Document Title:	**Non-Contact Detection of Fentanyl and Other Synthetic Opioids: Towards a Generalized Approach to Detection of Dangerous Drug Classes**
Author(s):	**Lauryn DeGreeff, Ph.D., Braden Giordano, Ph.D.**
Document Number:	**311353**
Date Received:	**January 2026**
Award Number:	**15PNIJ-22-GG-04418-RESS**

O-NIJ-2022-171186

IAA# 15PNIJ-22-GG-04418-RESS

Non-Contact Detection of Fentanyl and Other Synthetic Opioids: Towards a Generalized Approach to Detection of Dangerous Drug Classes

Principal Investigator:
Lauryn DeGreeff, Ph.D., Associate Professor
Department of Chemistry and Biochemistry
Florida International University
11200 SW 8th Street, CP 304
Miami, FL 33199
(305) 348-2606 | ldegreef@fiu.edu

Co- Principal Investigator:
Braden Giordano, Ph.D., Research Chemist
Chemistry Division, Code 6181
U.S. Naval Research Laboratory
4555 Overlook Ave. SW
Washington, DC 20375
(202)-404-6320
braden.giordano@nrl.navy.mil

TIN: 65-0177616 / DUNS: 07-129-8814

Project start/end date: January 1, 2023 – December 31, 2024
Award Amount: $642,632
Reporting term: Final Report
Submitted: January 30, 2025

Recipient: National Institute of Justice
Department of Justice, Office of Justice Programs
810 7th St. NW
Washington, DC 20531

Summary of the Project

The illicit use of potent opioids poses a danger to users, law enforcement, and the public. Non-contact detection of vapors associated with hazardous drugs enables a safe and effective tool for their presumptive identification. Commercially available handheld ion mobility spectrometry (IMS) was utilized in the vaporous identification of fentanyl and related synthetic opioids. The overall goal of this project was to improve the vapor identification of fentanyl and expand detection to include designer benzodiazepines (DBs) and related substances. The focus was on the preconcentration of vaporous surrogate compounds that are representative of the majority of the drug class. Furthermore, the proposed work provides a generalized analytical approach that is adaptable to other dangerous low volatility drug classes. The project was conducted in following five steps for the trace vapor identification of fentanyl and DBs:

Step 1: Headspace analysis of reference-grade material for the identification of a surrogate compound

Determination of vapor signature was completed using an optimized solid phase microextraction (SPME) coupled with gas chromatography and mass spectrometry (GC-MS) method. The headspace of the reference-grade material was sampled using SPME and analysis was completed using GC-MS analysis. Abundant compounds that were unique to the parent drug class were selected for vapor detection.

Step 2: Identification of acrylate-based coatings for the pre-concentration of vapor surrogate using Quartz Crystal Microbalance (QCM)

After determination of target compounds for vapor detection selection of an acrylate-based polymer was conducted. Five coatings were screened for analyte affinity: benzyl acrylate, phenyl acrylate, ethyl glycol methyl ether acrylate (EGMEA), dimethyl amino ethyl acrylate (DMAEA), and cyclohexyl acrylate. The polymers were applied to Quartz Crystal Resonators (QCRs) using Initiated Chemical Vapor Deposition (iCVD).

Vapor streams of target analyte were introduced into the QCM chamber using argon gas to carry vapors across the neat analyte and onto the surface of a QCR. This allowed the analyte vapor to interact

with the functionalized QCR surface. As vapor adsorption progressed, changes in mass (Δm) were recorded, with a plateau in the graph indicating QCR saturation or maximum adsorption.

Initial optimization was conducted using uncoated gold (Au) QCRs to determine ideal sampling conditions: analyte mass and argon flow rate. Each analyte was equilibrated in sealed 0.5 L Teflon jars for 24 hours and sampled in triplicate. Following optimization, the above mentioned five acrylate polymer coatings were screened for their analyte adsorption and desorption performance. Each coating was tested in triplicate using the optimal conditions. The optimal polymer coating was selected based on the amount of analyte vapor adsorbed by each coated QCR, with the highest-adsorbing coating identified as the most effective for selective vapor surrogate pre-concentration.

