

# *Trace Explosive Detection Research at NIST*

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# National Institute of Standards and Technology

Founded in 1901, NIST is a non-regulatory federal agency within the U.S. Commerce Department's Technology Administration. NIST's mission is to develop and promote measurements, standards, and technology to enhance productivity, facilitate trade, and improve the quality of life.



**NIST is the Nation's Reference Laboratory for chemical metrology research, standards, and data.**

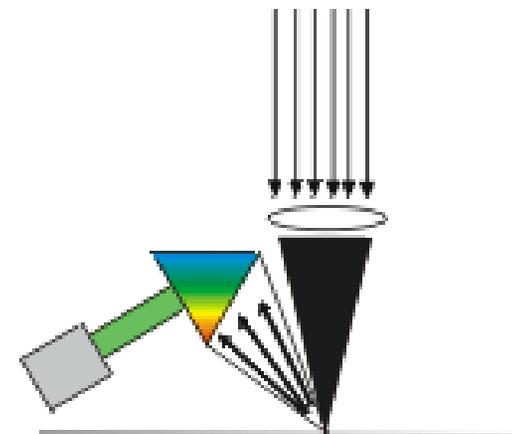
NIST has an operating budget of about \$839 million and operates in two locations: Gaithersburg, MD and Boulder, CO. NIST employs about 3,000 scientists, engineers, technicians, and support staff. About 1,600 guest researchers complement the staff.

# Surface and Microanalysis Science Division

Analytical Chemistry on the nanometer-micrometer spatial scale.

Primary Radiation: ions, electrons, photons, x-rays.

Secondary radiation: ions, photons, electrons, x-rays. Identifies chemical composition of sample (elemental, isotopic, molecular). Intensity tells us how much is present.



Basic Technique Development, Standards and Applications Using: SEM, Environmental SEM, TEM x 3, Electron Probes x 2, Auger x2, XPS x2, XRD x2, Micro-XRD, SIMS x 3, FIB, Laser Desorption MS, IRMS, autoradiography, SPM, NSOM, IR, visible, fluorescence and Raman Microscopes, GC, GC-MS, GC-IMS, particle counting and generation.

Applications: Semiconductors and thin film analysis, biological microanalysis, biomaterials, organic thin films, isotopic measurements, atmospheric chemistry, materials analysis, asbestos, explosives.

# NIST Program - Trace Detection Research

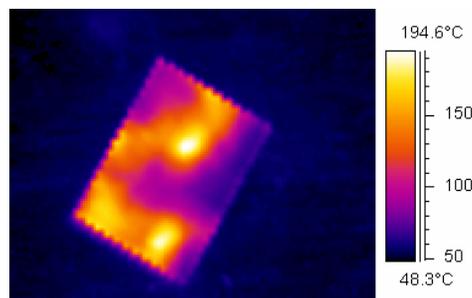


Study the performance characteristics of handheld, tabletop and portal-based explosive detection instruments. Develop a measurement infrastructure at NIST to optimize, calibrate and standardize these instruments. Support both new technology development and effective utilization by security screeners and 1<sup>st</sup> responders.

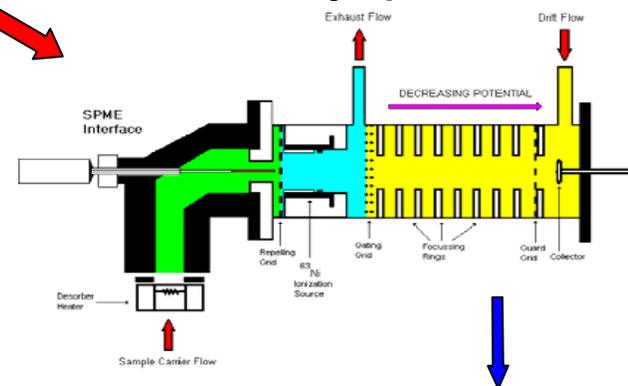
### Swipe Detection



### Heat Swipe



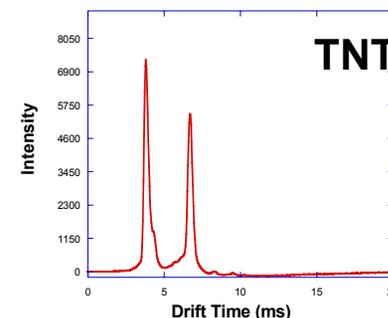
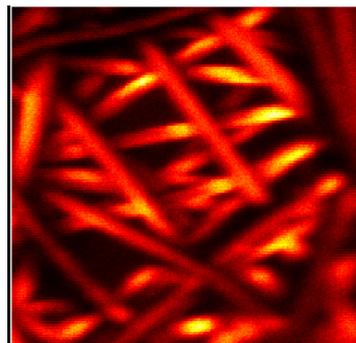
### Ion Mobility Spectrometer



### Portal Detection

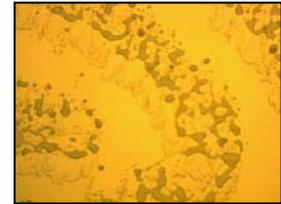


### Heat Collection Grid

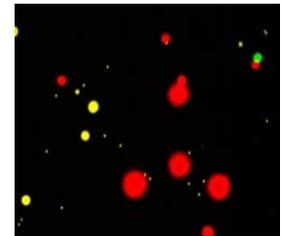


# Research being used to support “Development of A Measurement Infrastructure for Trace Explosive Detection at NIST”

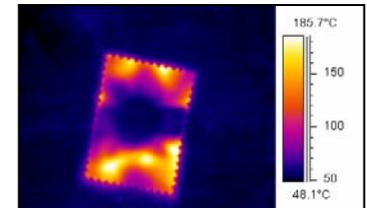
- What is being Sampled?
- Particle Removal/Collection
- Optimization of Operational Parameters
- Ion Mobility Spectrometry Fundamentals
- Standards for Trace Explosives Detection



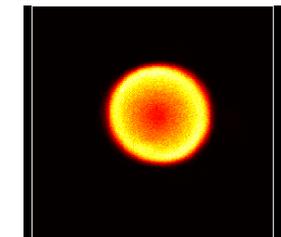
Optical micrograph of fingerprint



Fluorescence micrograph of spheres



IR Camera Image of Swipe

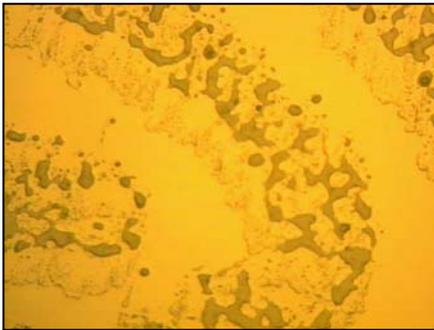


SIMS Image of TNT on Si

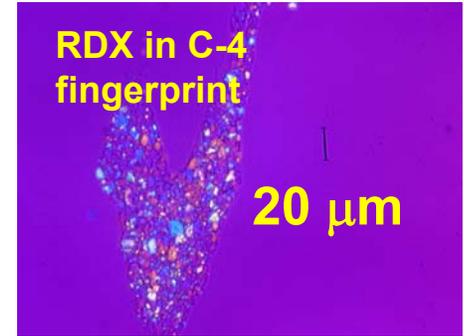
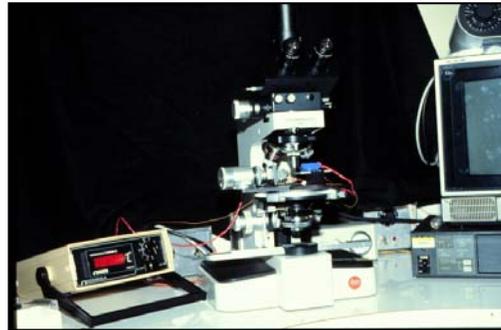
# What is the Sample?

Characterization of the nature and particle size distribution of trace explosive residues is critical for improving the design of trace detection equipment.

## Polarized Optical Microscopy



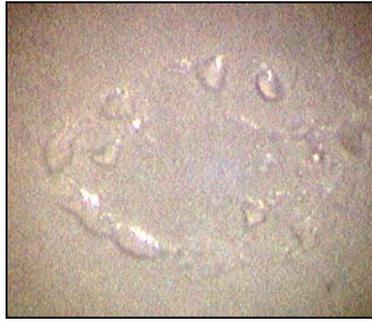
1 mm



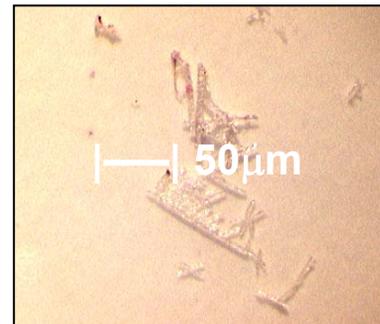
Semtex



Detasheet



C4 on Byteck



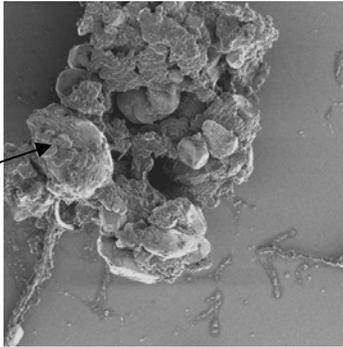
swiped

Luggage Handle

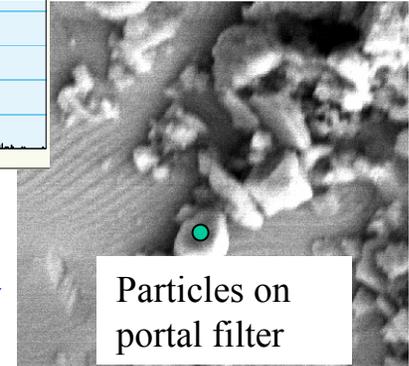
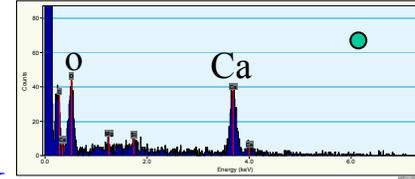


# Explosive particle sizing using FEG-SEM

RDX in C-4



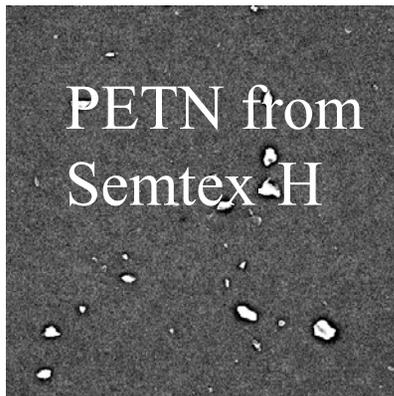
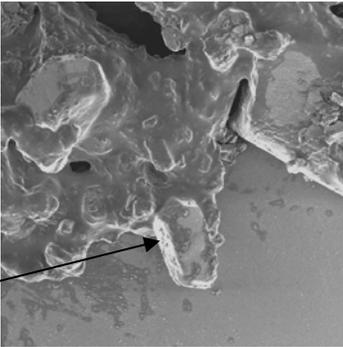
FEG-SEM



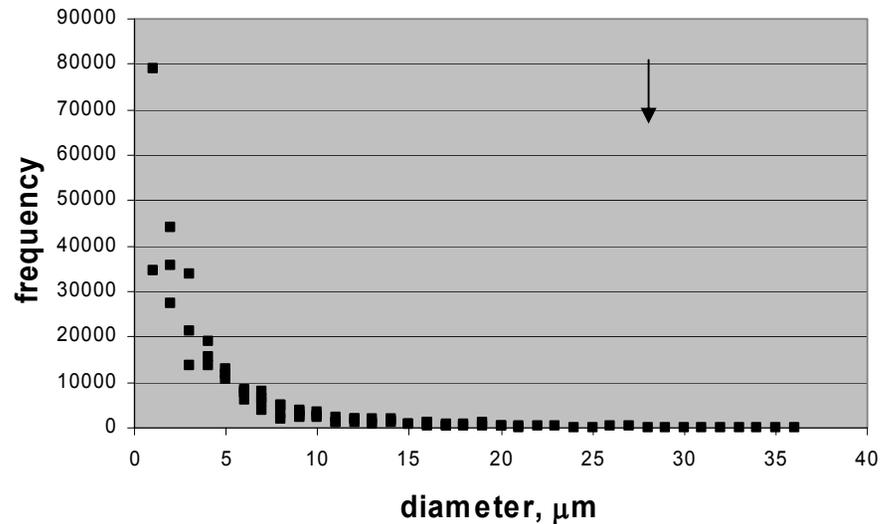
Particles on portal filter

Image courtesy of Hitachi at [www.hhtc.ca](http://www.hhtc.ca)

PETN in Semtex-H

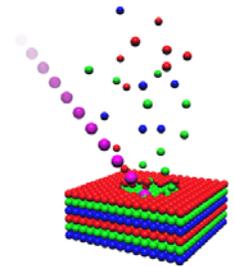


Particle size, PETN

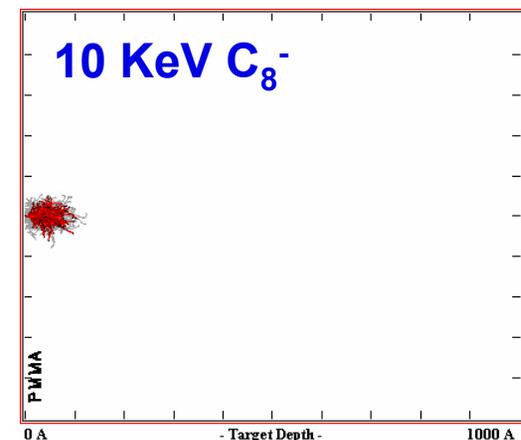
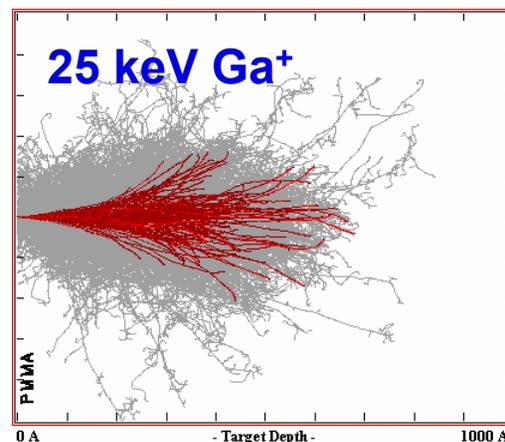
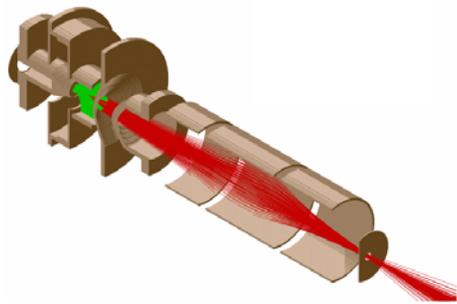
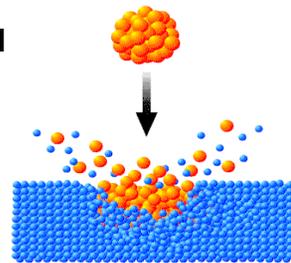


# Explosive Particle Analysis using Cluster SIMS

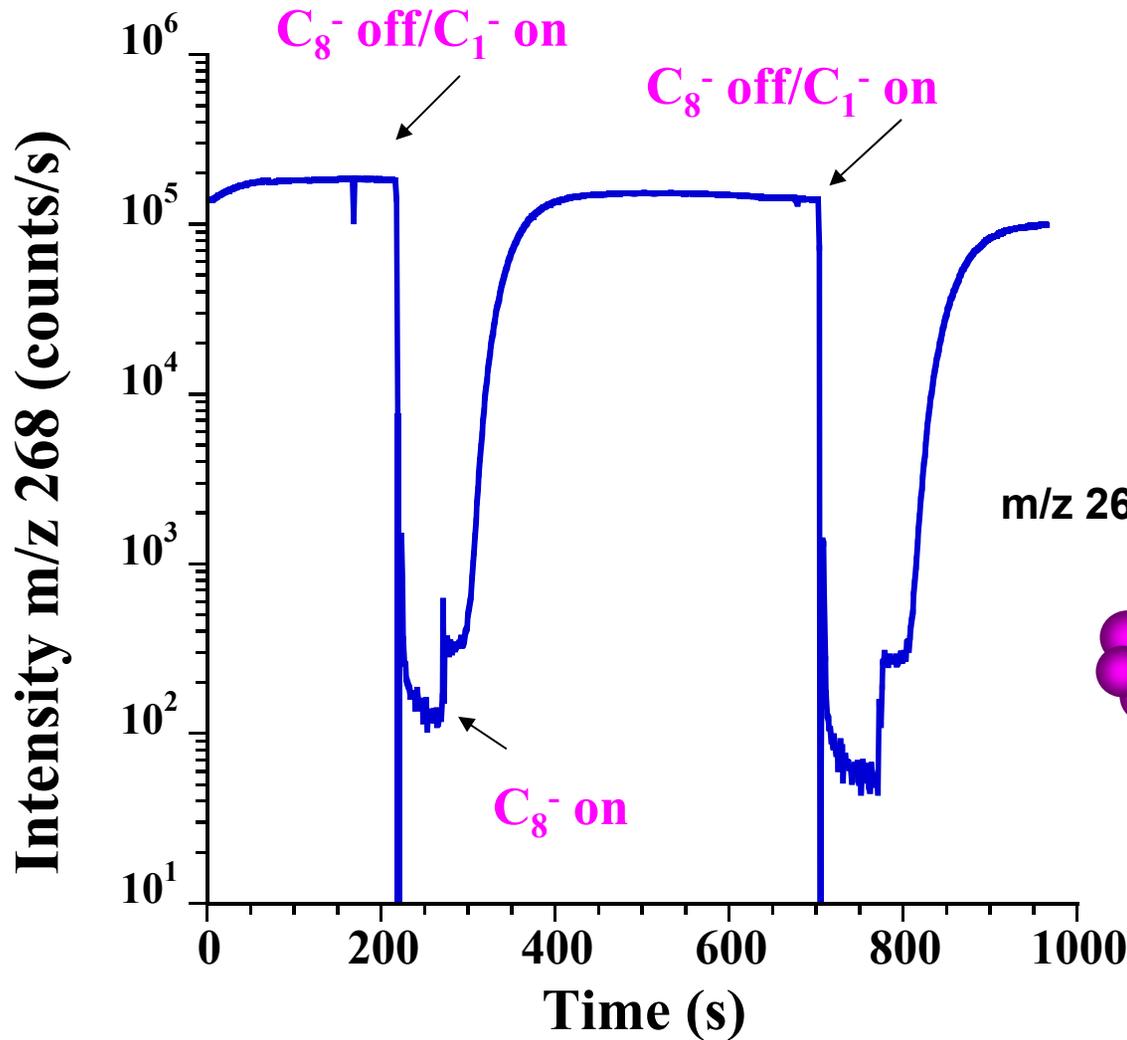
Primary Ion Bombardment on commercial SIMS instruments with polyatomic or cluster primary ion beam. ( $\text{SF}_5^+$ ,  $\text{C}_8^-$ ,  $\text{CsC}_6^-$ ,  $\text{CF}_3^+$ ,  $\text{Si}_5^-$ ,  $\text{Al}_7^-$ ,  $\text{C}_{60}^+$ ) on NIST magnetic sector and TOF-SIMS instruments.



