Development of a “Greener” Ionization source for Ambient Desorption Ionization with Nitrogen Gas

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ABSTRACT:

Analytical chemists employ copious quantities of organic solvents in their everyday work. Ambient ionization sources such as direct analysis in real time (DART) ionization source use inert gas to effect ionization of analyte in open air without using solvents. The original DART source was optimized for operation with helium, however this gas is derived from production of natural gas and thus requires mining for its production. Given that nitrogen is a ubiquitous, non-toxic component of the atmosphere we undertook a program to improve DART performance with nitrogen gas in order to determine its viability as a tool for reducing use of organic solvents for sample preparation and chromatography as desired for Green Chemistry.

ORIGINAL CARTRIDGE

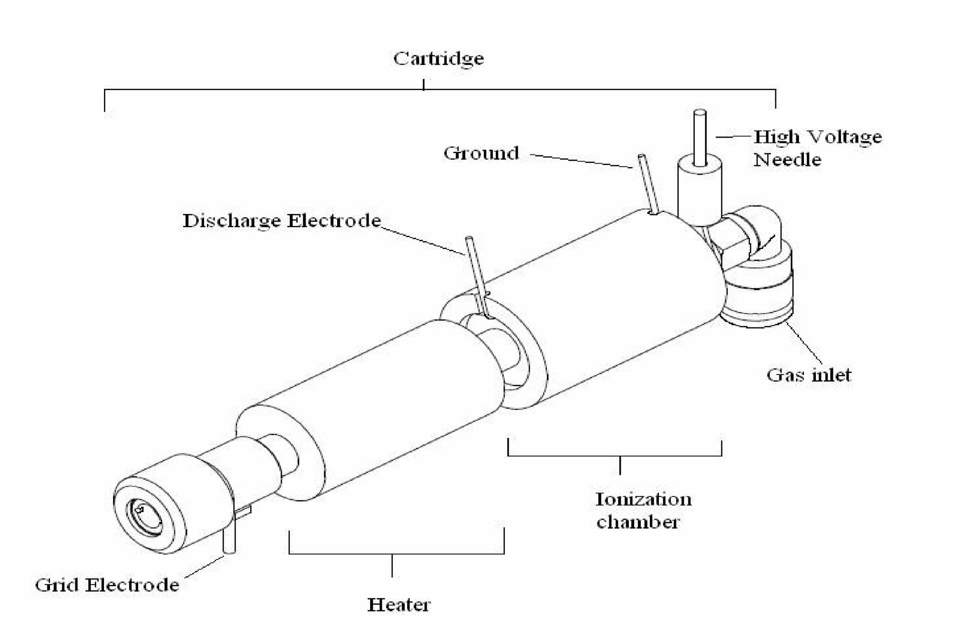

Figure 1. Original DART Cartridge Design Schematic

NEW CARTRIDGE

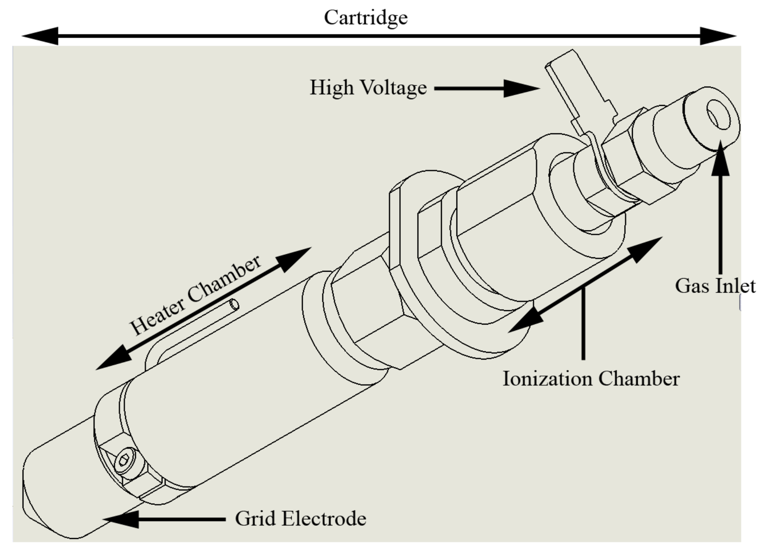

Figure 2. The new “Greener” DART Cartridge Design Schematic

DESIGN GOALS

- Improve sensitivity with nitrogen as the DART carrier gas
- Eliminate Oxygen entering into the ionization chamber through porous materials
- Reduce background
- Eliminate cement from design to improve durability
- Make cartridge components replaceable and serviceable
- Reduce the size of housing

EXPERIMENTAL

An investigation of the materials utilized to produce the various components of the DART source, its electrical configuration, and design was undertaken. A DART source with movable and exchangeable components suitable for prototyping was fabricated and subsequently used to document the effect of different materials, gas pressure, flow rate, and location of heating elements on ionization capability. Several designs were developed into prototype sources to enable comparisons of efficiency of ionization of a collection of test molecules with He and Nitrogen. Finally, implementation of the optimum materials and electrical configuration for promotion of better desorption ionization with Nitrogen was completed in this study.

