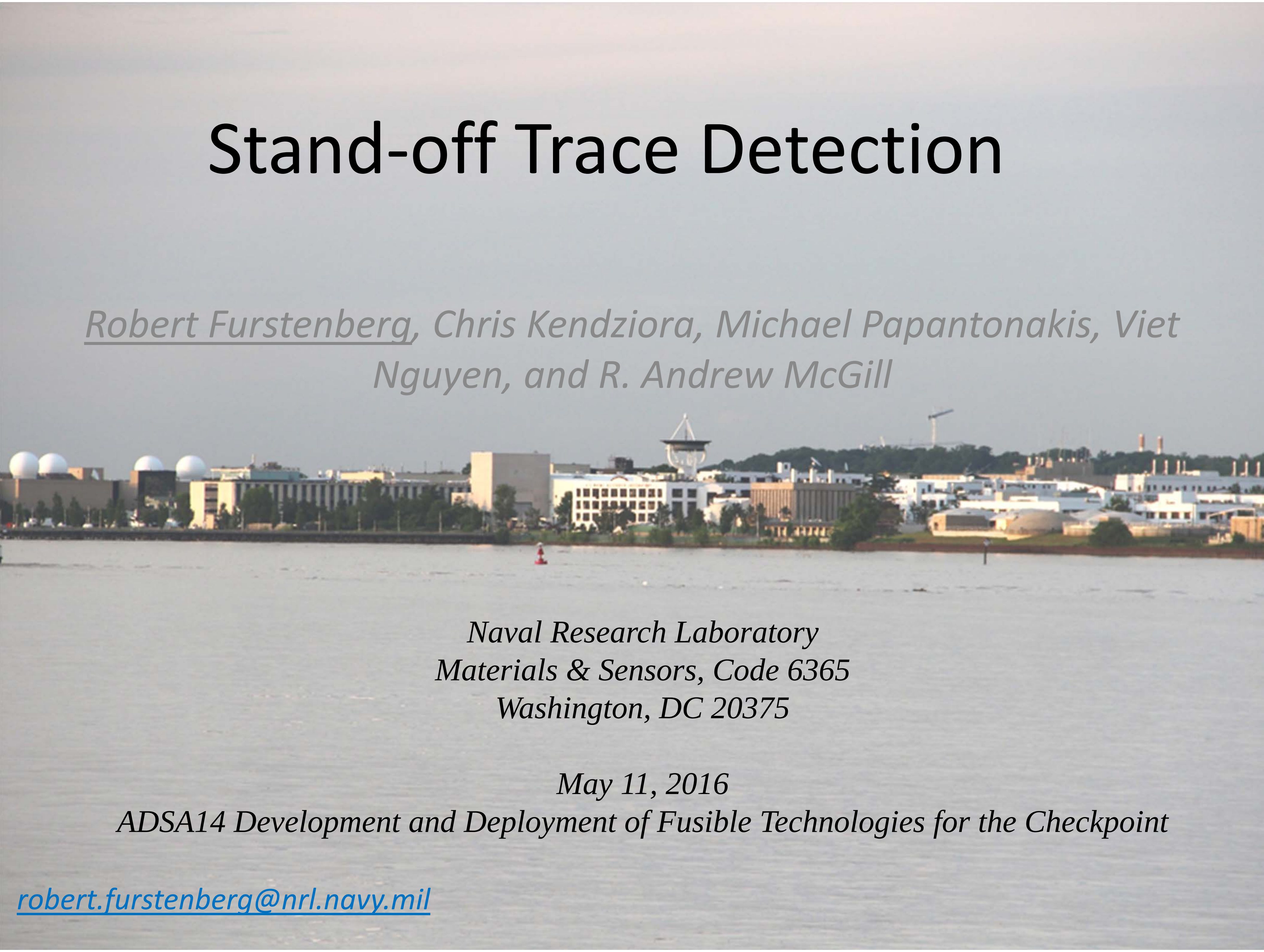


# Stand-off Trace Detection

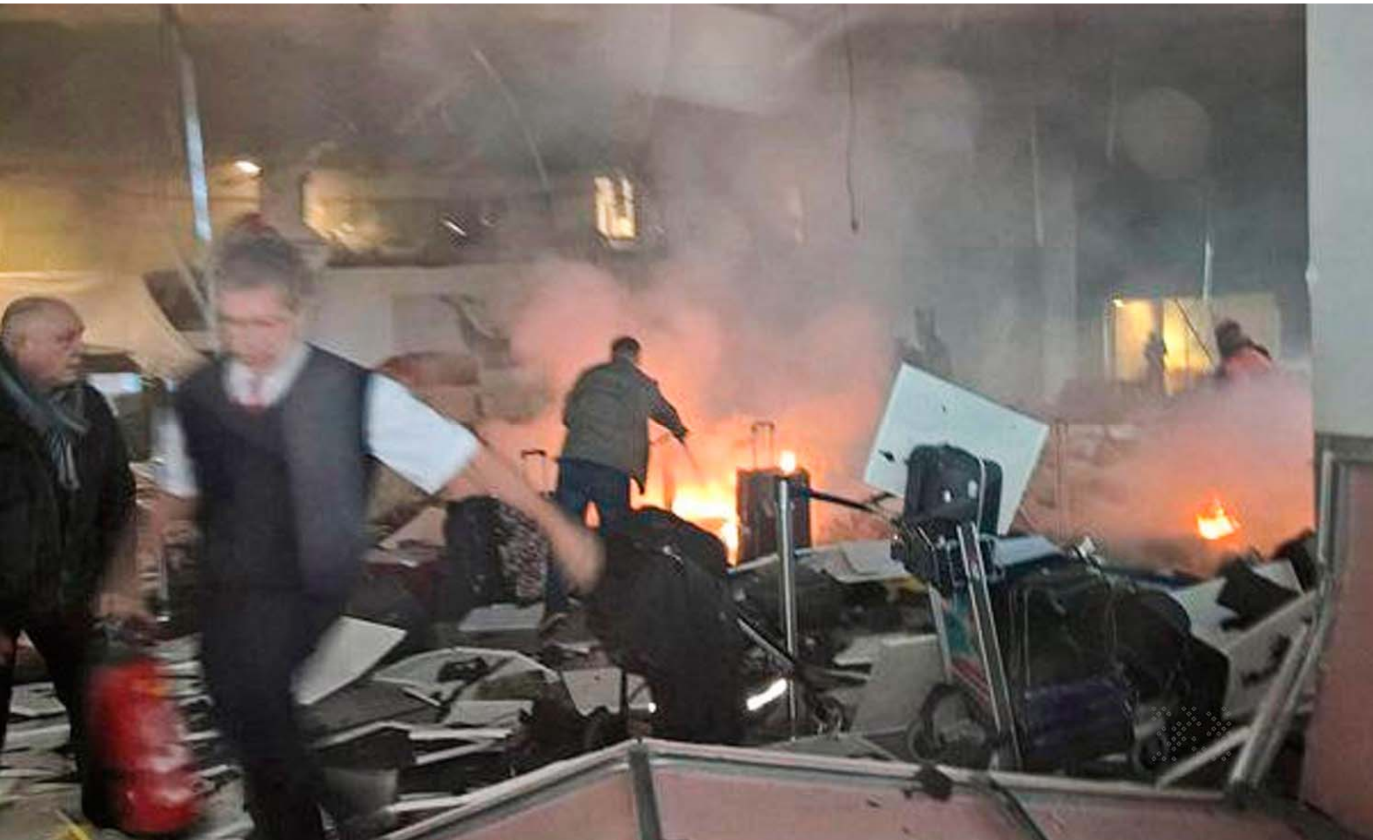
Robert Furstenberg, Chris Kendziora, Michael Papantonakis, Viet Nguyen, and R. Andrew McGill

*Naval Research Laboratory*
*Materials & Sensors, Code 6365*
*Washington, DC 20375*

*May 11, 2016*
*ADSA14 Development and Deployment of Fusible Technologies for the Checkpoint*

robert.furstenberg@nrl.navy.mil

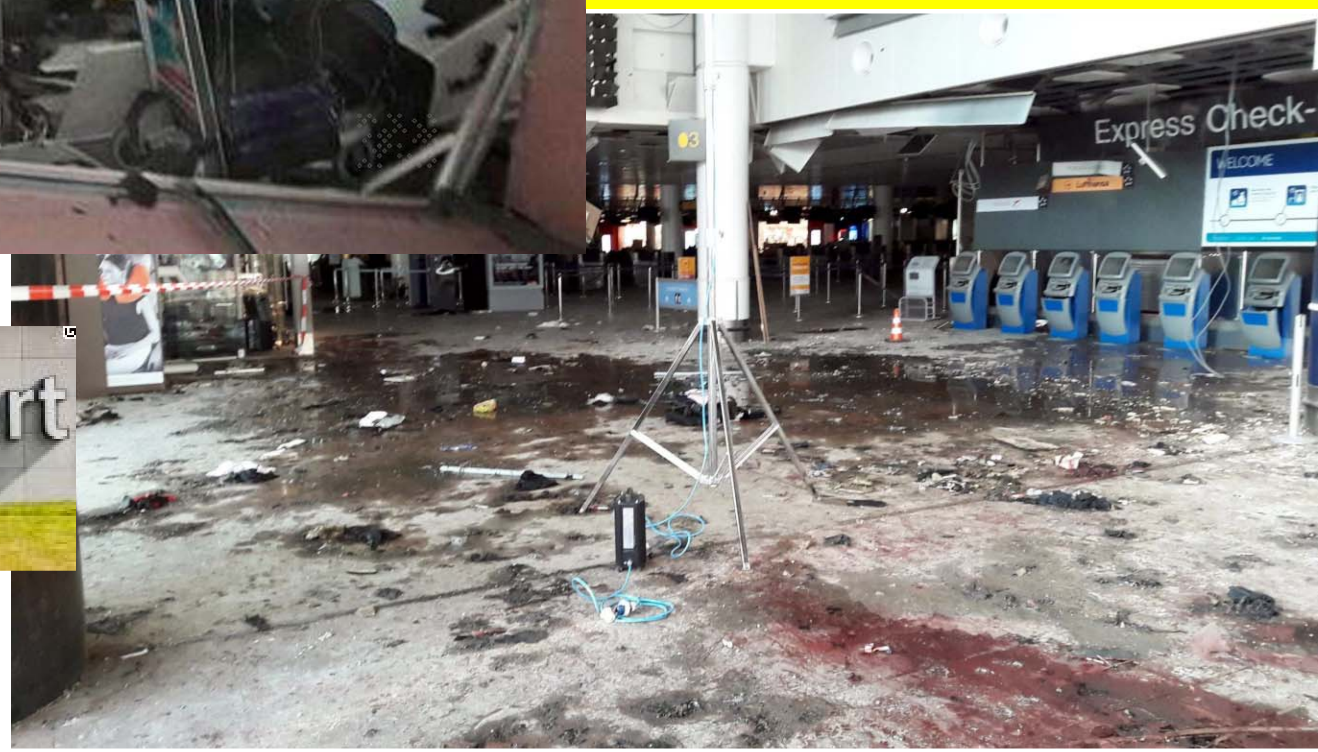
# Crowded Checkpoints = Attractive Soft Targets…



REDUCE CROWDING BY:

- Fast=no wait checkpoints
- Decentralized, multiple indoor/outdoor checkpoints using **stand-off detection**/surveillance

