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### **MOLECULAR BEAM STUDIES OF REACTION DYNAMICS**

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## Molecular Beam Studies of Reaction Dynamics

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### Scope of the Project

The major thrust of this research project is two-fold; 1) to elucidate detailed dynamics of simple elementary reactions which are theoretically important and to unravel the mechanism of complex chemical reactions or photo chemical processes which play an important role in many macroscopic processes and 2) to determine the energetics of polyatomic free radicals using microscopic experimental methods. Most of the information is derived from measurement of the product fragment translational energy and angular distributions using unique molecular beam apparatus designed for these purposes.

### I. Completed Work or In Progress

#### A. Molecular Beam Photoelectron Spectroscopy and Femtosecond Intramolecular Dynamics of $\text{H}_2\text{O}^+$ and $\text{D}_2\text{O}^+$

The 584 Å photoelectron spectra of supersonic molecular beams of  $\text{H}_2\text{O}$  and  $\text{D}_2\text{O}$  have been obtained with improved resolution. The spectroscopic constants of the  $\tilde{X}^2B_1$  and  $\tilde{A}^2A_1$  state ions, including  $\omega_1$ ,  $x_{11}$ ,  $\omega_2$ ,  $x_{22}$ , and  $x_{12}$ , are reported. For the first two electronic states of the ion, precise line splittings were evaluated with a least squares fitting procedure, employing sums of empirical instrument response functions and a linear background. A simulation of the vibrational manifolds of the  $\tilde{B}^2B_2$  state ions with combination progressions in the symmetry-allowed modes  $\nu_1$  and  $\nu_2$  failed to reproduce the diffuse photoelectron bands observed for both  $\text{H}_2\text{O}$  and  $\text{D}_2\text{O}$ . Autocorrelation functions were calculated from the photoelectron bands of all three electronic states. The  $\tilde{B}^2B_2$  state correlation functions exhibit ultrafast decay, occurring on a  $10^{-14}$  second timescale. The  $\nu_2$  motion appears to define the decay in the correlation function. This behavior supports a previously-proposed  $\tilde{B}^2B_2 - \tilde{A}^2A_1$  curve-crossing model for the nonradiative relaxation of the  $\tilde{B}^2B_2$  state ions.

B. On the High Resolution HeI Photoelectron Spectrum of the  $C_2D_4^+$  ( $X^2B_3$ ) Ground State.

The HeI (584Å) photoelectron spectrum of  $C_2D_4^+$  in its ground electronic state has been measured with a supersonic molecular beam. The improved resolution permits new vibrational fine structure to be observed and assigned. The disputed interpretation of  $\nu_2$  and  $\nu_3$  vibrations in  $C_2H_4^+$  ground state and the abnormal isotopic shift from  $C_2H_4^+$  to  $C_2D_4^+$  are explained by comparing the experimental results with a Franck-Condon factor calculation and a recent ab initio calculation. The torsional vibration ( $\nu_4$ ) is observed to be coupled significantly with the C=C stretching vibration ( $\nu_2$ ).

C. Spin-Forbidden Radiative Decay of the  $a^4\Pi_u$  State of  $O_2^+$ .

The spin-forbidden radiative decay of  $a^4\Pi_u$   $O_2^+$  has been measured in a radio frequency octopole ion trap. Photodissociation is used to probe the  $a^4\Pi_u$  population as a function of trapping time. We have found that the  $a^4\Pi_u$  state exhibits a multiple exponential decay, ranging from a few milliseconds to hundreds of milliseconds. The state dependence of the decay is seen in the photodissociation spectrum ( $b^4\Sigma_g^- \leftarrow a^4\Pi_u$ ), which changes dramatically from 0.1 ms to 100 ms. The major changes in the spectrum are simulated by assuming that the  $F_2$  and  $F_3$  spin components of the  $a^4\Pi_u$  state decay faster than the  $F_1$  and  $F_4$  components. We can account for this dependence on spin sub-level by assuming that the primary mechanism for radiative decay arises from spin-orbit coupling of the  $a^4\Pi_u$  and  $A^2\Pi_u$  states. Our results suggest that the  $a^4\Pi_u$  radiative lifetime of 0.22 s measured by O'Keefe and McDonald reflects the decay of only the longest living  $a^4\Pi_u$  sub-levels.

