Dissociative Recombination Chemistry and Plasma Dynamics

Final Technical Report - AFOSR Grant #FA9550-04-1-0035


June 16, 2008

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Advances made in the study of the dissociation dynamics and product branching ratios of energized transient neutral molecules formed by charge-transfer (DCE) and recombination of cations with free electrons (DR) are reviewed. The results obtained include detailed measurements of the three-body dissociation of H3, O4 and sym-triazine (HCN)3, as well as the product branching ratios for the two-body dissociation of CH5+ and H3O+. These results have significance for a number of Air Force applications, ons including ion-assisted ignition and combustion for hypersonic vehicle applications. This project aimed to extend our knowledge of the fate of transient neutrals produced in plasma environments while providing fundamental new information on the dynamics of two- and three-body dissociation processes. As noted below, significant successes in the study of DCE processes were achieved, while the efforts at carrying out DR measurements on the fast-ion-beam apparatus were not successful.
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1. Objectives

The objective of this research program was to study the dissociation dynamics and product branching ratios of energized transient neutral molecules formed by charge-transfer and recombination of cations with free electrons. The dynamics and product branching ratios for elementary dissociative-charge-exchange (DCE) and electron-ion dissociative recombination (DR) processes are relevant to a number of Air Force applications including ion-assisted ignition and combustion for hypersonic vehicle applications. DCE and DR processes play an important role in determining the fate of polyatomic ions in weakly ionized plasmas, and while cross-sections for DR reactions are known for a number of systems, only recently for small polyatomic ions have product branching ratios become available. This project aimed to extend our knowledge of the fate of transient neutrals produced in plasma environments while providing fundamental new information on the dynamics of two- and three-body dissociation processes. As noted below, significant successes in the study of DCE processes were achieved, while the efforts at carrying out DR measurements on the fast-ion-beam apparatus were not successful.

2. Statement of Work

This project aimed to study the dissociation dynamics and product branching ratios of transient neutral species produced by the neutralization of cations by charge exchange and recombination with free electrons. The focus was on species present in atmospheric and combustion plasmas, with an extension to the study of energetic material precursors like the carbon-nitrogen heterocycle sym-triazine (C$_3$H$_3$N$_3$). The experiments were carried out on a unique fast-beam multiparticle translational spectrometer. In the dissociative charge exchange measurements, a pulsed configuration with time-of-flight mass selection was used to interact ions of a specific mass with a Cs collision cell. In the dissociative recombination measurements, a continuous beam with a Wien mass filter was used to allow merging of low energy (eV) electron beams with high energy (keV) ion beams, for the purpose of studying DR processes under single-collision conditions. The merging was carried out in a specially designed trochoidal electron monochromator. Problems with electron beam current, ion-electron beam overlap and large background-gas derived signals were the primary impediments that were insurmountable given the resource and time constraints of the research project. The successful charge exchange experiments have provided important information on the energetics, dissociation dynamics and product branching ratios for the systems studied. In particular, unique insights into three-body dissociation processes in H$_3$ and sym-triazine were gained, along with a unique probe of the structure of CH$_5$$^+$ by measurement of the product branching ratios.

3. Accomplishments/ Findings

This research project resulted in a number of important advances in our understanding of the dissociation dynamics, energetics and product branching ratios for a number of dissociative charge exchange processes. In the sections that follow, the results obtained on H$_3$ and its isotopologs, O$_4$, CH$_5$ and its isotopologs and finally sym-triazine will be presented. The results on H$_3$ and sym-triazine in particular constitute benchmark studies of three body dissociation processes at an unprecedented level of detail. The DCE of CH$_5$$^+$ provides a further test of the fluxional structure of the non-classical carbonium ion, and the studies of O$_4$ provide new insights.
into the excited electronic states of these atmospherically relevant species. At the end of this section, the efforts to develop a next-generation merged-beam DR spectrometer will be reviewed.