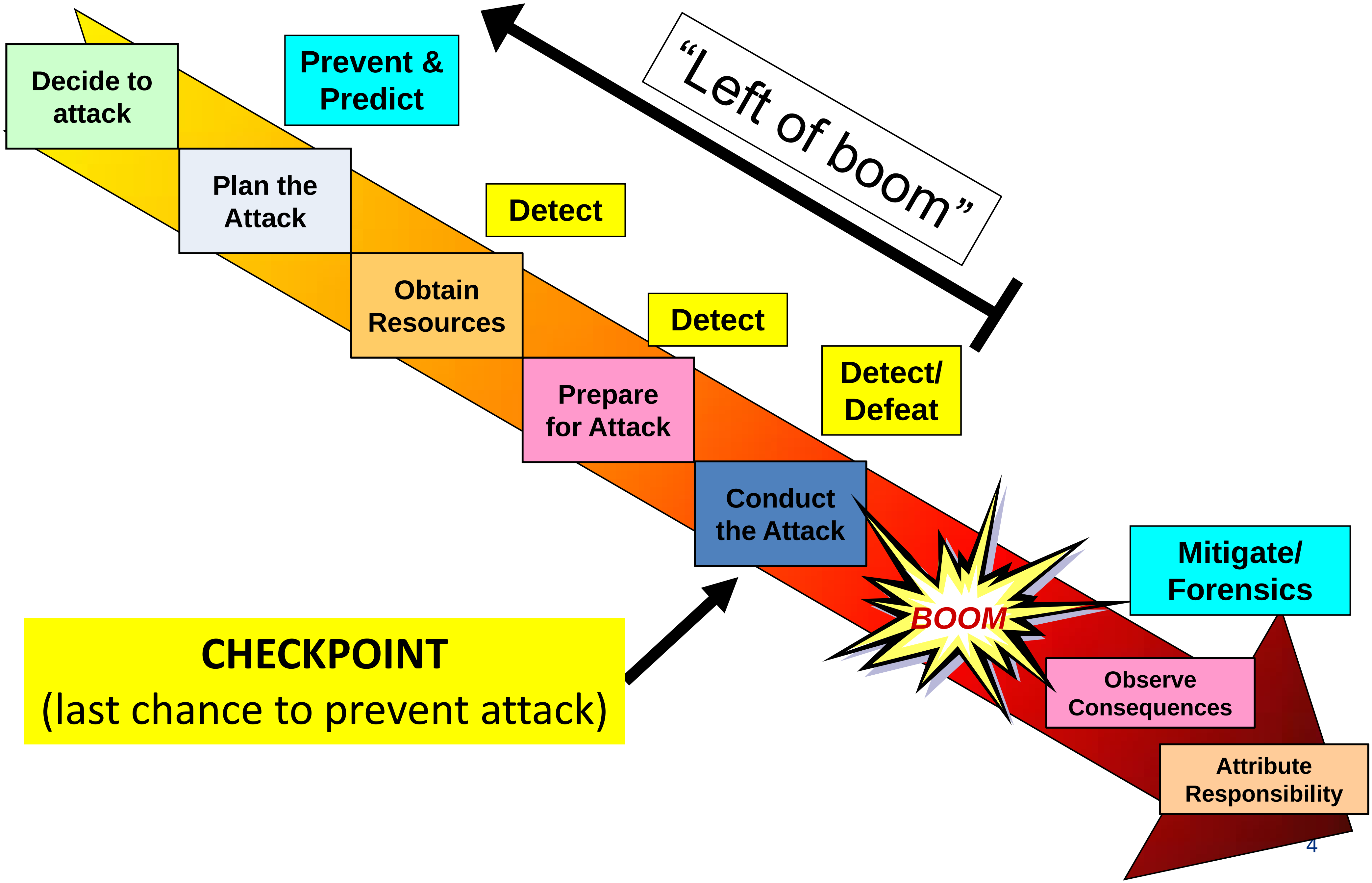
(Accept <100% scanned/chkpt!)




# Outline

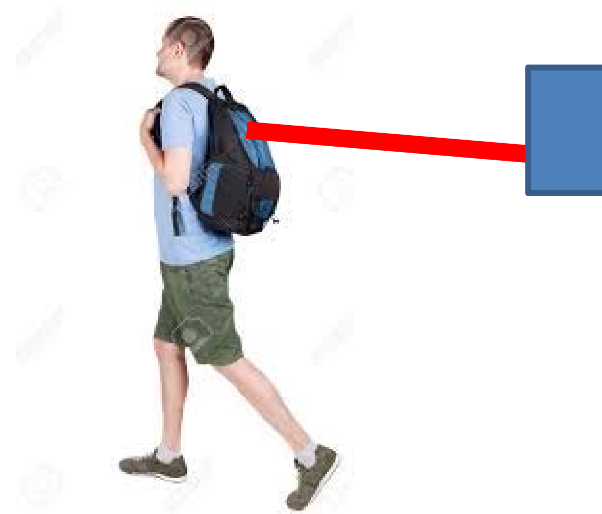
- **Motivation/Introduction**
- **Stand-off trace detection**
  - Photo-Thermal Infrared Imaging Spectroscopy
  - Laser Trace Vaporization (followed by IMS detection)
- **Realistic Trace Explosives Test Coupons**
  - Preparation
  - Analysis
  - Persistence studies
- **Conclusions**

# Exploit or attack the terrorist network



# “Left of boom” CONOPS

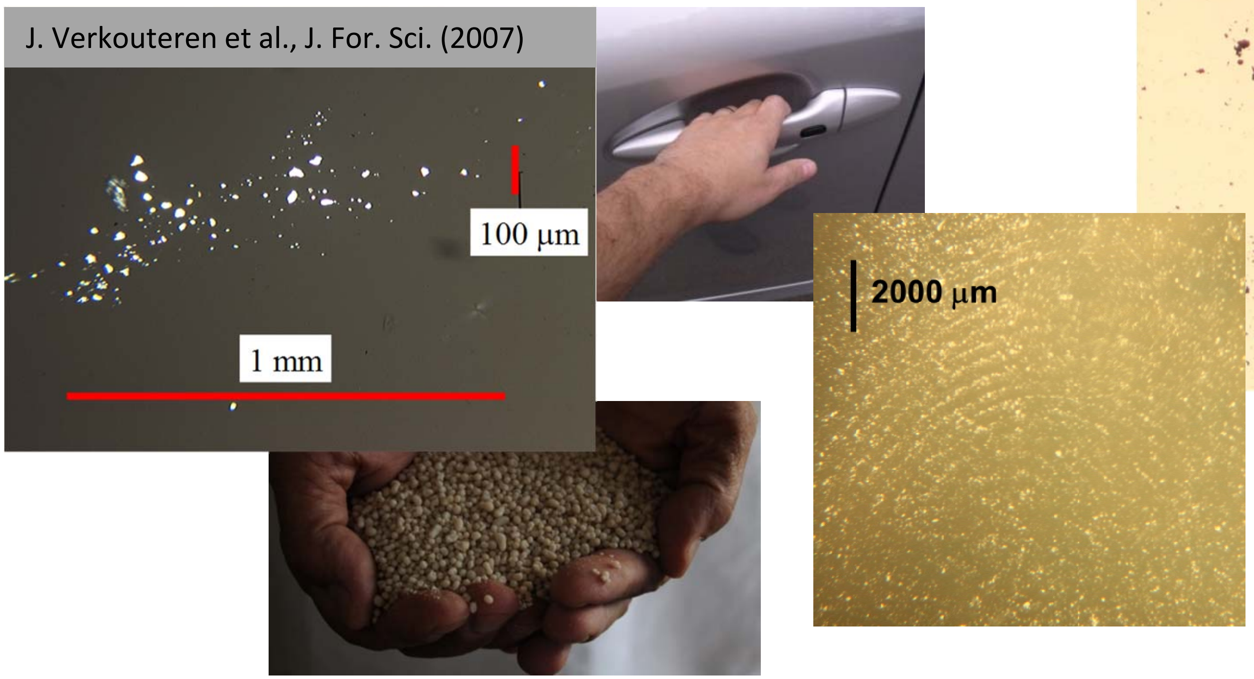
- Covert/Overt car screening
  - Probe car door handle at barrier
  - Determine presence of trace explosives
  - Probe inside trunk with handheld scanner

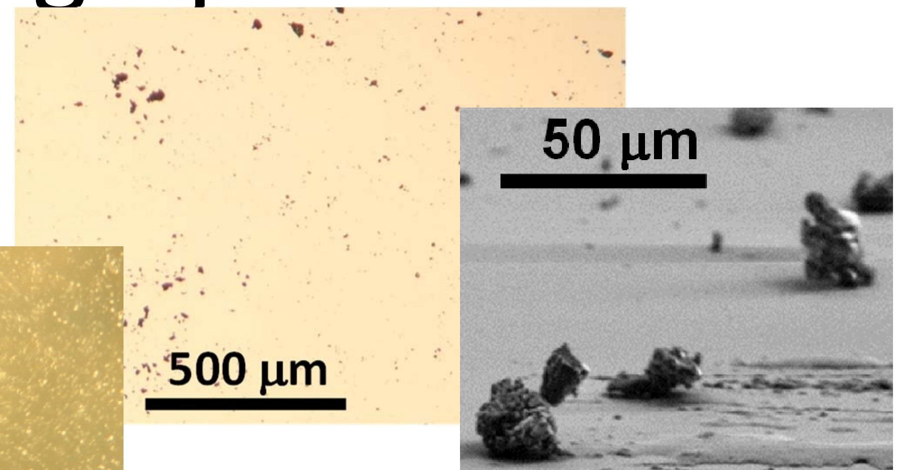



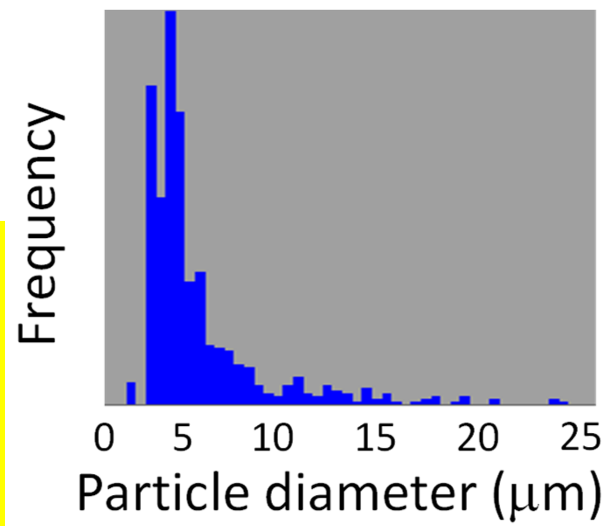
- Covert/Overt skin analysis
  - Determine presence of trace explosives

- Covert clothing/bags/shoes analysis
  - Determine presence of trace explosives
  - Uncooperative subjects (moving)

# Understand the source of trace explosives deposited in fingerprints




Fingerprint "ground truth":

- Mass coverage: ~ 1-10 μg/cm$^2$
- "Fill factor": ~ 0.05 - 1%
- Particle size: ~ 1-25 μm distribution
- Particle persistence hours, days, months
- Low vapor pressure => immeasurable vapor, can only detect solid particles!

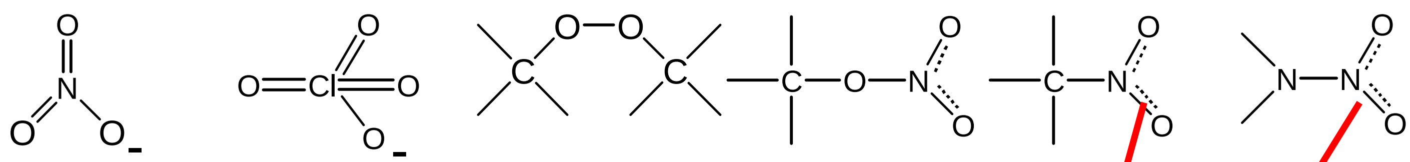
# Structural features of Explosives materials

## Principal chemical classes of explosives materials

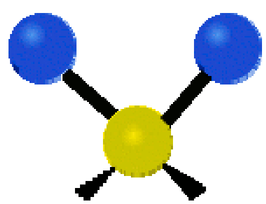
HME | Military

| Fuels | Nitrate (AN) | Perchlorate (KC) | Peroxide (TATP) | Nitrate Ester (PETN) | Nitro (TNT) | Nitramine (RDX) | Fillers |
|---|---|---|---|---|---|---|---|

