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Flowing afterglow studies of dissociative electron-ion recombination for a series of single ring compounds at room temperature

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ABSTRACT

A series of dissociative electron-ion recombination reactions has been studied at room temperature using a Flowing Afterglow with Langmuir Probe (FALP) to determine the rate constants. Studied species include protonated five and six membered single rings with the heteroatoms, N, O and S and with or without CH_3 substituent groups attached to the rings. The compounds studied are protonated benzene, $C_6H_7^+$; toluene, $C_6H_5CH_4^+$; pyridine, $C_5H_5NH^+$; pyrimidine, $C_4H_4N_2H^+$; 4-picoline, $C_6H_7NH^+$; cyclohexane, $C_6H_{13}^+$; 1,4 dioxane, $C_4H_8O_2H^+$ all six membered rings (all but the last two have π electrons in the ring) with furan, $C_4H_4OH^+$; pyrrole, $C_4H_5NH^+$; 1-methylpyrrole, $C_5H_7NH^+$; thiophene, $C_4H_4SH^+$ and pyrrolidine, $C_4H_9NH^+$ all five membered rings (with only the last mentioned having no π electrons in the ring). Comparison is made with literature data on some of the multi-ring systems (PAH's and PANH's). The involvement of π electrons in the rings is discussed. Protonated six membered rings have been shown to recombine ~2 times faster than five membered rings with the exception of 1,4 dioxane which has no π electrons. The situation is very different for the proton bound dimers, where five membered rings recombine somewhat more rapidly than six membered rings, again with 1,4 dioxane behaving very differently. These data are critically important in chemically modeling the Titan ionosphere which is presently being probed by apparatus onboard the Cassini spacecraft.

1. Introduction

Interest has increased in the electron-ion dissociative recombination (DR) of protonated single ring compounds since their discovery in the interstellar medium, ism, (C_6H_6, benzene) [1] and the atmosphere of Titan (C_6H_6, benzene; C_5H_5N, pyridine; C_7H_8, toluene) [2–4]. In particular, an enormous amount of detail on the atmosphere of Titan has been obtained by the highly successful Cassini space probe using the on board positive ion-neutral mass spectrometer (INMS) with a mass range from 1 to 99 amu and better than 1 amu mass resolution. The Cassini plasma spectrometer (CAPS) has detected positive ions up to 350 amu and negative ions from 20 to 8000 amu [5]. Some of these positive ions have been tentatively identified as naphthalene, anthracene derivatives and an anthracene dimer [2]. In these regions, polycyclic aromatic hydrocarbons (PAH's) and polycyclic aromatic nitrogen heterocycles (PANH's) have also been detected as the unidentified infrared bands and diffuse interstellar bands in the ism [6,7], and surprisingly as negative ions in the Titan atmosphere [2]. In the latter atmosphere, they are believed to be precursors for aerosol and tholin formation. For Titan, it has recently been suggested that sequential ion-molecule associations of ring compounds with the dominant ions (CH_3^+, $C_3H_3^+$, etc.) followed by dissociative electron-ion recombination would provide a route to the building up of multi-ring compounds from the observed, and likely, single rings [8]. For example, $CH_3^+ + C_6H_6$ (benzene) leads to an association compound with the composition of protonated toluene ($C_6H_5CH_4^+$). After recombination, the $C_3H_3^+ + C_6H_5CH_3$ (toluene) reaction could lead to the protonated 1,2 dihydronaphthalene ($C_{10}H_{11}^+$) (double ring). See Eq. (1).

$$\begin{array}{l} CH_3^+ + C_6H_6 \xrightarrow{M} H_3CC_6H_6^+(\text{Protonated toluene}) + M \\ (\text{Protonated toluene}) + e \rightarrow \text{Toluene} + H \\ C_3H_3^+ + \text{Toluene} \xrightarrow{M} C_3H_3^+ \cdot \text{toluene} + M \\ C_3H_3^+ \cdot \text{toluene} + e \rightarrow C_{10}H_{10}(1,2\ \text{dihydronaphthalene}) + H \end{array} \tag{1}$$

