References and Notes
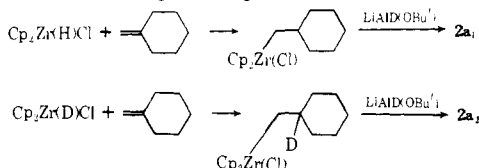
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(4) All reactions were performed under an atmosphere of dry argon. Solvents were dried by distillation from sodium or sodium benzophenone ketyl under nitrogen.

(5) Compounds 1a–d were prepared from Cp$_2$Zr(H)Cl and the corresponding olefin: J. Schwartz and J. A. Labinger, *Angew. Chem., Int. Ed. Engl.*, **15**, 33 (1976). Compound 1e was prepared from Cp$_2$ZrCl$_2$ and neopentyllithium.

(6) For example, hydrolysis of crude 2a with 2 N aqueous H$_2$SO$_4$ gave methylcyclohexane (90% by VPC) and H$_2$ (110% gas buret).

(7) Identified by ^{1}H NMR and hydrolysis to yield RH and ^{i}PrOH (GC/mass spectral identification). For R = A, ^{1}H NMR (benzene-d$_6$) δ 5.73 (10 H, s, Cp), 3.97 (1 H, septet, *J* = 6 Hz, –OCH–), 2.17–0.80 (13 H, br, alkyl group), 0.93 (6 H, d, *J* = 6 Hz, OCH(CH$_3$)$_2$).

A

(8) For 2a, ^{1}H NMR (THF-d$_8$, −30 °C) δ 5.82 (10 H, s, Cp), 1.7–0.9 (11 H, br, alkyl group), 0.35 (2 H, br, α-CH$_2$), −2.35 (1 H, br, hydride).

(9) A similar chemical shift has been reported for the bridging hydride in [bis(π-tetrahydroindenyl)zirconium dihydride]$_2$ (H. Weigold, A. P. Bell, and R. I. Willing, *J. Organomet. Chem.*, **73**, C23 (1974)) and is inconsistent with that for terminal hydrides in Zr(IV) species.^{1}

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(12)

Cp$_2$Zr(H)Cl + ⬡═ → Cp$_2$Zr(Cl) → (LiAlD(OBu^t)$_i$) → 2a$_i$

Cp$_2$Zr(D)Cl + ⬡═ → D Cp$_2$Zr(Cl) → (LiAlD(OBu^t)$_i$) → 2a$_j$

(13) S. Meyerson, T. D. Nevitt, and P. N. Rynalder, *Adv. Mass. Spectrom.*, **2**, 313 (1963). Spectra were also compared with those of authentic deuterated samples.

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(15) J. W. Lauher and R. Hoffmann, *J. Am. Chem. Soc.*, **98**, 1729 (1976).

(16) 2a$_1$ was stirred in dilute toluene solution for 3 h, during which time ≤5% methylcyclohexane was formed. Workup of the inorganic product with acetone gave B. The H/D ratio in the α position of the isopropoxide group

Cp$_2$Zr(OPr^i)
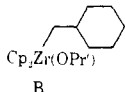
B

was determined as 1.06 by NMR integration. The H/D ratio in the β position of the alkyl group was determined by hydrolysis of the compound with 2 N H$_2$SO$_4$ and mass spectral analysis of the resulting methycyclohexane. The ratio of d$_0$/d$_1$' was ~1.

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Kerrie I. Gell, Jeffrey Schwartz*[19]

Department of Chemistry, Princeton University
Princeton, New Jersey 08540

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Multiphoton Dissociation of Molecules with Low Power Continuous Wave Infrared Laser Radiation

Sir:

