Table I. Bond Lengths (Å) and Angles (deg) for $ZnCl_2 \cdot B_2H_4 \cdot 2P(CH_3)_3$

	Bond Lengths		
Zn-Cl(1)	2.213 (1)	B(1)-H(B1b)	1.08 (4)
Zn-Cl(2)	2.208 (1)	B(2)-H(B2b)	1.20 (4)
Zn-H(B1b)	1.87 (5)	B(1)-H(B1a)	0.99 (5)
Zn-H(B2b)	1.90 (4)	B(2)-H(B2a)	1.11 (5)
B(1)-B(2)	1.814 (6)	B(1)-P(1)	1.912 (4)
		B(2)-P(2)	1.910 (4)
	Bond Angles		
Cl(1)-Zn-Cl(2)	114.7 (1)	B(1)-B(2)-H(B2b)	123.6 (21)
H(B1b)-Zn-H(B2b)	106.7 (18)	B(2)-B(1)-H(B1b)	120.2 (24)
Zn-H(B1b)-B(1)	97.2 (29)	B(1)-B(2)-H(B2a)	116.6 (22)
Zn-H(B2b)-B(2)	91.3 (24)	B(2)-B(1)-H(B1a)	114.2 (25)

$H_3)_3$ coordination in these complexes appears similar. Other metal compounds are being tested for the possible complexation with the diborane(4) adduct.

The symmetry of the $ZnCl_2 \cdot B_2H_4 \cdot 2P(CH_3)_3$ molecule is approximately C_2. The positions of the two bridge hydrogen atoms deviate little (0.06 and 0.13 Å) from the plane defined by the two boron atoms and the zinc atom, and each of the two terminal hydrogen atoms lies on the plane defined by its vicinal phosphorus atom and the two boron atoms.[7] The bond lengths and angles pertinent to this communication are listed in Table I. It is noted that zinc chloride did not react with $BH_3 \cdot P(CH_3)_3$ under the conditions of the $ZnCl_2 \cdot B_2H_4 \cdot 2P(CH_3)_3$ formation. The diborane(4) adduct appears to provide a favorable vicinal H-H distance for the chelation. The chelation by the two geminal hydrogen atoms, however, may be unfavorable because of the shorter H-H distance. It is noted further that, when a 1:1 mixture of $B_2H_4 \cdot 2P(CH_3)_3$ and $B_3H_5 \cdot 3P(CH_3)_3$[8] was treated with a 1-equiv quantity of $ZnCl_2$ in dichloromethane, $B_3H_5 \cdot 3P(CH_3)_3$ preferentially complexed with $ZnCl_2$. This observation suggests that the coordination of neutral borane adducts to metal centers is a general type of reaction and may further be developed to include other higher borane adducts.

Acknowledgment. The authors acknowledge support of this work by the U.S. Army Research Office through Grant DAAG 29-81-K-0101.

Supplementary Material Available: A listing of final atomic coordinates and isotropic thermal parameters (2 pages). Ordering information is given on any current masthead page.

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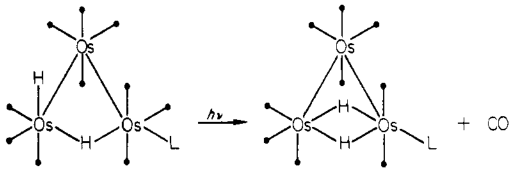
Photochemical Conversion of $H_2Os_3(CO)_{10}$(P-donor) to $H_2Os_3(CO)_9$(P-donor) in Solution and on High-Surface-Area Silica

Sir:

We wish to report our preliminary results concerning the photochemical generation of reactive intermediates from $H_2Os_3(CO)_{10}L$ (L = P-donor) in solution and on rigid oxide surfaces at 298 and 77 K. Our interest in these systems stems from that fact that the coordinatively unsaturated $H_2Os_3(CO)_9L$ species are well characterized[1] and are believed to be important in homogeneous catalytic reactions of olefins.[2] The relative importance of light-induced loss of CO from metal carbonyl clusters compared to metal-metal bond cleavage varies depending on the conditions under which the photochemistry is effected. Light-induced loss of CO from $Mn_2(CO)_{10}$[3] and *trans*-(η^5-C_5R_5)$Fe_2(CO)_4$ (R = H, $CH_2C_6H_5$, CH_3)[4,5] is the dominant photoreaction in rigid media at low temperature whereas net metal-metal bond cleavage dominates in fluid solution at 298 K. Relatively little is known regarding higher nuclearity clusters. The existence of both $H_2Os_3(CO)_{10}L$ and $H_2Os_3(CO)_9L$ complexes, therefore, provides an opportunity to investigate the mechanism of CO loss from these triangular clusters. Thus, the key issues in our study are establishment of (i) whether photoexcitation of $H_2Os_3(CO)_{10}L$ yields $H_2Os_3(CO)_9L$ and (ii) the mechanism for the light-induced chemistry. It has been previously established that $Os_3(CO)_{12}$ undergoes photosubstitution[6] and that $H_2Os_3(CO)_{10}(\eta^1$-SCHN-*p*-$C_6H_4F)$ undergoes light-induced loss of CO.[7] Our essential finding is that eq 1 represents the photochemistry at 298 K, but irradiation at low temperature does not result in CO loss as a primary photoreaction.



$H_2Os_3(CO)_{10}L$ $H_2Os_3(CO)_9L$ (1)

We have investigated three systems: $H_2Os_3(CO)_{10}(PPh_3)$, $H_2Os_3(CO)_{10}(PPh_2Et)$, and $[SiO_2]$–$L'Os_3(CO)_{10}H_2$ prepared from reaction of high-surface-area SiO_2 and $H_2Os_3(CO)_{10}(PPh_2CH_2CH_2Si(OEt)_3)$. The molecular species $H_2Os_3(CO)_nL$ (*n* = 9, 10; L = PPh_3, PPh_2Et, $PPh_2CH_2CH_2Si(OEt)_3$) were prepared according to the literature procedures or slight modifications thereof.[1,2,8] The modification of SiO_2 with $H_2Os_3(CO)_n(PPh_2CH_2CH_2Si(OEt)_3)$ (*n* = 9, 10) was carried out in a manner[9] similar to that previously reported.[10]

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[9] High-surface-area SiO_2 (400 m^2/g) purchased from Alfa was pretreated by heating at ~250 °C under vacuum (10^{-2} torr) for 48 h. A typical preparation of $[SiO_2]$–$L'Os_3(CO)_nH_2$ involves the suspension of ~1 g of pretreated SiO_2 in 100 mL of hexane. The $H_2Os_3(CO)_nL'$ (*n* = 9, 10; L' = $Ph_2PCH_2CH_2Si(OEt)_3$) was added to ~50 mM and the suspension stirred at 25 °C for 24 h under N_2. Elemental analysis establishes the coverage of cluster to be ~0.2 mmol/g or ~5 × 10^{-11} mol/cm^2 assuming that the surface area is 400 m^2/g. The solid was collected by filtration and washed repeatedly with hexane and dried under vacuum and under N_2. Control experiments using $H_2Os_3(CO)_n(PPh_2Et)$ (*n* = 9, 10) as the derivatizing agents yields no surface-confined metal carbonyls after washing with hexane.