3.A. Dynamics of Dissociative Charge Exchange

Studies of the DCE of polyatomic cations are relevant to plasma chemistry as they produce highly excited neutral species. The studies in this project have made use of charge exchange with Cs, which yields excited species 3.9 eV below the ionization potential of the target neutral as a result of ionizing the electron donor Cs atom. Using the fast 6-16 keV beams available on this apparatus, however, non-resonant charge-exchange processes can produce species nearly at the ionization potential of the neutral target for a small molecular cation like H$_3^+$. Our efforts complement and extend earlier studies of DCE$^{3,4,5,6,7,8}$ through the use of vibrationally cold cations produced in a supersonic expansion and a state-of-the-art multiparticle detector,$^9$ permitting studies of two- and three-body dissociative charge exchange processes.

In these experiments, a pulsed, fast (6 - 16 keV) mass-selected cation beam was passed through a 1-mm long charge-exchange cell with a pressure of $\approx 10^{-4}$ torr Cs. The neutral species produced by DCE recoiled out of the beam and were recorded by a multiparticle time- and position-sensitive detector. Given the parent ion mass and velocity, information from the multiparticle detector yielded a complete kinematic characterization of the dissociation. This experimental approach for studying the dissociation dynamics was identical to that used in our previous studies of dissociative photodetachment processes.$^{10}$ The time- and position-of-arrival of the products was recorded, yielding the product momenta and product energy and angular distributions in the center-of-mass frame. In the case of two-body dissociation, product kinetic energy and branching ratios are recorded. In the more exotic case of three-body dissociation, however, the details of product momentum partitioning can be examined. A convenient way to examine the three body dissociation dynamics makes use of a Dalitz plot that reveals directly how momentum is partitioned among the three products,$^{11}$ and two examples of Dalitz plots are given in the subsequent sections on the three-body dissociation dynamics of the low-lying Rydberg states of H$_3^+$, D$_3^+$, the H$_2$D$^+$ and HD$_2^+$. Figure 1 below in particular shows the Dalitz plot for a system of three equal mass products, as found in H$_3$ and D$_3$. The results of studies of the two- and three-body dissociation of O$_4^+$ are then reviewed, followed by the studies of CH$_5^+$ and the three-body dissociation of sym-triazine.

3.A.1. Three-Body Dissociation Dynamics of the Low-Lying Rydberg States of H$_3$ and D$_3$

The three-body dissociation dynamics of the low-lying Rydberg states of H$_3$ and D$_3$ have been studied by dissociative charge exchange with Cs, as reported in AFOSR publications 1 and 3 and Ph.D. thesis, publication 2. Capture of free electrons at or near the H$_3$ ionization threshold leads to both two and three-body DR processes, involving a number of the Rydberg states of H$_3$ with geometries near the $D_{3h}$ ground state of H$_3^+$. The lone dissociative state in this region, the ground $2p \ ^2E'$ state, provides the primary route to dissociation, however, non-adiabatic transitions from high-lying Rydberg states to the lower-lying Rydberg states are thought to play an important role in DR as they are most efficiently predissociated by the $2p \ ^2E'$ state. This degenerate dissociative state undergoes a Jahn-Teller distortion into upper and lower repulsive sheets as the nuclei are distorted from $C_{3v}$ symmetry. These sheets correspond to the three- and
two-body dissociation limits respectively. The coupling between the Rydberg states and the upper sheet of the $2p^2E'$ dissociative state governs three-body dissociation.

The low Rydberg states of $H_3$ can be conveniently prepared by charge-exchange of $H_3^+$ with Cs. Production of the cations in a supersonic expansion yields a rotationally and vibrationally cold source of $H_3^+$ and the isotopologs. The three-body dissociation kinetic energy release spectrum obtained for $H_3^+$ and $D_3^+$ at a 12 keV beam energy is shown in Figure 2. The $2s^2A_1'$, $2p^2A_2''$ states are nearly resonant for charge exchange with Cs, but as the large signals observed at high kinetic energy release (KER) for $H_3$ shows, states several eV off resonance can also be conveniently prepared, essentially all the way to the ionization potential. This effect is significantly reduced for $D_3$, as shown in the lower frame of Figure 2.

