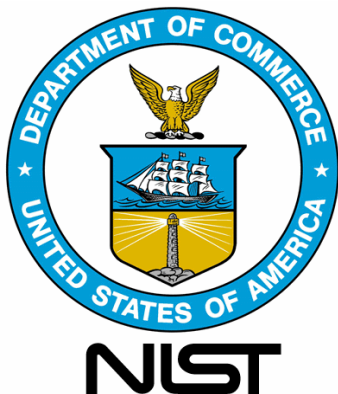# Advanced Metrology to Support IMS Trace Explosive Detection: NIST Capabilities and Progress

Greg Gillen, Robert Fletcher, Jenny Verkouteren, George Klouda, Cynthia Zeissler, Andrew Evans, DiAundra Davis, Maria Santiago, and Mike Verkouteren
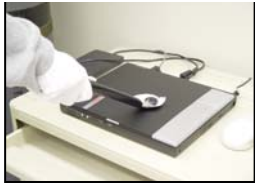
Surface and Microanalysis Science Division

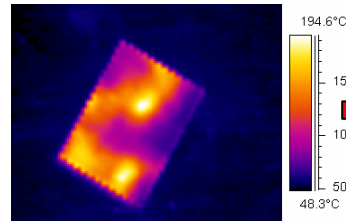**National Institute of Standards and Technology
Gaithersburg, Maryland**

ISIMS Conference, Gatlinburg, Tennessee, July 26-29, 2004
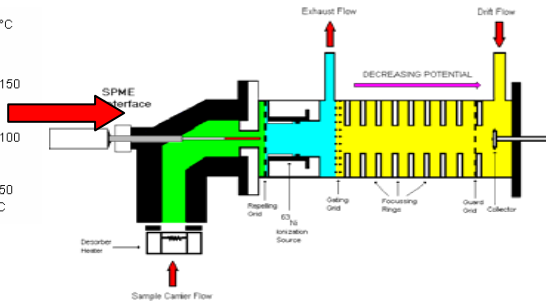
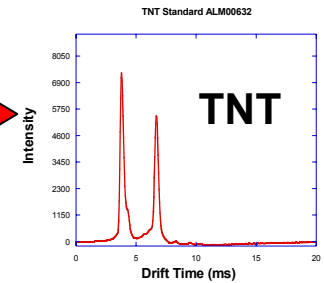# Trace Explosives Detection - Background

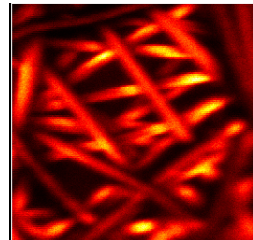**Swipe Sampling**

Swipe Heating

Ion Mobility Spectrometry
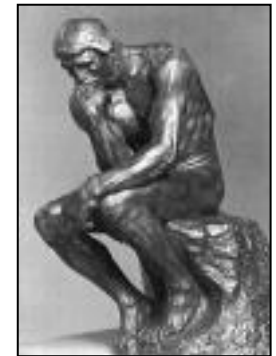
Data Processing

Portal Sampling

**Filter Heating
Vapor Preconcentration**

**Interpretation and
Decisions**

- Traces of material are transferred by handling

- Particles are key: VP HEs low; 10µm RDX particle → 1 ng

- Growing deployment: ~20,000 IMS instruments worldwide. 7000 at US airports. Also, embassies, stadiums, courthouses, etc

- Growing need to strengthen metrology and establish reliable standards to support widespread deployment. Address methods, performance metrics, training, intercomparability, measurement uncertainty, etc

Outline

Fundamental Sampling Issues

Portals

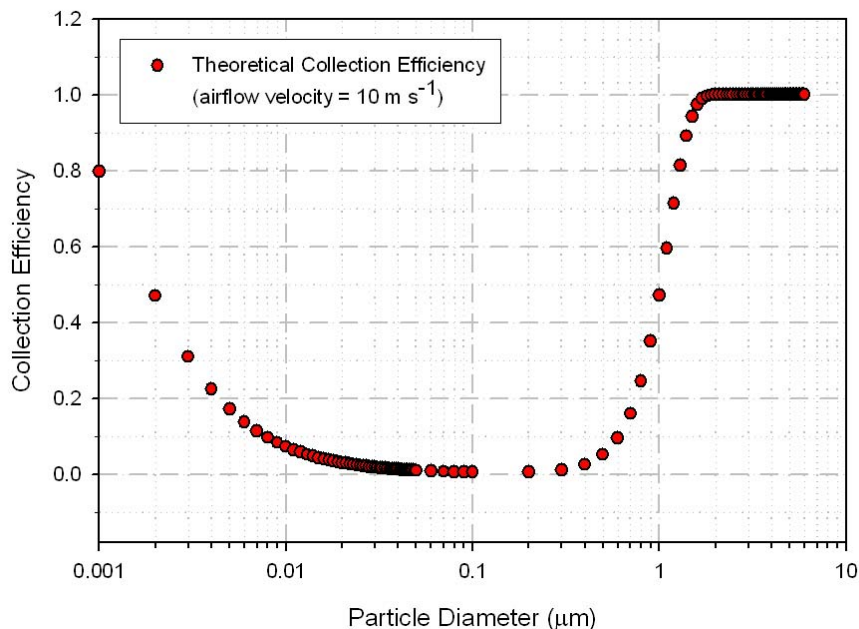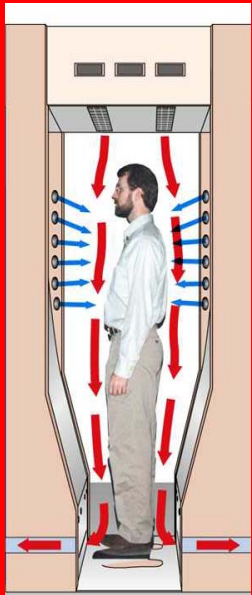*What's the sample?*

Vapor Preconcentration

Standard test-bed

Thermal desorption

Standards

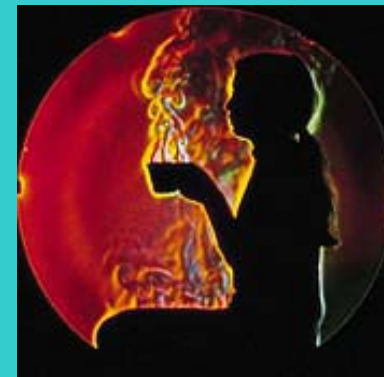*Ink-jet printing*

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Technology Administration, U.S. Department of Commerce

Particle Sampling Using Portals

Air Shower Design

Human Thermal Plume

Plot legend: Theoretical Collection Efficiency (airflow velocity = 10 m s^{-1})
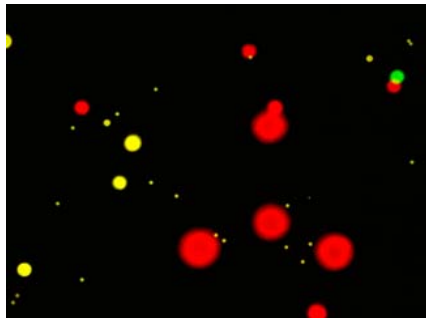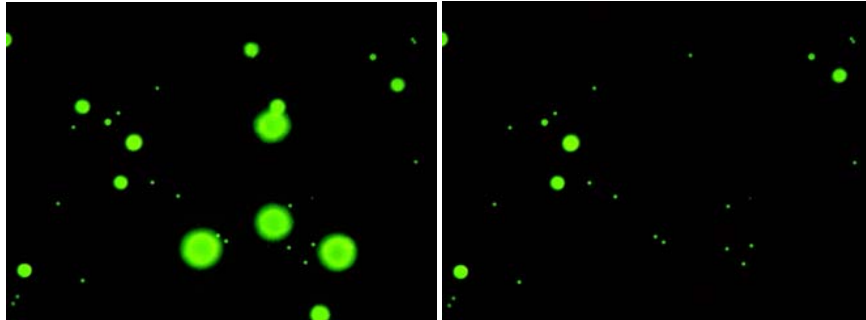
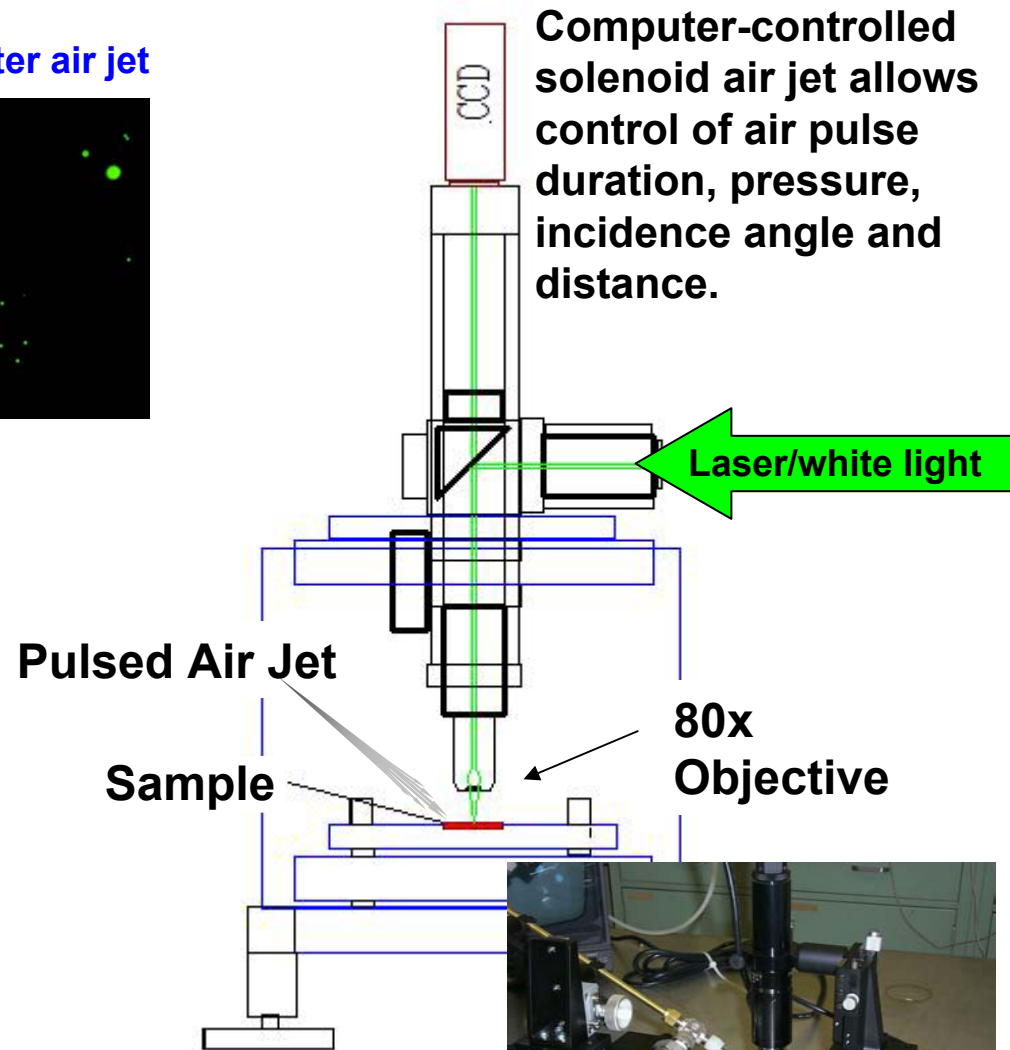Y-axis: Collection Efficiency
X-axis: Particle Diameter (μm)

