In-Situ and Instantaneous Detection of Aerosolized Chemical Threats Using Chip-Scale Mass Spectrometry

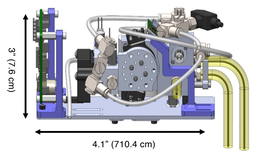
Detect-ION team including Gregory Vasquez, Gottfried Kibelka, and Ashish Chaudhary
Detect-ION I 6812 W Linebaugh Ave, Tampa, Florida

INTRODUCTION

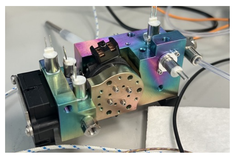
Non-volatile chemical compounds can persist in the atmosphere as aerosols in the size range of nanometers (nm) to micrometers (μm) range. Detection of these environments is paramount to swiftly and accurately detect acts of terrorism, industrial accidents, and environmental disasters. Detect-ION is developing a versatile chemical threat detection platform that collects, detects, and identifies aerosols of all sizes. The detector platform, called "**SPECTRA**L," comprises three novel low-SWaP subsystems, namely a miniature aerosol collector (**MAC**), a low-thermal-mass gas chromatograph (**LTM-GC**), and a chip-scale mass spectrometer (***µ*MS**) developed via prior IARPA funding.

COLLECTION

- **MAC** is a versatile aerosol collector, leveraging DI's patented Glass Capillary Array Collector (*GLAC*) design for efficient impaction (collection) and rapid desorption for sharp injection in the GC stage.
- The MAC is a dual-use collector, which is designed to operate as online and offline sample collection mode.



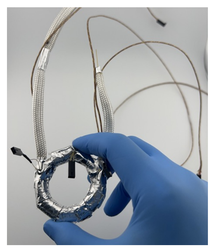
CAD model of the MAC 1.0



Prototype of MAC 1.0

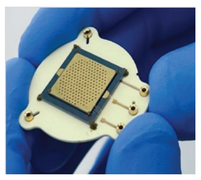
SEPARATION

- **LTM-GC** delivers low-SWaP gas chromatography with the versatility and performance of traditional oven GCs.
- DI's patent on-demand inline GC split design delivers higher material content to be transferred to the detector without compromising on injection quality
  - First segment of the GC column is used as a column trap or secondary collector.

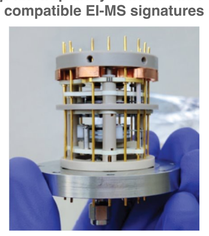


Prototype of 1x0-m LTM-GC

DETECTION



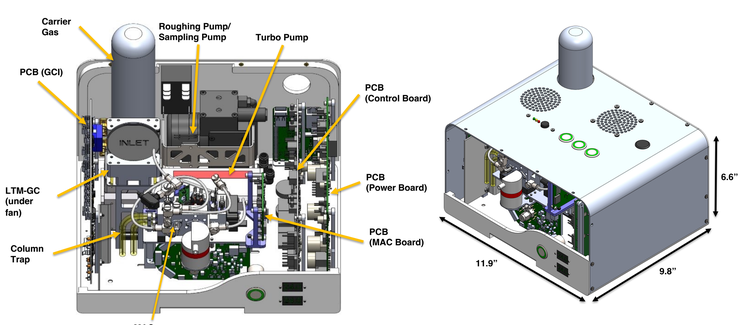
µ-ion trap array delivers NIST-compatible EI-MS signatures



Fully integrated *µ*MS on a miniature vacuum flange

- The ***µ*MS** uses microfabricated ion trap array and low-power electronics.
- A high-density array of chip-scale ion traps when operated simultaneously delivers low ppb level LODs.
- Using < 3 W, the *µ*MS delivers mass spectra from 40 -210 m/z at a resolution of 1 - 1.3 amu.
- *µ*MS generated mass spectra are electron impact (EI) NIST-compatible allowing the use of existing libraries for chemical identification.
- *µ*ms has been demonstrated in several variants of detector systems for a broad range (>200) of volatile and semi-volatile organic compounds in prior government-sponsored efforts.

