NIST Surface and Microanalysis Science Division
Trace Explosive Particle Detection Research

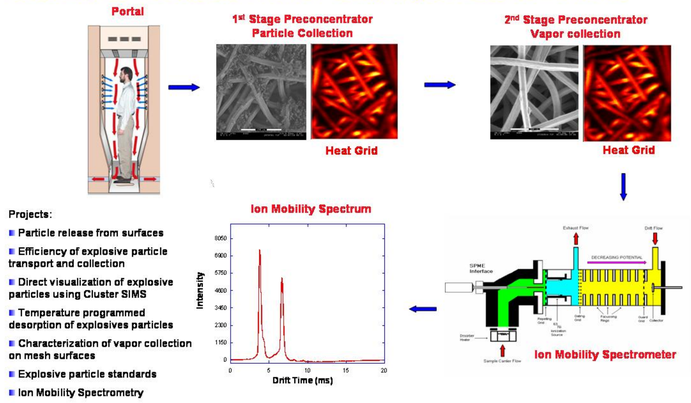
INTRODUCTION

New research programs at NIST: (1) Develop a measurement infrastructure to characterize, calibrate and standardize portal-based trace explosive detection systems. Funding from NIST Advanced Technology Program. Proposal developed with input from the Transportation Security Agency (TSA) Trace Explosives Detection Group, the Contraband Detection Technology Group at Sandia National Laboratory and the Gas Dynamics Laboratory at Penn State. (2) Collaborate with the TSA to study the performance characteristics of swipe trace explosive detection instruments.

EXPERIMENTAL GOALS

Use advanced microscopy and surface analysis tools at NIST to study the performance characteristics of each component of trace explosive detection portals/swipe detection instruments. Improve sensitivity, collection efficiency and operational reliability of explosive detection instruments. Conduct a complete microscopic and chemical characterization of individual explosive particles and their thermal degradation behavior during the sampling process. Perform research into the development of calibration procedures and NIST- traceable standard materials for explosive particle detection.

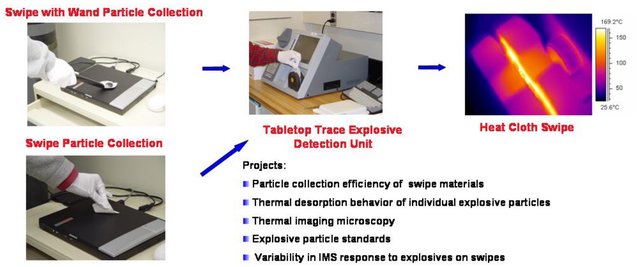
Trace Explosive Detection Portal- Sampling of Airborne Particles



Projects:

- Particle release from surfaces
- Efficiency of explosive particle transport and collection
- Direct visualization of explosive particles using Cluster SIMS
- Temperature programmed desorption of explosives particles
- Characterization of vapor collection on mesh surfaces
- Explosive particle standards
- Ion Mobility Spectrometry

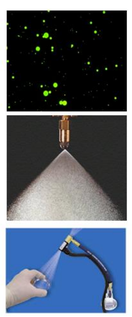
Swipe Detection of Explosive Particles



Projects:

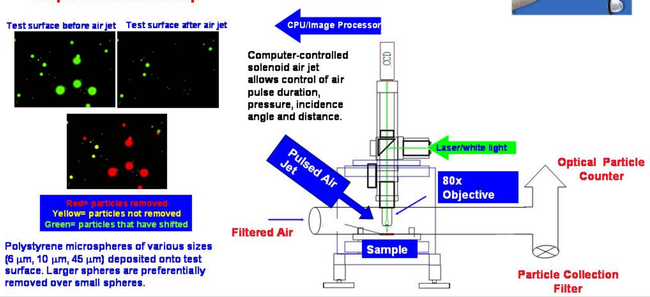
- Particle collection efficiency of swipe materials
- Thermal desorption behavior of individual explosive particles
- Thermal imaging microscopy
- Explosive particle standards
- Variability in IMS response to explosives on swipes

