.; Ott, K. C.; Grubbs, R. H. to be submitted for publication.

Mechanism of the Photochemical Disproportionation Reactions of $(\eta^5\text{-}C_5H_5)_2M_2(CO)_6$ (M = Cr, Mo, W)

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Very little is known about the mechanism of the photochemically induced disproportionation reactions of metal–metal bonded dimers.[1] For this reason we have been investigating the photochemical disproportionation reactions of the $Cp_2M_2(CO)_6$ (M = Cr, Mo, W; Cp = η^5-C_5H_5) complexes, an example of which is shown in eq 1. The photochemical reactions of metal–metal bonded dimers are generally thought to proceed via homolytic cleavage of the metal–metal bond,[2] but the disproportionation reactions might be an exception to this generalization; it has been suggested that these reactions proceed via photoinduced heterolytic cleavage of the metal–metal bond.[1a] We communicate here the results of experiments that strongly suggest that the disproportionation reactions of the $Cp_2M_2(CO)_6$ complexes result from neither homolytic nor heterolytic cleavage of the metal–metal bond but via a third pathway instead.

$$Cp_2Mo_2(CO)_6 \xrightarrow[NEt_3]{h\nu} CpMo(CO)_3^- + CpMo(CO)_2(NEt_3)_2^+ \quad (1)$$

Scheme I

$$\begin{gathered}
Cp_2M_2(CO)_5L \xrightarrow{h\nu} CpM(CO)_3 + CpM(CO)_2L \\
CpM(CO)_3 + CpM(CO)_2L \xrightarrow{\text{electron transfer}} CpM(CO)_3^- + CpM(CO)_2L^+ \\
CpM(CO)_2L^+ + L \rightarrow CpM(CO)_2L_2^+
\end{gathered}$$

Scheme II

$$\begin{gathered}
Cp_2M_2(CO)_5L \xrightarrow{h\nu} CpM(CO)_3 + CpM(CO)_2L \\
CpM(CO)_2L + Cp_2M_2(CO)_5L \rightarrow CpM(CO)_2L^+ + Cp_2M_2(CO)_5L^- \\
Cp_2M_2(CO)_5L^- \rightarrow CpM(CO)_2L + CpM(CO)_3^- \\
CpM(CO)_2L^+ + L \rightarrow CpM(CO)_2L_2^+
\end{gathered}$$

Irradiation ($\lambda > 290$ nm) of $Cp_2Mo_2(CO)_6$ (1.0×10^{-2} M) in degassed cyclohexane solution containing PPh_3 (5.0×10^{-2} M) proceeds as in eq 2.[3] The substitution product, $Cp_2Mo_2(CO)_5(PPh_3)$, was identified as an intermediate by its infrared absorption bands at 1968, 1900, and 1827 cm^{-1}.[4] The products $CpMo(CO)_2(PPh_3)_2^+$ and $CpMo(CO)_3^-$ were identified by their infrared absorption bands at 1974 and 1895 cm^{-1} and at 1771 cm^{-1}, respectively.[4] Note the small mole ratio of PPh_3 to metal complex (5:1) required to give the disproportionation products. This small ratio shows that the disproportionation is not attributable to a solvent-induced dipolar excited state as originally proposed.[5]

$$Cp_2Mo_2(CO)_6 \xrightarrow[PPh_3]{h\nu} Cp_2Mo_2(CO)_5(PPh_3) \xrightarrow[PPh_3]{h\nu} CpMo(CO)_2(PPh_3)_2^+ + CpMo(CO)_3^- \quad (2)$$