D. Photodissociation of  $CF_2BrCH_2I$  at 248, 266, and 308 nm.

The technique of photofragmentation translational spectroscopy has been used to study the photodissociation of  $CF_2BrCH_2I$  at excitation wavelengths of 248, 266, and 308 nm. The primary photofragments are  $CF_2BrCH_2$  and either  $I(^2P_{1/2})$  or  $I(^2P_{3/2})$ , although some C-Br bond fission does occur at 248 and 266 nm. A large fraction of the  $CF_2BrCH_2$  radical product contains enough internal excitation after the primary process to undergo secondary dissociation into  $CF_2CH_2$  and Br. Secondary dissociation is also observed to take place at 248 and 266 nm via absorption of a photon by the  $CF_2BrCH_2$  photofragment. By observing the threshold for the spontaneous secondary dissociation process, the reaction enthalpy for  $CF_2BrCH_2I \rightarrow CF_2CH_2 + Br + I$ , was determined to be  $67.5 \pm 2$  kcal/mol, which leads to:  $\Delta H_{f,0}^{\circ}(CF_2BrCH_2I) = -92.6 \pm 2$  kcal/mol. The c.m. translational energy distributions were derived for both the  $I(^2P_{1/2})$  and  $I(^2P_{3/2})$  dissociation channels resulting from primary C-I bond breakage. The  $I(^2P_{1/2})/I(^2P_{3/2})$  branching ratios are 3.3, 9.0, and 0.5 for excitation wavelengths of 248, 266, and 308 nm, respectively. The translational energy distributions also reveal that a major fraction of the  $CF_2BrCH_2$  product radicals are formed with high internal energies, averaging around 50 percent of the excess energy. The

angular distributions of dissociation products with respect to the laser polarization indicate that the primary photodissociation process for the ground and excited state channels at both wavelengths proceeds via a parallel transition--i.e., the transition moment must be nearly parallel to the C-I bond.

E. Photodissociation of CH<sub>2</sub>ClCH<sub>2</sub>I at 308 nm.

The technique of photofragmentation translational spectroscopy has been used to study the photodissociation of CH<sub>2</sub>ClCH<sub>2</sub>I at an excitation wavelength of 308 nm. The exclusive dissociation pathway is C-I bond breakage with formation of CH<sub>2</sub>ClCH<sub>2</sub> and I(<sup>2</sup>P<sub>3/2</sub>). The center-of-mass translational energy distribution of the photofragments reveals that an average of about 50 percent of the excess energy appears in translation. The angular distribution of dissociation products with respect to the laser polarization indicates that the photodissociation process proceeds via a parallel transition--i.e., the transition moment must be nearly parallel to the C-I bond. Exclusive production of ground state I(<sup>2</sup>P<sub>3/2</sub>) with a parallel polarization dependence is unexpected based on the prevailing picture for alkyl iodide photodissociation.

F. Determination of the C-H Bond Dissociation Energies of Ethylene and Acetylene by Observation of the Threshold Energies of H<sup>+</sup> Formation by Synchrotron Radiation.

To determine the C-H bond dissociation energies of ethylene and acetylene (R-H), we have measured the threshold energies of H<sup>+</sup> formation using synchrotron radiation in the wavelength region 58-70 nm. Subtracting the ionization potential of hydrogen atom (13.598 eV) from the observed threshold energies, we have deduced values of 5.06 ± 0.5 eV and 5.75 ± 0.05 eV for the C-H bond dissociation energies D<sub>0</sub>(R-H) of ethylene and acetylene, respectively.

G. Molecular Beam Photoelectron Spectroscopy of SO<sub>2</sub>: Geometry, Spectroscopy, and Dynamics of SO<sub>2</sub><sup>+</sup>.