It is now a recognized fact that infrared lasers can energize isolated molecules in multiphoton absorption processes.[1-3] While vibrational excitation of molecules with CW lasers has
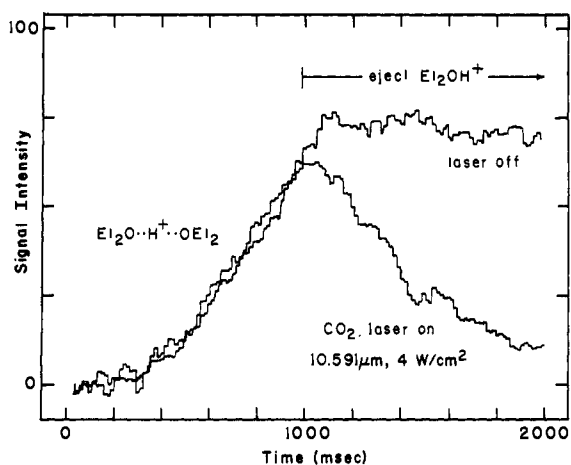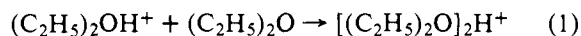
Figure 1. Ion intensity vs. trapping time for a typical multiphoton dissociation experiment. At a diethyl ether pressure of 5.5 × 10^{-7} Torr ions are formed by a 10-ms 70-eV electron beam pulse. The upper trace is the proton bound dimer signal with the laser off. Ejection of (C$_2$H$_5$)$_2$OH$^+$ beginning at 1-s trapping time halts further formation of the dimer. Continuous irradiation by the infrared laser at 4 W cm^{-2} coincident with ejection of (C$_2$H$_5$)$_2$OH$^+$ (lower trace) results in photodissociation of the dimer. At this pressure the time between collisions is ~50 ms.

been used to enhance bimolecular reaction rates[4] in the gas phase and to effect selective isomerization processes in matrices,[5] the majority of studies of multiphoton processes have utilized high intensity (>5 MW cm^{-2}) pulsed CO$_2$ lasers. Bloembergen and co-workers have recently demonstrated that dissociation probabilities of *isolated* molecules depend only on energy fluence and not on peak laser power.[3] Energy fluences used to effect multiphoton dissociation processes have typically been several joules cm^{-2}.[3] If it is only energy fluence that is important then there is the intriguing possibility that relatively low power CW lasers (several watts) can effect dissociation processes provided that a molecule can be irradiated under nearly collision free conditions for times approaching 1 s. We wish to report that such conditions can be established and multiphoton dissociation processes are readily observed thus adding a new dimension to this growing field on endeavor.
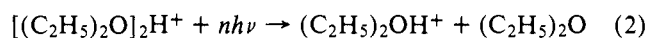
The experimental methodology is a straightforward application of techniques involving trapped ion cyclotron resonance spectroscopy (ICR)[6,7] which have been developed and applied to investigate the photochemistry of ions in the gas phase.[8-14] For the present experiments the output of a line tunable Apollo 550A CW CO$_2$ laser (bandwidth estimated to be 100 MHz) is directed transverse to the magnetic field through a 92% transparent mesh to irradiate ions in the source region of an ICR cell. The collimated 6-mm-diameter infrared beam is reflected back through the source region by a mirror finish on the bottom plate. Intensity variations across the ion spatial distributions are minimal owing to the small size of the ion packet.[15] Small translations (up to 3 mm) of the laser beam do not alter dissociation rates. A typical experiment thus involves pulsing the electron beam to generate ionic species which can be stored for several seconds, during which time reactions may occur. The ions are then mass analyzed to determine concentrations of the various species present.[7] The laser can be electronically gated to irradiate ions during any portion of the trapping sequence. Typical neutral pressures are in the range 10^{-7}–10^{-6} Torr, corresponding to neutral particle densities of 3 × 10^9 to 3 × 10^{10} molecules cm^{-3}. Typical ion densities are 10^5 ions cm^{-3}.

The major ions in the 70-eV electron impact mass spectrum of diethyl ether at long trapping times and low pressures are (C$_2$H$_5$)$_2$OH$^+$ and CH$_3$CHOC$_2$H$_5$$^+$.[16] At higher pressures the proton bound dimer is formed in processes 1.[17]

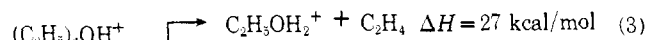$$(C_2H_5)_2OH^+ + (C_2H_5)_2O \rightarrow [(C_2H_5)_2O]_2H^+ \quad (1)$$

The enthalpy change for is estimated to be -31 ± 2 kcal/mol.[18] Although infrared absorption bands of the ions are unknown, multiphoton dissociation of species such as $[(C_2H_5)_2O]_2H^+$ was anticipated based on strong absorption bands of $(C_2H_5)_2O$ in the 9–10-μ region.[19]