Particle Removal From Surfaces



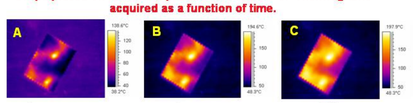
- Critical front end sampling process for both portals and swipe collection instruments. Need to identify fundamental processes involved in efficient particle removal from surfaces.
- Determine size distribution of particles removed from surfaces. If particle size distribution is known, portal can be designed for more efficient detection.
- Develop "standard surface" for particle release studies. Useful for both air jet removal using portals and swipe systems. Initial studies involve uniform deposition of a controlled number of fluorescent tagged polymer spheres on filter surfaces. Computer controlled air jet allows control of air pulse duration, pressure, angle and distance.
- Explore improved methods for particle removal using pulsed air jets, CO_2 jet spray, ionized air gun.

Experimental Setup

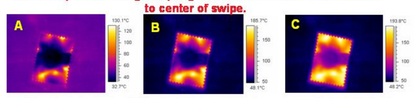


Infrared Thermograhy for Non-Contact, Spatially-Resolved Thermal Imaging of Swipes

Swipe placed on aluminum plate heated to 200 °C. Thermograms acquired as a function of time.



Same swipe after using sampling wand. Note variable heat transfer to center of swipe.



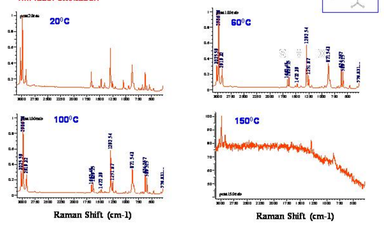
Note: Temperature scale not NIST calibrated. Data acquired using camera manufacturer's standard settings. Infrared camera calibration (NIST traceability) and determination of the emissivity of swipe materials is being conducted in a collaboration with the NIST Physics Optical Thermometry Group.

Efficient thermal desorption of explosive particles from the collector surface while minimizing degradation of the material is a key process for effective detection of explosives by ion mobility spectrometry. Variable heating of the collection medium is potentially a source of run-to-run variation. We have acquired a FLIR systems infrared camera for non-contact, spatially and temporally resolved temperature measurements. This data is being correlated with variable temperature Raman microscopy, scanning electron microscopy, optical microscopy and ion mobility spectrometry data.

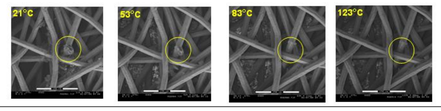
Micro-Raman Spectra of a PETN Particle vs. Temperature

Single PETN particle (~size 20 µm x 38 µm). Spectra acquired using 532.0 nm laser excitation

Pentaerythritol, tetranitrate
- $C_5H_8N_4O_{12}$
- Molecular Weight: 316.14



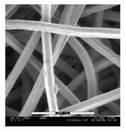
Morphological Changes in SEMTEX Particles as a Function of Temperature Using the Environmental Scanning Electron Microscope.



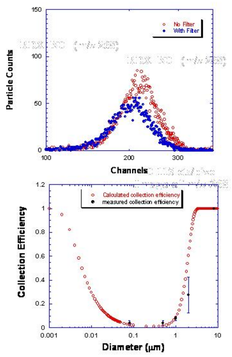
High Volume Sampling – Particle Collection Efficiency Testing of Portal Filters

Filters used for collection of explosive particles were selected for high air flow and to allow resistive heating. Is this configuration optimal for efficient particle collection of explosives of the targeted size range?

- Develop capability to characterize particle collection efficiency of portal filters.
- High air volume particle collection, what are special issues to be considered?
- Model filter collection efficiency and optimize filter design.
- Explore novel ways to improve collection efficiency of filters.



