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**COMbustion- and Re-Entry-PLasma research Using the ion
Storage facility DESIREE (COREPLUS)**

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14. ABSTRACT This project exploits the world-wide unique capability of the Stockholm University, Double ElectroStatic Ion Ring ExpERiment (DESIREE). This purely electrostatic storage facility enables the study of interactions between cations and anions at low and well-defined internal temperatures and centre-of-mass collision energies down to about 10 K and 10 meV, respectively with control over the reaction environment (temperature: ~10 K to 300 K) and the reaction collision energy (0.01 eV to 100s eV). This facility enabled initial investigations into neutralizing reactions relevant to: (1) Air-plasmas related to both laser-ignition and plasma-assisted combustion and (2) Plasmas generated around re-entrant space vehicles. Position sensitive multi-hit detector systems which have been extensively tested and proven to work in cryogenic environments was used in coincidence to measure correlations between reaction products in, for example, electron-transfer processes. Control over the reaction environment and the reaction collision energy means that any desired information, for examples: reaction cross-sections and fragment populations can be obtained over all conditions needed to accurately model these processes. Towards the end of this effort, C+ cations and C- anions have been simultaneously injected into the asymmetric and symmetric rings, respectively, and stored for several 10s of seconds. The entire apparatus has been running continuously at 12 K and < 10-12 Torr for the last 18 months. The 100 kV heavy-ion platform has been completed and commissioned. Currently, a hot-filament ion source (Nielsen-type) is installed, and mu-As of Ar+ and C+ cations have been extracted and transported into the ring. The 25 kV light-ion platforms has been running continuously for the last 12 months. Currently, a NEC SNICS II sputter ion source is installed, and mu-As of C-, C2- anions have been extracted and transported into the ring. At the time of this report, the residual gas-defining pressure in the device is sufficiently good that 10 keV C+/C- ions have been stored in the symmetric light-ion ring for 40 minutes. Lifetime issues, such as the limitation of the lifetime of 10 keV C+/C- ions in the asymmetric ring to 10 minutes, were also explored and remedied. Preparations have been made to test both ion sources to create the ions relevant to the COREPLUS project, drawing on both experiences from measurements at CRYRING (in Stockholm) as well as results from other storage rings, e.g., ELISA in Denmark.					
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Second year summary

The second year of the project has been defined by the commissioning period of the storage device, in which the following important DESIREE milestones have been reached:

- I) Within the last month, C^+ cations and C^- anions have been simultaneously injected into the asymmetric and symmetric rings, respectively, and stored for several 10s of seconds.
- II) The entire apparatus has been running continuously at 12 K and $< 10^{-12}$ Torr for the last 12 months.
- III) The 100 kV high energy platform has been completed.
 - Currently, a hot-filament ion source (Nielsen-type) is installed, and μ As of Ar^+ and C^+ cations have been extracted and transported into the ring.
- IV) The 25 kV light ion platform has been running continuously for the last 12 months.
 - Currently, a NEC SNICS II sputter ion source is installed, and μ As of C^- , C_2^- anions have been extracted and transported into the ring.
- V) At the time of writing, the residual gas-defining pressure in the device is sufficiently good that 10 keV C^+/C^- ions have been stored in the symmetric light-ion ring for 40 minutes.
 - A technical issue, which will be resolved in the next weeks, limits the lifetime of 10 keV C^+/C^- ions in the asymmetric ring to 10 minutes.

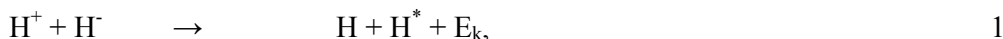
Preparations have been made to test both ion sources to create the ions relevant to the COREPLUS project, drawing on both experiences from measurements at CRYRING (in Stockholm) as well as results from other storage rings, e.g., ELISA in Denmark.

General Introduction

Mutual Neutralization

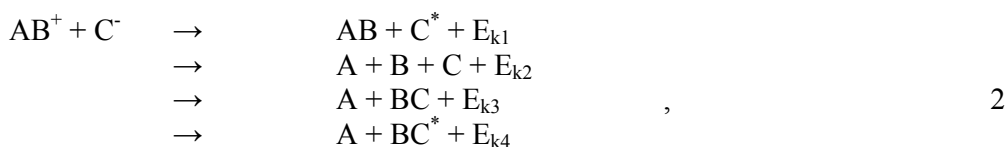
The majority of the visible universe exists in a state of weak ionisation, the so called fourth state of matter: plasma. Plasmas are ubiquitous, from those occurring naturally; interstellar molecular clouds etc., to those which are anthropogenic in origin; flames, in combustion engines, and in fusion reactors. The evolution of these plasmas is driven by the interaction of the plasma constituents; cations, anions and electrons, both with each other and their environment. One of the most important subsets of these reactions is recombination. Here, negative and positive charge carriers react to produce neutral products. This process is significant for two very important reasons. It is an ionisation reducing reaction, removing two ionised species and producing neutral products and, furthermore, these products may themselves be reactive radical species which can then further drive the evolution of the plasma.