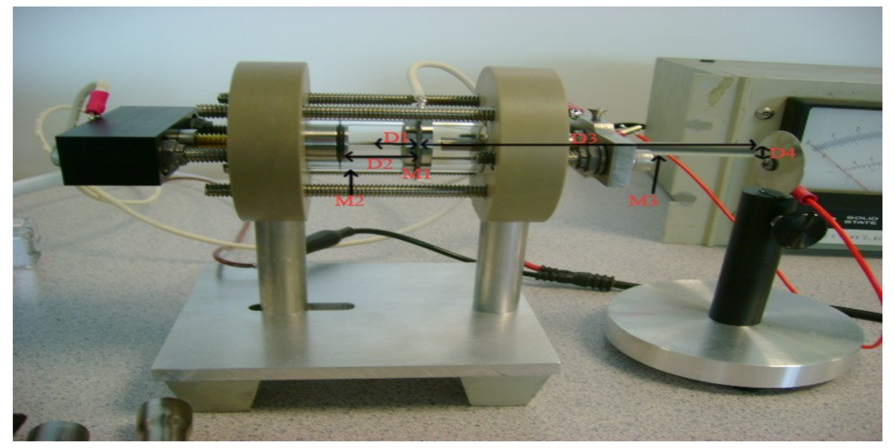

Figure 3

The DART cartridge breadboard permits adjustment of each component in the gun design.

- D1 is the variable length between the HV needle and the GND electrode.
- D2 is the variable length and material of the body of the Ionization Chamber.
- D3 is the variable length and material of the body of the Heater Chamber.
- D4 is the variable inner diameter of the heater chamber with variable material of the grid.
- M1 is the variable metal for the high voltage discharge.
- M2 is the variable material of the body of the Ionization Chamber.
- M3 is the variable material of the body of the Heater Chamber.

Using the breadboard unit enabled substitution of Major components such as various Ionization chamber tube materials and inner diameters in order to measure their desorption ionization capability. For each configuration, the breadboard was positioned in front of the API of a mass spectrometer and DART analyses of representative chemicals were completed to assess relative performance.

HARDWARE CHARACTERISTICS

- Body Materials
  - Alumina
  - Glass
  - Quartz
- Inner diameters of Body
- Discharge Materials
  - Stainless Steel
  - Titanium
- Grid Materials/ Mesh sizes
- Flow Rate
- Heater efficiency

ELECTRICAL CHARACTERISTICS

- Effects of different Voltages for glow discharge Needle.
- Comparison of configurations with and without the Discharge Electrode
- Effects of different Grid Voltages

Direct observation of the discharge was critical to the process. The discharge electrode position was infinitely adjustable in real time even with high voltage applied. The threshold for discharge was easily determined both visually and by inspection of the DART mass spectrum in ambient air.