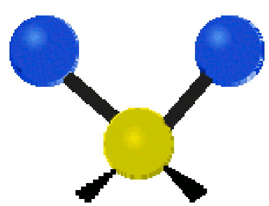
$C_nH_{2n+2}$
C(H)(OH)(O)
Al



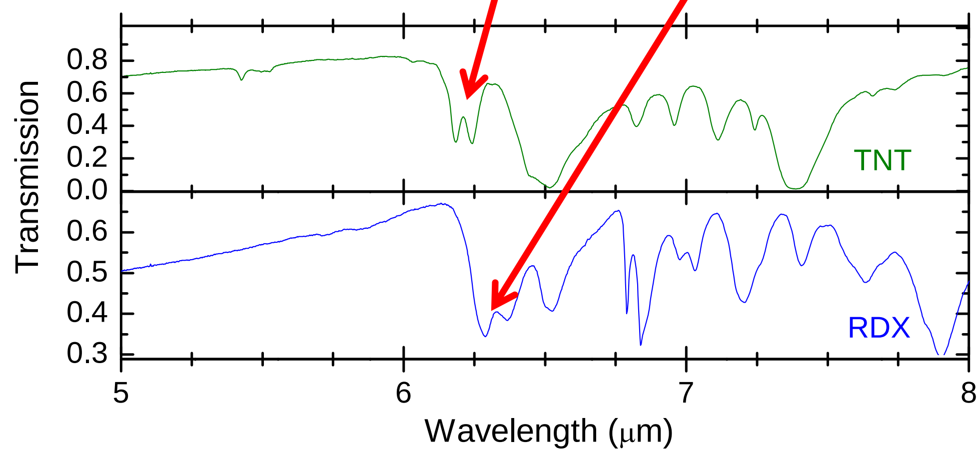
Ph-COOR
$-(CH_2C(CH_3)_2)n-$
Si-O-Si-OH



Symmetrical stretch



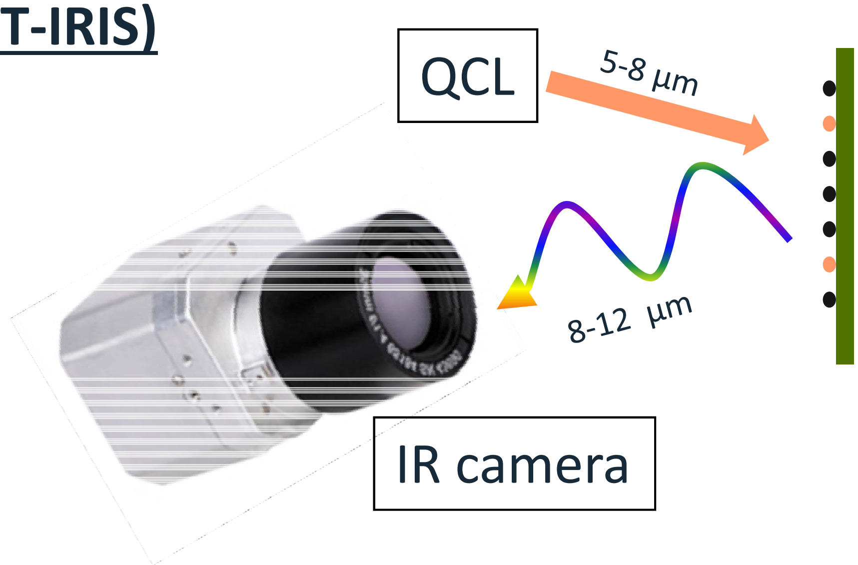
Anti-symmetrical stretch




**Common structural features targeted for IR detection**

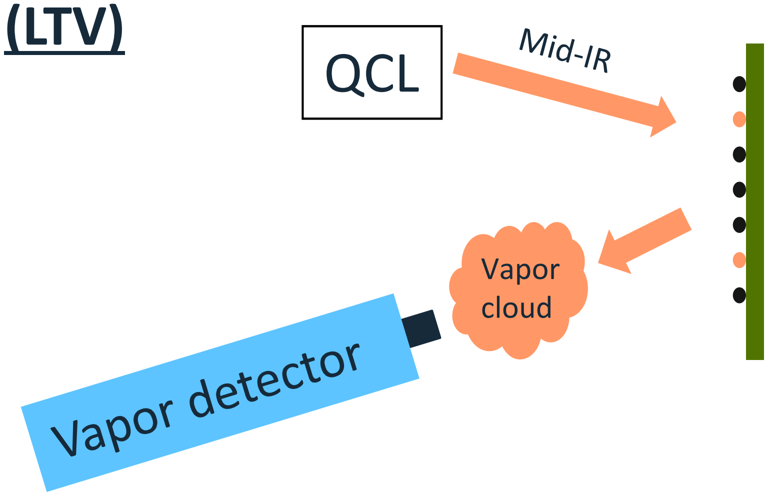
# Active infrared stand-off detection at NRL

## Photo-thermal IR Imaging Spectroscopy (PT-IRIS)




- Eye-safe IR light source
- Modest ~1°C “active” particle heating
- “Thermal” IR light emitted, collected and analyzed at a distance
- Successfully field tested a system; TRL 3-5
- Commercially available components; QCL, bolometer/MCT

## Laser Trace Vaporization (LTV)

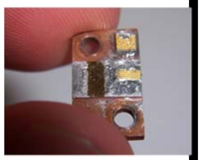


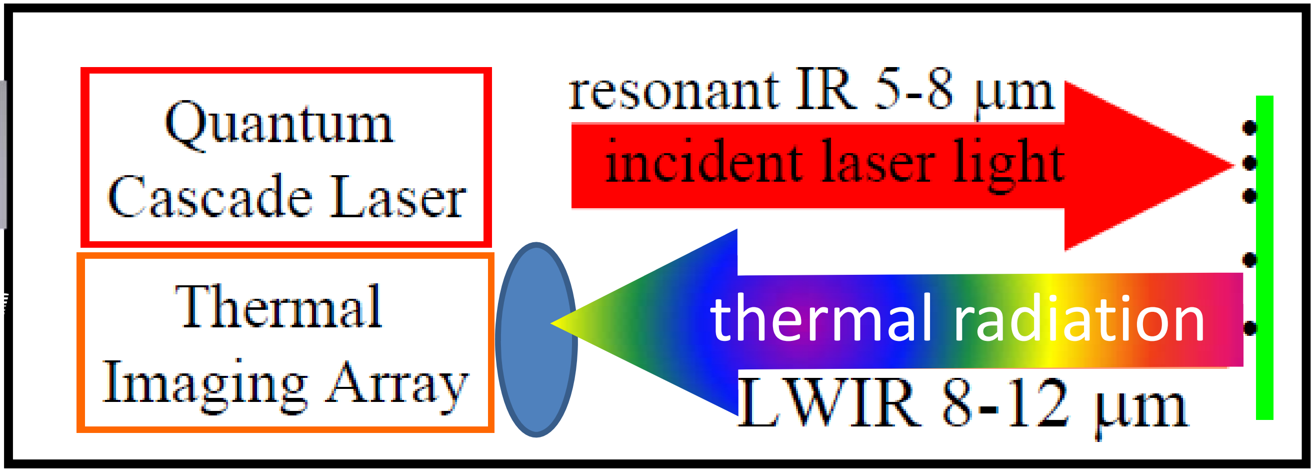

- Preferentially vaporization of materials of interest
- Higher laser powers than PT-IRIS (not eye-safe)
- Proximal interrogation of vapor (e.g. with IMS)

US Patents (NRL 6365): 8,101,915, 8,222604, 8,421,017, 8,421,018
Furstenberg, Kendziora et al, Applied Physics Letters, 93, 224103 (2008)
Furstenberg , Papantonakis et al., Proc. SPIE. 7665, 76650Q (2010)

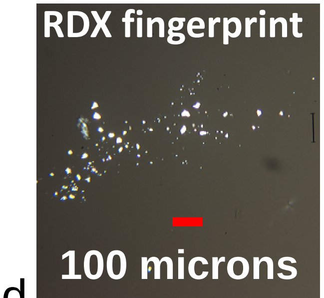
# Photo Thermal Infrared Imaging Spectroscopy (PT-IRIS)

# Photo-thermal Infrared Imaging Spectroscopy (PT-IRIS)






Surface contaminated particles of explosives



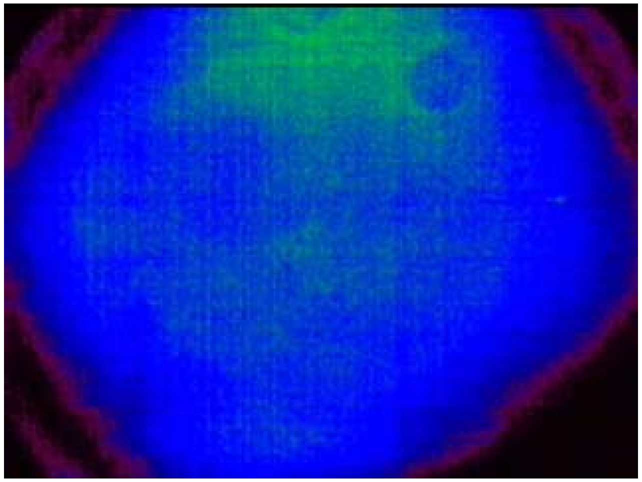

**PT-IRIS offers rich IR spectral features for detection using:**

1. Differential imaging
2. Wavelength selection (excitation)
3. Filtering wavelengths (collection)
4. Spatial contrast
5. Temporal kinetics

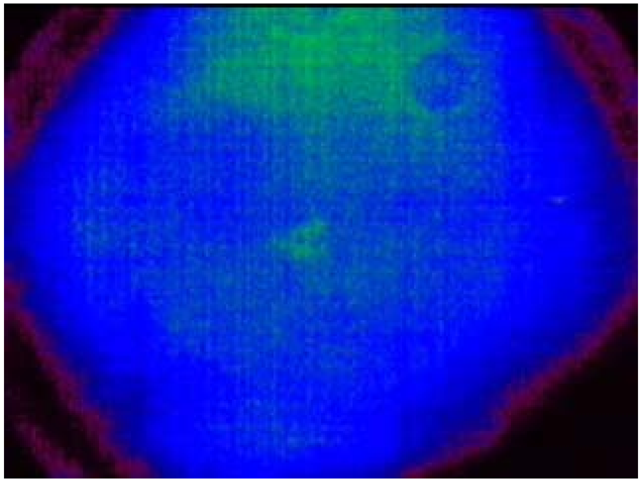
# (1) Differential imaging

1. Pulse or chop laser
2. “Laser Off” – “Laser On”

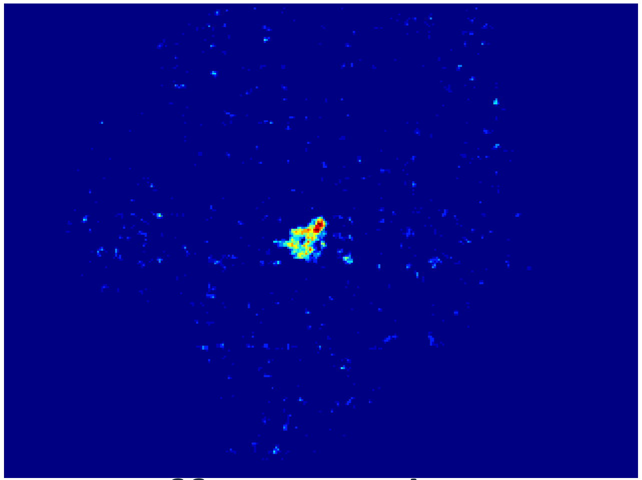
Thermal images of RDX on gold mirror illuminated with chopped QCL IR beam



Laser Off



Laser On

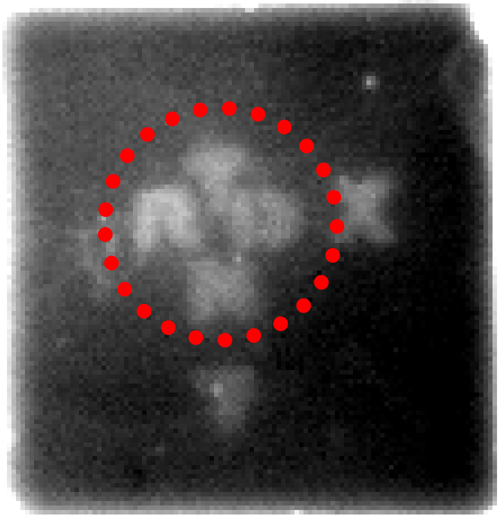


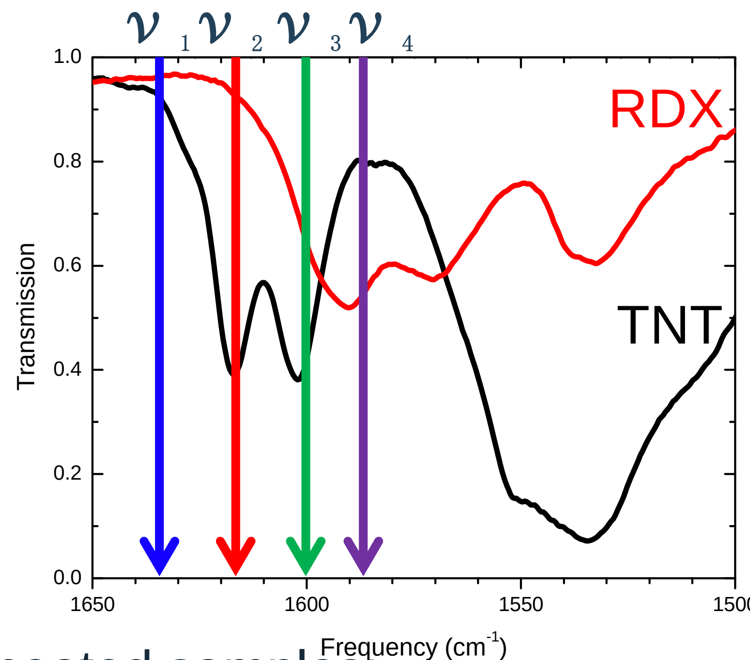
Differential

R. Furstenberg *et. al*, Applied Physics Letters, **93**, 224103 (2008)

# (2) Varying excitation λ; selectivity between explosives

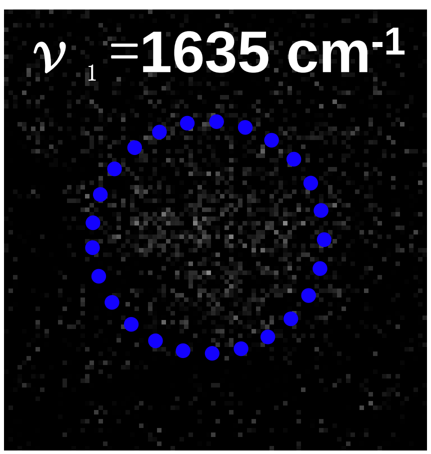
Sample of TNT and RDX illuminated by a heatgun: no laser



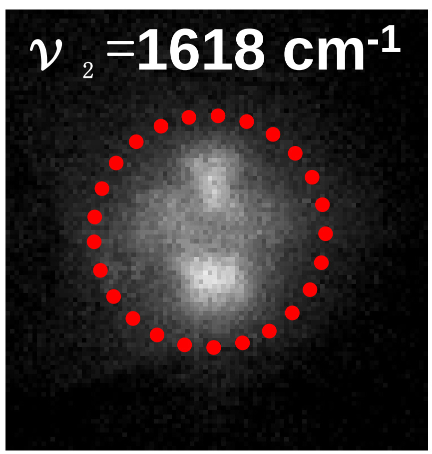



Differential images of QCL-heated samples:

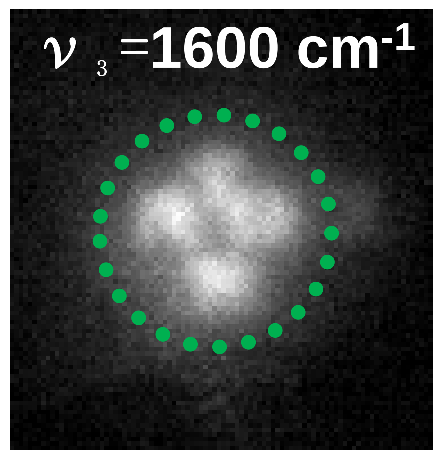
NOTE: Circles indicate the laser spot size.



