

TSL's efforts toward CBP detection needs



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TSL's efforts toward CBP detection needs

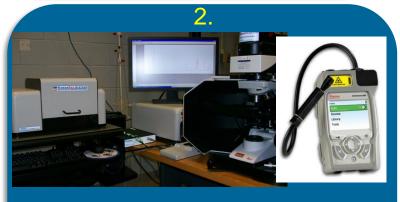
TSL Co-authors: John Brady, Barry Smith, Jeff Barber, Stephen Styk, Jason Stairs

and, specifically for the Vapor Detection work: John J. Brady, Kate Khan, Andrew Marr, Jonathan Canino, Garrett Corless, Eric Jeffas, Tierney Sugrue, Paul Flanigan, Ashly Starn, Linsey Kirschmann, Kyron Johnson, Inho Cho, Peter Hickey, Peter Glover, Paul Redfern, Steve LaLonde, Ketevan Chigogidze, Nancy Lugg, Marie-Josee Binette, Danny Fisher, Richard T. Lareau, Barry T. Smith, Erin Tamargo, Patricia McDaniel, and Leah Tanyag

OUTLINE

TSL's Primary Mission:

Enhance homeland security by performing test and evaluation of technologies to detect and mitigate the threat of improvised explosive devices and other contraband materials.

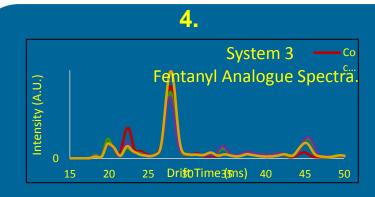


Forseti proj - high res. signatures to hand held deployed systems





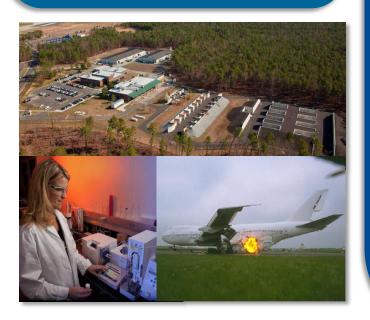
HVS Vapor trace detection of Cargo Containers



ETD Fentanyl Detection Assessment

TSL Overview

Primary Mission: Enhance homeland security by performing test and evaluation of technologies to detect and mitigate the threat of improvised explosive devices & other contraband materials.

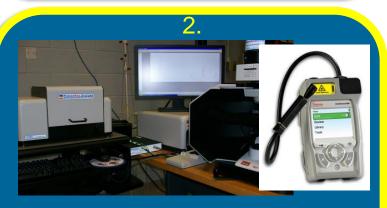


- Vulnerability and Mitigation: TSL works closely with TSA and other HSE stakeholders to identify explosives detection requirements and develop mitigation strategies.
- **Developmental T&E**: TSL works closely with industry counterparts to mature explosives and contraband detection technologies to meet customer requirements.
- *Independent T&E*: The ability of all screening equipment currently deployed in US Airports to meet TSA explosives detection requirements has been validated by TSL. (independent T&E)
- Applied Research: TSL scientists develop tools and methods to ensure we can cost-effectively test next-gen systems as they mature.
- Work For Others: TSL leverages its expertise in detection science to help affiliated agencies understand and develop explosive detection capability.

Forseti Signatures Project

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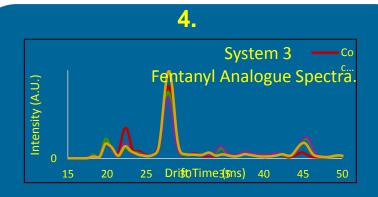


Forseti proj - high res. signatures to hand held deployed systems





HVS Vapor trace detection of Cargo Containers

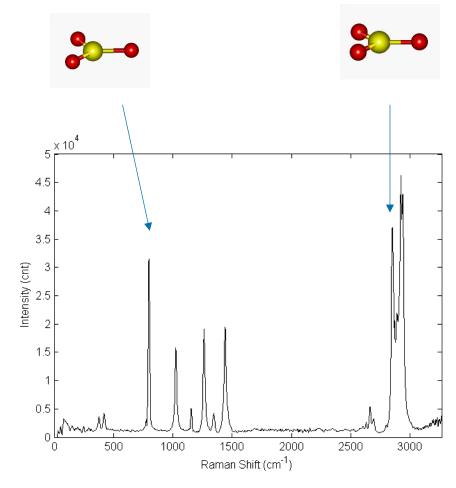


ETD Fentanyl Detection Assessment

What is Raman spectroscopy?







Pros/Cons of Raman spectroscopy

Pros

- Non-contact
- Measure through vial/clear packaging (some translucent)
- Raman SHIFT not absolute

Cons

- Weak requires laser
- Sampled at laser focus
- Fluorescence
- Heating/absorbance with dark materials

JIEDDO sponsored Started in 2010



Explosives Detection Subgroup



Rapid Deployment of Government Data onto Explosives Detection Systems for the Warfighter – Forseti Program

Signatures Goal

- •Develop a spectroscopic database of signatures for military, commercial and Home Made Explosives (HME) containing existing and emerging threats.
- •Provide the database to vetted industrial partners to develop or improve current security systems.
- •Shorten development time and lower cost by minimizing duplication of effort and therefore acquisition costs.

Methodology

- Acquire signatures of explosives at high resolution with laboratory equipment.
- Transform spectra to match resolutions and file formats of handheld systems.
- Test detection of handheld Raman and ATR-IR using government libraries and proprietary chemical identification algorithms.

Procedure

Variations in spectra among similar instruments arise from differences in:

- Experimental design
- Spectrometer configurations
- Detector technologies

~Libraries are device-dependent~

Procedure

- Spectra of acetone, acetonitrile, sodium chlorate, sodium perchlorate, potassium perchlorate, and C-4
- Record spectra with laboratory equipment
- Record spectra with handheld system
- Compare resolutions, line positions, and line strengths

Lab spectrometers



- Kaiser Optical Systems Raman RXN1-785 Analyzer
- 785 nm laser with Variable Power from 10 – 400 mW
- Holographic Grating with Range of 100 – 3450 cm⁻¹
- Spectral Resolution of 4 cm⁻¹.