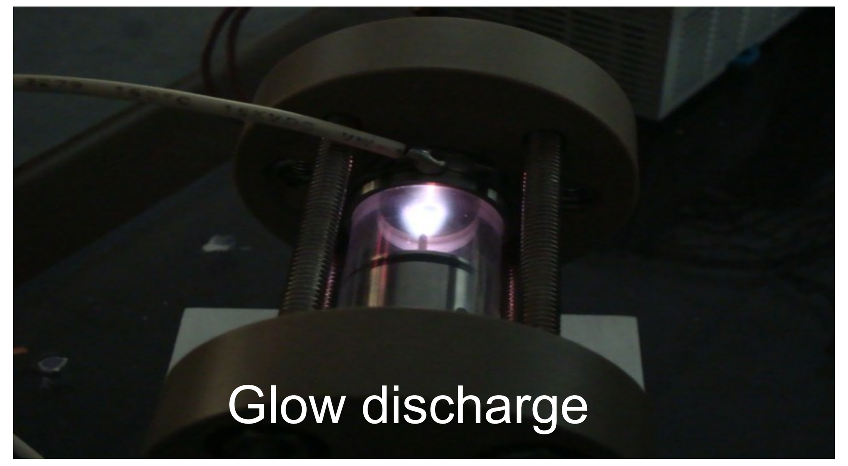

Figure 4. Picture of the breadboard DART discharge ionization region enabled visual inspection of the characteristics of the metastable formation.

HV Signal Analysis

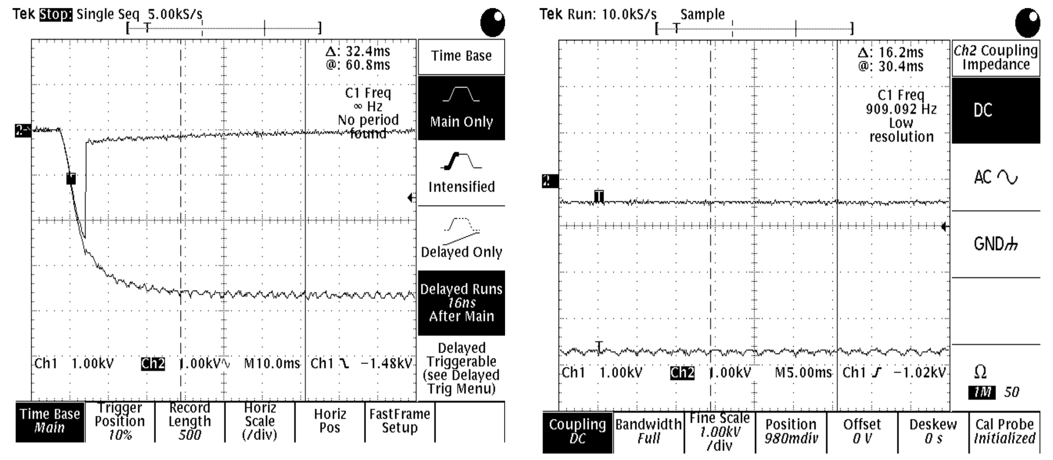

Figure 5. Above is an oscilloscope measurement of the HV signal at the spark of the glow discharge (Left) and at a constant glow discharge state (right). In both cases channel 1 shows the voltage potential at the HV Needle and Channel 2 shows the voltage potential drop at the output of the HV supply.

After varying the output of the HV supply we were able to determine that there was no change in sensitivity once the initial glow discharge was started. The original design used damping resistors to adjust the voltage drop across the HV needle and was wasting energy. After measuring the HV signal we were able to conclude that a current supply would be much more efficient that the damping circuit previously used. We were able to reduce power consumption by a factor of 3.

