REPORT DOCUMENTATION PAGE		Form Approved OMB No. 0704-0188

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1. REPORT DATE (DD-MM-YYYY)	2. REPORT TYPE	3. DATES COVERED (From - To)
June 2006	Final	1-Apr-2002 to 30-Nov-2005

4. TITLE AND SUBTITLE	5a. CONTRACT NUMBER
Mechanism and Dynamics of Explosive Combustion in Aerosol Fuels	
	5b. GRANT NUMBER
	F49620-02-1-0158
	5c. PROGRAM ELEMENT NUMBER

6. AUTHOR(S)	5d. PROJECT NUMBER
Tomas Baer	
Roger E. Miller	5e. TASK NUMBER
	5f. WORK UNIT NUMBER

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)	8. PERFORMING ORGANIZATION REPORT NUMBER
Department of Chemistry University of North Carolina Chapel Hill, NC 27599-3290	

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)

AFOSR/NL
875 N. Randolph Street
Arlington, VA 22203

AFRL-SR-AR-TR-06-0224

Dr Michael Berman

12. DISTRIBUTION/AVAILABILITY STATEMENT

Approved for Public Release: Distribution Unlimited

13. SUPPLEMENTARY NOTES

14. ABSTRACT

The research involves the production of high energy material particles with diameters between 1 and 5 •m and the study of its subsequent combustion by rapid laser heating or photolysis. Alternatively, the high-energy ionic liquids with no vapor pressure are placed on a plate inside the vacuum and irradiated with high intensity IR radiation. In either case, the reaction products are followed by single photon vacuum UV laser ionization in a time of flight (TOF) mass spectrometer. The purpose is the elucidation of reaction mechanisms in high density or condensed phases and the development of more efficient methods of combustion. The experimental set-up includes aerosol generation tools that inject the particles at atmospheric pressure into an aerodynamic lens, which focuses the particles onto the center of a 3 mm expansion nozzle, whereupon the particles are accelerated to velocities of 200 to 300 m/s depending upon their size.

15. SUBJECT TERMS

16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT	18. NUMBER OF PAGES	19a. NAME OF RESPONSIBLE PERSON
a. REPORT	b. ABSTRACT	c. THIS PAGE			
				12	19b. TELEPHONE NUMBER (Include area code)

Standard Form 298 (Rev. 8/98)
Prescribed by ANSI Std. Z39.18

The Mechanism and Dynamics of Explosive Combustion in Aerosol Fuels

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Project Description

The research involves the production of high energy material particles with diameters between 1 and 5 μm and the study of its subsequent combustion by rapid laser heating or photolysis. Alternatively, the high-energy ionic liquids with no vapor pressure are placed on a plate inside the vacuum and irradiated with high intensity IR radiation. In either case, the reaction products are followed by single photon vacuum UV laser ionization in a time of flight (TOF) mass spectrometer. The purpose is the elucidation of reaction mechanisms in high density or condensed phases and the development of more efficient methods of combustion. The experimental set-up includes aerosol generation tools that inject the particles at atmospheric pressure into an aerodynamic lens, which focuses the particles onto the center of a 3 mm expansion nozzle, whereupon the particles are accelerated to velocities of 200 to 300 m/s depending upon their size. The particles are sized by laser light scattering, ignited by a high power CO_2 laser pulse, and the products ionized by the 10.48 eV light produced by third harmonic generation of 356 nm light in a Xe cell or by tunable vacuum UV radiation prepared by 4-wave mixing in a Kr cell. Major findings include the following: Ion-ion recombination is very rapid in the laser ablation of ionic materials such as high concentration salt solutions. The modeling of the data indicate that the reaction is complete within 10 ns. When ionic liquids are ablated with a high intensity IR laser pulse more than 99% of the liquid is ejected into the vacuum as nano-droplets of inteact ionic liquids.

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Introduction

Liquid or solid state combustion and detonation processes have been the subject of considerable experimental(1-16) and theoretical(17-25) efforts. Yet, experimental methods for obtaining a detailed characterization of reaction intermediates and products in the condensed phase are limited and much less precise than in for instance the gas phase. Indeed, the need for novel approaches for studying liquid state combustion is made clear from the literature in this field. As Brill and James(26) have pointed out, "the most important reactions arise when the molecules are in the condensed phase in the presence of like molecules." However, rates and mechanisms of condensed phase combustion are extremely difficult to determine in part because no single mechanism is maintained during the course of the reaction, and simultaneous multiple mechanisms may well be the rule. Among the issues is the balance between ionic and radical reaction pathways as in the case of the base catalyzed reaction of nitromethane.(10,27-30) Ionic channels are essential in the early initiation stages, but become less important as the free radical channels take over.