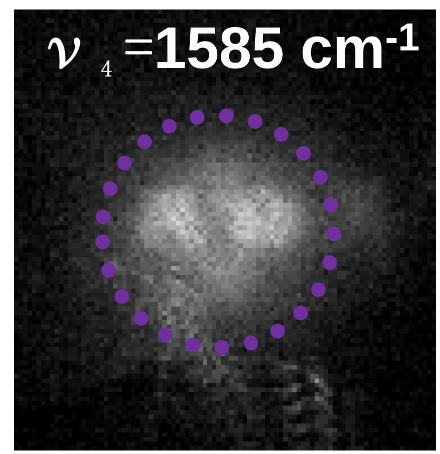

Off-resonance




TNT resonance

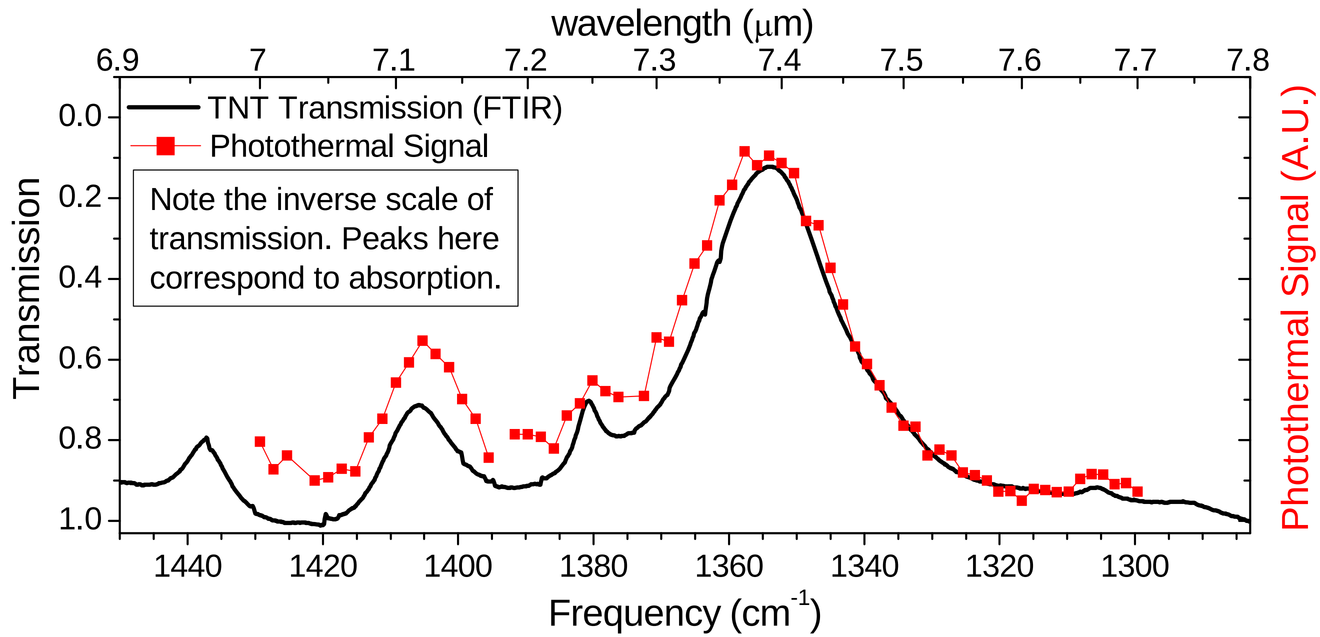



RDX & TNT




RDX resonance

# (2) Spectroscopy by varying excitation λ

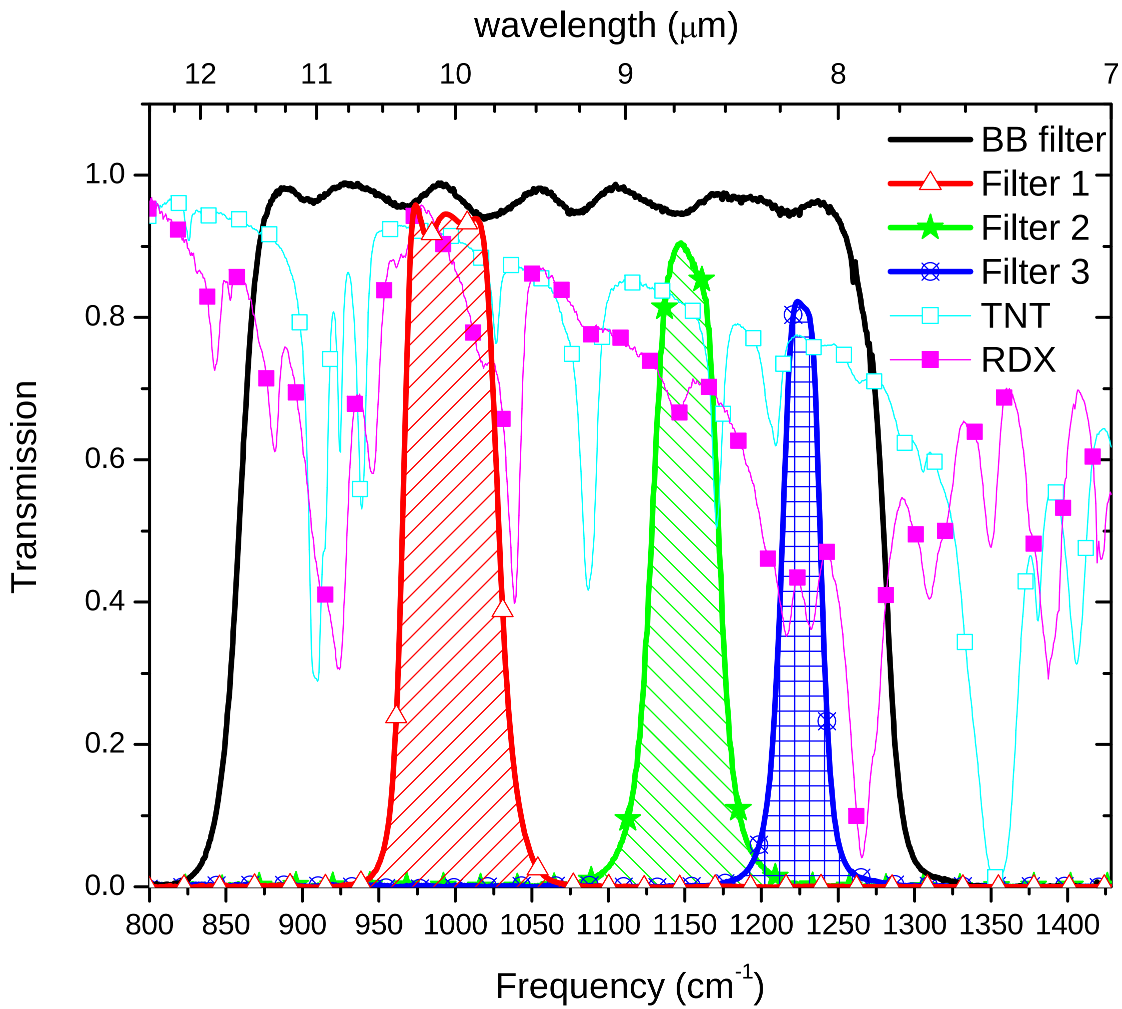



- More wavelengths improve selectivity, sensitivity
- Tunable QCL used here for PT signal from TNT
- 0.5 meter standoff, 30 mW, 7.0 – 7.7 mm (0.01 μm steps)
- Spectrum matches FTIR (all 5 absorption peaks)

PT-IRIS is effectively stand-off spectroscopy

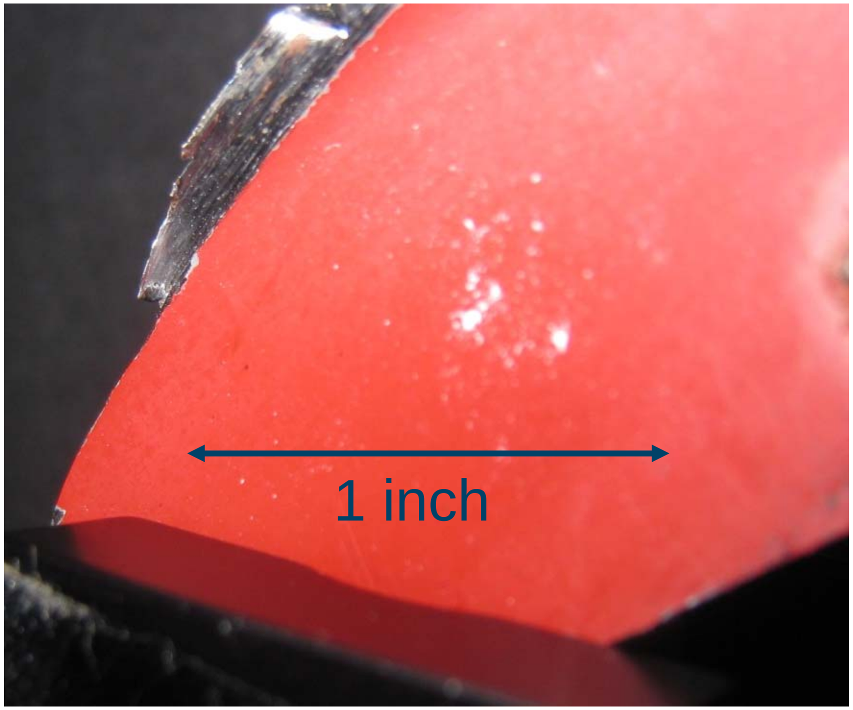
# (3) Signal enhancement by filtering on collected emission

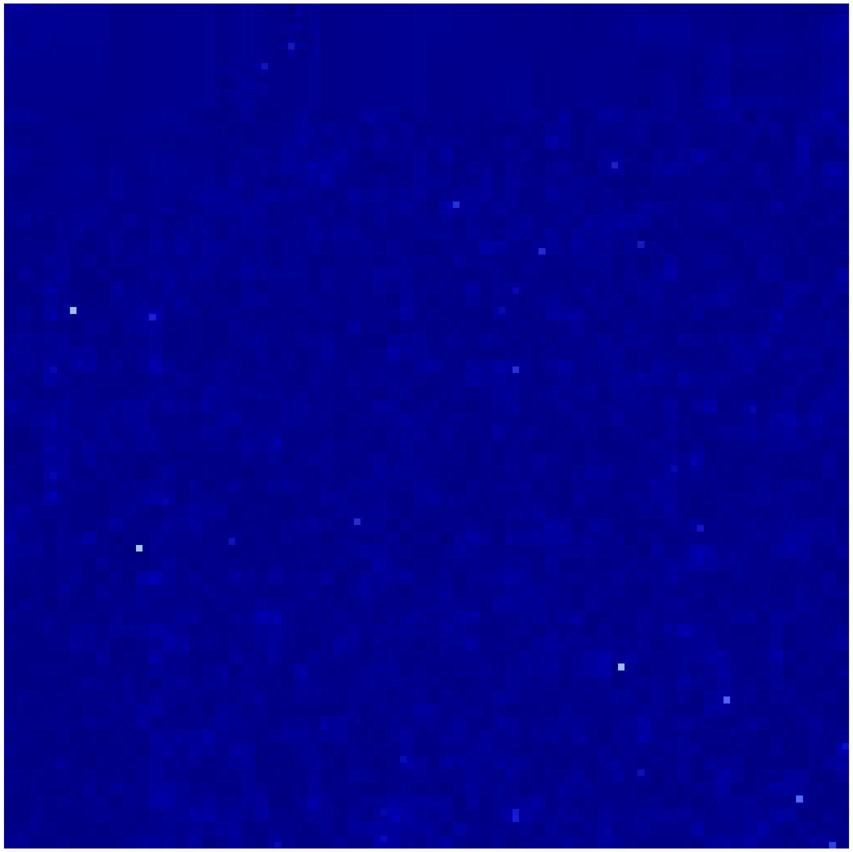
- Emission spectrum mirrors absorption spectrum by Kirchoff's Law
- Use collection LWIR filters to increase selectivity based on emissivity
- Filters which transmit or block the emission from different analytes
- These create a new analyte "signature" which can be compared with other known and unknown materials.
- These also help distinguish analytes from the substrate