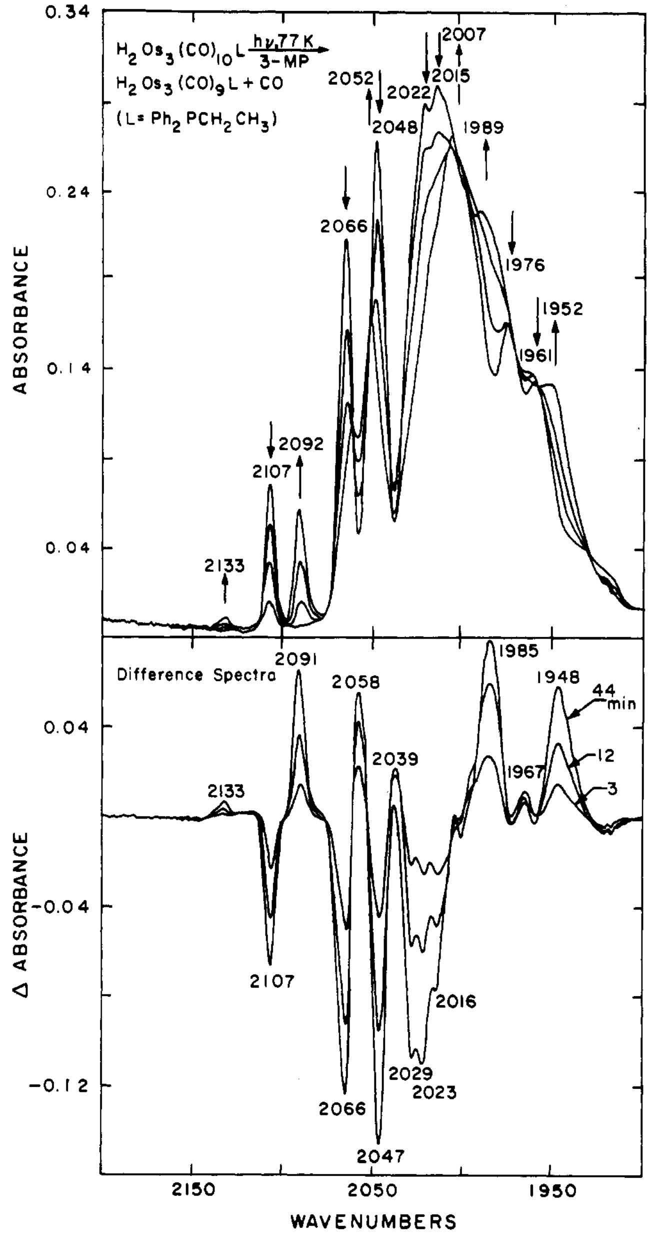

Figure 1. Top: Infrared absorbance changes accompanying near-UV irradiation of ~1 mM $H_2Os_3(CO)_{10}(PPh_2Et)$ in 3-methylpentane at 77 K. Bottom: Infrared difference spectra corresponding to changes shown in top. Positive peaks are product and negative peaks are starting material. Refer to Table I for band positions of starting material and products.

Characteristic IR and UV–vis spectral features for the molecular species and modified SiO_2 are given in Table I.

Near-UV irradiation (355 ± 20 nm, ~10^{-6} einstein/min) of ~0.5 mM $H_2Os_3(CO)_{10}L$ (L = PPh_3, PPh_2Et) in deoxygenated alkane media at 298 K leads to rapid IR and UV–vis spectral changes showing that eq 1 represents the photoreaction. The conversion is nearly quantitative to ~30% consumption of the starting material when CO is purged from the solution with Ar. The $H_2Os_3(CO)_9L$ photoproduct undergoes slower photoreaction to give unidentified products. Exposure of a solution containing photochemically generated $H_2Os_3(CO)_9L$ to CO at 1 atm leads to clean regeneration of $H_2Os_3(CO)_{10}L$, as expected. Consistent with this finding we note that low-extent conversion according to eq 1 is obtained in sealed vessels. The initial 366-nm quantum yield for the photoreaction is ~0.02.