- Bruker IFS 66v/S with FRA 106 Raman Accessory
- 500 mW CW Nd:YAG (1064 nm)
- LN₂ Germanium Detector
- Spectral Resolution of 2 cm⁻¹

Handheld systems



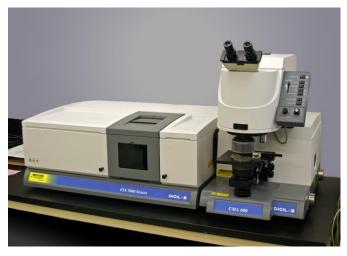
FirstDefender RMX 785 nm Raman System



DeltaNu Advantage 1064

Handheld systems on order 8 – 14 cm⁻¹ resolution

Laboratory to Handheld- 6/2018



Battelle-Columbus Digilab FTS-7000e



2011



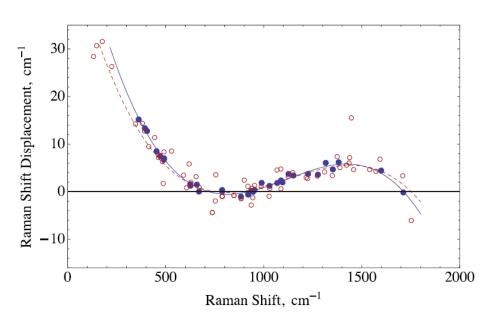
Therm in the control of the control

TSL
Bruker Vertex 70
2018

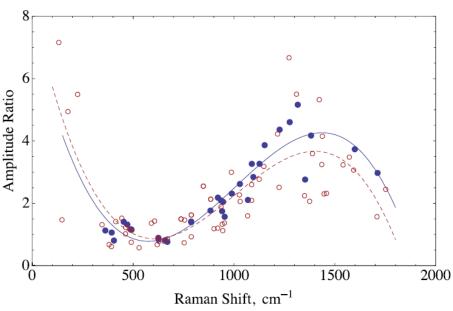


Corrections to spectra

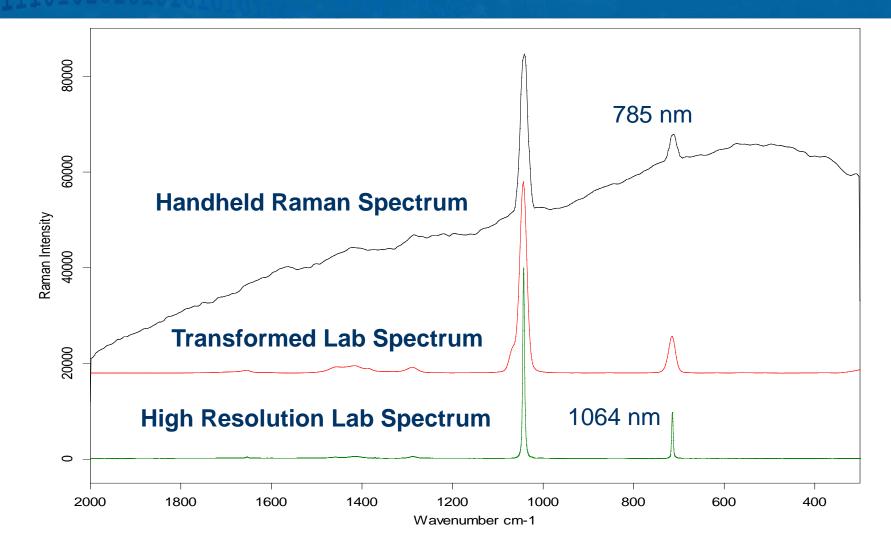
Raman shift correction



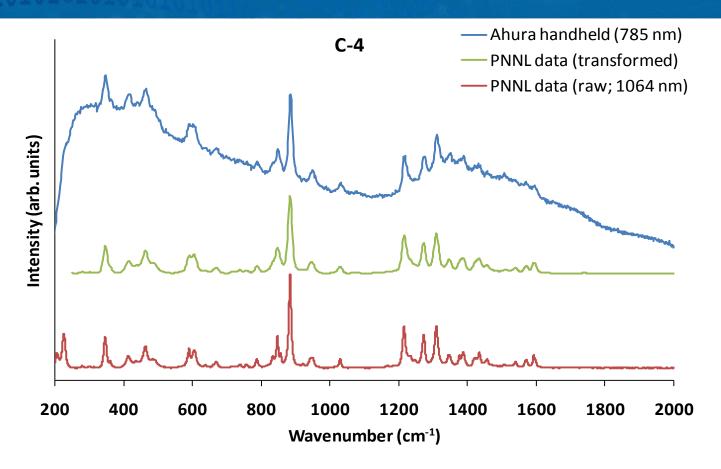
Intensity correction



Ammonium Nitrate - Successful Detection with Transformed Library

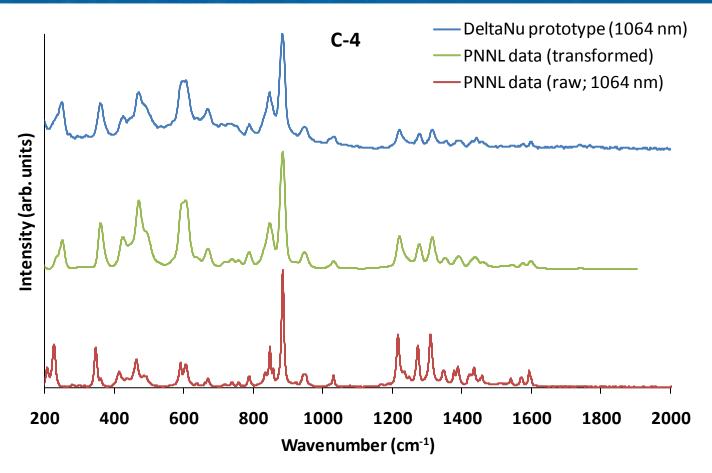


Ahura FirstDefender RMX



C-4 Spectra from PNNL Laboratory, Transformed, and Ahura Handheld

DeltaNu Advantage 1064



C-4 Spectra from PNNL Laboratory, TSL Transformed, and DeltaNu Handheld

Forseti Test Results

- Transformed library from high resolution spectra was highly successful across four separate devices
- Eight military and homemade explosives identified with imported libraries
- Twelve precursors and chemicals identified with imported libraries
- Vendor algorithms used imported libraries with no special adaptations to algorithms

See Weatherall et al., "Adapting Raman Spectra from Laboratory Spectrometers to Portable Detection Libraries," Appl. Spectrosc., vol. 67, pp. 149-157, 2013.

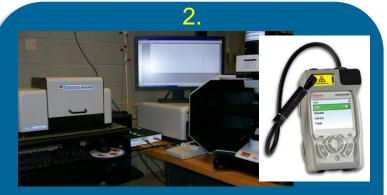
Current status

- TSL (explosives)
 - Transforming laboratory data for use on handheld systems (Forseti process)
 - Test effectiveness of transform function using vendor matching algorithm
 - Snowy Range Low Cost Raman (SnR LCR) first results 13 materials, scores 0.85-0.98
- CBP & DEA (fentanyls):
 - Collection of laboratory spectra of fentanyls

High Volume Sampling/ Vapor Detection

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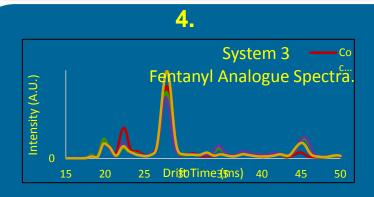


Forseti proj - high res. signatures to hand held deployed systems





HVS Vapor trace detection of Cargo Containers



ETD Fentanyl Detection Assessment

Introduction

- Explosives vapor detection (EVD) offers an alternative cargo screening capability
- Before EVD equipment can be used operationally, its performance must be assessed



- An understanding of the vapor concentration (i.e., energetic and nonenergetic components) produced in realistic scenarios → Standards
- Development of an assessment testbed, assessment methodologies and associated quality assurance and control methods





Previous work

Studies have been performed in the past using different pseudo-operational scenarios but...