# (4) Exploiting variations in spatial response

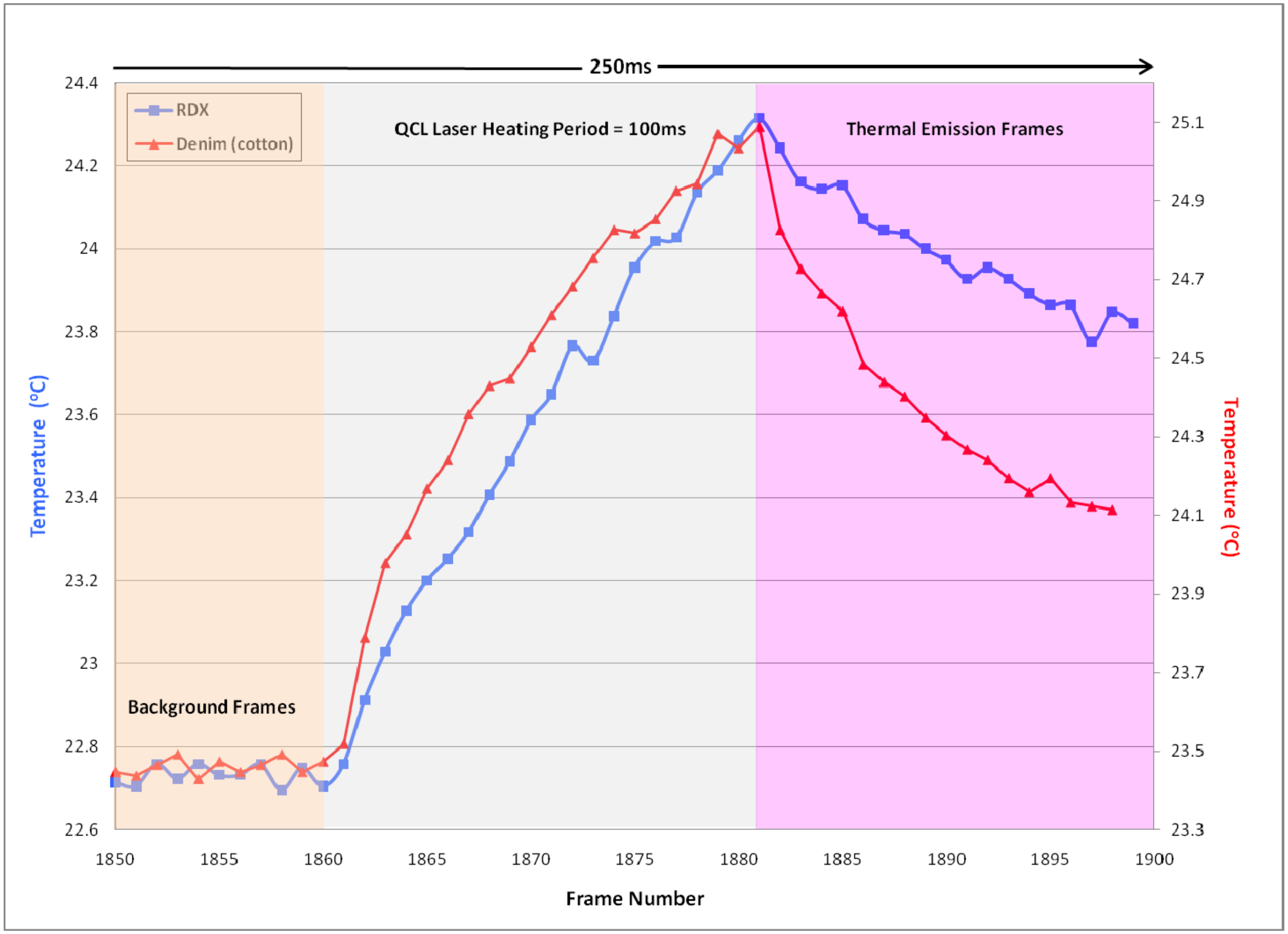
- RDX on a painted car panel
- 6.29 µm laser, 10 mm diameter beam, ~40 mW
- Laser manually directed around car panel



# (5) Signal contrast using temporal kinetics

- RDX on denim (cotton)
- Comparing temperature from pixels known to contain either RDX or denim
- Denim heats and cools faster than RDX
- Can identify RDX in the presence of denim




*Kendziora, MRS Fall 2011 Proceedings*

*Data taken with R. Jones; ITT*

# PT-IRIS summary

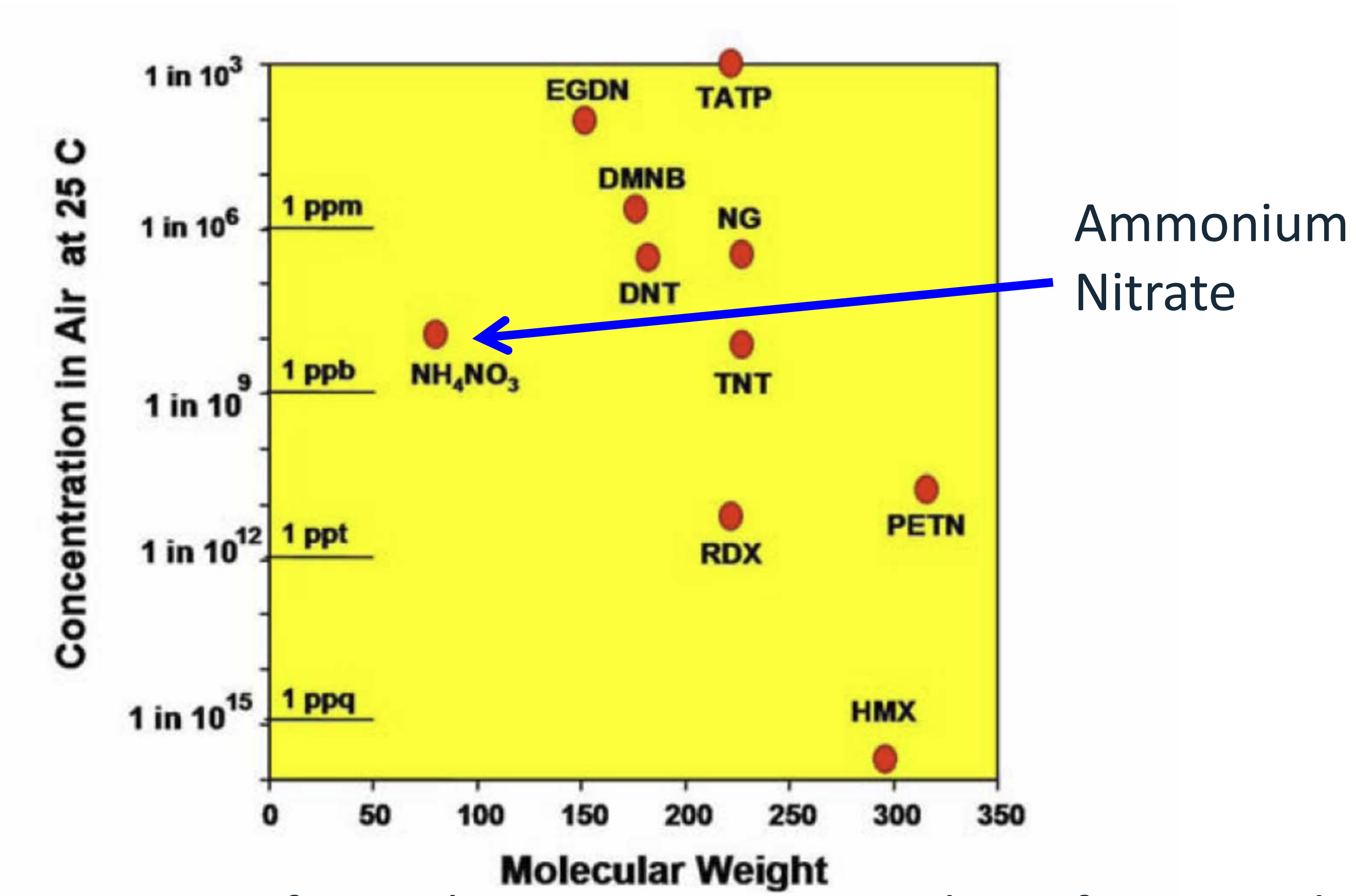
- **Eye-safe** IR lasers used to selectively heat trace amounts of explosives for photo-thermal imaging.
- PT-IRIS concept has been demonstrated on variety of substrates, analytes, in/out doors, and at significant standoff distances.
- Real-time, standoff, and imaging capabilities
- Chemical identification
- Potential covert operation
- Small QCL footprint allows integration with other capabilities

# Laser Trace Vaporization

# The Problem

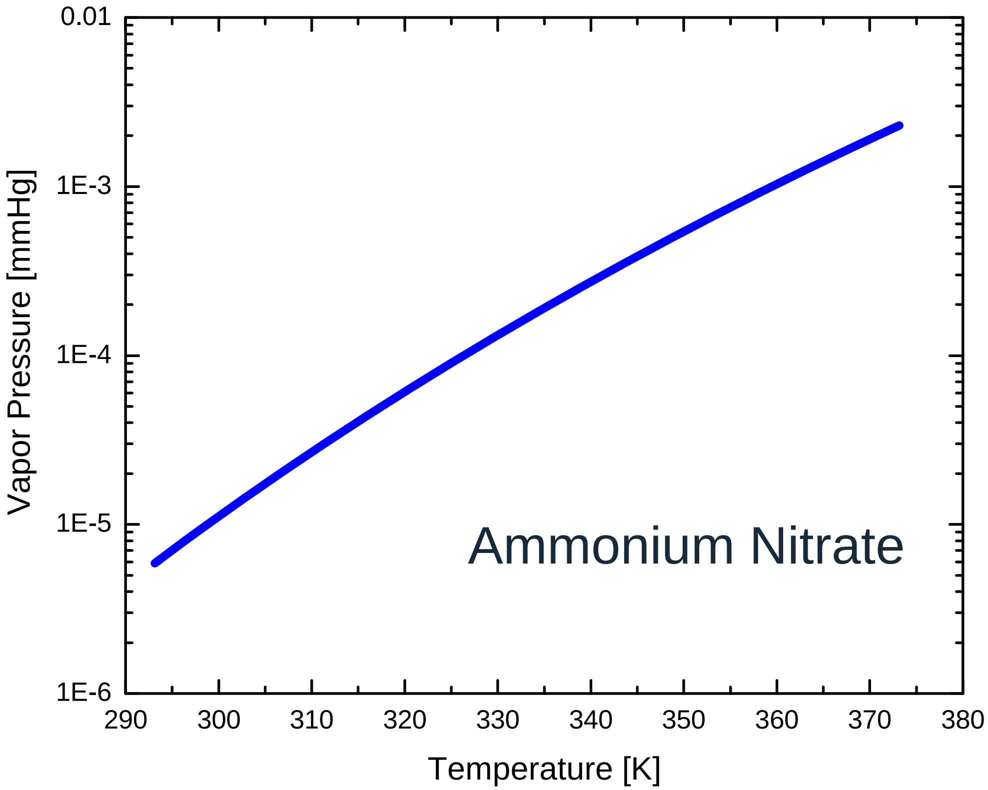
- Many explosives offer insufficient vapor for non-contact trace detection.
- Thus, despite the availability of mature vapor detectors, swabbing is primarily used.
  - surface particle swab process
  - air jets used to dislodge particles (portal)
- Current particle harvesting techniques:
  - time consuming
  - non-selective
  - surface contact method
  - high power consumption
  - high maintenance

