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Publication Date

1993-03-01

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Lawrence Berkeley Laboratory

UNIVERSITY OF CALIFORNIA

CHEMICAL SCIENCES DIVISION

Presented at the 1993 Combustion Research Contractors'
Meeting, Lake Harmony, Pennsylvania, June 2-4, 1993,
and to be published in the Proceedings

Molecular Beam Studies of Reaction Dynamics

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March 1993



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1993 Combustion Research Contractors' Meeting
Split Rock Resort & Conference Center
Lake Harmony, Pennsylvania, June 2-4, 1993

LBL-33919 ABS
UC-401

MOLECULAR BEAM STUDIES OF REACTION DYNAMICS

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This work was supported by the Director, Office of Energy Research, Office of Basic Energy Sciences, Chemical Sciences Division, of the U.S. Department of Energy under Contract No. DE-AC03-76SF00098.

Molecular Beam Studies of Reaction Dynamics

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Scope of Project

The major thrust of this research project is to elucidate detailed dynamics of simple elementary reactions that are theoretically important and to unravel the mechanism of complex chemical reactions or photochemical processes that play important roles in many macroscopic processes. Molecular beams of reactants are used to study individual reactive encounters between molecules or to monitor photodissociation events in a collision-free environment. Most of the information is derived from measurement of the product fragment energy, angular, and state distributions. Recent activities are centered on the mechanisms of elementary chemical reactions involving oxygen atoms with unsaturated hydrocarbons, the dynamics of endothermic substitution reactions, the dependence of the chemical reactivity of electronically excited atoms on the alignment of excited orbitals, the primary photochemical processes of polyatomic molecules, intramolecular energy transfer of chemically activated and locally excited molecules, the energetics of free radicals that are important to combustion processes, the infrared-absorption spectra of carbonium ions and hydrated hydronium ions, and bond-selective photodissociation through electric excitation.

Current Research and Recent Results

A. Primary Dissociation Processes

1. IR spectroscopy of ionic complexes of CH_5^+ . Ionic complexes of CH_5^+ have been investigated using infrared spectroscopy based upon vibrational predissociation. We studied $\text{CH}_5^+(\text{H}_2)$ and $\text{CH}_5^+(\text{CH}_4)_n$ ($n = 1, 2, 3$) in the frequency region from 2650 - 4150 cm^{-1} with 0.2 cm^{-1} resolution. In the IR spectra of $\text{CH}_5^+(\text{H}_2)$, the vibrational bands of the CH_5^+ group have been observed for the first time. They appeared as one broad feature which may indicate the floppy nature of CH_5^+ . Also, the H-H stretching band of H_2 in $\text{CH}_5^+(\text{H}_2)$ appeared as a rotationally resolved feature with line splitting and two anomalously intense peaks. Now we continue to study these features using a higher resolution IR laser in order to get information on the structure and intramolecular dynamics of CH_5^+ as well as $\text{CH}_5^+(\text{H}_2)$. In the IR spectra of $\text{CH}_5^+(\text{CH}_4)_n$ ($n = 1, 2, 3$), a trend in the frequency shifts and changes in intensity of the C-H stretching bands was found as the size of complexes increases from $n = 1$ to 3. From the trend we were able to get information on the solvation structure and dynamics of CH_5^+ with CH_4 .

2. VUV Photochemistry of Small Molecules. Using a new high power VUV excimer laser operating at 157 nm, the photochemistry of CO_2 , SO_2 , SiH_4 , CH_3Cl , CH_3Br and CH_2BrCl was studied via the photofragmentation translational spectroscopy technique.

