Atmospheric analysis using a microwave plasma ionization source and ion trap mass spectrometry

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Abstract

The on-line detection of both positive and negative ions has been demonstrated using a microwave plasma ionization source coupled with an ion trap mass spectrometer. Real-time xenon and krypton isotope measurements in air were made using either supplemental helium or atmospheric argon for plasma generation. The halogen signature from an organic molecule was detected as a negative ion using a helium plasma. Two instrument configurations have been assembled, one combining a microwave plasma source, an injection lens, and the ion trap; in the second assembly, a radiofrequency-only quadrupole mass filter was inserted between the lens and the ion trap. Most of the data reported here were acquired with the first configuration, although preliminary data with the second configuration has been taken. General operating parameters are discussed. The precision of xenon isotope ratio measurements ranged from 0.8% to 5% relative standard deviation, depending on the magnitude of the ratio. (Int J Mass Spectrom 178 (1998) 121–128) © 1998 Elsevier Science B.V.

1. Introduction

A recent report on a new detector for atmospheric xenon radionuclides is evidence that monitoring rare gases in the atmosphere is of timely interest [1]. Off-line methods for determining xenon and krypton in air use batch sampling methods and preconcentration or cryogenic steps in conjuction with mass spectrometry or gas chromatography/mass spectrometry [2–4].

On-line isotopic measurements for pure xenon and krypton gases were compared in a recent publication using two instruments, a quadrupole and a magnetic sector-field mass spectrometer, in combination with electron ionization [5]. Of the two, the sector instrument predictably produced measurements with greater accuracy and precision making it more suitable for discriminating between samples of different origin.

The desire for possible continuous xenon and krypton monitoring led to the choice of the rugged, highly sensitive ion trap mass spectrometer for the work described in this paper. Ion trap instruments have been developed for field use for applications ranging from the detection of volatile organic compounds in water by purge-and-trap gas chromatography/mass spectrometry [6] to direct sampling instru-

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Dedicated to the memory of Al Nier.

ments that permit rapid screening of environmental samples [7,8]. A microwave plasma source similar to the one discussed in this article was recently tested for rare gas isotopic analysis using a magnetic sector instrument [9]; Poths and Chamberlin report attaining 5% ionization efficiency routinely for pure Kr by using pure argon support gas and slightly lower efficiencies when analyzing for atmospheric Xe and Kr. The relatively high ionization efficiency provided by a microwave-induced plasma (MIP) source was an attractive advantage that we sought to exploit in direct monitoring. Operation of the microwave plasma source in the elemental analysis regime is another feature germane to direct analysis of Xe and Kr in air; possible isobaric interferences from airborne organic compounds can be eliminated, although under the appropriate conditions, a MIP source was used as a "soft" ionization source for organic compound determination [10].

A microwave plasma source was first combined with a quadrupole mass spectrometer in 1981 for the elemental analysis of solutions [11]. Olson and Caruso recently reviewed the expanding literature on MIP-mass spectrometry with attention to various sample introduction techniques [12]. MIP sources offer many advantages in almost all areas of analytical atomic spectroscopy including low operating costs, reduced power and gas consumption, increased sensitivity for high ionization potential elements, and choice of plasma gas [13,14].

The integration of the ion trap mass spectrometer with plasma sources is relatively recent. Researchers have explored the combination of both dc and rf glow discharge sources with the ion trap for the analysis of nonconducting and conducting solids [15,16]. Barinaga and Koppenaal combined an inductively coupled plasma ICP source with an ion trap for elemental and isotopic analysis [17]. Recent work by Eiden et al. incorporating an octopole ion guide/collision cell between the ICP and the ion trap has focused on the reduction or elimination of the problematic argon ion and other plasma-borne isobaric interferences that are typically seen with this source [18].

Most of our studies have focused on the combination comprising the MIP source, lens assembly, and ion trap. In our most recent work, a linear quadrupole operated in rf-only mode was inserted between the lens assembly and the ion trap and preliminary data has been acquired with that setup. Other researchers have investigated the use of a tandem quadrupole arrangement whereby a linear quadrupole precedes the ion trap; the linear quadrupole acts as a focusing device for externally generated ions entering the ion trap mass spectrometer. Morand and co-workers evaluated a tandem instrument in which a mass analysis quadrupole and an rf-only quadrupole used for beam transfer were interfaced with an ion trap; unwanted matrix ions could be removed from the beam injected into the ion trap by this method [19]. A hybrid combination has been used successfully by other researchers employing linear quadrupoles or octopoles for ion injection [20,21].