Step 3: Survey of confiscated samples using coated sorbent paper

It was imperative to confirm that the vapor surrogates identified in Step 1 are detectable from actual seized materials, and that the acrylate polymer selected in Step 2 can selectively adsorb these target vapors. Laboratory-grade filter papers were functionalized with the optimal polymer chosen in step 2, using iCVD. These coated filters were affixed to the insides of Teflon jar lids and used to headspace sample ten different seized drug samples from both the Kentucky State Police (KSP) crime laboratory and Maryland State Police (MSP) crime laboratory. After a one-week sampling period, the filter papers were returned to FIU for analysis. The samples were analyzed using an optimized and validated direct Thermal Desorption-GC/MS (dTD-GC/MS) method, previously developed through in-house sampling of reference-grade fentanyl.

Step 4: Integration of SiNW array with IMS

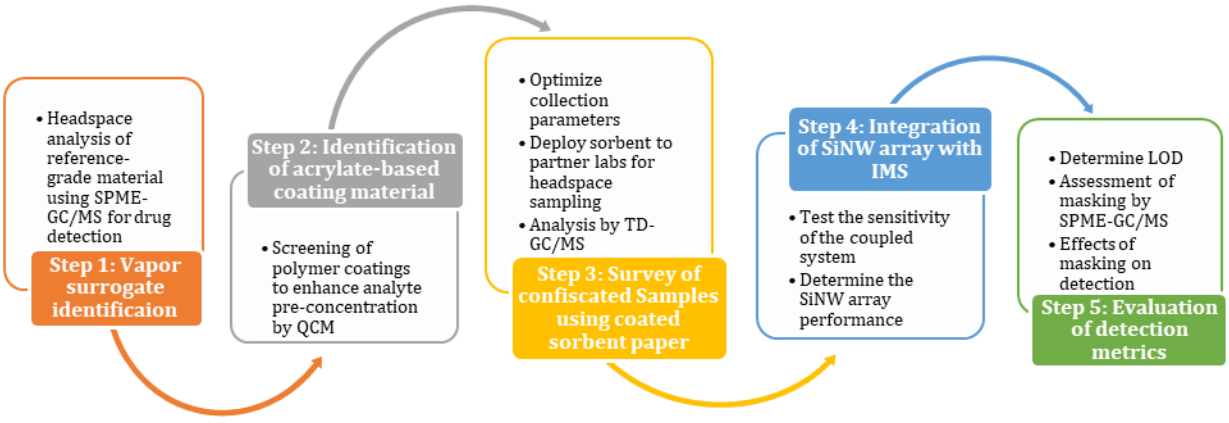
A Silicon Nanowire (SiNW) array was coated with the optimal polymer identified in Step 2 and integrated into a handheld IMS detector. To ensure effective analyte adsorption and desorption for efficient pre-concentration, sampling and desorption parameters have been optimized. Using the NRL-developed TESTbed vapor delivery system, flow rates, and exposure conditions were systematically varied to

determine the optimal conditions. GC/MS was employed to quantify analyte recovery and detect any thermal degradation, allowing fine-tuning for maximum efficiency and minimal artifact formation.

Step 5: Evaluation of detection metrics

The performance of the SiNW-IMS system will be evaluated against conventional IMS to assess its improved sensitivity and lower limit of detection (LOD) for target vapor analytes. This comparison will determine the effectiveness of the SiNW pre-concentrator in enhancing detection efficiency. Additionally, the system's ability to detect target vapors in the presence of commonly encountered interferences such as orange peels, coffee, mustard, automotive grease, adhesive tape, dryer sheets, acetone, ethanol, methanol, smoke, cleaning solutions, and deodorant will be assessed. These substances will also be tested individually to evaluate their potential to trigger false positives.