The identification of intermediates in any chemical reaction is challenging because only a few tools are available for conclusive product identification. High resolution IR in the gas phase is excellent in distinguishing molecular species, but is not very sensitive.(31,32) In fact, any spectroscopic method is very limited in identifying chemical species in the condensed phase because of the broad bands associated with each chromophore.(33) On the other hand, mass spectrometry is extraordinarily sensitive to species in the gas phase, and has thus been used to identify reaction products ejected into the vacuum.(4,10,30,34-36)

One of the difficulties in the study of combustion reactions is control of the experimental conditions, in particular the temperature. Because energetic reactions are by their very nature highly exothermic, the temperature of an uncontrolled reaction will increase during the course of reaction. Under these conditions, determination of an activation energy is indeed difficult. On the other hand, imposing isothermal conditions may result in a modified mechanism for the reaction. For instance, at low energies, trinitrotoluene (TNT) reacts by oxidation of the methyl moiety, CH_3, while $C-NO_2$ bond homolysis appears to be dominant at higher temperatures.(37) Wight and co-workers have addressed the issue of temperature control and have suggested a means to relate isothermal or non-isothermal conditions.(38,39) Our own work, to be summarized, has focused on measuring the temperature during the combustion process by monitoring the cracking pattern of a "thermometer" molecule.(40)

Finally, considerable discussion has emerged concerning the feasibility of using ionic liquids as combustion fuels. Ionic liquids have interesting properties such a very low vapor pressure and good solvation power. They have been proposed as solvents for synthesis and catalytic reactions.(41,42) Very recent theoretical work has centered on the structure and short time dynamics of these liquids.(43) We have begun our own study of these liquids using the methods to be described below.

The Experimental Approach

As shown in Figure 1, aerosols consisting of energetic materials are generated by a nebulizer or a variable orifice aerosol generator (VOAG) and injected into the aerodynamic lens(44-46) through a 100 μm, flow-limiting, orifice. The particles then travel through the lens suspended by the gas pressure of about 2 Torr. They are injected into the vacuum through a 3 mm nozzle, where they are accelerated to a velocity of 60-200 m/s, depending on their size. Because of the large number of collisions in the expansion and because the lens focuses the
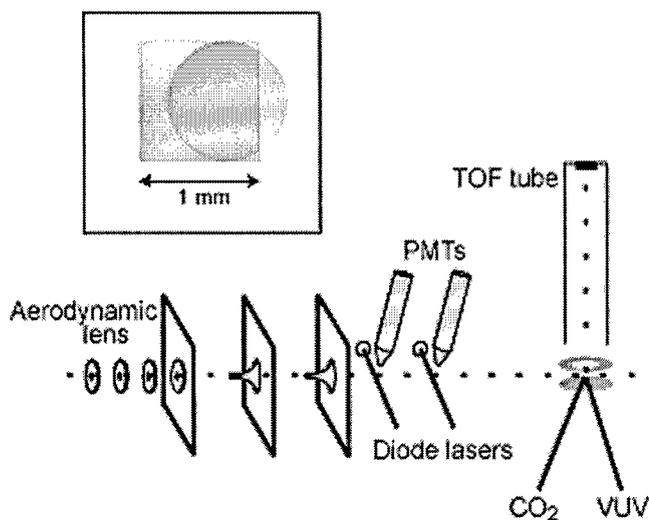
Figure 1: The experimental set-up in which aerosol particles are injected into the vacuum through a supersonic expansion. The particles velocity is determined by light scattering from the diode lasers. The CO_2 laser pulse initiates the combustion process, and the products are ionized in a time resolved fashion by single photon ionization with a vacuum ultraviolet (VUV) laser. The time of flight mass spectrum records all the masses from a single particle