RESULTS AND DISCUSSION: Cartridge Comparisons

In investigating the capability of the DART cartridge to support desorption ionization at ambient pressure we understood that ionization of molecules in air could not be used as an indicator of performance. This being the case experiments with targets coated with chemicals were used to benchmark the original DART cartridge unit. In all cases the yield of ions generated with Nitrogen was significantly less than that generated by using Helium Metastables.

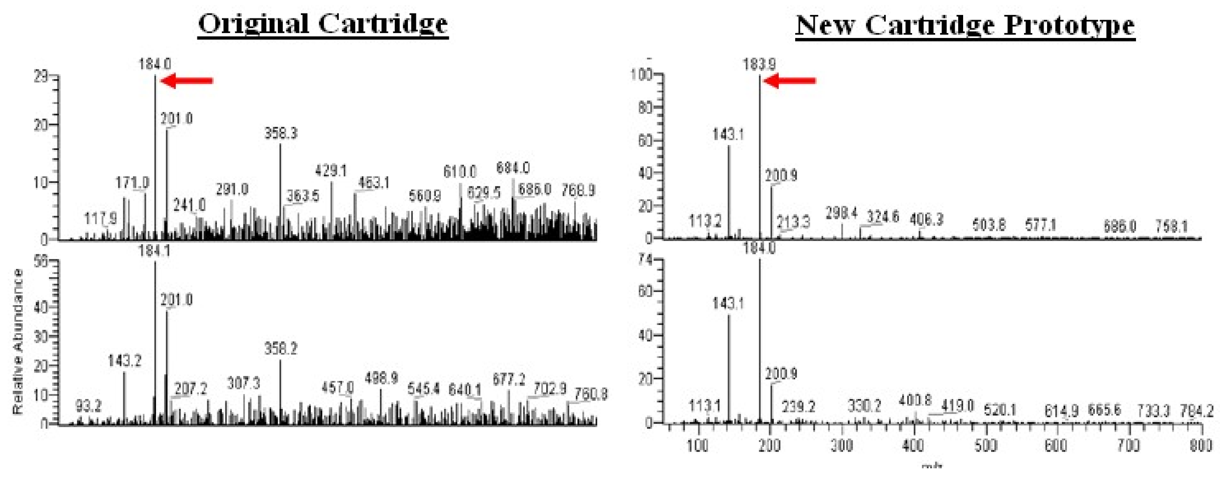

Figure 6. DART MS of Acephate with different cartridges.