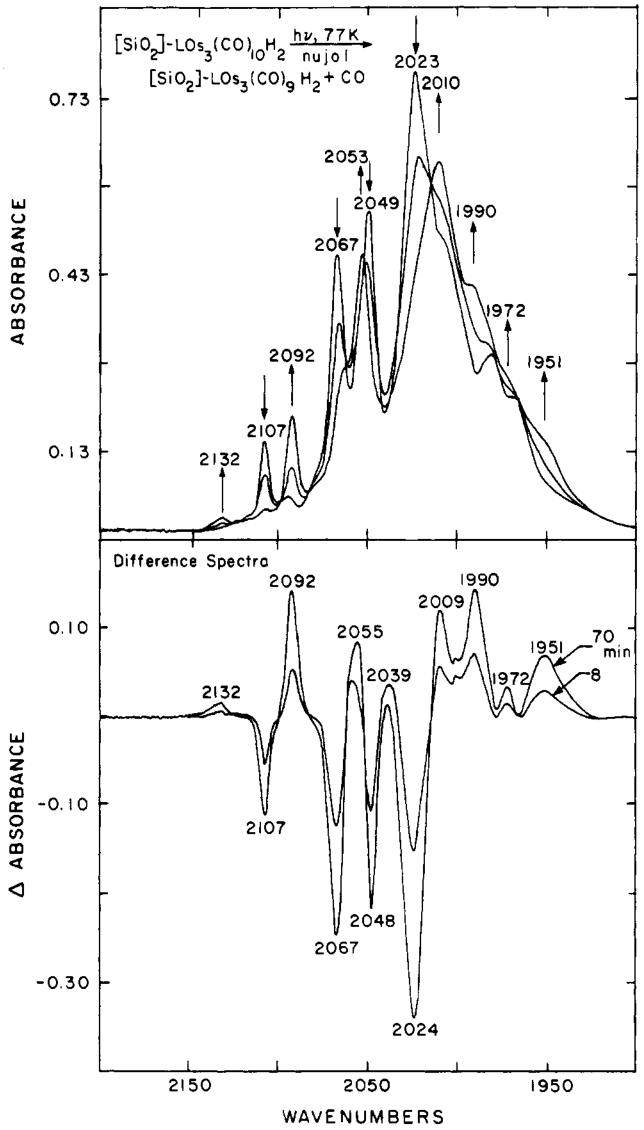

Figure 2. Top: Infrared absorbance changes accompanying near-UV irradiation of $[SiO_2]\text{-}L'Os_3(CO)_{10}H_2$ in a Nujol mull at 77 K. Bottom: Infrared difference spectra for the changes shown in the top. Refer to Table I for band positions of starting material and product.

As for $H_2Os_3(CO)_{10}L$ in alkane solution, we find that near-UV irradiation of a suspension of $[SiO_2]\text{-}L'Os_3(CO)_{10}H_2$ in deoxygenated alkane initially gives a nearly quantitative yield of $[SiO_2]\text{-}L'Os_3(CO)_9H_2$.[11] Again, exposure to CO regenerates the saturated cluster, $[SiO_2]\text{-}L'Os_3(CO)_{10}H_2$. The irradiation of $[SiO_2]\text{-}L'Os_3(CO)_{10}H_2$ does not yield detectable cluster in solution. Thus, the anchoring system is photoinert. The results with $[SiO_2]\text{-}L'Os_3(CO)_{10}H_2$ show that vacant sites on surface-bound clusters can be photogenerated.

Conceptually, the simplest mechanism for the conversion represented by eq 1 for both the homogeneous and suspended saturated cluster is dissociative loss of CO from the photoexcited cluster. However, the CO could originate from any one of the three chemically distinct Os atoms. We have begun an investigation of the mechanism of the photoreaction by monitoring the photochemistry at low temperature in order to trap intermediates.[12] Figures 1 and 2 show the IR spectral

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(11) UV–vis photoacoustic spectra of surface-confined materials were taken with a Princeton Applied Research Model 6001 photoacoustic spectrometer. IR spectra were obtained as Nujol mulls.

Table I. IR and UV-Vis Data for Relevant Complexes[a]