Note that associations with CH_3^+ followed by recombinations may also lead eventually to 1,2 dihydronaphthalene [8]. The recombination of these compounds also helps to maintain the ionization balance in the ism and the Titan atmosphere. In addition, in the association of these species with CH_3^+, it has been shown that the association component of the reaction mechanism is dependent on the presence or absence of π electrons in the rings [8]. It is of interest to pursue the recombination studies to determine whether recombination also exhibits a dependence on the pres-

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ence of π electrons. Note that since the Titan atmosphere is mostly N_2 (98%), heterocyclic rings incorporating N-atoms are likely to be an important component of the atmosphere. Few oxygen containing compounds have been observed in the Titan atmosphere (CO, CO_2, O) [9] so oxygen atoms in the ring are unlikely to be important there although they may be important in the ism where more oxygen containing species exist. As a contribution to an understanding of the chemistry of these regions, we have studied the dissociative electron-ion recombinations of the protonated six membered rings benzene, $C_6H_7^+$; toluene, $C_6H_5CH_4^+$; pyridine, $C_5H_5NH^+$; pyrimidine, $C_4H_4N_2H^+$; 4-picoline, $C_6H_7NH^+$; 1,4 dioxane, $C_4H_8O_2H^+$; cyclohexane, $C_6H_{13}^+$ and the five membered rings, furan, $C_4H_4OH^+$; pyrrole, $C_4H_5NH^+$, n-methylpyrrole, $C_5H_7NH^+$; pyrrolidine, $C_4H_9NH^+$ and thiophene, $C_4H_4SH^+$. Note that 1,4 dioxane, cyclohexane and pyrrolodine have no π electrons. Recombinations of a few single ring hydrocarbons have been studied previously $C_4H_5^+$, 53 amu; $C_6H_6^+$ (benzene), 78 amu; $C_6H_5CH_3^+$ (toluene), 92 amu; and the seven membered rings benzylium$^+$, 91 amu; tropylium$^+$, 91 amu; and cyclohetatriene$^+$, 92 amu [10,11] and there have been some studies with multi-ring systems (e.g., $C_{14}H_{10}^+$, anthracene ions) [12]. No theoretical calculations of recombination have been made for ions of this complexity. In the course of our recombination studies [8], and following previous work with CN containing ions [13,14], it has been found that association between the protonated reactant vapor [RV].

$$RVH^+ + RV + He \rightarrow (RV)_2H^+ + He \tag{2}$$

And RV can be competitive with recombination. This has complicated the studies since the recombination rate constants, α_e, can now vary with the RV concentration and, thus, the composition of the associating gas. Thus, it is essential to determine this variation and identify the reactant vapor concentrations where α_e is independent of the concentration and where single species such as RVH^+ or $(RV)_2H^+$ dominate the chemistry. This has enabled the recombination of proton bound dimers to be studied in addition to the protonated species. Note that plasma situations cannot always be created where only a single recombining species exists, in such cases the individual α_e were determined through chemical modeling, see Fig. 3b.

2. Experimental

These studies were carried out in a temperature variable flowing afterglow, which has been discussed in detail previously [15], and, thus, only a brief description will be given here. Helium carrier gas (Airgas UHP grade He) (with throughput 220 Torr l s^{-1} and pressure 1.5 Torr) is ionized in a microwave cavity and flowed along a flow tube (8 cm dia.; 1 m in length) to a downstream mass spectrometer/electron multiplier under the action of a roots blower pump. The electron density, [e], in the flowing plasma is determined by an electrostatic Langmuir probe operated in the orbital limited regime [16]. Gases (Ar and H_2) are added upstream sequentially into the flow to create Ar^+/e and H_3^+/e plasmas. Downstream of these additions, the reactant vapor (RV) is introduced into the flow to produce the protonated ion of interest by proton transfer from H_3^+ (reaction (3)) and the proton bound dimer by the association reaction (2) thus, generating the recombining plasmas of interest.

$$H_3^+ + RV \rightarrow RVH^+ + H_2 \tag{3}$$

Note that where there is a large difference between the proton affinities of the two neutral species the proton transfer reaction will be very exothermic and can result in fragmentation of the product ion. Where this occurs, an additional step is made in the chemistry to include an additional proton transfer from N_2H^+, HCO^+ or $C_6H_7^+$ so that the final proton transfer to produce RVH^+ is less exothermic and does not cause fragmentation. This method of proton transfer was utilized for the two ringed compounds cyclohexane and pyrrolidine. The electron density is observed to reduce along the flow tube as measured by the axially movable Langmuir probe. In this recombination controlled situation, [e] varies with distance, z, as seen in Fig. 1 and given by the equation.

