The ILIAMS project - an RFQ ion beam cooler for selective laser photodetachment at VERA

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Abstract

Selective laser photodetachment of anions is a novel technique for isobar suppression in Accelerator Mass Spectrometry (AMS). Ion-laser interaction times on the order of ms required for near-complete isobar suppression are achieved by retarding the ions in a gas-filled radio frequency quadrupole cooler. Inside this RFQ, the cooled anion beam is overlapped collinearly with an intense cw-laser beam. Within the Ion Laser InterAction Mass Spectrometry (ILIAMS) project at the University of Vienna, a dedicated injector beamline has been coupled to the VERA-AMS facility to explore and develop this method. In this work, experimental investigations on ion beam transmission, stability and elemental selectivity of the new setup are presented.

A 532 nm laser at 10 W transmitted power provides suppression factors larger than ten orders of magnitude for S^- and MgO^- under AMS conditions with simultaneous beam transmission for the ions of interest of up to 80%. The excellent ion identification capabilities of the subsequent AMS system also facilitate the study of destruction and formation of molecular anions inside the ion cooler. These kinetic and chemical reactions with the buffer gas provide additional elemental selectivity in certain cases, whereas others constitute a source of background.

1. Introduction

The idea to use the element selective process of laser photodetachment as a new means of isobar suppression in AMS was first explored almost 30 years ago [1]. This development was driven by the aim to overcome the ΔZ/Z- and velocity-dependence of established isobar separation techniques (cf. [2]) preventing measurements of high-Z trace isotopes at environmental levels. However, it was not until the use of an RFQ ion cooler for anions that laser ion beam interaction times became sufficiently long to facilitate continuous macroscopic depletion of interfering ion species using commercially available lasers [3, 4].

The main prerequisite of this technique, which is called Ion Laser InterAction Mass Spectrometry (ILIAMS) in the following, is that the electron affinity EA of the ion of interest has to be higher than EAs of disturbing ions. This can be fulfilled in either the atomic or a molecular anion system. Once such a system has been identified, cw-lasers with suitable photon energy are in general readily available due to big differences in EAs [5,6]. Furthermore, demands on the laser system other than power and beam quality are rather low, i.e. linewidth and wavelength stability are generally of no concern since photodetachment is a non-resonant threshold process. After an interaction time t between ions and a laser beam with photon flux Φ, the number N(t) of surviving ions with EAs smaller than the photon energy is simply given by

$$N(t)=N(0)\cdot e^{-\sigma\Phi t} \qquad (1)$$

where $N(0)$ is the initial number of ions and σ the photodetachment cross section.

Assuming a photon flux on the order of 10^{21} $cm^{-2}s^{-1}$ and typical photodetachment cross sections around 10^{-17} cm^2, efficient removal of isobars to AMS-relevant levels requires interaction times of at least several hundred of μs. At the same time, losses of the ions of interest need to be kept to a minimum.

At the University of Vienna, an RFQ ion cooler for selective laser photodetachment of negative ions has been developed [7]. In the following we will report on the coupling of this RFQ to the Vienna Environmental Research Accelerator (VERA) AMS facility as part of a new injection beamline and experimental results.

2. Methods

A comprehensive description of VERA is found in [8-10]. In this contribution only details of the new injection beamline of the ILIAMS setup as well as the performance of the RFQ ion cooler therein will be presented.

Figure 1 shows a schematic of the new injection beamline and the entire VERA AMS-facility. The major components of the new injector are a 40-sample-MC-SNICS ion source, a double-focussing 90° bending magnet with a bending radius of 0.35 m, an RFQ ion cooler and a set of 45° spherical Electrostatic Analyzers to couple the beam into a standard AMS-injector. The ion energy in the ILIAMS injector is limited to 30 keV by the design of the ion cooler. Hence, the maximum bending power of the 90° magnet of 8.4 MeV amu is sufficient to bend ions up to mass 280 at full injection energy. The image distance of the magnet is such that the nominal beam waist lies exactly at the cooler entrance. Electrostatic xy-steerers before and after the bending magnet allow to adjust ion beam position and direction at the cooler entrance. A Conidur® fine hole sheet made of stainless steel (Hein, Lehmann GmbH, Krefeld, Germany) acts as a 60-fold beam attenuator. The fine perforation of 0.05 mm at a sheet thickness of only 0.2 mm allows thinning out the ion beam without substantially reducing its phase space envelope. This attenuator is employed during injection of the stable reference beam into the ion cooler (cf. 3.1). The long beamline section following the ESA after the ion cooler is due to constraints in the available laboratory space.