Overview of project



OUTCOMES

Significant Results

Step 1: Headspace analysis of reference-grade material for the identification of a surrogate compound

Ten reference-grade designer and prescription benzodiazepines were analyzed using a SPME-GC/MS to investigate their headspace profiles. Due to the lack of consistent volatile organic compounds (VOCs) detected, additional analysis was conducted on seized benzodiazepine samples at the Maryland State Police crime lab at two different times. During the initial visit to the Maryland state crime laboratory, sampling was conducted for 1 hour at room temperature (RT). In a follow-up visit, the sampling conditions were adjusted to a longer duration of 4 hours at a temperature of 35°C. Benzophenone was identified in the headspace of the tested confiscated samples during this second session, and it was notably found in a greater number of samples from the first visit. Table 1 summarizes the presence or absence of benzophenone across different samples and conditions. While benzophenone was observed in some seized samples at the Maryland State Police crime lab, it was not detected in any reference-grade materials, indicating the need for further analysis of seized benzodiazepine samples to determine its reliability as a target vapor surrogate for benzodiazepines.

Table 1. Presence or absence of benzophenone in benzodiazepine samples following GC/MS analysis

Sample name	Drug	Amount	Appearance	Presence of Benzophenone	
				1st Visit	2nd Visit
P550170	Etizolam	0.019 g	White Powder	Y	Y
P550332	Clonazolam	0.224 g	White Powder	N	N
P550262	Clonazolam	0.090 g	Yellow Powder	Y	Y
P550150	Bromazepam	0.105 g	Off white crystaline powder	Y	N
P550203	Flubrotizolam	0.047 g	Chunky White Powder	N	N
P55060	Diclazepam	0.047 g	White Powder	N	N
P550151	Meclonazepam	0.081 g	Off white powder	Y	N
P550261	Flubrotizolam	0.090 g	Blue powder	Y	Y

Step 2: Identification of acrylate-based coatings for the pre-concentration of vapor surrogate using Quartz Crystal Microbalance (QCM)

Figure 1 illustrates the results of the QCM trials using gold QCRs (sensors). The mass of N-phenyl propenamide (NPPA) adsorbed onto the sensors was evaluated at various sample masses, using a constant flow rate of 100 mL/min. Based on the comparison of average NPPA vapor deposition across trials, 0.08 g was identified as the optimal sample mass for subsequent experiments.

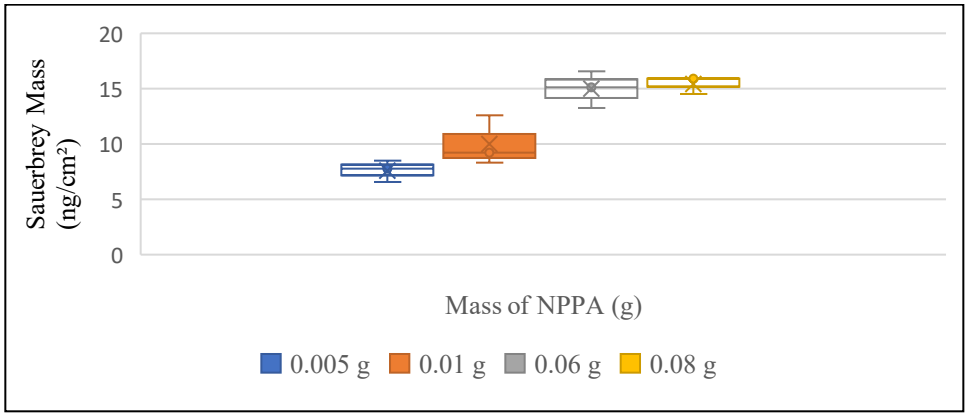


Figure 1. Mass of NPPA vapor adsorbed on the gold QCM sensor at a constant argon flow rate of 100.0 mL/min, with varying NPPA sample masses.

Figure 2 shows the results from varying the Argon gas flow rate while maintaining a constant NPPA mass of 0.08 g. Based on the comparison of adsorption performance, 100 mL/min was selected as the optimal flow rate for the experiment.