Optimizing Flow Rate For Nitrogen

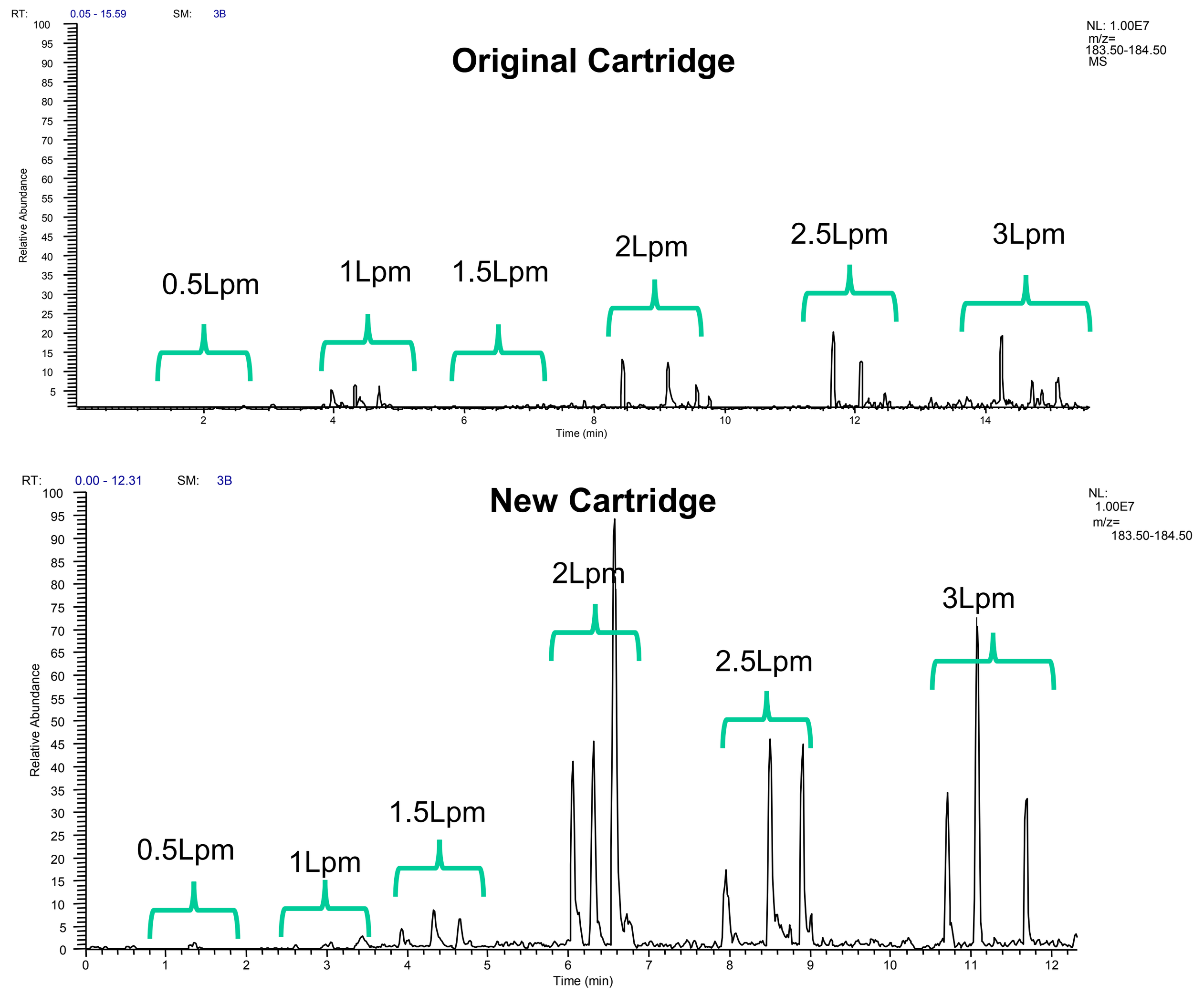

Figure 7. Mass chromatogram for multiple presentations of an Acephate sample for DART-MS at different gas flow rates.

