

Applications of Trace Detection in Combating the Opioid Epidemic

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So What? Who Cares?

- **Space:** Detection of synthetic opioids in international mail facilities
- **Problem:** Packages of synthetic opioids are difficult to detect due to small quantities and ever-changing chemical structure
- **Solution:** Trace detection technologies offer a viable option for interdiction when used in a multi-layered approach
- **Results:** Tools like IMS and TD-DART-MS can detect sub-microgram residues on the exterior of packages without the need to directly expose agents to powders
- **TRL:** 8/9
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Who We Are

NIST



Materials Measurement Lab



Surface & Trace Chemical Analysis Group

Focus areas:

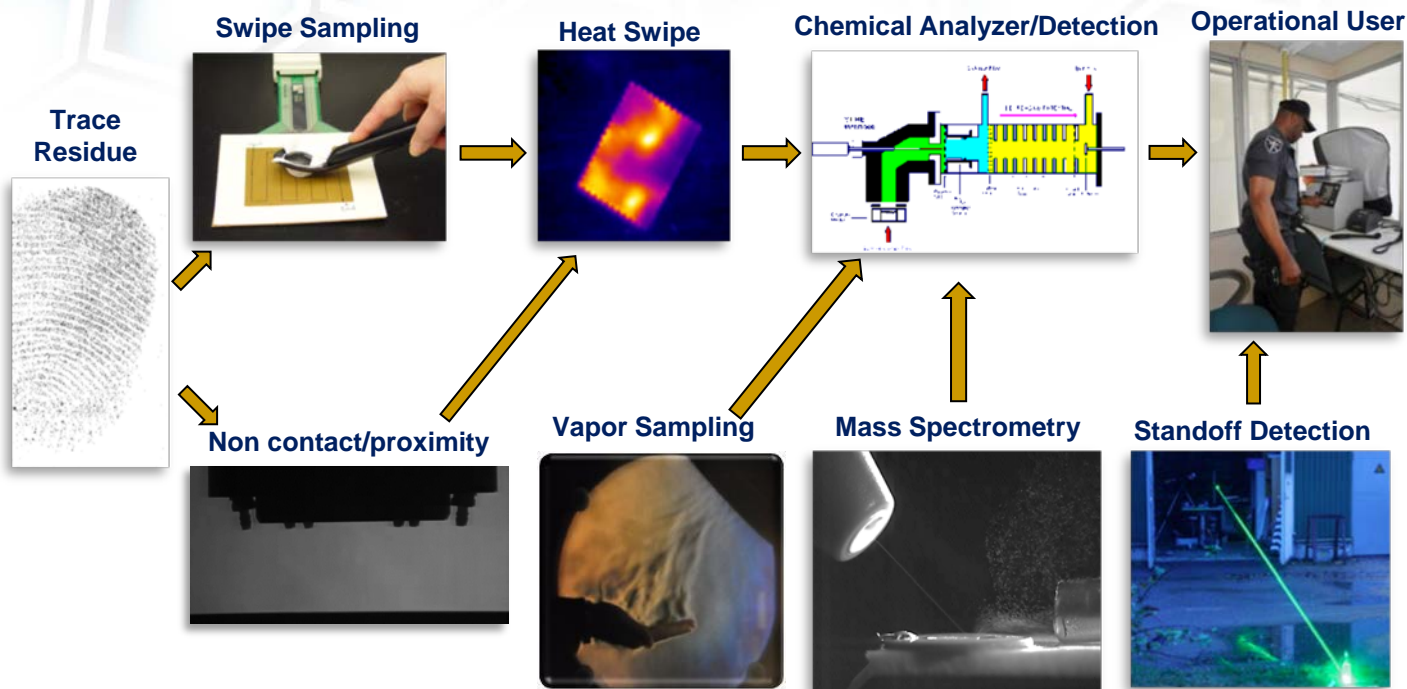
- **Trace Contraband Detection**
- **Forensic Science**
- **Advanced Mass Spectrometry**
- Additive Manufacturing & Inkjet Printing
- Nuclear Materials Characterization
- Standards Development