Although the simplest subset of these reactions to study has been electron-cation reactions, and ion-storage ring devices have proven to be an excellent tool providing extremely high quality data on these reactions [1], in many types of plasma, free electrons are neither the only nor even dominant carrier of negative charge, and atomic and molecule anions are present in significant quantities and may even dominate. However, the experimental requirements to study such neutralising anion-cation reactions are much more demanding than those to study electron-cation reactions. The rate at which the electron reacts with the cation depends on many parameters, for examples the collision energy, the internal energy of the cation, and the structure of the cation itself, and products from these reactions also need to be measured in coincidence if any insight into the reaction process is to be obtained. Replacing the electron with an anion adds significantly more complexity to the reaction, and here the process is called Mutual Neutralisation (MN):



where, typically, since the ionization energy is larger than the electron affinity, there is sufficient energy available in the collision to create internally excited products, e.g. A^* , with the remainder of the reaction energy given to the fragments as kinetic energy, E_k .

For reactions involving molecular species, the process becomes significantly more complicated, e.g.:



as molecular bonds may be broken and created, and reaction energy stored as ro-vibronic energy in molecular products – as opposed to purely electronic energy in atomic fragments.

The quality of MN data is significantly less satisfactory than electron-cation data. No storage ring data exists, and where only a limited number of single-pass merged beams studies are reported. The latter almost exclusively focus on high-energy, >3 eV, collisions between atomic ions (see e.g. [2]), and for those few studies involving molecules nothing is known about their internal energy - the interactions typically take place μs after the ions have been

created in (hot) sources (see e.g. [3]). Order of magnitude differences are observed between different apparatus for the same reactions, a fact also observed in the few studies at low <1 eV collision energies [2, 4].

The simplest and most fundamental MN process, the $H^+ + H^-$ reaction [5], also has considerable uncertainties. Recent theory [6] agrees with higher-energy data [7] and while significantly disagreeing with older low-energy data [8] a modified merged-beams apparatus reports better agreement [9]. The $H^+ + H^-$ system is special in the sense that both ions only have a single bound quantum state. However, the theoretical treatment is also highly complex, both due to the requirement to have extremely well defined potential energy curves, especially curves for the [by definition infinite number of] molecular Rydberg states which play a role in this reaction, but also by the fact that these processes occur over extremely long distances due to the Coulomb force between the reacting particles, requiring extremely large matrices and fine grids which makes good calculations exponentially expensive. Notwithstanding that, several recent theoretical treatments on this simplest of systems, using the highest quality curves, have shown agreement with reported experimental data at higher collision energies though, significantly, disagree markedly with the same data for lower energies.

MN has been studied in other apparatuses, e.g. the Flowing Afterglow (FA), which was initially developed to study ion-neutral reactions relevant to atmospheric physics, but now also used to study MN [10-14]. Data are obtained under thermal 300-500 K conditions and several interesting observations reported: diatomic anions recombine slower than polyatomic anions [14]; a previously unobserved plasma loss process proposed - electron-catalyzed mutual neutralization (ECMN) [13]; and an empirical formula [15] for the reaction rate fits 300 K data to $\pm 30\%$ [10]. However, disagreements exist between merged-beams and FA data for other electron-transfer reactions, see e.g. [1], and significant modelling of FA data is necessary [12]. Furthermore, the empirical model has only been minimally tested: FA data outside 300-600 K do not exist; internal energy is ignored; only rare gas cations are studied in reactions with anions having large binding energies. This is an unsatisfactory situation for such an important reaction. These issues clearly motivate need for high-quality merged beams data. For example, reactions with loosely bound anions only may be performed in a cold environment such as that in DESIREE due to the black body radiation-induced photodetachment [16,17]. Such merged beams data will test the MN rate model, the latest theory, and refute/confirm whether ECMN exists.

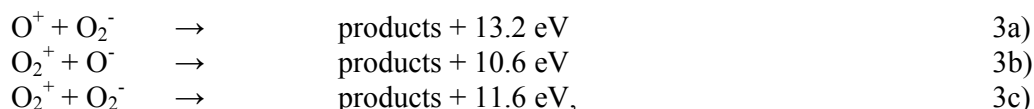
The construction at Stockholm University, Sweden, of a purely electrostatic storage facility, Double ElectroStatic Ion Ring ExpEriment (DESIREE), uniquely allows for studies of interactions between cations and anions at low and well-defined internal temperatures and centre-of-mass collision energies down to about 10 K and 10 meV, respectively [18,19]. Position sensitive multi-hit detector systems which have been extensively tested and proven to work in cryogenic environments [20] will be used in coincidence to measure correlations between reaction products in, for example, electron-transfer processes [16,18,19]. Control over the reaction environment (temperature: ~ 10 K to 300 K) and the reaction collision energy (0.01 eV to 100s eV) means that any desired information, for examples: reaction cross-sections and fragment populations, can be obtained over all conditions needed to accurately model these processes.