particles precisely in the middle of the nozzle, the aerosol particles travel with little divergence down the axis of the vacuum chamber. After passing two skimmers, the particles intersect two diode laser beams and scatter light to the two photomultipliers. This signal is used to detect the presence of a particle as well as to determine its velocity so that we can fire the pulsed infrared (IR) and vacuum ultraviolet (VUV) lasers when the particle reaches the TOF spectrometer. Up to here, the instrument is rather standard, and is similar to those used by other workers for studying atmospheric aerosols.(44,47-51) The unique feature in our experiment is the use of two pulsed lasers to interact with the particles.(52) The IR laser can be either a CO_2 laser, or an OPO laser tunable between 3.3 and 5 μm. This IR laser heats the particles to very high temperatures (1000 – 3000 K) and initiates the combustion process. Intermediates that escape the reaction zone are detected by ionization with the VUV laser. The VUV laser beam at 10.477 eV is created by 3rd harmonic generation in a mixture of Xe and Kr using the 3rd harmonic (355 nm) of a Nd-YAG Laser.(53) The resulting ions are mass analyzed by time-of-flight (TOF). Although a single particle generates a complete mass spectrum, we often signal average over 100 particles. Because the particles arrive at random times, the lasers must be fired at an irregular frequency. However, a home-built digital timing circuit ensures that the average frequency is maintained at 10 Hz so that the laser's temperature remains constant.

One of the interesting and important features about our experiment is that we can distinguish neutral molecules from pre-existing ions in the aerosol particle.(30) This turns out to be important in differentiating between ionic and neutral reaction channels. Neutral molecules are converted to ions only through photoionization with the VUV laser. On the other hand, pre-existing ionic species and ionic reaction products can be "lifted" out of solution simply through the vaporization of the solvent by the IR laser. The aerosols studied by this approach include nitromethane and some ionic liquids.

Because ionic liquids have no vapor pressure, it is possible to insert them directly into the vacuum system without the need to aerosolize them. This also avoids the use of solvents, which interfere with their decomposition. As shown in Figure 2, the sample (20-30 mg) is placed on the repeller plate. An IR laser (either 500 mJ from a CO_2 laser, or 10 mJ of an OPO laser) was then focused with a 25 cm lens onto the sample onto the sample. The OPO laser wavelengths were chosen to match the absorption peak of CH stretch bands around 2980 cm^{-1}. The ablated
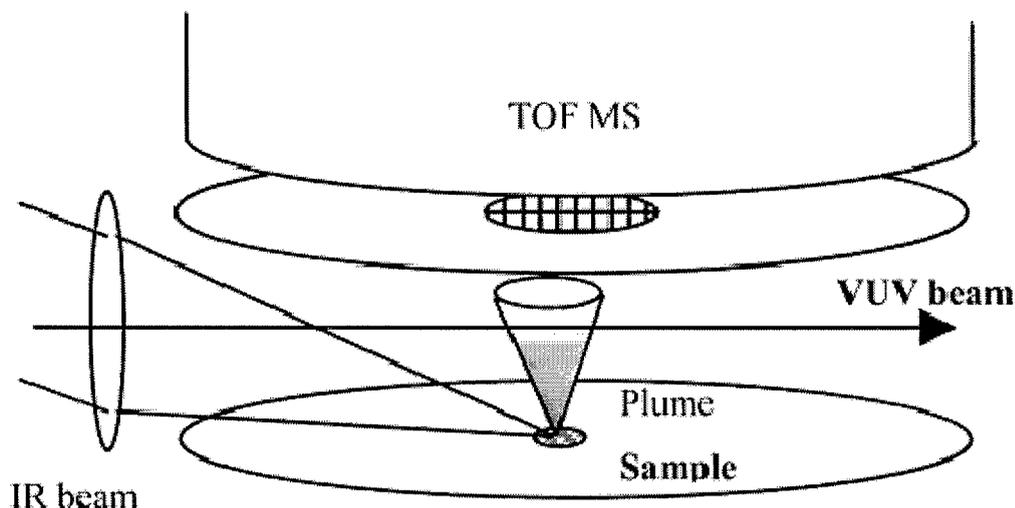
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Figure 2: Experimental arrangement for ablation of ionic liquids, a sample of which was placed directly onto the repeller plate of the mass spectrometer.

ions were extracted by applying a pulsed voltage to the ionization region after a variable delay time between 1 to 100 μs. In addition, the VUV laser could be fired to ionize any neutrals that have evaporated.