- ... there are challenges:
- Low levels of vapor present for detection
- Utility of laboratory-based instruments for benchmarking
- Numerous factors impact vapor concentrations (e.g., temperature, humidity, air flow, vapor sinks)

We sought to expand our current analytical capabilities for vapor benchmarking and perform fundamental characterization of vapor produced by explosives.







Research and Assessment Objectives

Headspace

- Determine "bouquet" of samples
- 20 mL vial
- Already in use

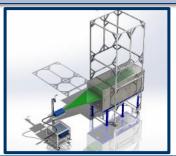
Adsorption

- Vapor-in
- Sources/sinks
- Vapor-out
- Quantitative vapor-flow measurement



Visualization

- 1/3-CONEX size
- Visualization
- Modular design
- Full instrumentation suite



Assessment

- CONEXContainers
 - LD3s
- Air Cargo Skids
- Multi-week evaluation



International Engagement

The US approach allows for comparison and validation without overlap and will continue into 2019.

International Engagement: Collaborators

The research of vapor and assessment of high volume samplers and analyzers is a joint international effort through CTTSO/TSWG bilateral agreements



Science and Technology















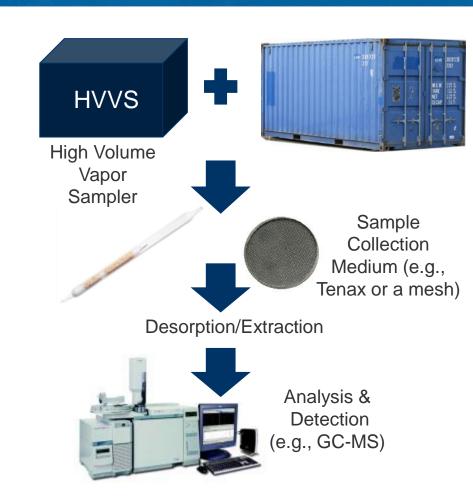


Vapor Trace Assessment

Assessment Goals

- 1. Determine whether high volume samplers and detectors are capable of sampling and detecting vapor from explosives from cargo containers such as CONEX containers, pallets, and LD3s in an open scenario (Easy Case)
- 2. Determine the bench mark vapor values (i.e., the quantity of explosive vapor for vapor sampling including the vapor signature such as the non-energetics) that is available under these specific scenarios

As a result, an RFI (RFI-16-14381) was opened and multiple participants responded and were accepted to partake in the assessment.



Threat Selection and Preparation

Materials and their equilibrium vapor pressures that were selected for use in the assessment based on volatility, the instrument's claimed detection capability, and availability

Material	Main Explosive Component/Signatures	Vapor Pressure @ 20 °C (Torr)	Source	
2,4-DNT	2,4-DNT	1.1027 x 10 ⁻⁴	Sigma Aldrich	
TNT	TNT	4.44512 x 10 ⁻⁶	TSL	
Tagged C-4	RDX	1.74986 x 10 ⁻⁹	TSL	
	DMNB (taggant)	9.42178 x10 ⁻⁴		
Untagged C-4	RDX	1.74986 x 10 ⁻⁹	TSL	





Experimental Design

- The pre-washed containers were in the same orientation and the same color to allow comparable heating/cooling profile
- Background samples of the containers and locations were obtained.
- Only one material per container to prevent cross contamination
- Multiple containers allowed for fresh samples to be obtained due to sampling rates and potential influx of "fresh" air.