species	temp, K	band maxima	
		ν, cm^{-1} (ϵ or rel O.D.)	λ, nm (ϵ or rel O.D.)
$H_2Os_3(CO)_9(PPh_3)$	298	2092 (5500), 2054 (14 900), 2012 (17 800), 2004 (4800), 1993 (7300), 1976 (3400), 1962 (1800)	560 (200), 357 (15 800), 302 (11 700)
	77	2093 (8800), 2053 (22 000), 2015 (11 000), 2009 (24 000), 2003 (5400), 1993 (11 000), 1988 (6400), 1973 (4500), 1961 (2500)	355 (30 000), 307 (16 000)
$H_2Os_3(CO)_{10}(PPh_3)$	298	2106 (2800), 2068 (7100), 2052 (7900), 2025 (15 000), 2018 (5600), 2008 (3600), 2000 (3200), 1984 (2900), 1972 (3000), 1932 (290)	388 (9800), 333 (9100)
	77	2109 (3500), 2069 (5800), 2051 (8500), 2027 (17 000), 2006 (7500), 2000 (2600), 1983 (2600), 1976 (5000), 1955 (4400), 1929 (510)	375 (17 000), 325 (13 000)
$H_2Os_3(CO)_9(PPh_2Et)$	298	2092 (4300), 2053 (11 200), 2013 (14 400), 2005 (4400), 1990 (5500), 1976 (3200), 1957 (1900)	352 (12 800), 297 (9800)
	77	2092 (6100), 2053 (14 000), 2011 (15 000), 2003 (2900), 1986 (6300), 1973 (3900), 1955 (3100)	346 (21 000), 296 (14 000)
$H_2Os_3(CO)_{10}(PPh_2Et)$	298	2106 (2200), 2066 (6600), 2051 (7800), 2025 (14 300), 2007 (3900), 1999 (2900), 1983 (2900), 1972 (3300)	385 (8100), 331 (7900)
	77	2107 (2200), 2066 (7700), 2049 (9800), 2030 (8900), 2012 (9700), 1993 (2300), 1988 (2300), 1977 (4900), 1961 (2400), 1929 (500)	379 (15 000), 326 (13 000)
$H_2Os_3(CO)_9(PPh_2CH_2CH_2Si(OEt)_3)$	298	2091 (1.00), 2052 (2.70), 2012 (4.75), 1988 (1.74), 1974 (0.82), 1954 (0.39)	560 (1.0), 354 (82), 300 (67)
$H_2Os_3(CO)_{10}(PPh_2CH_2CH_2Si(OEt)_3)$	298	2106 (1.00), 2066 (3.04), 2051 (3.55), 2025 (7.94), 2007 sh, 1983 (1.41), 1972 (1.38)	385 (1.00), 332 (0.96)
$[SiO_2]$-$L'Os_3(CO)_9H_2$[b]	298	2091 (1.00), 2052 (2.28), 2009 (2.82), 1988 (1.53), 1953 (0.56)	562 (1.0), 355 (19), 306 (20)
$[SiO_2]$-$L'Os_3(CO)_{10}H_2$[b]	298	2105 (1.00), 2064 (3.12), 2048 (3.60), 2021 (5.46), 1981 (1.82), 1968 (1.37)	385 (1.0), 320 (1.1)

[a] All data are for 3-methylpentane solutions, unless noted otherwise. [b] $L' = PPh_2CH_2CH_2Si(OEt)_{3-n}$ (n = 1-3). For surface-supported species, infrared spectra were recorded as Nujol mulls by FTIR, and UV-vis data were recorded with use of a photoacoustic spectrometer, PAR 6001.

changes accompanying the photolysis of the saturated clusters dissolved or suspended in an alkane at ~77 K. Note that the IR spectral changes for the surface-bound cluster are very similar to those for the dissolved analogue. Interestingly, the conversion represented by eq 1 appears to occur, as evidenced by the appearance of a feature due to free CO (2133 cm^{-1})[13] and the ~2092-cm^{-1} band associated with the cluster product shown in eq 1. However, the yield of the unsaturated species and free CO based on consumption of starting cluster increases with the fraction of starting material consumed. The highest yield of the unsaturated cluster is no greater than ~50%, and initially (<5% conversion) neither CO nor the cluster product in eq 1 is observed. Figure 3 compares the IR spectral changes upon irradiation of $H_2Os_3(CO)_{10}(PPh_3)$ at 298 and 77 K at 8% consumption of starting material. The unsaturated cluster forms cleanly (~2092 cm^{-1}) at 298 K, but none is detected at 77 K. At the lowest extent conversions there is a weak absorption for a product at ~2128 cm^{-1} (depending on L) that is likely associated with the primary photoproduct that does not involve CO loss. As the cluster product in eq 1 forms, CO forms and the ratio of these is constant, but the CO to cluster product ratio is ~2.0.[14] However, the amount of CO formed based on consumption of starting cluster never exceeds a ratio of 1.0. Warm-up of the irradiated sample to 298 K at the ~2% conversion stage yields regeneration of $H_2Os_3(CO)_{10}L$ and no detectable $H_2Os_3(CO)_9L$. The product at ~2% conversion is persistent in the dark at 77 K for at least 2 h. Warm-up (to 298 K) of irradiated samples when the 2092-cm^{-1} band is present yields some additional product and some regeneration of $H_2Os_3(CO)_{10}L$; $H_2Os_3(CO)_9L$ and $H_2Os_3(CO)_{10}L$ account for all Os (±10%) initially present.