The three lowest lying Rydberg states are resolved in Figure 2, allowing a detailed examination of the three-body dissociation dynamics by examination of the product momentum partitioning. The Dalitz plots for the $2s^2A_1'$, $2p^2A_2''$ and $3p^2E'$ states of $H_3$ (frames A, C and E) and the $2s^2A_1'$ and $2p^2A_2''$ states of $D_3$ (frames B and D) are shown in Figure 3. In addition, in frame F is shown the detector acceptance function for the KER = 2.7 eV $3p^2E'$ state of $H_3$. Since the detector acceptance is nearly unity even at this KER, the data reported here is raw data.

The dominant momentum partitioning among the three $H$ atoms, as indicated by the high-intensity regions of the Dalitz plots, provides a measure of the nuclear configuration at the point of dissociation for the $2s^2A_1'$, $2p^2A_2''$ and $3p^2E'$ states for $H_3$. The data for $H_3$ reveals that the $2s^2A_1'$ state undergoes three-body dissociation by a $C_{2v}$ distortion towards a linear configuration. The $2p^2A_2''$ state rotationally couples to the dissociative ground state largely preserving the initial symmetric configuration, and thus leading to nearly equal momenta among the products. The most complex dynamics are observed from the $3p^2E'$ state where totally symmetric and asymmetric features are observed with almost equal probability. This may be interpreted as capturing the Jahn-Teller distortion the nascent $3p^2E'$ state undergoes. $D_3$ exhibits subtle dynamical differences compared to $H_3$ as a result of the difference in the zero-point energy. For example, the distortions observed for both the $2s^2A_1'$ and $2p^2A_2''$ states for $D_3$ show a higher probability for dissociation from more distorted configurations compared to $H_3$. This data provides valuable information for the development and evaluation of both the potential energy surfaces and dissociation dynamics of the fundamental $H_3$ system.
3.A.2. Three-Body Dissociation Dynamics of the Low-Lying Rydberg States of HD$_2$ and H$_2$D

Studies of the isotopologs H$_2$D$^+$ and HD$_2$$^+$ were also carried out, as discussed in Ph.D. Thesis, AFOSR publication 2. DCE of these isotopologs occurs through excitation of the same series of Rydberg states, however, the symmetry of the system is reduced from D$_{3h}$ to C$_{2v}$ by the isotopic substitution. The mechanism in the asymmetric isotopologs is expected to be similar, however, the overall symmetry is reduced.

In the work carried out to date on the asymmetric isotopologs, the three-lowest Rydberg states of HD$_2$ and D$_2$H were prepared by charge-exchange of the corresponding cations with Cs. Translational spectroscopy measurements of both the two- and three-body DCE products were then performed. Well-resolved three-body kinetic energy release spectra were obtained as shown in Figure 4, allowing the three-body dissociation dynamics of the three lowest Rydberg states to be examined directly. The 2$s$ $^2A_1'$, 2$p$ $^2A_2''$ states are nearly resonant for charge exchange with Cs, but as the large signals observed at high kinetic energy release (KER) for H$_3$ shows, states several eV off resonance can also be conveniently prepared. This effect is progressively reduced for the heavier isotopologs going from H$_3$ to D$_3$, as seen in Figure 4.

Once again, the three lowest lying Rydberg states are resolved, allowing a detailed examination of the three-body dissociation dynamics using the Dalitz plot formalism. The Dalitz plots for asymmetric systems, with two out of three identical particles, have a different structure as shown in Figure 5. A comparison of the results obtained for H$_2$D and HD$_2$ with the previous study of H$_3$ and D$_3$ is given for the 2$s$ Rydberg state in Figure 6. The data for H$_3$ and D$_3$ clearly shows that the 2$s$ $^2A_1'$ state undergoes three-body dissociation by a C$_{2v}$ distortion towards a linear configuration. In the case of the isotopologs H$_2$D and HD$_2$, the Dalitz plots are not as easy to interpret. The intensity in the plots is centered around the expected points of

![Figure 3](image-url) - Dalitz plots showing the momentum partitioning for the low-lying Rydberg states of H$_3$ (A,C,E), D$_3$(B,D), and the detector acceptance at KER=2.7 eV (see text)

![Figure 4](image-url) - The center-of-mass kinetic energy release for the three-body dissociation of each isotopolog at 12 keV beam energy. The peak labels are states assignments, note that D$_3$/H$_3$ have the same state labels as do HD$_2$/H$_2$D,
of the fragment momentum partitioning in the three-body dissociation of the lowest-lying, 2s, Rydberg state of (A.) H₃, (B.) D₃, (C.) HD₂, and (D.) H₂D. The small circles on the plot represent the 1% probability level for the zero point wavefunction along the degenerate bending normal coordinates. 