In CO_2 photolysis an interesting spin-forbidden process was observed, leading to $\text{CO} + \text{O}(^3\text{P})$ products. The electronic branching ratio $\text{O}(^3\text{P})/\text{O}(^1\text{D})$ was found to be 0.06. The vibrational branching ratio for the $\text{CO}(v) + \text{O}(^1\text{D})$ was found to be $[\text{CO}(v=0)]/[\text{CO}(v=1)] = 1.3$. In the photolysis of SO_2 , a channel leading to $\text{S} + \text{O}_2$ products was observed, as well as the expected $\text{SO} + \text{O}$ channel. The molecules CH_3X ($\text{X} = \text{Br}, \text{Cl}$) were shown to eliminate H, X and HX upon irradiation at 157nm. In addition, the molecule CH_2BrCl was found to eliminate molecular BrCl.

The photochemistry of SiH_4 is interesting and relevant to the microelectronics industry (i.e. laser chemical vapor deposition of silicon thin films). It was previously thought that SiH_4 decomposes through H atom elimination to form the SiH_3 radical. We have shown, however, that molecular H_2 elimination, forming the $\text{SiH}_2(^1\text{A}_1)$ diradical is a major channel, thus altering our view of silane photochemistry.

3. Photodissociation Dynamics of ClO₂. The photochemical decomposition of the symmetric chlorine dioxide radical (ClO₂) in the atmosphere is of potential importance in the balance of global ozone. However, there has been considerable uncertainty regarding the excited state dynamics of this molecule. Two chemically distinct photodissociation pathways are thermodynamically possible upon electronic excitation at wavelengths shorter than 496nm:



Although it has generally been believed that channel (1) dominates, there has been considerable controversy regarding the possible existence of channel (2) since it leads to catalytic decomposition of ozone in the atmosphere. Although a number of groups have attempted to determine Cl atom quantum yields and identify the electronic state(s) of the O₂ molecule, the results have been largely inconclusive. We have studied the dynamics of these processes using photofragment translational energy spectroscopy with a tunable excitation laser and have clearly observed both fragment partners for both channels.

Although the Cl + O₂ channel is relatively minor (<5%), we find that both electronic states of O₂ are formed in the dissociation process with comparable yields. The Cl + O₂ channel results from a concerted unimolecular decomposition with a large fraction of the excess energy channeled into relative translational motion.

The ClO₂ (A²A₂ ← X²B₁) absorption spectrum possesses a well defined progression primarily resulting from excitation to the (v₁,0,0), (v₁,1,0), (v₁,0,2), and (v₁,1,2) levels of the excited electronic state. It is thus possible to prepare the electronically excited molecule in various well defined vibrational levels. We observe a considerable degree of state specificity in the photodissociation dynamics. Excitation of the symmetric bending or symmetric stretching modes of OCIO (A²A₂) leads to Cl + O₂ with a quantum yield of several percent. However, excitation of an asymmetric stretching mode at nearly the same energy leads to <0.4% yield of Cl + O₂. Such mode specificity in branching ratios for chemically distinct products is extremely unusual.

B. Reaction Dynamics

1. Ozone Reactions with Br, Cl Atoms. BrO and ClO radical species play very important roles in the catalytic destruction cycles of ozone in stratosphere. To further understand the mechanisms of these two important reactions, we have carried out the crossed molecular beams studies on these two systems.

$\text{Cl} + \text{O}_3 \rightarrow \text{ClO} + \text{O}_2$ (ΔH° = -39.0 kcal/mole) has been studied at four different collision energies from 6 kcal/mole to 32 kcal/mole. The Cl atomic beam is generated by thermal dissociation of Cl₂ in a high temperature graphite nozzle source. The ClO product angular distribution and time-of-flight (TOF) distribution have been measured at each collision energy. In general, our results show that there is a large translational energy release in products and product ClO is scattered in a wide range of angles. With collision energy increased, ClO lab angular distributions peak more in the forward direction with respect to Cl atom. In the center-of-mass (CM) frame, the translational energy release is large and accounts for 40-60% of the total available energy. Furthermore, the translational energy release is coupled with the center-of-mass angle, the kinetic energy release at the small CM angle is larger than that at the large CM angle. With the increase of the collision energy, the fraction of the total energy channeled into translation is increased and the difference between the fast and slow kinetic energy releases becomes larger as well. The center-of-mass angular distribution is predominantly sideways peaked and moves to more forward direction with the increase of the collision energy. The reaction Cl + O₃ is a direct reaction and the Cl atom is likely to attack the ozone molecule in the coplanar approach. The varied approaches of the Cl atom toward the ozone molecule would lead to a wide range of scattering angles and also the different types of kinetic energy releases.