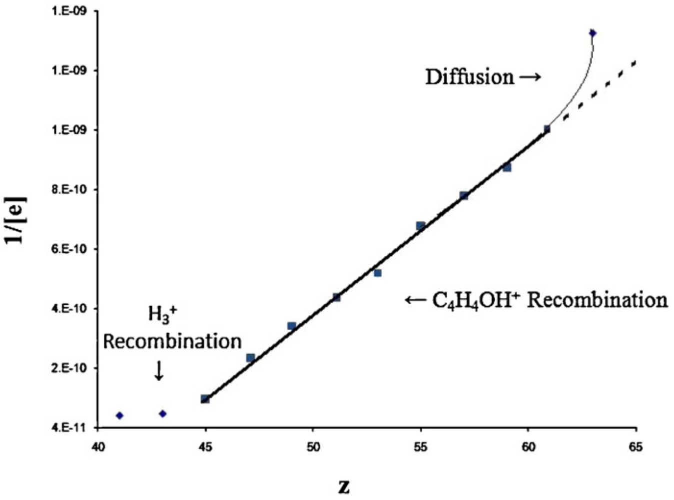

Fig. 1. Variation of 1/[e] versus distance, z, along the flow for the electron ion recombination of protonated furan at 300 K.

$$[e]^{-1} - [e]_0^{-1} = \frac{\alpha_e z}{v_p} \tag{4}$$

where α_e is the recombination rate constant, and v_p is the plasma velocity along the flow tube. Recombination rate constants, α_e, for this system are determined as a function of concentration [RV] and yield schematic plots of the form shown in Fig. 2a–c. Fig. 2a illustrates plateau regions where H_3^+, RVH^+ and $(RV)_2H^+$ are the recombining ions at the various RV concentrations. Obviously, it can be seen that without such a plot it is likely that erroneous rate constants would easily be obtained. An example for the 4-picoline system is shown in Fig. 3a where both the monomer and dimer plateaus can be seen in the data. Note that it is not always possible to cover all three regions as illustrated for RV being pyrrole (see Fig. 3b). At low [RV], the plasma will be that of H_3^+ since this recombines very slowly [17]. At higher [RV], proton transfer with RV dominates (reaction (3)) and the plasma is then controlled by RVH^+. This is shown in Fig. 2b; note that if recombination of RVH^+ dominates over association then no $(RV)_2H^+$ is observed. As [RV] is increased further, association (reaction (2)) becomes more rapid and dominates over RVH^+ recombination producing the proton bound dimer $(RV)_2H^+$, then giving a region dominated by $(RV)_2H^+$ and the recombination (see Fig. 2a).

$$RVH^+ + e \rightarrow \text{Products} \tag{5}$$

$$(RV)_2H^+ + e \rightarrow \text{Products} \tag{6}$$

If however, this association (reaction (2)) is very slow, RVH^+ will dominate up to large [RV] and this would give the form of α_e versus [RV] as shown in Fig. 2b depending on the association rates. Note that, the steps may not be well defined and curves with only indications of plateaus may be all that can be obtained, such as in Fig. 3b. Fig. 2a assumes that $\alpha_e((RV)_2H^+) > \alpha_e(RVH^+)$. If $\alpha_e((RV)_2H^+) < \alpha_e(RVH^+)$then the form will be as shown in Fig. 2c; note that this form has not been observed experimentally. An example of actual data for an RV of pyrrole is shown in Fig. 3a. Here steps are not quite so well defined, however values of $\alpha_e((RV)_2H^+)$ (with