A typical experiment monitoring $[(C_2H_5)_2O]_2H^+$ is shown in Figure 1. Ions are produced by a 10-ms electron beam pulse and stored for up to 2 s. At 1 s of trapping time the remaining $(C_2H_5)_2OH^+$ is ejected by double resonance in a time short compared with the time between collisions,[6,20] thus preventing further formation of the dimer. This is evidenced in Figure 1 (upper trace) by the constant abundance of $[(C_2H_5)_2O]_2H^+$ after 1 s. The laser, tuned to the 10.6-μ P(20) line of the 001–100 transition, is then turned on at 1 s of trapping time, coincident with the ejection of $(C_2H_5)_2OH^+$, and effects an exponential decay of the dimer (Figure 1, lower trace) with a *power of 4 W cm^{-2}!* At this laser power no appreciable photodissociation of $(C_2H_5)_2OH^+$ is observed, and the increase in abundance of this species exactly matches the decrease in abundance of the proton bound dimer, indicating that

$$[(C_2H_5)_2O]_2H^+ + nh\nu \rightarrow (C_2H_5)_2OH^+ + (C_2H_5)_2O \quad (2)$$

is the only decomposition pathway. For the data in Figure 1 a semilog plot of fractional dissociation yield as a function of irradiation time is linear, and the slope defines a photodissociation rate constant.[21] Photodissociation rate constants measured in this fashion are observed to decrease with increasing pressure, which is the expected effect of collisional deactivation processes.[22] The data extrapolate smoothly to zero pressure, yielding a collisionless rate constant which within experimental error is directly proportional to photon flux in the range of 1.0–4.0 W cm^{-2}. Dividing by photon flux yields a phenomenologically defined multiphoton dissociation cross section of $\sigma_D = 3.8 \times 10^{-20}$ cm^2, comparable with the value $\sigma_D = 1.5 \times 10^{-20}$ cm^2 estimated for multiphoton dissociation of SF$_6$.[2]

At lower pressures where dimer formation is not important, higher laser intensities (6–8 W cm^{-2}) lead to dissociation of $(C_2H_5)_2OH^+$. Of the two lowest energy pathways 3 and 4, only the former with $\sigma_D \simeq 2 \times 10^{-21}$ cm^2 is observed.[23]

$$(C_2H_5)_2OH^+ + nh\nu \quad \begin{cases} \rightarrow C_2H_5OH_2^+ + C_2H_4 & \Delta H = 27 \text{ kcal/mol} \quad (3) \\ \rightarrow C_2H_5^+ + C_2H_5OH & \Delta H = 53 \text{ kcal/mol} \quad (4) \end{cases}$$

A preliminary examination of the wavelength dependence of process 2 shows the photodissociation yield to be nearly constant over the 925–1090-cm^{-1} tuning region of the laser. This is not unexpected with 87 vibrational degrees of freedom and a rather flexible framework. We have also observed multiphoton dissociation of the molecular ion of perfluoropropylene to yield $C_2F_4^+$ and CF_2. In this case σ_D is strongly dependent upon wavelength with λ_{max} at 1047 cm^{-1} and a bandwidth of 25 cm^{-1}.[24,25] This wavelength dependence is similar to the 18-cm^{-1} bandwidth reported for high power, pulse laser dissociation of ^{32}SF$_6$.[26]

There are many possible extensions and applications of the present results which are currently being explored in our laboratory. Numerous aspects of the photophysics of multiphoton absorption processes can be studied, including collisional energy relaxation rates and IR emission lifetimes. Infrared absorption spectra of gas phase ions can be obtained by monitoring dissociation or by observing modified reaction rates as a function of wavelength. The low power requirements (<0.5 W cm^{-2}) imply that a wide range of available infrared lasers can be employed.[27] Because of the slow nature of the process,

the potential energy surface available to the decomposing molecule is thoroughly sampled below (but not above) the lowest energy decomposition pathway. This offers intriguing possibilities for mechanistic studies. In addition one can selectively heat ions and observe dissociation processes in cases where elevated temperatures would lead to degradation of neutral precursors (e.g., polypeptides) prior to ionization.

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(17) The kinetics of reaction 1 have not been investigated in detail. Observation of appreciable amounts of proton bound dimer only after several collisions have taken place (pressures >5 × 10^{-7} Torr and trapping times >0.5 s) suggests that reaction 1 is termolecular. However, bimolecular association reactions are known: M. K. Murphy and J. L. Beauchamp, *J. Am. Chem. Soc.*, **98**, 5781 (1976).

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(21) Since reaction 1 may initially impart up to 31 kcal/mol of internal energy to $[(C_2H_5)_2O]_2H^+$, the possibility of photolyzing vibrationally excited species requires consideration. Experiments in which the laser was turned on 500 ms after ejection of $(C_2H_5)_2OH^+$ produced the same multiphoton dissociation rates as when the laser was turned on coincident with ejection of $(C_2H_5)_2OH^+$. Since allowing additional collisions (~35 at 2 × 10^{-6} Torr) did not alter the photodissociation rate, $[(C_2H_5)_2O]_2H^+$ must have been thermalized prior to photolyses.

