Trace Explosives Sensor Testbed (TESTbed)

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NRL's Role in DHS S&T Overall Mission

- **Trace Explosives Sensor Testbed** designed and constructed for the evaluation of a broad range of detection systems, materials and sensors.

- **Independent validation and verification (IV&V)** of the new materials, sensors and detection systems (TRL 2-6) under development by DHS S&T for explosives vapor detection
  - prior to TSL certification testing
  - critical guidance to DHS and sensor developers early in project development

- **Promote advancement of explosives sensor development in wider community via**:
  - documentation in refereed journals
  - new hardware for vapor generation
  - new analytical verification approaches for trace explosive levels

- **Consultation and experimentation for solving any immediate and timely issues** that may arise for DHS S&T/TSA
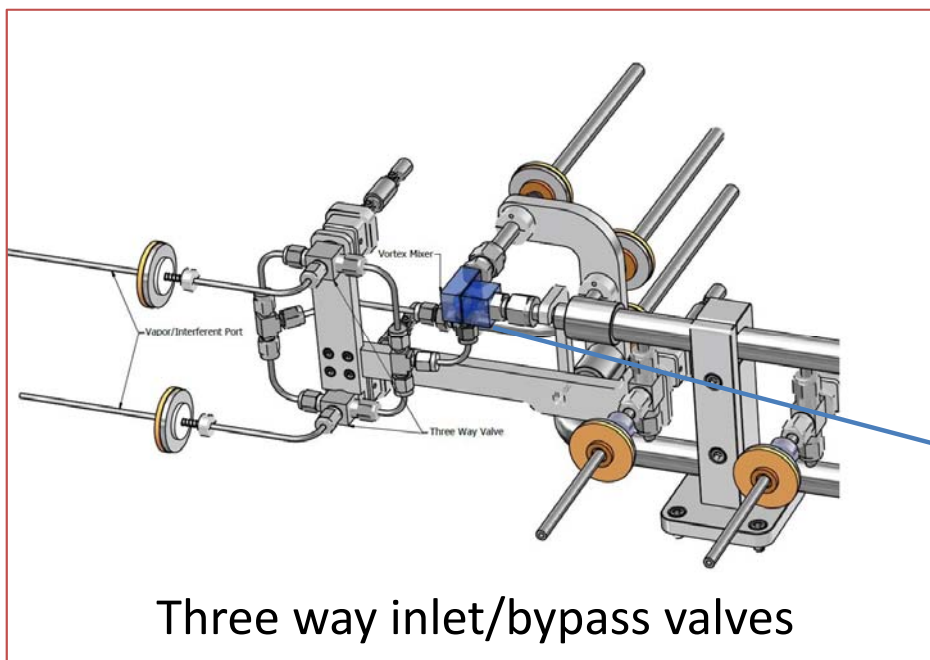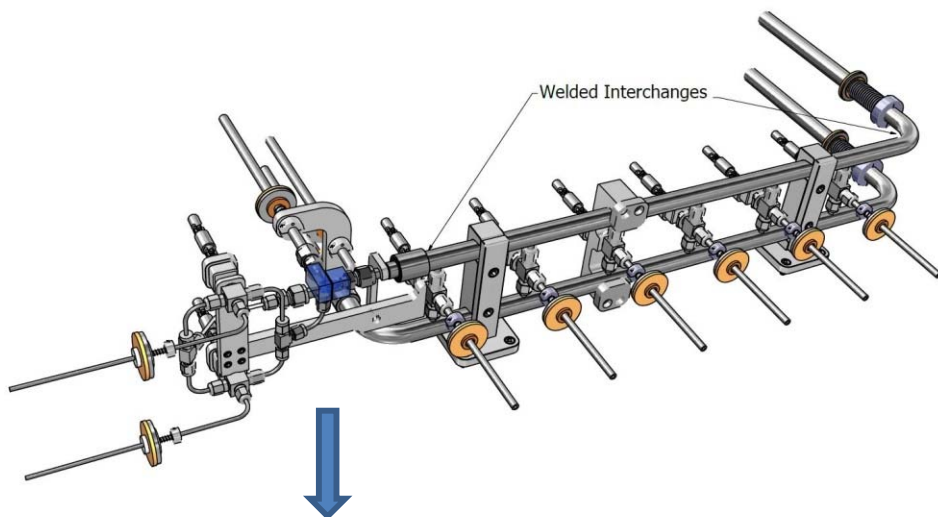
TESTbed

The TESTbed has dedicated computer control of a standardized vapor delivery system with an automated data collection system suitable for obtaining high quality data for sensor validation.

Key Features:
- Six identical sample ports
- Zero air source
- Four vapor generation sources
    - Permeation Tubes
    - Certified Gas Standards
    - Nebulizer/Pneumatic Flow System
    - Dynamic Headspace chamber
- Fully automated with a custom graphical user interface
- Housed in an Oven
    - Operational Temperature Range: 20 - 130 °C
    - Bake out to 150 °C
- Humidity control 0-85%

TESTbed Manifold

Welded Interchanges

Three way inlet/bypass valves

Vapor/Interferent Port
Vortex Mixer
Three Way Valve

Manifold key features:
- Sulfinert™ treated stainless steel tubing
- Dual distribution manifolds, one for clean air and the other for analytes
- Rapid switching between manifolds
- Two inputs to the analyte manifold
- Custom mixer for uniform mixture on analyte manifold
- Three way inlet/bypass valve
- Six sample ports for individual or simultaneous testing of multiple sensors
- Easily removed and exchanged
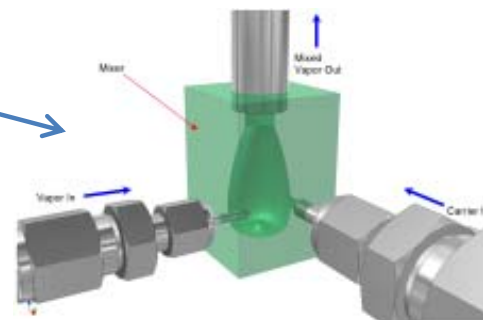
Mixer
Mixed Vapor Out
Vapor In
Carrier In
Vortex mixer

Conditioned Air

Environics and Miller-Nelson

Key features:
- Environics Series 7000 Zero Air Generator
  - Delivers up to 20 liters per minute, 30 psi of dry, contaminant-free air.
  - Free from
    - Water vapor
    - Particulates
    - <0.5 ppb Sulfur dioxide, hydrogen sulfide, oxides of nitrogen, nitrogen dioxide, ozone, carbon monoxide and hydrocarbons
- Miller-Nelson Test Atmosphere Generator controls and monitors the initial Flow Rate, Temperature, and Humidity level
  - Flow Rate : 2 - 20 L/min
  - Temperature : 20 - 35 °C
  - Humidity : 20 - 85 %RH

Commercial Test Vapor Sources

Kin-Tek FlexStream Automated Permeation Tube Oven

Permeation Tubes

Calibrated Gas Standards

- The FlexStream Automated Permeation Tube System controls both its oven temperature as well as the carrier gas flow rate through the oven module
  - 500 permeation tubes available
    - NH_3
    - HNO_3
    - DNT
  - Custom tubes optional

- Auxiliary flow controller for calibrated gas standards (Flow Rate: 10 - 1000 mL/min)