Instrument parameters were optimized for the sensitive detection of atmospheric xenon and krypton directly from air. The microwave plasma could be readily generated by using helium or atmospheric argon, and this utility was investigated. The rf voltage on the ring electrode during ion injection was a critical parameter; when this voltage was adjusted, i.e., when the electrodynamics trapping field was changed, resonances in Xe^+ and Kr^+ response were observed. This ion modulation effect, due to the interaction of supplementary fields with the main trapping field, has been investigated by many researchers [22–24]. The ion trap mass spectrometer was used to make isotope ratio measurements and results are discussed. Negative ion detection was demonstrated in the analysis of a chlorinated hydrocarbon.

2. Experimental

2.1. Instrumentation

A simplified schematic of the microwave plasma source-ion trap mass spectrometer is shown in Fig. 1. The first configuration referred to above is discussed here. The principal components of the system are the microwave discharge ionization source, the injection
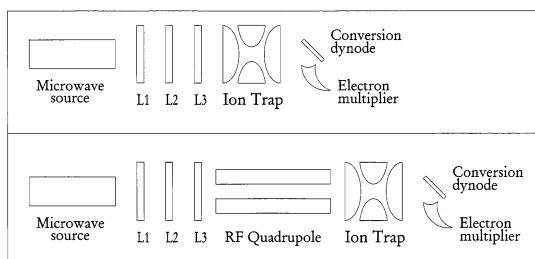
Fig. 1. The two MIP-ion trap configurations are shown here. In the second schematic, a linear quadrupole was inserted between the lens assembly and the ion trap mass spectrometer.

lens assembly, and the ion trap mass spectrometer. The lens assembly and the ion trap were housed in a stainless steel manifold. A 0.8-mm-diameter aperture in the manifold flange served as the interface between the plasma and the ion focusing region inside the vacuum manifold. Ions were focused through a custom-built three-element lens assembly and injected axially into a Finnigan ion trap mass spectrometer (Finnigan Corp., San Jose, CA). A Balzers turbomolecular pump (Pfeiffer Vacuum Technology, Nashua, NH; model TPU 510) backed by an Alcatel 21 roughing pump (Franklin Electric, Bluffton, IN) was used to achieve a base pressure in the manifold in the low 10^{-7} Torr range (uncorrected). A Varian Ion Gauge (Sugarland, TX, part no. 0564-K2500-303) and a buffer gas inlet were plumbed into the vacuum chamber.

Granville-Phillips variable leak valves (Boulder, CO; model 203) were used to regulate helium buffer gas pressure and air and ultrapure helium flow, if required, into the plasma gas feed line. Room air was leaked directly into the gas feed line supplying the MIP source for xenon and krypton analysis. Chemically active components of air (nitrogen, oxygen, carbon dioxide, and water) were removed from the air stream with a Sorbodyn-20 getter pump (Danielson Associates, Inc., Lisle, IL); inert gases such as xenon, krypton, and argon flowed unimpeded into the microwave plasma region. Direct analysis of xenon and kryton in air was not possible without getter pump removal of interfering active gases. Sample line pressure was monitored with a 10 Torr MKS Baratron

(MKS Instruments, Inc., Andover, MA). Room air as well as a certified gas standard (Matheson Gas Products, Inc., LaPorte, TX) containing 1.0 ppm each Xe and Kr in a helium matrix were analyzed. Negative ion detection was demonstrated in experiments conducted using a Dynacalibrator (VICI Metronics, Inc., Santa Clara, CA; model 340) to generate carbon tetrachloride vapor in a helium stream.

Three high voltage power supplies (Bertan Associates, Inc., Hicksville, NY; model 205B-03R-10R) were used to output dc voltages to the ion injection lenses, the lens support (located between the nearest injection lens and the entrance endcap), and the conversion dynode. A modification of the Scan/Acquisition Processor board in the Finnigan electronics inverted the +180 V gate pulse normally used during electron impact ionization. The 180 V gate signal was output to both halves of the split middle lens to permit positive ion penetration. For negative ion analysis, the gate pulse was restored to +180 V.