Summary of results

Ablation of ionic solutions: The study of ion-ion recombination

The base catalyzed combustion of nitromethane showed that an ionic pathway provided the dominant avenue for product formation. Our experiment allows us to distinguish ionic and neutral products that are formed in the combustion process. As shown in Figure 3, when the
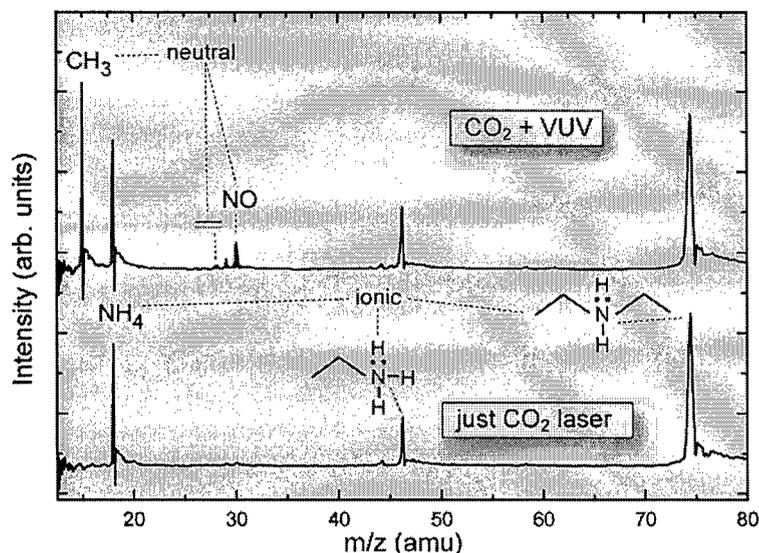

Figure 3. The mass spectra obtained by combusting nitromethane. The CO_2 laser only spectrum shows only the ionic species, while the upper spectrum shows also the neutral species that have been ionized by the VUV laser. From Woods et al. J.Phys.Chem. A (2001)

VUV ionizing laser is not turned on, only pre-existing ions are detected. New peaks resulting from the VUV laser shows neutrals that have ionization energies below the 10.5 eV of the laser. Because the pre-existing ions and the ionized neutral molecules are produced by different

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mechanisms, it is not obvious, how their peak intensities are related to their concentrations. We thus embarked on a study that addresses this question.
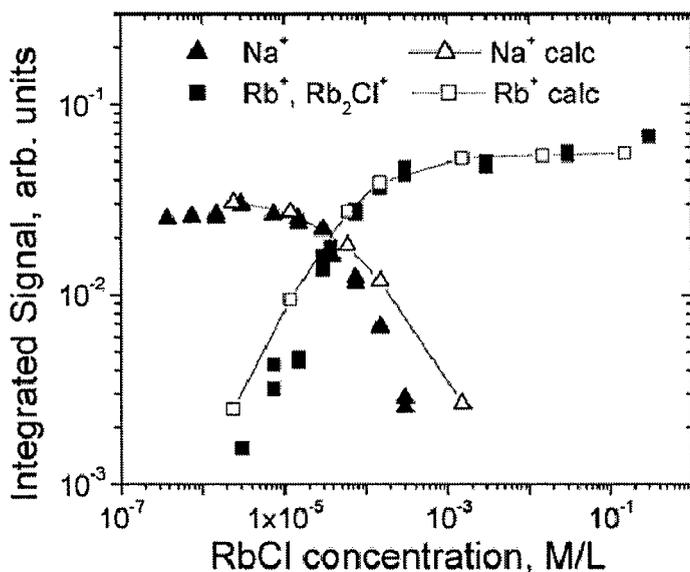

Figure 4. The ion signal as a function of RbCl concentration. The leveling off of the Rb^+ signal at the higher concentrations is a result of ion-ion recombination. The solid line through the points is a result of numerical modeling of the Thompson/Langevin 3-body recombination model. The only adjustable parameter is the temperature. From Dessiaterik et al. J.Phys.Chem.A (2003).