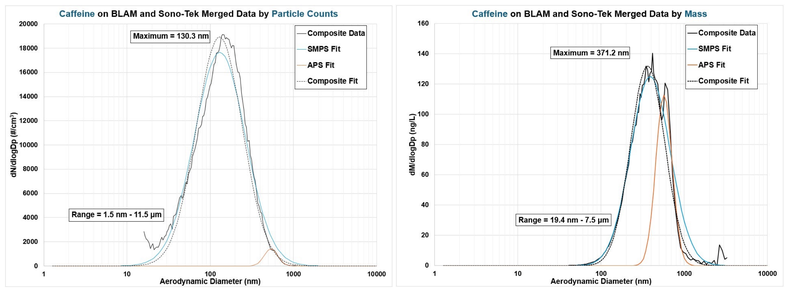
SYSTEM INTEGRATION



CAD model of the fully integrated SPECTRAL 1.0 platform (7.5 kg, 12 L) comprising of all the three subsystems described above, control electronics, sampling and vacuum pumps, compressed helium bottle and all the ancillary components needed for executing a versatile set of pneumatics, temperature control and thermal management maneuvers, as needed.

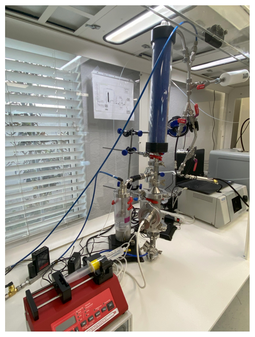
METHODS

To evaluate the SPECTRAL 1.0, a versatile flow-through aerosol generation testbed was developed. Using a CH Technologies Blaustein Atomizer (BLAM) and a Sono-Tek Atomizer (Sono-Tek), aerosols in a size range between 10 nm and 7 μm can be reliably generated. The aerosols are characterized using TSI's Aerodynamic Particle Sizer (APS 3321) and TSI's Scanning Mobility Particle Sizer (SMPS 3938).

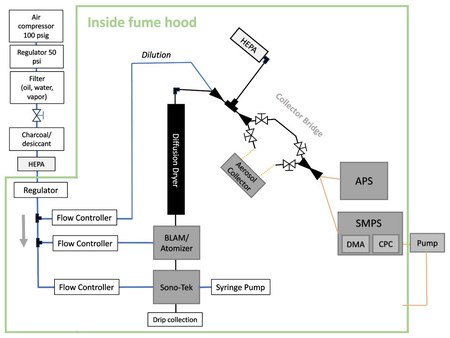


Size distribution of caffeine particles. Left: Particle count (dN/dlogDp) over particle diameter; Right: Mass distribution (dM/dlogDp) over diameter. Particles were generated by a combination of Sono-Tek and BLAM and characterized with APS and SMPS.

The aerosol testbed includes a collector bridge that enables effective determination of collection efficiencies as a function of particle size. To establish the ground truth, the collection and the desorption efficiencies for different aerosol collectors and a variety of aerosols are determined by collecting aerosol samples on commercial sorbent tubes that are subsequently analyzed on a Shimadzu QP2020NXNC GC-MS coupled with a TD-30 Thermal Desorption System.

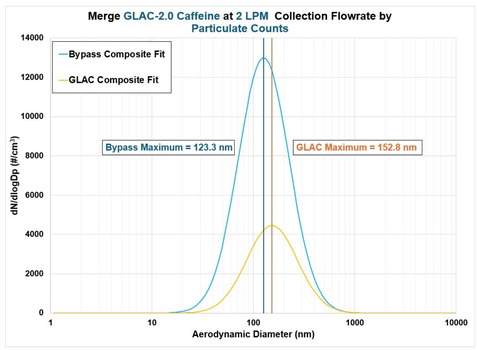


Flow-Through Aerosol Generation and Characterization Testbed



Schematic of the fluidics of the Aerosol Testbed. Aerosols are created using clean compressed air

Collecting aerosols directly from the testbed provides the reliable baseline for comparison. Collecting the aerosol after passing through the collector allows for the calculation of the collection efficiency while thermally desorbing collected material onto the sorbent tubes enables precise calculation of the desorption efficiency.

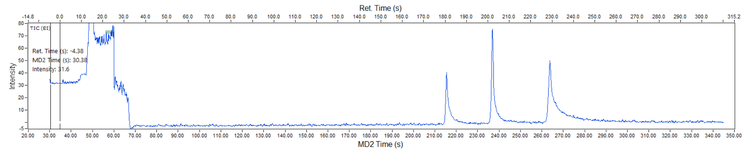


GLAC's collection efficiency based on APS/SMPS measurements is 89% and the desorption efficiency based on calibrated TD-GC-MS measurements is 80% resulting in an overall efficiency of 72% for Caffeine.