A Channeltron electron multiplier (model no. 4761G, Galileo Electro-Optics Corporation, Sturbridge, MA), permitting both positive and negative ion detection, was mounted in line following the ion trap exit endcap. High background noise, most likely due to energetic neutrals and photons from the microwave plasma hitting the electron multiplier, was observed initially whenever the plasma was turned on. The ion trap exit endcap (seven holes) was exchanged for an entrance endcap (single hole) and the electron multiplier detector was moved off-axis in a successful effort to reduce the interfering background noise. An ac potential was supplied to the two endcaps for axial modulation.

For the second configuration noted above a linear quadrupole from a residual gas analyzer (Model 100C, UTI Instruments, San Jose, CA) was inserted between the lens assembly and the ion trap. The quadrupole mass filter was controlled through the UTI electronics and was operated in a fixed rf-only mode. The same MIP source was used in this setup; the diameter of the opening between the source and focusing region was 3 mm in this case. The injection lens for the second configuration was assembled using

manufactured ion optics components (Kimball Physics, Inc., Wilton, NH).

2.2. Microwave plasma source

The plasma source used in our experiments is based on a concept by Vorburger et al. [25] with further refinements by other groups [26,27]. The ionization chamber consists of a ceramic ($A1_2O_3$) tube 1.3 cm o.d. $\times$ 10 cm long surrounded at one end by the microwave cavity. A microwave power generator (Model MPG 4M, Opthos Instruments, Inc., Rockville, MD) was operated at 10 to 12 W output, which provided the most stable signal. Operation at higher power reduced the rare gas response. A tesla coil was used to initiate the plasma in the source.

2.3. Parameters and operating conditions

We generated ion trap scan functions by using the Scan Editor program in the Finnigan software (Rev. B). A reasonable signal for Xe and Kr in air was produced by using a 200 ms ionization period during which the -180 V gate pulse was applied to the middle injection lens. The amplitude of the rf potential on the ring electrode during ion injection was chosen after a series of parametric experiments (see Sec. 3, Results and discussion for additional discussion); the rf potential was set to correspond to m/z 64 (corresponding to approximately 750 V_{0-pk} on the ring electrode) to maximize the detection of xenon, which has an abundance in air approximately 13 times less than krypton (87 ppbv versus 1.14 ppmv). The delay period following ion injection was set at 1 to 1.5 ms; longer delay periods (>8 ms) resulted in loss of krypton signal. An axial modulation signal of 486 kHz, 2.4 V_{pk-pk}, was applied to the endcap electrodes throughout the entire scan function period. Helium buffer gas was brought into the vacuum manifold through a leak valve at a pressure of approximately 6 to 8 $\times$ 10^{-5} Torr (uncorrected). The conversion dynode potential was set at -5 kV; the electron multiplier potential was 1500 V.

Optimization experiments were conducted to establish satisfactory operating conditions with maximi-

zation of xenon signal relative to krypton. An ionizing plasma could be generated using either supplemental helium or atmospheric argon from getter-pumped air. Reduced baseline signal was obtained by using 0.7 to 0.8 Torr helium in the gas feed line; however, eliminating the supplemental support gas and using an atmospheric argon-initiated plasma simplified instrument operation and further reduced baseline noise. The working air pressure range in the argon plasma mode was measured at 0.25 to 0.35 Torr in the plasma feed line.

For negative ion detection, power supply polarities were reversed so that the lens components, lens support, and dynode were biased positive. Operating parameters were adjusted to obtain an ion signal for Cl^-, but not exhaustively examined.

3. Results and discussion

3.1. Detection of Xe and Kr in air using the MIP, injection lens, and ion trap assembly

During initial optimization experiments when the main rf potential during ion injection was adjusted, resonances were observed in the Xe^+ and Kr^+ ion signals, that is, differences in ion efficiencies correlate with the amplitude of the rf voltage. These discrimination effects have been noted by other researchers studying ion injection in the quadrupole trap [22–24] and are due to the interaction of higher order fields with the quadrupolar trapping field.