The semi-empirical calculation by Murrell and co-worker suggested that the ClO product would be mostly forward scattered with respect to the Cl atom when Cl approaches the ozone molecule in a collinear pathway. The translational energy release in the products was predicted to be ~ 50% of the total available energy. Our results

qualitatively agree those from the semi-empirical calculation, however, an *ab initio* calculation on Cl + O₃ system is going to be very helpful.

Br + O₃ → BrO + O₂ ($\Delta H^\circ = -30.8$ kcal/mole) has been investigated at five different collision energies from 5 kcal/mole to 26 kcal/mole. BrO product angular distribution and TOF distribution have been measured at each collision energy. The results from Br + O₃ reaction are very similar to those of Cl + O₃ reaction. There is again a large translational energy release in products peaking away from zero and that product BrO is scattered in a large range of angles. With collision energy increased, BrO lab angular distributions peak more forward with respect to Br atom. Preliminary analysis for this reaction at 18 kcal/mole collision energy shows that the product kinetic energy release is in the range of 35%-50% of the total available energy and it is also dependent on the CM scattering angle. The BrO center-of-mass angular distribution peaks at 65° in the CM frame. It seems that both Br + O₃ reaction and Cl + O₃ reaction are involved with very similar mechanisms.

Ozone reactions with I atom and NO molecule (I + O₃ → IO + O₂ and NO + O₃ → NO₂ + O₂) are also important in atmospheric chemistry and will be carried out accordingly.

2. D + H₂ → DH(v,J) + H Reactive Scattering. Over the past few years we have set up a new crossed molecular beam machine to study rotationally state-resolved differential cross sections for the hydrogen exchange reaction $D + H_2 \rightarrow DH(v,J) + H$. A beam of D atoms is generated by laser photolysis of DI and crossed with a pulsed molecular H₂ beam. DH reaction products are state-specifically ionized a few centimeters downstream from the crossing point using Doppler-free (2+1) Resonance-Enhanced Multi-Photon Ionization (REMPI), and imaged onto a position-sensitive detector. By varying the time delay between the D-atom generation and the DH detection, we can map out the angular distribution of a specific ro-vibrational DH product state.

In the past year we have observed the first state-resolved DH⁺ signal which depended on both the operation of the DI and H₂ pulsed jets as well as the operation of both the DI photolysis laser and the DH detection laser, as required for D + H₂ reactive signal. A surprising observation in the experiments has been the occurrence of abundant DH formation believed to be result from collisions of D atoms produced in the DI photolysis volume, with various metal surfaces in the experiment, such as the differential wall between the DI chamber and the H₂ chamber. This source of DH background was not observed in the earlier D + H₂ experiments carried out about five years ago in this group on one of the universal crossed molecular beam machines. Some progress has been made towards reducing this DH background.

The conditions under which the DI and H₂ pulsed molecular beams need to be operated have been tested in detail. For the DI pulsed jet we have determined the optimum pressure and timing conditions that ensure the generation of an intense D-atom beam with a narrow velocity and angular distribution. A range of different H₂ source configurations have allowed us to deplete up to 70% of the D atom beam intensity.

The success of our new D + H₂ experiment relies to a large extent on the detection sensitivity for DH molecules that can be achieved. Using Doppler-free (2+1) REMPI with a counterpropagating ultra-violet laser beam arrangement, we have achieved a detection sensitivity for molecular hydrogen better than 10⁴ molecules/cc/quantum state. With this detection sensitivity we anticipate differential cross section measurements with countrates of several ions per lasershot.