Air-plasmas related to both laser-ignition and plasma-assisted combustion.

A recently demonstrated technique of laser-induced ignition for an aircraft combustion system has attracted funding with the aim of both optimising and modelling the ignition process.

However, modeling of the phenomena leading to avalanche and breakdown has so far proven to be a considerable challenge due to the unknown effects from electronegative oxygen, both O_2^- and O^- , during the avalanche phase. Data on the critical air ionization chemistry involving such oxygen ions, for example in their mutual neutralisation with their respective cations, must be obtained and understood if the chemistry of the pre-ionization processes leading to electron avalanche and breakdown are to be understood.

The following three oxygen-plasma related reaction systems are of interest, e.g.



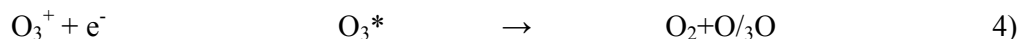
where the energy quoted corresponds to products formed exclusively in their ground state. Taking the first of these reactions in a little more detail:



We see that:

- 1) There are two main product channels “chemical branching”: one two-body and one three-body product channel, i.e., the latter channel includes dissociation of the O_2 .
- 2) There is sufficient energy in both channel D and E to create internally excited atomic oxygen products “electronic branching,” as well as rovibronically excited O_2 in channel 3ai).

Experiments conducted at CRYRING, an electron-cooler equipped heavy ion storage ring located at Stockholm University, on the associated electron-ion neutralisation reaction [1,9]:



showed that:

- 1) More than 98% of the reactions resulted in the production of the three O atoms.
- 2) The product O atoms were electronically excited though not in a statistical manner, i.e. the dynamics on the O_3 potential energy surface played a large role in the fragmentation process.

With this in mind, the three reactions here present many opportunities to probe the excited state dynamics created in such collisions, which otherwise would be impossible to study by usual (photonic) means (selection rules etc), and we plan on measuring the rate constants for the overall neutralization process together with the amount of dissociation and electronic state distribution of the O atoms.

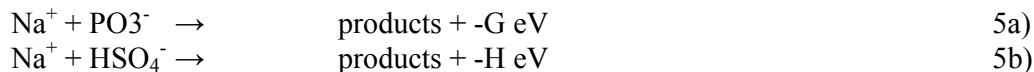
Such data are also applicable to numerous other systems including radio communication in the the D-region, reentry, and plasmas assisted combustion. Additionally, they are also benchmark systems for molecular dynamics calculations, and it will be particularly intriguing to compare the two systems involving three oxygens, where the energetics differ by 2.5 eV.

Plasmas generated around re-entrant space vehicles.

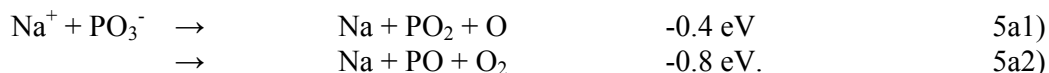
The aspect of reentry plasmas is also related to the second aspect of this proposal, namely the studies of plasmas generated by reentrant space vehicles as they traverse through the upper atmosphere. Such plasmas effect both ground-vehicle radio communication, as well as the

deleterious effects such plasmas cause to the heat-shielding and other exposed systems on the vehicle.

As part of this goal, in the second year of this proposal we plan on studying the following reactions with interest to re-entrant space vehicles:

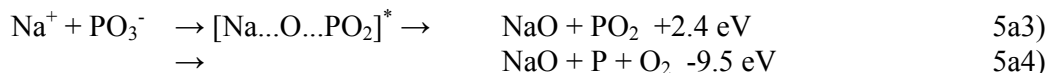


On both of these cases, in collisions at zero eV all possible direct electron transfer (i.e. long range processes without forming intermediate transient states) product channels are endothermic. Taking the first of these reactions in a little more detail, and looking at the first two exothermic channels:



We see that these channels have large activation energies and that there is a significant difference in these energies. Using the control over the interaction energies available in DESIREE, it will be possible to probe the “turning-on” of these channels individually.

Of further interest is the possibility that such reactions proceed through reactive intermediates, and not long-range charge transfer, i.e. making bonds between the two partners:



Reaction 5a3) is exothermic, meaning that this reaction is energetically open. If it is populated this will be measureable and we will immediately be able to make conclusions on the reaction process. It is worth noting that the least endothermic three-body channel, (5a4), which directly involves molecular products containing Na is endothermic by almost 10 eV. This means that channels 5a1) and 5a2) can be studied irrespective of whether channel 5a3) is populated as the former two processes are the only possible three-body product channels in collisions under 9 eV. These studies will allow for the first time a test as to whether a reactive neutralization is possible or efficient and has implications for other potential recombinations where neutralization is endothermic, e.g. atmospheric reactions with cluster ions.