Aerosol particles of known size and known RbCl concentration were vaporized by the CO_2 laser so that we know precisely the absolute number of ions in these droplets. We then measured the total ion current produced when the particles were vaporized. At very low concentrations (less than 10^{-5} M), the measured current is equal to the number of ions in the sample. However, as the concentration is increased, the measured current in Figure 4 does not keep up and saturates at high concentrations (above 10^{-2} M). At the same time, the Na^+ signal, due to a constant background concentration of sodium ions in the sample, decreases as the Rb^+ ion concentration increases. These results suggest that ion-ion recombination depletes the ion signal. We analyzed
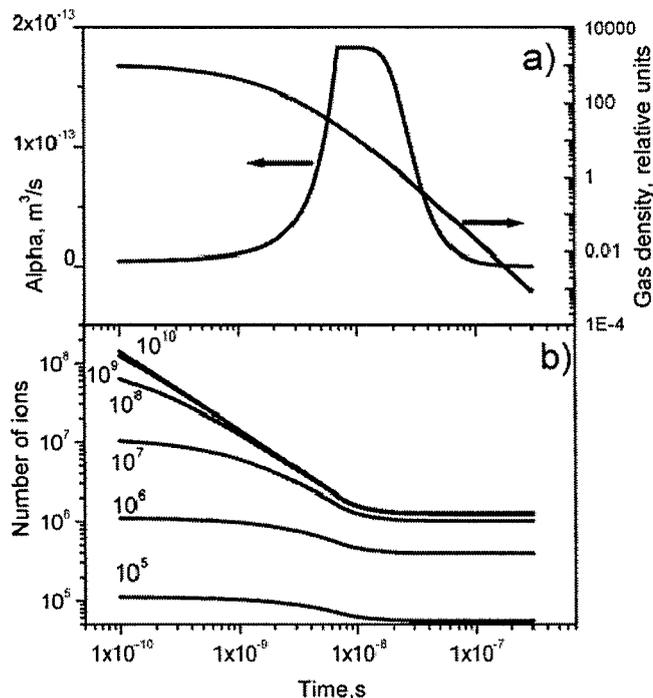

Figure 5: Calculated total gas density (upper graph) as a function of time after the IR laser pulse. We assume that the particle is hit with an infinitely narrow laser pulse and obtain the ion density (lower graph) as a function of time. What is evident is that all the ion-ion recombination has taken place within 10 ns. Furthermore the gas density rapidly drops leaving little time for 3 body collisions. In this calculation, we assumed that the gas density is uniform throughout the particle. This is certainly a very severe approximation, which could be modified in future calculations.

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this problem quantitatively with ion-ion recombination models based on a pressure dependent 3-body recombination rates proposed by J.J. Thomson(54) and P. Langevin.(55) The model assumes the following: the particle is heated instantaneously and all solvation forces cease. The aerosol particle then begins to expand at the speed of sound with a uniform gas density. The numerical solution to this problem resulted in a calculated signal intensity as shown by the solid lines in Figure 4. It is interesting that the only adjustable parameter is the assumed temperature of the expanding particle. The best fit, which was not very sensitive to the assumed temperature, was for a temperature of 2000K. This is very reasonable in light of our study with the thermometer molecules.

The really exciting aspect is that our model based on the Thompson/Langevin kinetics, permitted us to follow the concentration of ions in time as shown in Figure 5. This shows that the recombination reaction is complete within about 10-20 ns. Beyond that time, the remaining ions are ready to be detected. It is interesting that at this time the effective pressure is remains well above 1 atmosphere.

The other finding is the mechanism for ion extraction and coulomb explosion. Suppose that we wish to calculate the kinetic energy associated with the coulomb explosion arising from the repulsive interaction between the cations that are present in the ionization region at high density. First of all, the initial ions are closely associated with their anions so that the overall charge is neutral. In other words, we have a plasma. It requires a strong field to separate the positive and negative charges. However, in time, the total volume of the particles expands from the initial 2 μm to about 0.1 mm^2. At that point, the electric field required to separate the charges is much less so the positive and negative ion clouds can be completely separated. It is at this point that the coulomb explosion commences. Any calculation of the kinetic energy resulting from this coulomb repulsion must take into the account this effect.

Studies of Ionic Liquids:

Our initial efforts at investigating ionic liquids was based on the use of aerosol particles. However, the difficulty in preparing the pure ionic liquids without solvents, which interfere with their decomposition, led us to an alternative approach, which is shown in Figure 2. The following ionic liquids were investigated.