We have reinvestigated the HeI (584Å) photoelectron spectroscopy of SO<sub>2</sub> using a supersonic molecular beam. Improved resolution and rotational cooling allow us to observe several new features and to resolve explicitly the vibrational structure in the first six electronic states, in the first three photoelectron bands. The adiabatic ionization potentials (IP's) were accurately determined for all six states. The  $\tilde{X}^2A_1$  state is assigned to the  $\nu_2$  mode exclusively. Irregularity of the vibrational progression on the high IP side was observed for the first time. A potential barrier (to linearity) is proposed to interpret the irregular vibrational spacings in the  $\nu_2$  vibration. The barrier height is estimated to be 0.42 eV (3400 cm<sup>-1</sup>). The complex second band contains two states. Abnormal vibrational structure in the  $\tilde{A}^2A_2$  state is explained by the principal excitation of the  $\nu_3$  mode. A potential barrier is present in the  $\nu_3$  potential surface,

so that the ion has an asymmetric equilibrium geometry in this state. The barrier height is estimated to be less than  $220 \text{ cm}^{-1}$ . A new progression is resolved in the  $\tilde{B}^2B_2$  state, which is assigned to be a combination of  $\nu_2$  with  $2\nu_3$ . The  $\nu_2$  vibration is observed to be strongly coupled with the  $\nu_3$  mode. The true adiabatic IP for the  $\tilde{C}^2B_2$  state is determined for the first time, as  $15.902 \pm 0.003 \text{ eV}$ . The dynamics of the ion dissociation in this state is discussed and slow predissociations through the lower lying states are suggested. The  $\tilde{D}^2A_1$  and  $\tilde{E}^2B_1$  states are substantially broadened above the first few vibrational levels. Fast predissociations through the  $\tilde{C}^2B_2$  state are proposed to account for the spectral diffuseness. A weak band at about  $14.6 \text{ eV}$ , which had been assigned as a configuration interaction (CI) satellite band, is found to be a HeI  $\beta$  line ( $537\text{\AA}$ ) spectrum of the third band, due to the resolved vibrational structure. A true CI band at about  $17.5 \text{ eV}$  with resolved vibrational structure is observed. It consists of two  $\nu_1$  vibrational progressions, which look like two spin-orbit split components.

#### H. Radiative Decay Lifetimes of $\text{CH}_2^-$ .

Recently the presence and radiative decay of vibrationally excited  $\text{CH}_2^-$ , generated in a hot cathode discharge of methane, was established by measuring the time dependent photodetachment from excited states of  $\text{CH}_2^-$  as it radiatively relaxed in a high vacuum ion trap. The time dependence of the photodetachment was found to be consistent with an electron affinity of  $5250 \text{ cm}^{-1}$  ( $0.65 \text{ eV}$ ) for ground state  $\tilde{X}^3B_1$  methylene. The radiative decay lifetimes of the first three excited bending vibrations of  $\text{CH}_2^-$  were also tentatively assigned. Here, we report a more refined analysis of the experimental data along with theoretical ab initio determinations of the radiative decay lifetimes of the first 4 excited bending vibrational levels of  $\text{CH}_2^-$ . There is some discrepancy between the ab initio values ( $431, 207, 118, \text{ and } 68$  milliseconds for the  $\nu_2 = 1, 2, 3, \text{ and } 4$  levels respectively) and the experimental values ( $525, 70, \text{ and } 14$  milliseconds - for  $\nu_2 = 1, 2, \text{ and } 3$  respectively) for  $\nu_2 = 2$  and  $3$ . Possible reasons for this discrepancy are discussed but none of the alternatives are entirely satisfactory.

## II. In Progress or Future Work

### A. Reaction of $\text{O}^{18}$ with Unsaturated Hydrocarbons.

Since the molecular beam studies of reactions of oxygen atoms with unsaturated hydrocarbons were initiated, many surprising results have been found. It has been clearly demonstrated that the substitution reactions where O replaces H,  $\text{CH}_3$  or halogen atoms are the major channels, and the 1,2 H atom migration followed by C-C bond rupture is not as important, although 1,2 migration of Cl atoms was seen.