The interest of the Air Force in studies into MN reactions is shown in several papers on the subject, see e.g. [10-14], as well as their finding of this project. These papers demonstrate the first thermal studies of ion-ion recombination which have been reported in many years, and have yielded important and unique data. However, and of implicit relevance both to this proposal and to the Air Force, these studies are restricted to reactions involving noble gas cations. The DESIREE facility has no such restrictions and, furthermore, allows not only the neutral reaction products to be identified but also the electronic state of the atomic species.

Methods and discussion

A schematic of the whole facility is shown in Figure 1, while a picture of the facility “in reality” is shown Figure 2.

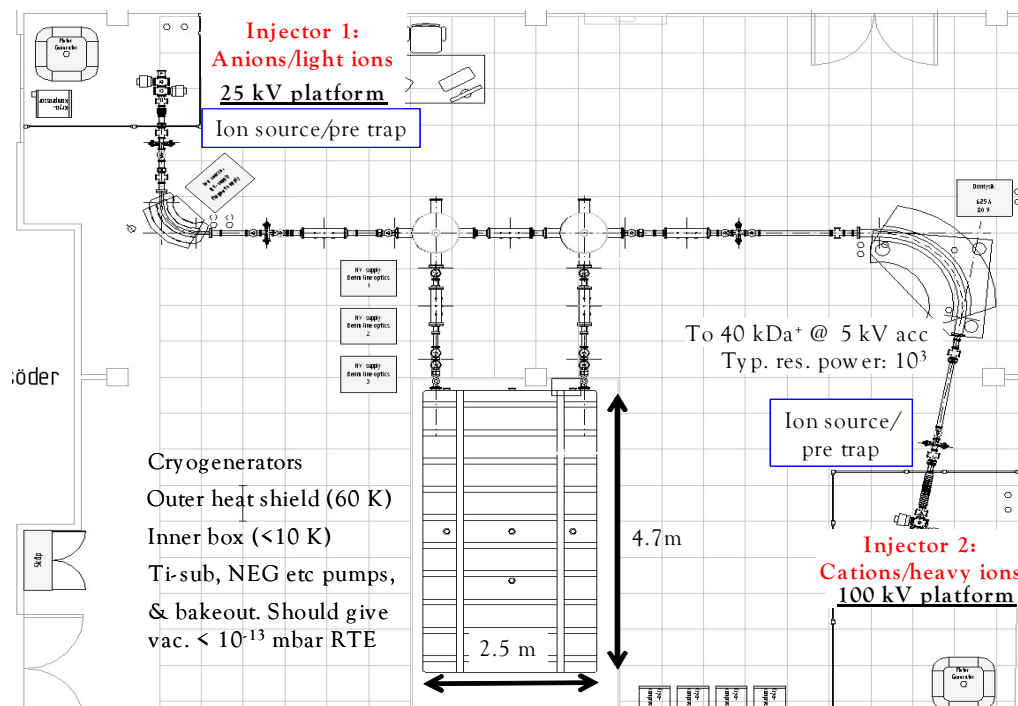


Figure 1: A technical overview of the DESIREE facility: the main vacuum vessel, the two ion source platforms, injection beam lines, and some general physical characteristics