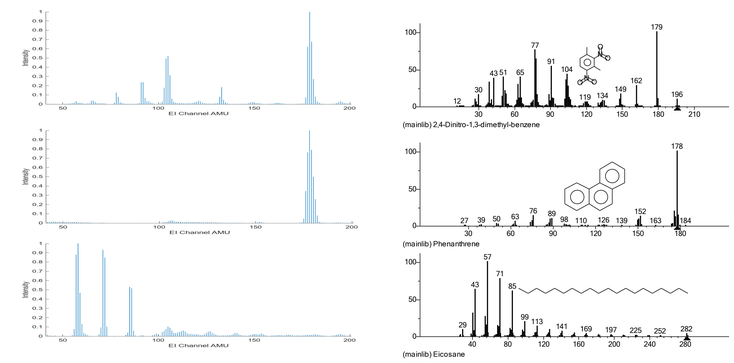
RESULTS

The SPECTRAL system can detect a wide variety of organic aerosols, currently up to a volatility that translates to a gas chromatographic retention index of 2400.

The low internal dead volume (0.3 mL) of the GLAC in combination with short paths inside the heated MAC enables rapid thermal desorption of collected aerosols onto the LTM-GC for optimal chromatography.



Total Ion Chromatogram (TIC) of 20 ng each 4,6-Dinitro-m-xylene, Phenanthrene, and Eicosane. (Shimadzu injector 250 °C, 50 cm hydroguard 0.320 mm ID, oven 250 °C isotherm, split ratio 5, Carrier Gas: 50 kPa (7.25 psi) constant pressure, transfer line in air and column connector heated to 220 °C, 10 m MXT-5, 250 μm ID, 0.25 μm phase, MS vacuum tube 61 °C, vacuum feedthrough 160 °C.

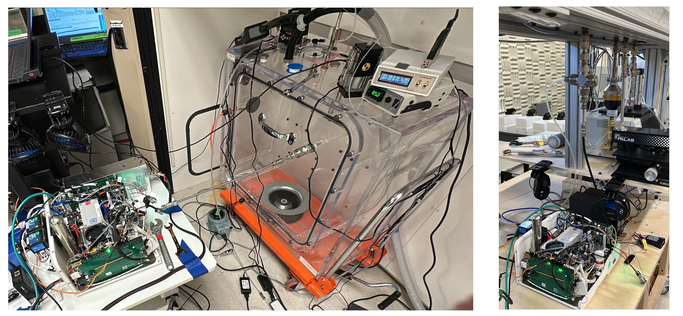


Left: Mass spectra generated by *µ*MS; Right: EI-MS NIST spectra for comparison

KEY MISSION CAPABILITY

SPECTRAL's efficiency across the collection, desorption, and detection stages allows <1 ng of limits of detection in sampling duration as short as 2 min. The overall system latency for such sampling method is <15 min, which includes embedded smart cleaning. Therefore, SPECTRAL can be applied to collect trace-level chemical threat signatures in a dynamic threat environment.

Detect-ION plans to complete integration of SPECTRAL 2.0 in April 2025 and expects the performance to surpass that of SPECTRAL 1.0. A part of the ongoing development is the development of aerosol threat libraries and the implementation of an automated search algorithm for the identification of compounds based on GC retention index and mass spectra.



SPECTRAL 1.0 integrated with the government testbed developed at U.S. Naval Research Laboratory, Washington, DC for benchmark testing.

Present and Past Funding

This research is based on work supported by the Office of the Director of National Intelligence (ODNI), Intelligence Advanced Research Projects Activity (IARPA), via FA8650-17-C-9108, FA8650-19-C-9100 , 20120800003 and **140D0424C0008** and has some technology elements in part sponsored by DARPA via HR001119C0034and DIU/DTRA via HQ00342390001. The views and conclusions contained herein are those of the authors and should not be interpreted as necessarily representing the official policies or endorsements, either express or implied, of the ODNI, IARPA, DARPA, DIU, DTRA or the U.S. Government. The U.S. Government is authorized to reproduce and distribute reprints for Governmental purposes notwithstanding any copyright annotation thereon.