In Fig. 2, the background-subtracted responses for a major Kr and Xe ion are plotted against the fundamental parameter β. These data were acquired in a series of experiments using the certified helium standard and matrix helium was used to generate the ionizing plasma. Ions were injected at different values of the fundamental rf voltage, which correspond to different values of β_{inject}. The values of β_{inject} were calculated by using the approximation described by Carrico [28]. An earlier study by Williams et al. [24] showed that maximum injection efficiency occurred at $\beta_{inject} = 0.1$. The present study intentionally avoided these low values of β_{inject}, because, at the rf voltage
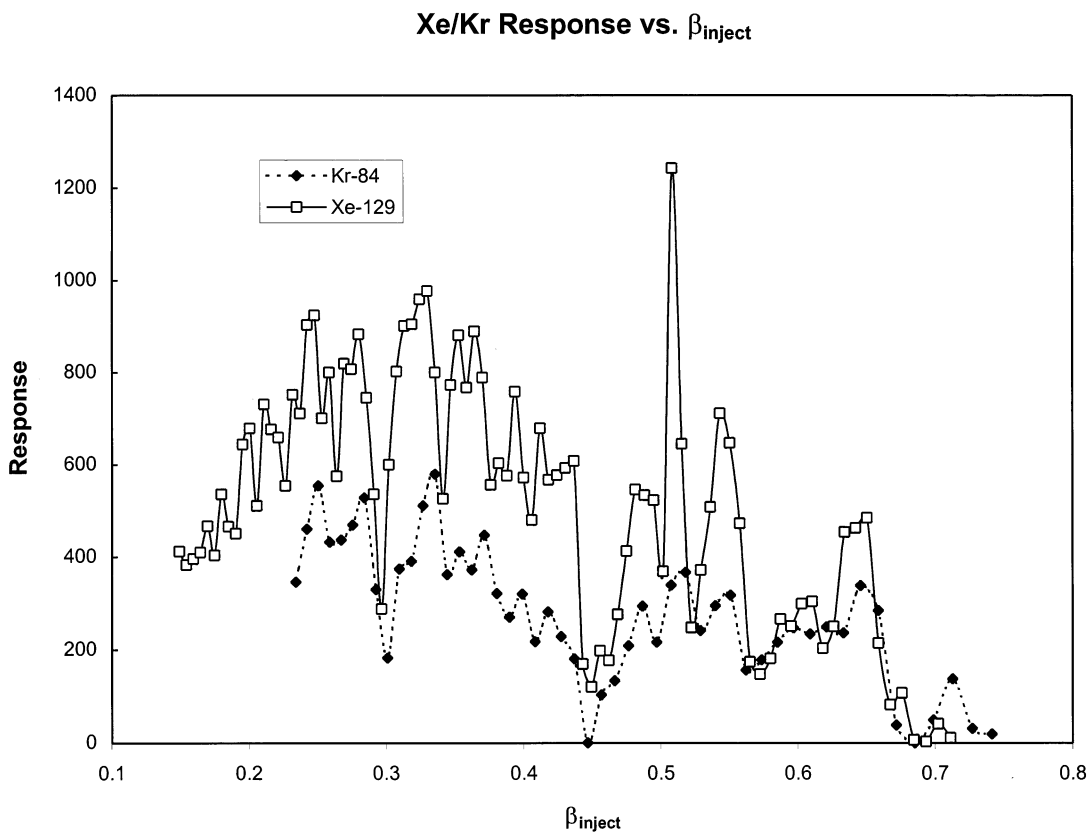
Xe/Kr Response vs. β_{inject}

Fig. 2. The response for two of the major rare gas isotypes vs. β_{inject}. ^{84}Kr is represented by the solid black circles; the response for ^{129}Xe is shown by the open white squares.

corresponding to β_{inject} = 0.1, nitrogen from air is efficiently stored in the ion trap. This, in turn, can lead to space-charge effects and unwanted ion-molecule reactions. Several maxima in both Kr (1.4 ppm and Xe (87 ppb) responses were observed. At a given fundamental rf voltage, the β_{inject} values for Kr and Xe are different. If the simultaneous detection of both elements is desired, a compromise value for the rf voltage during injection, which governs the low-mass storage cut-off value, must be used. At a low mass cut-off during injection of 64 Da, both xenon and krypton were detected at their natural concentrations in air. This low mass cut-off value corresponds to β_{inject} = 0.55 for ^{84}Kr and β_{inject} = 0.33 for ^{129}Xe (Fig. 2). The same resonance experiments were repeated later by using an argon-initiated plasma in the direct analysis of atmospheric Xe and Kr. Similar maxima and minima as shown in Fig. 2 were observed.