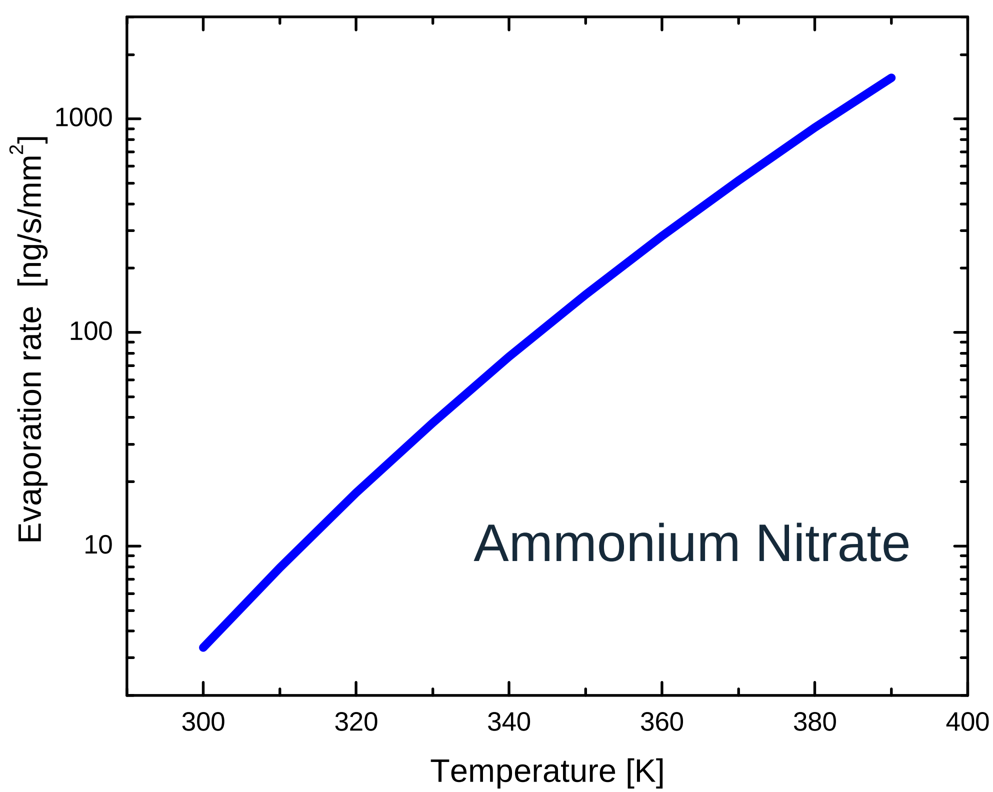
# Vapor Pressure of Explosives




- The vapor pressure range for explosives spans > 12 orders of magnitude
- Low vapor pressure means:
  - Particles persist on surfaces for long times
  - Very little vapor exists for detection!
- Vapor signature is exponentially stronger at elevated temperatures!

# Vapor Pressure vs. Temperature



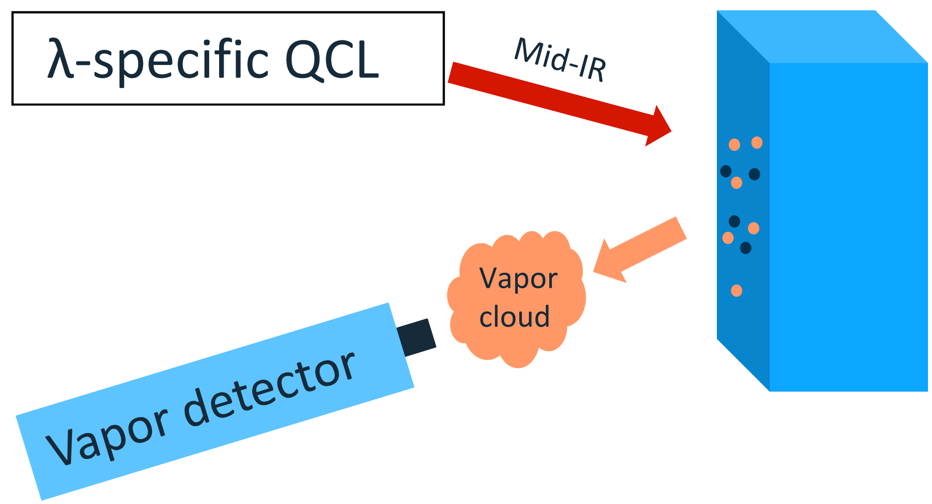




**Small increase in temperature = LARGE increase in evaporation!**

We need to heat explosives particles.
<u>But</u>, we shouldn't heat anything else on the surface!

# Laser Trace Vaporization (LTV)

- Many explosives have low vapor pressures
- Vapor pressure, and evaporation rate,  have an exponential dependence on temperature
- We used an Ion Mobility Spectrometer (IMS) to validate signature enhancement



# LTV: Prompt vaporization of explosives (RDX)

LTV video of RDX on polycarbonate

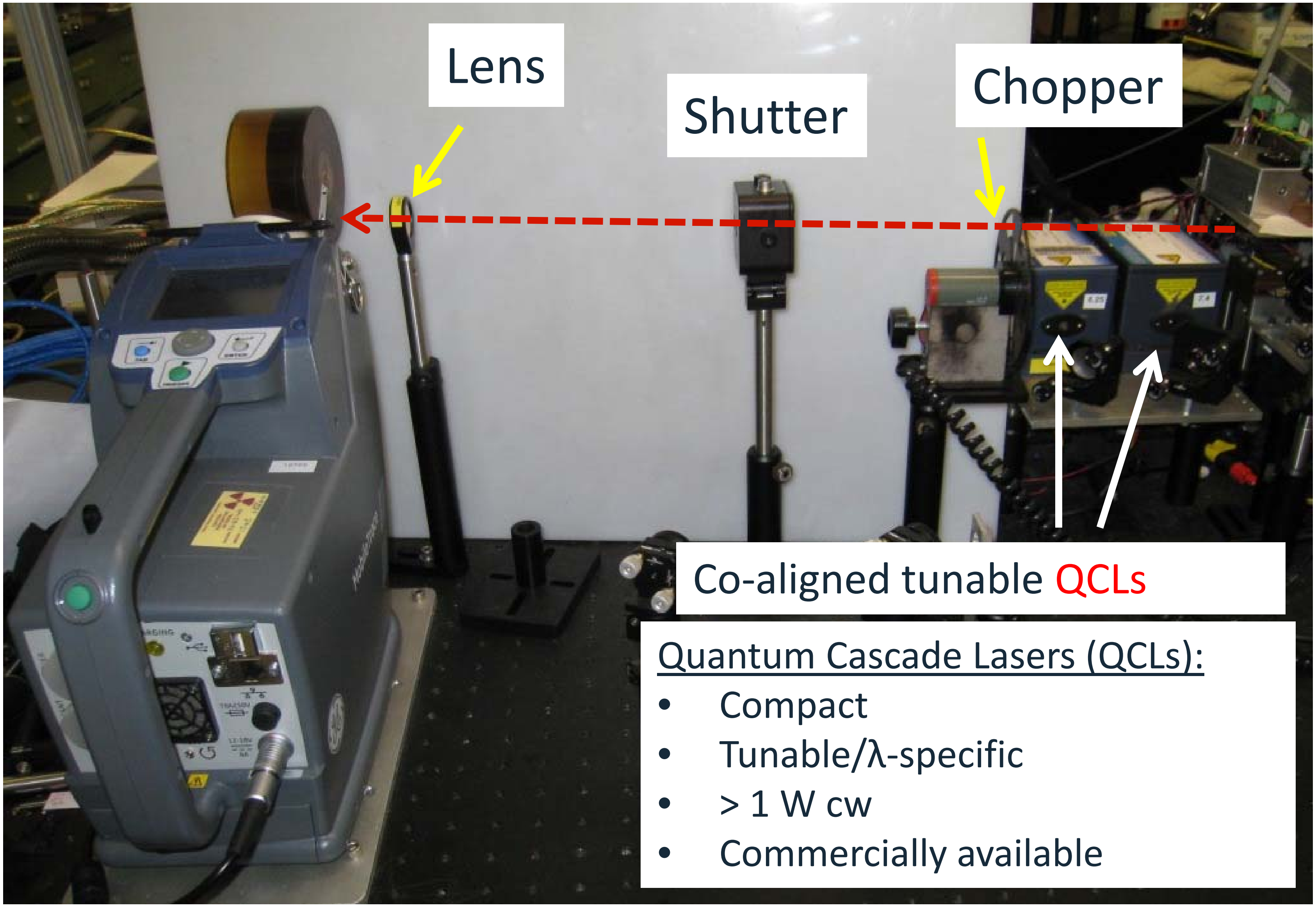
White = RDX particles

Black = Laser cleared

159 mW, focused
600 μm line width
Cleared area is 2 x 5 mm
Real time video

Sub-micron RDX particles spray-coated on polycarbonate

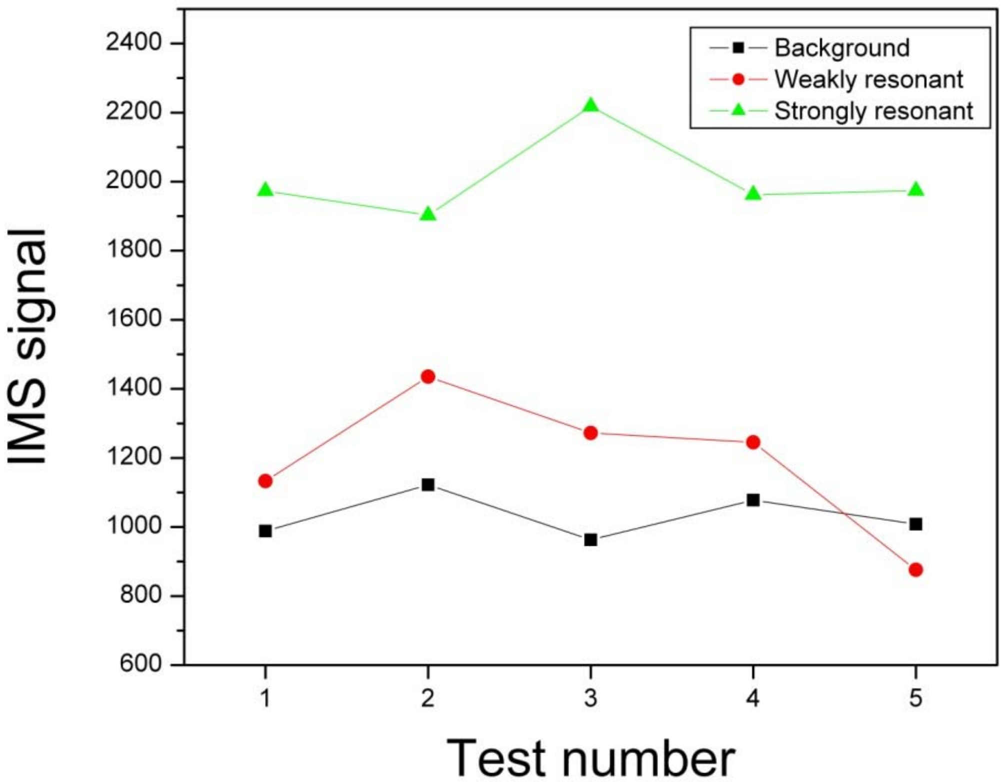
# LTV test bed

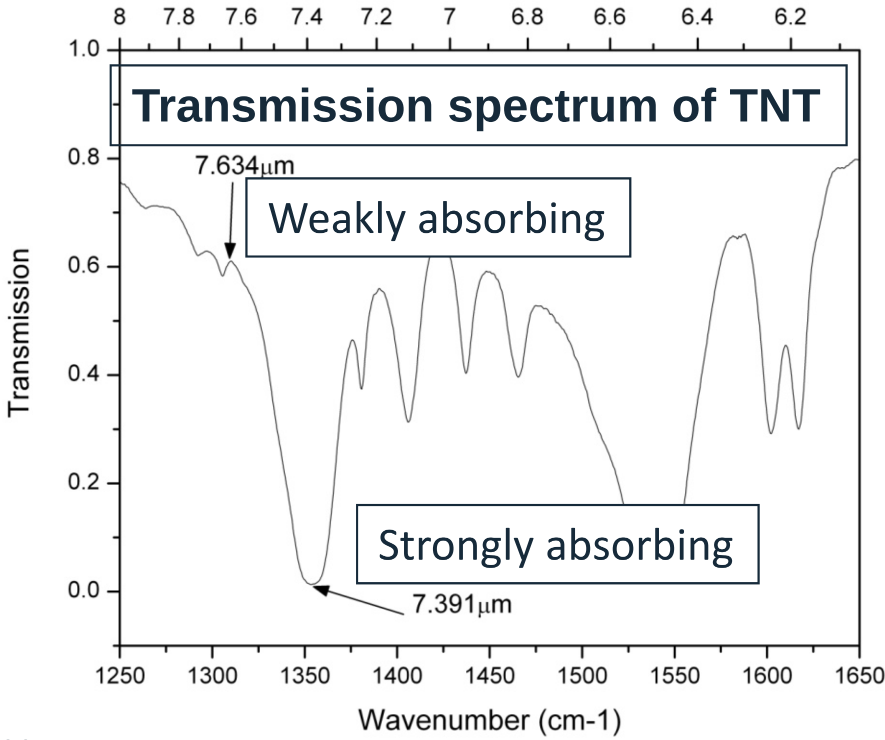



Quantum Cascade Lasers (QCLs):