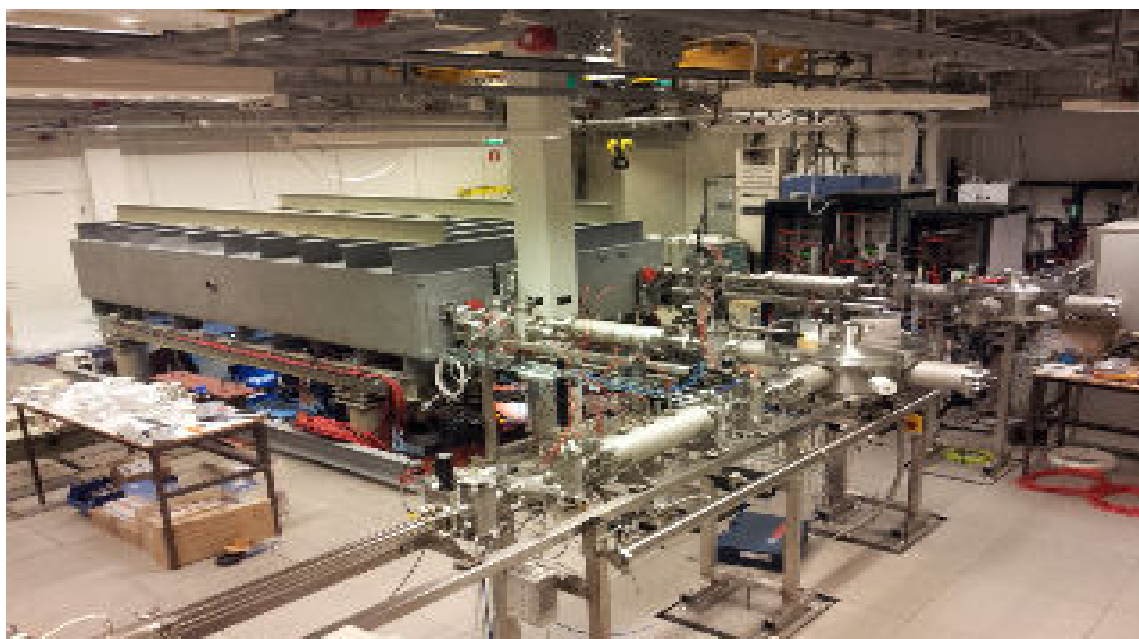


Figure 2: A picture of the DESIREE facility. Here, the main vacuum vessel is shown back and centre, while the light ion 25 keV platform is to the right.

Figures 3 and 4 show the heart of the DESIREE experiment, with a schematic and picture, respectively.

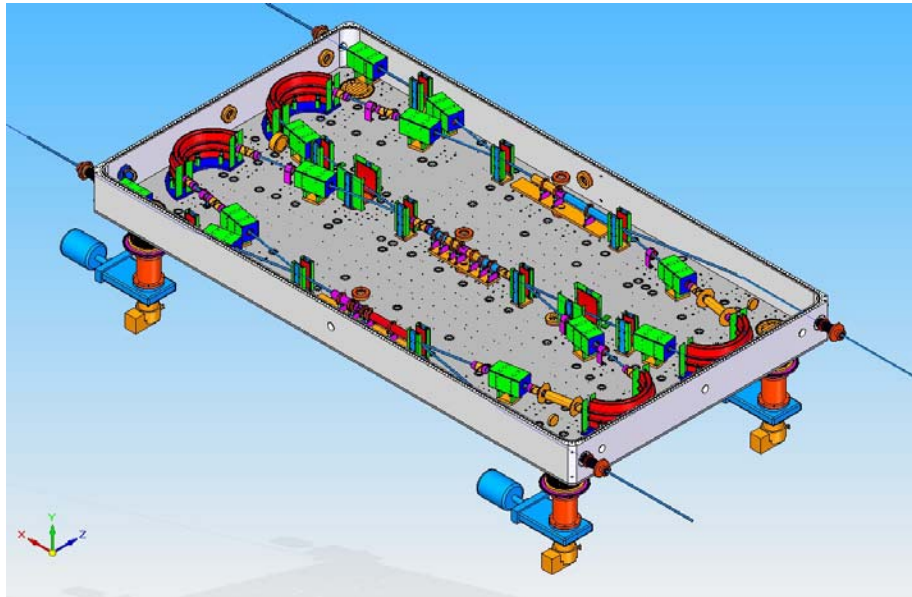


Figure 3: A technical schematic of the heart of the DESIREE facility: the double-walled vacuum vessel, showing the open ion optics, the two separate storage rings, and the unique merging region.



Figure 4: A picture of the heart of the DESIREE facility: the double-walled vacuum vessel, showing the open ion optics, the two separate storage rings, and the unique merging region

A complete technical paper describing in detail the facility was published in 2011 [18]. The first commissioning paper, discussing actual operation of the facility and the long-time storage of ions in a cryogenic environment, was published in 2013 [19], and a third paper concerned with the simultaneous storage of oppositely charged ion species is being written and will be submitted in the next few months [21]

In brief, ion sources located on the source platform are used to create the anions and cations of interest. These ions are then transported into one of the two rings inside the DESIREE vacuum vessel, where they will be stored for several to many tens and hundreds of seconds. During each revolution the ions pass through a common straight section (merging region) where, under the right conditions, they will interact, and the products from this interaction will be detected.

Before the right conditions are sent, during storage, infra-red active ions will thermalize to the surrounding temperature, ~ 12 K, via photon emission. After a sufficient storage time, the conditions in the merging region, which is operated as a segmented drift tube, are controlled to allow interactions to take place and neutrals products formed from these interactions will no longer be deflected by the ion optics and they strike a detector where their position and difference in arrival time is recorded.

The voltage on the various segments which comprise the drift tube region is steered externally and is used to control the relative velocity, and hence collision energy, between the oppositely charged ion species. With this method collision energies down to 10 meV can be reliably achieved, the lower limit being set both by the ion optics in the storage ring and the energy spread of the broadest of the ion beams. Neutral particle rates as a function of the collision energy are then obtained, and these data are then used to extract a kinematically-complete picture of the interaction which occurred.