The rare gases in air were ionized by a microwave plasma supported with either ultrapure helium or argon from room air pumped through the getter; the ionized species were then injected into the ion trap to produce a stable signal for atmospheric Xe and Kr. Figure 3 shows a mass spectrum for the two inert gases obtained by using an atmospheric argon-initiated plasma. Many of the naturally occurring isotopes for krypton (m/z 82, 83, 84, and 96) and xenon (m/z 129, 130, 131, 132, 134, and 136) are seen in the plot. The small peaks at m/z 79 and 81 have been tentatively assigned to bromine and the peak at m/z 127 to iodine. The peak at m/z 80 is most likely the argon dimer ion.

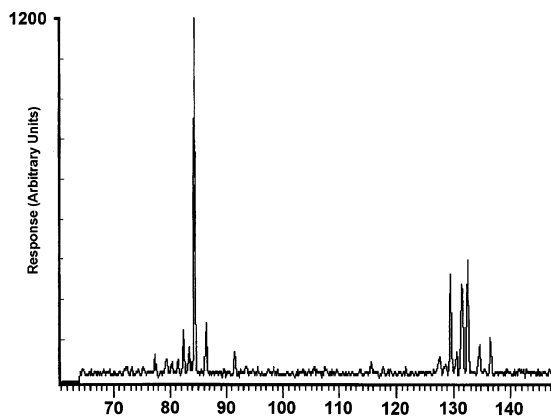The natural abundance in air of the major xenon

Fig. 3. In this mass spectrum of naturally occurring krypton and xenon in room air, argon, which occurs at 0.93% abundance in the earth's atmosphere, was used to initiate the plasma.

isotopes ratioed to ^{129}Xe is shown in Table 1. In the second row, the observed isotope ratios (relative to ^{129}Xe) are ratioed to natural isotope ratios. A number other than 1 indicates the difference from natural abundance that was seen in the ion trap mass spectrometer. Although the observed isotope abundances differ from natural concentrations, our main concern in these experiments was with the degree of precision that could be obtained and not necessarily the accuracy.

Sample flow rate, i.e. the amount of air being leaked into the instrument per unit of time, is important to optimize for best ionization efficiency. Air flow rates into the sample line were measured directly using a bubble flow meter. Flow rates for air ranged from 0.2 to 0.45 ml/min into the plasma region; the latter rate was measured when conditions were optimized for the atmospheric argon plasma. Many factors contribute to the overall efficiency of the instrument for rare gas detection. Among these are the source efficiency, that is, the ratio of ions created to total available atoms. The efficiency of a similar MIP source was measured at 2.1%—5% for Xe in air [9]. It is likely that the higher flow rates and concomitant higher source pressures of these experiments reduce that ionization efficiency by 10- to 100-fold. The efficiency of the guiding optics, injection into the ion trap, and trapping efficiency (of ions entering the trap, the number that attain stable orbits) are other important factors that reduce the final number of atoms of xenon that are detected per unit time. Injection efficiencies from an external source into the ion trap of 10%, and even higher with specialized techniques, have been reported [29–31]. Based on the ionization efficiency and signal response in these experiments, ion injection and trapping efficiencies are estimated to be about 1% or slightly higher.

The degree to which precision improved with instrumental changes in the microwave plasma ion trap can be reviewed in Table 2. Room air was directly sampled for all measurements with supplemental helium serving to initiate the microwave plasma. Each series of results summarizes data from five independent measurements of xenon concentration in air. The average background-subtracted response for each isotope was ratioed to averaged ^{129}Xe signal. The precision in the original configuration ranged from 3.3 to 24.8% relative standard deviation. The endcap change and off-axis adjustment of the

Table 1
Comparison of the observed isotope ratios for Xe obtained with the ion trap mass spectrometer (relative to ^{129}Xe) with expected ratios. In the last row, deviation from 1 indicates the degree of difference between observed and natural ratios

Isotope of Xe	128	129	130	131	132	134	136
Natural abundance (ratioed to ^{129}Xe)	0.07	1	0.16	0.80	1.02	0.39	0.34
Observed/natural	2.82	1	1.36	1.07	0.90	0.77	1.05

Table 2
Each number represents the % relative standard deviation obtained from a series of five independent measurements. In each case, average response at a particular isotope was ratioed to averaged ^{129}Xe response