- Collaboration with TSA

- Characterize particle removal efficiency of air jets

- Model and measure particle collection efficiency of portal filters

- Explore ways to improve efficiencies of particle removal and collection

Particle Removal From Surfaces - Methodology

Test surface before air jet **Test surface after air jet**

Red= particles removed
Yellow= particles not removed
Green= particles that have shifted

Computer-controlled solenoid air jet allows control of air pulse duration, pressure, incidence angle and distance.

CCD

Laser/white light

Pulsed Air Jet

80x Objective

Sample

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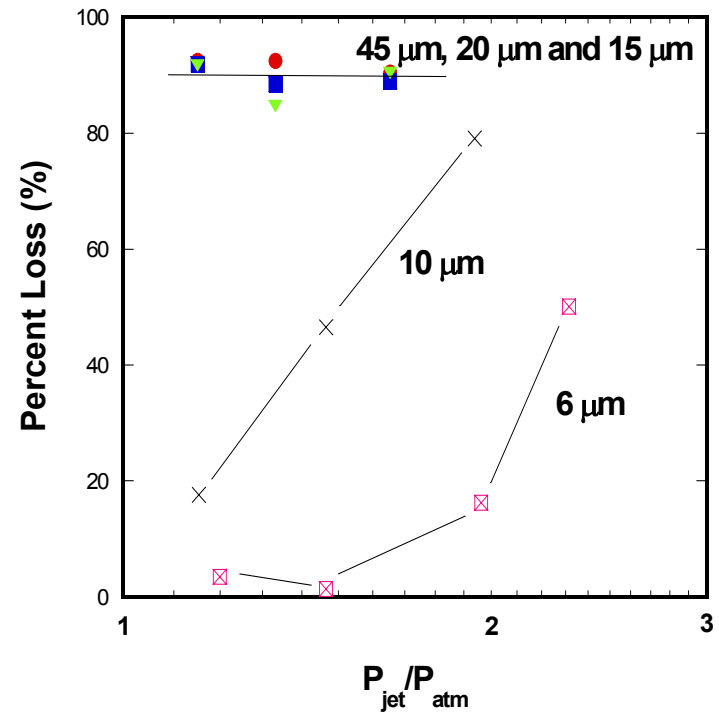# Particle Removal from Test Surfaces Using Air Jets
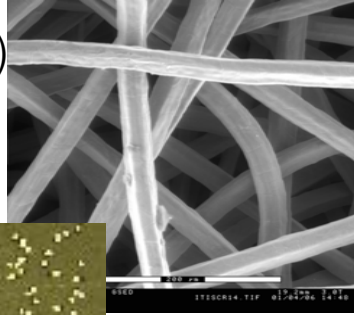
Polycarbonate Surface
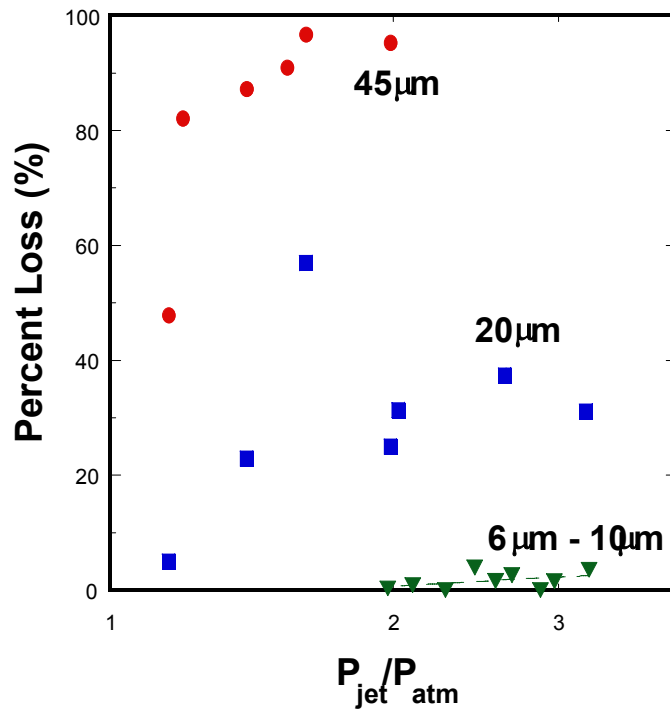
Muslin Cloth

Particle Collection Efficiency of Filters

Air Shower Filter
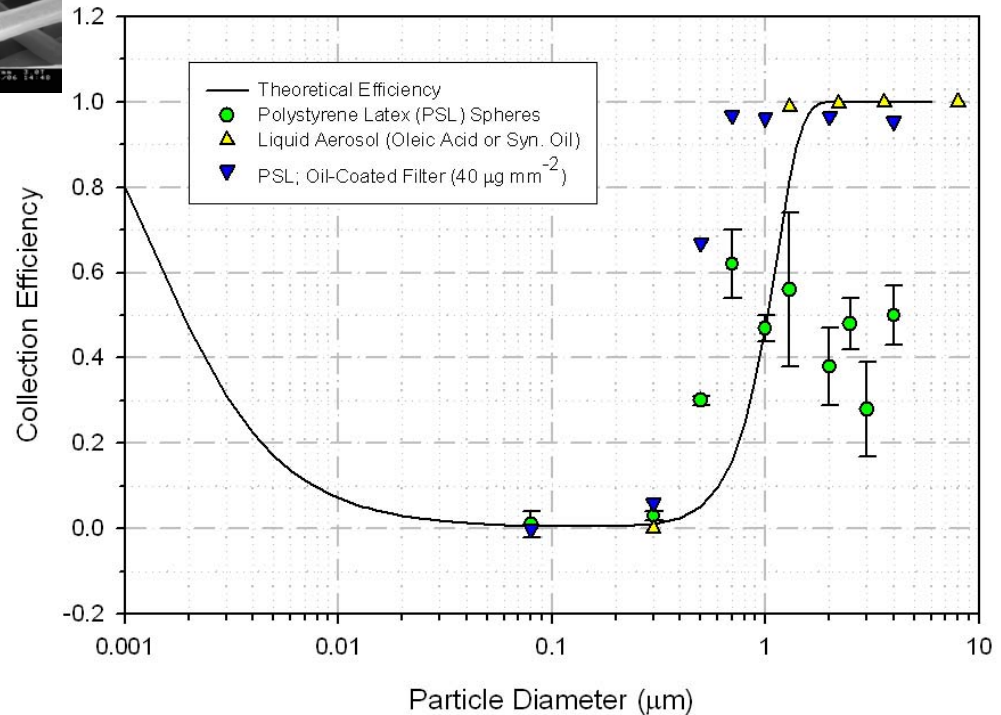- Metal Fiber Mesh (25-50 μm)
- Graded Pore Structure

X-Section
(700 μm)

Air Shower Portal Design

Collection Efficiency Results:

- Consistent with theoretical predictions for oil-coated filter or "sticky" particles

- Polystyrene latex spheres + dry filter interaction deviates from prediction

- Verified: efficiency optimized when targeting and sampling coarse particulate matter

Legend:
- Theoretical Efficiency
- Polystyrene Latex (PSL) Spheres
- Liquid Aerosol (Oleic Acid or Syn. Oil)
- PSL; Oil-Coated Filter (40 μg mm^{-2})

X-axis: Particle Diameter (μm)
Y-axis: Collection Efficiency

Outline

Particle Sampling Issues

Portals

What's the sample?