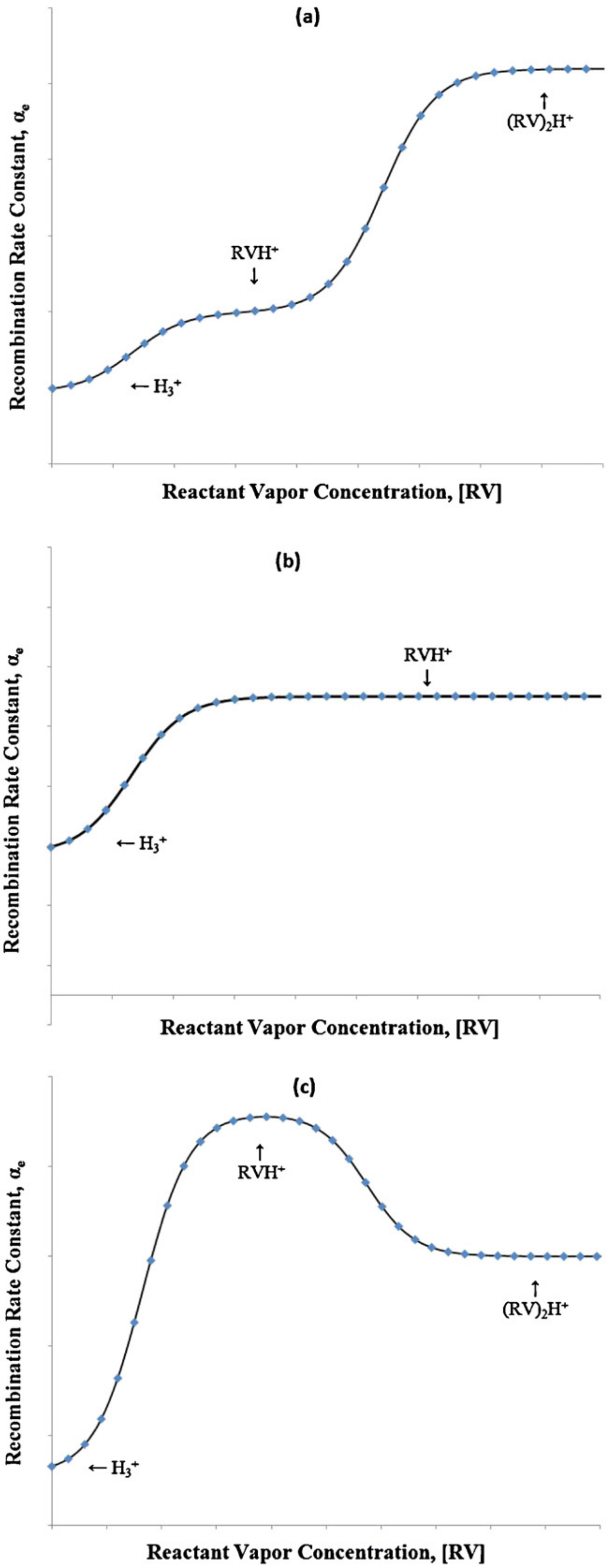

Fig. 2. Schematic of the variation of α_e with reactant vapor concentration, [RV], showing the regions where H_3^+, RVH^+ or $(RV)_2H^+$ dominate the recombination. (a) Plateaus in recombination for RVH^+ and $(RV)_2H^+$ with $\alpha_e(RVH^+) < \alpha_e((RV)_2H^+)$ and asymptotic approach to H_3^+ recombination at low reactant vapor concentrations. (b) Plateau in recombination for RVH^+ with no production of $(RV)_2H^+$ and an asymptotic approach to H_3^+ recombination at low reactant vapor concentrations. (c) Plateaus in recombination for RVH^+ and $(RV)_2H^+$ with $\alpha_e(RVH^+) > \alpha_e((RV)_2H^+)$ and asymptotic approach to H_3^+ recombination at low reactant vapor concentrations.