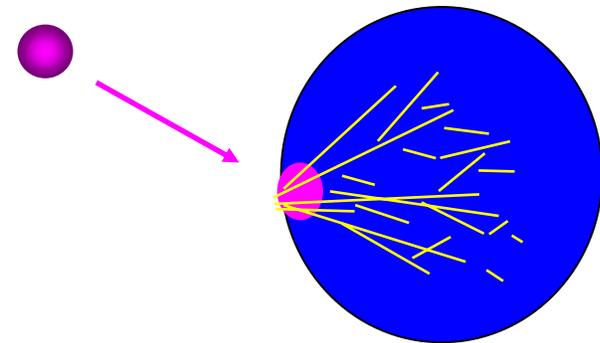
- Low penetration depth - Energy of Component Atom =  $E_o * M_c / M_{\text{total}}$
- Higher sputter/secondary ion yields.
- Reduction in accumulation of beam-induced damage for some organic molecules.



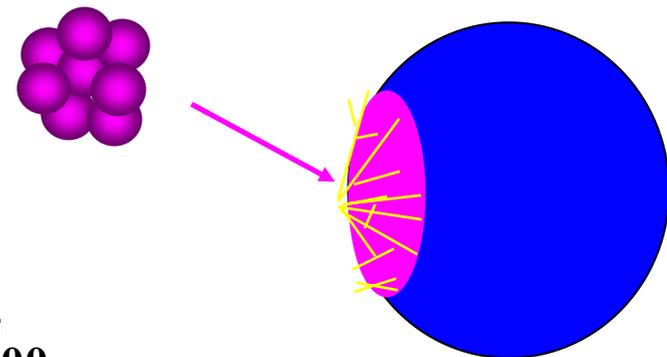
# C<sub>8</sub><sup>-</sup> Depth Profile of RDX Particles



## Explosive Particle



m/z 268 Monoatomic Ion Beam – low signals with extensive subsurface damage of the sample by probing ion beam.

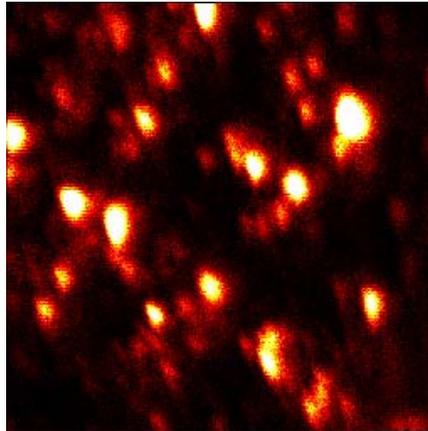
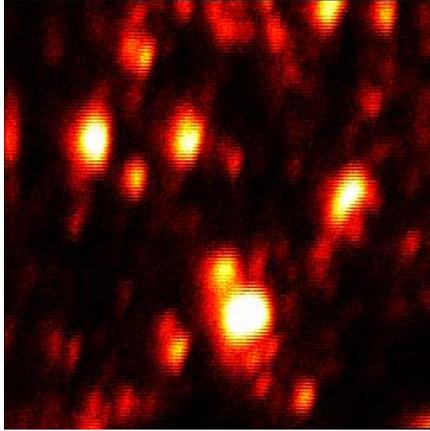


Cluster Ion Beam- high signals with minimal subsurface damage by probing cluster beam

$C_1 = 5e14 \text{ ions/cm}^2$   
 $C_8 = 1.3e14 \text{ cluster ions/cm}^2$   
 $(7.8e14 \text{ atoms/cm}^2)$

# Microbeam SIMS Imaging of PETN and RDX Particles on Silicon (Static SIMS, C<sub>8</sub><sup>-</sup>)

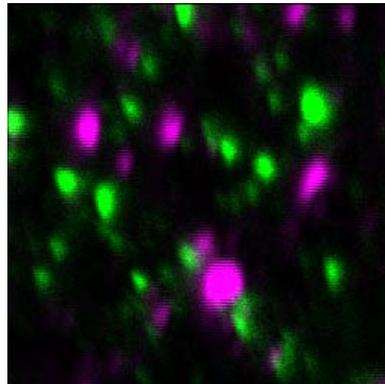
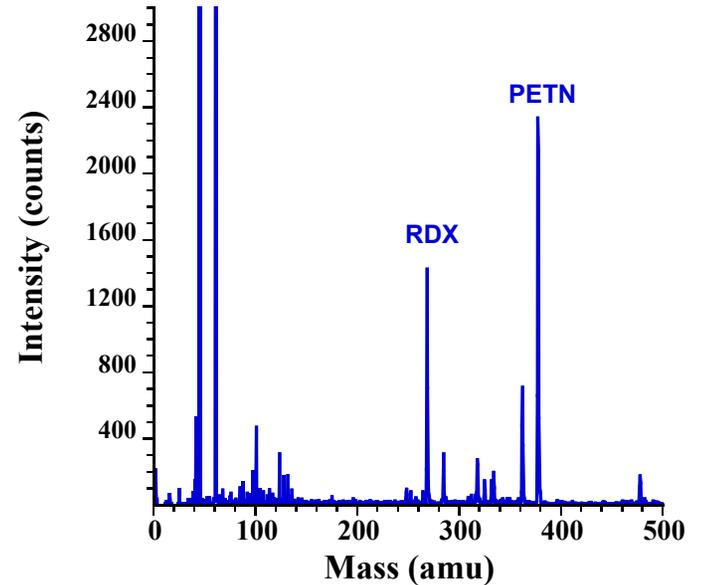
400 μm



PETN (0-440 cts/pixel)

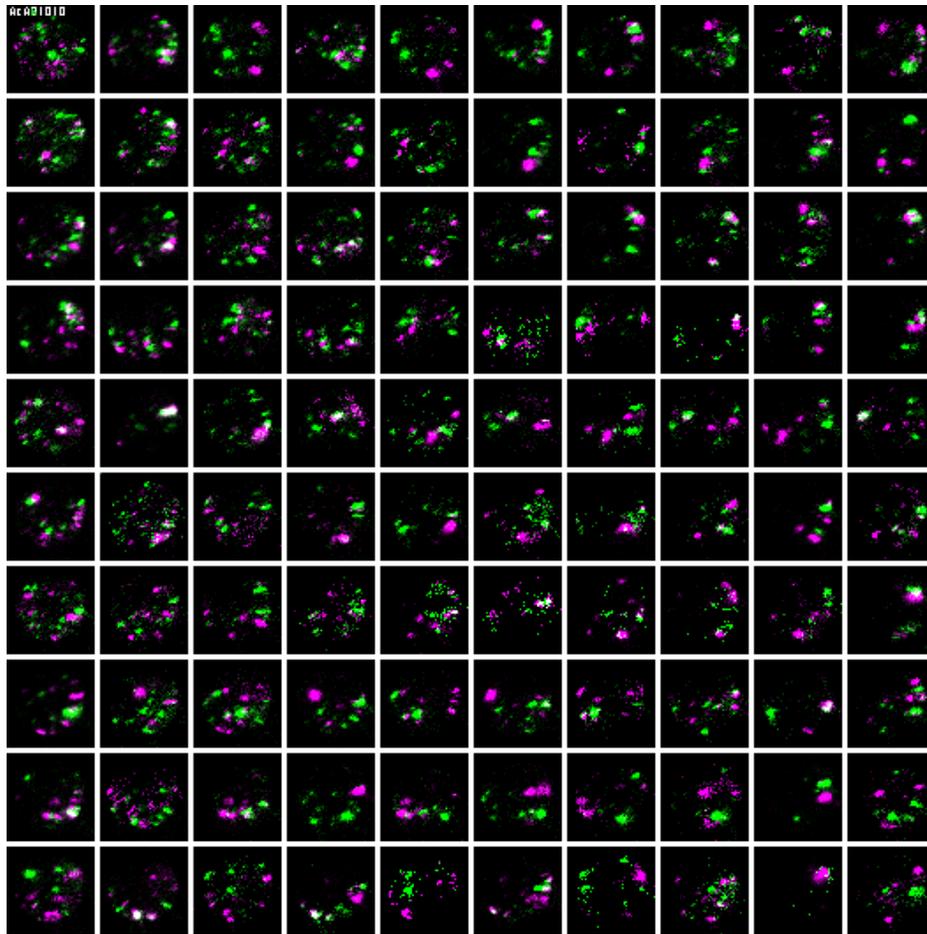
RDX (0-270 cts/pixel)

### Mass Spectrum After Image Acquisition



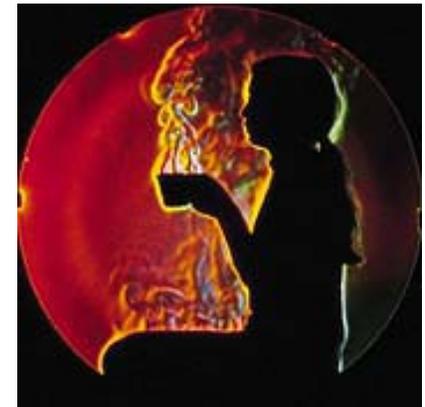
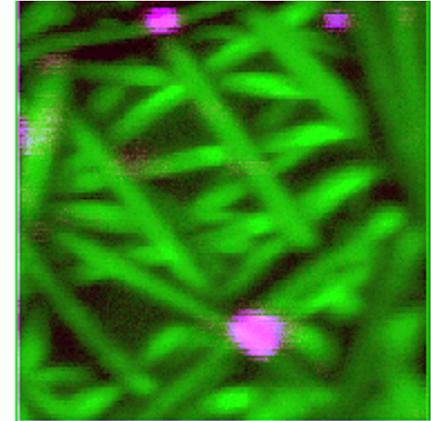
30 s image acquisition

# PETN and RDX Microbeam Particle Search (C<sub>8</sub><sup>-</sup>)



RDX  
PETN

Portal Grid + PETN

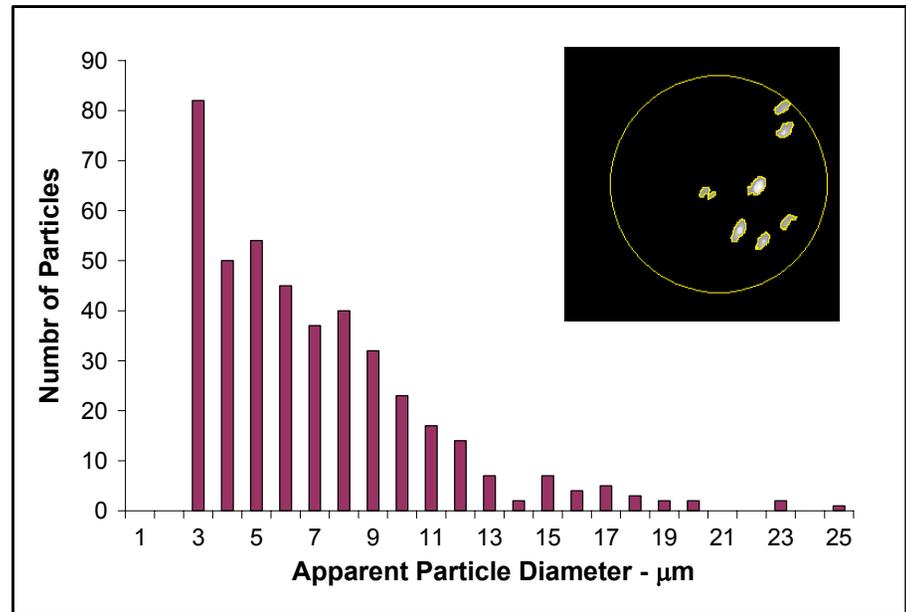
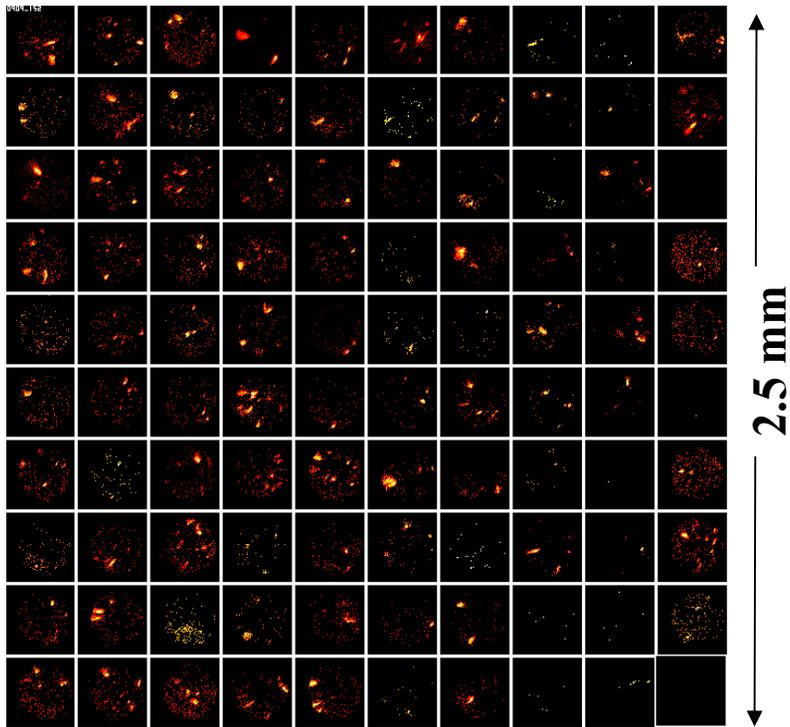
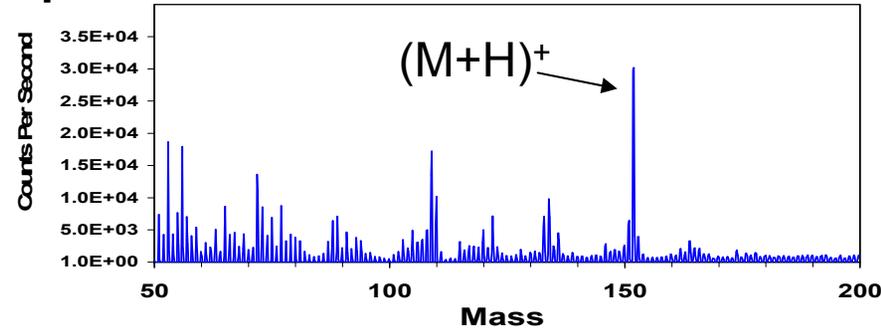


Determine sample characteristics  
Determine particle transport characteristics of human thermal plume

~880 PETN particles detected, 2 hours analysis time

# 1. Cluster SIMS Analysis of Fingerprint (SF<sub>5</sub><sup>+</sup>)

- 10 x 10 particle search in microscope mode.
- Acetaminophen particles in human fingerprint.
- Image <sup>29</sup>Si for 10 sec, (M+H)<sup>+</sup> for 20 sec
- Dose 8.5x10<sup>13</sup> ions/cm<sup>2</sup>
- Total time 63 minutes (25% overhead).