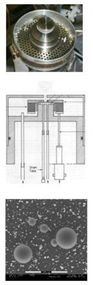
Metal fiber filter



Generate Test Aerosol

Aerosol
- Vibrating Orifice Aerosol Generator
Solid or liquid aerosol, e.g., Sodium Chloride, Ammonium Fluorescein, Oleic Acid
- Particle size Calibration to Aerodynamic Diameter via Sedimentation Velocity
- 100 % Collection Efficiency for 8 µm Aerodynamic Diameter (liquid particles)

Polystyrene Spheres
- Aerosol produced by Collision Nebulizer
- 27 % Collection Efficiency for 2 µm Aerodynamic Diameter (see plot)



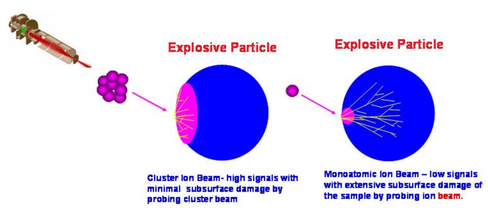
Measure particle collection efficiency of Berkaert 60AL3 portal filters using monodisperse test aerosols

Metal fiber filter testing unit

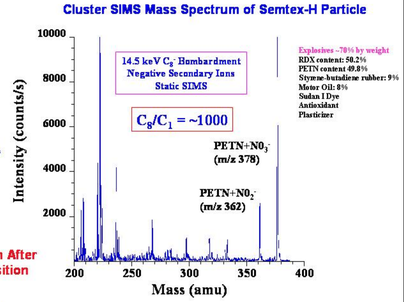
Preliminary Results
Observed efficiency of 60 AL3 is consistent with theoretical filter efficiency curve. Collection efficiency is very dependent on size of collected particles.

Particle Collection Efficiency Testing using Secondary Ion Mass Spectrometry

In a typical collection we are looking for a very few explosive particles in a large background of environmental particles. Even pure standard materials typically contain many non-explosive particles. We need to develop analytical methods to individually identify and count explosive particles. The unique Cluster Secondary Ion Mass Spectrometry Capability at NIST is being evaluated for this purpose. The use of energetic cluster primary ion beams can give orders of magnitude improvements in characteristic molecular signal with little degradation of the sample. This approach allows for the rapid identification and imaging of explosive particle distributions on surfaces.

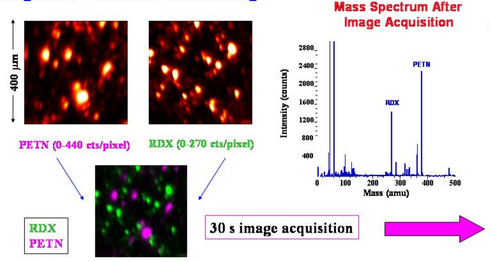


Cluster SIMS Mass Spectrum of Semtex-H Particle



Explosives ~ 70% by weight
RDX content: 50.2%
PETN content 49.8%
Styrene-butadiene rubber: 9%
Motor Oil: 8%
Sudan I Dye
Antioxidant
Plasticizer

Organic Particle Searching



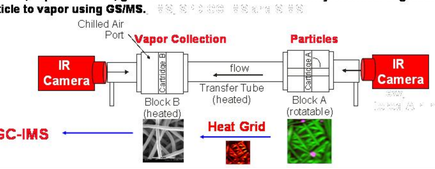
PETN and RDX Particle Overlay Showing Distribution of Explosive Particles on Surface

Using computer controlled movement of the sample stage, a series of individual images are taken to produce a mosaic of the distribution of explosive particles on a surface. The intact parent molecular ion signal is used to identify the particles. Particle size, location & identification are tracked.

Characterization of 2nd Stage Preconcentrator

The efficiency of the conversion of explosive particles to vapor and the subsequent collection of this vapor on the second stage preconcentrator grid is another key component of portal detection systems. A testing system is under construction to evaluate the efficiency of 2nd stage preconcentration technology for explosive vapor collection.