3. Cl Atom Reaction with NO₂ Molecule. The endothermic reaction $Cl + NO_2 \rightarrow ClO + NO$ ($\Delta H^\circ = 8.6$ kcal/mole) is the reaction to connect NO_x and ClO_x groups in atmospheric chemistry. Because ClNO₂ is a stable molecule, a collision long-lived complex is expected to form in this reaction. By the unique feature of the crossed molecular beam experiment, collision energies could be adjusted to probe the energy dependence of reaction probability in this endothermic reaction. Threshold region could be well studied by lowering collision energy.

We have studied this reaction at three different collision energies from 2 kcal/mole above the threshold to 15 kcal/mole above the threshold. The ClO product CM angular distributions have the forward-backward type symmetry which clearly confirms that Cl + NO₂ reaction proceeds through a long-lived complex. With the increase of the collision energy, the forward component in the CM angular distribution is also increased which might

collision energy, the forward component in the CM angular distribution is also increased which might demonstrate the transition from a long-lived complex mechanism to a direct reaction with the shortening of the complex lifetime.

Future Plans

A. Primary Dissociation Processes

1. Primary Dissociation of Hydrocarbons by IR Multiphoton Excitation. With the proposed development of a high power IR laser covering 2-5 μ , it will be possible to deposit a large amount of energy by multiphoton excitation through C-H or O-H stretching vibration. Primary dissociation of larger hydrocarbons containing 6-10 carbon atoms will be investigated. Of special interest will be the dissociation of various isomers.

2. Investigation of Energy Flow from High Frequency Modes to Low Frequency Modes in Unimolecular Decomposition. The relative efficiencies of energy flow among high frequency modes and low frequency modes can be examined if a molecule can be found which contains two weak chemical bonds of comparable bond dissociation energies and one of the dissociating bonds is coupled strongly to high frequency modes and the other to low frequency modes. $\text{CH}_3\text{CH}_2\text{OH}^+$ satisfies these conditions. This molecule has two dissociation channels forming $\text{CH}_3\text{CHOH}^+ + \text{H}$, and $\text{CH}_2\text{OH}^+ + \text{CH}_3$. These two channels of either H atom or CH_3 radical removal from the central C atom are competitive and require about 20 kcal/mol of energy. If O-H stretching vibration is excited by a direct overtone excitation beyond the dissociation energy level, the branching ratio measured as a function of excitation energy will reveal the nature of energy flow from the high frequency O-H stretching mode. If the energy is indeed first distributed among high frequency modes before flowing into low frequency modes, one would expect a C-H bond rupture to dominate, contrary to the results expected from a statistical theory.

Comparison of the results of this experiment with those of another experiment in which $\text{CH}_3\text{CH}_2\text{OH}^+$ is deposited with the same amount of internal excitation with different initial conditions will be very revealing. We intend to pursue this by selecting the internal energy of $\text{CH}_3\text{CH}_2\text{OH}^+$ by using the ion-electron coincidence technique.

3. H and H_2 Elimination from Hydrocarbon Free Radicals Excited by UV Photons. Understanding the energetics and decomposition pathways of hydrocarbon free radicals is crucial to describing combustion processes. Despite their importance, the dissociation of these species has not been studied extensively using molecular beams. Under the collisionless conditions of the molecular beam elucidation of the primary processes that are a result of the intrinsic dynamics of the dissociation is feasible. The development of molecular beam sources that can generate a high number density of these transient species should allow their detailed study using the technique of photofragment translational spectroscopy. The loss H and H_2 are the major dissociative pathways of simple hydrocarbon free radicals and, therefore, the recent modification of one crossed laser-molecular beam machine to allow detection of the H and H_2 photofragments is an important improvement. The advent of H and H_2 detection to our photodissociation apparatus should not only facilitate the study of these radicals but also allow unambiguous determination of the product branching ratios. Since H and H_2 elimination are the *only* energetically accessible channels, reinvestigation of methyl radical photodissociation at 193 nm is planned as the first system. Propargyl (C_3H_3) radical is one of the simplest conjugated free radicals and is postulated to be important in the formation of aromatic compounds, such as benzene, in flames. The photodissociation of propargyl radical is, therefore, important in understanding the properties of combustion intermediates. Ethyl radical and vinyl radical will also be investigated to determine their dissociative properties.