The RFQ ion cooler itself has been extensively described in [7], so only its major features are covered here. Its central part is a 951 mm long quadrupole electrode structure terminated by insulated 3 mm apertures at both ends. The radius inscribed by the quadrupole rods is 4.35 mm. A weak longitudinal DC guiding field of typically 2 V/m (maximum 15 V/m) is created by four long plate electrodes wedged in between the RF-rods. A set of aperture lenses counteracts beam expansion during deceleration and allows focusing of the beam during reacceleration. Ion residence times inside the RFQ range from 500 μs to several tens of ms depending on injected ion current. The buffer gas pressure, which under routine operation is pure Helium (Air Liquide, class 5), is leaked in at the center of the cooler tube via a manually operated bleeding valve. The entire cooler structure is set to a potential a few ten Volt below the cathode potential in the ion source to facilitate electrostatic deceleration of the ions. Currently, this is achieved by a separate HV-power supply. A difference-stabilized HV-setup is foreseen in the future.

The RF is supplied by two resonant circuits coiled in opposite direction, which are driven via a ferrite transformer by a 200 W E&I 1020L RF power amplifier (Electronics & Innovation Ltd., Rochester, NY, USA) and a function generator DS345 (Stanford Research Systems, Sunnyvale, CA, USA). A binary set of 9 purpose-built switchable inductors ensures resonance conditions for different frequencies. Typically, below 10 W of RF power is necessary to achieve zero-to-peak-voltages of around 300 V at frequencies of a few MHz. However,

around 30 W of primary power are required due to significant power reflection at the ferrite transformer.

The entire cw laser setup fits onto a 1.5x1 m laser table placed adjacent to the beamline. The view port of the bending magnet in cooler direction is equipped with a MgF_2 window to allow laser beams of wavelengths between 200 nm and 6 μm to enter with > 94% transmission. Likewise, the 45° ESA after the ion cooler has an 8 mm hole in its outer half-sphere and a MgF_2 window at the viewport to allow the laser beam to exit. Commercial calorimetric power meters are used to monitor the transmitted laser power. Until now, all experiments have been conducted with a 532 nm 18 W VERDI-V18 cw laser (Coherent Inc, Santa Clara, CA, USA), but a 984 nm (10 W) and a tunable IR 2000-3000 nm (<5 W) are also available in the laboratory.

All components including the cw laser have been fully integrated into the VERA control system "*AccelNET*" (NEC, Middleton, WI, USA) via a separate Beckhoff control system (Beckhoff Automation GmbH & Co. KG, Verl, Germany) and a parameter exchange server developed in-house. This allows to operate ILIAMS and VERA remotely and to run fully-automated measurements.

The absence of slits at the beam waist between the ILIAMS magnet and the RFQ ion cooler unfortunately limits the mass resolution of the setup in its present state to $m/\Delta m \approx 150$. Figure 2 shows a simulation with SIMION 8.1 (Scientific Instrument Services, Inc., Ringoes, NJ, USA) of neighboring beams at masses 273 to 277. In combination with the injection lens system, the separation of the different mass beams is too small to fully block the neighboring beams by the 3 mm entrance aperture or any of the preceding aperture lenses. This is also observed experimentally when injecting HfF_5^- and WF_5^- into the ion cooler (cf. Figure 4). With all components optimized for injection of mass 277 ($^{182}WF_5^-$), about 1% of $^{180}HfF_5^-$ (m=275) is still transmitted. Based on detailed studies of the ion optics, additional x-slits will be mounted between magnet and ion cooler to overcome this problem.