Xenon isotope ratio measurements	128	130	131	132	134	136
% RSD[a]	24.8	9.3	3.3	4.1	5.9	9.1
% RSD[b]	5.0	2.7	1.2	0.8	0.9	1.8

[a] Data were obtained with a helium plasma and room air.
[b] Data obtained using helium plasma and room air. The electron multiplier was moved off axis and the exit endcap exchanged for an entrance endcap.

electron multiplier significantly improved isotope ratio precision, with relative standard deviations (RSDs) ranging from 0.8 to 5.0% due to the reduced background noise. Similar improvements were noted for krypton isotope ratio measurements. When room air was used to support the plasma, the improved precision was maintained with RSDs ranging from 1.1 to 3.9% for xenon and 0.9 to 3.6% for krypton.

3.2. Negative ion detection

Detection of negative ions may offer advantages in the analysis of some types of materials. The elements with the highest electron affinities are the halogens such as fluorine, chlorine, and iodine. Halogen signatures from the plasma ionization of volatile chlorocarbons may be more readily detected in the negative ion made. Configuring the lens assembly to transmit negative ions was readily accomplished. This capability was demonstrated in the analysis of carbon tetrachloride. When CCl_4 vapor was swept into the microwave plasma region in a helium stream, the distinctive isotopic pattern for Cl^- at 35 and 37 Da was seen.

3.3. MIP, injection lens, linear quadrupole, and ion trap assembly

To further decrease background noise, a quadrupole mass filter was inserted between the lens assembly and the ion trap and tested with a fixed rf voltage. Averaged mass spectra from the data acquisition program are compared in Fig. 4. The mass spectrum shown in (A) was acquired with the original setup, i.e., without the linear quadrupole in place. In (B), the mass spectrum was obtained by using the tandem configuration. Data in both cases were obtained from the analysis of room air using atmospheric argon to generate the plasma. With the mass filter in place, the later mass spectrum shows much less interference from the background signal that extends across the scan range in (A). The signal-to-noise ratio in (B) has improved by at least an order of magnitude.
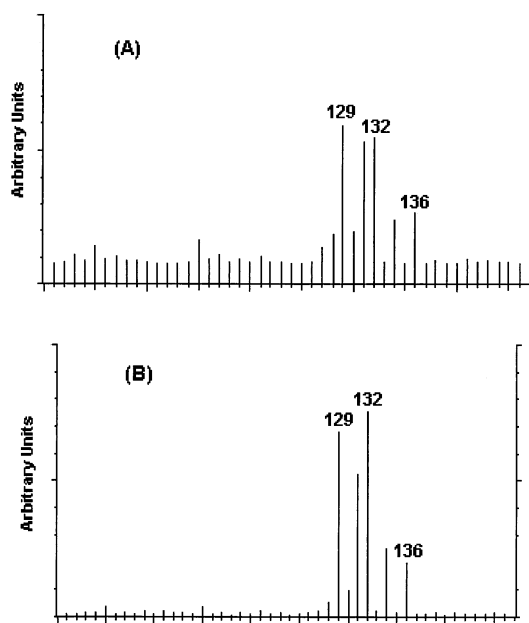

Fig. 4. Mass spectra of xenon and krypton in room air obtained with two different instrument setups are shown for comparison. In (A), the configuration consisted of microwave plasma, lens assembly, and ion trap. In (B), a linear quadrupole operating in rf-only mode was inserted between the lens assembly and the ion trap, resulting in a cleaner signal with reduced background interference.

4. Conclusion

The results shown here demonstrate the successful integration of a microwave induced plasma source and an ion trap mass spectrometer and point toward a promising tool for elemental isotopic analysis. The precision of isotope ratio measurements improved as changes were made to the system; these measurements indicate an emerging area for further study in ion trap research. The insertion of a quadrupole mass filter between the ion optics and the ion trap has reduced background interference from the plasma substantially. Further study with this configuration is needed to fully characterize the resolution and isotope ratio precision. Additional refinement and development remains to be done, especially in the latter area, in order to achieve high precision in isotopic ratio analysis. Sources contributing to the instability of the signal need to be identified. Possibilities may include inconsistencies in sample flow related to the sample

and gas flow setup, and incomplete getter pump removal of contaminating active gases. The physical-mechanical aspects to be further studied include orifice dimension, lens optics, and the spacing between components.

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