These major findings are now well accepted among combustion chemists, but there are still some questions remaining about the relative importance of 1,2 H migration in the two reactions  $O + C_2H_2$  and  $O + C_2H_4$  and their energy dependence. Much of the difficulty in carrying out definitive studies of some of the minor reaction channels is due to the high background of CO in the ultrahigh vacuum mass spectrometric detector and the difficulty in distinguishing  $C^{13}H_3$  from  $O^{16}$ .

The use of  $O^{18}$  isotopes, in spite of the high cost, should alleviate all these difficulties and provide a more complete picture of these important combustion reactions. Experiments are currently being carried out on  $O + C_2H_4$  and  $O + C_2H_2$ , to determine the relative importance of H migration channels forming  $CH_3 + HCO$  and  $CH_2 + CO$ , compared to the substitution channels  $C_2H_3O + H$  and  $C_2HO + H$  at various collision energies. Preliminary results have been obtained.

#### B. Dynamics of Endothermic Substitution Reactions.

The promotion of endothermic reactions, by various forms of reagent excitation for atom-diatomic systems, depends critically on the nature of the potential energy surface. For systems with an early barrier, such as  $CH_3Br + I \rightarrow CH_3 + IBr$  and  $CF_3Br + I \rightarrow CF_3 + IBr$ , translational energy has been shown to be more effective than vibrational excitation in promoting chemical reactions.

For an endothermic substitution reaction, such as  $Br + C_2H_2Cl_2 \rightarrow C_2H_2BrCl + Cl$  or  $Br + C_2H_2Cl_2 \rightarrow C_2HBrCl_2 + H$ , the intermediate  $[C_2H_2Cl_2Br]^+$  is a stable radical species and extensive vibrational energy randomization is expected to take place. If the intramolecular relaxation is faster than the chemical reaction, a statistical theory can be used to predict the decay of the collision complex formed. One would immediately conclude that the decay back to the reactant  $Br + C_2H_2Cl_2$  is most exothermic and it should be the major channel, and the substitution reaction will not be important near the threshold, unless the dynamic aspects are more important than the statistical aspects. In other words, the endothermic substitution reactions will be extremely good systems to use to understand the intramolecular dynamics of chemically activated species, especially if non-statistical behavior is observed. We have obtained results on the reactions of Br atoms with 1,1 dichloroethylene and 1,2 dichloroethylene. These two molecules will provide a good comparison of the relative importance between the kinematic aspect of collisions and chemical effects.

#### C. Vibrational Spectroscopy of Hydrogen Bonded Cluster Ions.

In spite of the recent progress made in the high resolution IR spectroscopy of simple, stable molecular ions, there is essentially no information available on the vibrational spectroscopy of molecular cluster ions. Recently, there was a breakthrough in the laboratory in obtaining the spectra of  $H_3O(H_2O)_n^+$  using two different methods. The first method uses  $H_2$  as a messenger and first attaches a  $H_2$  to  $H_3O(H_2O)_n^+$  and then observes the absorption of IR photons by  $H_3O(H_2O)_n \cdot H_2$  through the



vibrational predissociation process,  $\text{H}_3\text{O}(\text{H}_2\text{O})_n^+ \cdot \text{H}_2 \xrightarrow{h\nu} \text{H}_3\text{O}(\text{H}_2\text{O})_n^+$ . The second method uses a high power  $\text{CO}_2$  laser to detect the absorption of a tunable photon by cluster ions by carrying out IR multiphoton dissociation of the excited cluster ions.

These methods will be further developed and will be used for the investigation of vibrational spectroscopy of various hydrated ions, and ammoniated ions,  $\text{NH}_4(\text{NH}_3)_n^+$  ( $n=1,2,3,4$ ), as well as carbonium ions, such as  $\text{C}_2\text{H}_5^+$  and  $\text{C}_2\text{H}_7^+$ .

The structure of these types of carbonium ions has been the source of considerable controversy. The classical structure has the odd hydrogen localized on a carbon. In the early 1950's, a symmetrical bridging structure was proposed. This structure soon gained widespread acceptance only to be challenged more recently. Vibrational and rotational spectra of these compounds will unambiguously resolve their structures.

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