3. Results and Discussion

3.1 Transmission and stability

Among the major prerequisites of AMS is that unknown sample and reference sample are measured under identical conditions, implying that the total ion detection efficiencies both for stable and rare isotopes are constant. Slight current dependencies of measured isotopic ratios are nevertheless observed at many AMS facilities (e.g. [11]) and usually attributed to small variations in accelerator transmission. Unfortunately, the RFQ-ion cooler exhibits a strong

variation in transmission with ion current when stable reference isotope beams of several μA are injected. These are however often required in AMS in order to reach sufficient statistics of the rare isotope within reasonable time. This current-dependency of the RFQ has already been discussed in [7]. Higher ion currents lead to increased phase space of the ion beam when leaving the ion source and shorter residence times inside the RFQ. Furthermore, losses inside the RFQ increase as the stored charge approaches the charge limit of the RFQ and ions are increasingly getting pushed out of the potential well by space charge. Also the RF-heating of stored ions is growing once they are pushed away from the potential minimum in the cooler axis by space charge, resulting in higher ion energy spreads.

As these are fundamental limitations, no changes in this behavior were expected with integration of the cooler into the new injector. This is confirmed by transmission data for various $^{37}Cl^-$-currents in the μA range shown in Figure 3a. They were recorded during a routine AMS measurement with the ILIAMS setup lasting over 48 hours. To overcome the problem of current dependency of ratios, a phase space conserving beam attenuator is now employed to reduce the intensity of the stable reference isotope beam by a factor of 60. Figure 3b shows the transmission of the attenuated $^{37}Cl^-$ ion beams for the same set of data. With injected ion currents of only several tens of nA no more current dependency of transmission is observed. The reproducibility of individual samples in routine AMS measurements with normalization to the attenuated stable isotope is better than 3% and the whole system including the laser was tested to be stable over several days. The attenuator is inserted via a motorized translator, which takes roughly 5 s. This limits the minimum switching time between stable isotope and rare isotope ion beams. More details on measurement strategy and results of the first AMS-measurements of ^{36}Cl with ILIAMS can be found in [12].

3.2 Isobar suppression – fragmentation and formation of molecules:

The behavior of the RFQ cooler for the molecular anion system MgO^- and AlO^- has already been investigated on the ILIAS test bench [7]. However, this setup did not provide any mass analysis of the transmitted ion current. Hence, only upper limits for the isobar suppression factors based on the total ejected ion current could be given. The mass-composition of the extracted ion beam and the fraction of the anion under investigation therein could not be determined. Nevertheless, a suppression factor of $>10^4$ for MgO^- vs AlO^- could be demonstrated at 10.8 W transmitted power of the 532 nm laser [7].

The new ILIAMS setup benefits from the full mass analysis of the VERA injector and, if necessary, the entire subsequent AMS facility. This allows a detailed analysis of the composition of the ejected negative ion beam. Interestingly, fragmentation and formation of molecules as well as pickup of fragments by injected ions are all observed in pure He buffer gas. Figure 4a shows mass spectra obtained when injecting AlO^- at mass 43, MgO^- at mass 40 and 41 as well as $^{37}Cl^-$ into the ion cooler without laser light. The distinct peaks at masses 16 and 17 when injecting AlO^- and MgO^- clearly show that $^{16}O^-$ and $^{16}OH^-$ are both formed when oxygen-containing anions are injected. Since these ions have no stable trajectories inside the RFQ under routine operating conditions for masses around 40, the ion cooler was specifically set for the low masses. While the ILIAMS RFQ is not operated as a quadrupole mass filter but as an ion guide (i.e. no DC voltage is applied to the RF electrodes), only ions within roughly ±30% mass deviation from the tuned ion mass are efficiently transported through the RFQ for a given RF frequency and amplitude (see [7] for details and experimental data). Hence, the transmission for the injected masses 37 to 43 was below 1% during these experiments with RFQ settings optimized for ions around mass 16. The injector mass scans in Figure 4a were recorded using a Faraday cup placed directly after the accelerator for enhanced mass resolution. The plotted currents are thus convoluted with the charge state distribution of each ion species. The fact that the ratio between O^- and OH^- changes by almost an order of magnitude when injecting mass 41 ($^{25}MgO^-$ and $^{24}MgOH^-$) instead of 40 ($^{24}MgO^-$) is a very strong indication that injected elements rather than impurities in the He buffer gas are the main source for these anion species. Both scans were conducted from the same sputter sample after more than an hour of presputtering and less than 15 min apart. The only parameter changed was the ILIAMS magnet, which was switched from mass 40 to 41. Another interesting feature is the peak at mass 32, which was observed when injecting 1.5 μA of AlO^-. The same peak appeared in mass scans when injecting $^{24}MgO^-$ and tuning the ion cooler for mass 32. This peak indeed belongs to O_2^-, which was confirmed by identification of $^{16}O^+$, $^{16}O^{2+}$ and $^{16}O^{3+}$ after VERA's high-energy-side analyzing magnet and the absence of any other noticeable peak. We attribute this to the formation of O_2^- from single O and O^- originating from the dissociation of injected anions. As a quick verification experiment, also $^{16}O^-$ was injected into the ion cooler and resulted in the formation of 10 pA of mass 32. A $^{37}Cl^-$ beam in the ion cooler produces no peaks above background at lower masses.