(22) The mechanism of low intensity multiphoton excitation differs in one respect from the pulsed laser experiments because the time scale of the experiment allows for spontaneous emission as an important route to deactivation of vibrationally excited species. Typical infrared radiative rates (calculated assuming a dipole transition moment of 0.01–0.20 D and λ = 1000 cm^{-1}) are ~1–100 s^{-1}, competitive with the observed dissociation rates (<10 s^{-1}). For laser intensity of 1 W cm^{-2} and transition moments mentioned above the Rabi frequency is >10^5 s^{-1}. Therefore the photodissociation rate-determining step is not the initial excitation of a ground-state ion.

(23) The $CH_3CHOC_2H_5^+$ ion is also observed to photodissociate at ~8 W cm^{-2}. Loss of ethylene to give CH_3CHOH^+ is the lowest energy pathway; this could not be confirmed owing to the complexity of competing reactions. The cross section was not measured but is estimated to be <2 × 10^{-21} cm^2.

(24) The observation of a sharp frequency dependence indicates that this experimental technique probes true photochemistry as opposed to heating effects in the ICR cell. Furthermore, if ion decompositions were due to heating of the cell or neutral gases, the rate of the lowest energy process in the diethyl ether system (reaction 3) would be comparable with or faster than the rate of $[(C_2H_5)_2O]_2H^+$ decomposition. Similarly, in the C_3F_6 system

the fragment ion $C_3F_5^+$ is unperturbed by the infrared radiation despite the availability of a decay channel whose threshold is ~72 kcal/mol: J. L. Beauchamp and B. S. Freiser, *J. Am. Chem. Soc.*, **96**, 6260 (1974). The presence of an ion whose concentration is unaffected by the laser radiation establishes that operation of the ICR cell is not perturbed by the infrared beam.

(25) D. S. Bomse, R. L. Woodin, and J. L. Beauchamp, unpublished work.
(26) R. V. Ambartzumian, *Proc. Nordfjord Conf., 1976,* 150 (1976).
(27) The short coherence time of the laser (~10^{-8} s) indicates that even conventional incoherent infrared sources in conjunction with a *monochromator* or band pass filter should make it possible to observe slow multiphoton dissociation through the *entire* infrared spectral range. A 300-W commercially available blackbody source provides 0.29 W of radiation at 1000 cm^{-1} (25-cm^{-1} band) and 1.1 W at 3000 cm^{-1} (also over a 25-cm^{-1} band). These powers should be adequate to effect photodissociation in view of the present experiments.
(28) NSF Predoctoral Fellow.

R. L. Woodin, D. S. Bomse,[28] and J. L. Beauchamp*

Contribution No. 5728
Arthur Amos Noyes Laboratory of Chemical Physics
California Institute of Technology
Pasadena, California 91125

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Figure 1. Possible structures for $BC_4H_5Fe(CO)_3$.

Synthesis of $BC_4H_5Fe(CO)_3$. An Example of the Conversion of an Organometallic Compound into a Metallocarborane

Sir:

Traditionally, organometallic compounds are viewed as polyhapto-ligand metal complexes and it was only recently that Wade pointed out that such compounds can also be viewed as cage systems in which the metal acts as a single atomic member of the cage.[1] In such a model, organometallic compounds are related to boranes, compounds for which cage geometry is related in a simple way to the number of valence electrons assigned to the framework.[2] If this model is more than an academic extrapolation, then organometallic compounds should exhibit some cagelike chemical or physical characteristics. In terms of the electronic states of the cations, we have already demonstrated similarities between B_5H_9, $B_4H_8Fe(CO)_3$, and $C_4H_4Fe(CO)_3$[3] and, in addition, have shown that in one way the photochemical behavior of $B_4H_8Fe(CO)_3$ is similar to that of $C_4H_4Fe(CO)_3$.[4] Herein is reported the preparation and characterization of $BC_4H_5Fe(CO)_3$ from the short-time photolysis of $C_4H_4Fe(CO)_3$ in the presence of B_5H_9. This observation both demonstrates the conversion of an organometallic compound into a metallocarborane[5] and supports the usefulness of the cage analogy in areas outside of borane chemistry.