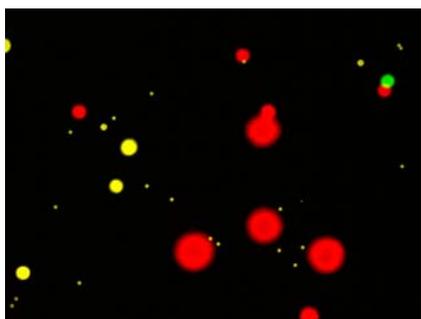
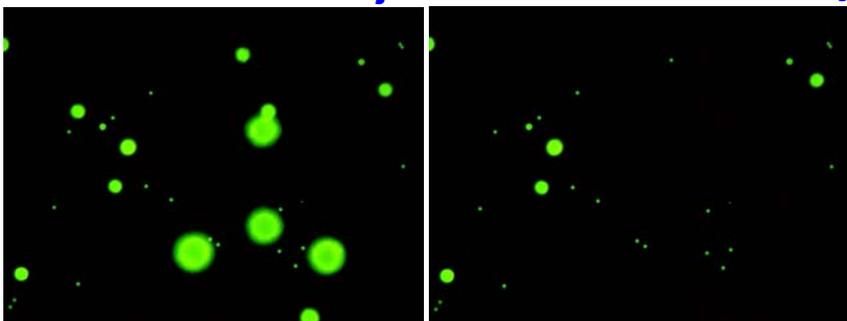


← 2.5 mm →

451 particles identified

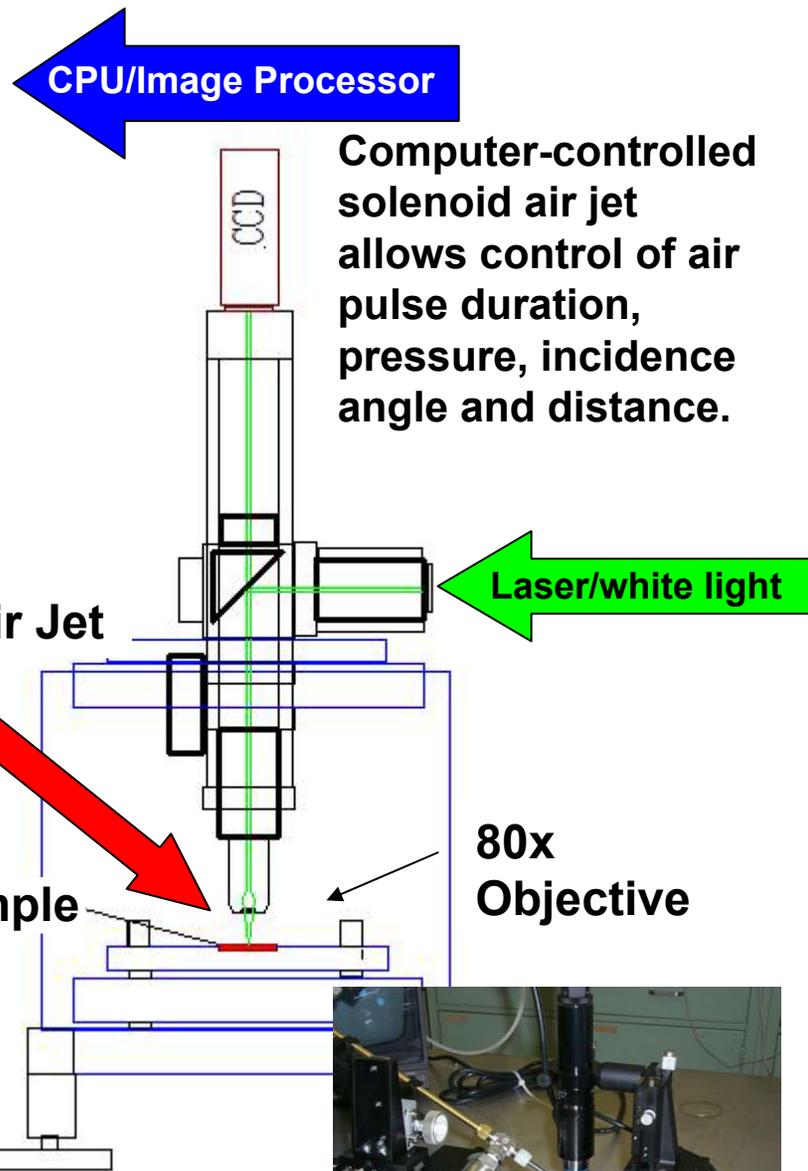
# Particle Removal From Surfaces

Test surface before air jet      Test surface after air jet



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

Polystyrene microspheres of various sizes (1  $\mu\text{m}$ , 6  $\mu\text{m}$ , 10  $\mu\text{m}$ , 45  $\mu\text{m}$ ) deposited onto test surface. Larger spheres are preferentially removed over small spheres.

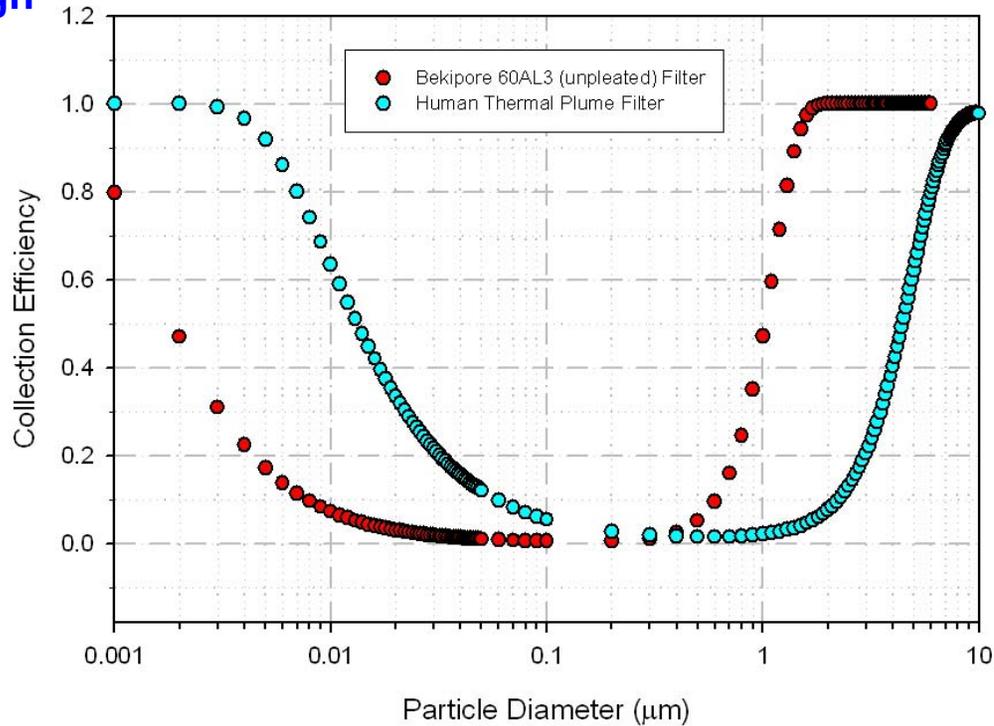
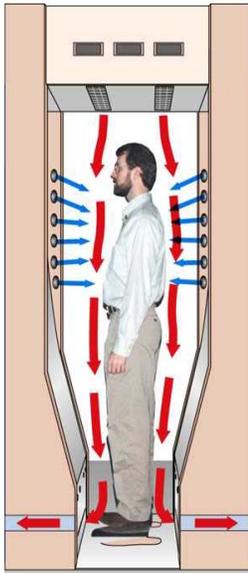


Computer-controlled solenoid air jet allows control of air pulse duration, pressure, incidence angle and distance.

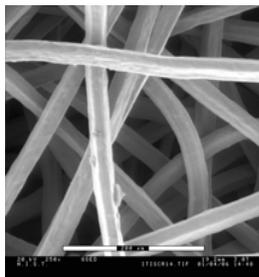
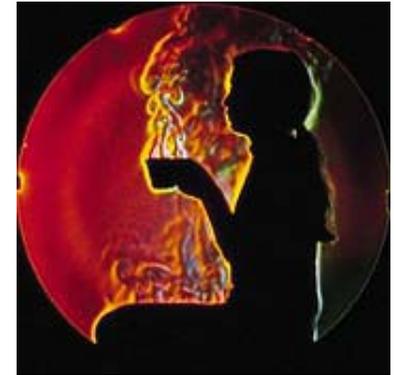


# Particle Collection Efficiency

## Air shower Design



## Human Thermal Plume

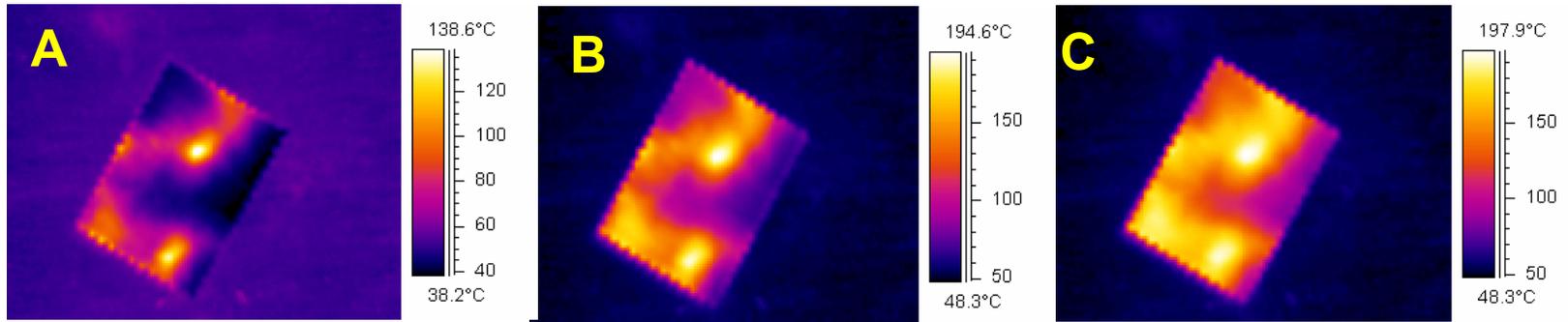


Metal filter

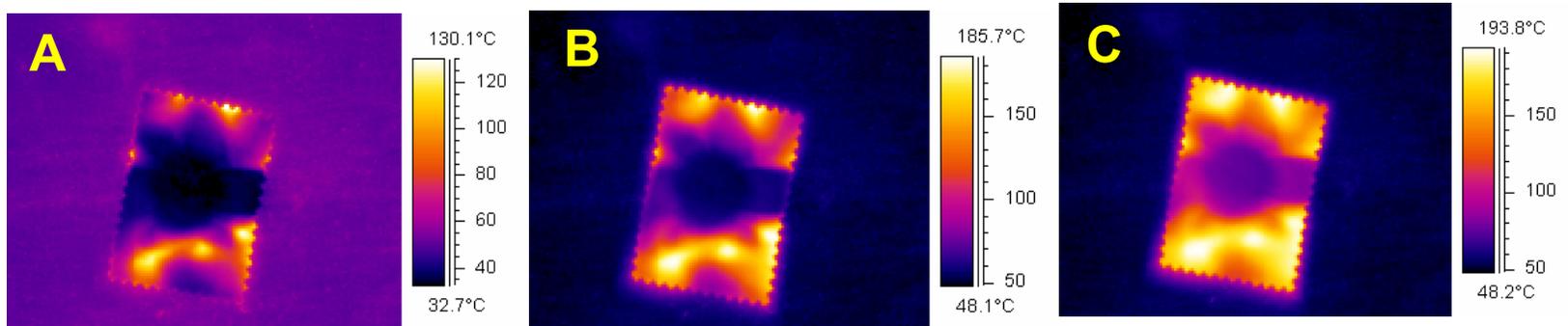
- Develop capability to characterize particle collection efficiency of portal filters.
- High air volume particle collection, what are special issues to be considered?
- Model filter collection efficiency and optimize filter design.
- Explore novel ways to improve collection efficiency of filters.

# IR Thermometry for Spatially and Temporally Resolved Temperature Mapping

Swipe placed on aluminum plate heated to 200 °C. Thermograms acquired as a function of time (0-10 seconds).

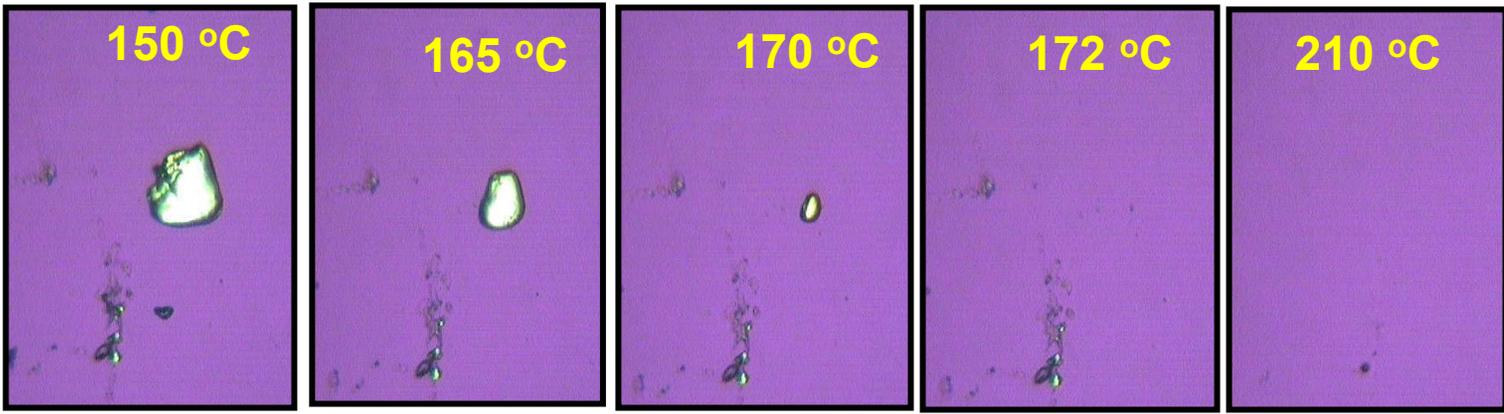


Same swipe after using sampling wand. Note variable heat transfer to center of swipe (0-10 seconds).

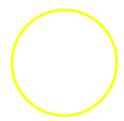
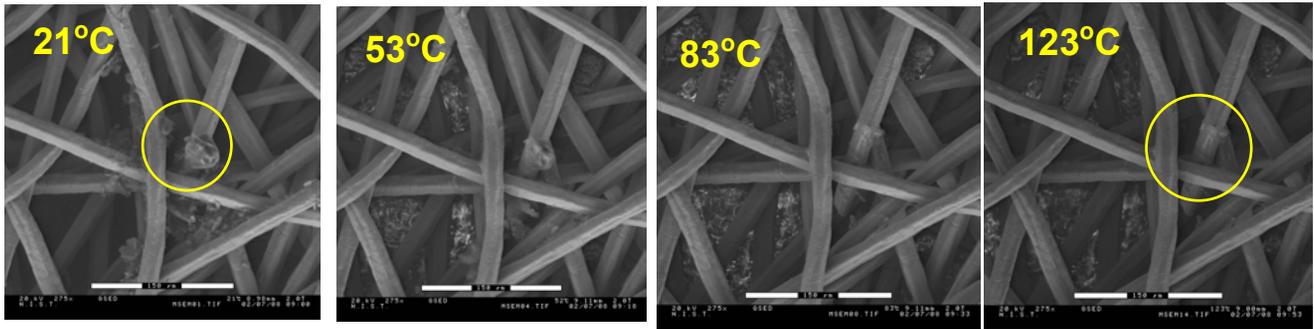


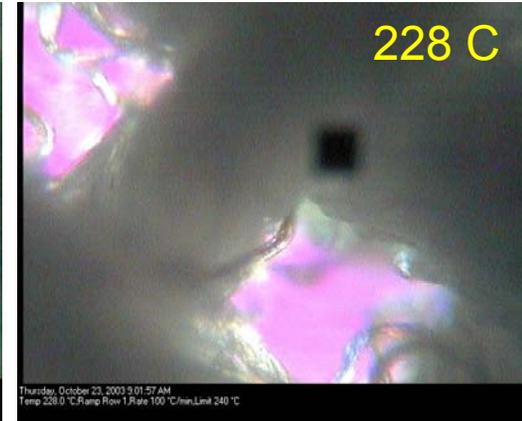
Note: Displayed temperature scale not NIST calibrated. Data acquired using camera manufacturer's standard settings. Infrared camera calibration (NIST traceability) and determination of the emissivity of swipe materials is being conducted in a collaboration with the NIST Physics Optical Thermometry Group.