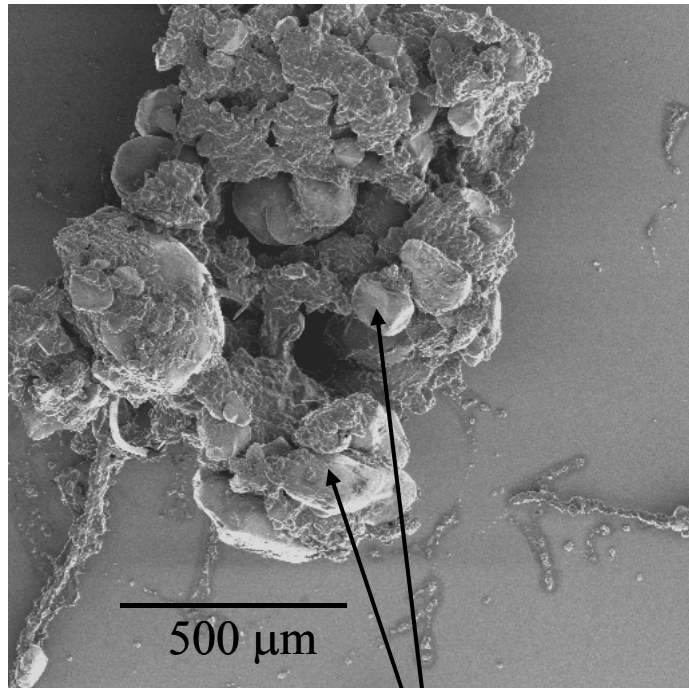Vapor Preconcentration

Standard test-bed
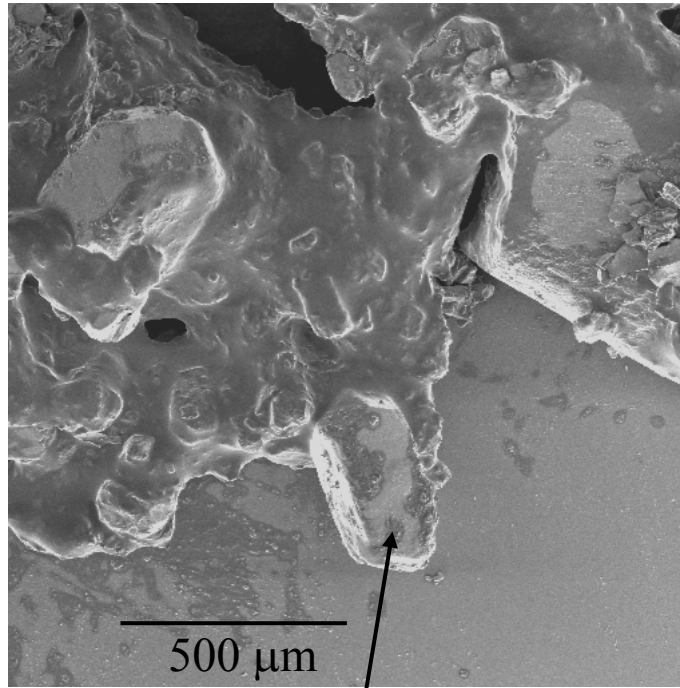
Thermal desorption
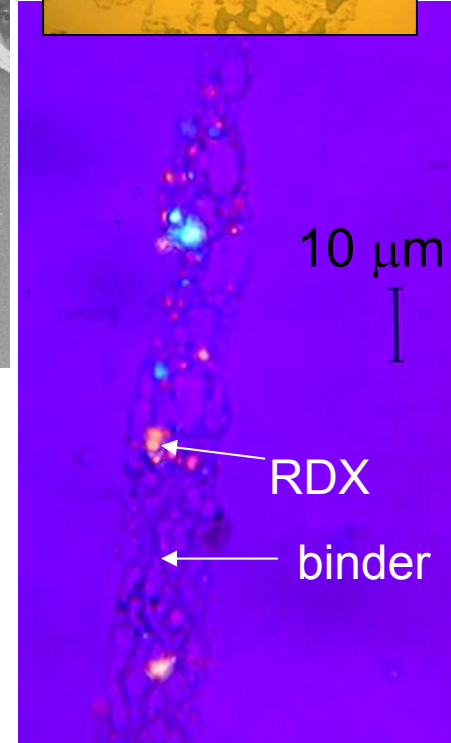
Standards

Ink-jet printing

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Formation of Explosives Residues

fingerprint

RDX in
C-4

Mil-specs for C-4:
75% 850 μm, 25% 44 μm

PETN in
Semtex-A

10 μm

RDX
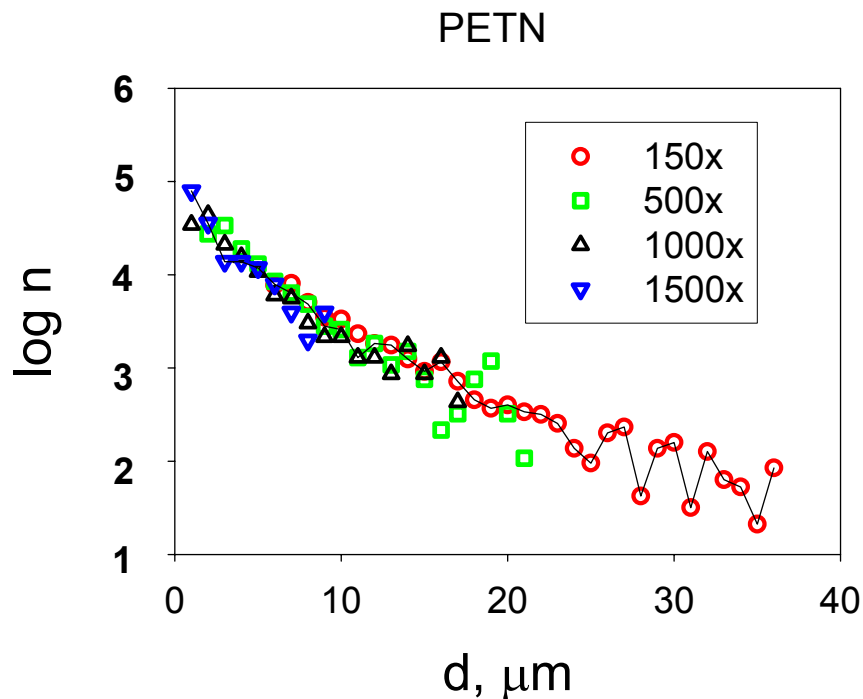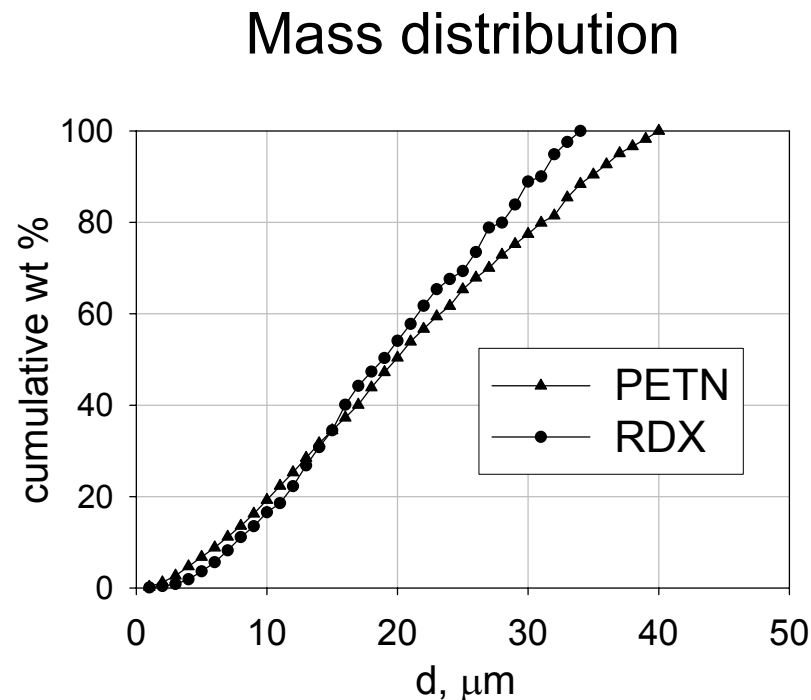
binder
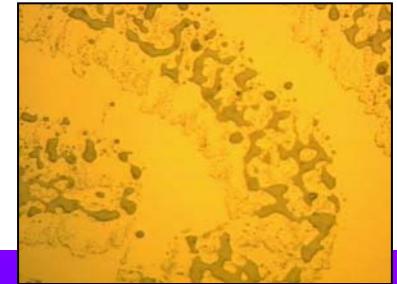
Polarized Light
Microscopy Image

Particle Size & Mass Distributions of Explosives in Residues

PETN

- Extract crystals from C-4 and Semtex-A
- Crush, deposit on filter
- Count by imaging random fields at 4 magnifications

Mass distribution

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Outline

Sampling Issues

Portals

What's the sample?