Results

DESIREE milestones

Many technical problems were still unresolved at the start of the COREPLUS project. However, almost all the facility-critical (and project dependent) milestones were reached during the timeframe of the project:

1. Within the last month, C^+ cations and C^- anions have been simultaneously injected into the asymmetric and symmetric rings, respectively, and stored for several 10s of seconds.
2. The entire apparatus has been running continuously at 12 K and $< 10^{-12}$ Torr for the last 18 months.
3. The 100 kV heavy-ion platform has been completed and commissioned.
 - 3.1. Currently, a hot-filament ion source (Nielsen-type) is installed, and μ As of Ar^+ and C^+ cations have been extracted and transported into the ring.
4. The 25 kV light-ion platforms has been running continuously for the last 12 months.
 - 4.1. Currently, a NEC SNICS II sputter ion source is installed, and μ As of C^- , C_2^- anions have been extracted and transported into the ring.
5. At the time of writing, the residual gas-defining pressure in the device is sufficiently good that 10 keV C^+/C^- ions have been stored in the symmetric light-ion ring for 40 minutes.
 - 5.1. A technical issue, which will be resolved in the next weeks, limits the lifetime of 10 keV C^+/C^- ions in the asymmetric ring to 10 minutes.

Ion and neutral particle detection

There are several in-situ detectors available for monitoring the number of ions stored in each ring as well as the number of products produced during the mutual neutralisation interactions in the common merging section.

The number of ions stored in the rings can be monitored by using split-electrode pick-up devices located at each end of the merging section. As the ion bunches pass through the split-electrode pick-up, an image-charge is created that is related to the number of ions in the bunch and its distance from the electrode: the use of a set of split electrode pickups then allows the centre of the ion bunch to be determined as well as the ion current. Furthermore, as the positive and negative ions generate oppositely charged images charges, several important parameters such as their relative positions and their currents can be determined at the same time. Figure 5 shows example signals generated by the pick-ups when the carbon anions stored.

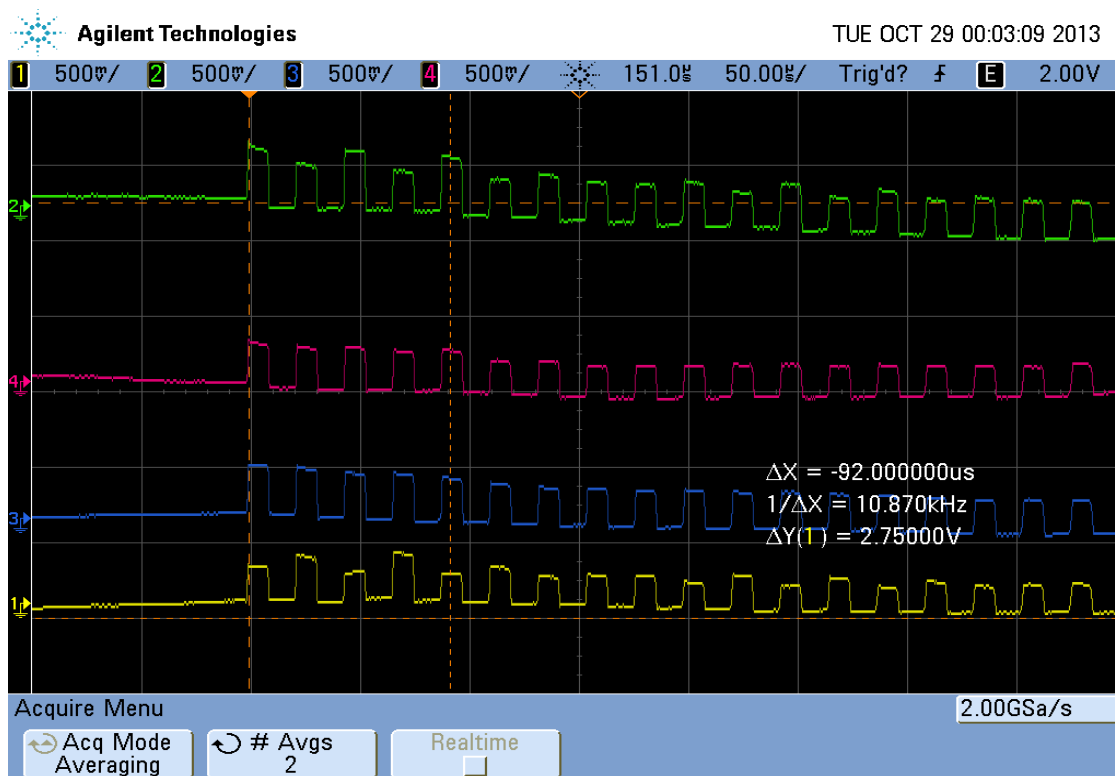


Figure 5: Example signals showing the image charges created by the stored ions on each of the segmented pick-up elements.

The combination of the signals from the pick-up detectors with the neutral particle detector located 1.5 m distance away, and discussed next, from the last element in the merging region will allow absolute rate coefficients, and hence reaction cross-sections to be determined.

Figure 6 shows the “lab-side” of the neutral particle “imaging detector”: an MCP-phosphor screen (PS) inside vacuum, with windows to couple the light from the PS out into the lab where it can be monitored [20].