Buffer gases other than He enhance certain reactions or provide reaction partners. These processes may boost elemental selectivity in addition to laser photodetachment but are possible sources of background as well. Negative ion gas reactions have been studied at eV

energies at University of Toronto in a similar RFQ setup, which aims at isobar separation for AMS solely by reactions of anions with buffer gas [13]. They report e.g. on the identification of YF_3O^- after the RFQ when injecting YF_3^- into NO_2 buffer gas. Furthermore, a strong suppression of WF_5^- versus HfF_5^- was observed in O_2 buffer gas [14]. This was attributed to the formation of oxyfluorides, although the exact processes remained elusive. Since such technique may enable measurements of the astrophysically interesting trace isotope ^{182}Hf [15,16], we decided to investigate the behavior of $^{182}WF_5^-$ in different buffer gas mixtures by mass analysis of the ejected ion current. The results are shown in Figure 4b. The sputter target used for this experiment was a 1:1:1 by weight mixture of HfF_4, W and PbF_2. The ILIAMS magnet was set for mass 277 although, as already mentioned above, the mass resolution of the setup in its present state is too low to fully suppress neighboring masses. Hence a distinct peak at mass 278 (mostly $^{183}WfF_5^-$) and even a small fraction of the much more intense $^{180}HfF_5^-$ peak (mass 275) is still visible in the spectrum. The different buffer gases were premixed in a small pressure bottle, which lasted for about 10 hours of operation at the typical buffer gas pressure of 0.25 mbar. This was more than sufficient as each mass scan in Figure 4b lasted less than 10 min, after which the buffer gas was exchanged. The dominant process is the pickup of O to form $^{182}WF_5O^-$, which even occurs at a noticeable rate in pure He (nominal purity ≥ 99.999 mol%). Addition of 1% N_2 to the He gas results in a clearly increased reaction rate, the original beam is reduced by ~30%. While there is ~40 pA less $^{182}WF_5^-$, the peak at mass 293 attributed to $^{182}WF_5O^-$ increases by just the same amount. The source of O for this process is likely to be O-containing molecules around mass 277. The presence of heavier collision partners in the form of N_2 buffer gas presumably leads to an increased destruction rate of these molecules and provides more reaction partners for $^{182}WF_5^-$. Deliberate addition of 1% O_2 to pure He buffer gas further enhances this reaction channel with >75% of the $^{182}WF_5^-$ transformed into $^{182}WF_5O^-$. At higher O_2 levels of 10% in the buffer gas, no more $^{182}WF_5^-$ is ejected from the RFQ. At the same time, also $^{182}WF_5O^-$ is now strongly reduced indicating either some further reaction to masses outside the investigated range or neutralization of these anions. The formation of $^{182}WF_4O^-$ was not observed in any of the buffer gases. In analogy to WF_5O^-, also the formation of HfF_3O^- from HfF_3^- in He buffer gas has been experimentally verified.