Vapor Preconcentration

Standard test-bed

Thermal desorption

Standards

Ink-jet printing
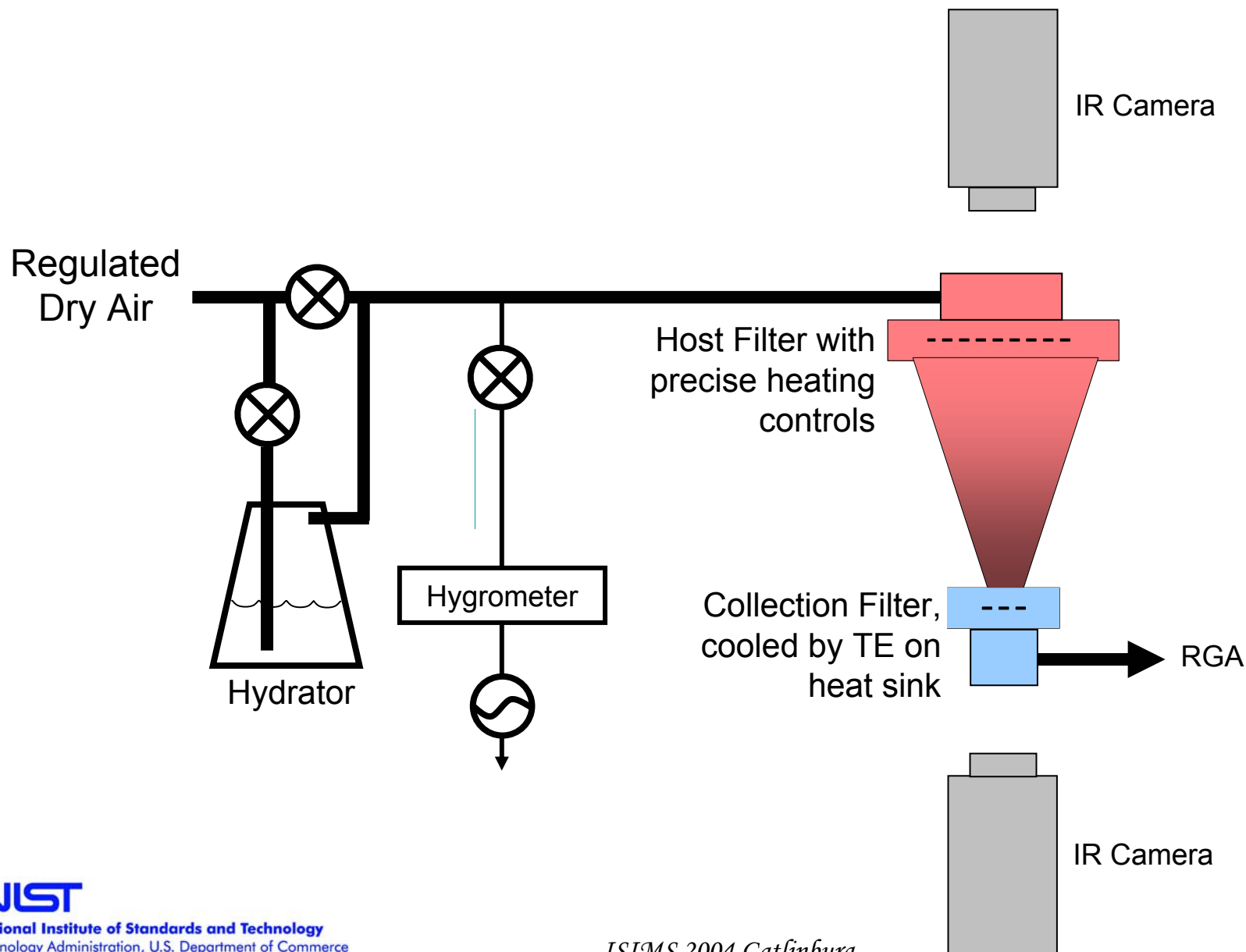
Vapor Preconcentrator
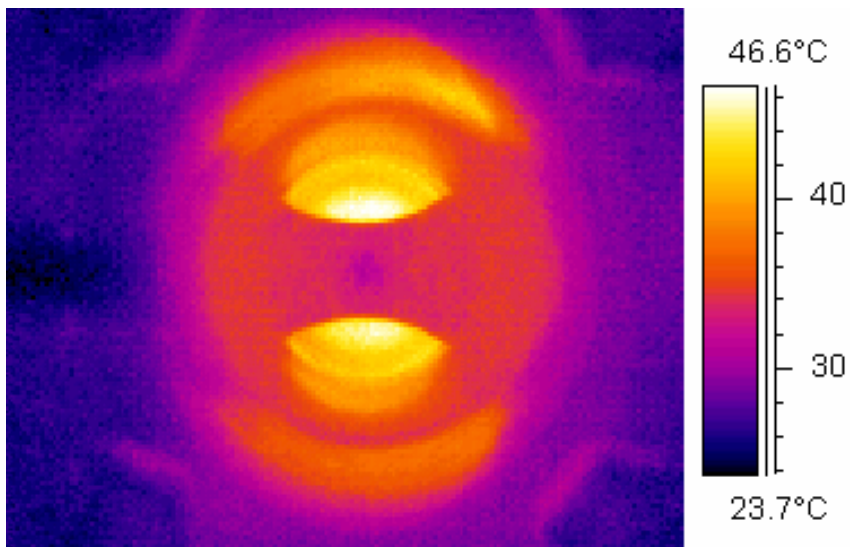## NIST Standard Test Bed

- ## Control over
  - ### – Air stream rate and humidity
  - ### – Host filter heating and configuration
  - ### – Transfer tube temperature and configuration
  - ### – Collection filter material and temperature

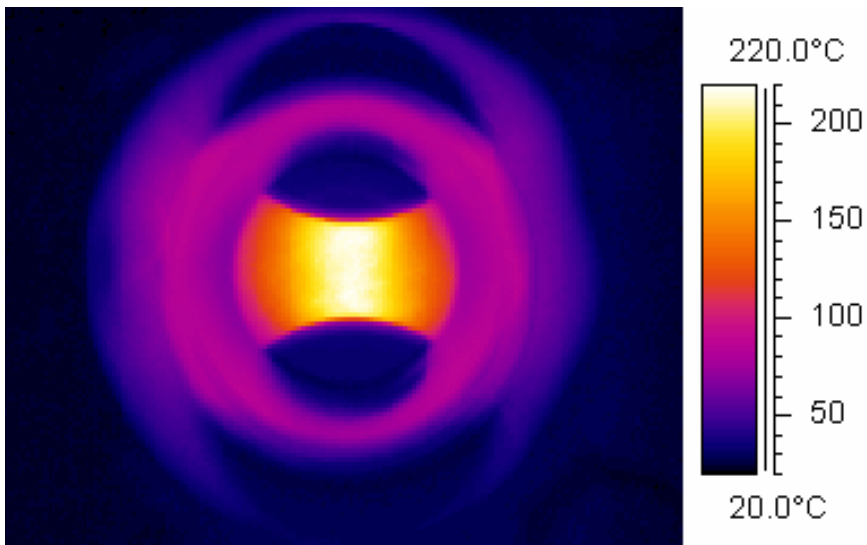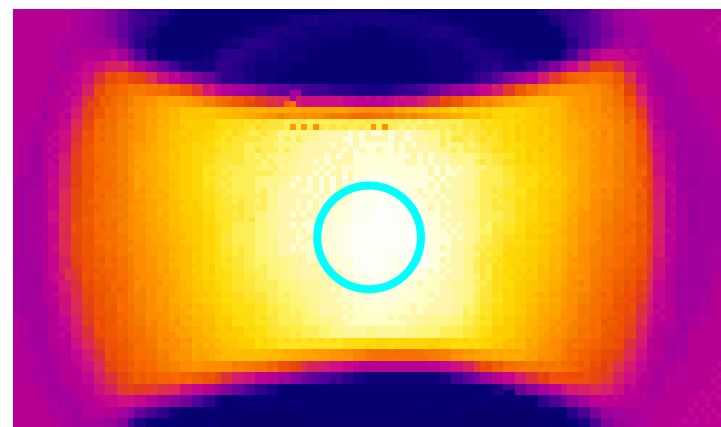# NIST Vapor Preconcentrator System

IR Camera

Regulated Dry Air

Host Filter with precise heating controls

Hygrometer

Hydrator

Collection Filter, cooled by TE on heat sink

RGA

IR Camera

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Solvent evaporating

IR Thermometry of Host Heating Filter

Flash Vaporization of Explosive

Thermal Focusing via Concave
Resistive Heating Grid
T = 220 °C with s = 3.0 °C in
target area

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Vapor Collector System
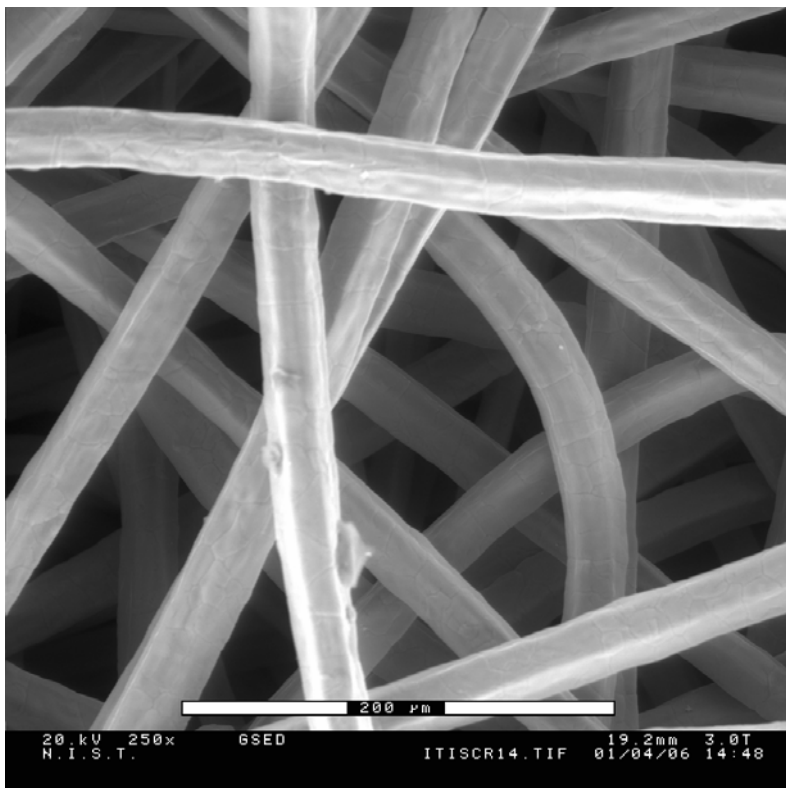
SS 316L Fiber Mesh
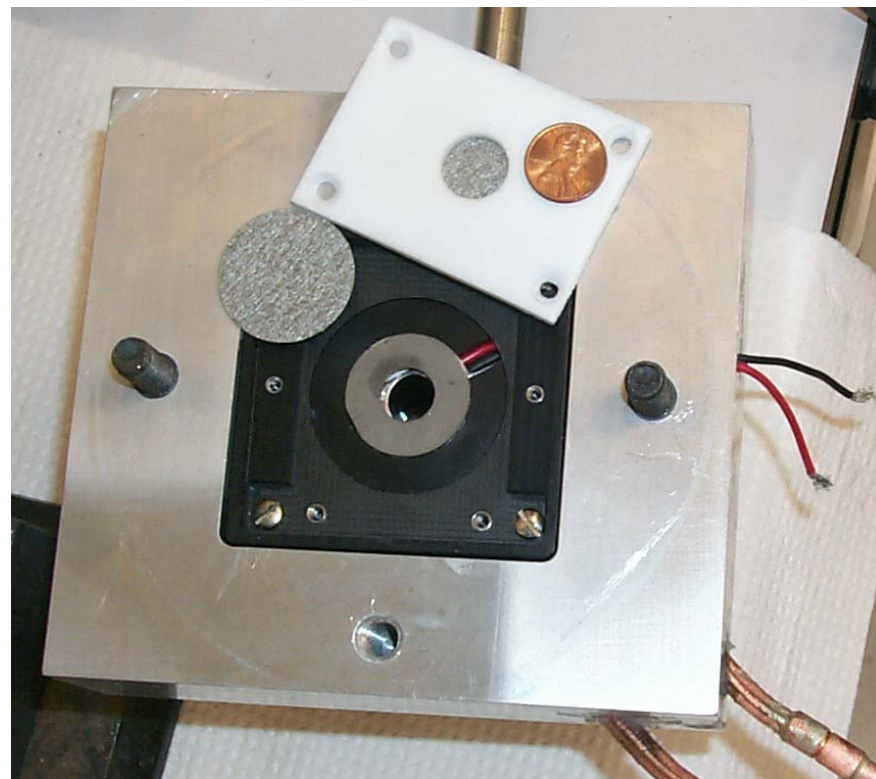
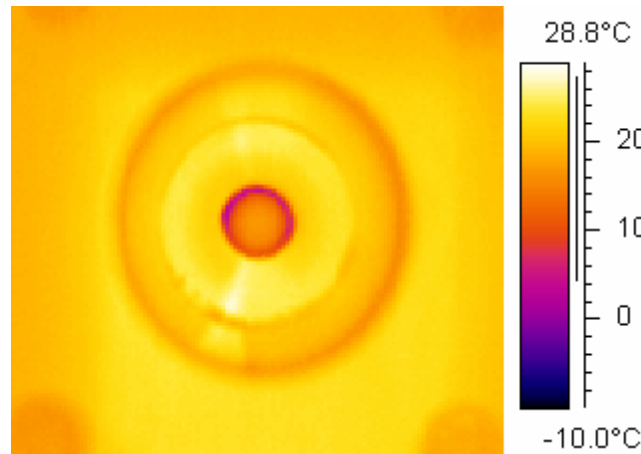Heat-Sink Block, Thermo-Electric Cooler, Collector, and PTFE Mask