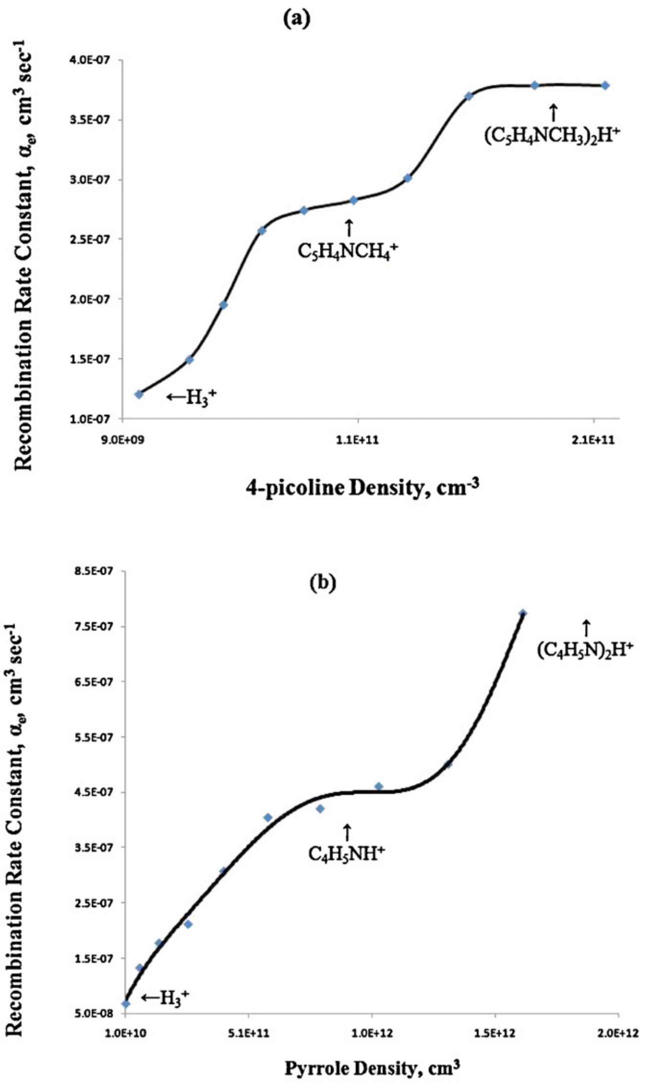

Fig. 3. (a) Experimental data for the recombination rate constant, α_e, of protonated 4-picoline as a function of 4-picoline reactant vapor concentration showing both the monomer and the dimer plateaus; and (b) experimental data for the recombination rate constant, α_e, of protonated pyrrole as a function of pyrrole reactant vapor concentration. This shows that the plateaus for the dimer cannot be seen in all cases.

lower accuracy) and $\alpha_e(RVH^+)$ can still be obtained by chemical modeling. The overall model has the form.

$$\underset{\alpha_3\downarrow e H_2,k_{-3}}{H_3{}^+} \overset{RV,k_3}{\rightleftharpoons} \underset{\alpha_M\downarrow e}{RVH^+} \underset{k_{d,He}}{\overset{RV,He,k_a}{\rightleftharpoons}} \underset{\alpha_D\downarrow e}{(RV)_2H^+} \tag{7}$$

where α_3, α_M and α_D are the recombination rate constants of $H_3{}^+$, the monomer RVH^+ and the dimer $(RV)_2H^+$ respectively. K_3 and k_{-3} are the forward and reverse rate constants of reaction (3) and k_a and k_d are the forward and reverse rate constants of the association reaction (2). It should be noted that in modeling of the data, fitted values were used for the k rate constants. Changes in these values by a factor of 4 did not significantly influence the fitted recombination rate constants, i.e., by ~15%. To reduce the association, reaction mixes of 0.5–1.5% by volume reactant vapor (RV) in He were made. The reactant vapor was obtained by putting a pure sample of the reactant in to a glass vial and then purifying it through a series of pump-freeze thaw cycles in which all the impurities gases suspended within the liquid would be driven off and pumped away. This permits a mix to be made which in low concentrations gives the ability to study a wider dynamic range of concentration [RV] in

Table 1
Room temperature (300 K) recombination rate constants, α_e ($cm^3\ s^{-1}$) for the protonated monomer (RVH^+) and proton bound dimer ($(RV)_2H^+$) for the compounds indicated.

