under acidic conditions. The synthetic 593A dihydrochloride (mp 280–290 °C dec) was identical in TLC behavior and spectral (^{1}H NMR, ^{13}C NMR, and MS) properties with natural 593A dihydrochloride.[15]

Acknowledgment. We thank the Robert A. Welch Foundation and Rice University for support of this work.

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(9) Prepared from 3,3-diethoxypropanal in four steps [(1) CH_2=$CHCH_2MgBr$, THF, reflux; (2) MsCl, Et_3N, CH_2Cl_2, 0 °C; (3) O_3, MeOH-CH_2Cl_2 (1:9), −78 °C; (4) Me_2S, Et_3N, MeOH-CH_2Cl_2 (1:9), room temperature] in 50% overall yield, bp 68–70 °C (0.6 mm).
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Photochemistry and Photocatalytic Activity of a Polynuclear Metal Carbonyl Hydride: Dodecacarbonyltetrahydridotetraruthenium

Sir:

We report here our preliminary findings concerning the photochemistry and photocatalytic activity of the polynuclear hydride $H_4Ru_4(CO)_{12}$. While mononuclear hydrides and di- and trinuclear clusters have received considerable study,[1] the only other tetranuclear carbonyl species that have been the object of detailed photochemical studies are $[(\eta^5\text{-}C_5H_5)Fe(CO)]_4$[2] and $HFeCo_3(CO)_{12-n}L_n$ ($L = PPh_3$; n = 0, 2)[3] which undergo metal-to-solvent charge-transfer oxidation[2] and complex, inefficient declusterification,[3] respectively. Interesting photoreactions of $H_4Os_4(CO)_{12}$ and $Ir_4(CO)_{12}$ have been reported,[4,5] but the nature of the primary chemical result from irradiation has not been established. The $H_4Ru_4(CO)_{12}$ cluster and its substituted derivatives are known catalyst precursors for olefin isomerization and hydrogenation[6-8] and thus afford us a special opportunity with respect to studying light-activated catalysis, since the actual active species may be only one step away from the precursor $H_4Ru_4(CO)_{12}$.[6-8]

The $H_4Ru_4(CO)_{12}$ complex was synthesized according to the literature procedure.[9] The yellow-orange complex exhibits an intense, near-UV absorption maximum at 362 nm (ϵ 17 500 M^{-1} cm^{-1}) with a tail into the visible in hydrocarbon solvents. Near-UV irradiation (355 ± 20 nm, 1.2 × 10^{-6} einstein/min) of the complex alone in deoxygenated isooctane solution at 25 °C and a concentration of ~5 × 10^{-4} M gives slow decomposition to unidentified products, but as a function of time the decomposition becomes markedly slower when the sample is sealed. Irradiation under the same conditions but in the presence of L [L = $P(OMe)_3$ or PPh_3] results in clean infrared spectral changes; data in Figure 1 are representative. The infrared bands in the CO stretching region that are associated with the product are identical with those reported[10] for $H_4Ru_4(CO)_{11}L$. Continued near-UV irradiation results in additional infrared spectral changes consistent with further functionalization of the cluster to form $H_4Ru_4(CO)_{12-n}L_n$ (n = 1, 2, 3, 4), but, as shown in Figure 1 for L = PPh_3, monosubstituted clusters can be generated essentially quantitatively before multiple substitution products appear. The 366- or 436-nm quantum yield for the photosubstitution (eq 1) is 5 ± 1 × 10^{-3} for either $P(OMe)_3$ or PPh_3 and a concentration of L = 0.01 or 0.1 M.

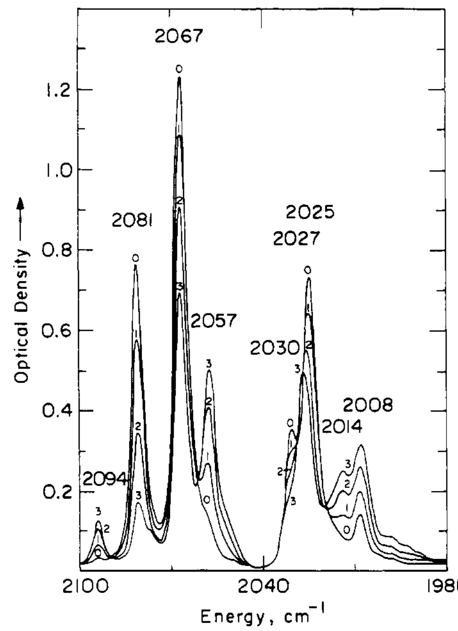

Figure 1. Infrared spectral changes accompanying near-UV (355 nm) irradiation of $H_4Ru_4(CO)_{12}$ (~5 × 10^{-4} M) in the presence of PPh_3 (~10^{-1} M) in *n*-pentane solution at 25 °C. Bands at 2081, 2067, 2030, 2025, and 2008 cm^{-1} are due to $H_4Ru_4(CO)_{12}$ and those growing with irradiation at 2094, 2057, 2027, 2014, and 2008 cm^{-1} are due to $H_4Ru_4(CO)_{11}PPh_3$. Curves 0, 1, 2, and 3 are after 0-, 20-, 40-, and 75-s irradiation, respectively.