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



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

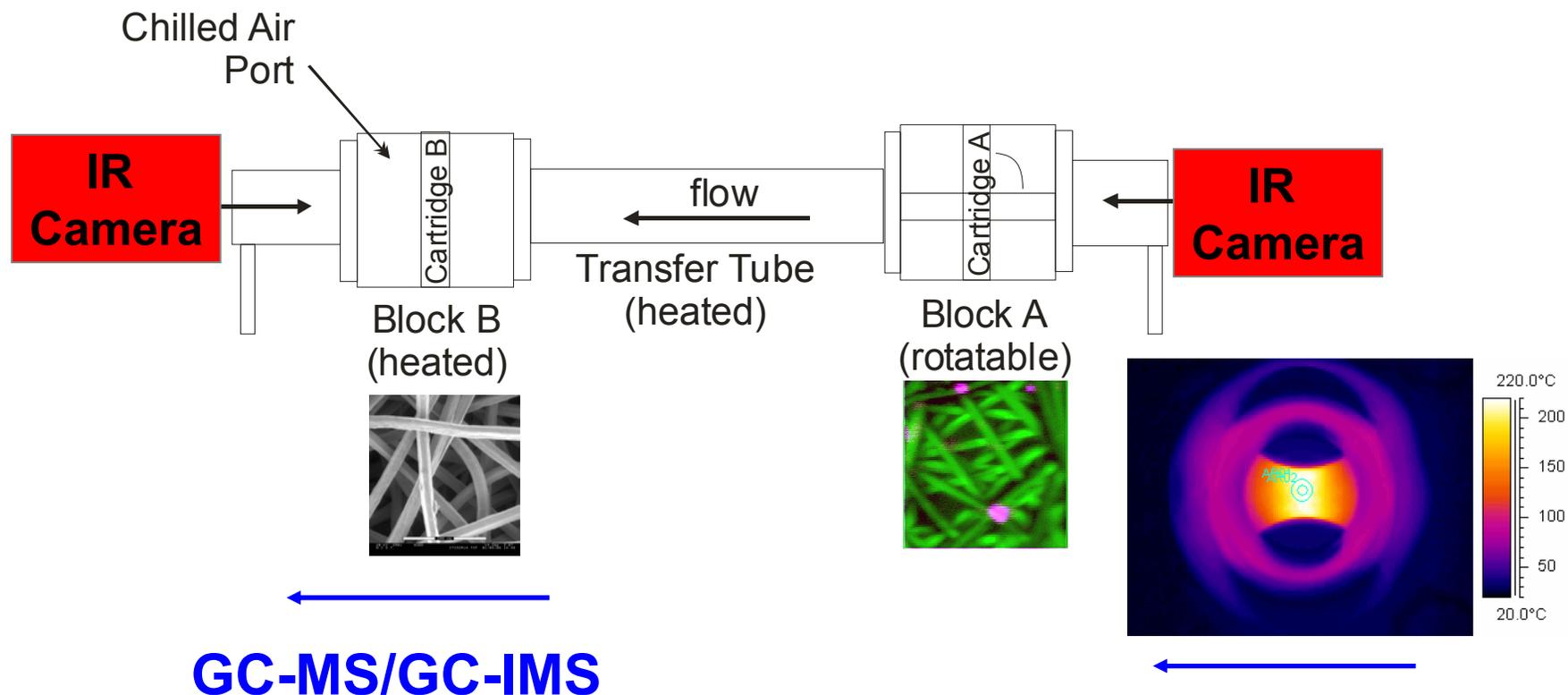




C-4 on muslin, -15 C temperature differential

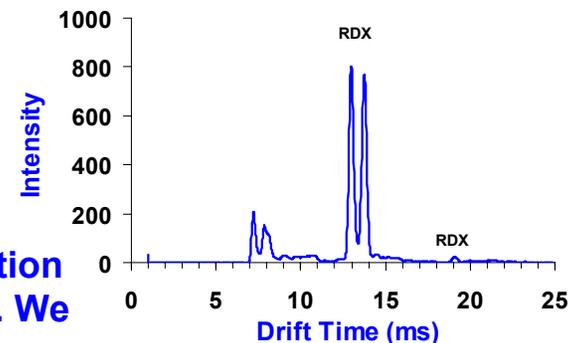
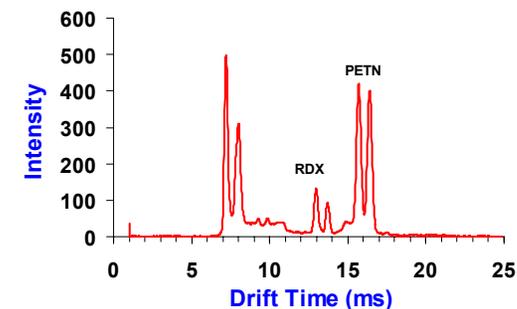
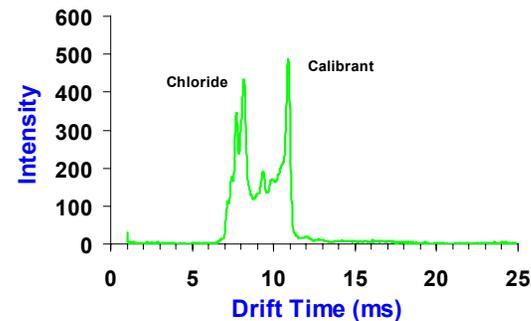
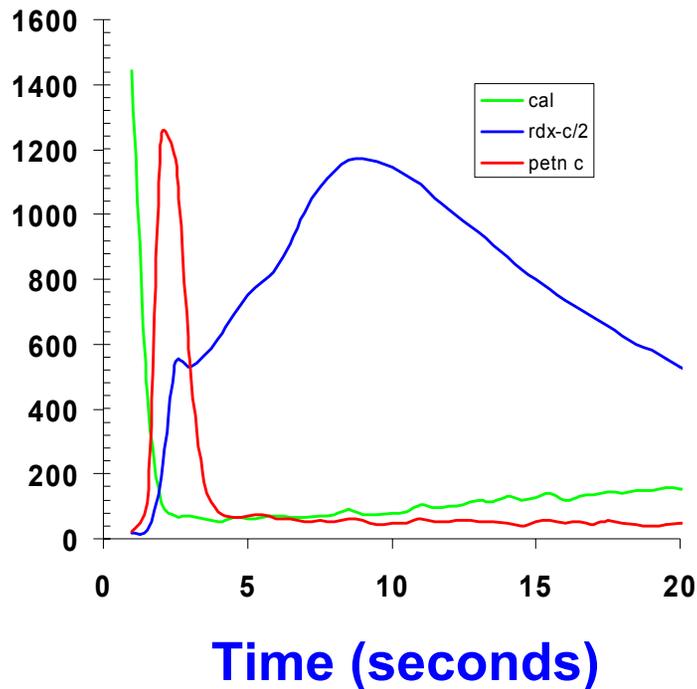
# Characterization of 2<sup>nd</sup> Stage Preconcentrator

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 has been constructed to evaluate the efficiency of 2<sup>nd</sup> stage preconcentration technology for explosive vapor collection.



# Ion Mobility Spectrometry Studies-1

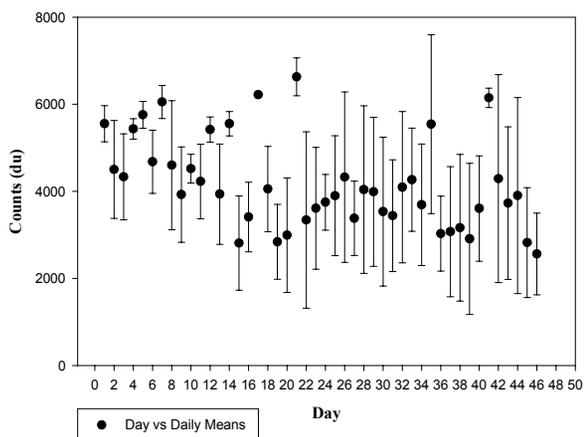
## TSA Semtex Standard 440 ng on swipe



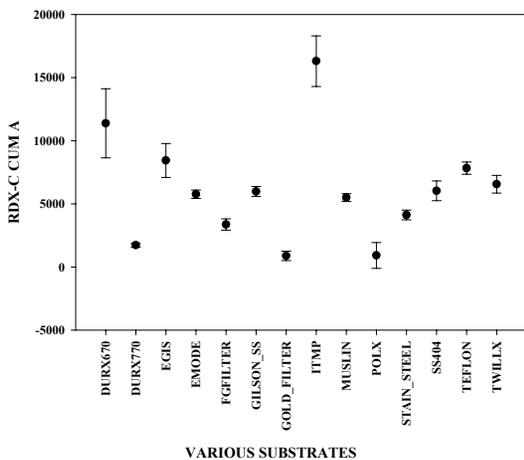
Temporal thermal desorption IMS profiles provide additional information about the composition of the sample and its behavior during heating. We are studying the influence of particle size and desorber temperature on the desorption profiles. This will be correlated with temperature programmed Raman, Optical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

# Ion Mobility Spectrometry Studies-2

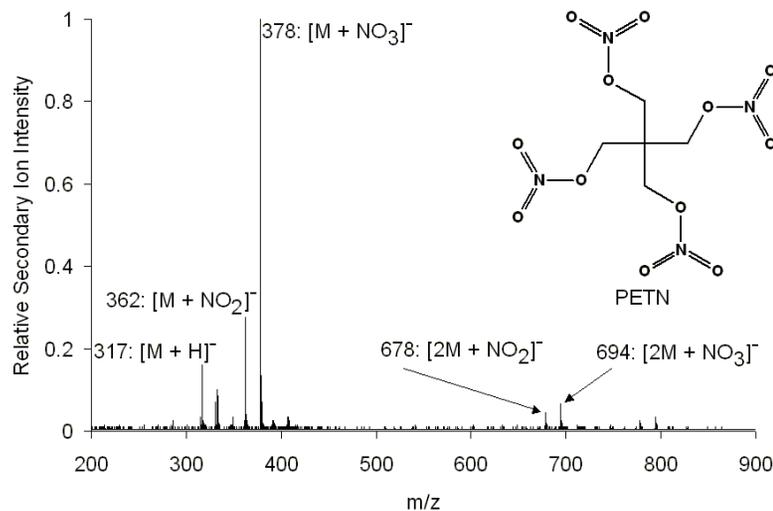
RDX-C (Cum Amp) Data Collected for 1  $\mu\text{g/mL}$  RDX Solution  
February 13, 2003 - April 21, 2003  
Data Set Mean =  $4165.5 \pm 1135.6$



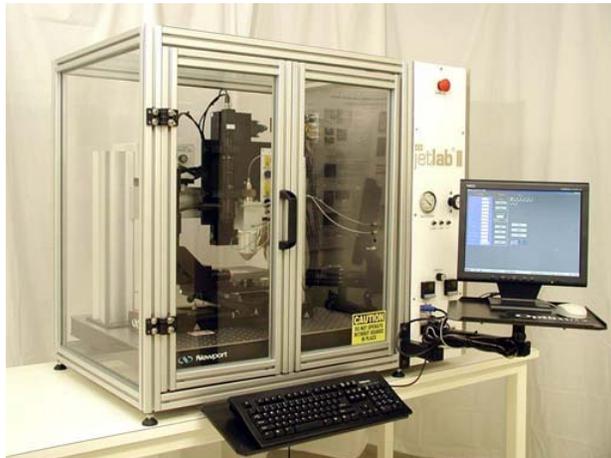
RDX INTENSITY VS. SUBSTRATE



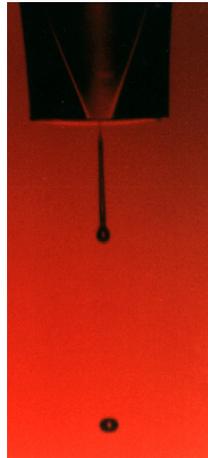
Statistical variability  
Quantitative Analysis  
Spectral Library  
Trace Constituents  
Smokeless Powders



# Preparation of Printed Explosives Standards

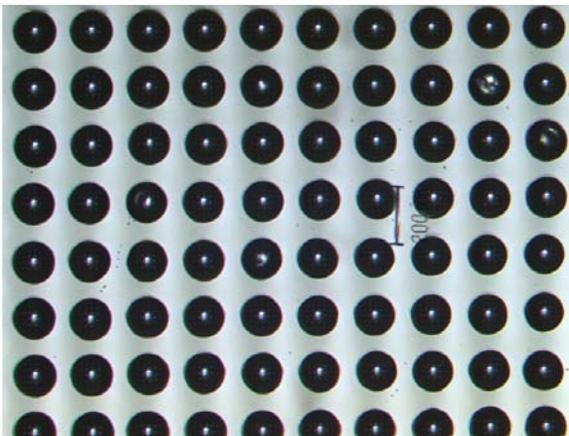


MicroFAB Jet Lab II

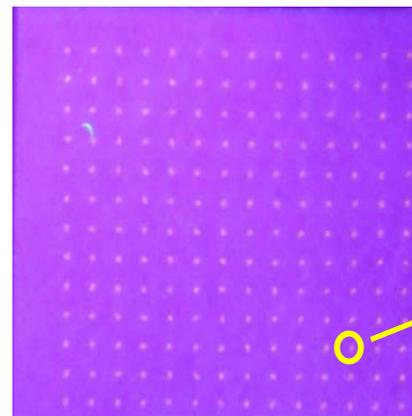


- Use MicroFab JetLab II inkjet printer to print explosive/narcotic standards on a variety of surfaces. Experiment with polymer/explosives mixtures.
- 2 types of standards: testing collection methodologies and IMS response.
- Use advanced surface analysis tools to characterize droplet characteristics.

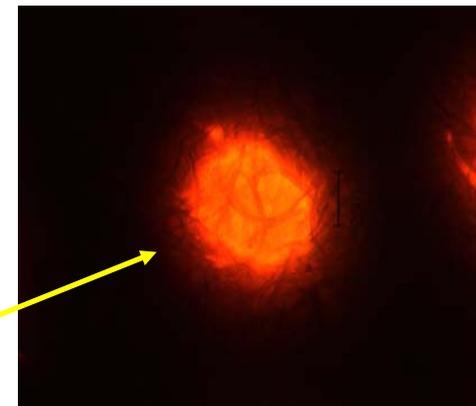
## Polymer Droplet Array



## Rhodamine + TNT

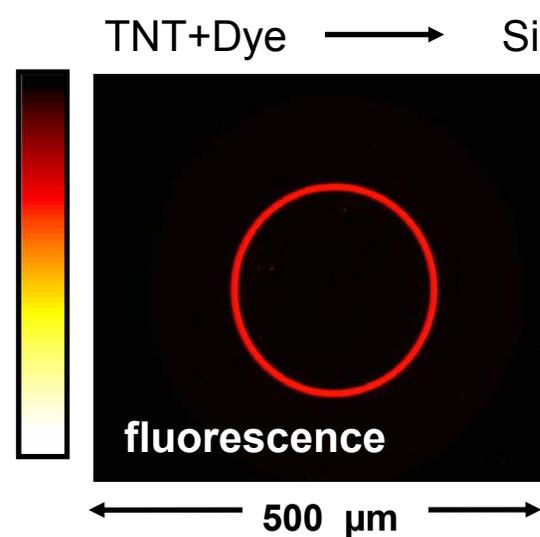
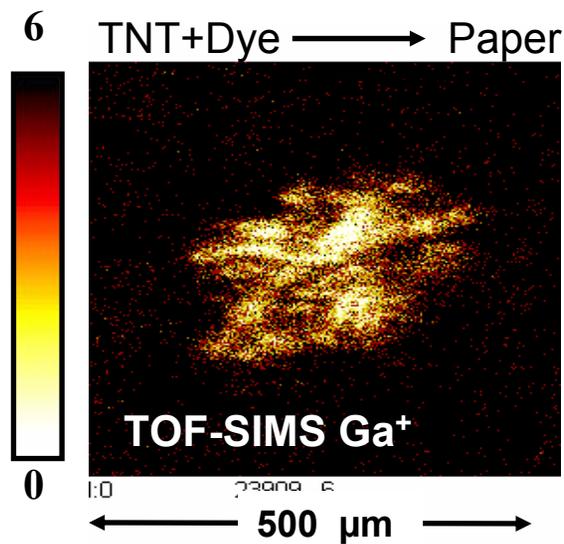
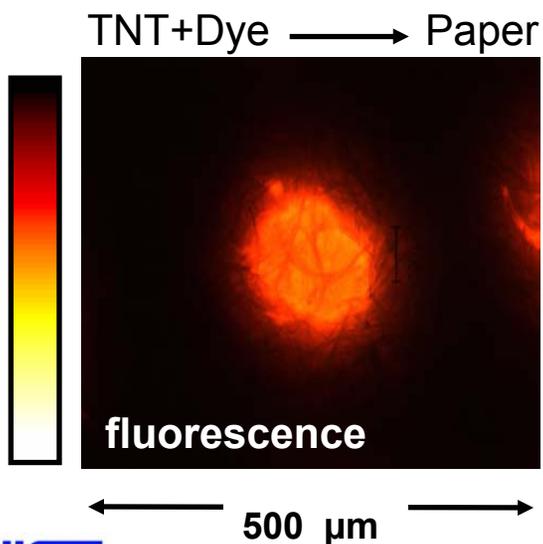
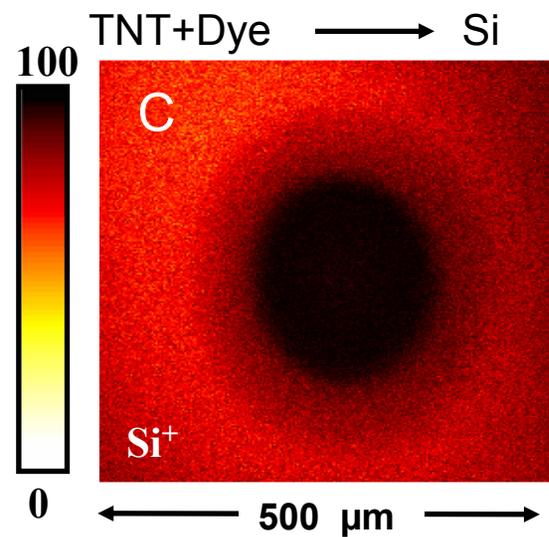
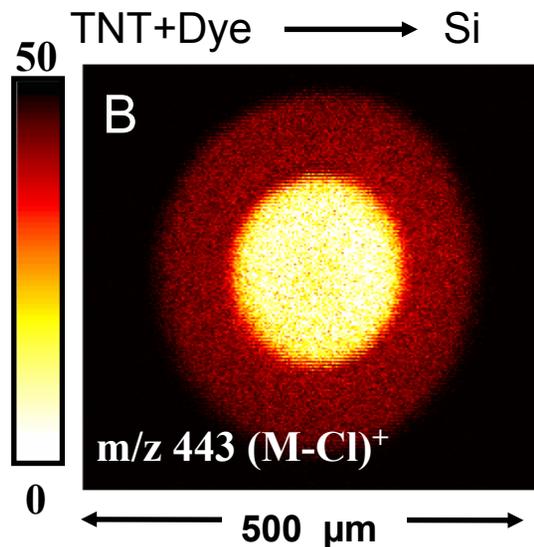
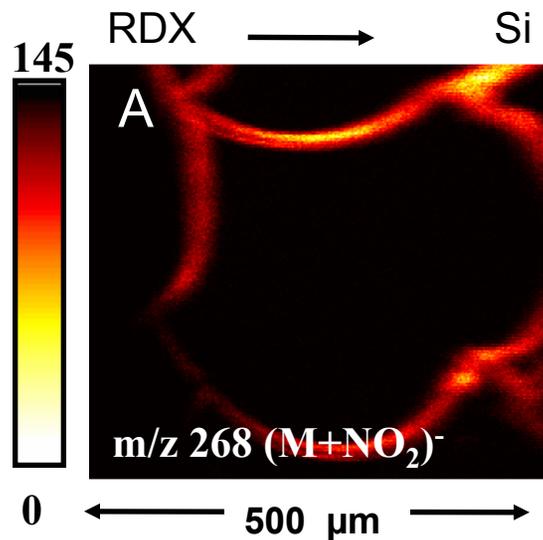