While reactions of isobaric molecular anion species are generally beneficial, there are also cases of the opposite. This happens, e.g., when molecular reactions contribute to the transmission of anions by evading selective photodetachment through quick enough transmutation once entering the laser interaction region in the RFQ. One example would be

C_3^- with an electron affinity EA of 1.995(25) eV [6], which is easily detached by our 2.33 eV Verdi laser during ^{36}Cl measurements. Collisions of C_3^- with pure He buffer gas can however result in the transmutation into C_2^- [12]. With an EA of 3.269(6) eV [6], this anion is immune against photodetachment by 2.33 eV photons. The reaction rate increases with ion injection energy into the buffer gas. In the absence of any mass filter between the RFQ cooler and injection into the accelerator, such processes may contribute to background in the final detection setup. With all these new results at hand, we started to assess isobar suppression factors in the RFQ cooler by both laser photodetachment and possible molecular reactions combined. Table 1 contains suppression factors recently achieved with our setup for three anion systems of interest for AMS. In most cases it was even necessary to deliberately spike test samples with isobaric contamination like MgO or S at the % level in order to determine the isobar suppression factor of the ILIAMS setup. Given the 10+ orders of magnitude suppression both for MgO^- vs. AlO^- and S^- vs. Cl^-, any further atomic isobar suppression in the detection system for these elements becomes obsolete. Thus, low charge states with high stripping yields can be employed. The full experimental details on the benefits and implications of this novel technique for ^{36}Cl AMS measurements at VERA are presented in [12].

4. Conclusions and Outlook

The ILIAMS setup has been fully integrated into the VERA facility, which now allows studies of isobar suppression by laser photodetachment at AMS sensitivity levels. The entire setup has been tested to be stable over several days of measurement and opens ample possibilities for studies of element-selective photodetachment as well as reactions of molecules with buffer gas and ambient molecular fragments at eV energies. Given the experimental results, we emphasize the importance of a mass filter after the RFQ prior to injection of the ion beam into the accelerator in order to achieve highest isobar suppression.

Further experiments are foreseen to investigate the degree of WF_5^- vs HfF_5^- suppression in a He-O_2 buffer gas to determine the feasibility of ^{182}Hf measurements. This anion system also illustrates one of the main experimental challenges of this novel technique regarding new trace isotopes: The electron affinities of many molecular anion species are still unknown or only theoretical calculations exist. Hence selection of an appropriate molecular system and corresponding cw-laser requires some experimental studies. In the case of Hafnium- and Tungsten-pentafluorides, the only experimental stake so far is the finding that the

photodetachment cross section for WF_5^- is a factor of 100 higher than for HfF_5^- at 266 nm [17].

In addition to the scarcity of electron affinity data, kinetic and chemical reactions of molecular anions in the RFQ may strongly enhance isobar suppression, but could also render certain anion species unsuitable for this technique. Among the promising candidates for applications of the ILIAMS technique in the near future are $^{53}MnO^-$ (1.375(10) [18]) / $^{53}CrO^-$ (1.221(6) [6]), $^{60}FeH^-$ (0.481(7) [6]) / $^{60}NiH^-$ (0.481(7) [6]), $^{93}ZrF_5^-$/ $^{93}NbF_5^-$ (experimental EA values given in eV where known).

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Table 1

anions	AMS-isotope	suppr. factor
MgO^- vs AlO^- (1.60 eV vs 2.66 eV)	^{26}Al	10^{11}
S^- vs Cl^- (1.60 eV vs 2.66 eV)	^{36}Cl	$3x10^{10}$
BaF_2^- vs CsF_2^- (EAs unknown)	^{135}Cs	$>10^4$

Experimental anion suppression factors by laser photodetachment measured with He-buffer gas and 10 W transmitted laser power at 532 nm (2.33 eV). Where known, experimental electron affinities are given [5,6]

Figure captions:

Figure 1: Schematic drawing of the ILIAMS setup and its location within the VERA AMS facility. Electrostatic components are shown in yellow, magnetic components in blue and components for ion generation and detection in green.