B. Reaction Dynamics

1. Reactions of CH_3 with Unsaturated Hydrocarbons. Radical addition to unsaturated bonds is the primary mechanism in most chain polymerizations and is also important in the formation of soot in combustion processes. The recent development of a novel molecular beam source, capable of generating an intense number density of methyl radicals, presents the possibility of studying the dynamics of reactions involving CH_3 and unsaturated hydrocarbons. Although the H atom addition to ethylene is more rapid than to acetylene the trend is reversed for CH_3 addition. This has been attributed to a larger pre-exponential factor for the $\text{CH}_3 + \text{C}_2\text{H}_2$ reaction, offsetting the

increase in the activation energy. Ab initio calculations agree qualitatively with these findings and suggest a late activation barrier to both of these reactions. Clearly these important reactions need to be examined under crossed molecular beams conditions to experimentally determine the thermodynamics and activation barriers.

2. Reaction of O(¹D) with Methane and Ethane. The dissociation of O₃ in the throat of a pulse molecular beam source using He as a carried gas is an excellent way to produce an intense pulsed O(¹D) beam. The reactions of O(¹D) with methane and ethane will form highly vibrationally excited methanol and ethanol as reaction intermediates. These intermediates will dissociate by eliminating OH, H, H₂ or H₂O. The highly vibrationally excited methanol and ethanol are also reaction intermediates of the reactions of CH₃ and C₂H₅ with OH. Radical-radical reactions are extremely difficult to pursue in a crossed molecular beams experiment. However, in these cases, the same reaction intermediates can be prepared by the insertion of O(¹D) into C-H bond. When a high power IR laser becomes available it is also possible to investigate the dissociation of methanol and ethanols by the IRMPD approach.

3. Heterogeneous Reaction of Atoms with Solid Surface and Chemisorbed Molecules. In the scattering of H atom with LiF, surprisingly, the formation of HF products were observed. With our new beam surface apparatus, we intend to carry our systematic investigation of reactions of solids with gaseous atoms and radicals. Three types of reactions will be pursued: (1) Reactions of Cl, O and H with graphite; (2) reactions of chemisorbed C₂H₄ on Pt with various atoms; and (3) reaction and decomposition of CH₄ on a metal oxide surface with or without continuous exposure to a stream of O₂ for the understanding of catalytic oxidation of CH₄.

4. Pulse Pyrolysis of Organometallic Transition Metals for Crossed Molecular Beam Studies of Transition Metal Atoms. Because of extremely low vapor pressures, the production of an atomic beam of transition metal from the vapor is extremely difficult. The laser ablation was often used for the production of cold transition metal clusters. However, the intensity is rather limited for carrying out a crossed molecular beams experiment.

A possible alternative way of producing an intensive transition metal atom beam is by the pyrolysis of organometallic compounds during the pulsed supersonic expansion. The heated tube for pulsed beam expansion has to be of sufficient temperature to induce complete dissociation, and even if some of the transition metal atoms are condensed on the heater inner surface during the expansion, it should re-evaporate during the off cycle. A pulsed beam source capable of operating at 3000°C will be needed for this purpose.

Acknowledgement

This work was supported by the Director, Office of Energy Research, Office of Basic Energy Sciences, Chemical Sciences Division, of the U.S. Department of Energy under Contract No. DE-AC03-76SF00098.

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