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IR Thermometry of Filter During Vapor Collection

Transfer Tube at 180 ºC
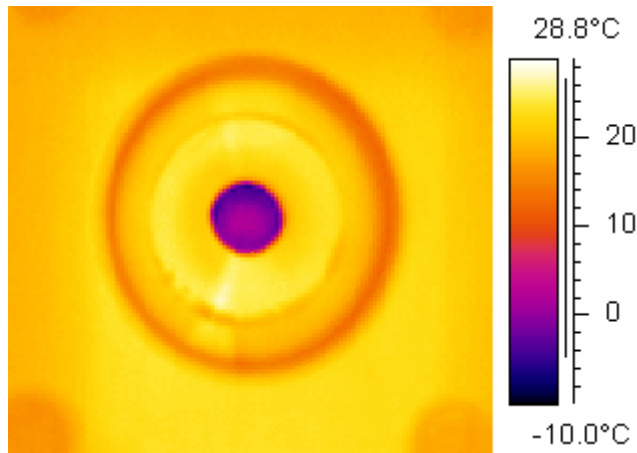
Air Flow = 0

Air Flow = 0.1 m/s

Air Flow = 1.3 m/s

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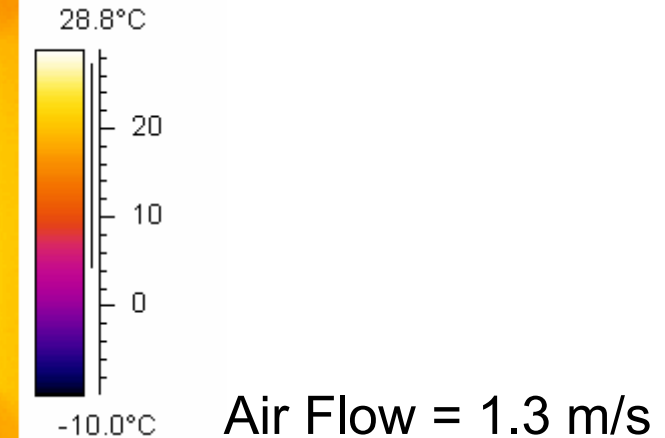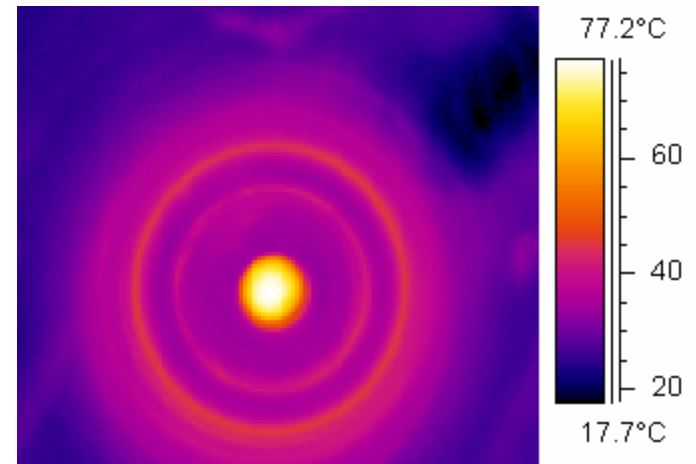
Test-of-Concept
2-level full factorial design

- Explosive (RDX, PETN, TNT)
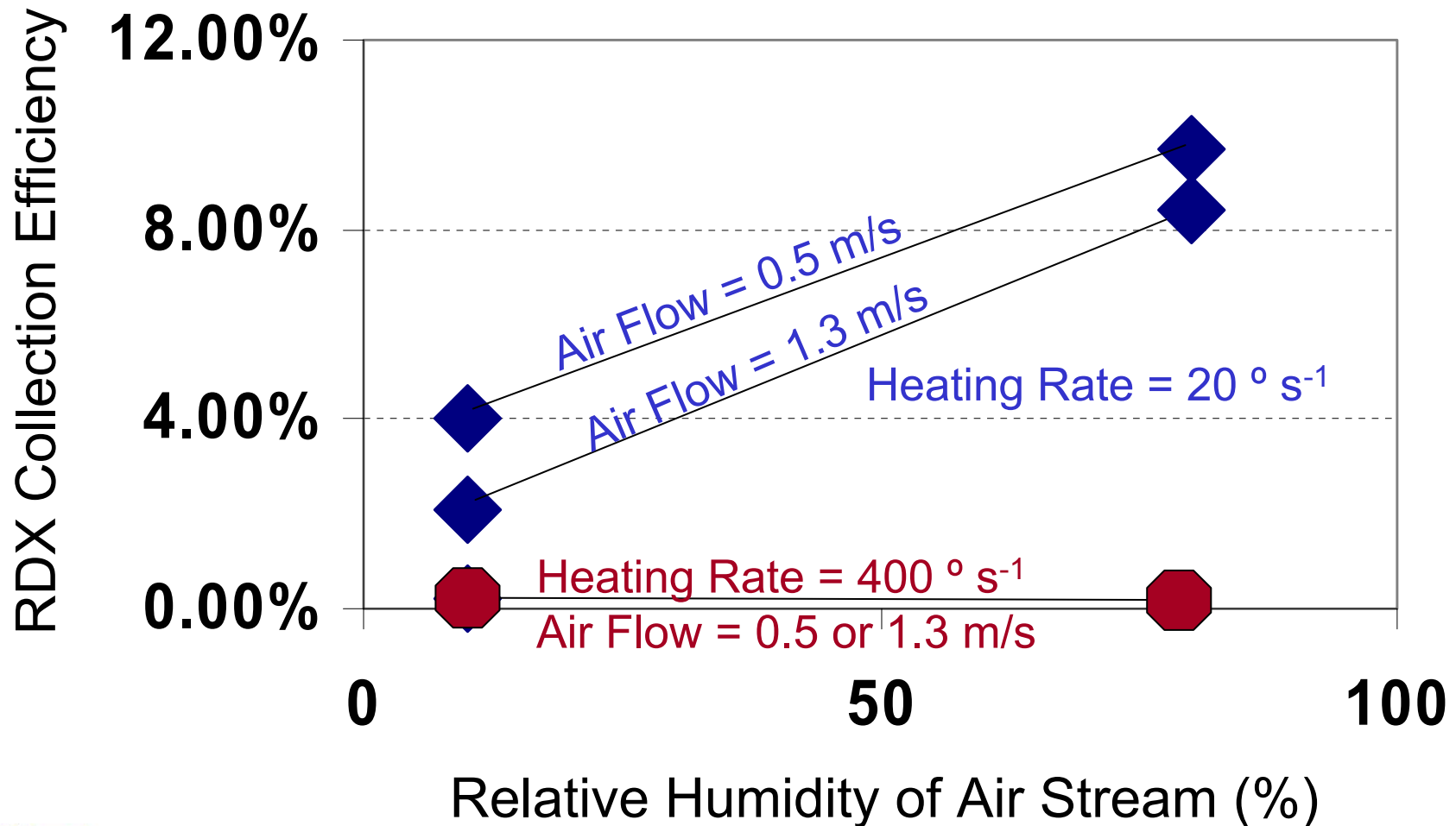- Heating rate of host grid (20 °/s, 400 °/s)
- Relative humidity of air flow (10 %, 80 %)
- Air flow through collector (1.3 m/s, 0.5 m/s)

Run	Heating Rate	Relative Humidity	Air Flow
1	+	+	+
2	+	–	+
3	+	+	–
4	+	–	–
5	–	+	+
6	–	–	+
7	–	+	–
8	–	–	–

Results for Vapor Collection of RDX

Bekipor AL3 (SS 316L) Filter

T_{max} (Host Filter) = 220 ºC, (Transfer Tube) = 200 ºC, (Collection) = 70 ºC

RDX Collection Efficiency vs. Relative Humidity of Air Stream (%)

- Air Flow = 0.5 m/s
- Air Flow = 1.3 m/s
- Heating Rate = 20 º s^{-1}
- Heating Rate = 400 º s^{-1}
- Air Flow = 0.5 or 1.3 m/s

Outline

Sampling Issues

Portals

What's the sample?

