

Mass Spectrometry of UF₆ in a Micro Ion Trap*

William B. Whitten, Peter T. A. Reilly, Guido Verbeck, and J. Michael Ramsey

Oak Ridge National Laboratory
Oak Ridge, Tennessee, USA 37831-6142
whittenwb@ornl.gov

ABSTRACT

We have been exploring the miniaturization of ion trap mass spectrometry with cylindrical ion traps as small as 0.5 mm. One application we are addressing is the isotopic analysis of UF₆. Besides the usual problems of mass resolution and instrumental sensitivity, the chemical reactivity of UF₆ adds additional restrictions on construction materials, ionization sources, and detectors. Results of experiments at the ORNL UF₆ test loop are reported.

Key words: uranium isotope ratios, mass spectrometry

* Research sponsored by the U.S. Department of Energy, National Nuclear Security Administration, Office of Nonproliferation Research and Engineering, and Office of International Safeguards, under contract DE-AC05-00OR22725 with Oak Ridge National Laboratory, managed and operated by UT-Battelle, LLC.

INTRODUCTION

Mass spectrometers are usually considered to be laboratory instruments because of their size, weight, and power requirements. However, we have shown that ion trap technology can be used to perform mass spectrometry using electrode structures of submillimeter dimension. In scaling down the trap dimensions, other parameters such as vacuum pumping requirements, operating voltages and frequency, and overall system size and weight all become more favorable, making possible a fieldable instrument that should be hand portable for use as a survey instrument. Our plan is to incorporate this micro ion trap mass analyzer in a hand-portable mass spectrometer system for isotopic analysis of UF_6 . The portable mass spectrometer is under development for DOE's Office



Figure 1. Battery-powered prototype micro ion trap mass spectrometer.

of Nonproliferation Research and Engineering. A photograph of the battery-powered prototype is shown in Figure 1. The mass spectrometer system consists of a battery power supply, electronics and data package, a scroll and turbo vacuum pump, vacuum chamber, and ion trap analyzer. The latter has an electron gun ionization source, three electrodes to form a 1-mm diameter ion trap, some focusing electrodes and grids, and a channel-electron multiplier ion detector. During operation of the instrument, the pumps, interior of the vacuum system, and the components of the mass analyzer are exposed to the vapor being analyzed. The present study was designed to explore the suitability of various components and structural materials for operation in the presence of highly reactive UF_6 vapor.

The micro ion trap mass spectrometer is based on cylindrical rather than hyperbolic geometry for ease of fabrication. The trap consists of a ring electrode with 1-mm diameter hole and two end cap electrodes, as shown in Figure 2. For operation as a mass analyzer, a hot filament electron gun for sample ionization and a channel electron multiplier for ion detection are added. Ions can be trapped within the ring electrode if a radiofrequency voltage of suitable amplitude is applied to the electrode assembly.¹ A mass spectrum of the trapped ions is generated by ramping the voltage applied to the electrodes. At a certain voltage that depends on the mass of the ion, the ion trajectory becomes unstable and ions of that mass that are present in the trap escape to the detector. The resulting detector signal versus voltage can be converted to a mass spectrum.

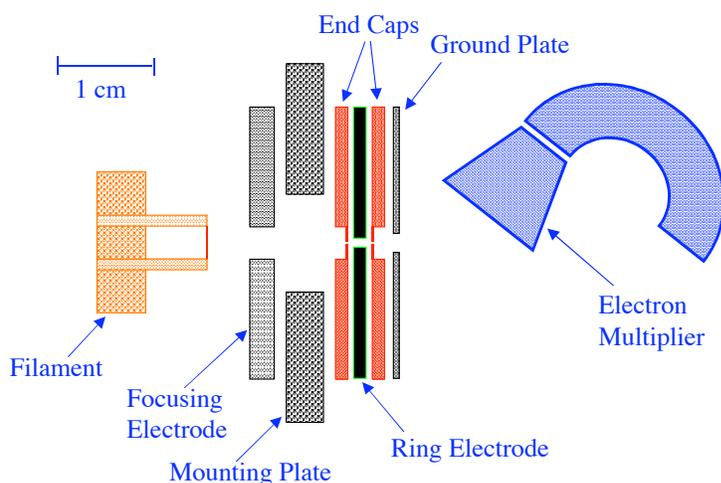


Figure 2. Micro ion trap mass analyzer.