Compound, RV	Ion formulae	Protonated monomer	Proton bound dimer	Monomer ion mass (amu)	Ion concentration in the titan atmosphere (cm^{-3})
6-Membered rings		$\alpha_e(RVH^+)$[a]	$\alpha_e((RV)_2H^+)$[a]		
Benzene	$C_6H_7^+$	8.0(−7)[b]	~5.0(−7)	79	18.0
Pyridine	$C_5H_5NH^+$	8.5(−7)[b]	~6.0(−7)	80	4.0
Pyrimidine	$C_4H_4N_2H^+$	1.35(−6)[b]	~7.0(−7)	81	0.7
Toluene	$C_6H_5CH_3H^+$	3.8(−7)	~1.3(−6)	93	2.4
4-Picoline	$C_5H_4NCH_3H^+$	2.83(−7)	3.79(−7)	94	0.9
1,4 Dioxane	$C_4H_8O_2H^+$	2.35(−7)	~4.3(−6)	89	4.2
Cyclohexane	$C_6H_{13}^+$	1.3(−6)	~3.9(−6)	85	0.15
5-Membered rings					
Furan	$C_4H_4OH^+$	4.1(−7)	~7.3(−7)	69	1.8
Pyrrole	$C_4H_5NH^+$	4.0(−7)	~9.4(−7)	68	4.0
1-Methylpyrrole	$C_4H_4NCH_3H^+$	5.0(−7)	~1.0(−6)	82	0.22
Pyrrolidine	$C_4H_9NH^+$	2.7(−6)	~7.0(−7)	72	0
Thiophene	$C_4H_4SH^+$	>7.0(−8)	N/A[c]	84	0

[a] (−7) means × 10^{-7}.
[b] Values obtained previously [8].
[c] Indicates not available.

cases where rapid association is present. This has proven to be the only method by which data could be obtained in the protonated monomer region. Rate constants are accurate to ±15% for protonated monomers and ±20% for the proton bound dimers. Where the proton bound dimer was not clearly observed, only approximate values could be given for the rate constants.

3. Results and discussion

The data that have been obtained at room temperature for a series of recombination rate constants for RVH^+ and $(RV)_2H^+$ are given in Table 1 for RV being benzene, C_6H_6 (Sigma >99.5%); toluene, $C_6H_5CH_3$ (Sigma 99.8%); pyridine, C_5H_5N (Sigma 99.9%); pyrimidine, $C_4H_4N_2$ (Sigma 99.9%); 4-picoline, C_6H_7N (Sigma 98%); 1,4 dioxane, $C_4H_8O_2$ (Sigma 99.8%); cyclohexane, C_6H_{12} (Sigma ≥99.7%); six membered rings and the five membered rings, furan, C_4H_4O (Sigma 99+%); pyrrole, C_4H_5N (Sigma 98%) and 1-methyl pyrrole, C_5H_7N (Sigma 99%); pyrrolidine, C_4H_9N (Sigma 99.5%) and thiophene, $C_4H_4SH^+$ (Sigma 98%). Note that the data for benzene, pyridine and pyrimidine have been published previously and are included here only for completeness and comparison [8]. To help indicate the relevance that the data may have for Titan, the measured concentration of ions of the same mass to charge as the studied ions has been included in table. A great deal of information can be gleaned from this table.