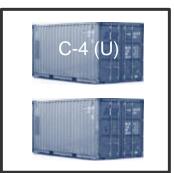
Crane Site: Analyze blank and DNT containers without commerce



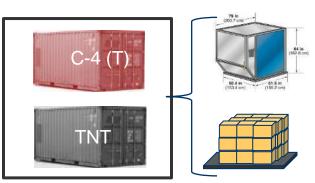
Bunker Area: Analyze containers without commerce











Explosives were placed on a table in a CONEX, in a box in a pallet, or in a LD3 container.

C-4 (T) represents tagged material; C-4 (U) is untagged.

Threat Preparation – Air Cargo



Pallet



- Bulk C-4, TNT, and DNT were individually placed into an LD3 container or a pallet.
- For the LD3, the material was placed in an open Tupperware container on the bottom of the container.
- For pallets, a box containing the bulk material was placed in the middle of the simulated pallet.



Experimental Design - Sampling Schedule

- The goal was to obtain samples from a specified container three times per day (i.e., morning, after lunch, and at the end of the day).
- Schedule allowed time to analyze samples, transition equipment, and for re-equilibration to occur.
- August 15-26, 2016 in Atlantic City,
 NJ: Higher temperature provides higher concentration

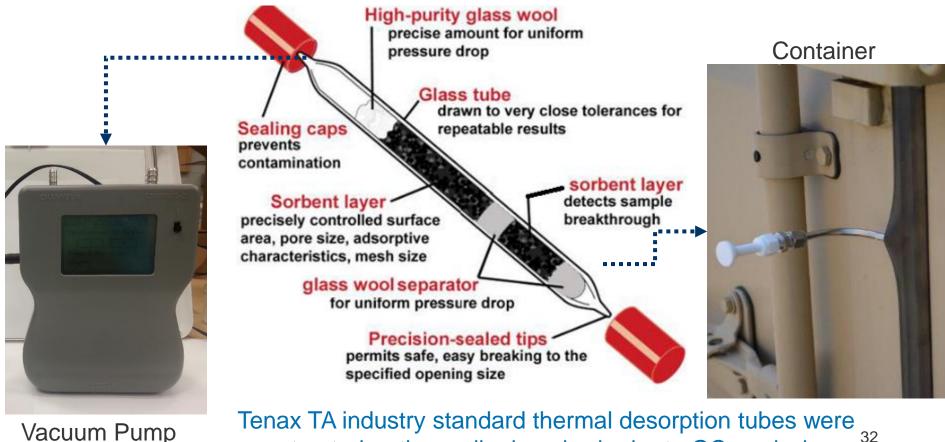


Location	Container Code	8/18	and all	alate 817	lage 818/	7116 8/19/2 ¹
Crane Site	LD3-Blank1	-	Y	-	-	-
	C-Blank1	-	Y	_	-	-
	C-Blank2	-	Y	-	-	-
	C-DNT1	-	Y	-	-	-
	CP-DNT1	-	Y	-	-	-
	Air Blank	-	Y	-	-	-
Bunker Area	C-TNT1	Y	-	-	-	-
	C-TNT2	Y	-	-	-	-
	C-TNT3	Y	-	-	-	-
	C-C4T1	-	-	-	-	Y
	C-C4T2	-	-	-	-	Y
	C-C4T3	-	-	-	-	Y
	C-C4U1	-	-	-	Y	-
	C-C4U2	-	-	-	Y	-
	CP-TNT	-	-	Y	-	-
	CL-TNT	-	-	Y	-	-
	CP-C41	-	-	Y	-	-
	CL-C41	-	-	Y	-	-
	C-Blank3	Y	-	-	Y	Y
	Air Blank	Y	Y	Y	Y	Y

Example Week 1 Sampling Schedule for Participant 1

Vapor/Signature Analysis

Signature assessments were conducted throughout the evaluation in an effort to determine the quantity of vapor available for detection



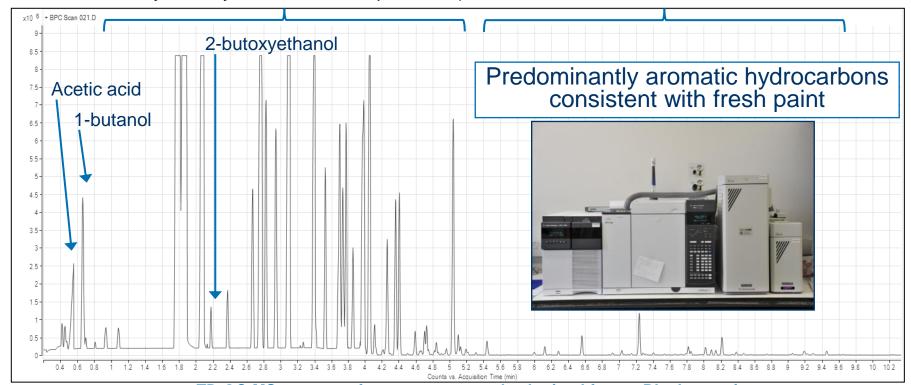
Tenax TA industry standard thermal desorption tubes were 32 extracted or thermally desorbed prior to GC analysis.