← 2.5 cm →



← 150 μm →

# Microbeam $SF_5^+$ SIMS Imaging of InkJet Droplets



# Conclusion



The NIST Surface and Microanalysis Science Division is 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. The use of advanced microanalytical and surface analysis techniques is a key component of this program. Key areas of focus include:



- Explosive particle release from surfaces
- Explosive particle collection
- Ion Mobility Spectrometry detection of explosives
- Standards for explosive detection

**For more information:**

**NIST**

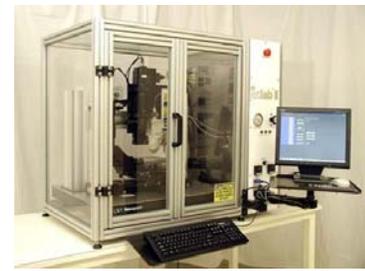
[greg.gillen@nist.gov](mailto:greg.gillen@nist.gov)

[robert.fletcher@nist.gov](mailto:robert.fletcher@nist.gov)

**TSA**

[richard.lareau@faa.gov](mailto:richard.lareau@faa.gov)

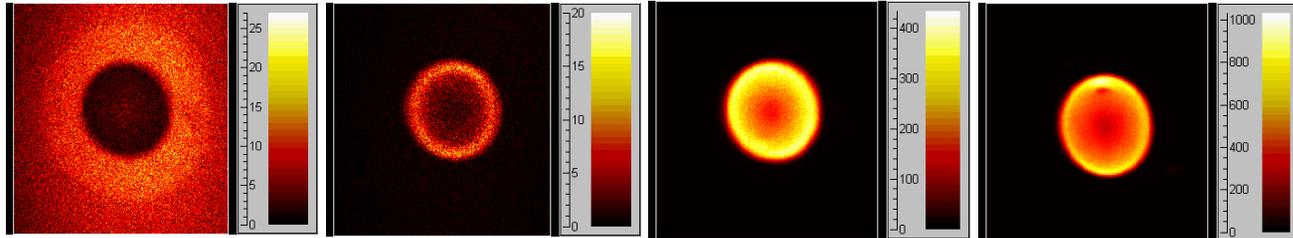
# NIST Uses for JetLab II



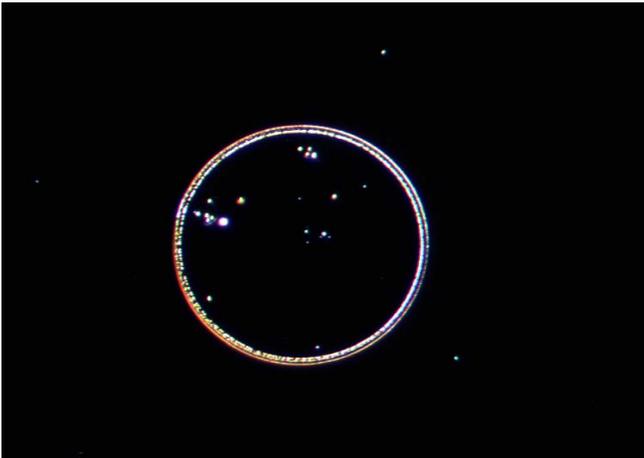
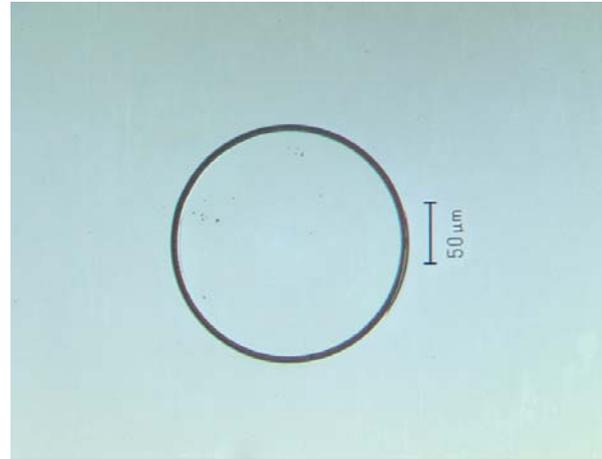
- **Pure Explosive Standards:** IMS screening response, air jet removal, wipe removal, instrument calibration.
- **Composite Explosive Standards:** same as above.
- **Production of composite fluorescent sphere/high explosives standard**
- **Narcotics Standards:** same as samples above
- **Drug Delivery Sphere Production**
- **DNA/Biomolecule Arrays.** Standards for surface analysis techniques.
- **Generation of individual particle arrays**

# Acknowledgements

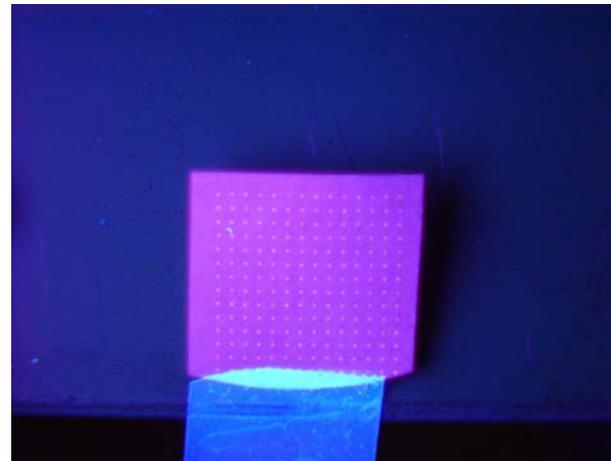
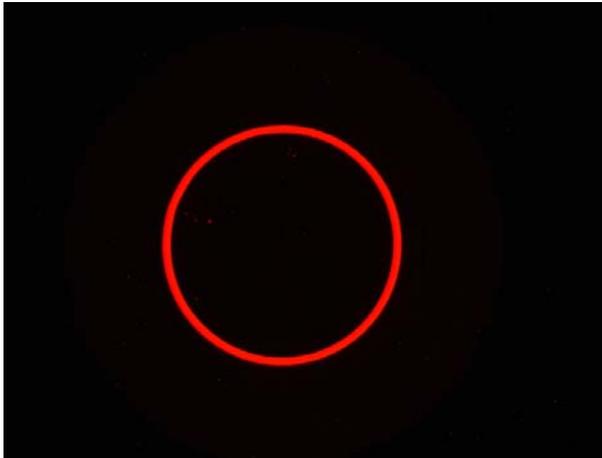
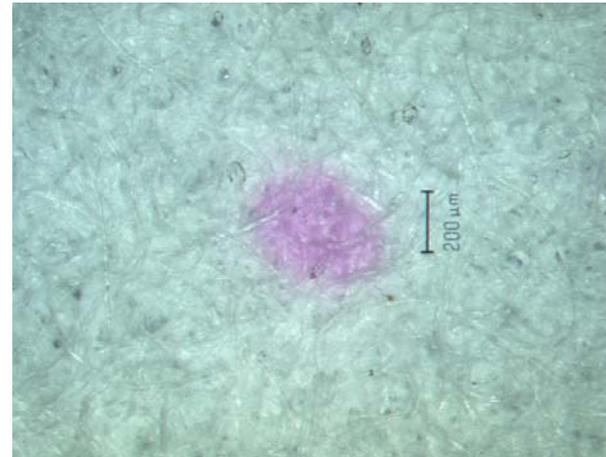
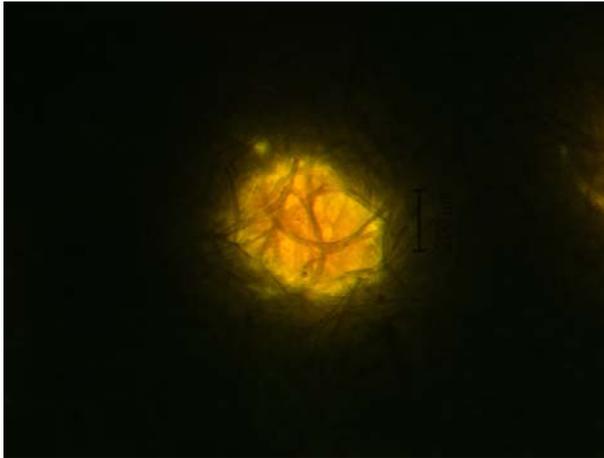
- Analytical Microscopy Research Group at NIST
- Richard Lareau, Transportation Security Administration, Trace Detection Group
- Mike Boldman, Senior Engineer, MicroFab Technologies
- Funding from NIST Advanced Technology Program, Office of Law Enforcement Standards, Office of Domestic Preparedness, Department of Homeland Security.



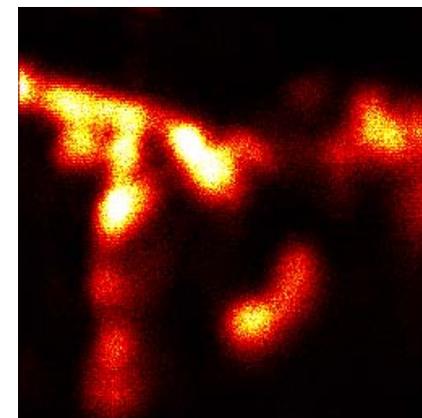
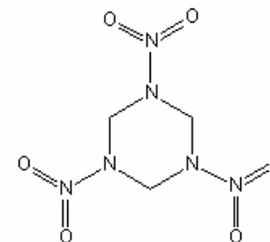
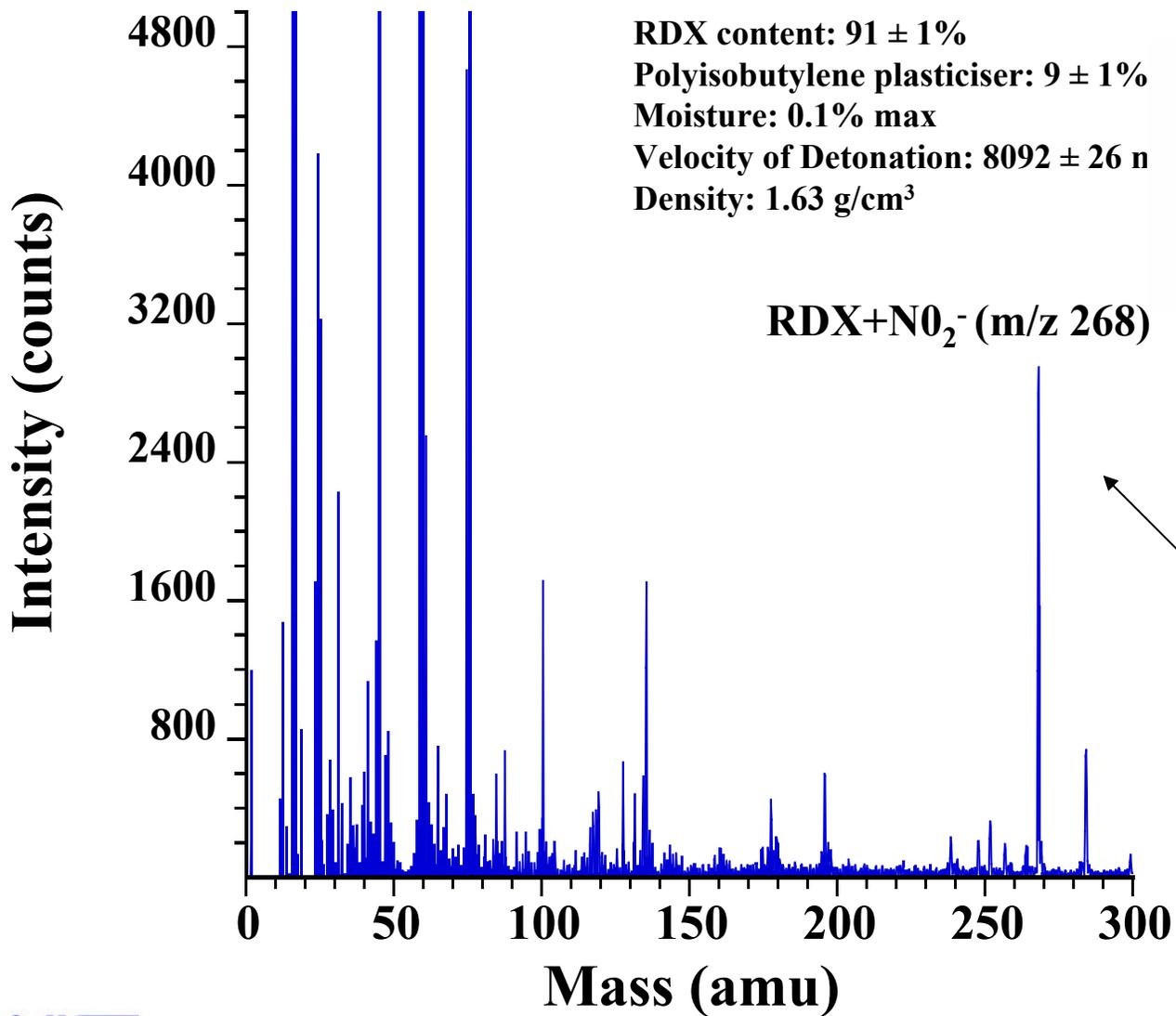
# InkJet Printed Samples



# InkJet Printed Samples

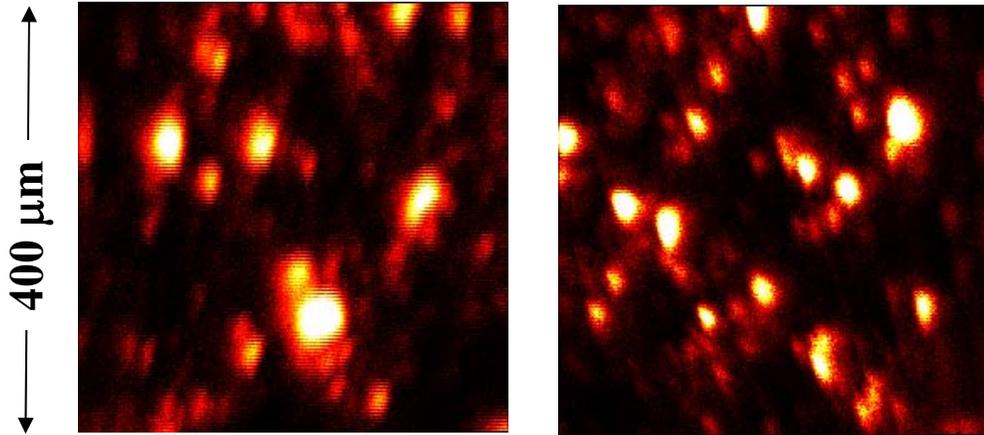


# Mass Spectrum of C4 Particle on Silicon



500  $\mu$ m  
0-115 cts/pixel  
**Image of m/z 268**

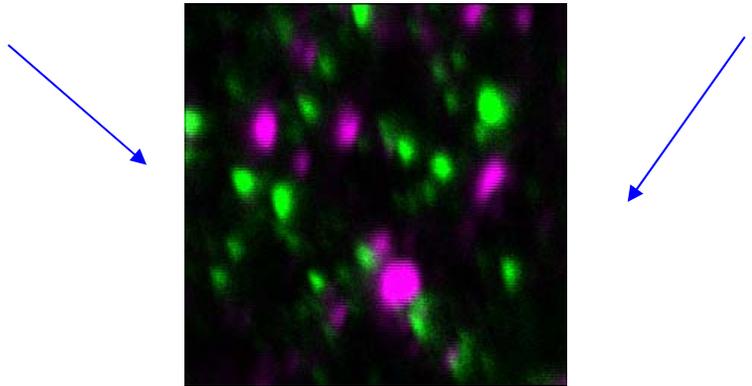
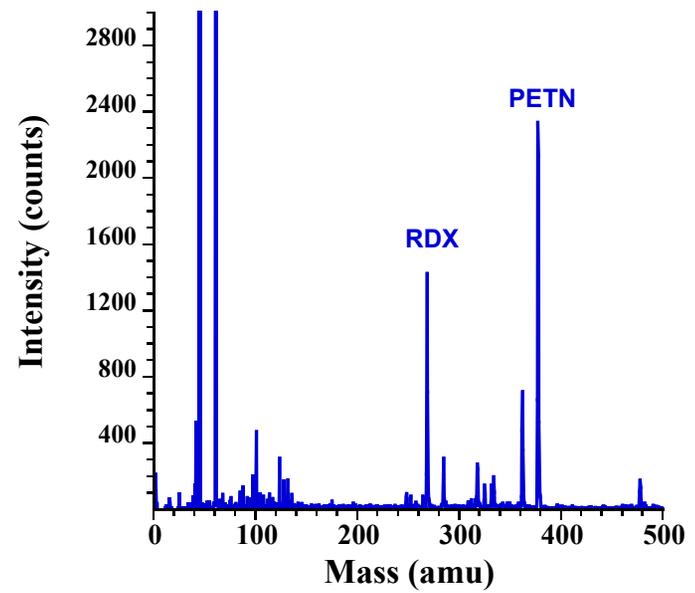
# Microbeam C<sub>8</sub>-Imaging of PETN and RDX Particles on Silicon