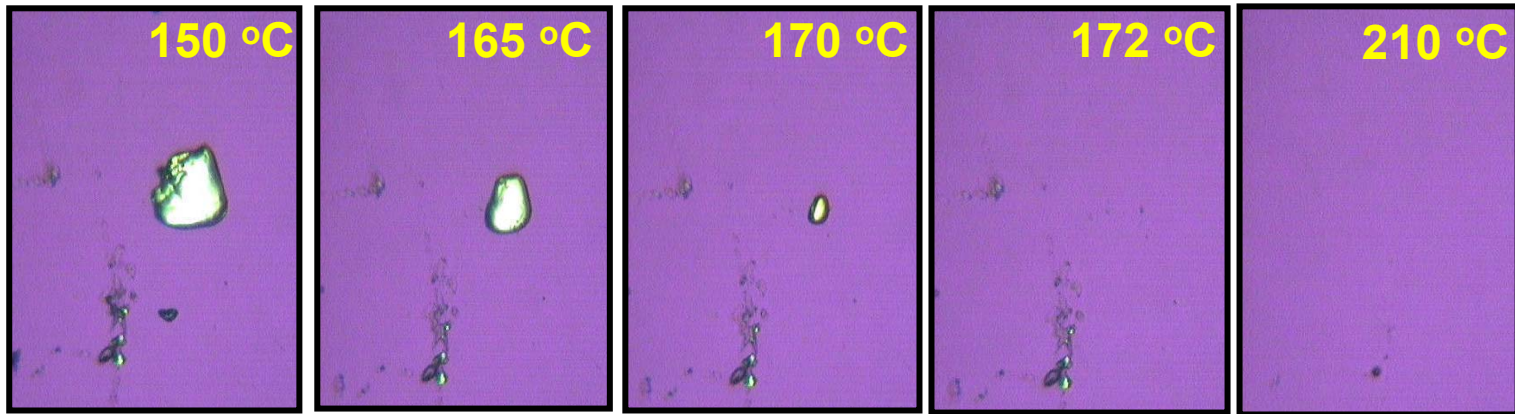Vapor Preconcentration

Standard test-bed

Thermal desorption
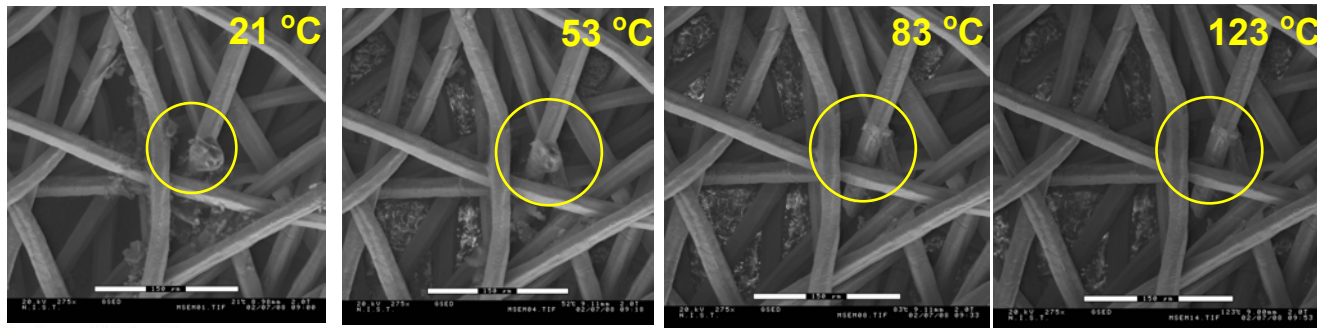
Standards

Ink-jet printing

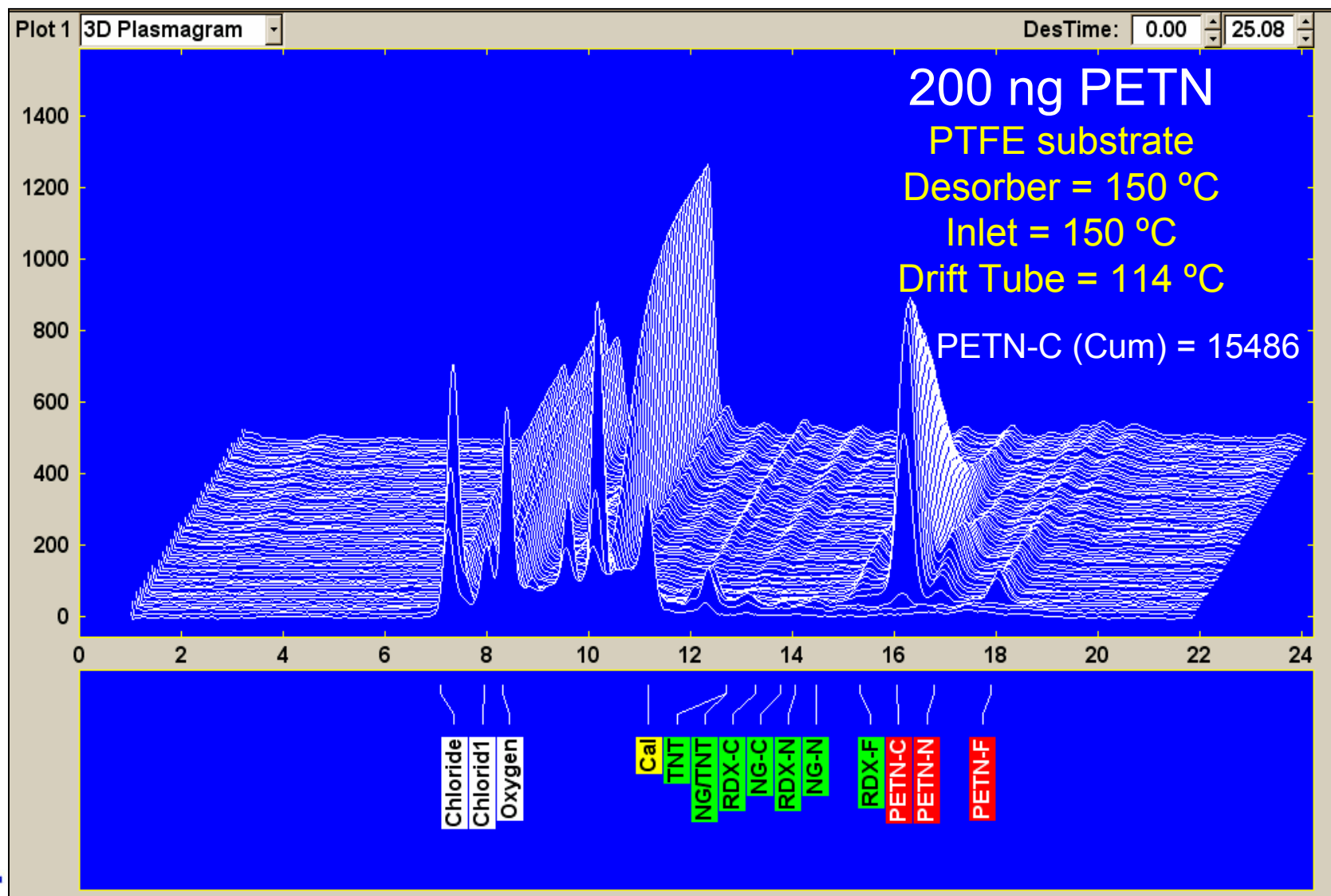# Individual Particle Morphological and Compositional Changes During Heating

Morphological Changes in C4 Particles as a Function of Temperature Using Temperature Programmed Optical Microscopy
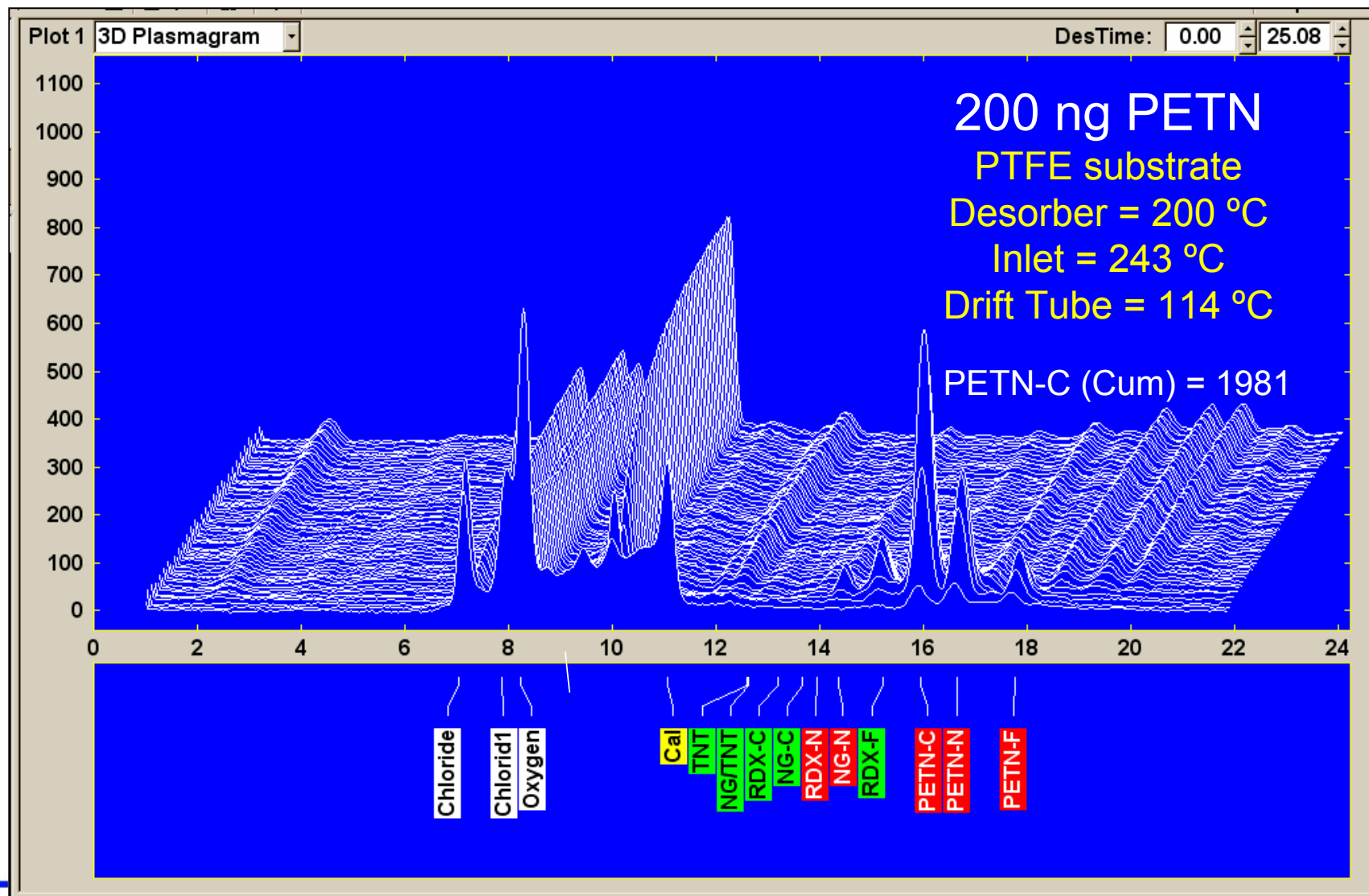