Current Opioid-Related Efforts

1. Collaborate with agencies to address current needs, anticipated needs, and gaps
2. Establish a platform for the comparison of technologies, focusing on the unique challenges of synthetic opioids
 - Complete a detailed method optimization study
 - Determine the effects of mixtures and complex background matrices
 - Evaluate the expected false positive rates
3. Develop / accelerate novel detection techniques
 - How can we obtain rapid, reliable, and low-cost detection and/or identification
4. Identify & quantify opioid background in operational environments
 - Develop methods to measure background and understand its significance
5. Measure trace residue persistence
 - How are these compounds effected by the environment
6. Investigate novel implementations of new technologies into workflows

Trace Contraband Detection Approach



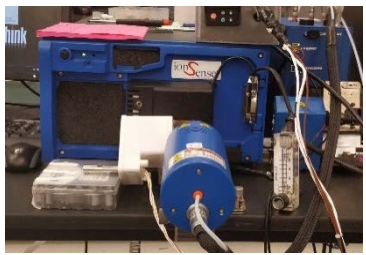
One of our major program areas is to provide the basic measurement and standards infrastructure to support optimization and calibration of existing trace detection technologies and to assist in the development of effective next generation technologies.

Potential Application for Opioid Screening

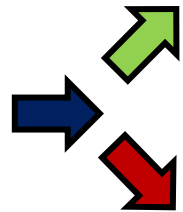


www.dlpartpanda.com

Package Received



Sample Outside of Package for Residue (IMS or TD-DART-MS)



www.istockphoto.com

Package Continues



www.agilent.com

Package Pulled Aside for Further Testing

Strengths and Limitations of Trace Detection

Trace detection targets the invisible (μg to ng) levels of residue that are present on surfaces through intentional or unintentional contamination, without having to come in contact with the bulk material.

Benefits

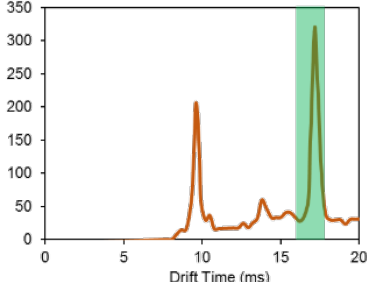
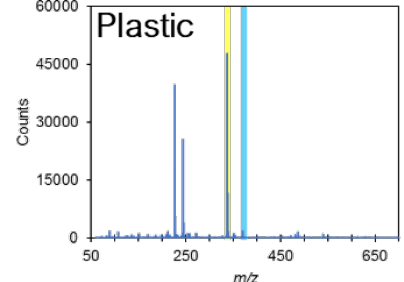
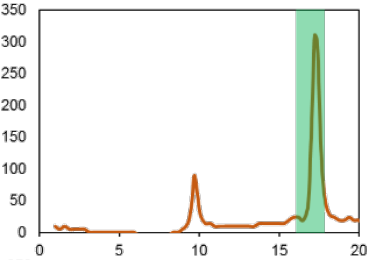
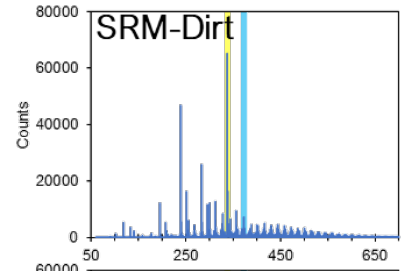
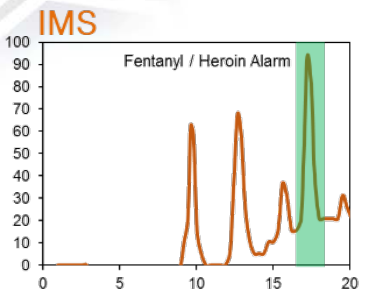
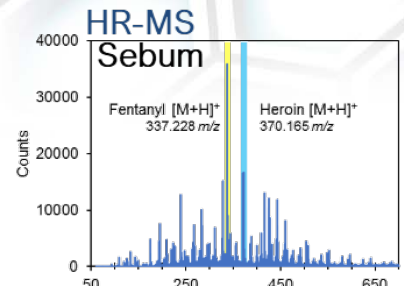
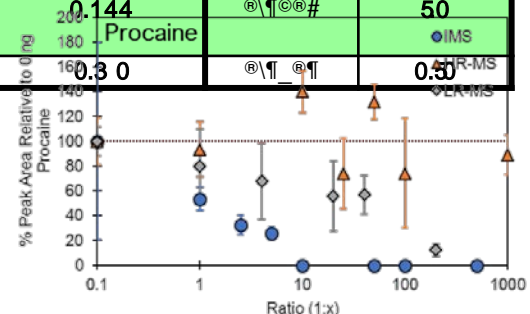
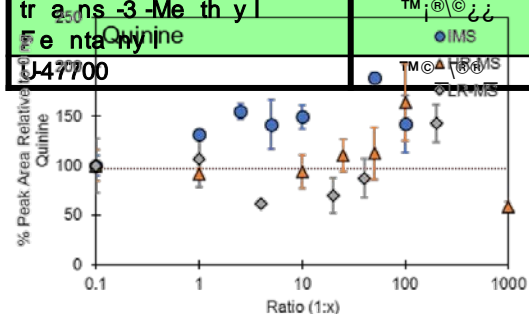
- Safety
 - Trace sampling potentially lowers exposure risks
 - Use to help determine the appropriate PPE
- Speed
 - Analyses can be completed in seconds
- Rapid Adaptability
 - Libraries can be updated for emerging threats
- Sensitivity & Specificity
 - ng to sub-ng detection limits
- Wide Threat Detection Range
 - Narcotics, explosives, CWA's, etc.