PETN (0-440 cts/pixel)

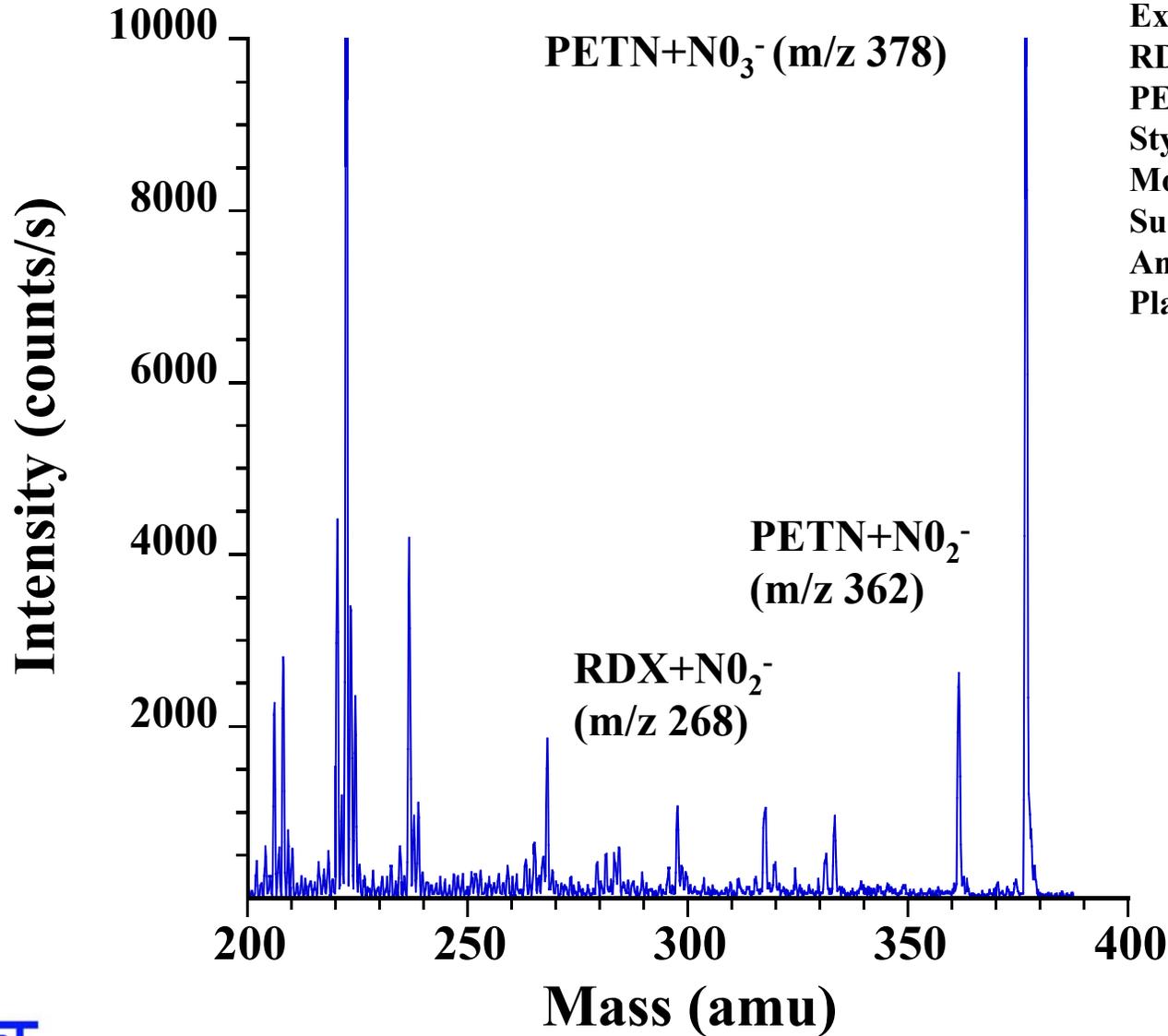
RDX (0-270 cts/pixel)

### Mass Spectrum After Image Acquisition



30 s image acquisition

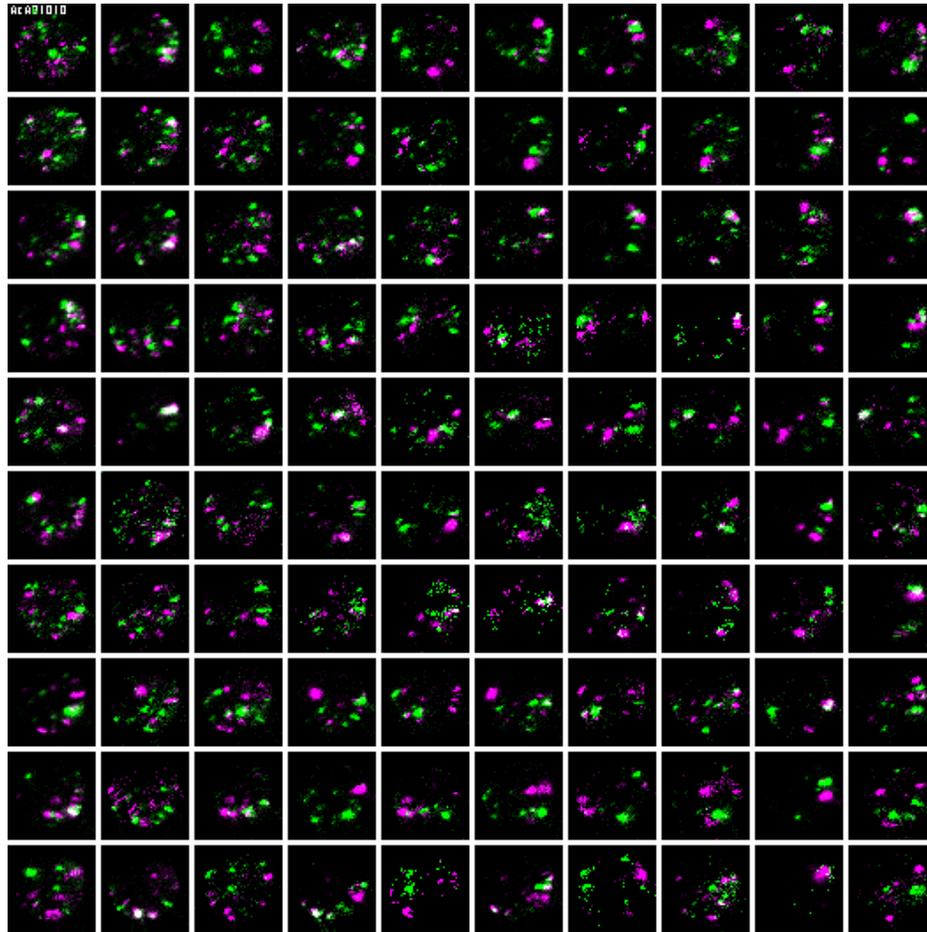
# Mass Spectrum of Semtex-H on Silicon



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

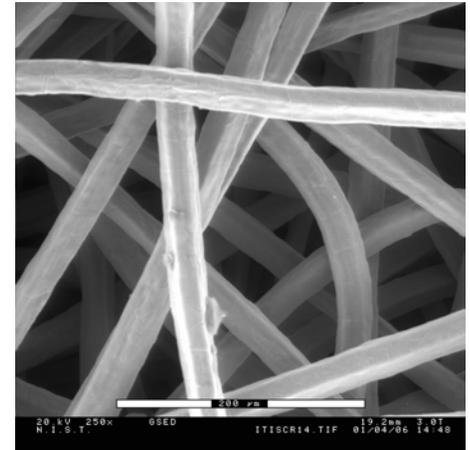
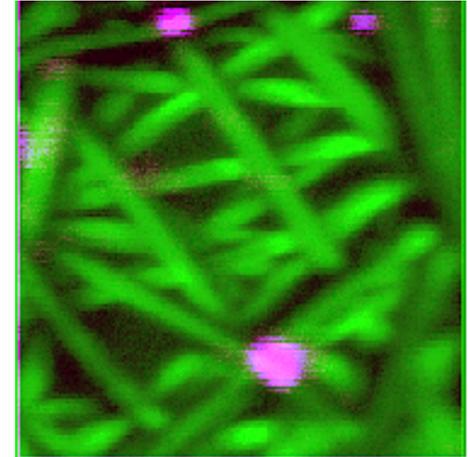
# PETN and RDX Particle Overlay Showing Distribution of Explosive Particles on Surface

RDX  
PETN

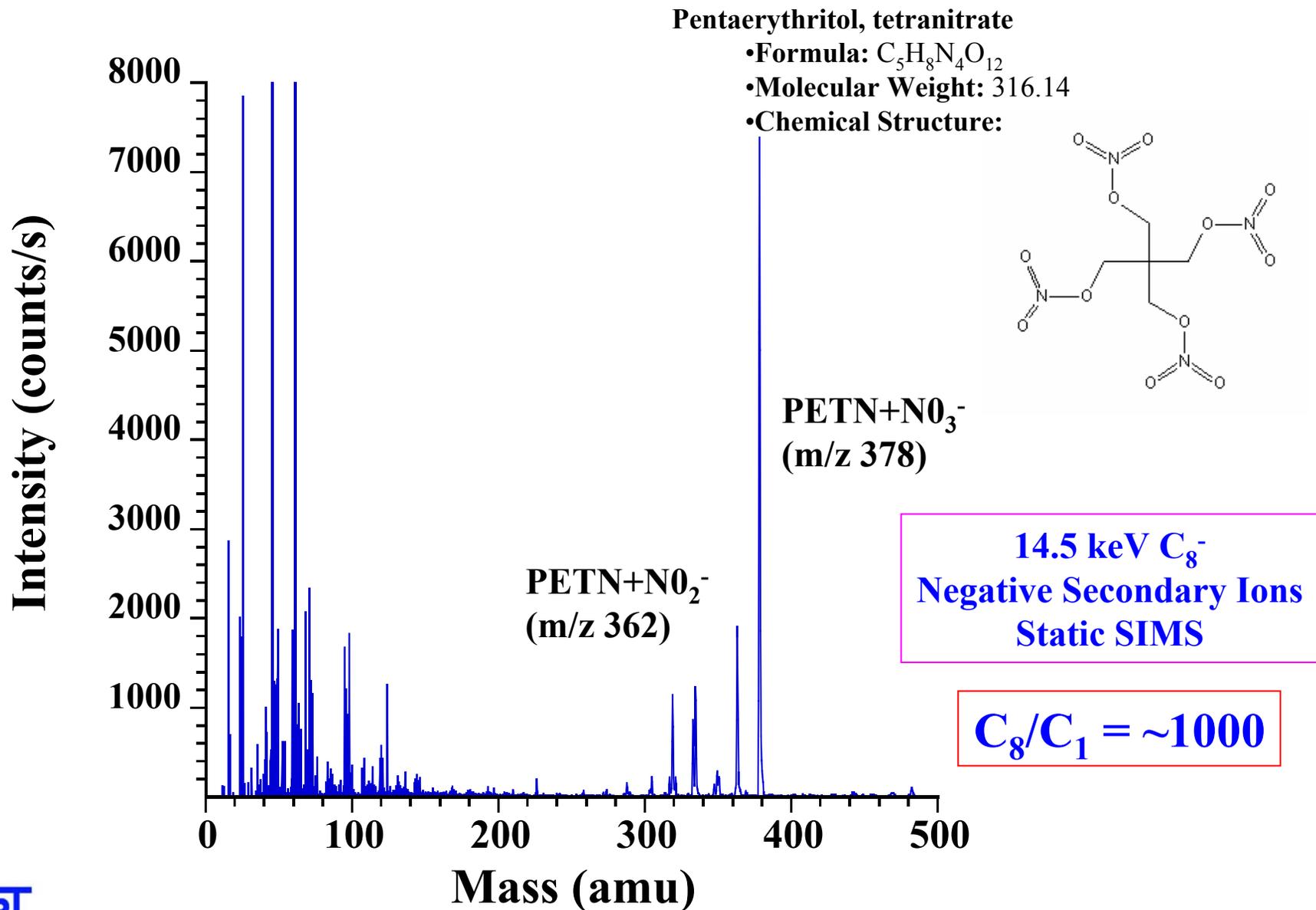


3.5 mm

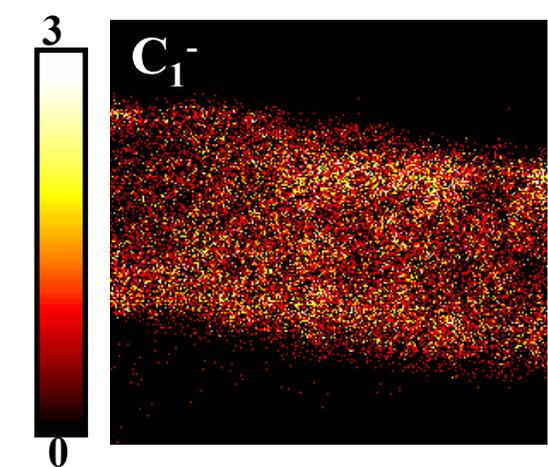
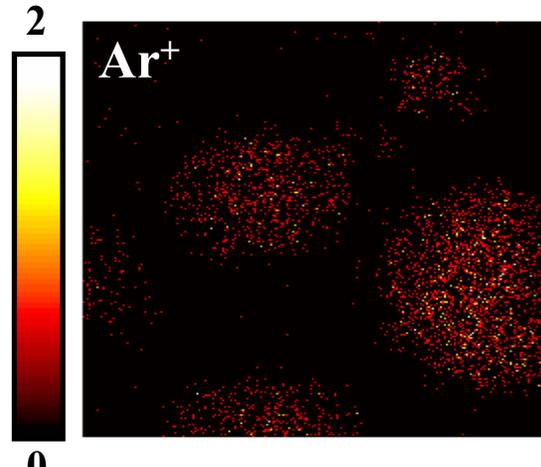
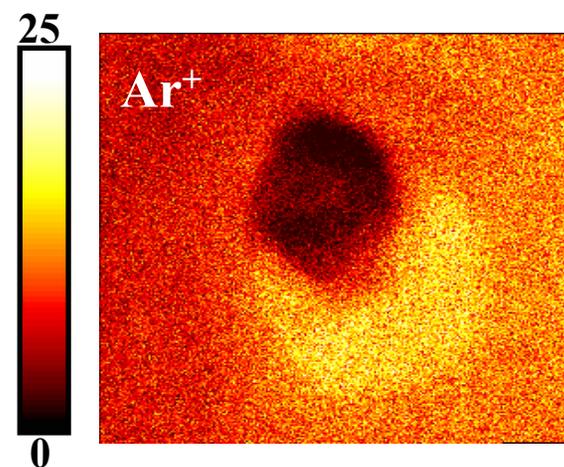
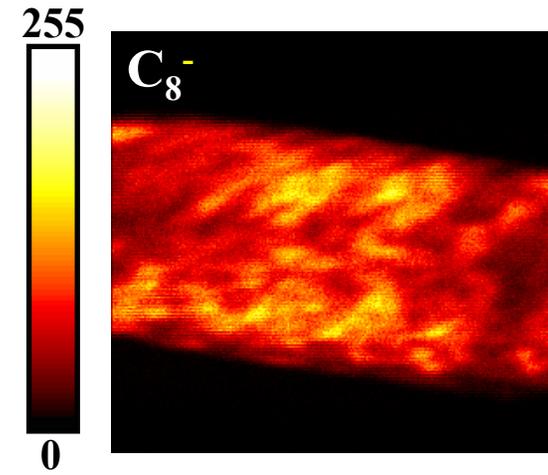
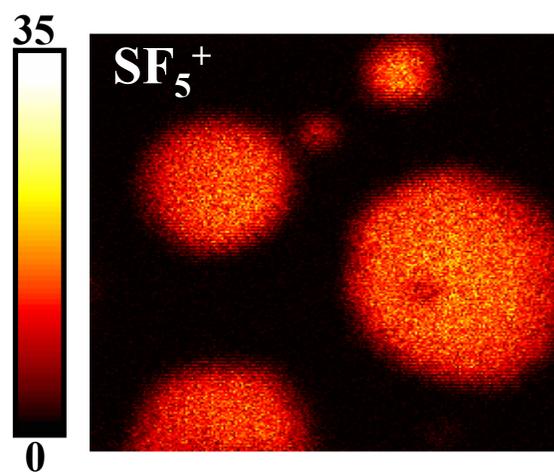
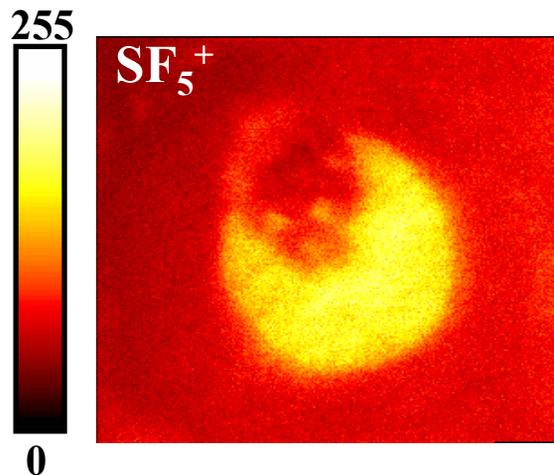
3.5 mm



# Mass Spectrum of PETN Particles on Silicon



# Chemical Imaging with Cluster Primary Ions



← 250  $\mu m$  →

Cocaine deposit on silicon. Static SIMS image of protonated molecular ion at  $m/z$  304.