**Figure 5.** Dalitz plot illustrating the correlation between the partitioning of the fractional square of product momentum with product momentum vectors. Qₓ and Qᵧ denote the degenerate bend normal modes for C₂ᵥ symmetry for the Qₓ, Qᵧ and symmetric stretch motions, which vary with isotopic substitution as shown in Figure 1. A detailed understanding of these results, however, will require further theoretical analysis. Presently, the primary observation that can be made from the Dalitz plots is that in both isotopologs the heavy (D atom) products in general receive a greater partitioning of energy than the light product. This may have important implications for isotopic fractionation processes in plasmas and the interstellar medium.

3.A.3. Two- and Three-Body Dissociative Charge Exchange of O₄⁺ with Cs

Measurements of the two- and three-body dissociation dynamics of the DCE of O₄⁺ with Cs have also been carried out, as reported in the Ph.D. thesis, AFOSR publication 2. The results are consistent with the formation of O₄⁺ in both charge-localized O₂⁻O₂⁺ and covalent O₄⁺ structures. The work done on this system yields the first study of the three-body dissociation of O₄⁺, providing information on the state-specific gas-phase solvation energetics for the charge-localized structure containing 3sσ and 3pλ Rydberg states of O₂ with ground state O₂(X ³Σ_g⁻). The ozone production channel O₄⁺ → O₃ + O was not observed, nor was the production of stable O₄.

Figure 7 shows the KER spectrum obtained for the two-body dissociation of O₄ to O₂ + O₂ at several different O₄⁺ beam energies. The major feature with a KER near 1 eV is consistent with production of O₂(B ³Σ_u⁻) + O₂(X ³Σ_g⁻) and provides impetus for theoretical studies on the PES for O₄⁺ in the configurations of possible cation geometries. The high energy peaks near 6 and 7 eV in the 12 keV spectrum indicate a dissociation process imparting translational energy well above that required to dissociate a single O₂ molecule, consistent with a strongly bound covalent cation precursor. Neutralization of a covalent precursor is the most plausible route to producing the nascent O₄⁺ in a highly repulsive region of the neutral potential energy surface. The narrowness of the peaks suggest dissociation from a highly symmetric geometry that is possibly the quartet Dᵥ₄ structure predicted by theoretical studies on the O₄⁺ cation.¹⁶,¹⁷
Perhaps the most interesting aspects of the $O_4^+$ DCE study are observed when a correlation between the 12 keV $O_4^*$ KER spectrum of the three-body dissociation channel ($O_4^* \rightarrow O_2 + O + O$) and the fractional square of the momentum imparted to the surviving $O_2$ fragment is made (Figure 8). Two main features are observed in this plot—a progression of vertical features and another consisting of diagonal features. The vertical features are characteristic of a charge localized $O_2-O_2^*$ complex, as they imply that the KER of the fragments are independent of the momentum partition for the dissociating $O_2$ molecule. The vertical progression is consistent with charge exchange to a $O_2\cdots O_2(3s\sigma)$ complex followed by dissociation to $O_2(X^3\Sigma^{-}\,\nu=0-3) + O(1D) + O(3P)$ products. The diagonal features in the correlation plot show a dependence of the KER on $O_2$ fragment momentum partitioning, suggestive of a covalently bound $O_4$ precursor, although the exact origin of the features has yet to be understood. These results provide significant new empirical data on the dynamics of the $O_4$ system and motivation for excited state calculations of $O_4^*$ near cation geometries.