$$H_4Ru_4(CO)_{12} + L \xrightarrow{h\nu} H_4Ru_4(CO)_{11}L + CO \qquad (1)$$

Dinuclear, metal–metal-bonded, metal carbonyls generally undergo very efficient metal–metal bond homolysis subsequent to optical excitation,[11-13] while trinuclear complexes undergo inefficient declusterification.[3,14-16] Presumably, the trinuclear complexes may undergo efficient metal–metal bond homolysis, but low declusterification yields result from efficient recoupling of the tethered radical centers. In the tetranuclear complexes where the lowest excitations involve transitions between orbitals delocalized over four metal atoms and where each metal atom is directly bonded to three others, it is less likely that complete metal–metal bond scission obtains. Rather, the optical excitation apparently results in metal–ligand cleavage as generally obtains for mononuclear metal carbonyls having metal-centered lowest excited states.[1] At this point we cannot

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Table I. Photocatalytic Chemistry Using $H_4Ru_4(CO)_{12}$[a]

A. Stoichiometric Reduction of Olefins

olefin	irrdn time, h	% convn[b]	product(s) (%)
1-pentene	7	69	*n*-pentane
3,3-dimethyl-1-pentene	1.5	35	3,3-dimethylpentane
	5	64	
	7	80	
cyclopentene	13	51	cyclopentane
1-pentyne	23	74	1-pentene (73)
			cis-2-pentene (10)
			trans-2-pentene (13)
			n-pentane (4)
2-pentyne	23	60	*cis*-2-pentene (75)
			trans-2-pentene (1)
			1-pentene (24)
			n-pentane (<1)

B. Photocatalytic 1-Pentene Isomerization

irrdn time, h	initial [1-pentene], M	% reductn[b] to *n*-pentane	% isomn[c] to 2-pentenes	t/c[d]	Φ_{isomer}[e]
1	0.1	27	24.6	1.20	0.33
3	0.1	55	70.3	1.26	0.33
7	0.1	69	93.0	1.63	0.19
2	2.0		12.5	1.8	1.59
4	2.0		24.3	1.8	1.54
6	2.0		41.9	1.9	1.77

C. Photocatalytic Hydrogenation of Olefins (10 psi of H_2)

olefin	irrdn time, h	turnover no.[f]	product(s) (%)
cyclopentene	24	206	cyclopentane
3,3-dimethyl-pentene	24	127	3,3-dimethylpentane
1-pentene	24	84	*n*-pentane + equilibration of pentenes
1-pentyne	24	11	1-pentene (66)
			cis-2-pentene (13)
			trans-2-pentene (10)
			n-pentane (11)
2-pentyne	24	137	1-pentene (2)
			cis-2-pentene (19)
			trans-2-pentene (73)
			n-pentane (6)

[a] All experiments were carried out in deoxygenated, dry benzene solutions of 5×10^{-4} M $H_4Ru_4(CO)_{12}$; 1-cm^3 samples in Pyrex ampoules were irradiated at 25 °C with a GE Blacklite (355 ± 20 nm) providing ~1.2×10^{-6} einstein/min incident on the sample. Thermal controls in all cases showed little or no conversion to products. Except where noted otherwise, the olefin concentration is 0.1 M. [b] Based on the assumption that $H_4Ru_4(CO)_{12}$ can transfer two molecules of H_2. [c] Percent conversion of 1-pentene to 2-pentenes. [d] t/c is ratio of *trans*- to *cis*-2-pentene isomerization products. [e] Number of 1-pentene molecules reacted per photon incident. [f] Number of reduced molecules after 24 h per molecule of $H_4Ru_4(CO)_{12}$ initially present.

rule out light-induced metal-metal bond cleavage followed by substitution of the radical center(s).[1,11,17] However, it is worth noting that the disubstituted cluster arises from irradiation of the monosubstituted cluster, whereas substitution of two radical centers would give some disubstituted product as a primary photoproduct. Finally, in this regard we note that CO bonded to transition metal surfaces can be photodissociated by optical excitation and the CO loss is not due to trivial heating effects.[18] Thus, our clean CO photosubstitution of $H_4Ru_4(CO)_{12}$ may serve as a model for photodissociation of nonbridging CO from metal surfaces.