Nebulizer and a Pneumatic Flow System

Pneumatically Modulated Liquid Delivery System (PMLDS)
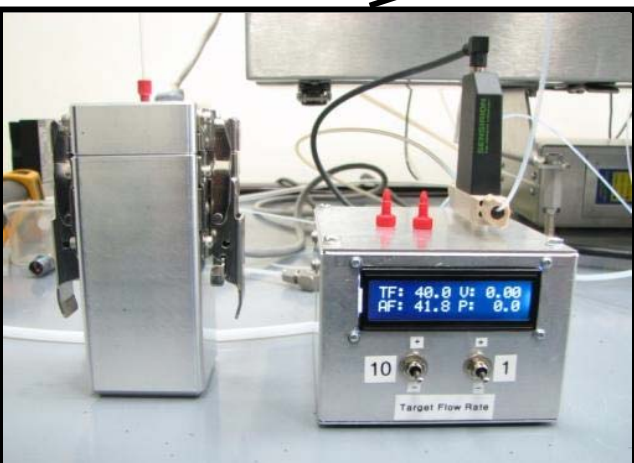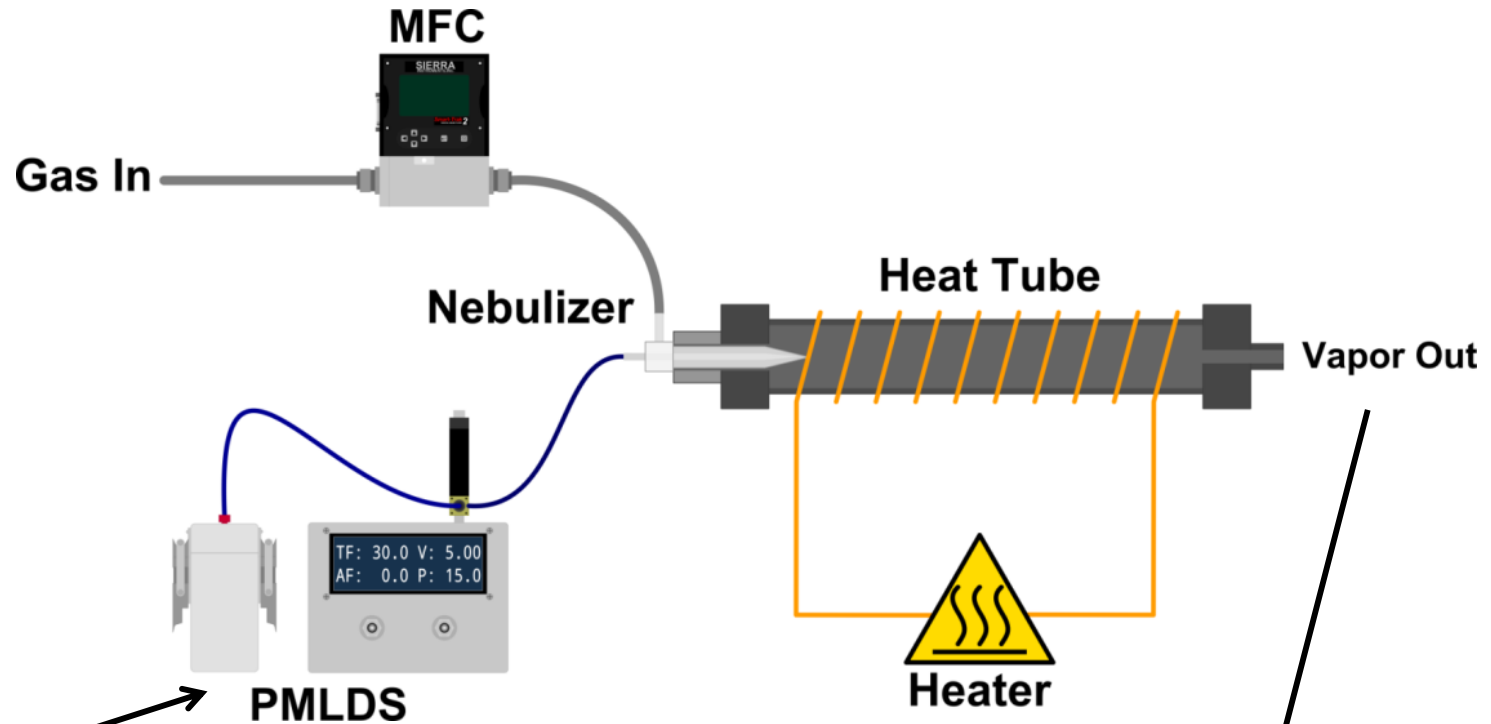
Pulse-Free, Efficient Vaporization:
- Heat tube vaporizes water/analyte
- Suitable for TNT, RDX, and PETN
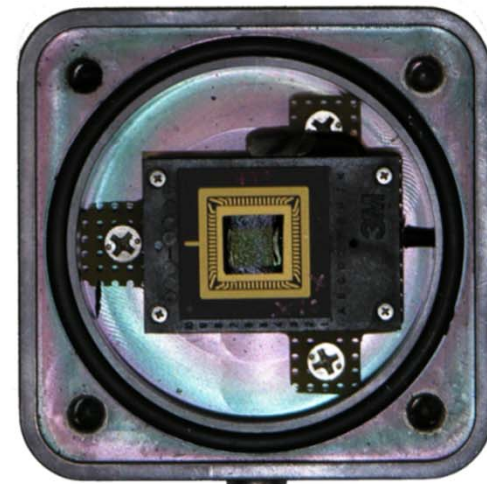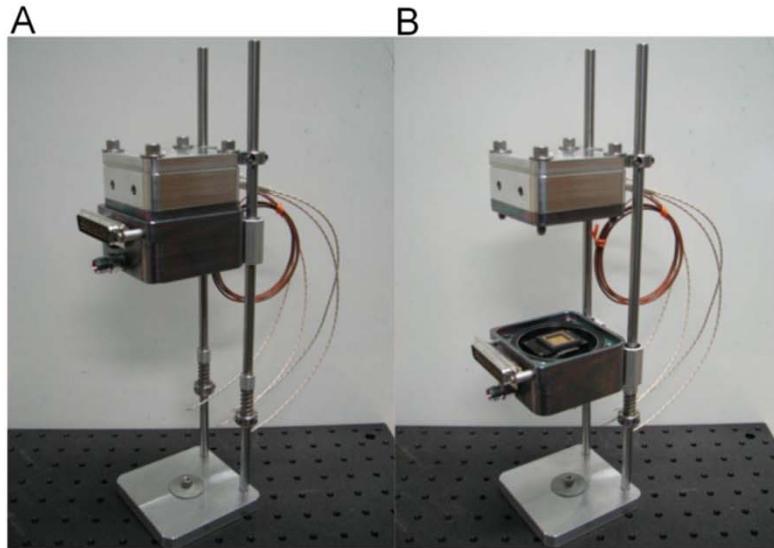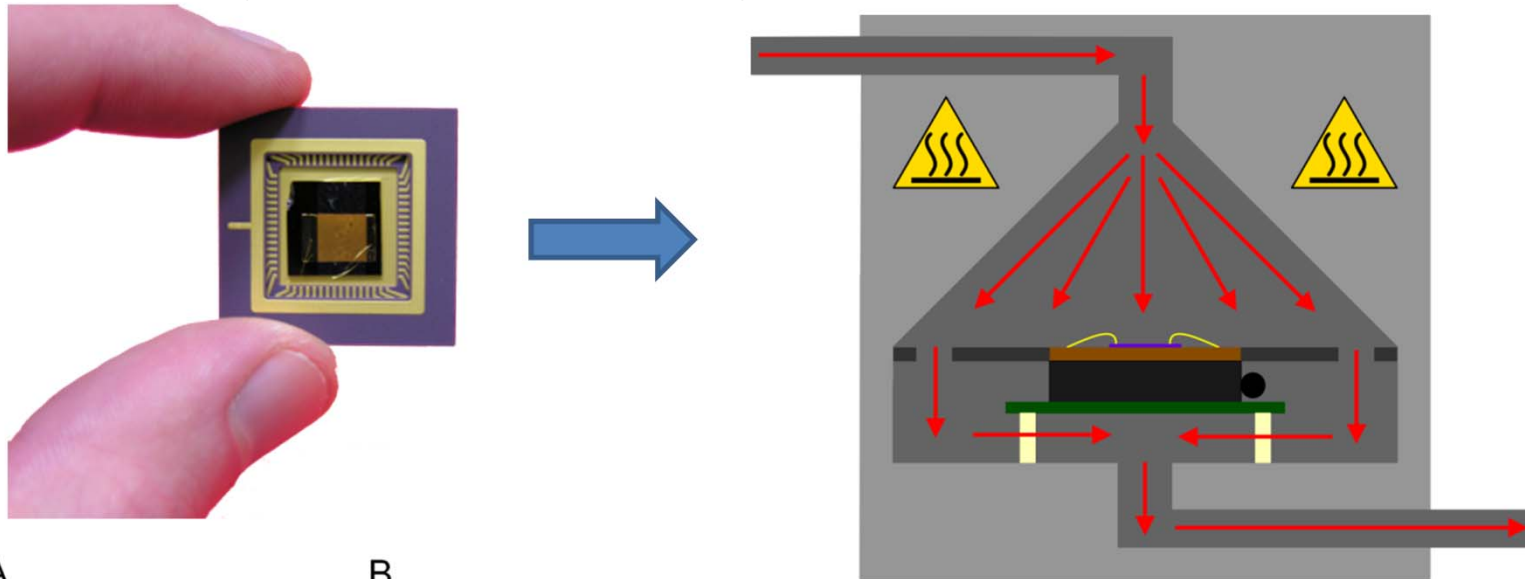- Simple interface and computer control