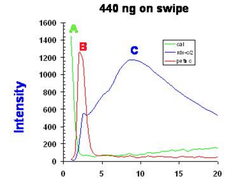
- **Primary filter (particle collection)– deposit explosive particles/solutions. Vary heating parameters, filter material, particle type and loading.**
- **Collection filter (vapor collection) – vapor collection efficiency to be measured as function of collector temperature, filter material, explosive used, gas flow and time. Evaluate efficiency/chemical degradation in the conversion of the particle to vapor using GS/MS.**



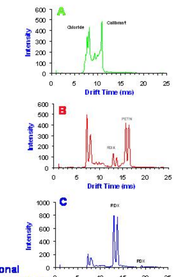
Fundamental Aspects of Ion Mobility Spectrometry

Temporal Variation in Response to Explosives

TSA Semtex Standard
440 ng on swipe



Temporal thermal desorption IMS profiles provide additional information about the composition of the sample and its behavior during heating. We are studying the influence of particle size and desorber temperature on the desorption profiles. This will be correlated with temperature programmed Raman, SIMS and ESEM analysis.



New IMS instrument acquired by NIST for studying fundamental aspects of ion mobility spectrometry. Useful addition to existing capabilities in IMS, GC-MS and MS

- **GC-IMS Mode for quantitative analysis, improved identification of complex samples. Analysis time < 3 minutes.**
- **SPD-GC-IONSCAN- direct headspace analysis of solid samples. Can also be used to examine portal grids.**
- **SPME Interface**

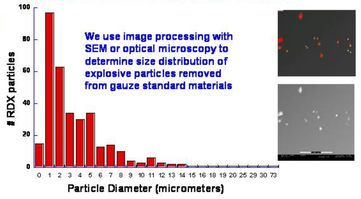
Standards for Explosive Particle Detection - Microscopic Characterization of Test Materials

What are the chemical and morphological properties of standards currently used to test portal systems? At the microscopic level, what are we trying to sample with the portal system?

RDX Particles on Gauze Test Material

Penn State Standard RDX on Gauze

We use image processing with SEM or optical microscopy to determine size distribution of explosive particles removed from gauze standard materials



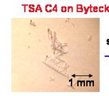
Standards Research

Currently, for particle removal and swipe efficiency experiments we are using uniform deposition of various sizes of monodisperse, fluorescent tagged, polymer microspheres. Ongoing studies include:

- **Attempt to incorporate a known amount of explosive into/onto the tagged spheres. These composite spheres would allow for single particle metrology and counting as well as providing explosive concentration standards for IMS.**
- **Direct preparation of explosive particles using fluidized bed or vibrating orifice particle generators.**
- **High Explosives adsorbed onto a solid particulate matrix (NIST Analytical Chemistry Division).**

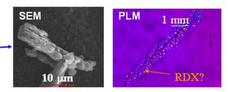
TSA Test Explosive Standard Materials

TSA C4 on Byteck



swiped

Luggage Handle



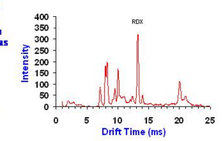
Deposit of dendritic crystals that stays intact after swiping to luggage handle

Each dendrite is two-phased with µm-sized RDX? crystals buried by overgrowth phase. Overgrowth phase is also primarily C, N, O, but with some areas showing Na contamination.

Semtex

Detasheet

Non-crystalline droplet like material-What will be sampled in Portal?



Ion Mobility spectrum of RDX doped polystyrene sphere

Summary

The NIST Surface and Microanalysis Science Division is currently involved in a research program to develop a measurement infrastructure for the improvement, characterization and standardization of both portal and swipe-based trace explosive detection instruments. Key areas of focus include:

- **Explosive particle release from surfaces**
- **Explosive particle collection**
- **Ion mobility detection of explosives**
- **Standards for explosive detection**

For more information:

NIST
greg.gillen@nist.gov
robert.fletcher@nist.gov

TSA
richard.lareau@tc.faa.gov