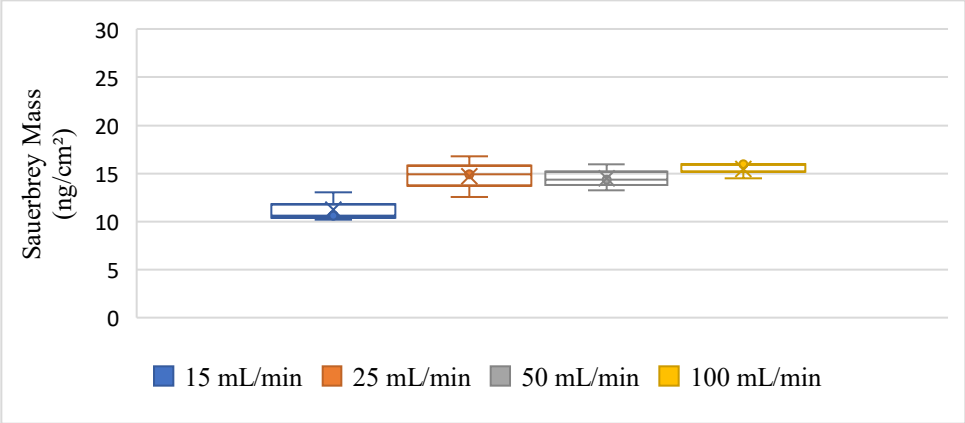


Figure 2. Mass of NPPA vapor adsorbed on the gold QCM sensor at varying Argon flow rates, while maintaining a constant NPPA mass of 0.08 g.

As depicted in Figure 3, all coated sensors adsorbed a greater mass of analyte vapor onto their surfaces compared to the uncoated gold sensor. Among them, the EGMEA-coated sensor exhibited the highest analyte uptake, demonstrating superior adsorption performance relative to both the other coated sensors and the gold sensors.

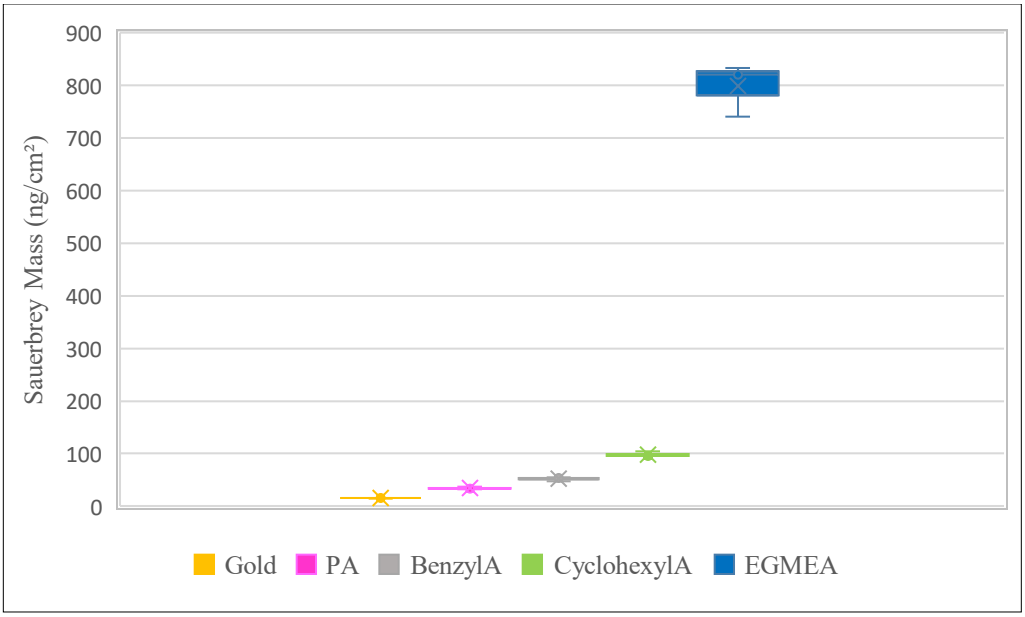


Figure 3. Trends in NPPA vapor adsorption on gold and polymer-coated QCM sensors, based on triplicate trials conducted under optimized experimental conditions.