Morphological Changes in SEMTEX Particles as a Function of Temperature Using the Environmental Scanning Electron Microscope

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Desorption of PETN at 150 Degrees

Plot 1 | 3D Plasmagram | DesTime: 0.00 | 25.08

200 ng PETN
PTFE substrate
Desorber = 150 ºC
Inlet = 150 ºC
Drift Tube = 114 ºC

PETN-C (Cum) = 15486

Chloride   Chlorid1   Oxygen

Cal   TNT   NG/TNT   RDX-C   NG-C   RDX-N   NG-N   RDX-F   PETN-C   PETN-N   PETN-F

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Desorption of PETN at 200 Degrees

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Swipe IMS Measurement Factors

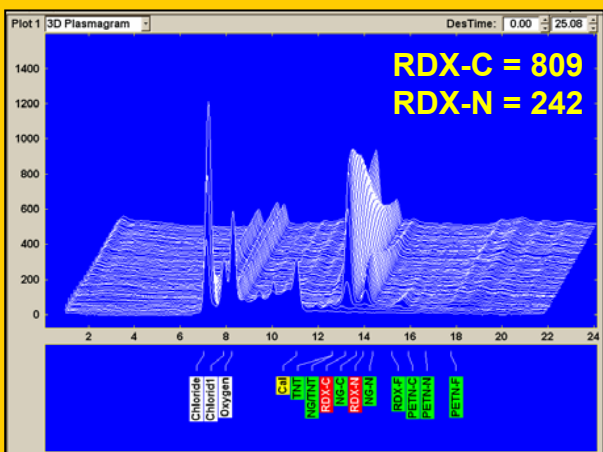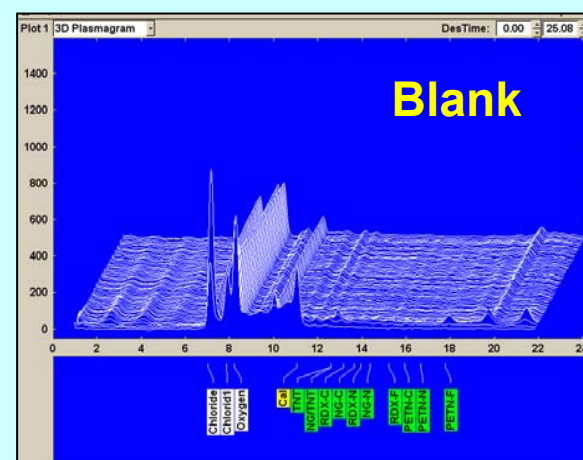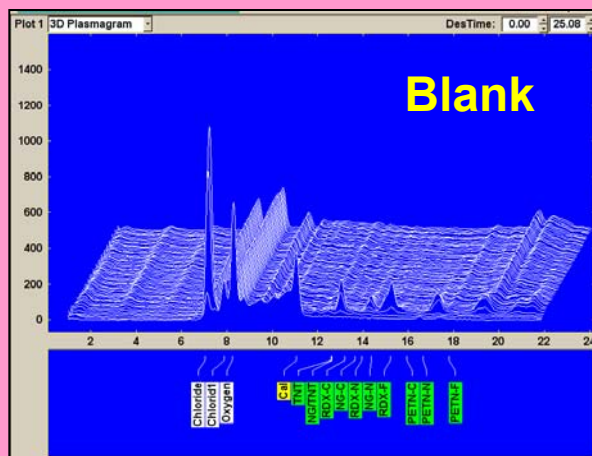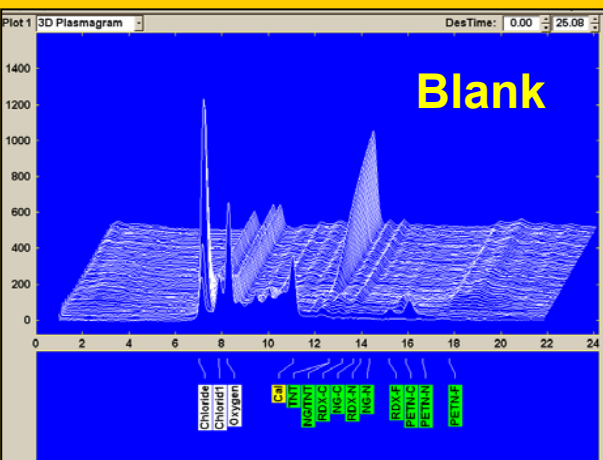4 ng RDX; Desorber = 200 ºC, Inlet = 243 ºC, Drift Tube = 114 ºC
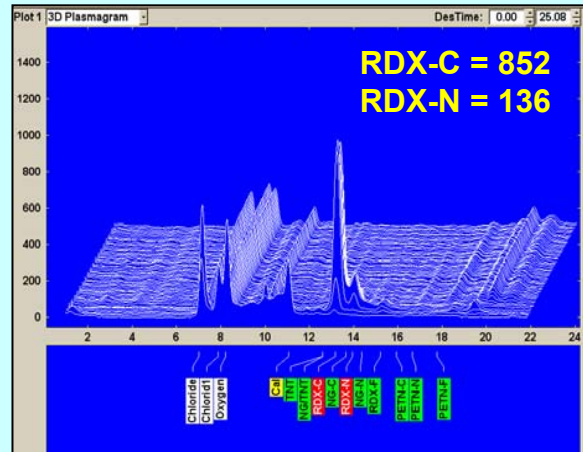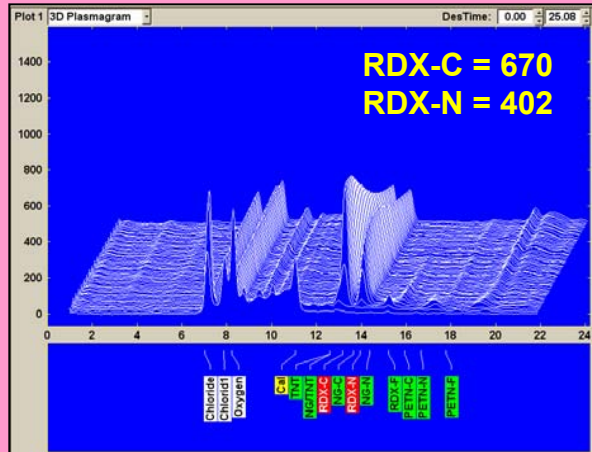
Outline

Sampling Issues

    Portals
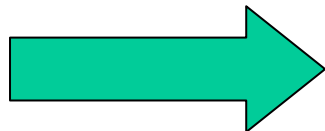
    What's the sample?

Vapor Preconcentration

    Standard test-bed
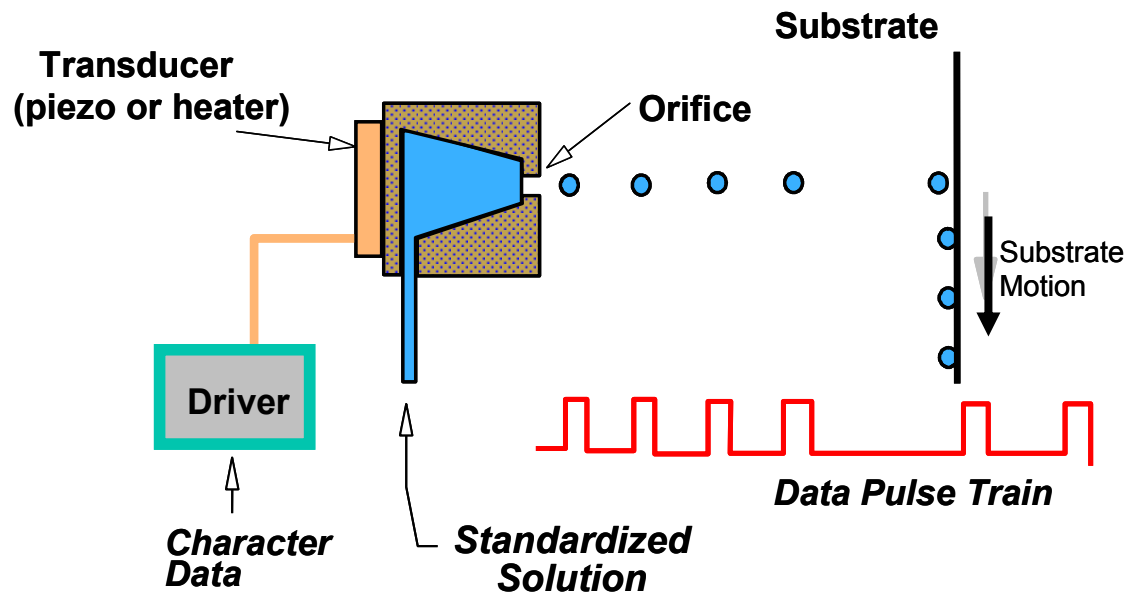
    Thermal desorption

Standards

    Ink-jet printing

Metrological Ink-jet Printing

New use of technology to produce millions of low-cost, realistic, and reliable standard materials for:

- **training and performance testing of first responders and screeners**
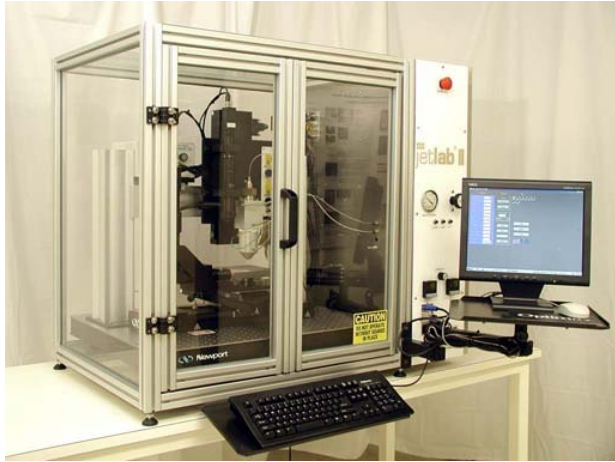- **frequent calibration and performance testing of detection equipment**

Assume 10,000 tabletop IMS instruments x 2 calibrations/day = 20,000 day
   >7 million reference materials consumed per year.