Figure 2: SIMION 8.1 simulation of the injection section of the RFQ ion beam cooler including the first 5 cm of the RFQ buffer gas section for neighboring mass beams: m=273 (black), m=274 (green), m=275 (red), m=276(cyan), m=277(blue). Lateral distances in the top view plot are magnified by a factor 2. The input ion beam parameters are based on phase space measurements at the object slits of the ILIAMS magnet. Calculated ion beam transmissions of 43% (m=275), 15% (m=274,276) and 1.5% (m=273,277) with all injection components optimized for mass 275 closely match the experimental situation with a mass resolution of $m/\Delta m = 150$.

Figure 3: Ion beam transmission of $^{37}Cl^-$ through the RFQ ion cooler at 0.31 mbar He as a function of injected ion currents during 48 hours of ^{36}Cl-AMS. The injected $^{37}Cl^-$current is measured in the Faraday cup in front of the RFQ cooler, and the RFQ transmitted $^{37}Cl^-$ current is measured in an offset-Faraday cup following VERA's injection magnet (cf. Figure 1). Data points stem from 20 min runs on different sputter targets, a full turn of the target wheel takes around 6 h, the start time of each turn is given in the plots. During a 20 min run, unattenuated and attenuated ion beam transmissions are measured almost simultaneously (less than 1 min apart) every 5 min. The plotted data points are averages over a run. (a) shows the current dependency of transmission for the unattenuated stable reference beam as a function of ion current. (b) shows the transmission values as a function of ion current for the same set of samples but with the fine hole sheet attenuator inserted. Other than this, no prior beam collimation out of the ion source is employed.

Figure 4: Mass scans of the anion beams ejected from the RFQ with the VERA injection magnet. (a) Comparison of mass scans with the RFQ optimized for transmission of O^- (m=16) when injecting MgO^-, $MgOH^-$, AlO^- and $^{37}Cl^-$ into pure He buffer gas. Note that the $^{16}O^-$ /$^{16}OH^-$ obviously changes with the amount of H-containing molecules injected. Hence, the source of hydrogen is unlikely to be impurities in the He buffer gas. (b) Comparison of mass spectra when injecting mass 277 ($^{182}WF_5^-$) and tails of neighboring masses into various buffer gases. Pickup of O by WF_5^- to form WF_5O^- is the most prolific reaction observed in this mass range.

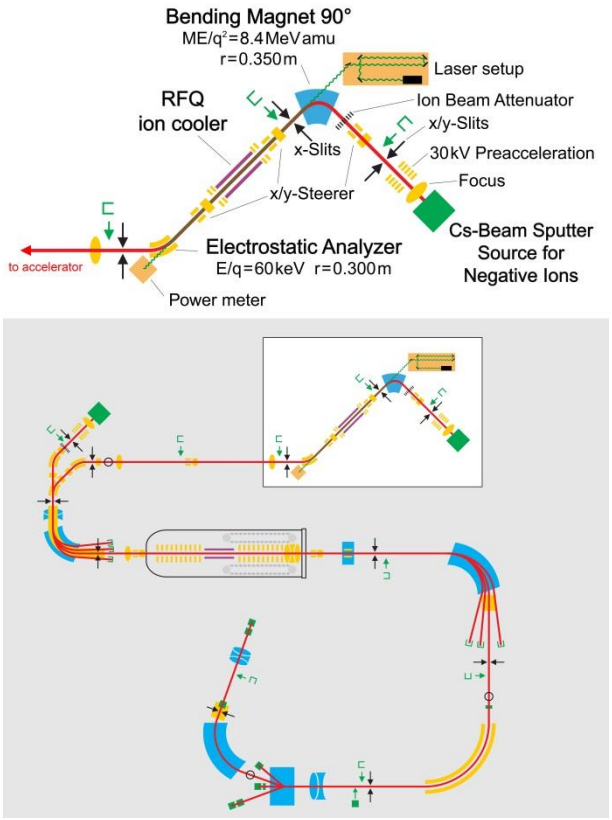
Bending Magnet 90°
ME/q²=8.4MeVamu
r=0.350m
Laser setup
RFQ
ion cooler
Ion Beam Attenuator
x/y-Slits
x-Slits
30kV Preacceleration
Focus
x/y-Steerer
Cs-Beam Sputter
Source for
Negative Ions
to accelerator
Electrostatic Analyzer
E/q=60keV r=0.300m
Power meter