3.A.4. Dissociative Charge Exchange of $CH_5^+$ and Isotopologs

Fuel-derived hydrocarbon cations were a focus of interest in this research, and beyond preliminary studies of $C_3H_7^+$, the most successful pure hydrocarbon cation experiment examined the DCE of $CH_5^+$ and the product branching ratio between $H + CH_4$ vs. $H_2 + CH_3$. In addition to this fundamental information, it was found that the study of the DCE of $CH_5^+$ with Cs, coupled with quasiclassical trajectory calculations on an $ab$ in $itio$ potential energy surface for $CH_5$, provides a novel probe for the structure of the non-classical $CH_5^+$ cation. The theoretical studies on this system were carried out by Joel Bowman and co-workers at Emory University, and provide results that are directly comparable to the experimental branching ratios into $CH_4 + H$ and $CH_3 + H_2$ products and
the product kinetic energy release distributions. Experimentally, it is found that the CH₄ + H channel is favored by 11:1. The theoretical branching ratios were found to be 14:1 for fluxional CH₅⁺, and 34:1 when CH₅⁺ was taken to be a semi-rigid molecule. These results are consistent with a fluxional CH₅⁺ ground state as reported in AFOSR publication 5. Further studies of the branching ratios for deuterated CH₅⁺ isotopologs have also been carried out and will be reported in a future collaborative publication.

3.A.5. Three-Body Dissociation of Sym-Triazine

Studies of the two- and three-body DCE of the sym-triazine cation have also been carried out. The three-body dissociation dynamics have received the most attention so far, owing to the fact that the symmetric concerted three-body dissociation process was directly observed for the first time. These studies have allowed us to probe the dissociation dynamics of the highly symmetric (D₃h) aromatic C-N heterocycle (C₃N₃H₃), with its complex electronic structure. In these experiments, the three-body dissociation of sym-triazine to three HCN products was investigated by translational spectroscopy and high-level ab initio calculations. Dissociation was induced by charge exchange between the sym-triazine radical cation and cesium at beam energies of 12 and 16 keV, and a symmetric three-body dissociation channel was clearly observed by a non-resonant charge exchange process at the higher incident beam energy. Krylov and co-workers at USC have calculated the excited-state energies and electronic couplings, indicating that sym-triazine is produced in the 3s Rydberg and π*←n manifolds. Analysis of the topology of these manifolds along with momentum correlation in the dissociation products suggest that the 3s Rydberg manifold is characterized by a conical intersection of two potential energy surfaces and leads to stepwise dissociation, while the π*←n manifold consists of a four-fold glancing intersection that leads to a symmetric concerted reaction. This work is currently in final review in the journal Science, and acceptance is anticipated. A subsequent full account of the study is also in preparation.


A major focus of this research project was the construction of a new generation merged beam apparatus for studying dissociative recombination of free electrons with molecular cations. This effort did not succeed, however, primarily as a result of insufficient electron beam current, insufficiently characterized ion-electron beam overlap and large background-gas derived signals. These problems were found to be insurmountable given the resource and time constraints of the research project. A brief review of the effort is worth documenting further, however. A trochoidal electron monochromator was constructed and characterized, allowing the merging of a low energy electron beam (1 – 3 eV lab energies) with a 16 keV cation beam. To increase the duty cycle of the experiment, the cation beam was converted to a continuous beam, using a Wien filter for mass selection. Detection scheme modifications that were necessary with the continuous ion beam were completed and verified by studies of the DCE of O₂⁺ with Cs. As the effort continued, significant effort was expended to reduce the ultimate vacuum in the interaction region, with pressures as low as 2 x 10⁻¹⁰ torr achieved. However, the signal from collisions with background gas were still too large.
In the continuous beam mode of operation, the apparatus achieved beam currents of 3 – 5 nA currents for species like $\text{H}_3^+$ and $\text{O}_2^+$. The trochoidal monochromator implemented for the production and merging of energy-selected electron beams with the cation beam was constructed and characterized, achieving electron beam currents in excess of 100 nA at a nominal laboratory energy of 2.2 eV. This electron energy was used in extensive experiments on a 12 keV beam of $\text{H}_3^+$, where merging with 2.2 eV laboratory energy electrons reaches a relative collision energy in the center-of-mass frame near 0 eV, where the DR cross section is highest. Later experiments were also carried out at 16 keV with electrons of 3 eV lab energy. To the best of our knowledge, this continuous electron beam was overlapped with the continuous ion beam, but in fact we had no diagnostic for this beyond the current transmitted through sequential apertures. In the end, in spite of signal levels estimated to be several hundred events per hour, we were unable to successfully recover signals from the electron beam interacting with the ion beam above the random background noise.