Mass resolution of the instrument can be improved by adding a supplemental alternating voltage applied axially between the two end cap electrodes.² Ions are ejected from the trap with high efficiency when the frequency of the supplemental voltage is simultaneously resonant with the secular frequency of the ion and with a nonlinear resonance of the cylindrical trap. We have called this process double resonance ejection.² The ion signal integrated over the entire mass spectrum is essentially

independent of the resonance condition because the total number of ions in the trap depends only on the sample pressure and electron beam current. However, because of the increased mass resolution, the height of the isotopic peaks becomes much higher when the double resonance condition is satisfied. An example of a mass spectrum obtained in this way is shown in Figure 3. This is a spectrum of the isotopes of xenon with a mass resolution of approximately 1000.²

EXPERIMENTAL

The purpose of the present investigation was to study the effects of exposure to uranium hexafluoride vapor on the sensitive components of a micro ion trap mass spectrometer. Since any components exposed to UF_6 vapor become radiologically contaminated to some degree, we constructed an ion trap test chamber and vacuum system that could be used for the compatibility study and an identical system for developmental work in our nonradiological laboratory. A drawing of the micro ion trap analyzers constructed for the tests is

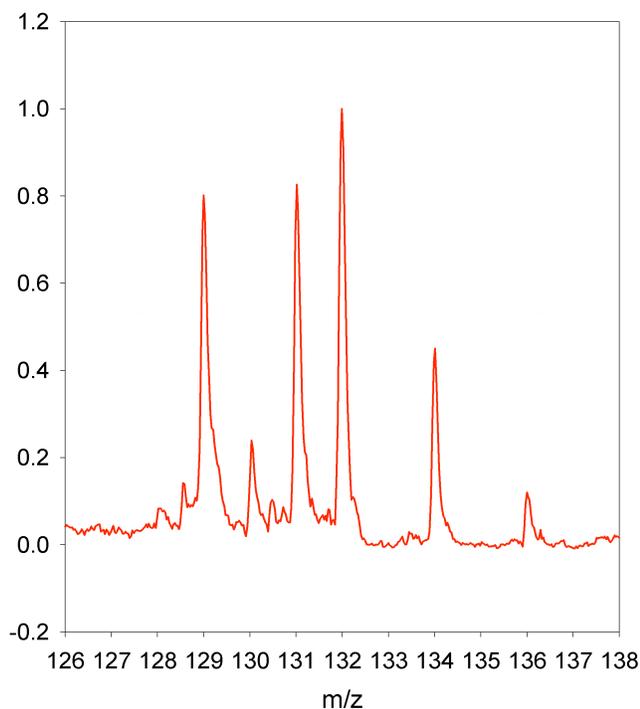


Figure 3. Mass spectrum of xenon isotopes.

shown in Figure 4. From right to left, a tungsten filament and 3-element Einzel lens form the electron gun for electron-impact ionization, three electrodes form a 1-mm diameter quadrupole ion trap, followed by a grid and channel-electron multiplier for ion detection. A 70-l/s turbo-drag pump was mounted on the vacuum chamber with an ion gauge to monitor the internal pressure. Connections to the various components from the electronics instrumentation are via vacuum feedthroughs so only the inside of the chamber and its contents are subject to contamination. A photograph of the analyzer assembly is shown in Figure 5.

