



Expanding the range of threats detected with a miniature mass spectrometer by the integration of novel sample inlets

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Abstract

A next generation chemical detection system has been developed using a cylindrical ion trap based mass spectrometer. While mass spectrometry has the potential to provide security forces, homeland defense, and first responders with highly specific detection, reduced false alarm rates, and a wider range of detectable threats; portable mass spectrometers are limited to the detection of samples in the vapor phase. However, in many deployments, threats are present in forms other than vapor (e.g., liquid, particulate, etc.). To enable a mass spectrometer to detect non-gaseous threats, users are typically required to prepare samples off-line for introduction to the instrument (e.g., SPME, pyrolysis, etc.). While proven in a laboratory setting, these preparation techniques require expertise, consumables, and increase the logistical burden for field applications. The system proposed here overcomes these challenges by presenting several mechanisms for enabling the collection and analysis of low volatility and non-vapor samples in the field.

The instrument developed by the authors is a small (< 20 l volume, < 8 kg weight) mass spectrometer. The instrument is capable of analysing a wide range of compounds over a 10 – 450 amu mass range with unit resolution. The system is capable of measuring concentrations to 100 ppb with its integrated MIMS inlet. A 10 l/s turbo / roughing pump combination allows higher gas loads than typically seen in portable instruments. Example spectra will be presented illustrate the resolution and sensitivity of the combined systems.

To enable particulate (e.g., explosives) and 'sticky' (e.g., CWA) substances to cross the vacuum barrier into the mass spectrometer, a novel thermal desorption system is being developed. The sample inlet allows collection of explosive particulate using a swipe method similar to those in use today. However, to allow transfer of the vaporized explosive particulate into the vacuum chamber, the desorber is configured to evacuate the volume prior to desorption thus allowing direct injection of the sample into the instrument without the need for external ionisation or ion transport (e.g., ion funnel or quadrupole ion guide).

A novel pre-concentrator has been developed that allows the sensitivity of the mass spectrometer to be increased by a factor of 10³ – 10⁵ utilizing both adsorption and evacuation combined in one device. A directly coated sorbent heater allows faster response than typical sorbent columns. The extremely high gain allows the detection of low volatility substances (e.g., explosives and CWAs).

Miniature Mass Spectrometer

The instrument developed by the authors is a small (< 20 l volume, < 8 kg weight) mass spectrometer based on an ion trap architecture. In short, the instrument is capable of:

- 10 – 450 amu mass range (covering most explosives, TICs, and CWAs)
- < 1 amu resolution (FWHM)
- Sample-to-sample time: < 1 second (30 seconds with described pre-concentrator or desorber)
- Power consumption: < 45 W
- Sensitivity: < 200 ppb VOCs (< 1 ppb with pre-concentrator)

Figure 1 below shows three variants of the instruments.



Figure 1 – Photographs of 3 variants of 1st Detect miniature mass spectrometer including bench-top (left), handheld (center), and sub-component (right)

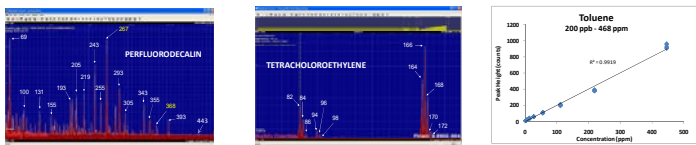


Figure 2 – representative spectra including perfluorodecalin (left), tetrachloroethylene (center), and a calibration for toluene (right) showing linear response over 200 ppb – 468 ppm

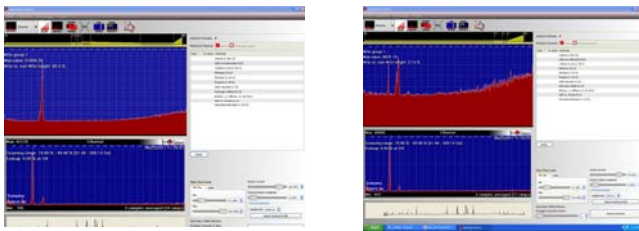


Figure 3 – Example of MSⁿ (MS/MS) mode of operation

Purge and Trap

The enable collection of threats agents from non-gaseous media, a novel purge and trap system has been developed utilizing the preconcentrator presented previously. A reduced pressure headspace is created over a liquid sample, and air bubbled through the system to release analyte. Preconcentration of the sample has been achieved with customized adsorption matrix in a geometry optimized both for maximum adsorption collection as well as efficient high yield desorption via direct introduction into the mass spectrometer at reduced pressure. The integrated design of the preconcentrator with the chemical detection system is targeted at sensitive analysis of vapor samples.