The low-temperature photochemistry rules out dissociative loss of CO as the primary reaction upon photoexcitation of $H_2Os_3(CO)_{10}L$, although $H_2Os_3(CO)_9L$ is formed as given in eq 1 at 298 K. The fact that more CO than $H_2Os_3(CO)_9L$ is formed at 77 K indicates the presence of at least one other CO loss product that can back-react or give $H_2Os_3(CO)_9L$ upon warming to 298 K. Additional studies are under way to establish the structure of the photogenerated intermediates that may derive from a geometrical rearrangement, metal–metal bond cleavage, or loss of hydrogen. Loss of the P-donor

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(12) Complexes were dissolved at 298 K to 0.1–1.0 mM to ensure site–site isolation upon cooling to 77 K. Cooling to 77 K often caused precipitation of these complexes in alkane media. Samples were run at several concentrations to ensure that results are not a consequence of aggregation. IR spectra were recorded at 77 K on a Nicolet 7199 FTIR, and UV-vis spectra were recorded with a Cary 17. The Dewar and irradiation procedures have been described previously: Kazlauskas, R. J.; Wrighton, M. S. *J. Am. Chem. Soc.* **1982**, *104*, 6005.

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(14) The absorptivity of CO in the matrices used is ~300 M^{-1} cm^{-1} on the basis of control experiments involving photodissociation of CO from $M(CO)_6$ (M = Cr, W). This allows quantitative determination of the number of CO's lost per molecule. The details of the determination of CO absorptivity will be published elsewhere.

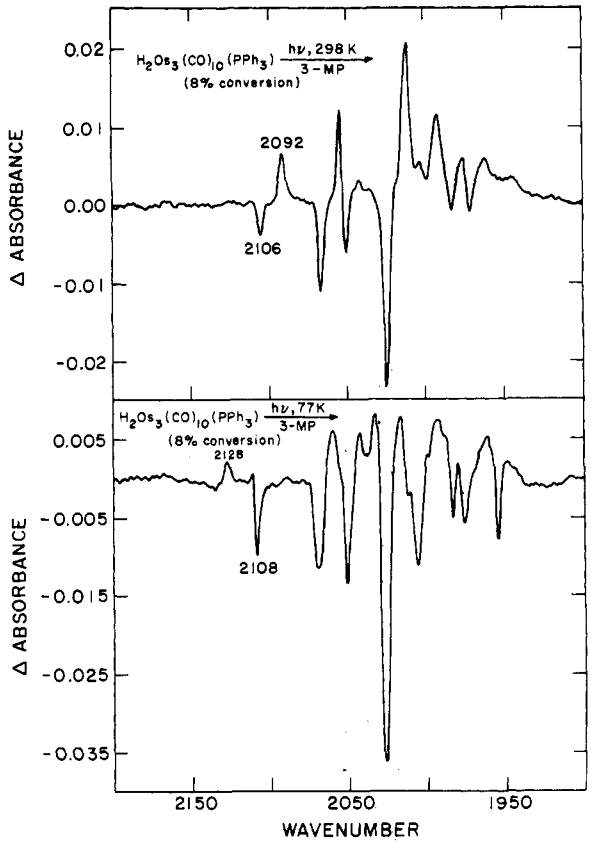

Figure 3. Comparison of difference spectra upon near-UV irradiation at 8% consumption of starting 1 mM $H_2Os_3(CO)_{10}(PPh_3)$ in 3-methylpentane at 298 and 77 K. Note that at 298 K $H_2Os_3(CO)_9(PPh_3)$ is formed (2092 cm^{-1}) whereas nearly none is detected at 77 K. Further irradiation at 77 K does produce $H_2Os_3(CO)_9(PPh_3)$ as for $H_2Os_3(CO)_{10}L$ shown in Figures 1 and 2.

is apparently not efficient, since we do not find release of Os species into solution from the irradiation of $[SiO_2]-L'Os_3(CO)_{10}H_2$. Interestingly, the low-temperature photochemistry of $H_2O_3(CO)_{10}L$ departs from the low-temperature photochemistry of $Mn_2(CO)_{10}$[3] and $(\eta^5\text{-}C_5R_5)_2Fe_2(CO)_4$[4,5] that do lose CO upon photoexcitation in rigid media but give metal-metal bond cleavage upon photoexcitation in fluid solution.

Acknowledgment. This research was supported in part by the Office of Naval Research and the National Science Foundation.