Dynamic Head Space Sample Chamber
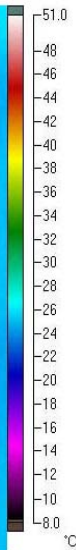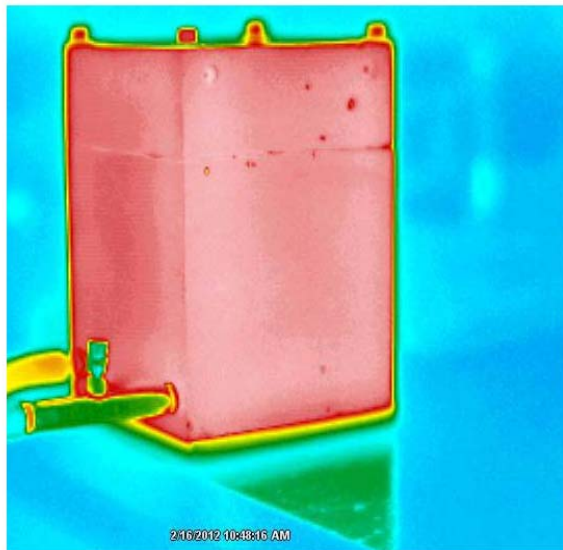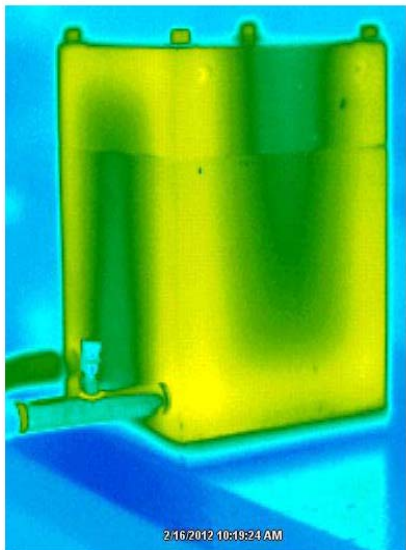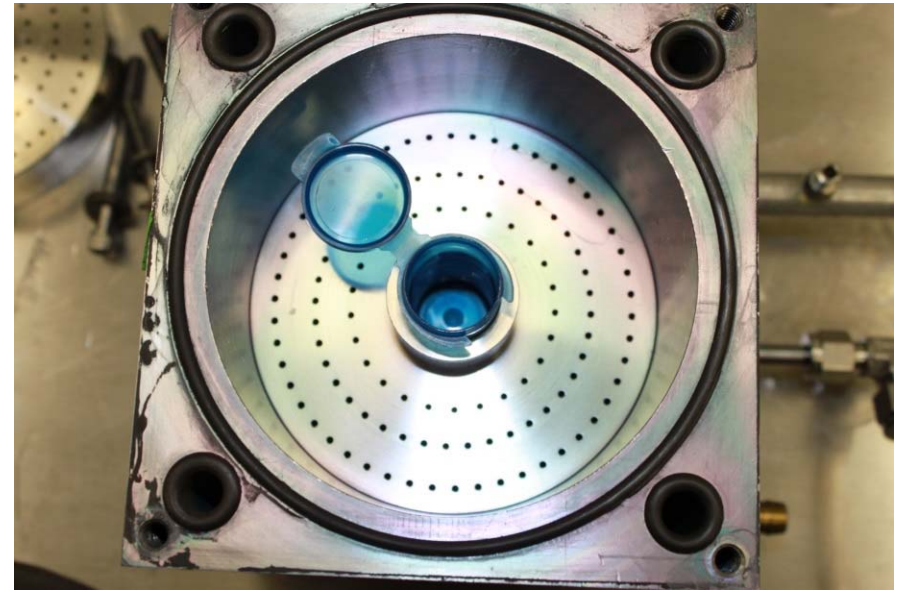
Impactor Chamber

- Chamber for electronic sensors such as chemiresistors, SAW
- Temperature controlled, Sulfinert^{TM} coated sample chamber
- Sensor up to 1"x1" in a PGA chip, connected with a ZIF

Optical Chamber

Impactor

Lateral

- Chemiluminescence and fluorescence or fluorescence quenching
- Temperature controlled, Sulfinert^{TM} coated chamber
- Sample Size 1"x1"
- Photomultiplier tube (Hamamatsu R1288A-27) for chemiluminescence
- Fiber optic probe for the fluorescence
  - Avantes and features 12 illumination fibers of 200 μm surrounding a single detection fiber of 600 μm

Computer Control

- Touch screen interface allows user to control/monitor/store system parameters for all components
- Standard test protocols preloaded
- Custom test protocols easily developed, implemented and stored
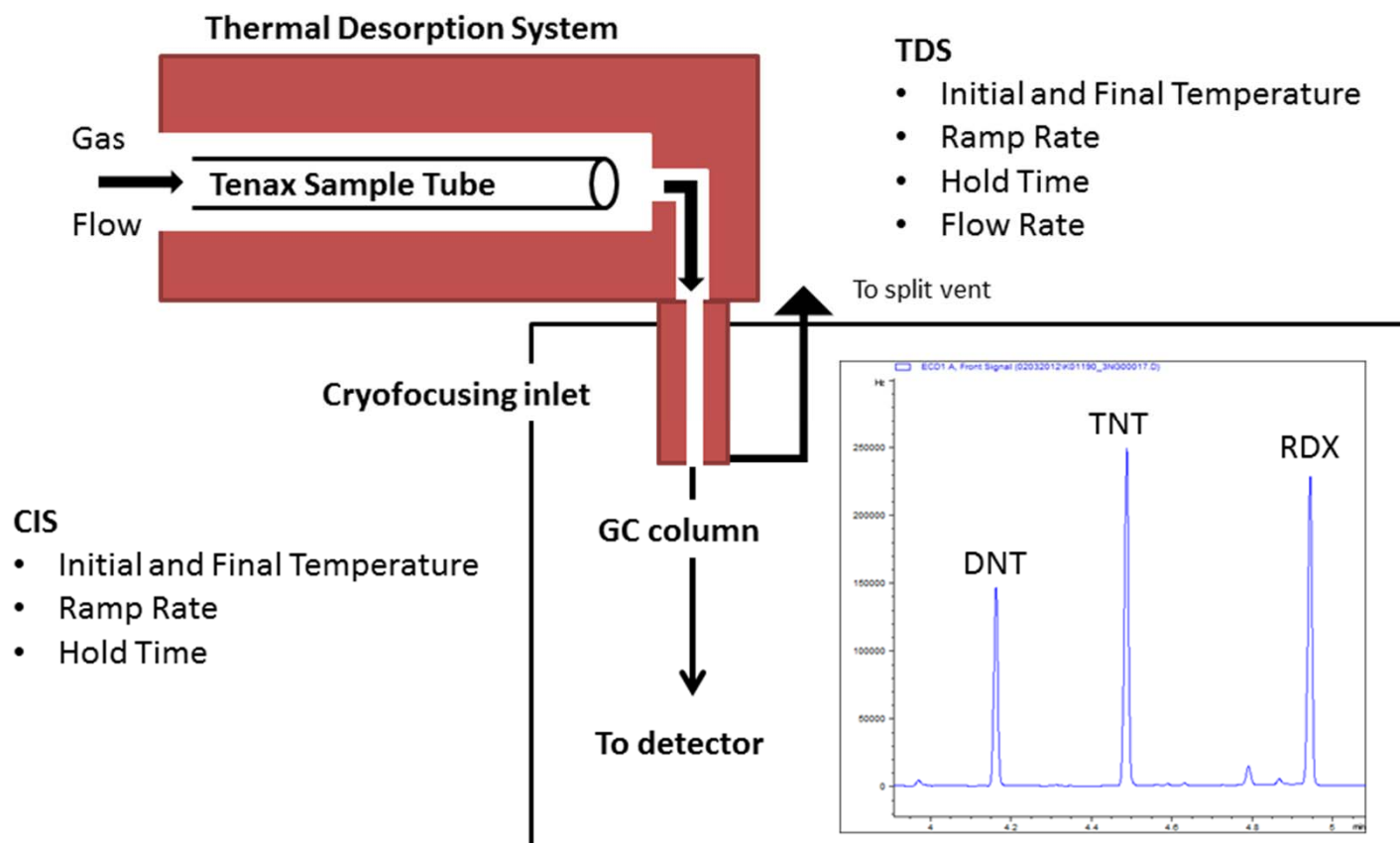
TESTbed Validation