Ionic liquid	MW	MP ^{0}C
1,3-Dimethylimidazolium methyl sulfate (Me$_2$Im$^+$MeSO$_4^+$)	208.24	*
1-Ethyl-3-methylimidazolium chloride (EtMeIm$^+$Cl$^-$)	146.62	77-79
1-Ethyl-3-methylimidazolium nitrate (EtMeIm$^+$NO$_3^-$)	173.17	38-41
1-Butyl-3-methylimidazolium chloride (BuMeIm$^+$Cl$^-$)	174.67	55
1-Hexyl-3-methylimidazolium chloride (HeMeIm$^+$Cl$^-$)	202.72	-85

The IR laser ablation showed that the dominant ion produced is Cl$^-$ loss product. Examples are shown in Figure 6 where four of the five salts show mostly the Cl$^-$ loss product along with some m/z 83 ions. This latter ion is a result of fragmentation in which the long side chain is lost.
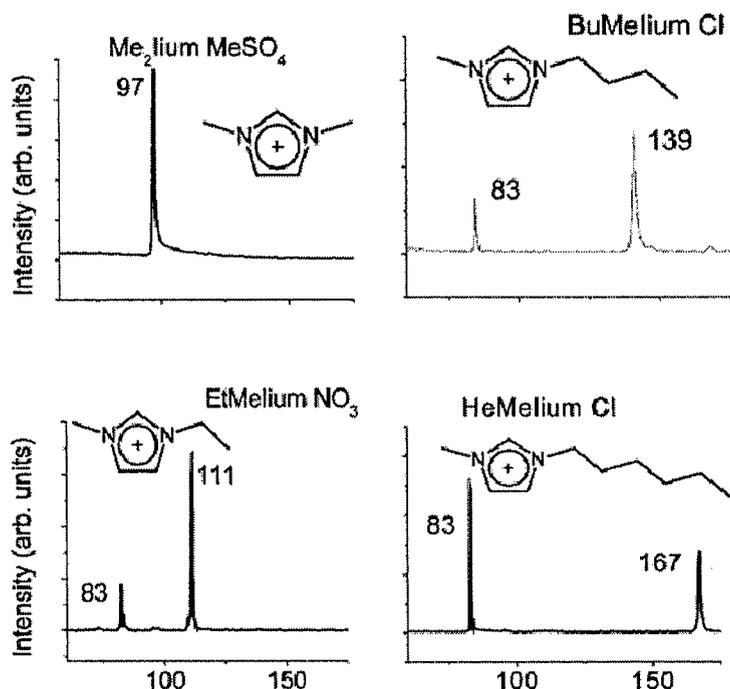
Figure 6: IR laser ablation mass spectra of several imidazolium ionic liquids. The dominant ion is a result of loss of the anion part of the salt.

When the VUV laser is fired some microseconds after the IR laser, we see a strong signal that looks very different. This is shown in Figure 7 for the case of the 1-hexyl salt.
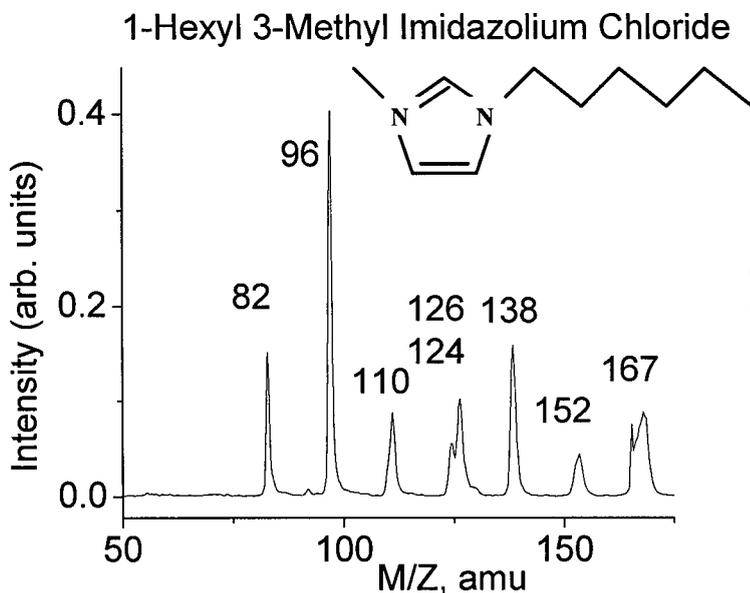

Figure 6. The mass spectrum obtained when the VUV laser is fired some 20 μs after the IR laser.