The 6-membered rings, for which data are quoted, all have six π electrons excluding 1,4 dioxane and cyclohexane (which have none) and all of the 5-membered rings, except pyrrolidine, also have six π electrons. For the protonated monomers, the rate constants are generally larger for the 6-membered rings relative to the 5-membered rings by about a factor of 2. The only exception is 1,4 dioxane, which has a much smaller rate constant ($2.3 \times 10^{-7}\ cm^3\ s^{-1}$ relative to the average six membered ring rate constant, $\sim 9 \times 10^{-7}\ cm^3\ s^{-1}$) and no π electrons. This indicates that in this case there may be a dependence on the presence and absence of π electrons. Although, cyclohexane has a large monomer rate constant but no π electrons. However, the rate constants for five and six membered rings would be expected to be similar since the same numbers of π electrons are involved in both cases. The fact that there is a factor of ~2 difference (smaller for five membered rings) may be related to the difference in the mobility of the electrons in the six- and five-membered rings. The situation for the proton bound dimers is very different with the 5-membered rings having rate constants slightly larger than the 6-membered rings. Again 1,4 dioxane and cyclohexane are the main exceptions, with rate constants larger than the other species. Thus it appears that in this case, the π electrons may be influencing the rate constants. It is also of interest to note that the rate constant given for thiophene is a lower limit for the monomer species due to the adverse effects of thiophene on the metal of the Langmuir probe. No information on the thiophene dimer could be obtained. It can also be noted that the addition of a methyl substitution may decrease the rate constant in six membered rings. This can be the observed when comparing 6-membered rings with their methyl substituted and non methyl substituted forms, such as when benzene and pyridine are compared to toluene and 4-picoline. There seems to be the opposite effect with the five membered rings, pyrrole compared to n-methyplyrrole. The protonated ring compounds can also be compared with chain hydrocarbons. Lehfaoui et al. [18] studied the recombination's of a series of such ions varying in complexity from CH_5^+ to $C_8H_{17}^+$ (CH_5^+, 7.0×10^{-7}; $C_2H_5^+$, 6.0×10^{-7}; $C_3H_7^+$, 8.3×10^{-7}; $C_4H_9^+$, 8.3×10^{-7}; $C_5H_{11}^+$, 7.2×10^{-7}; $C_6H_{13}^+$, 7.6×10^{-7}; $C_7H_{15}^+$, 5.5×10^{-7};and $C_8H_{17}^+$, 8.2×10^{-7}, all $cm^3\ s^{-1}$). The α_e for these species are somewhat smaller than those in Table 1 for six membered rings showing that there are factors in play which increase the efficiency of ring recombination. The situation is however not clearly defined as can be seen from comparisons with a series including larger ring compounds varying from $C_3H_3^+$, 7×10^{-7}; $C_5H_3^+$, 9×10^{-7}; $C_6H_6^+$, 1×10^{-6}; $C_7H_5^+$, 7×10^{-7} to $C_{10}H_8^+$, $3 \times 10^{-7}\ cm^3\ s^{-1}$ [19]. Here there is no clear pattern of increasing α_e with increasing ion complexity. The overall picture though is followed when O-atoms are added to the ring with its electron withdrawing nature, resulting in a reduction in the rate constant (to $2.35 \times 10^{-7}\ cm^3\ s^{-1}$) for 1,4 dioxane. This may offer an explanation for the rate constant difference between cyclohexane and 1,4 dioxane monomer.

Also, previous data on the six membered rings benzylium, $C_6H_5CH_2^+$ and cycloheptatriene, C_7H_8 and the seven membered ring tropylium c-$C_7H_7^+$[11] are in keeping with the present measurements. In our studies for the six membered rings with no side groups, α_e increases as the number of N-heteroatoms in the ring is increased from zero to two. As substituent groups, CH_3 are added, the α_e also increases in some cases.

The situation is dramatically different for the proton bound dimers (see Table 1). The α_e for the O-containing six membered ring, dioxane, and for cyclohexane, which have no π electrons are much larger than all of the other six membered rings by about a factor of five. More data are needed to definitely establish these trends and offer possible clues to the origins of these effects.

4. Conclusions

The data on dissociative electron-ion recombination obtained in this study have contributed significantly to chemical modeling of the Titan ionosphere and influencing the ionization balance. It has been shown that protonated six member rings recombine somewhat more rapidly than their proton bound dimers and protonated five member rings. Thus, multi rings made up of six membered rings are likely to grow more rapidly in the Titan atmosphere enhancing naphthalene, anthracene, coronene, etc. rather than azulene with its five membered ring. For substituted rings, the rate constants are generally larger as the degree of heteroatom and, in limited cases, CH_3 group substitution increases. This has provided more data for modeling the growth of multi-rings which has recently been suggested to occur in association of the major ions, $CH_3{}^+$, $C_3H_3{}^+$, etc. followed by recombination. These ring compounds are known to be stable and it is thus likely that the ring will remain intact upon recombination making these species the precursors to further ring growth.

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