System has been characterized with the following analytes

- Ammonia from 10 ppb-10 ppm
- Nitrogen Dioxide from 10 ppb-10 ppm
- Carbon Monoxide from 1 ppm – 1000 ppm
- Methyl Salicylate
- Hexane
- DNT from 10 ppt_v - 50 ppb_v
- TNT from 640 ppq_v – 10 ppb_v
- RDX from 850 ppq_v – 1 ppb_v
- PETN from 12 ppt_v - 1 ppb_v
- TATP from 5 ppb_v – 5 ppm_v

Confidence Check: Standard protocol developed using 2,4-DNT

Analytical Validation Method

Thermal Desorption System

Gas

Tenax Sample Tube

Flow

TDS
- Initial and Final Temperature
- Ramp Rate
- Hold Time
- Flow Rate

To split vent

Cryofocusing inlet

CIS
- Initial and Final Temperature
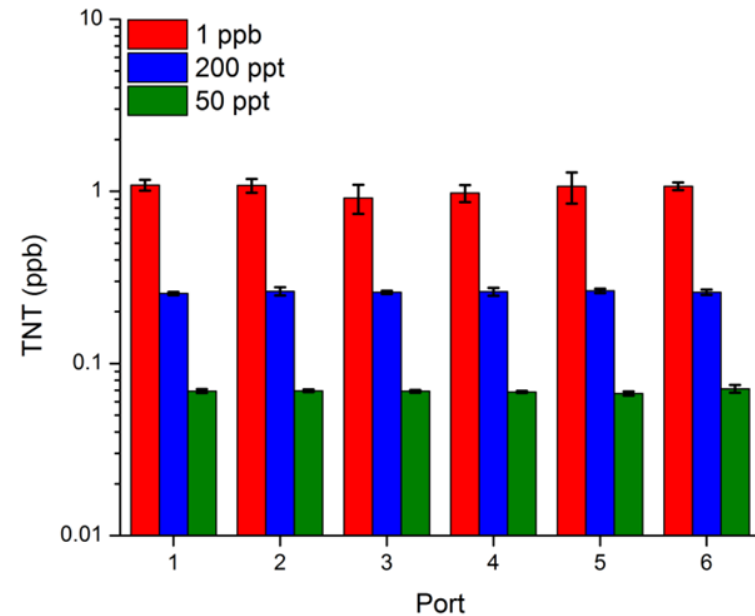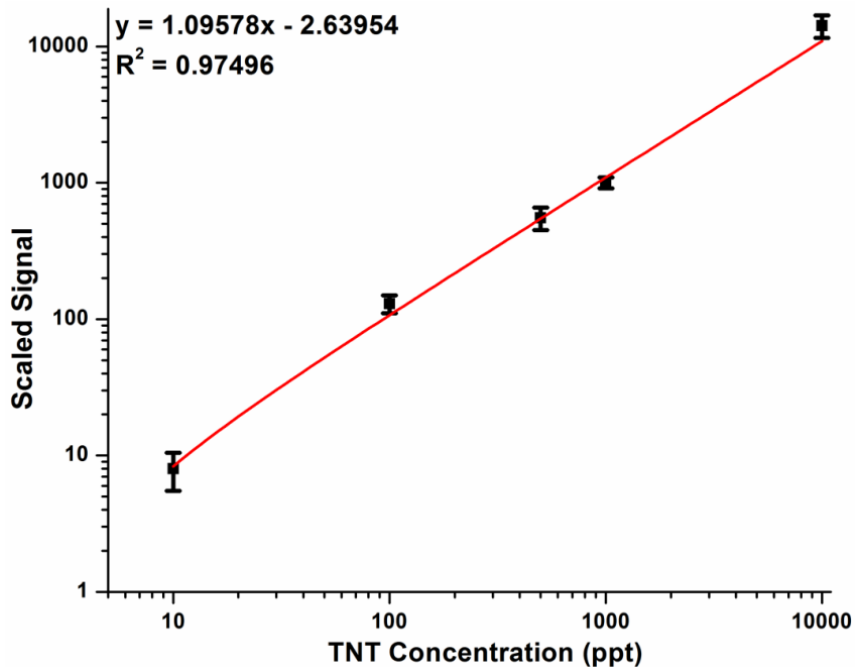- Ramp Rate
- Hold Time

GC column

To detector

DNT TNT RDX

Explosives validated using Tenax/TDS-CIS-GC-ECD showing accuracy and precision within the experimental limits of the protocol
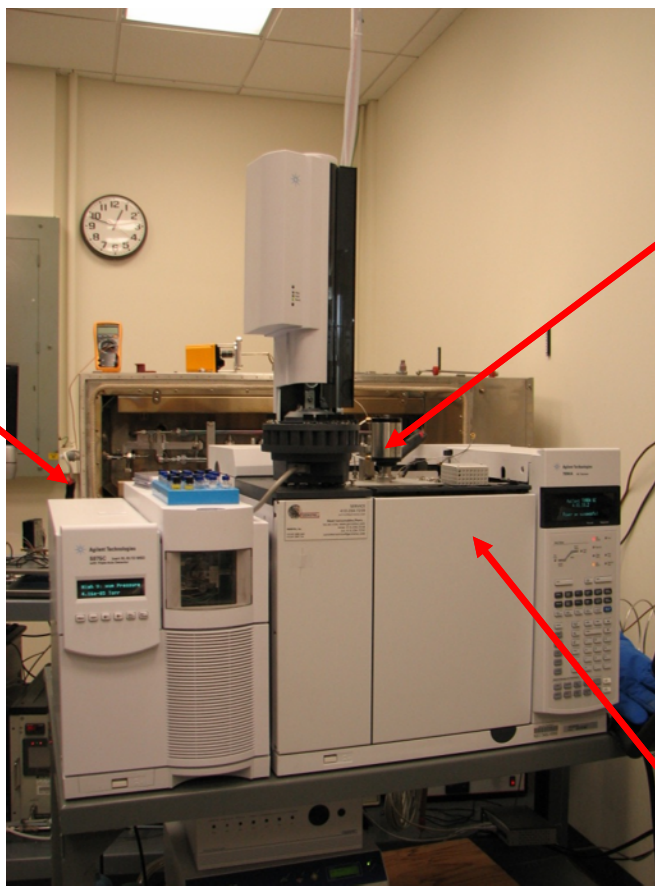
TNT from 10 ppt – 10 ppb

- Analytical methods developed for **DNT, TNT, RDX, PETN, TATP**
- Manuscripts submitted and/or published for each of these analytes

Online Verification

Agilent 5975C MSD

- Electron Impact
  - Rapid Identification of 1000 compounds
- Chemical Ionization
- Negative CI
  - Selectivity
  - Low Limits of Detection
    - ➤ TNT = 40 fmole
    - ➤ RDX = 100 fmole