Vapor/Signature Analysis

CONEX containers were found to provide a complex background

Aromatic hydrocarbons
(toluene → xylenes → trimethyl benzenes
→ ethyldimethyl benzenes → natphthalene)

Larger aliphatic compounds

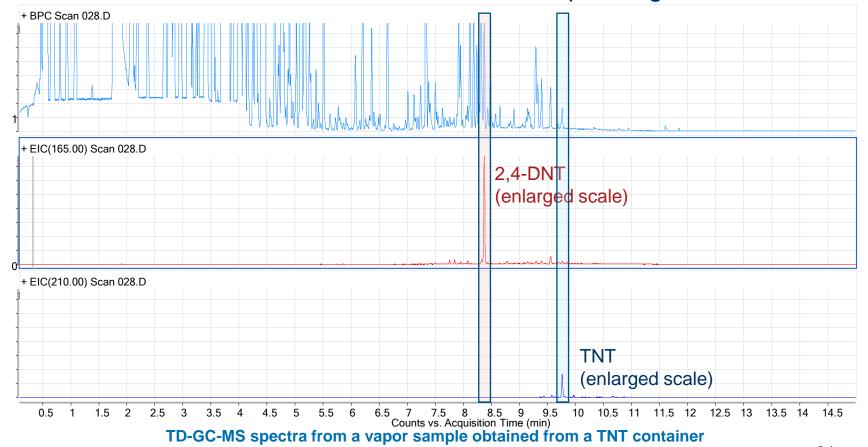


TD-GC-MS spectrum from a vapor sample obtained from a Blank container

Can we see signatures from the target material in this complex background?

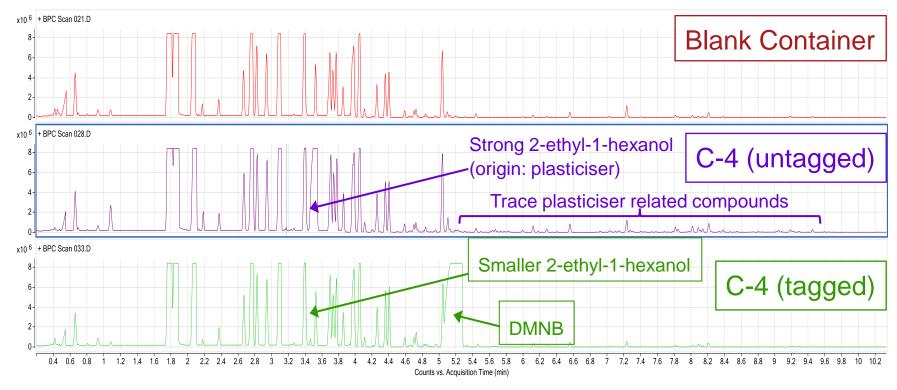
Vapor/Signature Analysis - TNT

CONEX containers were found to provide a complex background, but TNT and DNT* were observed in the corresponding containers.



Vapor/Signature Analysis – C-4

C-4 containers were found to contain cyclohexanone, 2-ethyl-1-hexanol, DMNB (tagged only), etc.



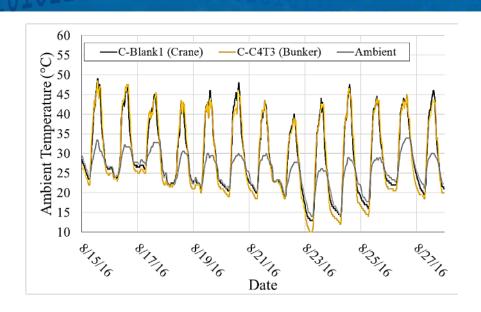
TD-GC-MS spectra from a vapor sample obtained from a various containers

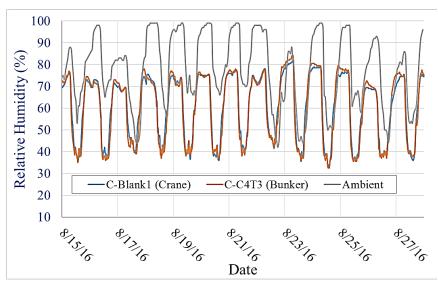
Vapor/Signature Analysis

Material	Concentration Range (ng/L)	Potential Temperature Correlation
2,4-DNT	250 - 410 [*]	Υ
TNT≎	18 - 24	Υ
DMNB	8,800 - 13,900	Υ
RDX	-	-

Non-energetic signatures were also observed such as 2-ethyl-1-hexanol, cyclohexanone and taggants (e.g., DMNB)

Assessment Results



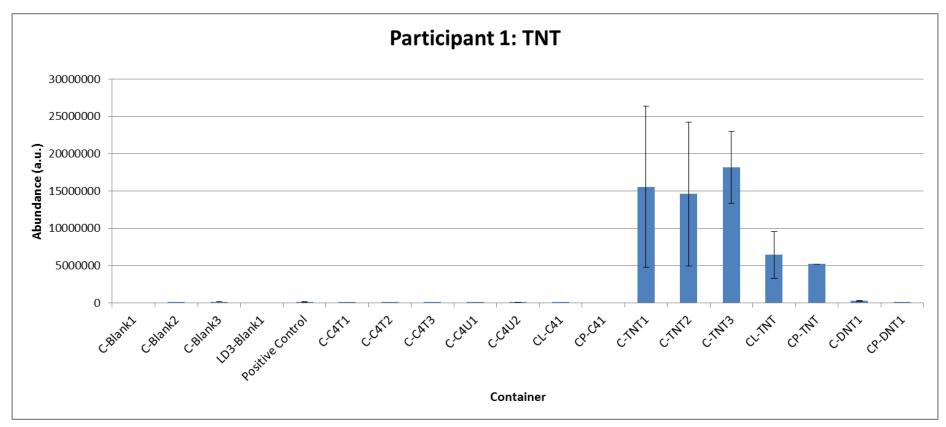


Containers showed a significant increase in temperature (~10°C) above ambient temperature conditions with an average of **45°C**