The mass spectrum is dominated by the parent molecule and fragmentation of the hydrocarbon chain. We have established that these fragment peaks arrives from dissociative photoionization of a single neutral precursor. This has been a long standing problem that is not easily resolved. These studies have demonstrated for the first time the mechanism for ablation and vaporization of ionic liquids. IR laser irradiation generates a substantial number of ions that are created by the

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loss of the Cl⁻ ions. Some of the energetic ions dissociate by breaking the long hydrocarbon chain. At the same time, and in much greater number (about 10^7 times more), IR laser irradiation creates neutral molecules by the elimination of HCl. During this process, the chloride ion grabs the ring hydrogen atom between the two nitrogen atoms. At the same time, the R group moves to take the place of the H atom. The net result is the production of an immidazole molecule.
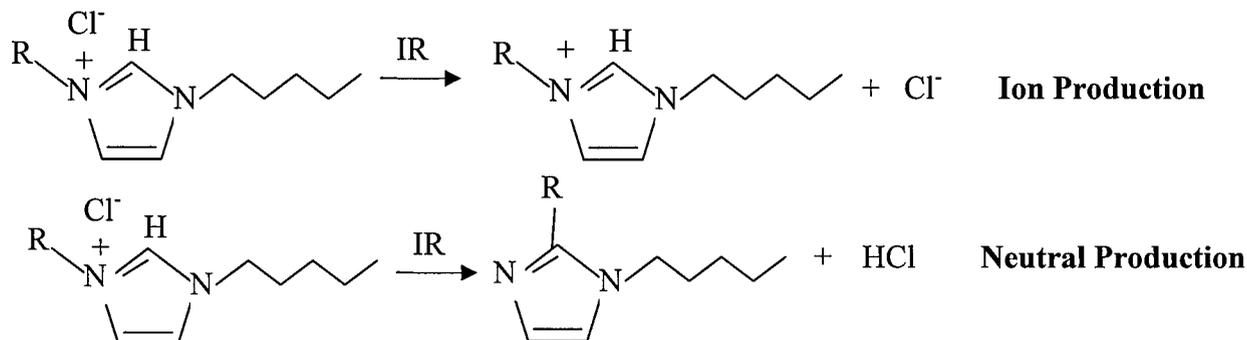
What happens is that the ablation causes the chloride ion to grab the hydrogen atom at the top of the immidazolium ion. At the same time, the R group moves over to take its place. The net result is that we vaporize an immidazole molecule, which can then be ionized with the vacuum UV light. The major evidence for this is the similarity of the mass spectra obtained with the VUV laser and those obtained from the corresponding immidazole molecules. Secondly, if the fragments observed with the VUV laser had come from neutral fragments generated in the IR ablation process, we would have observed ions such as C_4H_9 or C_4H_8. None were observed. Thus, all of the ion peaks observed in the VUV mass spectrum must have come from the immidalole precursor.

 We have also determined by gravimetric methods that the bulk of the ablation process results in the ejection of nanodroplets of intact salt. By quantitative comparison of all the data, we have determined that the IR laser ablation process results in the following products, and that the ablation process does not generate energetic fragments.

Salt Particles	99%
$R_1R_2Im + HX$	1%
R_1R_2IiumX	0.1%
$R_1R_2Iium^+ + X^-$	10^{-7}%
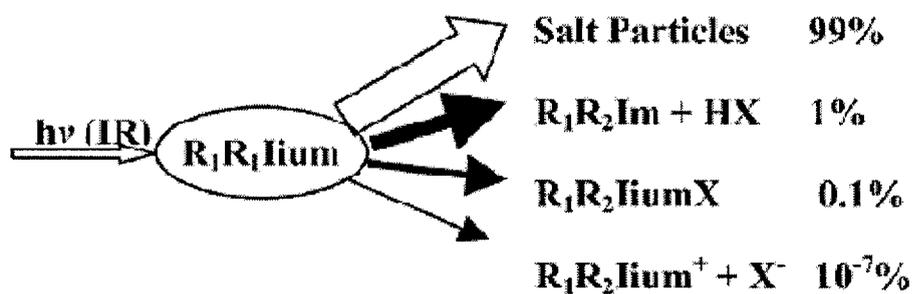
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Papers published with support of AFOSR:

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Personnel Supported by AFOSR grant:

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