Figure 1

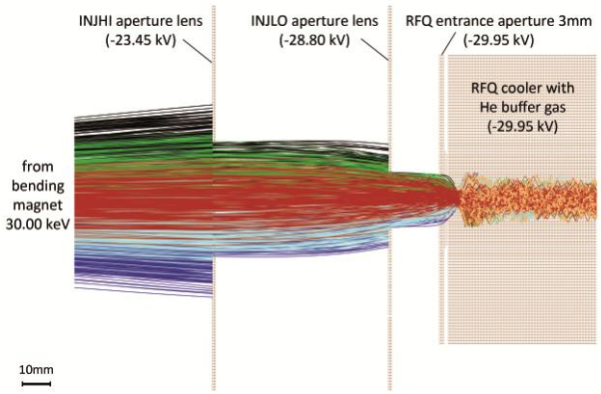
INJHI aperture lens
(-23.45 kV)
INJLO aperture lens
(-28.80 kV)
RFQ entrance aperture 3mm
(-29.95 kV)
RFQ cooler with
He buffer gas
(-29.95 kV)
from
bending
magnet
30.00 keV
10mm

Figure 2

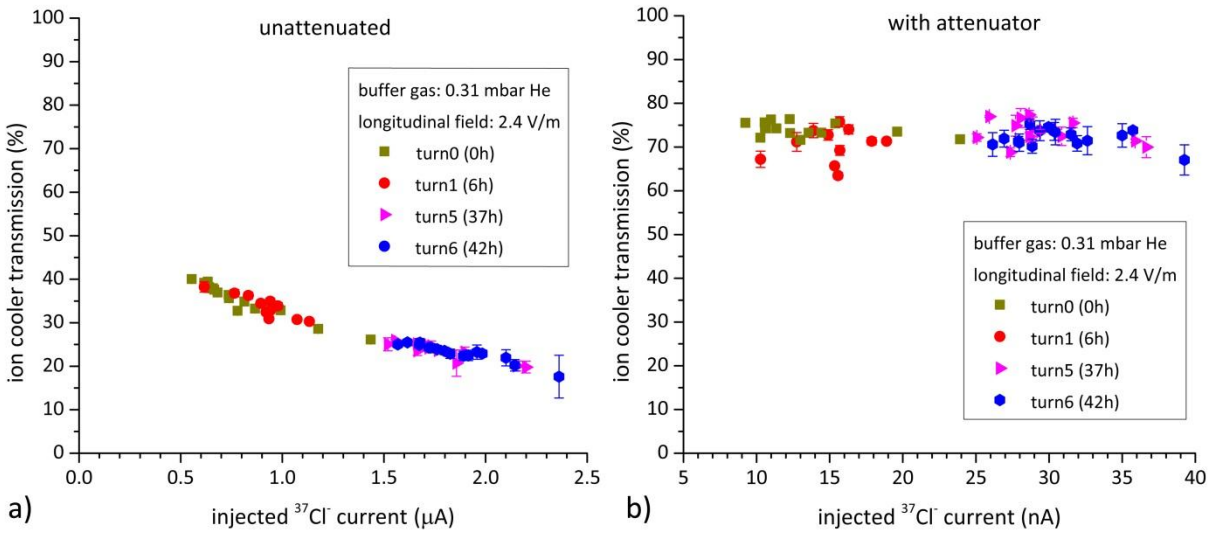
unattenuated
buffer gas: 0.31 mbar He
longitudinal field: 2.4 V/m
turn0 (0h)
turn1 (6h)
turn5 (37h)
turn6 (42h)
ion cooler transmission (%)
injected $^{37}Cl^-$ current (µA)
a)
with attenuator
buffer gas: 0.31 mbar He
longitudinal field: 2.4 V/m
turn0 (0h)
turn1 (6h)
turn5 (37h)
turn6 (42h)
ion cooler transmission (%)
injected $^{37}Cl^-$ current (nA)
b)