Drawbacks

- Mixtures
 - Some limitations with complex mixtures
- Cost
 - Higher upfront cost than colorimetric
- Library Dependence
 - Most systems require search libraries
- Maintenance
- Training

Platform for Comparison

Compound Name	DART-MS		IMS	
	Base Peak +30V (m/z)	LOD ₉₀ (ng)	K ₀	Sensitivity (ng)
Acetyl Fentanyl	TMⓂTM\ⓂⓂⓂ	0.222	Ⓜ\Ⓜ#_#	50
Carfentanyl	TM_ \ⓂTMⓂ	0.197	Ⓜ_#\ⓂⓂ	50
Cyclopropyl Fentanyl	TM##\Ⓜ _	0.165	Ⓜ_#\Ⓜ#	1.0
Fentanyl	TMⓂTM#\ⓂⓂⓂ&	0.142	Ⓜ\Ⓜ _&_	1.401
para-Fluoroisobutyryl Fentanyl	TM@_ \ⓂTM_	0.351	Ⓜ\ⓂⓂⓂⓂ	1.0
Furanyl Fentanyl	TM#\Ⓜ _Ⓜ#	0.199	Ⓜ\ⓂⓂⓂⓂTM	50
trans-3-Methylfentanyl	TM_ \Ⓜ _&_	0.144	Ⓜ\ⓂⓂⓂ#	50
Quinine	ⓂIMS	200	ⓂIMS	50
Procaine	ⓂHR-MS	160	ⓂHR-MS	50
U-47700	TMⓂⓂⓂⓂ	0.30	Ⓜ_#_Ⓜ	0.50



Can Trace Be Used To Detect Opioids?

- We are working with forensic labs and DHS components to understand if trace opioid detection is operationally possible
- To date >200 items sampled
- Overall 92 % accuracy in correct detection
- ***If used to determine the presence of synthetic opioids, 100 % accuracy***
- Typically micrograms to milligrams of material present
- False positives / negatives commonly attributed to samples containing plant material

Quantitative measurements from a package purchased on the dark web.