Mechanism aside, the photosubstitution of $H_4Ru_4(CO)_{12}$ represents the successful functionalization of a large cluster by photochemical means; this provides the foundation for studies aimed at specific levels of substitution and functionalization of large clusters. As detailed below, such chemistry is important in understanding the light-induced catalytic activity of the $H_4Ru_4(CO)_{12}$ where the first step in the thermal catalysis is logically loss of CO followed by substrate binding.[6-8]

Near-UV irradiation of 5×10^{-4} M $H_4Ru_4(CO)_{12}$ has been carried out in the presence of 1-pentene and other olefins. Analysis of the organic products in the case of 1-pentene shows that the 1-pentene is catalytically isomerized to *cis*- and *trans*-2-pentene and slowly, stoichiometrically reduced to *n*-pentane. When the irradiation is carried out under the same conditions but also under 10 psi of H_2, we find catalytic generation of *n*-pentane. Data for 1-pentene and other olefins are given in Table I. Either stoichiometric or catalytic photoreduction of 2-pentyne initially yields principally *cis*-2-pentene; 1-pentyne initially yields principally 1-pentene. These results indicate that, once the conversion to the alkene takes place, the product is exchanged for another alkyne molecule at a rate which is faster than equilibration of the alkene product among its three isomers and faster than subsequent reduction to the corresponding alkane. Large turnover numbers have been found (Table I). By using a higher light intensity, we find turnover rates of >2000/h for isomerization of 1-pentene and >60/h for hydrogenation for 2 M 1-pentene and 10 psi of H_2 at 25 °C.

It is logical that photochemical loss of CO leads to catalytically active Ru species. At 2 M 1-pentene the observed quantum yield for alkene isomerization is ~1.6 where photosubstitution of CO by phosphines is occurring with a quantum yield of only 5×10^{-3}, consistent with the generation of a thermally catalytically active species. The ratio of isomerization to photosubstitution quantum yields indicates that each catalytically active species turns over ~300 times before another photon is needed. At 0.1 M 1-pentene the isomerization quantum yield is only 0.3, presumably because the active species may be competitively scavenged by the photoejected CO. At 10 psi of added CO, all isomerization and reduction activity of 0.1 M 1-pentene is suppressed. The suppression of catalytic activity by CO is consistent with the notion that photochemical CO loss is the key step in the photoactivation of $H_4Ru_4(CO)_{12}$. The mechanism of alkene reaction is therefore likely to be first photosubstitution to form a labile olefin complex followed by hydride transfer and then reductive elimination to form reduced olefin. Isomerization of alkenes may occur by reversible hydride transfer or by a π-allyl-hydride mechanism.

The only IR-detectable Ru product from irradiation of $H_4Ru_4(CO)_{12}$ in the presence of 1-pentene is $H_2Ru_4(CO)_{13}$.[19,20] Recall, vide supra, that $H_4Ru_4(CO)_{12}$ does not lead to identifiable products in the absence of olefin or other potential ligands. Thus, the 1-pentene would appear to serve as a hydride acceptor and the resulting $H_2Ru_4(CO)_n$ fragment scavenges CO to yield the $H_2Ru_4(CO)_{13}$. Comparison of the photochemically produced $H_2Ru_4(CO)_{13}$ with authentic $H_2Ru_4(CO)_{13}$, prepared as previously described,[19] confirms the product identity. The chemical yield of the photochemically produced $H_2Ru_4(CO)_{13}$ can be as high as 67%, but under irradiation it too effects the reduction of 1-pentene to *n*-pentane and the Ru product has not been identified.[21] Characterization and isolation of the presumed $H_4Ru_4(CO)_{11}$(alkene) has proven difficult. Irradiation routinely yields a weak IR peak at ~2097 cm^{-1} that may be attributable to the alkene complex, but this species has not been produced in sufficient concentration to allow characterization. To contrast our results on $H_4Ru_4(CO)_{12}$, note that irradiation of $H_4Os_4(CO)_{12}$ in the presence of $RCH{=}CH_2$ yields $H_3Os_4(CO)_{11}(HC_2HR)$.[4] It would appear that, if such a species is produced from the $H_4Ru_4(CO)_{12}$, it is far more thermally and/or photochemically labile, since our main product, $H_2Ru_4(CO)_{13}$, does not contain hydrocarbon ligands.