Substrate

**Transducer
(piezo or heater)**

Orifice

Substrate
Motion

Data Pulse Train

Driver

*Character
Data*

*Standardized
Solution*

**Advantages**

- Precise delivery of known number of droplets with known mass and concentration
- Large dynamic range in jetting frequency and droplet size
- Wide choice in jetted compounds (explosives, narcotics, matrix) and substrate target

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NIST Ink-jet Printing System for Development of Trace Explosive Reference Materials

JetLab II Trace Explosive Printer System

TNT printed directly on test strips for use in IMS calibration

IMS intensity (arb. units) vs ng TNT
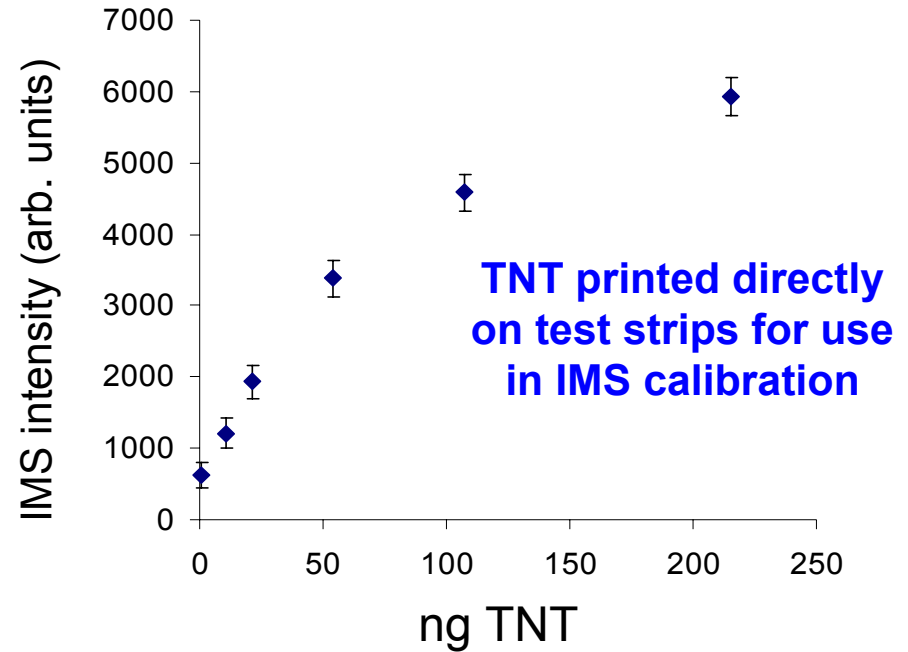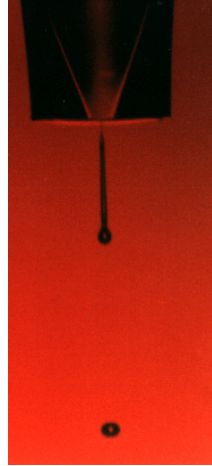
Fluorescein + TNT

← 2.5 cm →

Fluorescein + RDX

← 2.5 cm →

ISIMS 2004 Gatlinburg
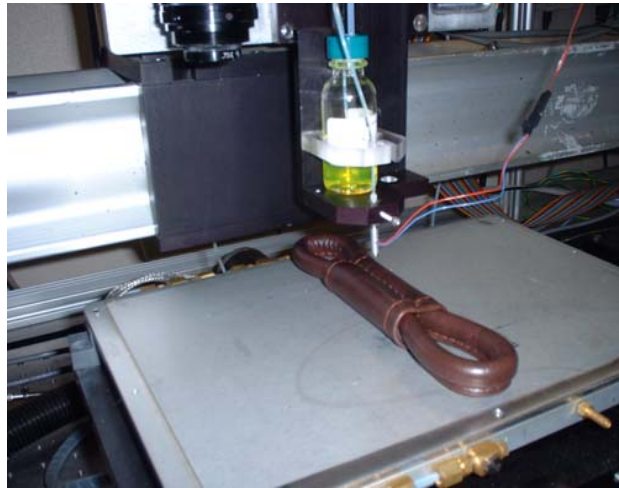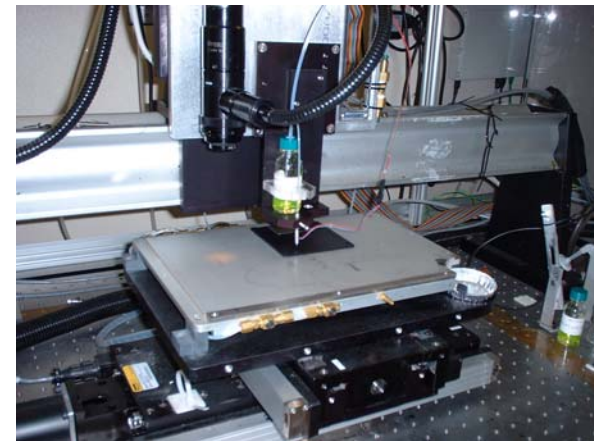
Ink-jet Printing on Novel Test Surfaces
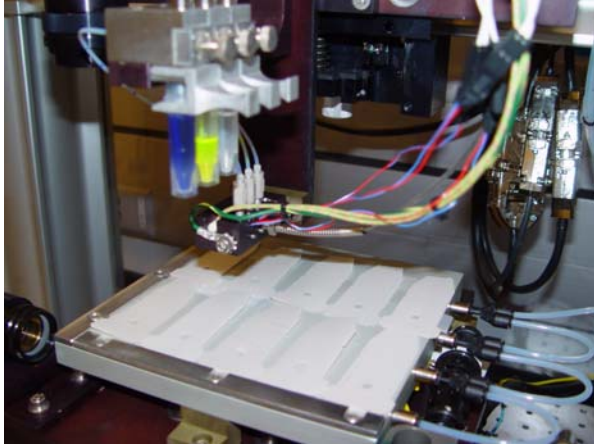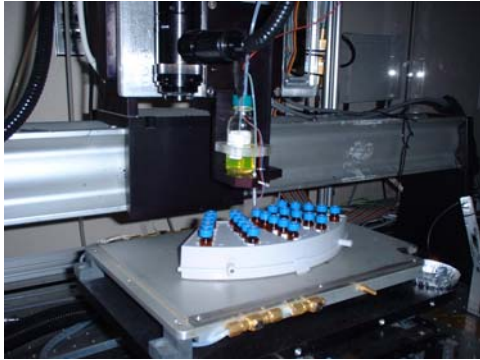

IMS Swipes

Luggage handles

Floppy Disks

Methods for Determination of Explosive Mass Deposited on Substrate

- Known concentrations in reservoirs, known frequency, known orifice size

- Isotope dilution GC/MS analysis of mass of explosive in many droplets

- Volume measurement of single droplet from optical monitoring of formation using strobe illumination

- Weighing of multiple drops

- Measurement of drop size on non-wetting surface

- Visual observation of droplet arrays using fluorescent and optical dyes

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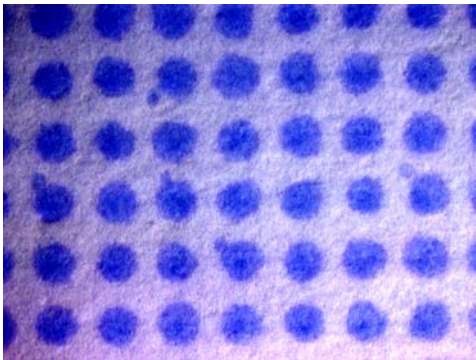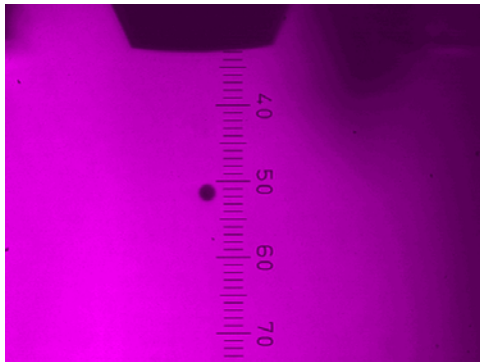
Summary

- NIST trace explosive detection program
  - Focused on particle sampling and metrology issues
  - Microanalytical approaches
- Air-jets preferentially remove:
  - larger particles from surfaces
  - more particles from cloth (porous) surfaces
- Portal collection efficiency optimized with larger particles
  - model validated with sticky particles or sticky surfaces

Summary, cont.

- Nature of explosive particles
  - most mass distributed in larger particles (> 10 μm)
  - size & mass distributions similar in residues
  - IMS response from single large particle >> LD
- Preconcentrator test-bed demonstrated
  - Factors: heating rate ~ explosive type > humidity > air flow
- Ink-jet printing of explosives demonstrated
  - Precision, reproducibility, versatility
  - Continuing characterizations, calibrations, comparisons, evaluations of uncertainty

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Technology Administration, U.S. Department of Commerce

A Metrological Vision of the Future

Intelligent and adaptable detection systems

- *Smart portals*
    - Optical pattern recognition systems (human features, attire, fabric)
    - Adaptable air jets and particle collection
    - Programmed preconcentrator
    - Chemical pattern recognition systems
    - Interactive international database

Cheap, instantly available, and reliable calibrations

- *Modified ink-jet technology*
    - Particle detection
    - Vapor detection