Another approach to producing energized neutrals near the ionization potential is to return to a Cs charge-exchange cell and use resonant excitation of Cs from the ground 6s state to higher-lying levels using the Ti:Sapphire laser coupled with an optical parametric amplifier available in the laboratory. Cosby and co-workers carried out experiments using this technique in the mid-1990’s. This approach allows tuning the level of excitation created by resonant charge exchange, asymptotically approaching excitation equivalent to the capture of a zero energy electron by a cation. If further charge exchange measurements are carried out in the future, this will be an important technique to apply.

4. Personnel Associated with the Research Effort

Over the period of this research grant, in addition to the P.I. three graduate students received full or partial support of their research efforts. The graduate students include Dr. Christopher M. Laperle, who received his Ph.D. during the first year of this grant and Jennifer Mann, with an expected Ph.D. completion date of September 2008 and John Savee, with an expected Ph.D. completion date of January 2009. The P.I. has obtained institutional support to continue supporting these two students through the end of their Ph.D. studies. Dr. Laperle recently accepted a tenure-track Assistant Professor position at Providence College in Providence, RI, an undergraduate institution.

5. Publications

Over the course of this grant, the following publications have been published or submitted with AFOSR support.


6. Interactions/Transitions

6.a. Meetings

The P.I. gave a number of invited talks on this research program, as detailed here:

National and International Meetings:


Invited talks at various institutions:
78. "Dissociative charge exchange dynamics.", Physical Chemistry Seminar, Department of Chemistry, Texas A&M University, April 8, 2004.
79. "Dissociative charge exchange dynamics.", Physical Chemistry Seminar, Department of Chemistry, Rice University, April 9, 2004.


88. "Three-body dissociation dynamics of the low-lying Rydberg states of H₃.", Physical Chemistry Seminar, Department of Chemistry, University of Southern California, Los Angeles, CA April 18, 2005.
93. "Three-body dissociation dynamics of the low-lying Rydberg states of H₃.", Dept. of Chemistry and Biochemistry, University of California, Los Angeles, October 31, 2005.

The PI's group members also presented the following poster presentations on this research project:


In addition to these presentation at meetings, the PI was also the organizer of two scientific meetings that received AFOSR support during this research project:

Chair, Gordon Conf. on Photoions, Photoionization and Photodetachment, Buellton, CA Jan. 29 – Feb. 3, 2006

Chair, 29th International Symposium on Free Radicals, Big Sky, MT Aug. 12-17, 2007
6.b. Consultative/Advisory Functions

During the period of this grant, the PI attended the AFOSR Molecular Dynamics Contractor's meetings in 2004, 2005 and 2007, engaging in productive discussions with AFRL and researchers from other universities engaged in AFOSR sponsored research. In addition, the PI engaged in a number of discussions with AFRL researchers and contractors including Dr. Skip Williams, Dr. Rainer Dressler, Dr. Albert Viggiano, Dr. Yu-hui Chu, Dr. Thomas Miller, Dr. Anthony Midey and Dr. Dale Levandier. These discussions focused on the chemistry of oxygen cluster ions, charge exchange and dissociative recombination processes.

6.c. Transitions

There have been no significant transitions during this period.

7. New Discoveries, Inventions or Patent Disclosures

Outside of the results reported in the accomplishments/new findings section above, there is nothing further to report here.

8. Honors/Awards

In the period of this grant, the principal investigator, the P.I. Prof. Robert E. Continetti was named the Kurt Shuler Scholar in Physical Chemistry at the University of California, San Diego. He was also appointed to serve as Chair of the Department 2006-2009.

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