The details of the mechanism of the light-induced chemistry are still under study, but for now the principal finding is that catalytic chemistry of olefins can be induced at lower temperatures than needed thermally. Previous studies[6-8] show that $H_4Ru_4(CO)_{12}$ and its substituted derivatives are thermal catalysts for alkene isomerization and hydrogenation, but the temperatures used are at least in the 70-80 °C range; we find no thermal chemistry on the same time scale as our photoreactions at 25 °C. We find an initial *trans*- to *cis*-2-pentene ratio from 1-pentene to be near that found thermally,[6a] and the principal formation of 1-pentene from 1-pentyne and *cis*-2-pentene from 2-pentyne at low extent conversion parallels findings from the thermal catalysis.[6b] Thus, it would appear that the same catalyst is involved thermally and photochemically. Finally, the effect of added CO is to suppress both thermal[6] and photochemical olefin reactions. On these grounds and the photosubstitution chemistry of $H_4Ru_4(CO)_{12}$ we assert that photoinduced ejection of CO is contributory to achieving the catalytically active species, as is proposed in the thermal chemistry.[6] These data do not yet constitute unequivocal proof that the Ru_4 core remains intact during catalysis, since small amounts of very active mononuclear catalysts may yet be present. However, the photosubstitution does take the system a step closer to the catalyst at lower temperatures than ordinarily needed. The $H_4Os_4(CO)_{12}$ photochemistry in the presence of alkenes is clearly related to our work[4] and has led to isolable, apparently inert, Os_4 species that may be the result of CO ejection from $H_4Os_4(CO)_{12}$, but loss of hydrogen also occurs and it is not clear how this happens.

Preliminary results show that $H_2Os_3(CO)_{10}$, $H_3Mn_3(CO)_{12}$, and $H_4Re_4(CO)_{12}$ can also effect stoichiometric reduction of olefins when irradiated at 25 °C. We do not imply that the metal core necessarily remains intact in these instances,[22] but rather we mention these as examples to illustrate that other polynuclear hydrides aside from $H_4Ru_4(CO)_{12}$ yield reactive reducing agents when irradiated.

Acknowledgments. We thank the National Science Foundation and the Office of Naval Research for support of this research. Partial support from GTE Laboratories, Inc., is also gratefully acknowledged. M.S.W. acknowledges support as a Dreyfus Teacher–Scholar grant recipient, 1975–1980, and J.L.G. as an Arthur D. Little Fellow in the Department of Chemistry MIT, Spring, 1979.

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(21) $H_2Ru_4(CO)_{13}$ is a known thermal catalyst for 1-pentene isomerization at ~70 °C: Vaglio, G. A.; Osella, D.; Valle, M. *Transition Met. Chem.* **1977**, *2*, 94. We find slow 25 °C, catalytic isomerization that can be accelerated by near-UV irradiation. We detect no thermal conversion to *n*-pentane at 25 °C.
(22) Indeed, recent results show that $H_3Mn_3(CO)_{12}$ is declusterified by light.[16]

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Convenient Method for Regiospecific Carbon–Carbon Bond Formation at the γ Position of Allylic Halides

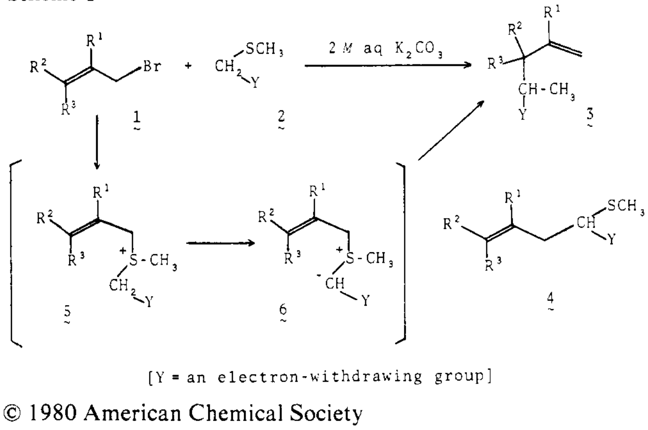
Sir:

We here disclose a new synthetic reaction for the regiospecific formation of a carbon–carbon bond at the γ position of an allylic bromide (**1**). The method simply involves stirring **1** and the α-methylthio ketone (**2**, Y = COR) in the presence of 2 M aqueous K_2CO_3 to give exclusively a substitution product (**3**, Y = COR) of the S_N2' type without contamination of any of its regioisomers (**4**, Y = COR) (Scheme I).

A typical procedure is as follows. To a mixture of 1 equiv of **2** and 1.6 equiv of **1** was added 2 equiv of 2 M aqueous K_2CO_3. This mixture was stirred for several days (see Table I) and extracted with CH_2Cl_2; the dried extract was evaporated; and the residue was column chromatographed (silica gel) or distilled to provide **3**.[1,3] It is noteworthy that, in every case, **4** was not detected by an NMR analysis of the reaction mixture.[4] The yields of **3** obtained by the reaction of (methylthio)acetone (**2**, Y = $COCH_3$) and ω-(methylthio)acetophenone (**2**, Y = COPh) with a variety of allylic bromides are shown in Table I.

For the present reaction, the mechanism involving nucleophilic attack of the carbanion of **2** on the γ position of **1** seems unfeasible by the following reasons: (i) The acidity of **2** is too low to be deprotonated by K_2CO_3; (ii) even if the carbanion

Scheme I



[Y = an electron-withdrawing group]

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