Gerstel Online Cooled Inlet

- Adsorbs at 250 mL min^{-1}
  - 10 mL-10 L adsorption Vol.
- Cyro-cooled sorbent bed
- Rapid desorption (12°C sec^{-1})
- Variety of sorbents
  - Tenax TA^{TM}
  - CarboTrap C
  - CarboTrap B
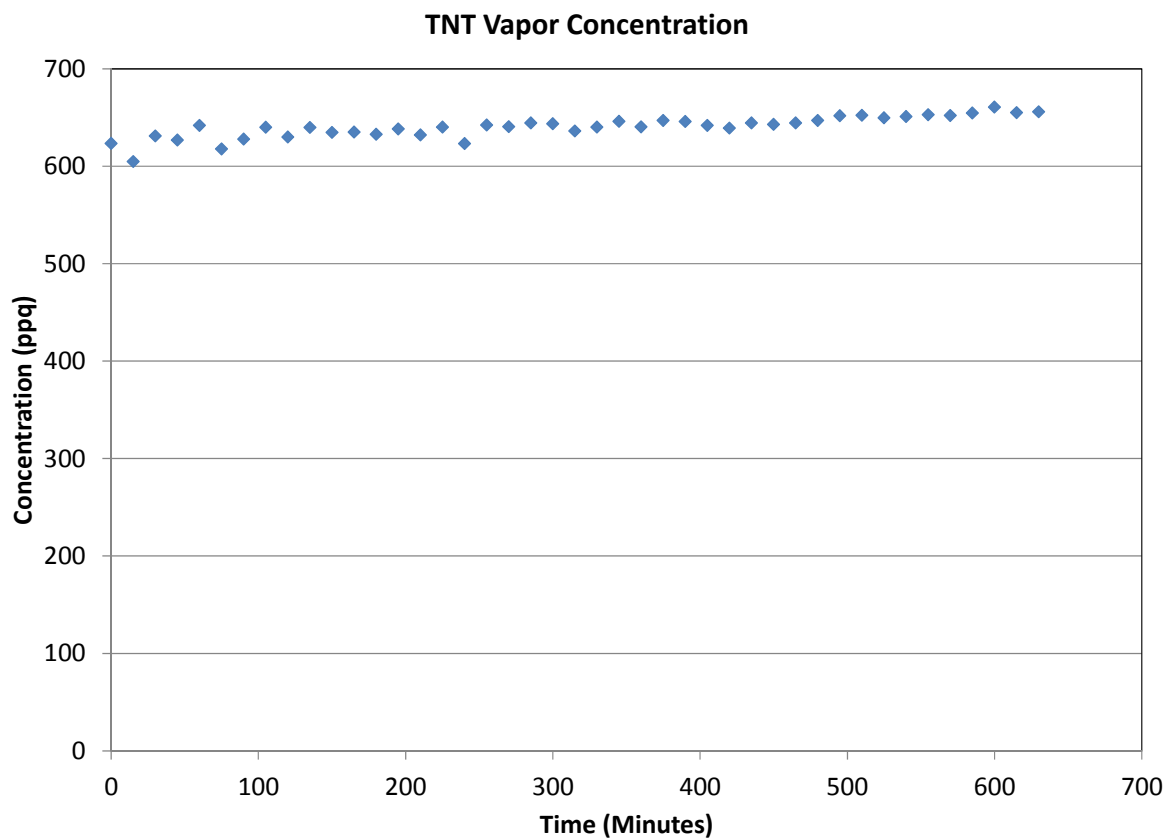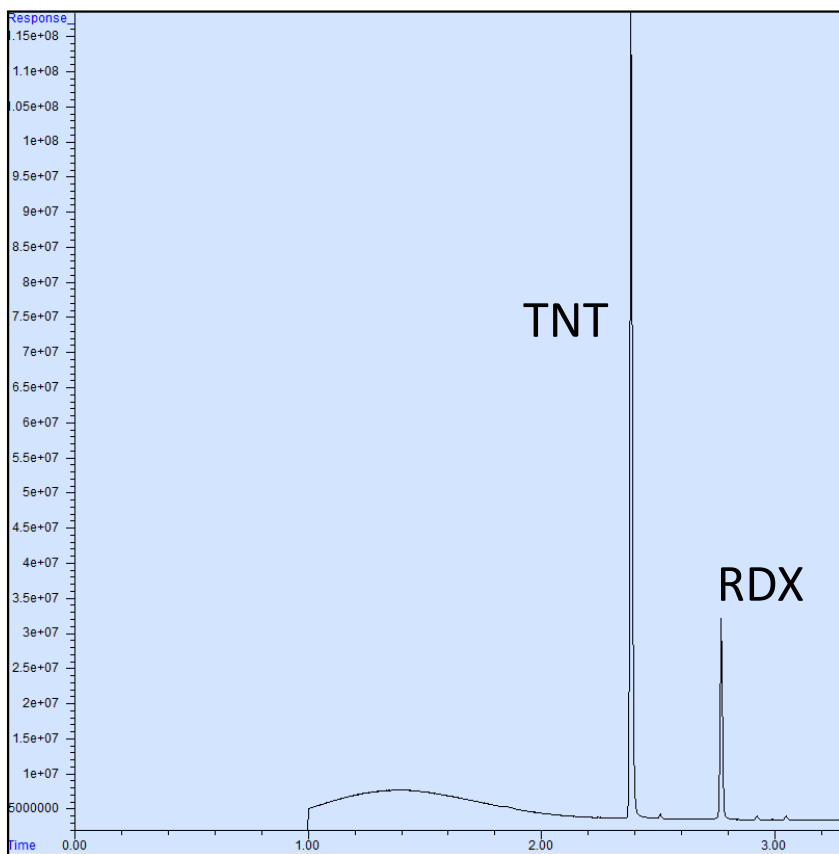 - Silanized Glass
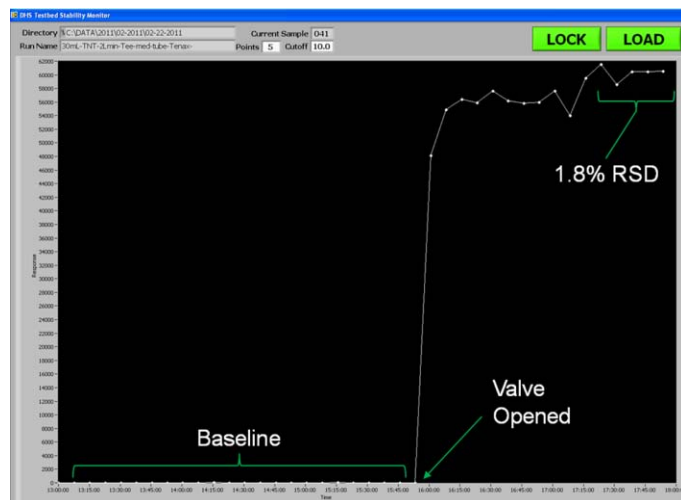
Agilent μECD

- Selectivity
- Low Limits of Detection
  - ➤ TNT = 440 fmole
  - ➤ RDX = 1.1 pmole
  - ➤ PETN = 1.5 pmole