Disproportionation reactions similar to the one in eq 2 occur for the $Cp_2M_2(CO)_6$ (M = Cr, Mo, W) dimers in hexane solution with ligands other than PPh_3. A list of ligands that lead to disproportionation includes NEt_3, CH_3CN, pyridine, aniline, $AsPh_3$, $P(O\text{-}i\text{-}C_3H_7)_3$, and $P(OCH_3)_3$. In each case, irradiation initially produces the monosubstituted dimer, $Cp_2M_2(CO)_5L$ (identified by IR spectroscopy), and then this complex photolyzes to give the ionic products $CpM(CO)_2L_2^+$ and $CpM(CO)_3^-$. In no case were ionic products observed to form without the initial formation of the monosubstituted dimer. In order to check the idea that the monosubstituted dimer is an intermediate in the formation of the ionic products, the complex $Cp_2Mo_2(CO)_5(PPh_3)$ was synthesized[6] and its photochemistry studied. Irradiation (290 nm) of this complex (1.0×10^{-2} M) in hexane solution containing 0.1 M PPh_3 gave $CpMo(CO)_2(PPh_3)_2^+$ and $CpMo(CO)_3^-$. The products were identified by infrared spectroscopy.[4] No infrared bands other than the ones attributable to these products were observed.

The disproportionation reactions show a marked wavelength dependence. Irradiation of $(MeCp)_2Mo_2(CO)_6$[7] in cyclohexane containing excess PPh_3 at 505, 435, 405, and 366 nm[8] does not give the ionic disproportionation products; only the monosubstituted product $(MeCp)_2Mo_2(CO)_5(PPh_3)$ is formed.[9] The quantum yield for substitution of $(MeCp)Mo_2(CO)_6$ by PPh_3 ($[PPh_3] = 0.018$ M, cyclohexane solution) at 405 nm is 0.04 ± 0.01. Disproportionation does not occur until the excitation wavelength is 290 nm. At 290 nm the quantum yield for disproportionation of $(MeCp)_2Mo_2(CO)_5(PPh_3)$ is 0.40 ± 0.04 in

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(1) (a) Allen, D. M.; Cox, A.; Kemp, T. J.; Sultana, Q. *J. Chem. Soc., Dalton Trans.* **1976**, 1189–1193. (b) Hieber, W.; Beck, W.; Zeitler, G. *Angew. Chem.* **1961**, *11*, 364–368.

(2) (a) Wrighton, M. *Chem. Rev.* **1974**, *74*, 401–430. (b) Grinley, D. S.; Wrighton, M. S. *J. Am. Chem. Soc.* **1975**, *97*, 4908–4911. (c) Grinley, D. S.; Bock, C. R.; Wrighton, M. S. *Inorg. Chim. Acta* **1977**, *23*, 85–94. (d) Kidd, C. R.; Brown, T. L. *J. Am. Chem. Soc.* **1978**, *100*, 4095–4103. (e) Hudson, A.; Lappert, M. F.; Nicholson, B. K. *J. Chem. Soc., Dalton Trans.* **1977**, *98*, 551–554. (f) Morse, D. L.; Wrighton, M. S. *J. Am. Chem. Soc.* **1976**, *98*, 3931–3934. (g) Geoffroy, F. L.; Wrighton, M. S. "Organometallic Photochemistry"; Academic Press: New York, 1979.

(3) All irradiations were carried out with a 200-W high-pressure mercury arc. Solutions were degassed with a stream of argon or nitrogen. The 290-nm wavelength band was isolated with a Corion interference filter.

(4) Haines, R. J.; Nyholm, R. S.; Stiddard, M. H. B. *J. Chem. Soc. A* **1968**, 43–46.

(5) The disproportionation reactions were initially observed to occur in neat solvents such as pyridine, Me_2SO, and DMF. It was suggested that the polar solvent molecules induced a dipole in the excited state, $^{\delta-}M–M^{\delta+}$, which subsequently relaxed to give ionic products.

(6) Barnett, K. W.; Treichel, P. M. *Inorg. Chem.* **1967**, *6*, 294–299.

(7) The methylcyclopentadienyl compounds were used in some of these experiments as they were more soluble in alkane solvents than their cyclopentadienyl counterparts.

(8) These wavelengths were isolated with interference filters from Edmund Scientific Co.

(9) The CO stretching region of the infrared spectrum of $(MeCp)_2Mo_2(CO)_5(PPh_3)$ is identical with that of $Cp_2Mo_2(CO)_5(PPh_3)$.

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