Rapid Ion Trap Mass Analysis of Inorganic Water Clusters Separated by Differential Mobility Spectrometry

Timothy Vazquez; Theresa Evans-Nguyen

Department of Chemistry, University of South Florida, Tampa, FL 33620

Overview

Purpose: Inorganic separation of radionuclide analogs through differential mobility spectroscopy and detection using an ion trap mass spectrometer.

Methods: Interfaced a home-built differential mobility spectrometer (DMS) with a custom built ion trap mass spectrometer. Ions are introduced by nanospray to the DMS. Control electronics from a Sionex unit were used to apply the dispersion and compensation voltage potentials to the DMS electrodes through the Sionex Expert software. Introduction of ions to the ion trap is done through the use of a capillary directly through the ring electrode of a 3-D quadrupole ion trap. We operate the quadrupole ion trap by frequency scanning the fundamental RF in a continuous mode of introduction and analysis.

Results: Implementation of high-speed digital frequency scanning into quadrupole ion trap. Design, fabrication, and interfacing of DMS components to a custom quadrupole ion trap.

Introduction

DMS: Inorganic separation using traditional chromatography often requires long run times. This type of analyses while valuable is not compatible with rapid speciation of inorganics in a fieldable setting. DMS however, has been demonstrated to effectively pre-filter using an orthogonal separation mechanism to the mass spectrometer (MS).[1] This removes the need for chromatography for select species. DMS offers enhanced sensitivity for targeted chemical detection. Sensitivity is enhanced by rapidly suppressing chemical noise prior to MS introduction thereby lowering the performance requirements of the MS. This feature can be capitalized on for portable instrumentation that allows for field analysis using MS. Potential applications include process analytical technologies, medical diagnostics, and explosives detection.

Ion Trap: The quadrupole ion trap MS is operated using previously studied[2] high-speed digital frequency scanning. This scanning mode allows for larger mass ranges and high acquisition speeds as fast as 1 ms/spectrum which facilitate the fast run times of the DMS. Instrumental development of DMS interface to a custom built ion trap MS is described herein.

Methods

High-Speed Digital Frequency Scanning:

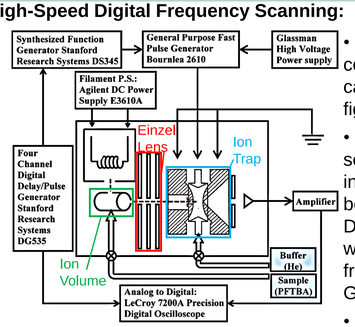

Figure 1: Experimental block diagram. Adapted from reference 2.

- Electronics for continuous mode can be seen in figure 1.
- Instrumental setup of DMS interface to DIT can be seen in figure 2. DIT components were repurposed from a Finnigan GCQ Plus.
- Reference standards are introduced through Ion volume.

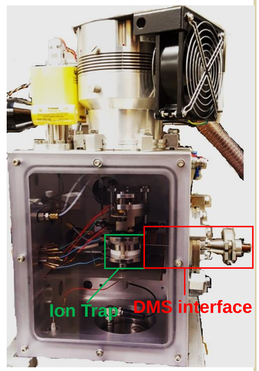

Figure 2: Quadrupole Ion Trap with DMS interface

Methods – Xenon Reference Standard

LeCroy Wavepro 7200A Precision Digital Oscilloscope was used to obtain spectra for Xenon which can be seen in figure 3.

- Xe partial pressure: 1.20 x 10^{-7} torr
- He partial pressure: 1.64 x 10^{-3} torr
- Filament current: 1.17 A
- Detector voltage: 2275 V
- Cycle rate: 6 Hz
- Scan Rate: 60 Hz
- Fundamental voltage: 500 V_{rf}
- Supplemental voltage: 0 V

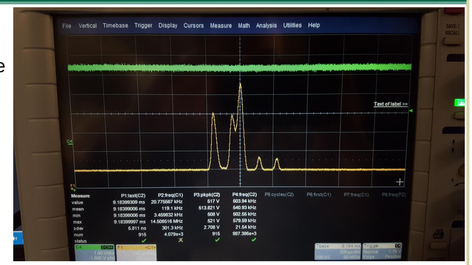

Figure 3: Xenon standard with He as buffer gas

Results - DMS Setup

DMS Flange: A Vespel flange encased two stainless steel electrodes which can be seen in figure 4. The electrodes inlet ions through a volume consisting of the following dimensions 4 mm wide, 0.05 mm height, and 15 mm deep. The flange also contained counterbored holes for screws to position the electrodes while also providing the necessary voltage via the repurposed Sionex unit. An o-ring slot as well as four fastening holes to the base of the flange to secure it to other instrumentation.