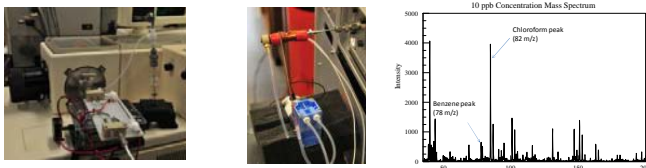
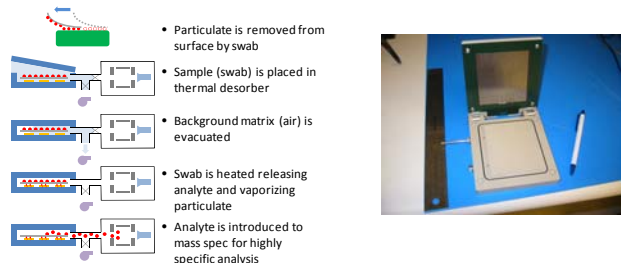


Figure 4 – Photograph (left and center) of water sampler and representative data (right) showing measurement of 10 ppb benzene and chloroform in water

Evacuated Desorber

To enable particulate and 'sticky' substances to cross the vacuum barrier into the mass spectrometer, a novel thermal desorption system is being developed. The sample inlet allows collection of explosive particulate using a swipe method similar to those in use today. However, to allow transfer of the vaporized explosive particulate into the vacuum chamber, the desorber is configured to evacuate the volume prior to desorption thus allowing direct injection of the sample into the instrument without the need for external ionization or ion transport (e.g., ion funnel or quadrupole ion guide).

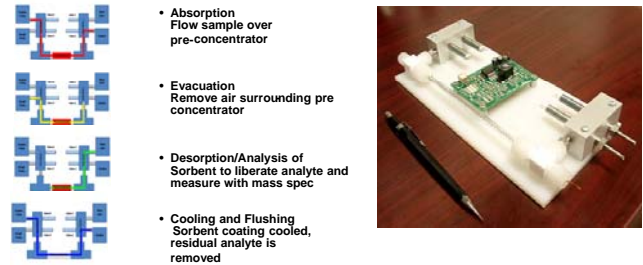


- Particulate is removed from surface by swab
- Sample (swab) is placed in thermal desorber
- Background matrix (air) is evacuated
- Swab is heated releasing analyte and vaporizing particulate
- Analyte is introduced to mass spec for highly specific analysis

Figure 5 – schematic (left) and photograph (center and right) of evacuated desorber

Pre-Concentrator

A novel pre-concentrator has been designed that leverages the selective sorptive capabilities of advanced materials with a novel design that significantly reduces the analysis time compared to currently deployed instruments. In the design sorbent materials are placed in a tube with a heating element. The electrical leads of the heater is connected to a power supply controlled by a computer system. Because the tube housing the sorbent is evacuated thus minimizing the dead volume effect prior to thermal desorption, the overall concentration gain is a product of the adsorption and evacuation gains yielding a typical total gain in the range of 10⁴. Typical analysis times are less than 30 seconds.



- Absorption
Flow sample over pre-concentrator
- Evacuation
Remove air surrounding pre-concentrator
- Desorption/Analysis of Sorbent to liberate analyte and measure with mass spec
- Cooling and Flushing
Sorbent coating cooled, residual analyte is removed

Figure 6 – schematic (left) and photograph of pre-concentrator

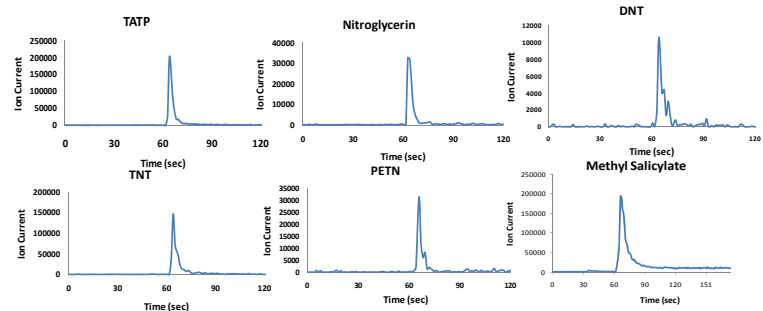


Figure 7 – Desorption profiles for representative explosives and CWA simulants

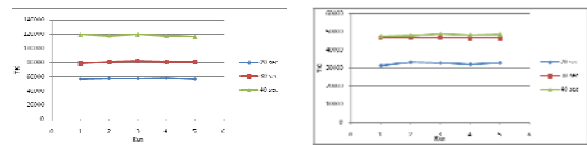


Figure 8 – Repeatability and Linearity performance of pre-concentrator

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