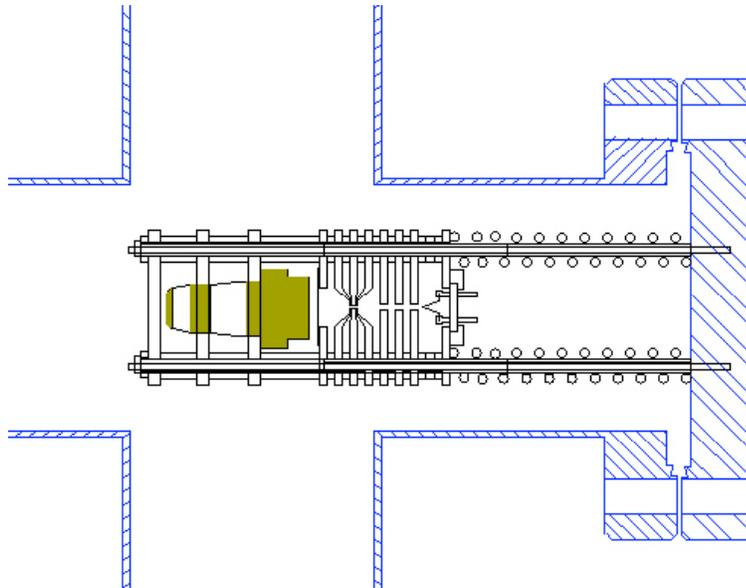


Figure 4. Diagram of the micro ion trap mass analyzer test assembly, mounted on a 4.5-inch flange.

The components that are most sensitive to surface reactions are the electron multiplier/detector, filament of the electron gun, and the turbo pump with aluminum blades operating at close tolerance. The electron multiplier relies on secondary electron emission at the surface to develop its gain and surface conductivity to establish the electric field within the device. The filament operates at elevated temperatures and is of small diameter.

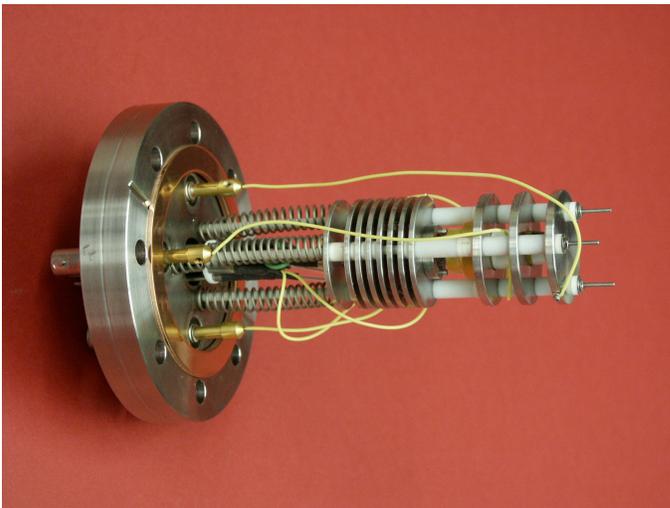


Figure 5. Photograph of the ion trap mass analyzer.

We have shown previously that the electron gun in combination with the ion detector can be used as a pressure gauge. Ions generated by electron impact are detected with gain by the channel electron multiplier. These measurements can be made without the radiofrequency instrumentation required to operate the ion trap. Since all of the critical components are involved in such measurements, these measurements represent a simple way to determine the compatibility of the components with UF_6 vapor.

The assembly shown in Figures 4 and 5 was mounted on a small rack containing the necessary power supplies and measuring instruments and transported to the UF_6

test loop at Oak Ridge National Laboratory. At this facility, we admitted a small amount of UF_6 to the chamber through a leak valve while pumping continuously. To study compatibility, we maintained a pressure of 1×10^{-5} Torr for nearly 2 hours while the pressure was monitored both with the electron gun – multiplier experiment and with a commercial ion gauge. This pressure is the largest sample pressure that we normally use for a mass spectral measurement. The results of this experiment are shown in Figure 6, where the multiplier output current is plotted versus time. The current declined initially while the detector surface was responding to the UF_6 vapor but then approached a steady state value. An isotope ratio determination with the ion

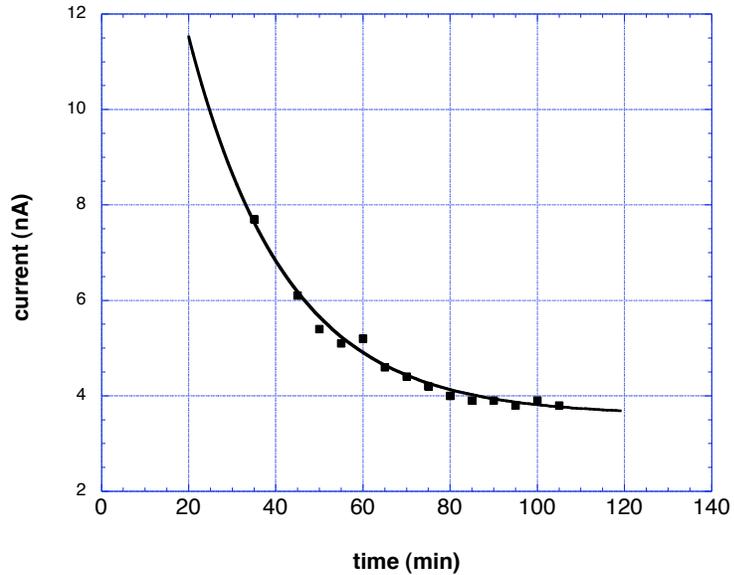


Figure 6. Time dependence of the detector current.