- Compact
- Tunable/λ-specific
- > 1 W cw
- Commercially available

# Wavelength selectivity of TNT on PE

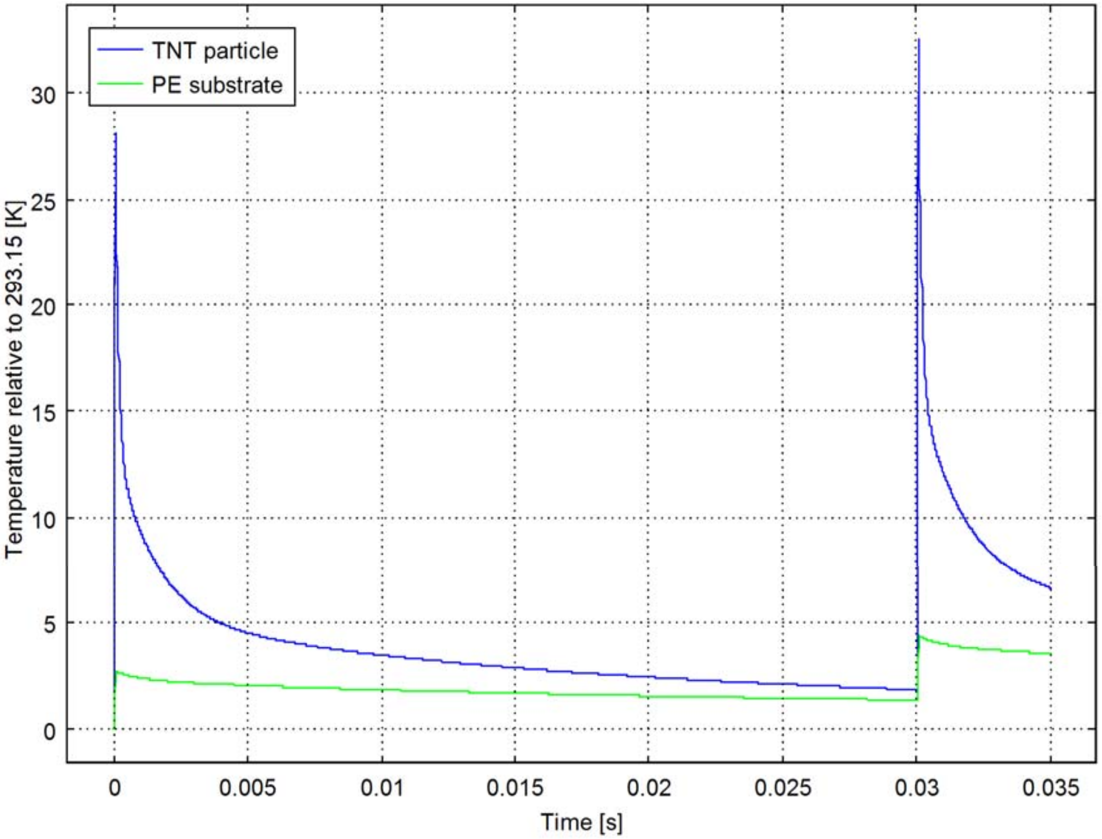


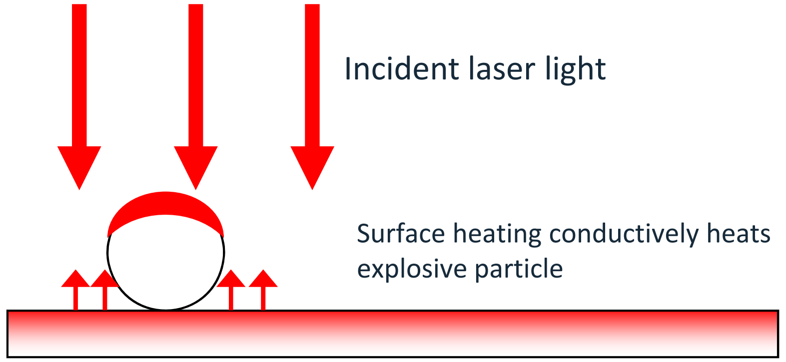




130 mW
4.5 mm beam spot (unfocused)
1 µg TNT

On the IR transparent substrate, enhancement only from resonant wavelength

# Comparing heating of surfaces and particles




Due to surface heating of substrate

- ➢ All laser energy absorbed by substrate (heated) at either λ – "optically thick"
- ➢ Surface absorption important for longer heating times

**Shorter pulses provide better thermal contrast**

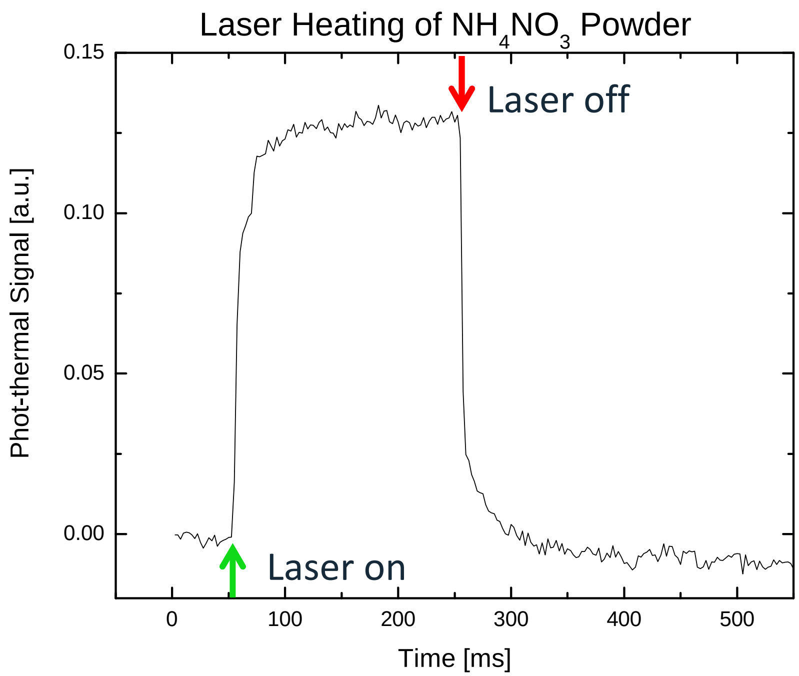
Input:

- particle diameter: $d_{particle} = 2\ R_{particle} = 69\ \mu m$
- Laser Power: 100mW; 100 μm x 100 μm spot
- Assumed absorption lengths:
  - in TNT: $\delta_{absorption} = 1\ \mu m$
  - in the substrate: 100 μm
- 50 μs laser pulse

Simulation by J. Großer (German ESEP)

# Measurement of thermal kinetics

- High speed MCT infrared imaging array
- Measured laser heating of AN particles
- 50-80 micron AN particles, 200 ms laser pulse
- Time scale agrees with modeling



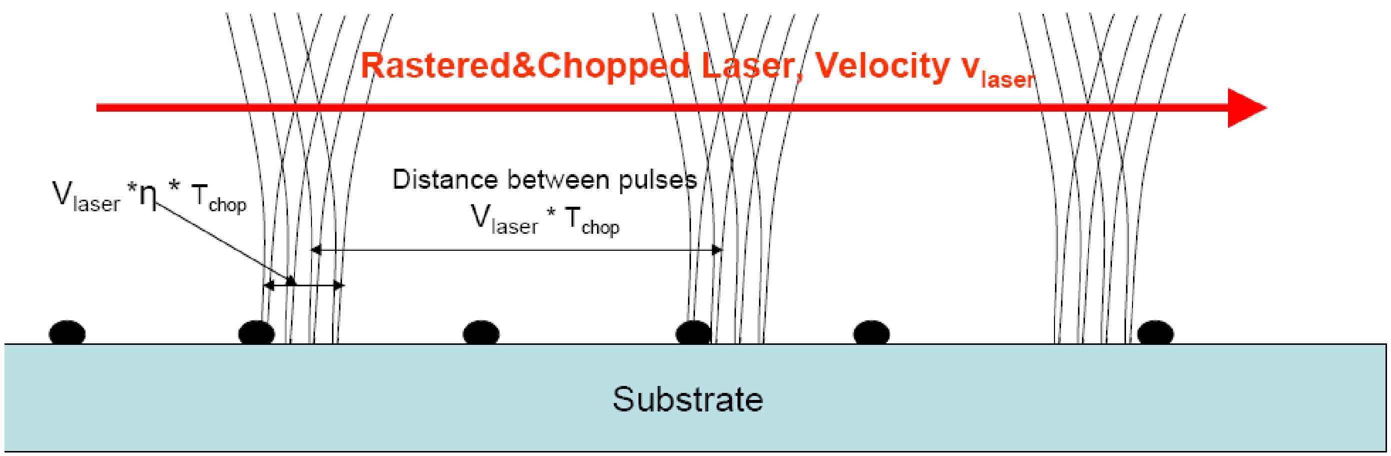

- Reaches thermal equilibrium in ~ 50 ms

  $\Rightarrow$ Again, use shorter laser pulses!!!

# Strategies to improve LTV selectivity

- Can optimize the laser pulsing with a better understanding of substrate/particle properties
- Pulse/chop laser
- Raster beam

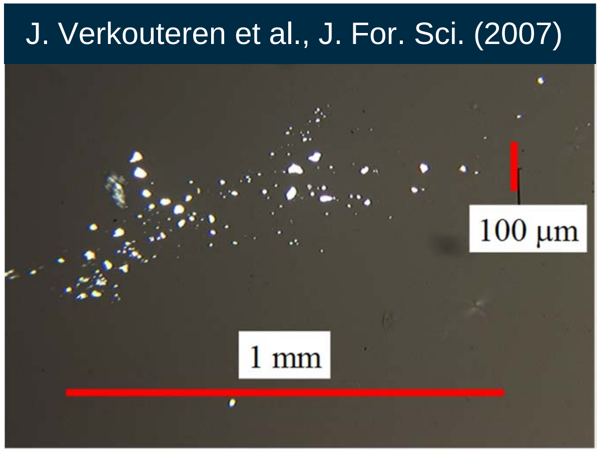
➢ Limit heating of substrate/interferent while providing material vaporization



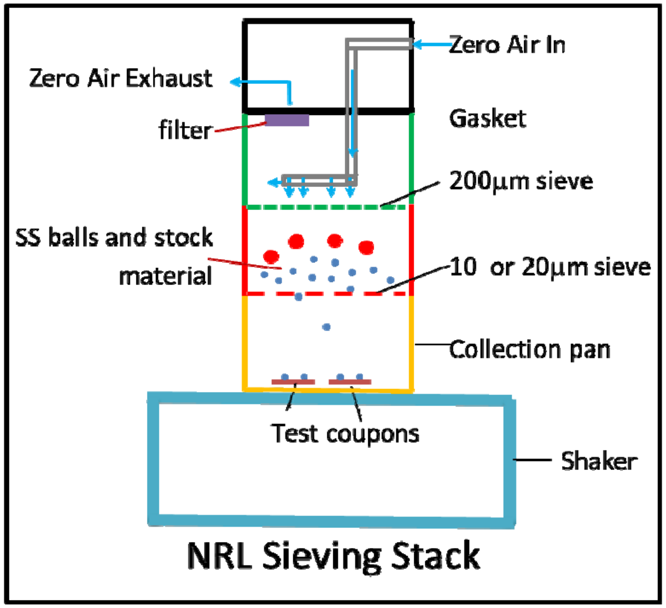
# LTV Summary

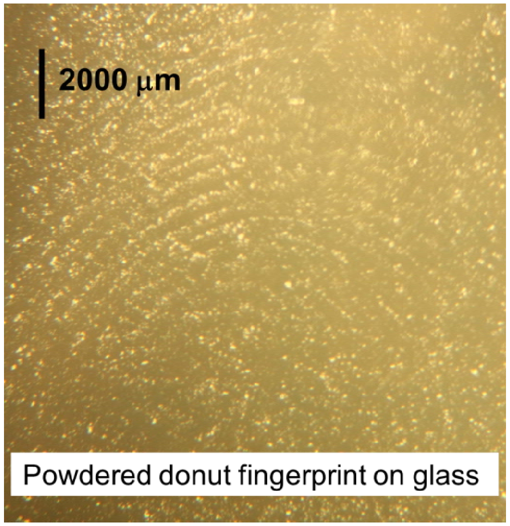
- LTV can provide the following detection advantages for low vapor pressure materials (AN, TNT, RDX)
  - Adds sensitivity for detection of low vapor pressure analytes
  - Selective vaporization from a contaminated surface
  - Faster response time
  - Non-contact or standoff
- Small footprint of QCLs enables interfacing with hand-held vapor detectors or integration into existing platforms
- LTV can easily be adapted for other materials of interest