Irradiation at 360 nm of a light yellow solution of 2.1 mmol of $C_4H_4Fe(CO)_3$ and 2.3 mmol of B_5H_9 in 3.5 mL of diethyl ether[6] for 15 to 30 min in a Rayonet photochemical reactor resulted in a dark brown solution and the production of 0.05 mmol of noncondensables. The reaction mixture was fractionated and 1.8 mmol of $C_4H_4Fe(CO)_3$ was recovered along with a few milligrams of a new compound.[7] The new compound is more volatile than $C_4H_4Fe(CO)_3$ but less volatile than B_5H_9, and purification was achieved by repeated fractionation on a vacuum line.

The new compound has an empirical formula $FeBC_7O_3H_5$ ($^{56}Fe^{11}B^{12}C_7^{16}O_3^1H_5^+$, calcd 203.9682 amu, obsd 203.9684 amu; $^{56}Fe^{10}B^{12}C_7^{16}O_3^1H_5^+$, calcd 202.9716 amu, obsd 202.9718 amu). The parent ion in the mass spectrum fragments by the sequential loss of three CO molecules, and the envelopes of the parent ion and first three fragment ion clusters are consistent with a molecule containing a single boron atom. The gas phase infrared spectrum exhibits bands at 2930 (m, CH), 2550 (m, BH), 2080 (s, CO), and 2030 (vs, CO) cm^{-1}.[8] The 25.2-MHz ^{11}B FT NMR spectrum in CD_2Cl_2 contains
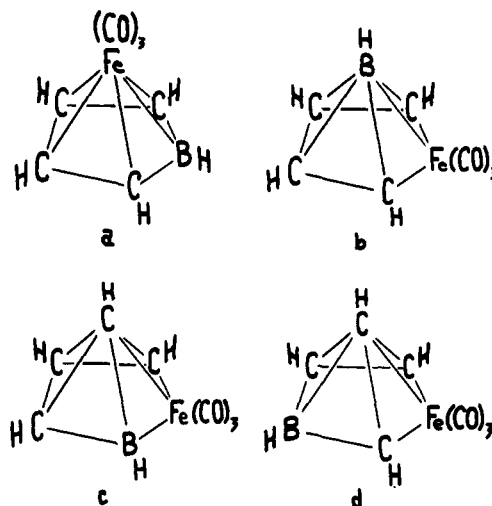
a single doublet with chemical shift of 11.8 ppm ($J = 140$ Hz) downfield relative to $BF_3 \cdot O(C_2H_5)_2$. The 100-MHz ^1H FT NMR spectrum in C_6D_6 consists of two multiplets (14-Hz fwhm) at δ 4.62 and 0.95 having relative areas of ~1:3.[9] On ^{11}B decoupling a broad peak (40-Hz fwhm) appeared at δ 2.57 with relative area 1. Under probe conditions[10] the signals attributed to the new compound decay with a half-life of ~1 h.

The new compound is formulated as $BC_4H_5Fe(CO)_3$, and it is isoelectronic with the known compounds $B_5H_9Fe(CO)_3$[11] and $B_3C_2H_7Fe(CO)_3$.[12] Four likely structures for this compound are shown in Figure 1. All four structures are nido frameworks in agreement with the presence of 16 skeletal electrons (2 from $Fe(CO)_3$, 2 from BH, and 3 from each CH) in a six-atom cage.[2] Structure a is that of the ligand borole bound to $Fe(CO)_3$, substituted derivatives of which have been reported recently.[13] This structure is ruled out on the basis of the proton spectra. Structure b is ruled out for the same reason and also because the ^{11}B chemical shift suggests a boron in the environment of a basal position rather than an apical position.[14] Neither structure c nor d can be ruled out on the basis of the available data. However, structure c is preferred as it is easier to rationalize the nearly equal chemical shifts of three CH protons.[15] It is interesting that the new compound has a basal $Fe(CO)_3$ group like $B_5H_9Fe(CO)_3$ rather than an apical $Fe(CO)_3$ like $B_3C_2H_7Fe(CO)_3$.

The mechanism for the formation of $BC_4H_5Fe(CO)_3$ is unknown and undoubtedly complex. Other products are formed in the reaction but those partially identified thus far do nothing to illuminate the situation. It is intriguing to note, however, that here the insertion of boron into the carbon ring takes place with the metal carbonyl fragment being retained in the product, whereas in the case of insertion of carbon into a metal bound borane cage the metal carbonyl fragment was lost.[4,16] Although the particular reaction reported here is poor in terms of yield, it does suggest a new approach to the preparation of unusual metallocarboranes.

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