Figure 3

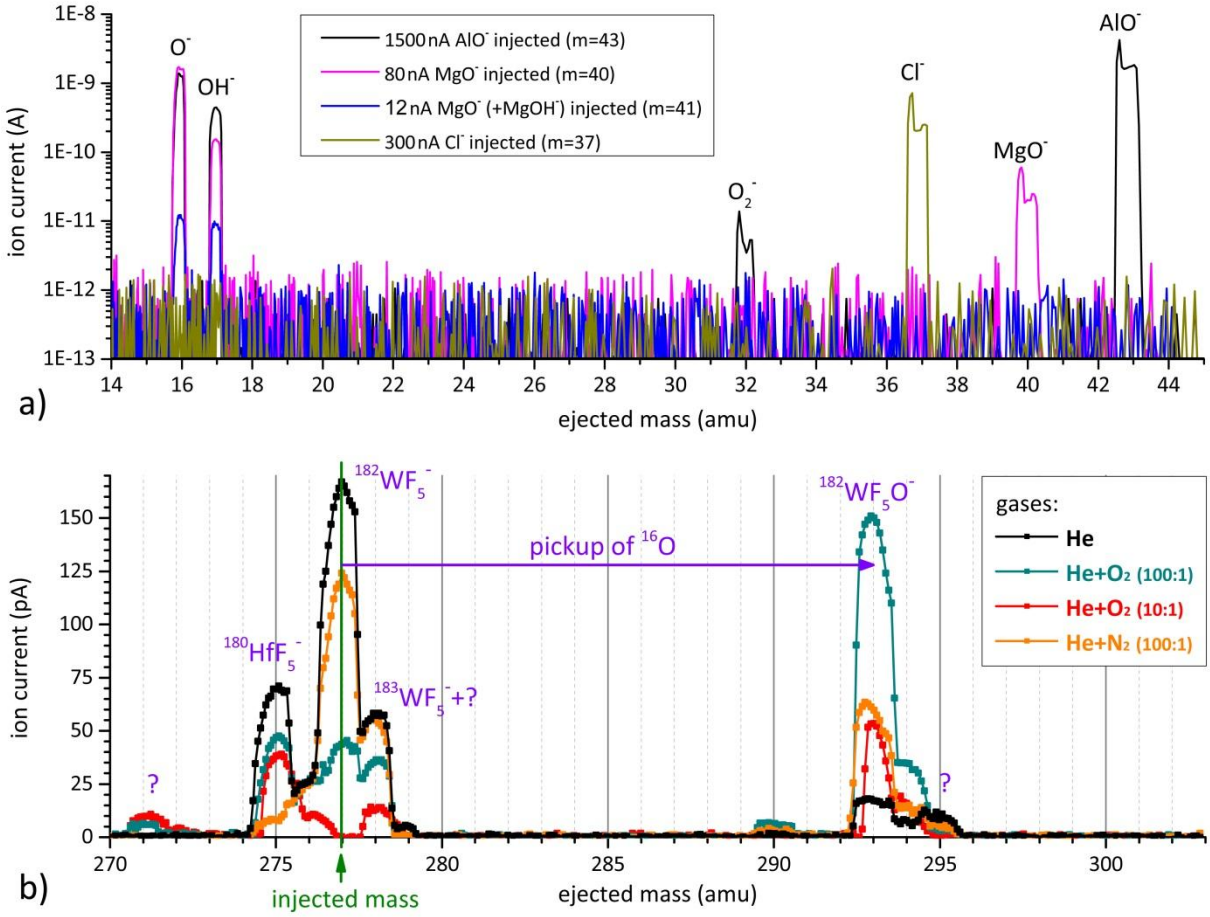
1500 nA AlO⁻ injected (m=43)
80 nA MgO⁻ injected (m=40)
12 nA MgO⁻ (+MgOH⁻) injected (m=41)
300 nA Cl⁻ injected (m=37)
O⁻
OH⁻
O2⁻
Cl⁻
MgO⁻
AlO⁻
ion current (A)
ejected mass (amu)
a)
182WF5⁻
pickup of 16O
182WF5O⁻
180HfF5⁻
183WF5⁻+?
?
?
gases:
He
He+O2 (100:1)
He+O2 (10:1)
He+N2 (100:1)
ion current (pA)
injected mass
ejected mass (amu)
b)

Figure 4