Sample	Furanyl Fentanyl	Fentanyl	Cocaine
E-packet shipping label	0.35 µg	0.75 µg	0.10 µg
E-packet	0.11 µg	3.3 µg	0.21 µg
Play-Doh packaging	0.35 µg	0.12 µg	0.06 µg
Baggie containing drug	10.0 µg	0.06 µg	0.15 µg

Inner Packaging	Extract	Percent Occurrence	Result Type
Drug Detected	Same Drug Detected	79 % (n = 151)	True Positive
Drug Detected	No Drug Detected	1.5 % (n =3)	False Positive
Drug Detected	Different Drug Detected	2.5 % (n = 5)	False Positive
No Drug Detected	Drug Detected	4 % (n = 7)	False Negative
No Drug Detected	No Drug Detected	13 % (n = 25)	True Negative
Overall Accuracy:		92 %	

Trace Degradation Study

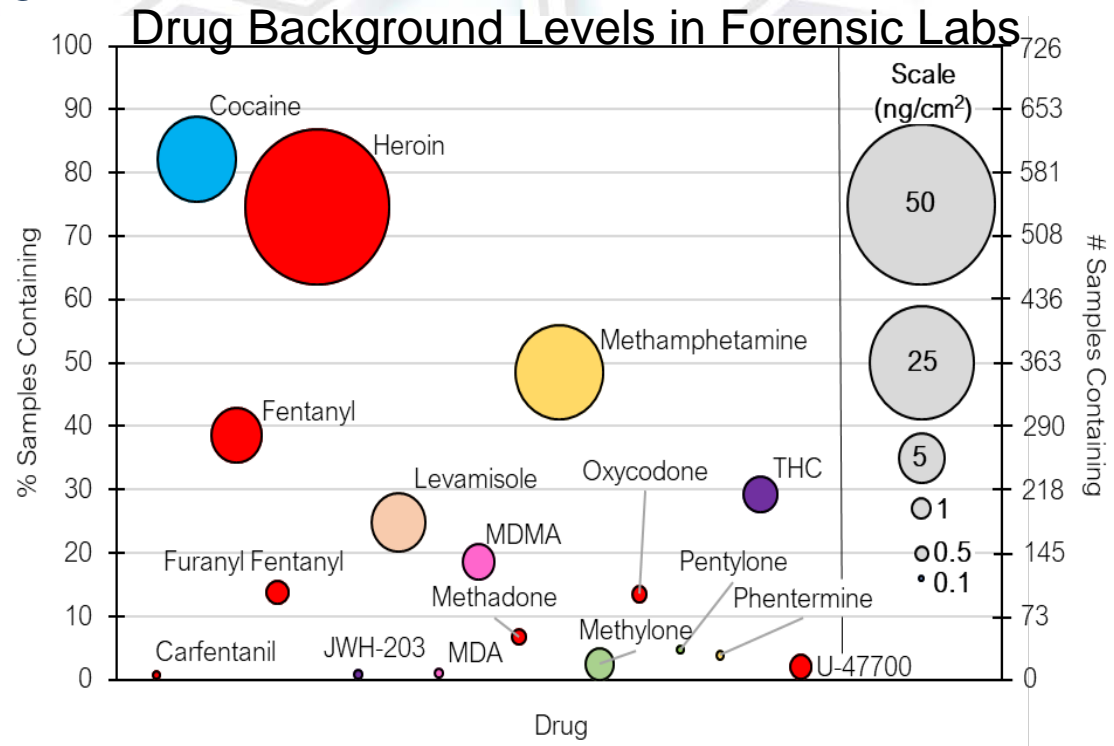
Day 7	-4 °C	Lab	30 °C	Ozone	47 °C	UV
Fentanyl	Green	Orange	Orange	Red	Orange	Red
Furanyl	Green	Orange	Orange	Red	Red	Red
Heroin	Green	Green	Green	Green	Red	Red
Day 35	-4 °C	Lab	30 °C	Ozone	47 °C	UV
Fentanyl	Green	Orange	Red	Red	Red	Red
Furanyl	Green	Orange	Orange	Red	Red	Red
Heroin	Green	Green	Green	Green	Red	Red

% loss	< 20 %	20 % - 50 %	> 50 %
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- Use inkjet printing and simulated environmental exposures to evaluate trace residue stability
- Significant and rapid decay is observed in harsh environments
- Stability is preserved for samples stored in refrigerated conditions
 - Important for standards development
- Currently expanding opioids, investigating other NPSs, and examining the effects of adulterants / cutting agents

Operational Background

- Deployment of detection technologies requires an understanding of the background of the environment
- Background effects data quality, detection rates, and analyst safety
- Working with labs over the last two years to measure, interpret, and provide feedback on drug background levels
- Over 20 labs have participated resulting in over 700 samples
- Recently begun sampling mail facilities



Initial Results Show Substantially Lower Backgrounds in IMFs.