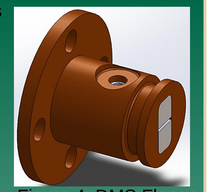

Figure 4: DMS Flange

Figure 5: DMS Interface

DMS Interface: Seen in figure 5, a custom machined 7075 aluminum interface is supported by a KF 25 flange and allows the inlet of atmosphere through 1/4" tubing. Fastening bore holes inserted for DMS flange positioning and sealing.

DMS Interface to DIT: DMS flange and interface joined with M3 screws and Viton 1/16" fractional width o-ring. 1/4" to 1/16" Yor-lok reducing straight union followed by a 5" length of stainless steel capillary. Different capillary inner diameters (ID) were tested with best results obtained at an ID of 0.005". Capillary inlets into machined 3-D quadrupole ion trap while being insulated with a PEEK fitting that can be seen in figures 6 and 7.

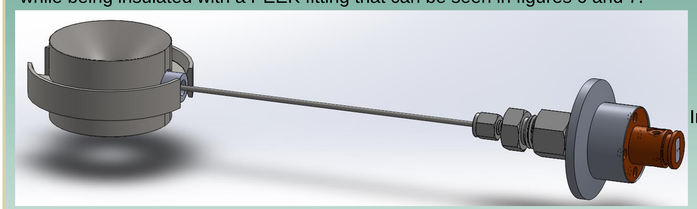

Figure 6: DMS Interface to DIT

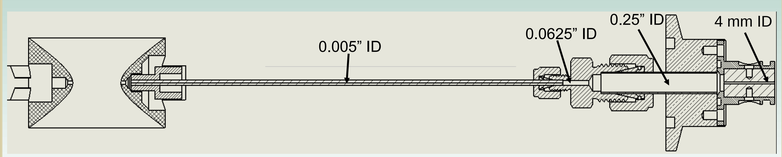

Figure 7: Cross-section view of DMS to DIT

Results - Nanospray Setup

Thermo-Finnigan nanospray setup with PicoTip emitter will be used as the ionization source. 3-D stage seen in figure 7 used for precision alignment. A high voltage power supply provides voltage to PicoTip Emitter. Nanospray injection occurs orthogonally with respect to DMS to avoid saturation This can be seen in figures 8 and 9.

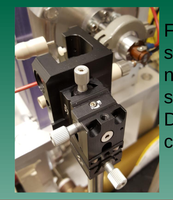

Figure 7: 3-D stage, nanospray setup, and DMS configuration

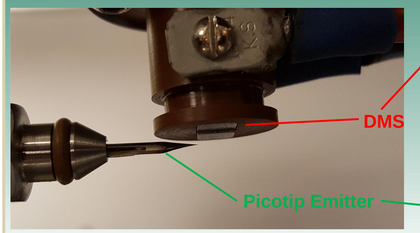

Figure 8: Nanospray positioning

Figure 9: Spray from PicoTip to DMS

Conclusions

Ion transmission into the trap may be complicated by quadrupolar field dynamics and shallow potential well depths of the ring electrode. Application of a bias voltage to the capillary or through directing ions into the external ion volume and einzel lens as seen in figure 1 could potentially overcome this issue. 0.010" ID and 0.005" ID capillary sizes were tested with the lowest attainable pressures being 1.72 x 10^{-2} torr, N_2 and 1.54 x 10^{-3} torr, N_2; respectively. The lower pressure obtained from the 0.005" made it more ideal to use for further testing. The pressure from this capillary could still be too high which can create greater chemical noise and reduce the resolution of the spectra obtained.

Future Work

- Utilize different cycle rates to radionuclide samples starting with cesium using DMS-MS.
- Design, fabricate, and implement a reaction chamber to install at the front end of the DMS. Chamber will consist of a 1-cm reaction cell flowing heated nitrogen gas to reduce adduct formation and impurities.
- Optimize conditions to obtain higher resolutions and determine isotopic abundance ratios through DMS-MS.

Acknowledgements and References

This work was supported by the Defense Threat Reduction Agency, Basic Research Award # HDTRA1-11-1-0012.

1. Sinatra, F. L.; Wu, T.; Manolakos, S.; Wang, J.; Evans-Nguyen, T. G. *Analytical Chemistry* vol. 87 issue 3 February 2015. p. 1685 – 1693. DOI: 10.1021/ac503466s

2. Wang, D.; van Amerom, F. H. W.; Evans-Nguyen, T. *Analytical Chemistry* vol. 85 issue 22 November 2013 p. 10935 – 10940. DOI: 10.1021/ac402403h