Figure 6: A picture showing the phosphor screen anode of the imaging detector. The green-blue spot corresponds to the detection of neutral atoms created from collisional detachment of stored ions with those few residual gas molecules that remain in the common straight section

Here, the light comes from the neutral particles, resulting from the collisional neutralisation of the stored ions with the residual gas particles, striking the MCP and, eventually, causing the PS to phosphoresce.

There are two similar “imaging-type” detectors in the main vacuum vessel, and these monitor the neutral particles produced from residual gas collisions in each of the non-common straight sections, i.e., the neutrals produced exclusively from anion-rest gas and cation-rest gas collisions. The following two figures, Figure 7 and Figure 8, plot examples showing the number of neutrals atoms measured as a function of storage time of C^- anions [19] in the symmetric ring, and C^+ cations in the asymmetric ring, respectively.

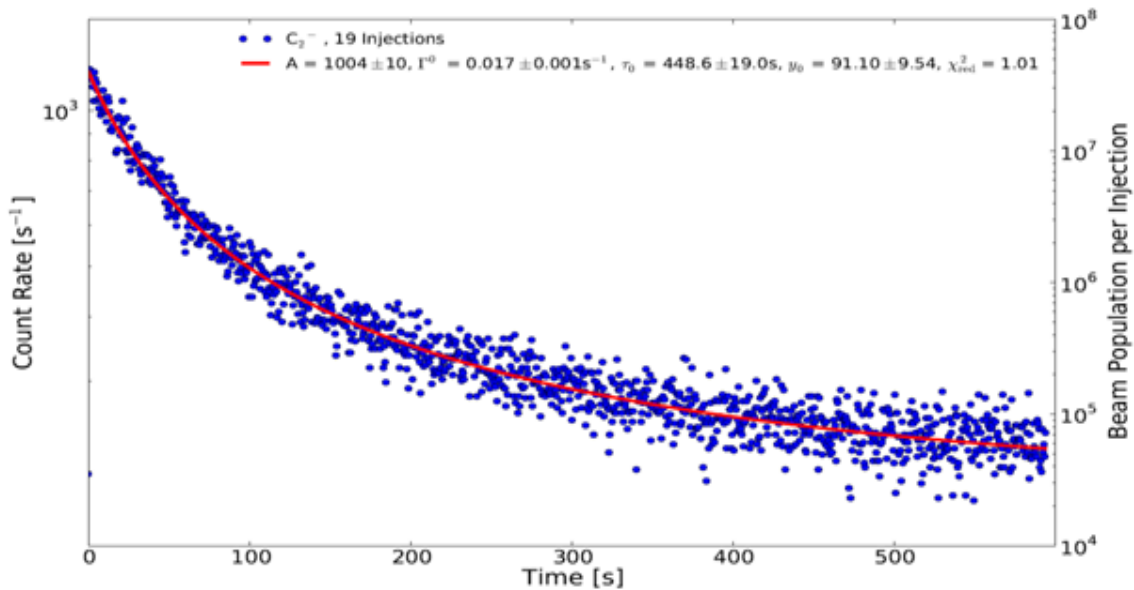


Figure 7: The number of neutrals atoms measured as a function of storage time to indicate the storage lifetime of C^- anions in the symmetric ring

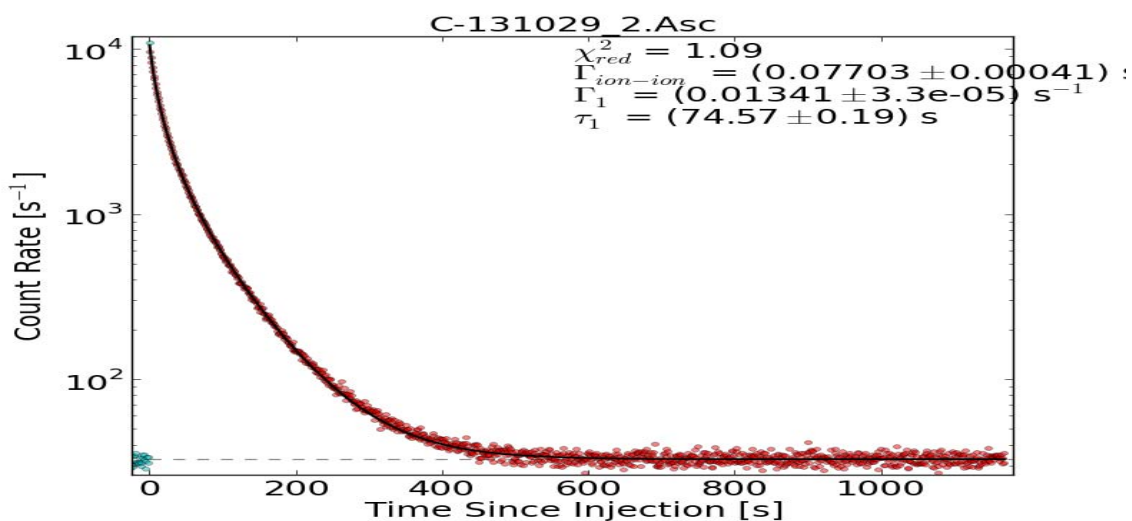


Figure 8: The number of neutrals atoms measured as a function of storage time to indicate the storage lifetime of C^+ cations in the asymmetric ring