Takeaways

- Trace detection is a viable tool for triaging, presumptive testing, or intelligence gathering of opioid-related packages
- Trace residue exists on the exterior of drug packages and persist long enough to potentially be leveraged for a safe, non-intrusive analysis
- Implementation of trace detection, requires understanding of the operational background, though this concern is lower in postal screening environments
- Trace detection tools can provide useful, complimentary, information to traditional analyses

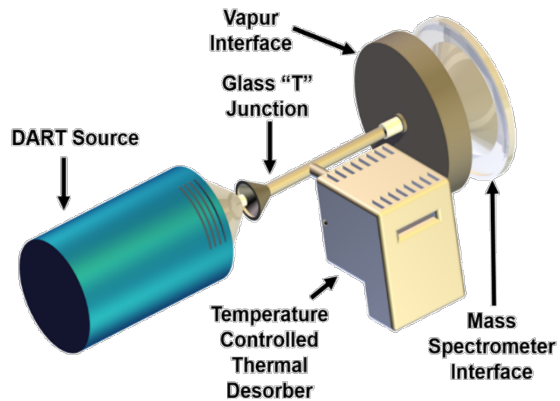


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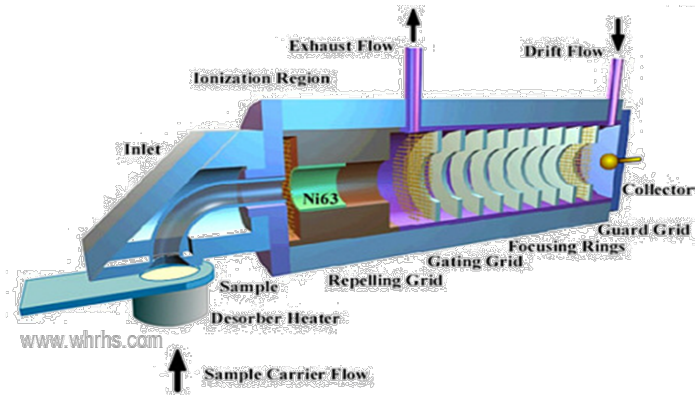
TD-DART-MS

- Heated nitrogen metastable gas stream for sample desorption and ionization
- Can be coupled with portable or laboratory grade mass spectrometers
- Provides increased safety to examiners due to enclosed configuration



IMS

- Small and portable with lower resolution than MS
- Thermal desorption followed by ionization (^{63}Ni , photo, APCI)
- Ions are separated as they pass through a drift tube counter to a flow of gas
- Evaluated 6 different systems to date



Quantitative Results

- LC-MS/MS being utilized to understand the level of contamination
- Significant differences between powder and pill levels observed
- Exterior levels of drugs typically exceeded 1 μg on the outside of the bag
 - Pharmaceutical pills presented lower levels
- Exterior levels of drugs not in the exhibit (noise) were typically well below 1 μg
 - Potential for signal thresholding for better determination of whether or not a drug is likely to be present in an exhibit

	Cocaine	Fentanyl	Heroin	MDMA	Meth.	U-47700
Average (μg)	6.45	5.90	197.02	22.03	20.57	1.05
Median (μg)	1.92	2.64	39.39	11.70	14.01	1.05
Max Recovered (μg)	46.62	34.92	2430.00	106.80	54.09	1.29

Street Samples

- Work with forensic laboratories to evaluate the ability to analyze real world samples
- Complex make-up and low concentrations can make detection difficult