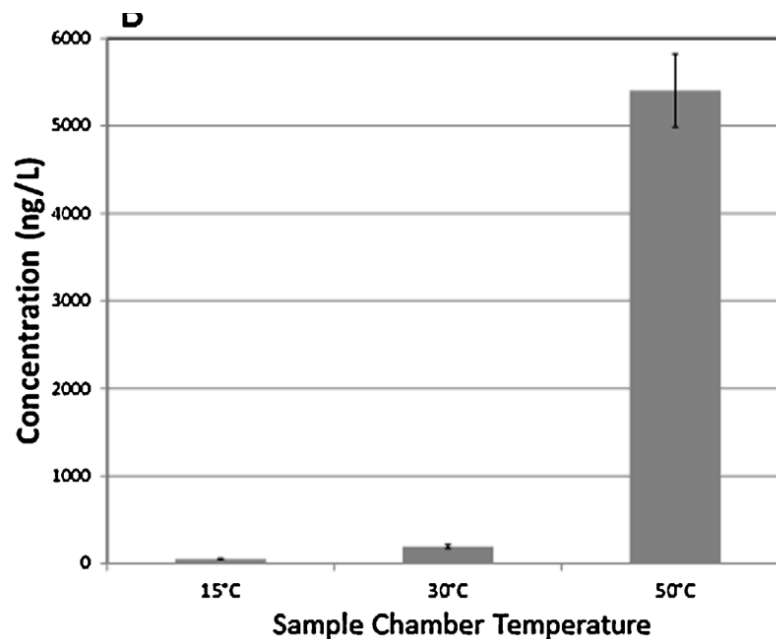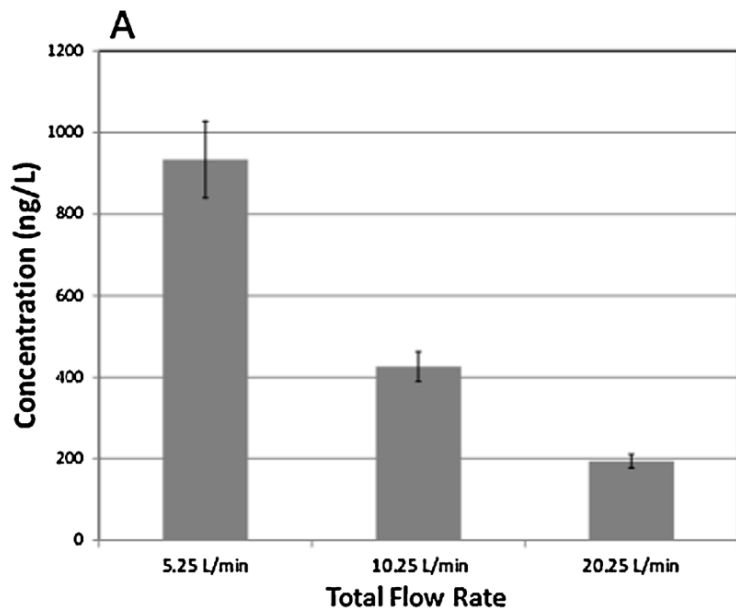# Parts per Quadrillion Generation of TNT

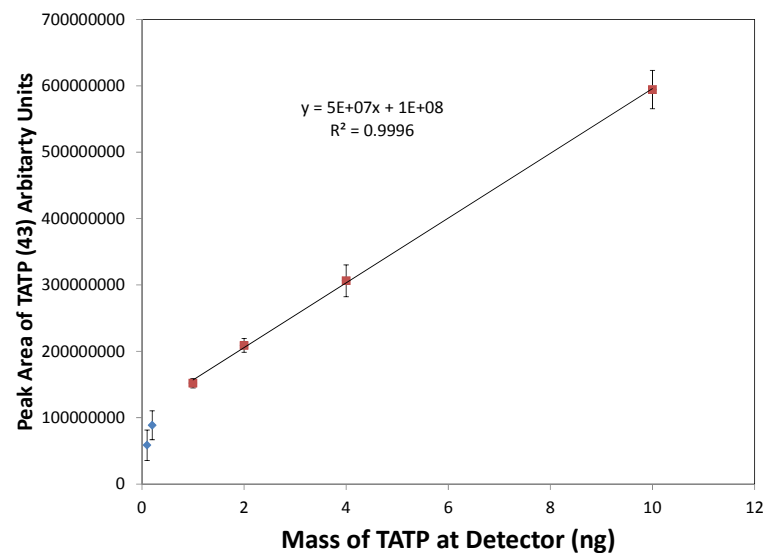A representative GC chromatogram of TNT and RDX collected on a Siliconert coated glass tube at 10°C
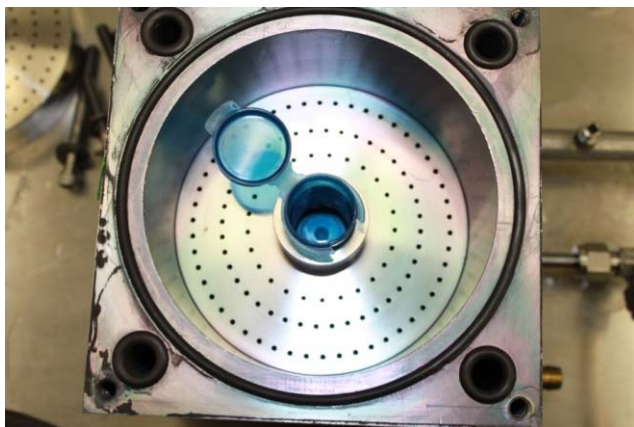

Average Concentration – 640 ppq
RSD – 1.7%

Online Programmable Temperature Vaporization (PTV) GC/MS for TATP

- TATP dynamic range of 50-5400 ng/L
- Verified using TDS-CIS-GC/MS

Vapor Validation – Lowest Concentration Quantified (LCQ)

Summary of the vapor concentrations detected in our laboratory. Realize that lower vapor concentrations are achievable with longer sample times.

| Explosive	Sat. Vapor Conc.*	TDS-CIS-GC		On-line CIS-GC	
		Sample Time (Vol.)	LCQ	Sample Time (Vol.)	LCQ
TNT	~9 ppb_v	60 min (6 L)[a]	3.4 ppt	13 min (0.66 L)[b]	640 ppq
RDX	~5 ppt_v	60 min (6 L)[a]	4.3 ppt	13 min (0.66 L)[b]	850 ppq
PETN	~11 ppt_v	30 min (3 L)[a]	12 ppt		
TATP	~63 ppm_v			1 min (0.025 L)[a]	5.5 ppb

[a] Sample was collected on Tenax-TA sorbent at or near 25°C
[b] Sample was collected on a Silconert coated glass tube at 10°C

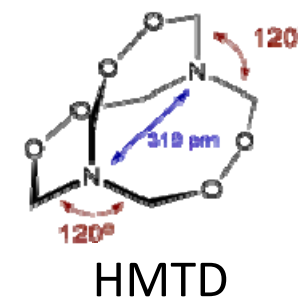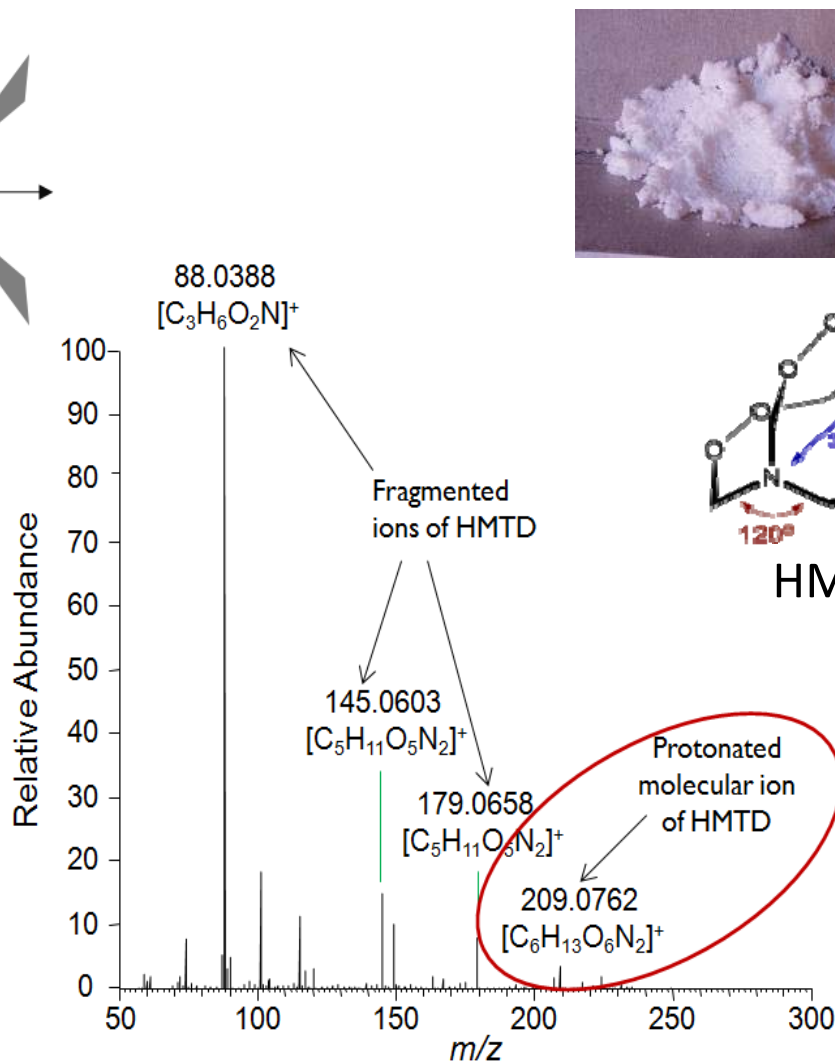* From "The Vapor Pressure of Explosives," Ewing *et al.* Trends in Analytical Chemistry, Vol. 42, 2013, 35-48