trap can be made in 10 to 100 seconds depending on the desired precision, so two hours

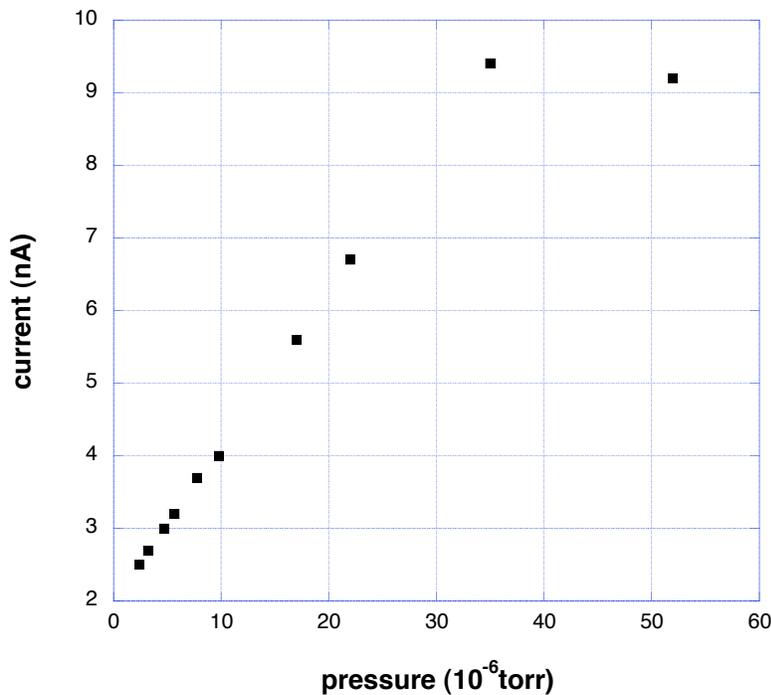


Figure 7. Pressure dependence of detector current.

represents a large number of determinations. However, the approach to steady state implies that the surface changes had equilibrated at the maintained UF_6 pressure so that operation for extended times is likely.

The second experiment at the termination of the first was to measure the detector current as a function of UF_6 pressure, similar to the previous experiments we had done with nitrogen and helium gas. The results of this experiment are shown in Figure 7. The current increased linearly with pressure up to the 1×10^{-5} Torr value where the components had been passivated. At higher pressure, the gain decreased due to surface

modification at the higher exposure, and at 5×10^{-5} Torr, the current was visibly decreasing with time. It is likely that the gain would again approach a steady state value but since this pressure was much higher than would be used in a mass spectral measurement, we terminated the experiment. After pumping the system over a weekend, the multiplier gain partially recovered but again decreased at higher UF_6 pressures.

CONCLUSIONS

The results of these experiments show that chemical reactions of UF_6 and its decomposition products with the mass spectrometer components should not impair measurements. Furthermore, UF_6 responds to electron impact ionization and ion detection much as other gases that we have studied in similar experiments.

ACKNOWLEDGEMENT

This research was sponsored by the U.S. Department of Energy, National Nuclear Security Administration, Office of Nonproliferation Research and Engineering, and Office of International Safeguards, under contract DE-AC05-00OR22725 with Oak Ridge National Laboratory, managed and operated by UT-Battelle, LLC. We would like to thank Lee Trowbridge for the use of the test loop facility and Darrell Simmons for his assistance in performing the experiments.

REFERENCES

- ¹ O. KORNIENKO, P. T. A. REILLY, W. B. WHITTEN, J. M. RAMSEY. Rev. Sci. Instrum. 70 (1999) 3907.
- ² J. MOXOM, P. T. A. REILLY, W. B. WHITTEN, J. M. RAMSEY. Rapid Commun. Mass Spectrom. 16 (2002) 755.