Containers showed lower humidity compared to ambient conditions

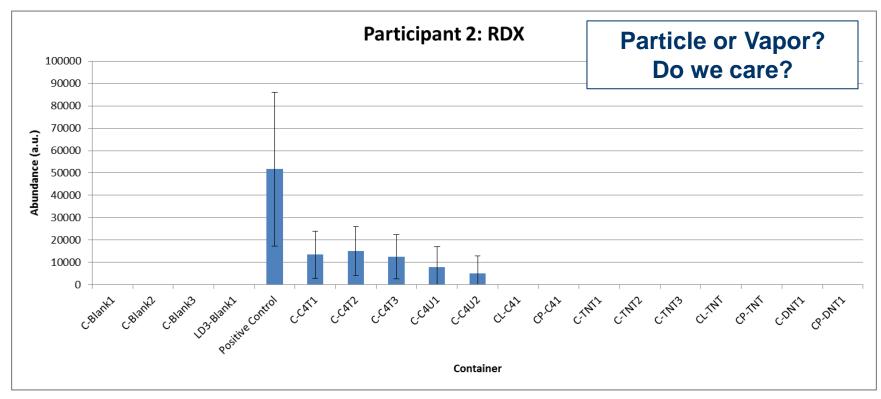
Data suggests that the air exchange between the container and the ambient environment may be low

Assessment Results: TNT



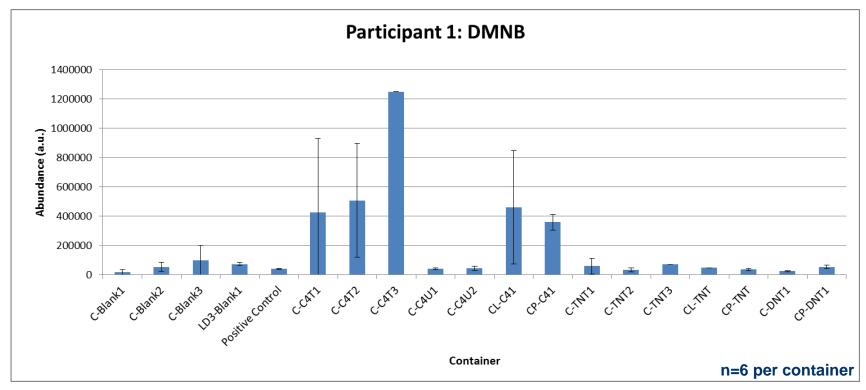
- Detected in containers throughout the assessment
- Possible correlation with temperature

Assessment Results: C-4 (RDX)



- Detected in containers throughout the assessment
- No observed correlation with temperature

Assessment Results: C-4 (DMNB)



- Detected in containers throughout the assessment but also found in the background samples
- No observed correlation with temperature

Suggests that the addition of non-energetics could aid in the detection of these materials

Assessment Results: Summary

 Weather conditions (principally temperature and humidity) seemed to be weakly correlated with vapor availability and may have increased the observed levels of vapor

Material	Participant				
	1	2	3	4	5
DNT	Р	_	Р	Р	_
TNT	Р	Р	?	Р	_
DMNB	Р	_	Р	?	_
RDX	_	Р	?	_	_
BKGND Issues	?	Υ	Υ	?	?

P = Potential? = Inconclusive— = Not observed

- In the open sample scenario (Easy Case) evaluation of some technologies demonstrated the potential capability to detect materials of interest
 - Particle vs. vapor? Other signatures of interest? Non-energetics?
- Other species (e.g., EGDN and AN) were commonly observed, however the origin of these was not explained by the experimental design and was not observed in the signature analysis samples
 ₄₁

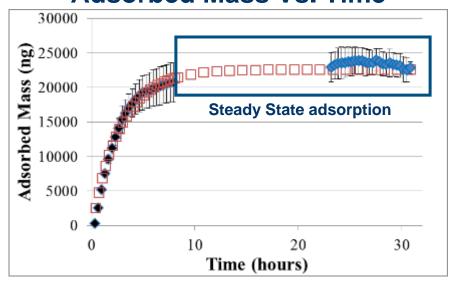
Way Forward

Adsorption





Adsorbed Mass Vs. Time



- Engage collaborators and participants to disseminate information for their continued development
- Through funding from TSWG (PM: Patricia McDaniel) and EXD (PM: Kumar Babu) we will:
 - Continue fundamental studies to determine the vapor levels available for detection and account for adsorption and permeation
 - Quantitatively assess the collection efficiency of HV filters/traps
 - Continue to evaluate high volume sampling systems under different, more realistic scenarios
 - Air cargo & Marine containers
- ADD Drug Vapor Detection assessment.

2017 & 2018 Vapor Trace Assessment

- The TSL has conducted a second vapor trace assessment of high volume sampling (HVS) systems under different, more realistic scenarios
 - Air cargo & Marine containers
 - Data analysis is underway
- Will be conducting a third assessment of HVS and analyzers this summer (August/September 2018)
 - If interested in participating, please contact the TSL.





Non-Energetic Species

The vapor pressure of some energetic materials may be low enough to prevent collection of sufficient explosive molecules.

Potential Signature Species for Explosive Materials				
Methyl, Dinitrobezene*				
2-ethyl-1-hexanol				
Cylcohexanone				
Taggants (e.g., DMNB)				

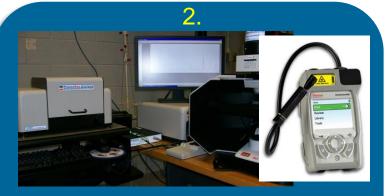
The materials listed above are non-energetic components that were observed during our signature analysis work for explosive materials and could serve as potential additions to your library to aid in identifying the material of interest.

ETD assessment for Fentanyl detection

1.