HMTD: Ambient Ionization using FAPA
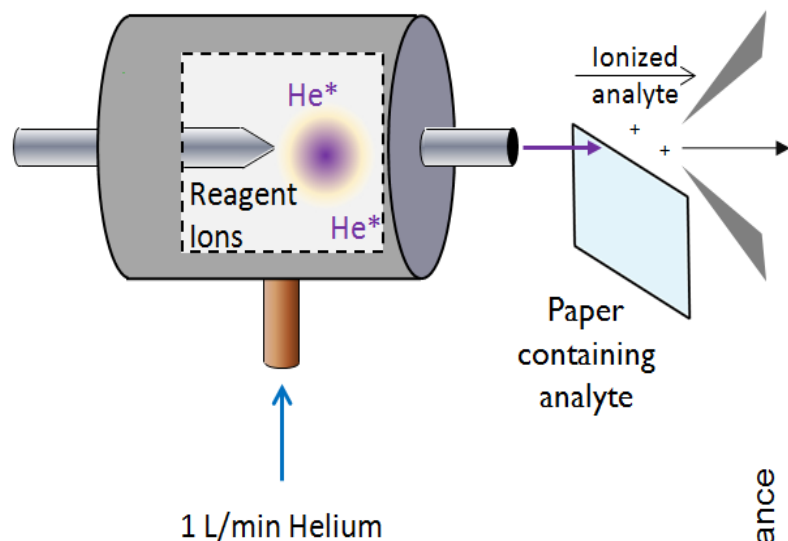
Protonated molecular ion (209m/z) and fragment ion detected
*estimated LOD in low ng range

Mass spectrum peaks:
- 88.0388 $[C_3H_6O_2N]^+$
- Fragmented ions of HMTD
- 145.0603 $[C_5H_{11}O_5N_2]^+$
- 179.0658 $[C_5H_{11}O_6N_2]^+$
- 209.0762 $[C_6H_{13}O_6N_2]^+$ — Protonated molecular ion of HMTD

HMTD

HMTD parent molecule observed using Flowing Atmospheric-Pressure Afterglow-Mass Spectrometry (FAPA-MS) with sample deposited onto a piece of paper at the nanograms/mm² level
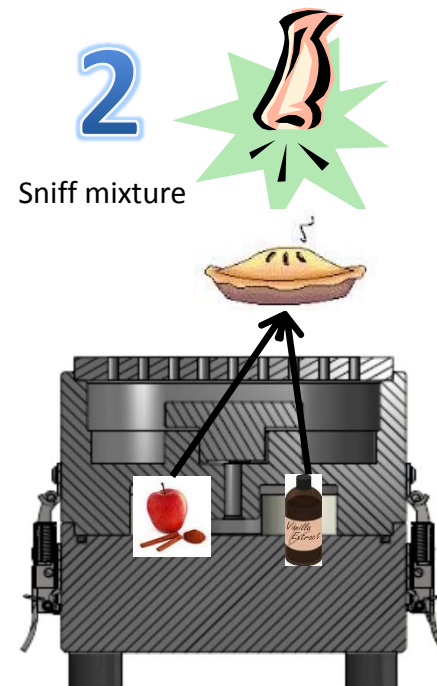
Related Program:
Mixed Odor Delivery Device (MODD)

A device has been designed to safely contain separated binary explosive components and deliver a mixed vapor to canine or instrumental detectors

1 Sniff components

2 Sniff mixture

Passive design
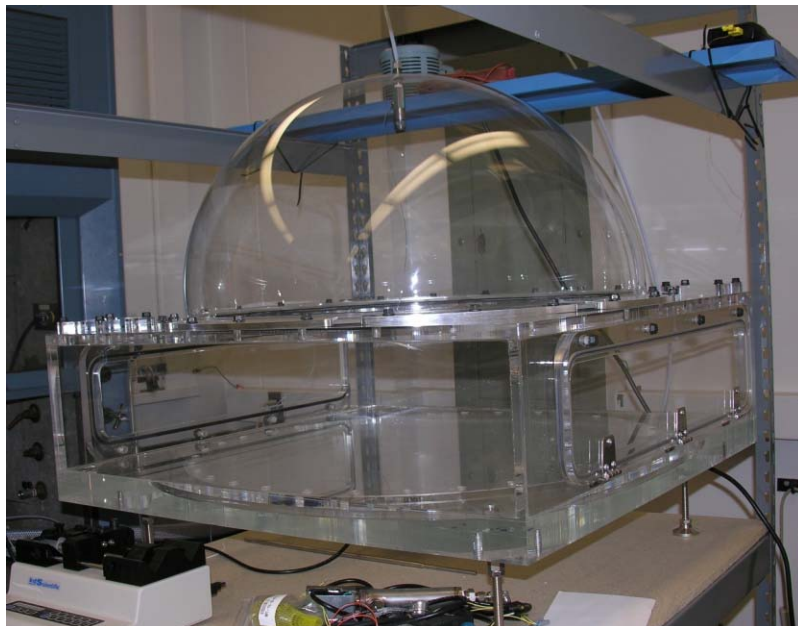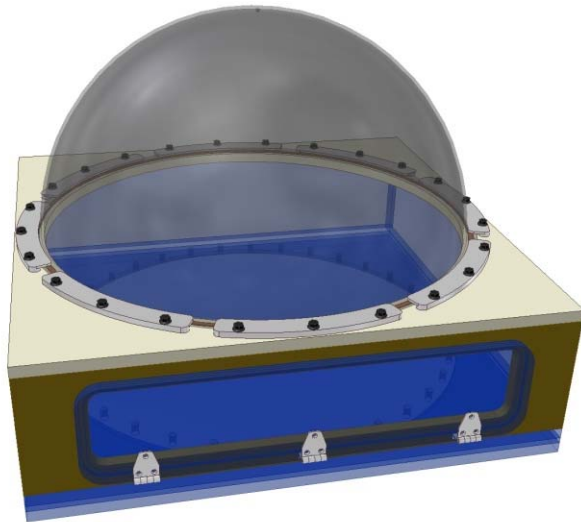
Active design with heated chamber