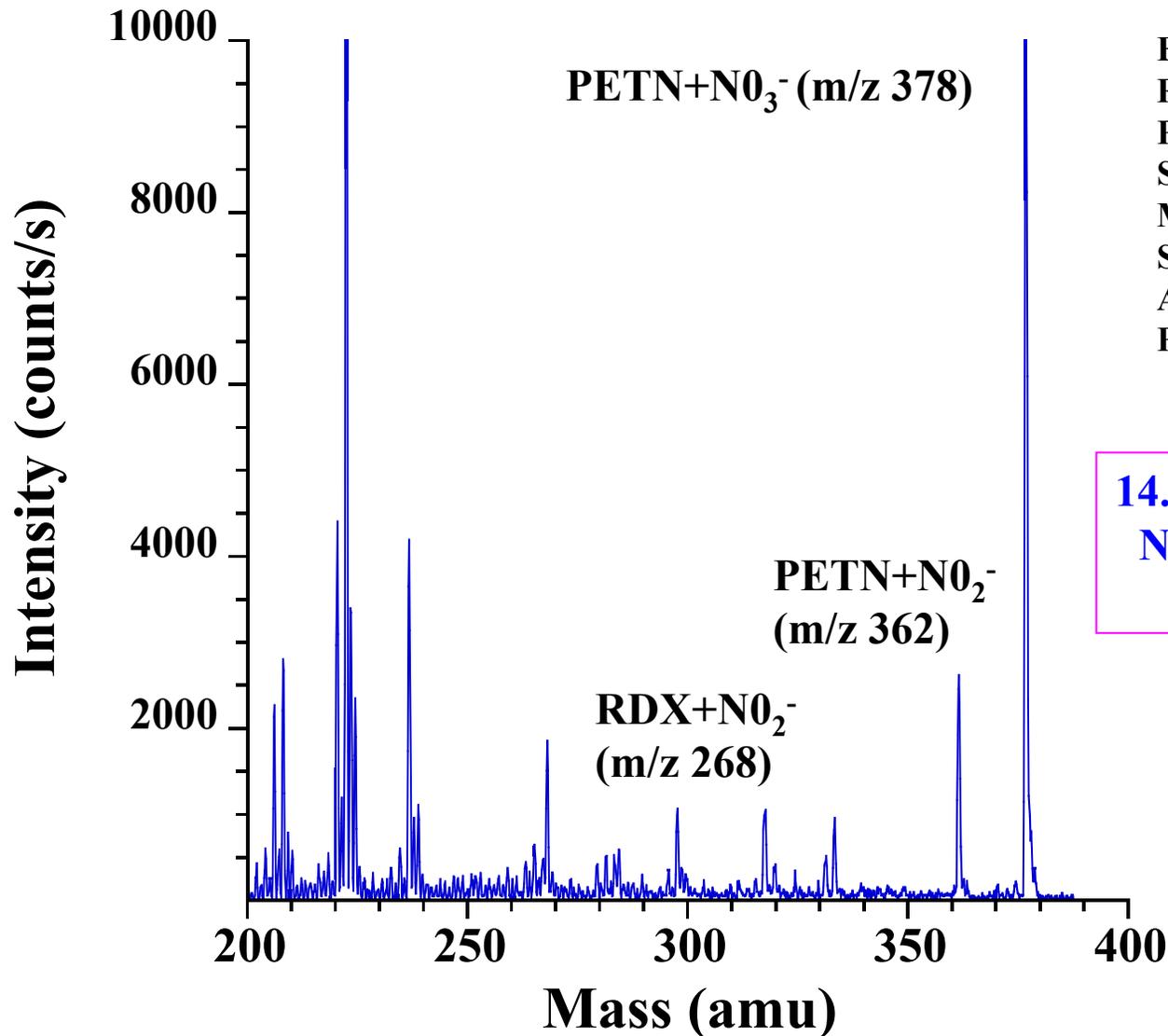
← 500  $\mu m$  →

Spray drug delivery system. Static SIMS image of active steroid component  $\sim m/z$  364.

← 150  $\mu m$  →

Organic salt on human hair. Static SIMS image of molecular ion at  $m/z$  242.

# Cluster SIMS Mass Spectrum of Semtex-H Particle



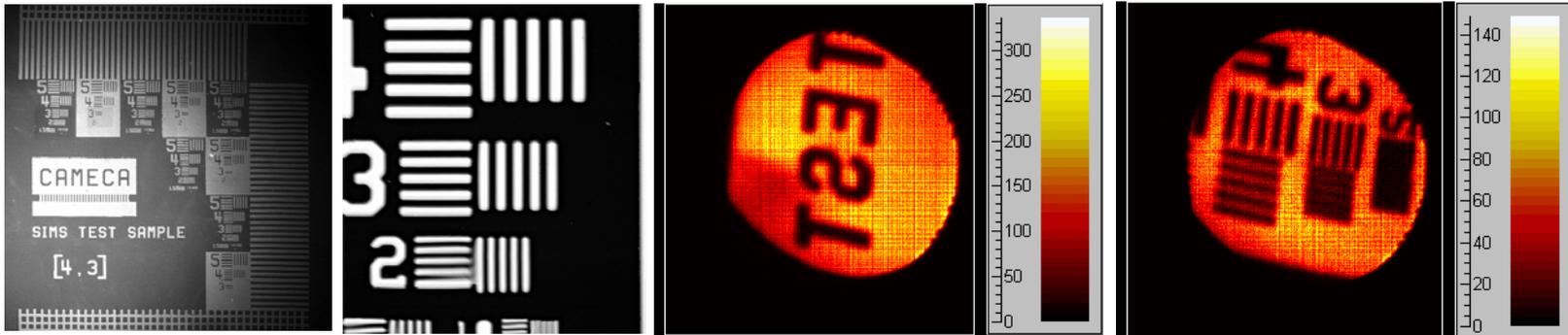
Explosives ~70% by weight  
RDX content: 50.2%  
PETN content 49.8%  
Styrene-butadiene rubber: 9%  
Motor Oil: 8%  
Sudan I Dye  
Antioxidant  
Plastizier

14.5 keV C<sub>8</sub><sup>-</sup> Bombardment  
Negative Secondary Ions  
Static SIMS

C<sub>8</sub>/C<sub>1</sub> = ~1000

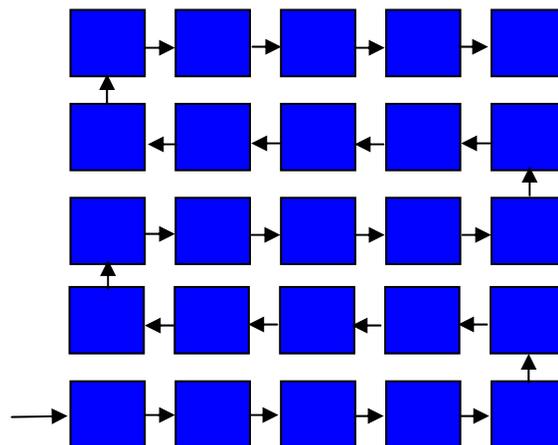
# Equipment for Organic Particle Searching

- Cameca IMS 4F with  $SF_5^+$  or  $C_8^-$  primary ion beams.
- Extended mass range, fast RAE, post acceleration option.
- Charles Evans and Associates PXT Software.
- Lispix image processing software from NIST.
- Samples from trace explosives detection group (TSA).
- Spatial calibration using Cameca Si/Ta test sample calibrated on FEG-SEM.



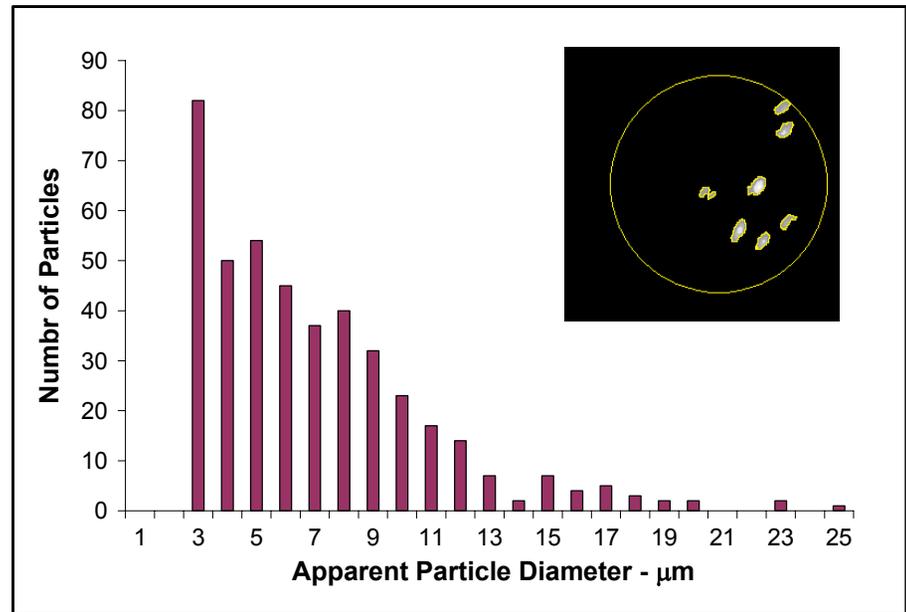
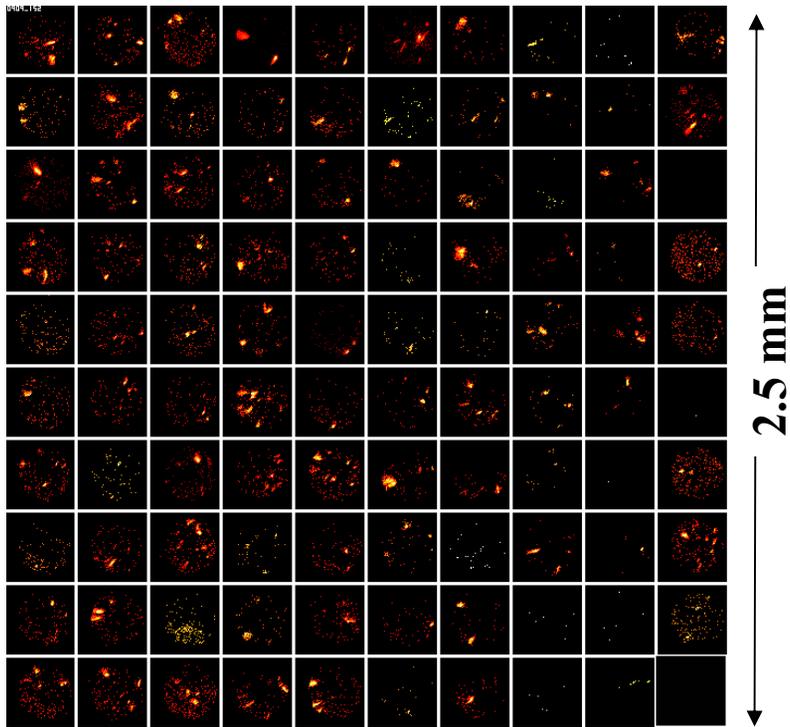
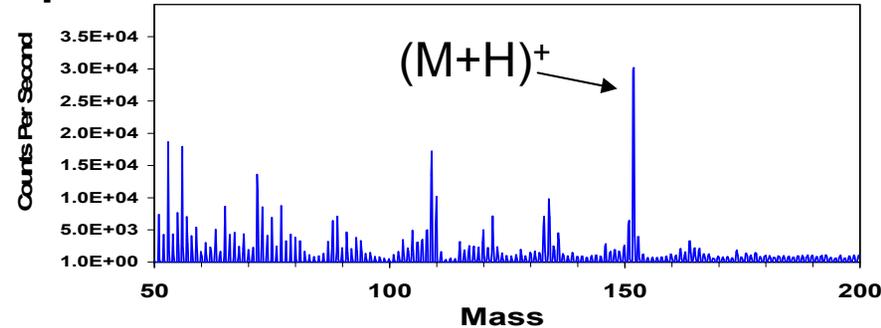
# Procedure for Organic Particle Searching

- Acquire images of characteristic molecular ions in microscope (dynamic) or microprobe imaging mode (static).
- Acquire each image for ~ 20-60 sec. Primary current typically 0.5-3 nA into 250  $\mu\text{m}$  raster. Dose (per image field) during acquisition  $\sim 1 \times 10^{14} - 1.7 \times 10^{15}$  ions/ $\text{cm}^2$ .
- Microprobe imaging using static SIMS conditions  $5 \times 10^{-11}$  amps, 500  $\mu\text{m}$  raster, dose during acquisition  $\sim 2-7 \times 10^{12}$  ions/ $\text{cm}^2$ .
- Presputter used as needed.



# 1. Cluster SIMS Analysis of Fingerprint ( $SF_5^+$ )

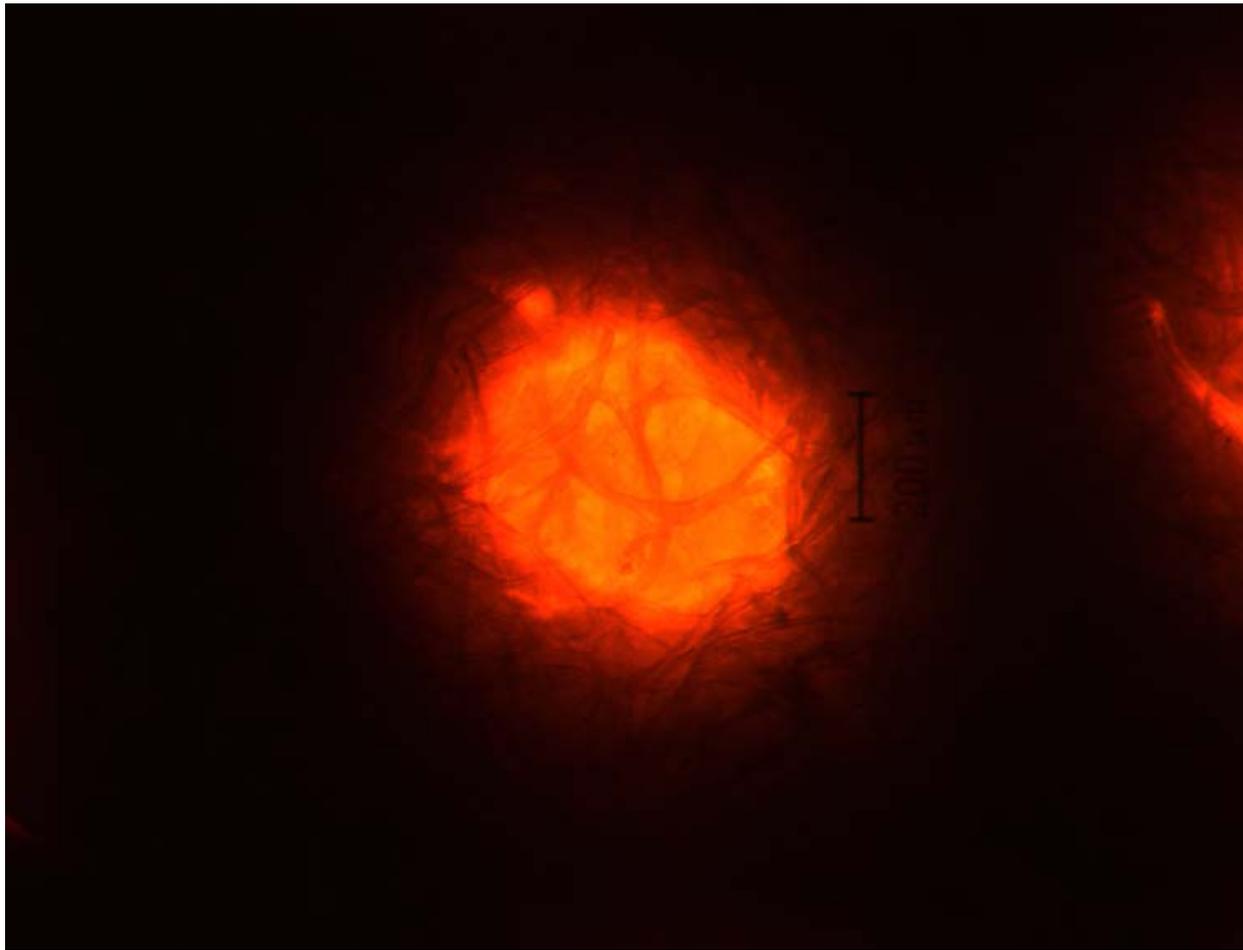
- 10 x 10 particle search in microscope mode.
- Acetaminophen particles in human fingerprint.
- Image  $^{29}Si$  for 10 sec,  $(M+H)^+$  for 20 sec
- Dose  $8.5 \times 10^{13}$  ions/cm $^2$
- Total time 63 minutes (25% overhead).