Registry No. $H_2Os_3(CO)_{10}(PPh_3)$, 56398-26-6; $H_2Os_3(CO)_{10}(PPh_2Et)$, 78885-55-9; $H_2Os_3(CO)_9(PPh_3)$, 88510-52-5; $H_2Os_3(CO)_9(PPh_2Et)$, 75835-64-2; $H_2Os_3(CO)_9(PPh_2CH_2CH_2Si(OEt)_3)$, 88510-53-6; $H_2Os_3(CO)_{10}(PPh_2CH_2CH_2Si(OEt)_3)$, 88510-54-7; SiO_2, 7631-86-9; CO, 630-08-0.

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σ- and π-Acidity of $As(OTeF_5)_3$

Sir:

We have been exploring the use of $P(OTeF_5)_3$[1] and $As(OTeF_5)_3$[2,3] as extremely bulky substitutes for carbon monoxide in metal complexes. The pentafluoroorthotellurate $(OTeF_5)$[4] group has been substituted for fluorine or chlorine in a wide variety of main-group and high-valent transition-metal compounds.[5-7] By using various physicochemical criteria, including molecular structures (group site preferences),[8,9] NMR chemical shifts,[9-11] Mössbauer isomer shifts,[11] chemical reactivities,[12] and the thermal stabilities[13] of some main-group $OTeF_5$ derivatives, it has been repeatedly demonstrated that the electronegativity of $OTeF_5$ and fluorine are practically equal. This suggests the electronic similarity $P(OTeF_5)_3$ and $As(OTeF_5)_3 \sim PF_3$, $AsF_3 \sim CO$. Herein we report that $As(OTeF_5)_3$ behaves chemically like AsF_3 in its ability to form adducts with simple Lewis bases (σ-acidity) and to form complexes with low-valent transition metals (π-acidity). The occurence of both σ- and π-acidity in the same molecular species is rare.

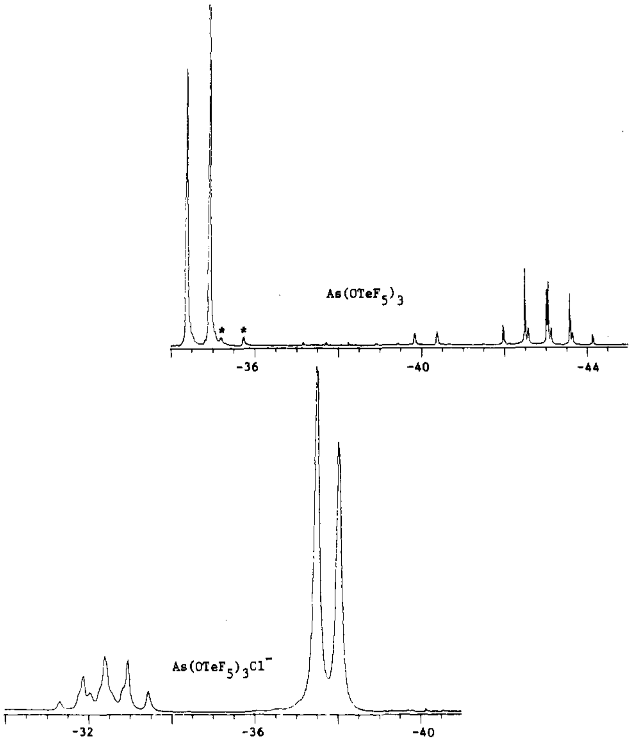

Figure 1. ^{19}F NMR spectra (338.8 MHz, 22 °C) of $As(OTeF_5)_3$ (upper trace) and $P(n\text{-}Bu)_4{}^+As(OTeF_5)_3Cl^-$ (lower trace). Both samples were 0.10 M in 50:50 CH_2Cl_2/CD_2Cl_2. Chemical shifts (ppm) are upfield of internal $CFCl_3$. Only half of the ^{125}Te satellites are visible in the upper trace. The two peaks marked with asterisks are due to an unidentified impurity.

The slightly volatile liquid $As(OTeF_5)_3$ was prepared by a literature procedure.[3] The ^{19}F NMR spectrum of this compound in CH_2Cl_2, shown in Figure 1 (top), is an AB_4X pattern

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