- Safely monitor dangerous IED mixtures: e.g., ANFO or AN/Al
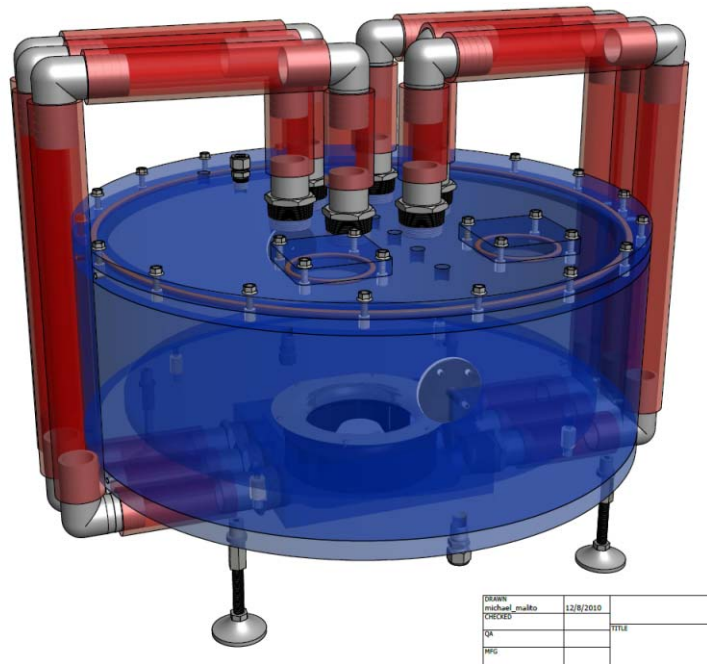- Vapor mixed from four separate PFA jars
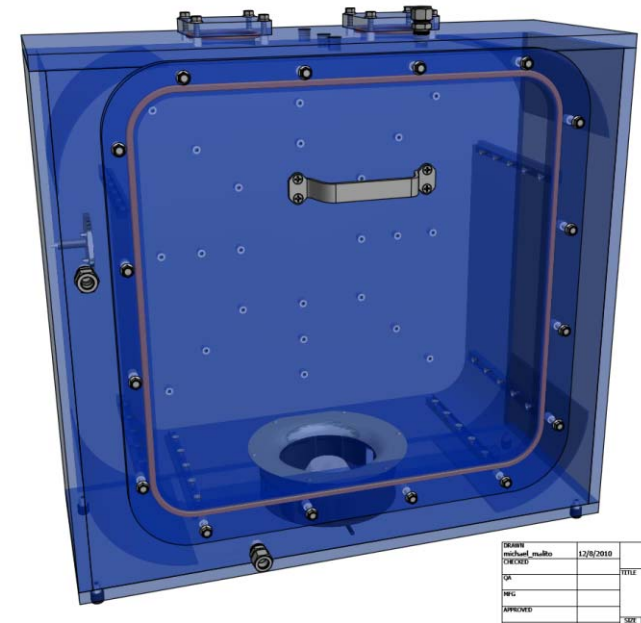
High Vapor Pressure Analyte Chambers
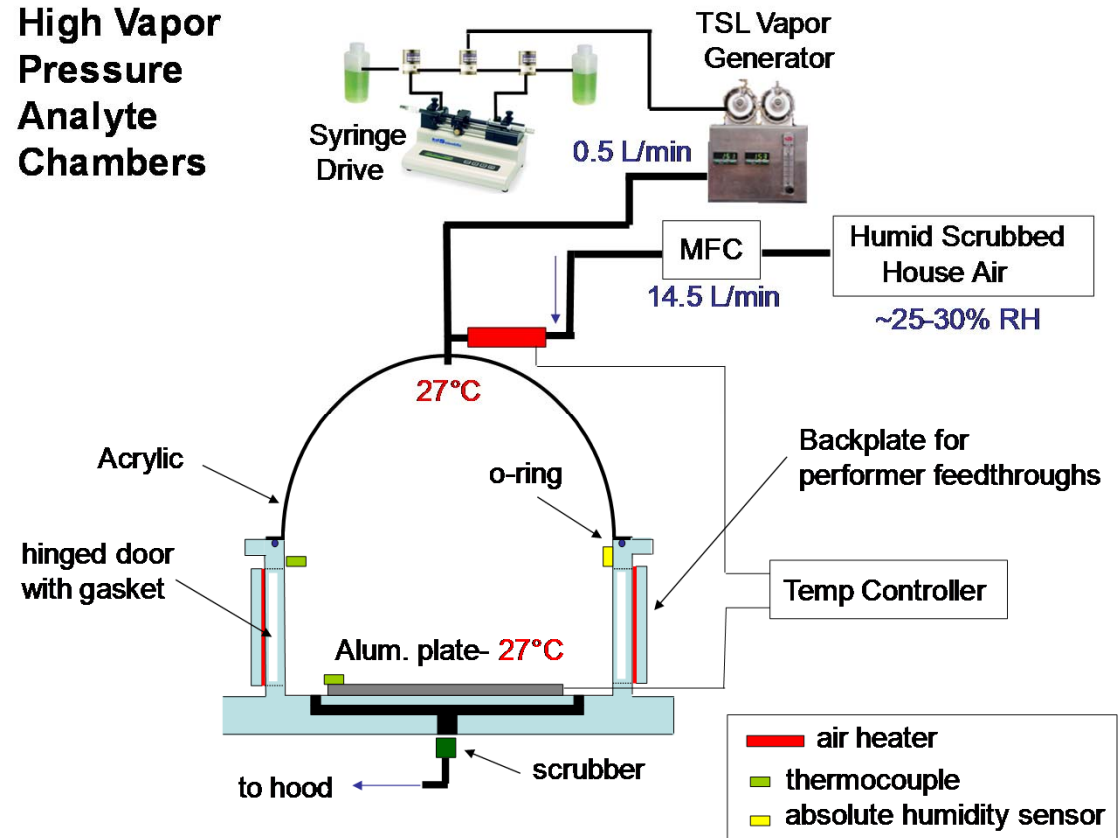
The testbed provides uniform, reliable methods for evaluating large scale, explosives detection systems for personnel and platforms protection.

Horizontal Chamber Design for Particles

Vertical Chamber Design

- Evaluation of large scale explosive detection systems for personnel and platforms' protection
- Fan included to promote circulation
- Particles can be introduced, e.g. Arizona Road Dust, to generate explosive/particle mixtures

LASR-Sensor Lab

Sensor Lab Facilities

- Walk-in environmental chamber
  - 10' X 15'
  - Temp: -30-50ºC, Humidity: 10-95% RH
  - Walls that can be washed down
- CSZ Temperature/Humidity /Altitude Chamber
- Aerosol Lab with wind tunnel

Other Facilities

- Desert High Bay
- Littoral High Bay
- Tropical High Bay
- Reconfigurable Prototyping High Bay
- Power and Energy Lab
- Human-System Interaction Labs
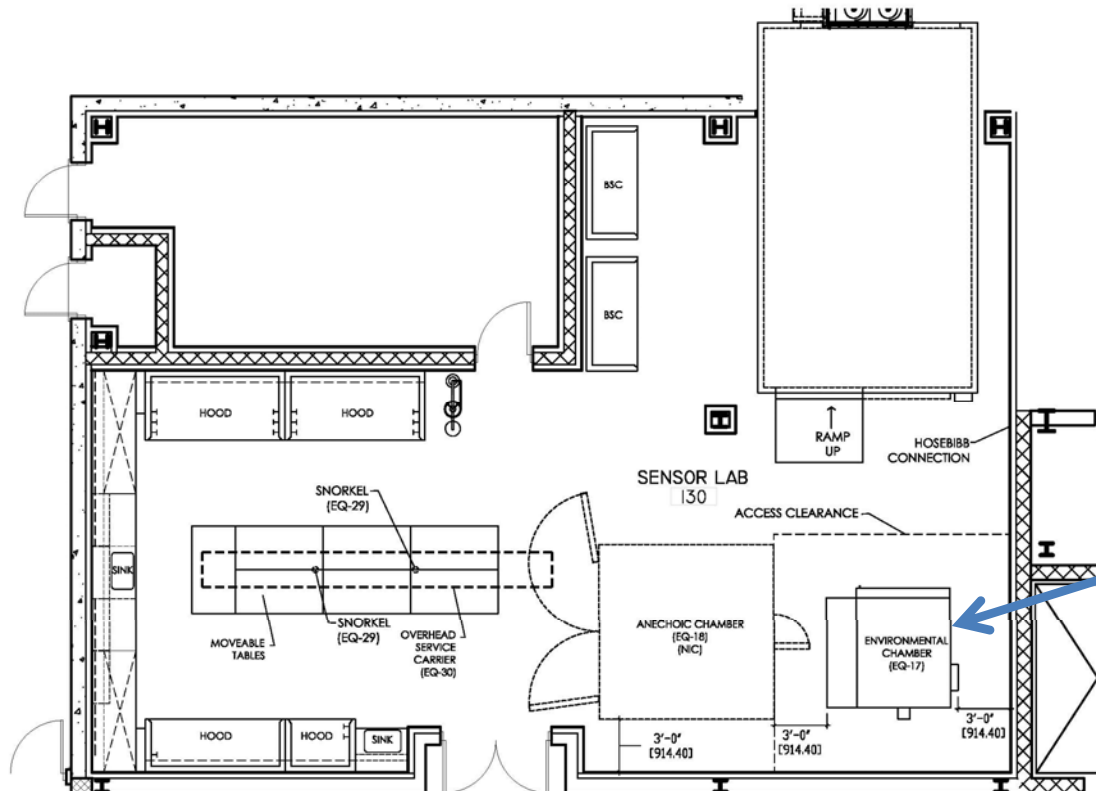
Acknowledgements

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