Inner diameter comparisons

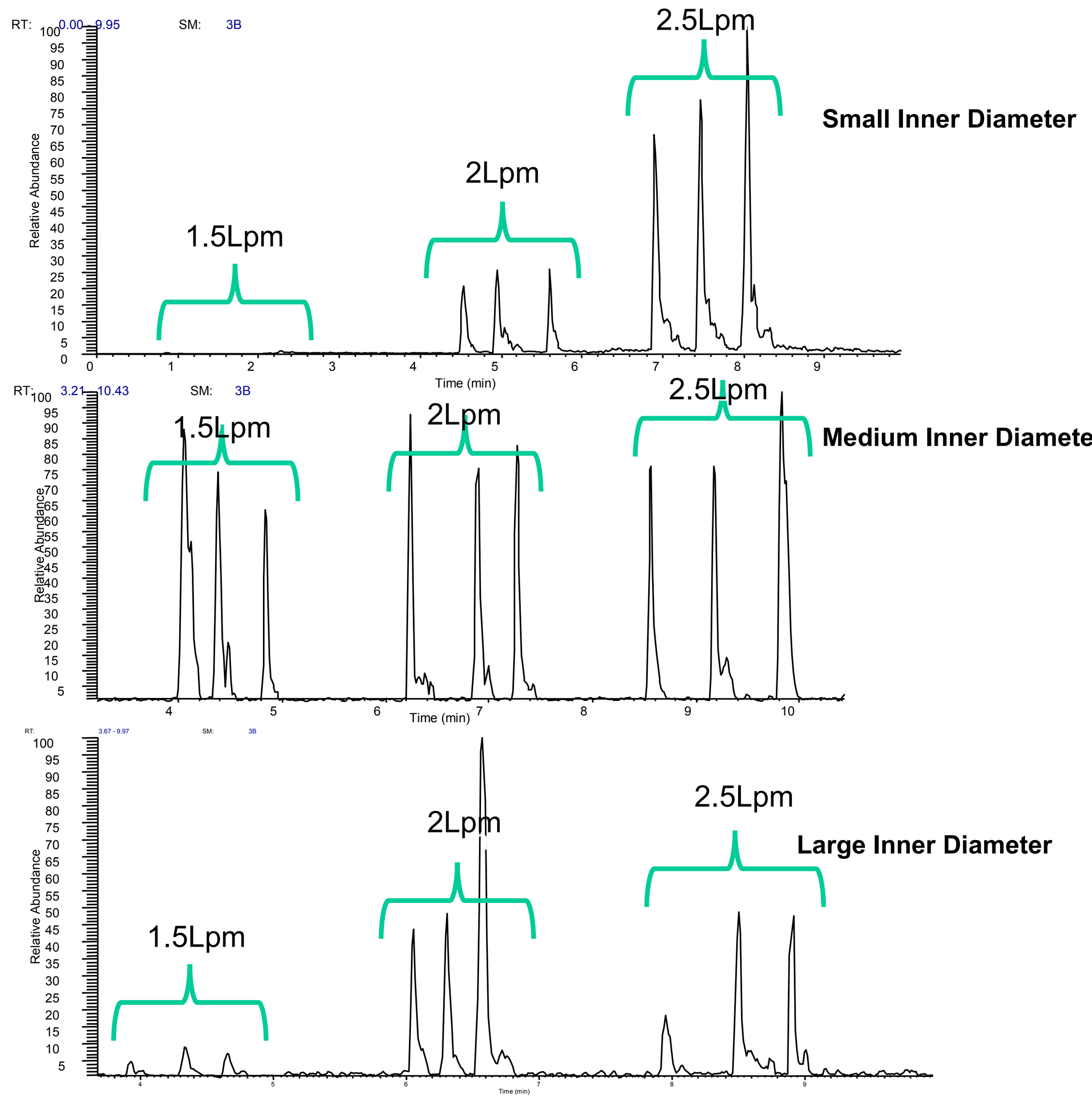

Figure 8. A series of commercially available sized tubes were selected to examine their effect on the generation of ions. Analysis of a variety of compounds were selected over various temperature ranges in order to compare the pros and cons of each size.

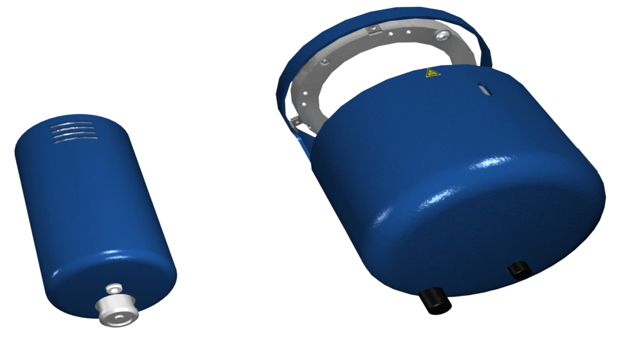

Figure 9. Above are the two DART sources side-by-side for comparison. The new source (left) is just over 3” in diameter and has active cooling. The original DART 100 source (right) is 6” in diameter and has a passive cooling system.

CONCLUSION

Development of a DART desorption ionization source with improved nitrogen carrier gas functionality has been completed. A functional “breadboard” device enabled measurement of ion production with different designs. Performance of various components individually and as complete assemblies allowed us to develop a commercial source with equivalent performance to the original DART using helium and improve performance relative to the original DART operated with nitrogen.