← 2.5 mm →

451 particles identified

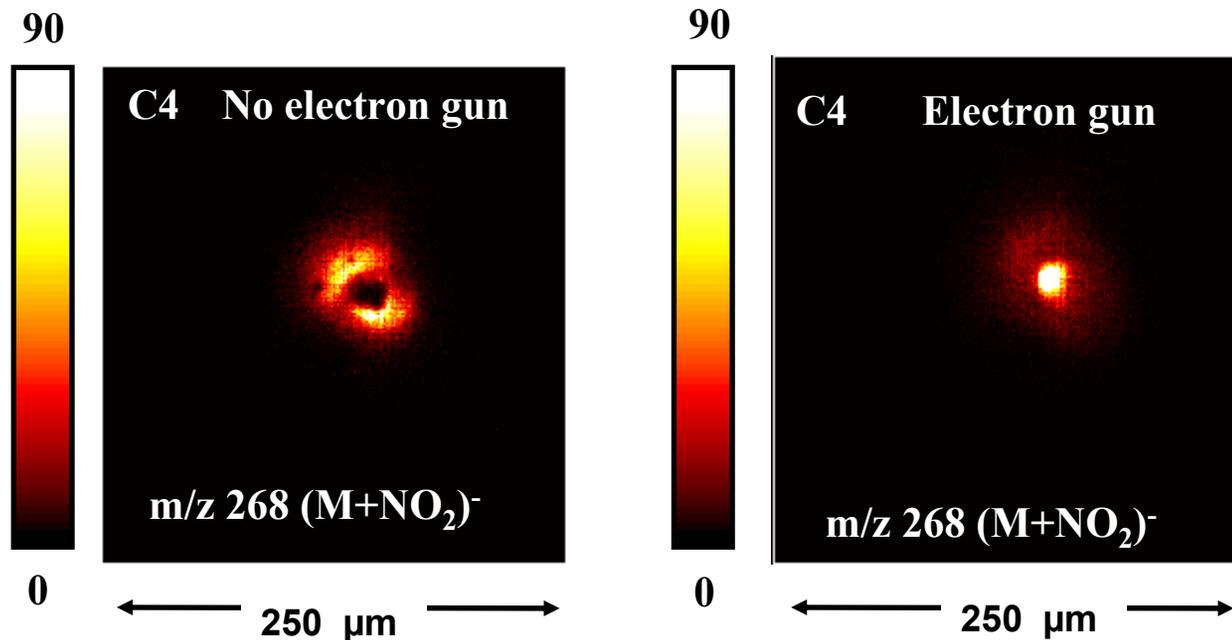
# TNT+Rhodamine B Inkjet Droplet



# Measurement Issues

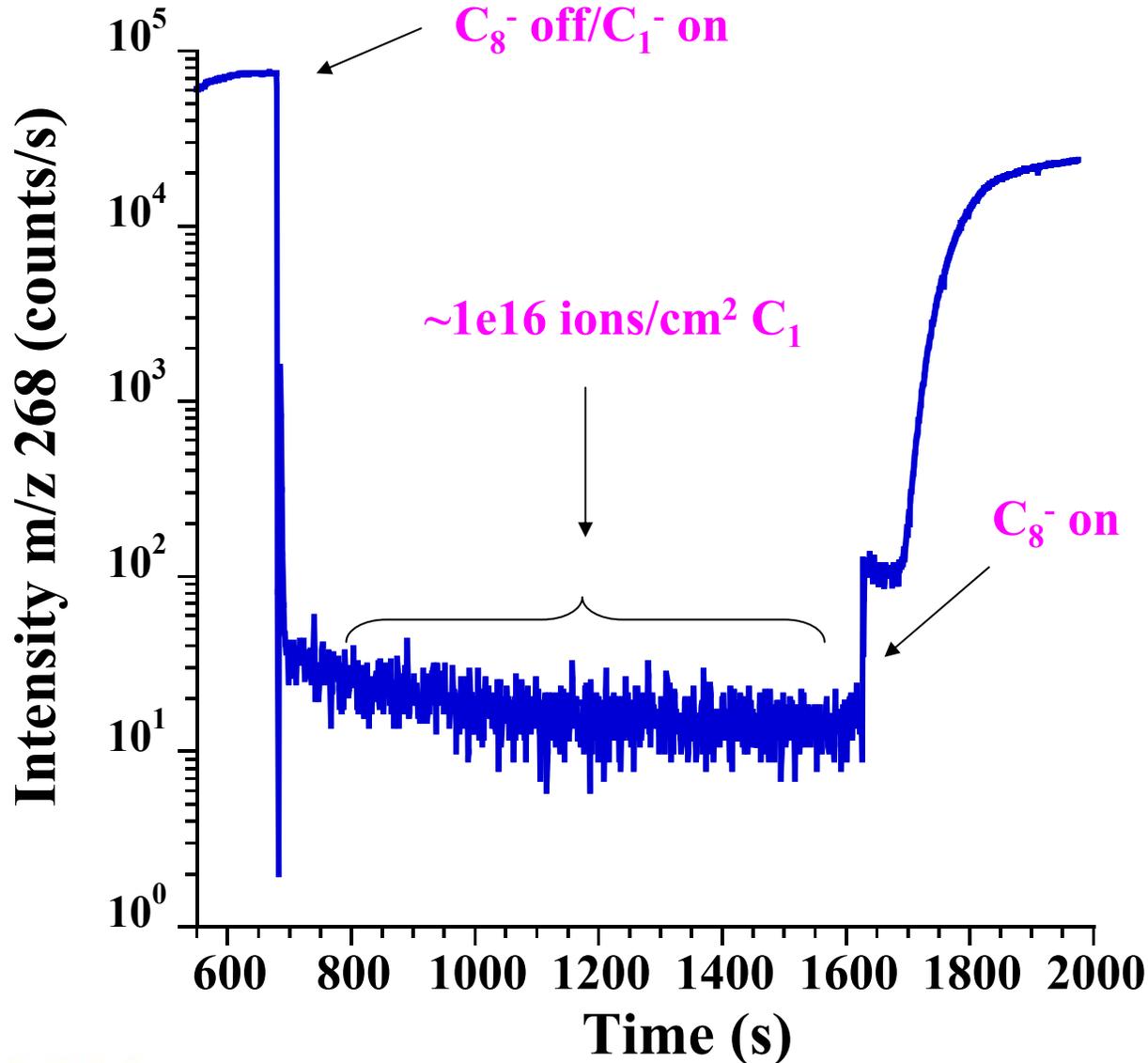
## Particle samples are insulators

- $\text{SF}_5^+$  Au/Pd sample coating (sometimes works). Normal incidence electron gun.
- $\text{C}_8^-$  charging typically not much of a problem.

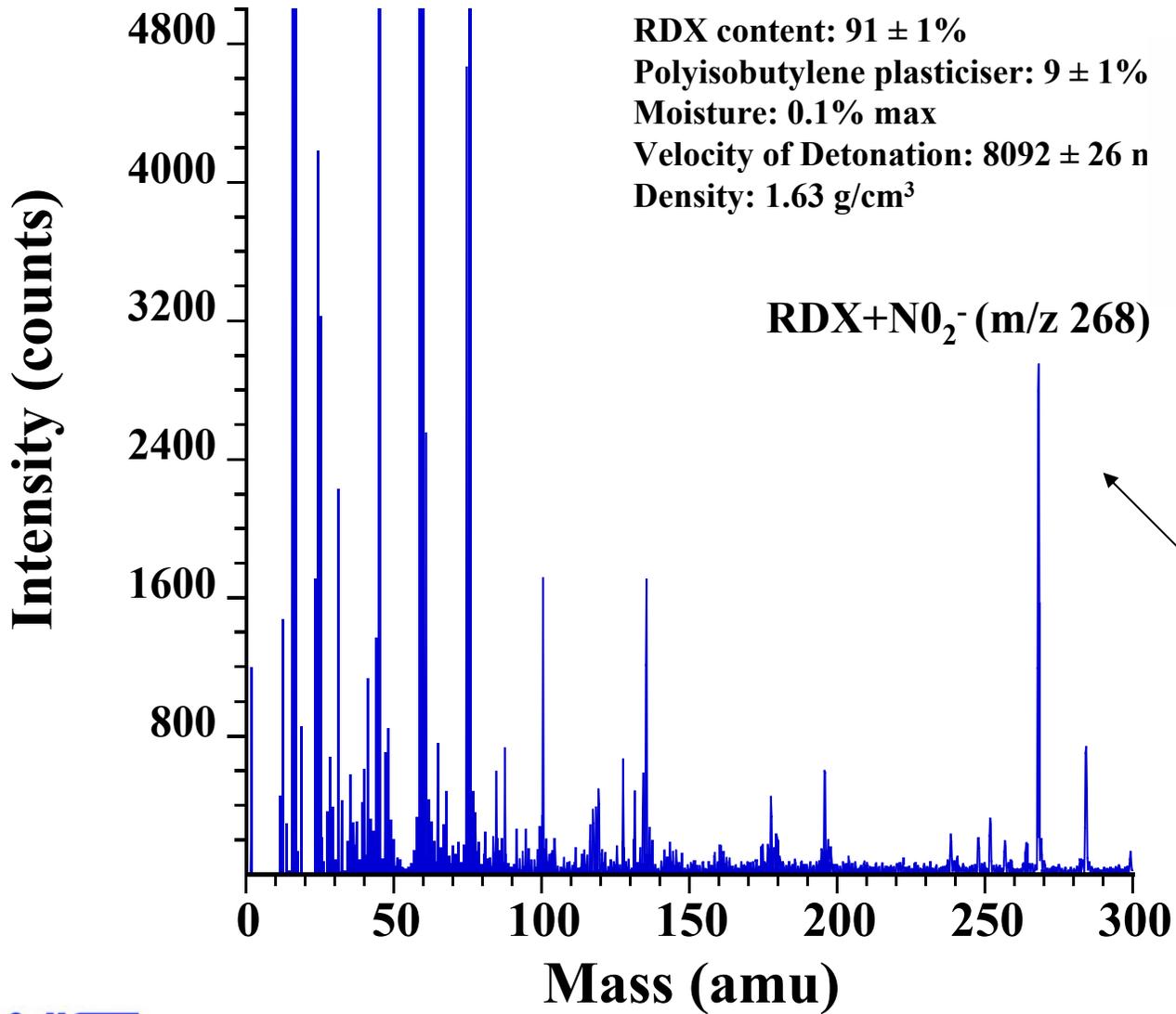


**\*Resputtering of material may be serious problem**

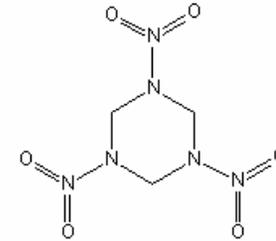
# Depth Profile of Individual RDX Particles



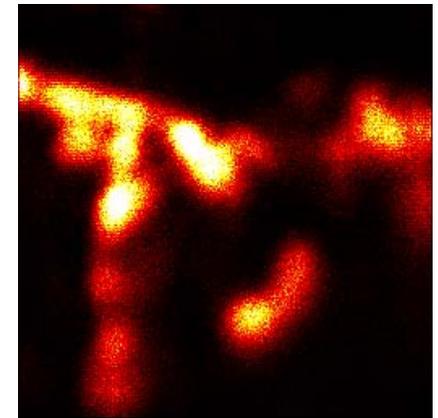
# Cluster SIMS Mass Spectrum of C4 Particle



**RDX content:  $91 \pm 1\%$**   
**Polyisobutylene plasticiser:  $9 \pm 1\%$**   
**Moisture: 0.1% max**  
**Velocity of Detonation:  $8092 \pm 26$  n**  
**Density:  $1.63 \text{ g/cm}^3$**



**RDX+N<sub>2</sub><sup>-</sup> (m/z 268)**



← 500 μm →

0-115 cts/pixel

Image of m/z 268

# Characterization, Optimization, Calibration and Standardization of Trace Explosive Detection Units



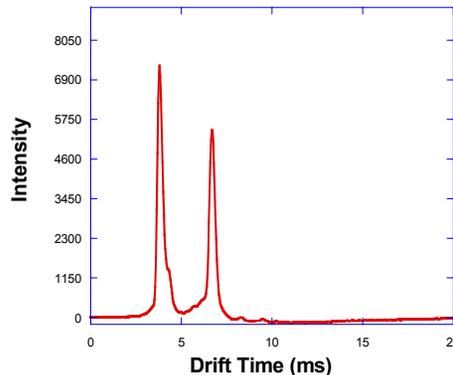
Use advanced surface and microanalysis capabilities at NIST to study the performance characteristics of portable and tabletop trace explosive detection instruments. Conduct research to optimize, calibrate and standardize these instruments. Emphasis on use of this technology by First Responders and Airport Security



### Swipe Detection

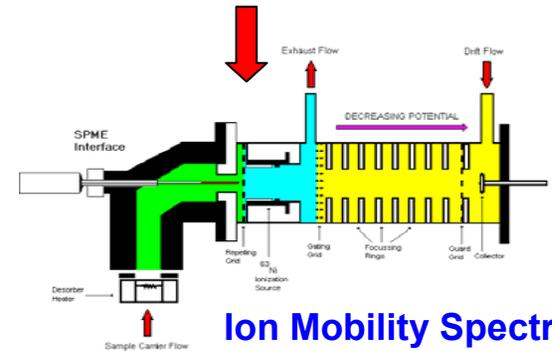
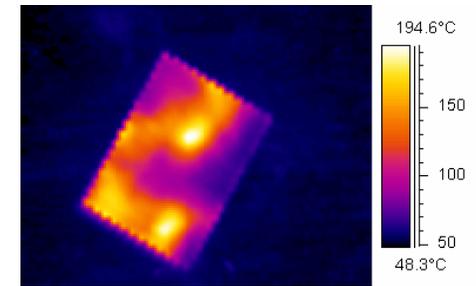


TNT Standard ALM00632



### Explosive Detection

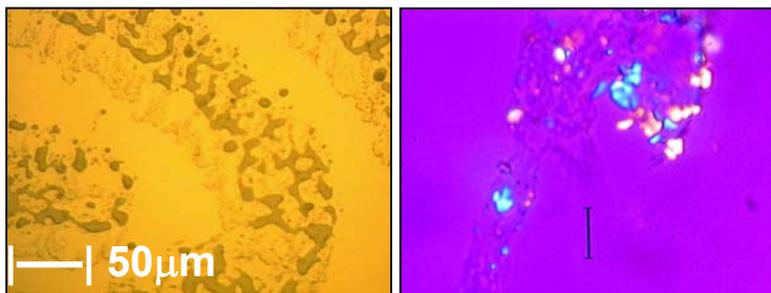
### Swipe Heated to Desorb Explosive Vapors



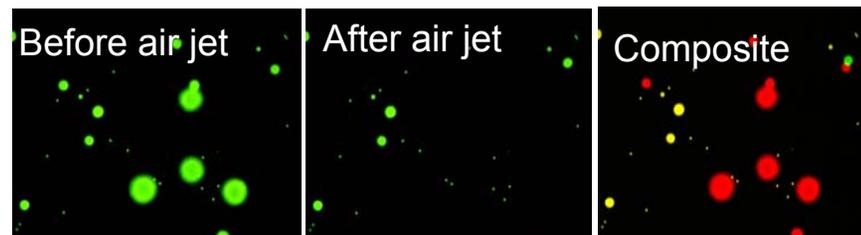
### Ion Mobility Spectrometer

# Research being used to support “Development of Measurement Infrastructure for Trace Explosive Detection at NIST”

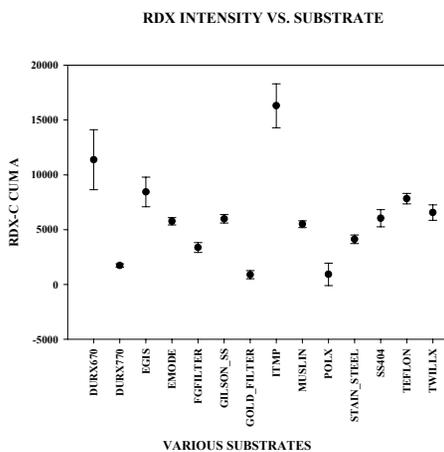
## What is being Sampled?



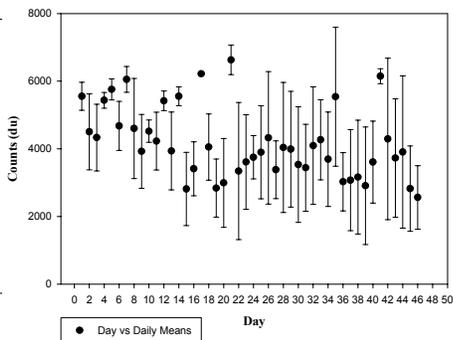
## Particle Removal/Collection



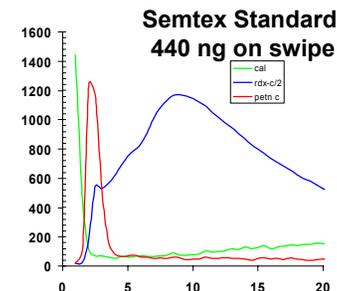
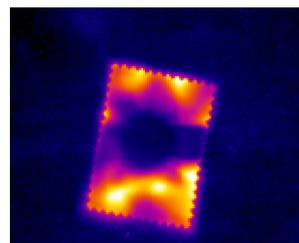
## Instrument Performance



RDX-C (Cum Amp) Data Collected for 1 µg/mL RDX Solution  
February 13, 2003 - April 21, 2003  
Data Set Mean =  $4165.5 \pm 1135.6$



## Optimize Operational Parameters



## Explosive Standards



# Procedure for Organic Particle Searching

- Acquire images of characteristic molecular ions in microscope (dynamic) or microprobe imaging mode (static).
- Acquire each image for ~ 20-60 sec. Primary current typically 0.5-3 nA into 250  $\mu\text{m}$  raster. Dose (per image field) during acquisition  $\sim 1 \times 10^{14} - 1.7 \times 10^{15}$  ions/ $\text{cm}^2$ .
- Microprobe imaging using static SIMS conditions  $5 \times 10^{-11}$  amps, 500  $\mu\text{m}$  raster, dose during acquisition  $\sim 2-7 \times 10^{12}$  ions/ $\text{cm}^2$ .
- Pre-sputter used as needed.