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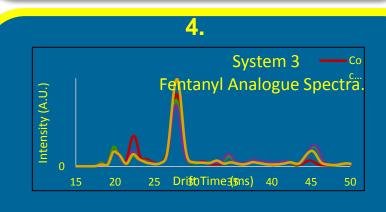


Forseti proj - high res. signatures to hand held deployed systems





HVS Vapor trace detection of Cargo Containers



ETD Fentanyl Detection Assessment

Mission Alignment with CBP

- In recent years, there has been an increased influx of smuggled potent synthetic opioid compounds, which cannot be rapidly detected upon importation into the U.S.
- The DHS Science and Technology Directorate (S&T) received a direct request from the U.S. Customs and Border Protection (CBP) Office of Field Operations (OFO) for support in identifying potential technology solutions for the non-intrusive inspection (NII) and rapid detection of synthetic opioid materials at high-throughput international mail facilities
- To support CBP's request, DHS S&T's Chemical and Biological Defense Division (CBD) created the Synthetic Opioid Detection Program, led by Dr. Rosanna Robertson
- Dr. Robertson has requested TSL developmental test and evaluation (DT&E) support to identify potential detection solutions via tech foraging of near COTS systems

Developmental Test & Evaluation

S&T CBD DT&E Feasibility Request

- Capability assessments for COTS technologies identified via technology scouting
- Optimization of prototypical technologies solicited via an industry prize competition

1) Primary Screening

- Collect data for large-bulk quantities of synthetic opioid and/or surrogate materials using COTS X-Ray transmission and/or Computed Tomography (CT) systems
- Provide initial data to OEMs to facilitate the development of software/algorithms
- Dependent on availability of bulk materials

2) Secondary Screening

 Conduct technical assessments for presumptive/confirmatory synthetic opioid detection technologies (i.e. Raman/FTIR, ETD, colorimetric/assay kits) that have the potential to provide alarm resolution capabilities

3) Critical Response

 Conduct short duration data collection activities to develop solutions for capability gaps associated with the detection of emergent synthetic opioid materials

TSL has the detection science SME and capability to provide T&E for primary and secondary screening technologies in support of the CBP mission.

Test Article Development

Test Articles

- A stream of commerce assessment to create synthetic opioid concealments as well as false alarm sets – contractor visits to international mail facilities
- Assessment of material properties relevant to specific detection modalities
- 1) Material Safe Handling and Storage
 - Provide administrative support to identify regulatory requirements
 - Develop LSAPs and/or SOPs for the safe handling/storage of bulk materials
- Test Set Build
 - Create test articles representative of those observed within the operational screening environments of international mail facilities
 - Identify synthetic opioid surrogate materials
- 3) Field Sample Analysis and Characterization
 - Analysis and characterization of field samples obtained from international mail facilities to support requirements development

Test article support will focus on the identification of synthetic opioid test sets to be used for T&E.

ETD Capability Assessment

- Due to similarities in the sampling and analysis of trace explosive and illicit drug residues, Explosive Trace Detection (ETD) systems are often utilized for the trace detection of illicit drugs
 - A formal standard exists for the trace detection of explosives; no equivalent standard exists for illicit drugs
 - Need for verification and validation of trace illicit drug detection technologies
- Illicit Drug Detection Working Group
 - ➤ Developed Tier I Trace Illicit Drug Detection Standard → synthetic opioid analytes not included
 - Considering potential technology solutions for capability gaps associated with the screening of emerging illicit drugs, such as synthetic opioids
- ETD Fentanyl Detection Capability Assessment
 - Initially requested by the TSA Office of Requirements and Capabilities Analysis (ORCA)
 - Assessment of current gold standard ETD systems against emergent synthetic opioid threats identified as capability gaps

Objective

determine if ETD systems can be used for the trace detection and identification of synthetic opioid materials.

Emergent Threats

Synthetic Opioids

- Fentanyl and various analogues of fentanyl being readily observed by DHS component organizations in operational settings
 - Commonly encountered fentanyl derivatives:
 - Fentanyl
 - Acetyl fentanyl
 - Furanyl fentanyl
 - Carfentanil
 - **❖** U-47700

- 4-Fluoroisobutyrylfentanyl
- Acryl fentanyl
- Butyryl fentanyl
- Valeryl fentanyl
- Benzylfentanyl

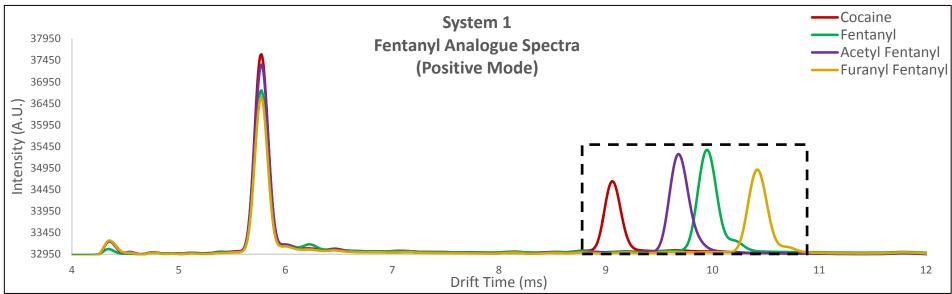
- 3-methyl fentanyl
- Acetyl norfentanyl
- Allylfentanyl
- Cyclopropyl fentanyl
- Tetrahydrofuran fentanyl

- Key Considerations
 - Safety
 - 1) Personal Protective Equipment (PPE)
 - 2) Toxicity levels and decontamination protocols/procedures
 - 3) Laboratory Safety Activity Plan (LSAP) developed prior to capability assessment
 - Detection
 - 1) Are significant Ion Mobility Spectrometry (IMS) signals produced for various fentanyl analogues?
 - 2) Can ETD systems differentiate specific fentanyl analogues from one another?

Need for safe, reliable, efficient solution(s) for safe handling and testing of various synthetic opioid materials and detection technologies.