Analysis of these data was carried out in a similar fashion to that reported by Reinherd *et al.* in measurements of the lifetime of He^- anions in a cryogenically cooled ion trap [17,22]: part of a proof-of-principle R&D programme supporting DESIREE. Using this analysis, about 1.2 million anions are transported into the storage ring every injection [19].

However, in these examples, it is quite clear that the ion-beams have different lifetimes. In principle, the lifetime should be defined by the loss of ions through collisions with residual gas particles, and since the ions see identical residual gas number densities in both rings, the lifetime of the cations is determined by another process. In this particular case, it is the fact that an unfiltered power supply I used to switch between the injection of the cations into the ring and their storage. This lack of filtering means the small ripples on the supply voltage (which are 0.01 V in 1000 V) are sufficient to destabilize the ions during each revolution that they are no longer stored.

Conclusions

We are almost ready to begin the first merged beams measurements. The “two” storage rings are working and have been used to store ions successfully. More importantly, just recently, both cations and anions have been simultaneously injected and stored in the asymmetric and symmetric rings, respectively.

The signals due to the image charge the ions generate as they pass through the pick-up electrons has been used to determine the centre-of-mass of both the stored ion beams.

The micro-channel plate detector which is used to determine the position and difference in arrival times of the neutral particles arising from the mutual neutralisation reaction is being commissioned and, after a camera-upgrade expected to occur in the next few weeks, will begin operation. The detectors which monitor the interactions of the ions with residual gas particles in the separate straight sections of the ring are working as designed and are used to determine the collisional lifetime of the ion beams.

All of these detectors are required if we are to be able to successfully characterise the properties of the rings as they relate to ion storage and beam manipulation as well as understanding the interaction of the cations and anions in merged-beams interactions.

“Take-home message”

Within the next month the first mutual neutralisation measurements will be made in DESIREE, under cryogenic conditions and for well defined centre of mass collision energies ranging from a few meV to a few hundred eV.

Knowledge transfer

Two, direct and refereed, papers will have come out from work related to DESIREE, where the COREPLUS project has supported my participation in this work: The commissioning paper [19], and the simultaneous storage of oppositely charged ions [22]. Complimentary papers related to the initial experiments, which the COREPLUS project will support me (due to the differences in the financial year start-end dates) will also be prepared.

Under the two-year duration of the project, I have been invited to speak about the DESIREE facility and the experiments I will undertake on more than ten different occasions: many of which have been at international conferences (in France, China, the United States, Germany, and Sweden, and with one forthcoming in India), while two have been national in Sweden. Several collaborators have visited the laboratory under the project duration, hosted by myself, both those with immediate AFOSR connections to the COREPLUS project, e.g., Dr. Nick Shuman, Kirtland AFB, as well as those with links to AFOSR-funded research and interest in DESIREE, e.g., Prof. R. E. Continetti, UCSD. Other collaborators with interest in DESIREE have also been hosted by me.

Future considerations.

Everything necessary for the reactions proposed in the COREPLUS project, i.e. the materials necessary for the creation of the ions to be investigated, have been purchased (both anions and cations) and source testing on several of these ions has been started. Here, production and optimisation of O^- , O_2^- and PO_3^- will be undertaken. The relevant cations are more straightforward as these ions have been created before and similar ion sources will be used.

The initial commissioning experiments after C^+/C^- will focus on atomic ion collisions, e.g. of relevance here O^+/O^- due to their relative simplicity compared to molecular ion collisions allowing a more detailed investigation of the parameter space necessary to understand exactly what we are measuring.

It is expected that we will then move on to the next simplest type of collisions, i.e. atom-di-atom and several of the reactions proposed in this project will be studied here, e.g., $O^\pm/O_2^\pm$. Finally, presentation of the current status of DESIREE as well as the initial experiments is planned for several relevant conferences.

In discussion with several interested parties in the DESIREE project, most significantly, Al Viggiano and Mike Berman, I will be submitting a “continuation” proposal to the COREPLUS project. This will again address the reactions mentioned here, but coupled with a rigorous programme of mutual neutralisation reactions tailored to test the various models which have been proposed for these reactions, see e.g. [15], as well as experimental observations on the reaction properties, see e.g. [14], by extending the range of possible anion and cation reaction partners which are beyond the reach of FA instruments. Finally, testing of an interesting, yet previously unobserved, plasma loss process in recent FA measurements: electron-catalyzed mutual neutralization (ECMN) [13], will also be investigated.

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