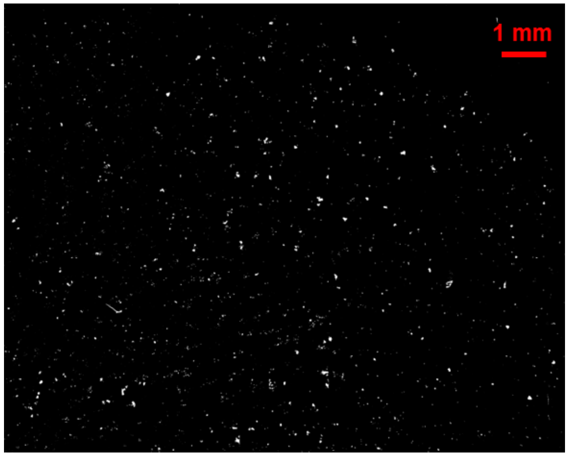
# Realistic Test Coupon Preparation







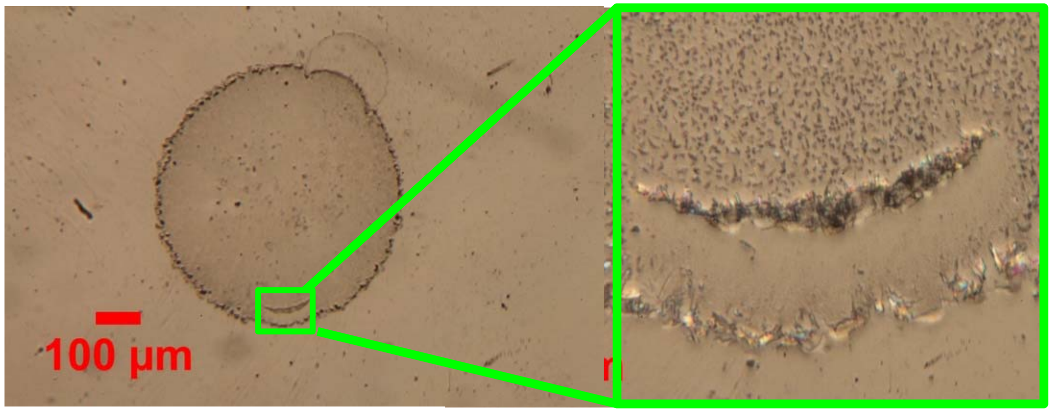



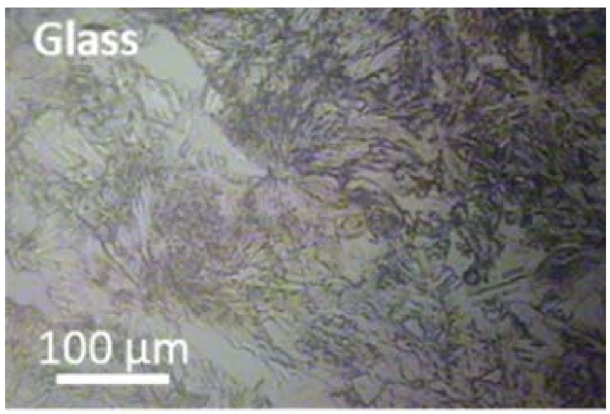

Any detection technique should be tested against realistic (fingerprint-like) targets!

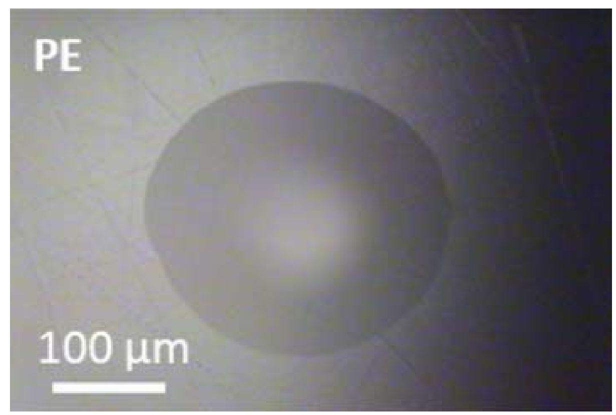
# Inkjetting

RDX
on glass




TNT
on glass



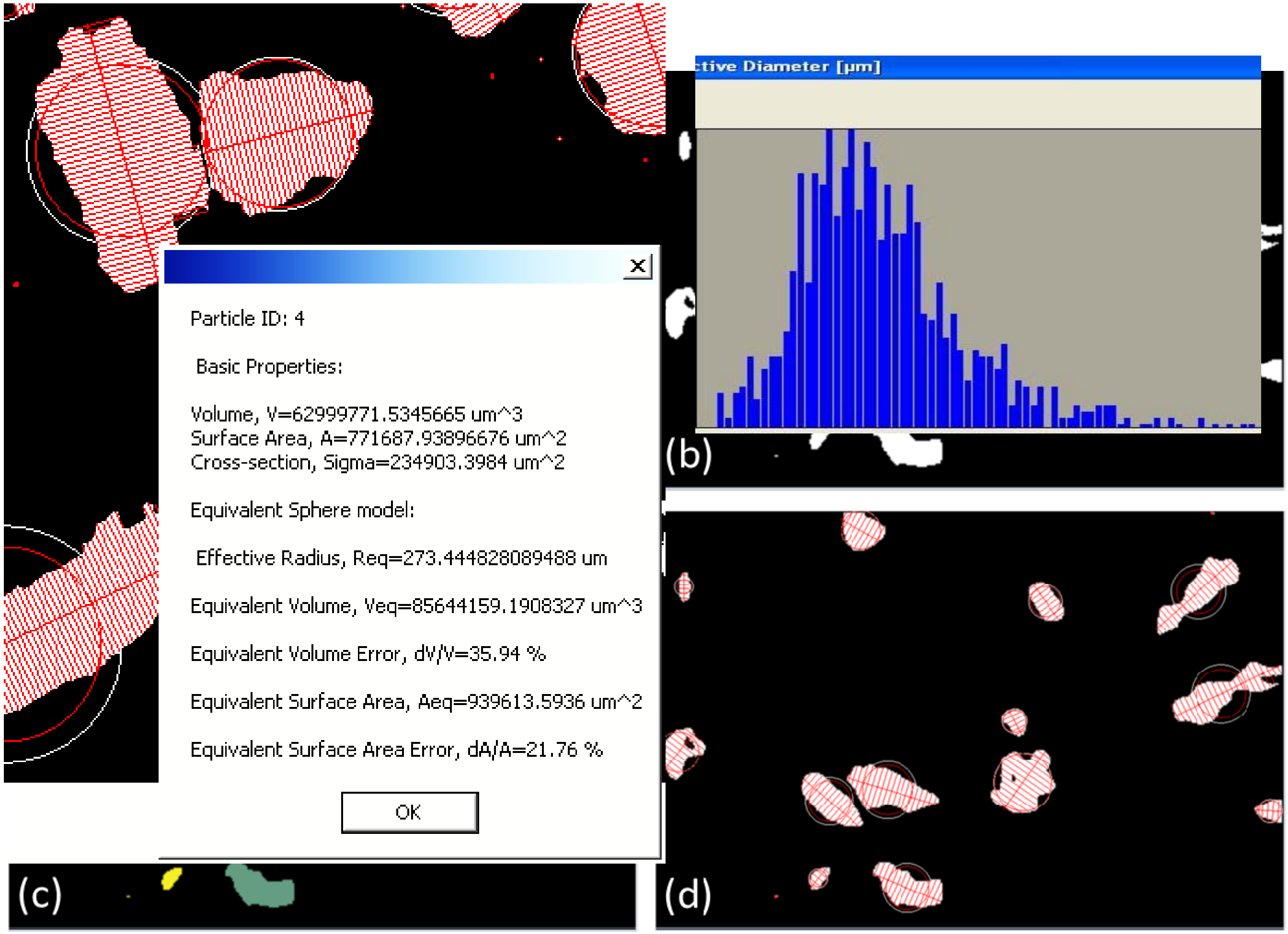

Sucrose
on PE

- Analyte distribution is controlled by solvent wetting effects and can produce a variety of structures after drying.

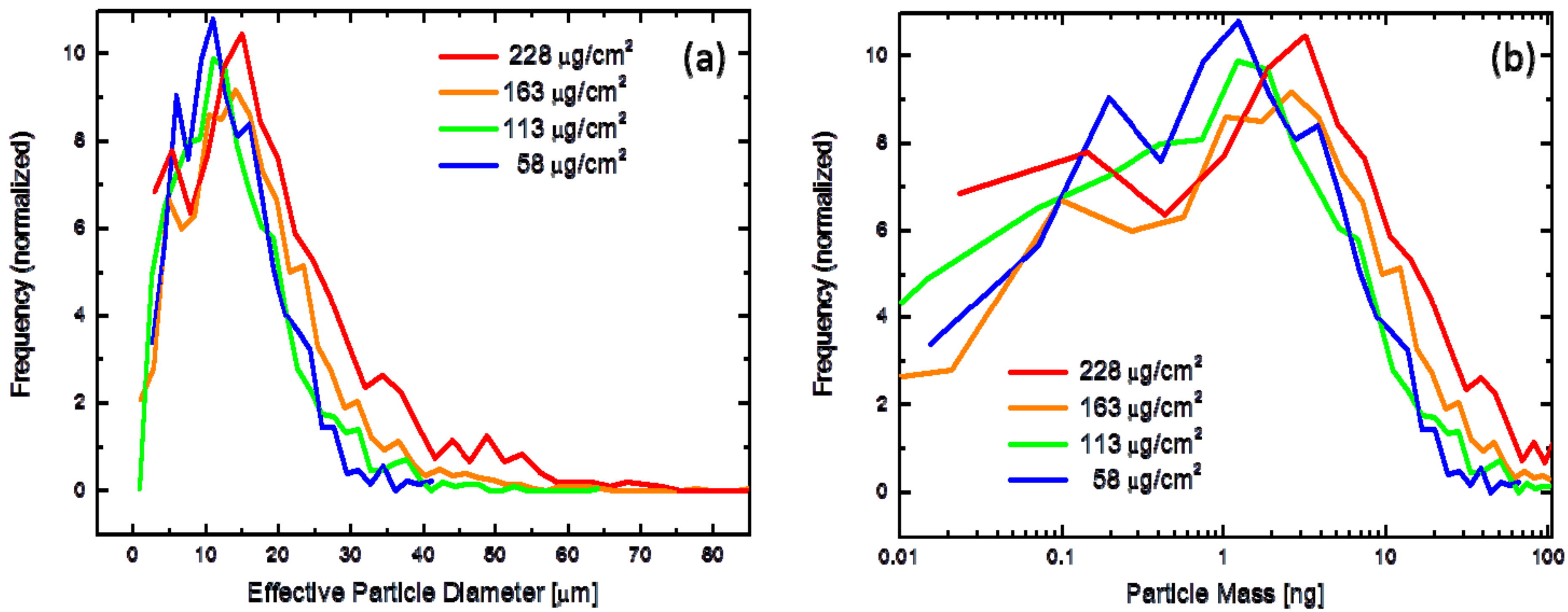
# Particle imaging analysis

## NRL ParticleMath

1. Import optical image
2. Scale image
3. Particle analysis
   - thresholding
   - locate particles/clusters
   - identify axis
   - axial slices
   - compute volume
   - effective diameter
4. Compute loading and particle size distribution



# Sieved RDX particle size & mass distributions




- 20 μm sieve produces particles in 5-20 μm range of interest
- > 20 μm indicative of clustering
- 10 μm average particle size = 1.2 ng

# Test Coupon Summary

- Sieving and inkjetting were compared with fingerprinting as techniques for fabricating test coupons for optical spectroscopy
- Sieving provides test coupons with comparable fingerprint metrics
- Inkjetting provides good control of areal density but poor control of desired fill factor, particle sizes or particle contact with the substrate - important considerations to optical spectroscopy techniques.
- A new particle imaging analysis technique, NRL ParticleMath was used to quickly characterize fingerprint or sieved test coupons and validated by UV-visible spectrophotometry.

# Conclusions

- Presented stand-off detection techniques suitable for improved checkpoint screening:
  - PT-IRIS: All-optical trace detection
  - LTV: Create vapor (where there was none) for IMS detection
- Presented new test coupon preparation technique

# Stand-off Trace Detection

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