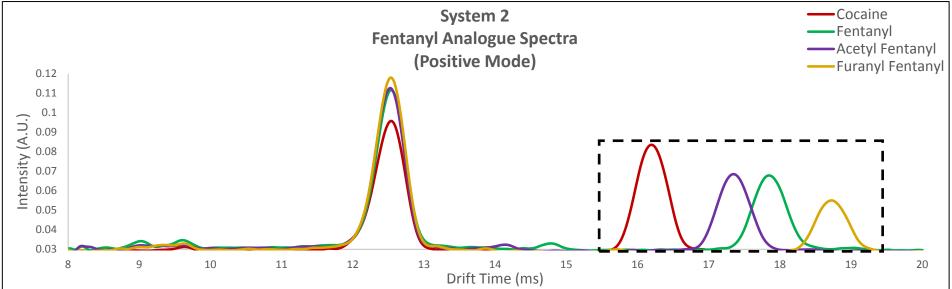
Assessment Overview

- 1) Analytes (commercially available standards)
 - Fentanyl
 - Acetyl fentanyl
 - Furanyl fentanyl
- 2) ETD Systems
 - 4 gold standard COTS ETD systems
 - System 1 → illicit drug detection specific configuration
 - Systems 2/3/4 → TSA configuration
- 3) Sample Preparation (n=5)
 - Direct deposition of working solution onto appropriate ETD sampling media
 - 100 ng mass loading level
- 4) Controls
 - Positive Control → Cocaine (positive mode); RDX (negative mode)
 - Negative Control → Methanol



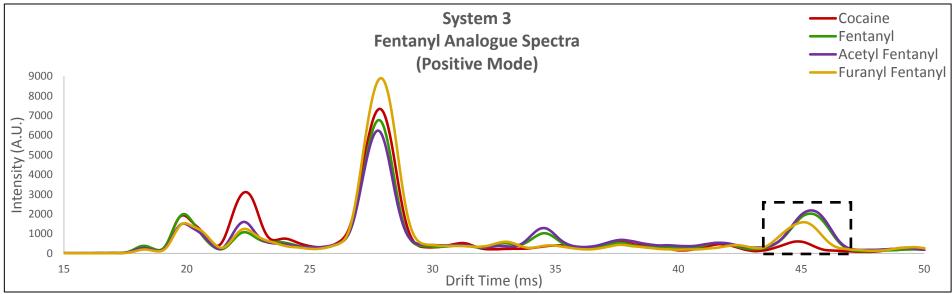
- · Specific narcotics configuration used
- Existing detection channels for various illicit drugs and fentanyl analogues
- Well-resolved, distinct peaks observed
- Rapid clear down times observed

Analytes	Drift Time (ms)	Reduced Mobility (K ₀)	Perf	etection forma ms/Tr	nce
Cocaine	9.064	1.150	5	1	5
Fentanyl	9.952	1.047	5	1	5
Acetyl Fentanyl	9.688	1.076	5	1	5
Furanyl Fentanyl	10.42	1.000	5	1	5



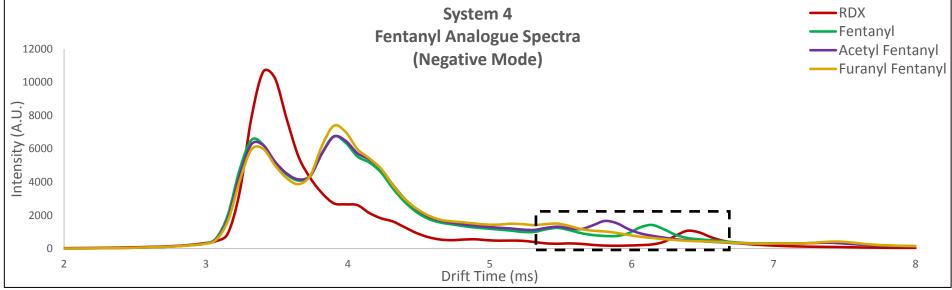
- TSA configuration used; no existing detection channels for fentanyl analogues
- Fentanyl analogues distinctly separated in IMS spectra
- · Rapid clear down times observed
- Specific detection channels for fentanyl analogues could be programmed into system

Analytes	Drift Time (ms)	Reduced Mobility (K ₀)
Cocaine	16.180	1.150
Fentanyl	17.907	1.039
Acetyl Fentanyl	17.362	1.072
Furanyl Fentanyl	18.725	0.994



- TSA configuration used; no existing detection channels for fentanyl analogues
- Significant overlapping of fentanyl analogue features observed in IMS spectra
- Prolonged clear down times observed for fentanyl and acetyl fentanyl

Analytes	Drift Time (ms)	Reduced Mobility (K ₀)
Cocaine	27.895	1.150
Fentanyl	45.362	0.707
Acetyl Fentanyl	45.362	0.707
Furanyl Fentanyl	45.099	0.711



- TSA configuration used; no existing detection channels for fentanyl analogues
- Fentanyl analogue features only observed in negative mode;
 Cocaine (positive control) observed in positive mode
- Low IMS signals observed for fentanyl and acetyl fentanyl
- No significant IMS signal observed for furanyl fentanyl

Analytes	Drift Time (ms)	Reduced Mobility (K ₀)
RDX	6.390	1.510
Fentanyl	6.140	1.571
Acetyl Fentanyl	5.810	1.661
Furanyl Fentanyl	-	-

Conclusions

- 1) ETD Systems 1 and 2 capable of detecting and identifying specific synthetic opioid materials
 - System 1 → existing narcotics configuration, which includes fentanyl channels
 - System 2 → no existing detection channels for fentanyl analogues; could be programmed
 - Distinct, well-resolved peaks observed for cocaine and each fentanyl analogue
 - No clear down issues observed on either ETD system
 - Due to high resolution/specificity, newly emerging fentanyl analogues could likely be identified on either system, but would need to be programmed into algorithm retroactively
- ETD System 3 did not display the potential capability to specifically identify fentanyl analogues
 - Based on observance of overlapping IMS spectral features for the fentanyl analogues under test, a "fentanyl class" could be developed.
 - Potential for proactive detection of newly emerging fentanyl analogues
- 3) ETD System 4 displayed very low average signal intensity for each fentanyl analogue
 - Need to further investigate why analogue features were observed in negative mode

Disclaimer

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