The DMAEA-coated sensor did not exhibit the typical adsorption-desorption behavior during trials with NPPA, as illustrated in Figure 5. The anomalous behavior observed with the DMAEA-coated QCR refers to its deviation from the expected adsorption-desorption pattern typically seen in QCM experiments. In a normal trial, as illustrated in Figure 4, a polymer-coated QCR exposed to a vapor like NPPA would show an initial decrease in frequency (indicating mass gain from adsorption), followed by a plateau range (indicating saturation of mass adsorption on to the QCR surface) and finally a return toward baseline during the desorption phase once the analyte vapor stream is removed.

However, in the case of the DMAEA-coated QCR (Figure 5), the typical trend was not observed. This irregular response suggests a possible interaction between NPPA and the DMAEA coating that may have

interfered with normal analyte adsorption and desorption. Due to this anomalous behavior, the DMAEA polymer was excluded from further screening for NPPA preconcentration.

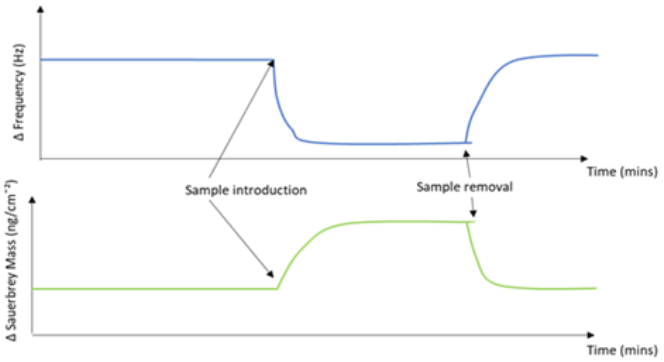


Figure 4. Typical adsorption–desorption profile from a single trial displayed by Biosens3 software: Δ Frequency vs. Time and Sauerbrey Mass vs. Time

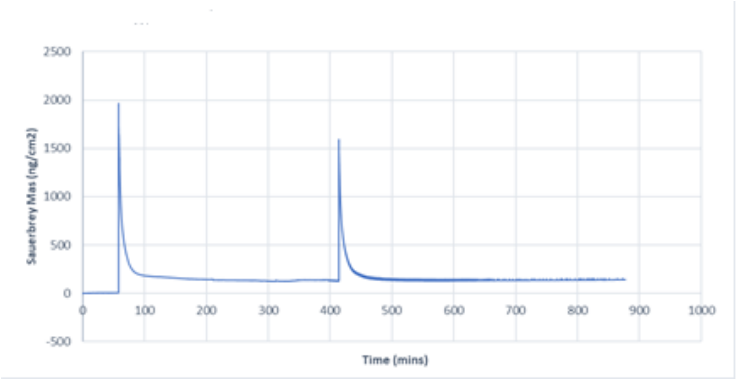


Figure 5. Anomalous behavior of DMAEA-coated sensor with NPPA vapor under optimized conditions

Step 3: Survey of confiscated samples using coated sorbent paper

Table 2 outlines the optimized dTD-GC/MS method for analyzing coated filter paper headspace sampled with reference-grade and seized fentanyl. It details the key parameters that were fine-tuned to maximize NPPA recovery and enhance the sensitivity of detection from the coated filter paper.

Table 2: Final optimized TDU-GC/MS method

Parameter	**Value**
GC-MS	
Purge Flow	3 mL/min

GC Oven Ramp	30°C/min
Flow Rate	1.94 mL/min
TDU	
Transfer Temperature	250°C
TDU Ramp	25°C/min
Initial Temperature	20°C
CIS	
CIS Ramp	12°C/min
Initial Temperature	-10°C

Figure 5 shows the average mass of NPPA collected from one-quarter of the EGMEA-coated filter paper sampled with reference-grade fentanyl and pure NPPA at FIU. The sampled filter papers were analyzed using the optimized TDU-GC/MS method outlined in Table 2. No detectable NPPA was observed after 48 hours of sampling with 100 mg of fentanyl. However, extending the sampling duration to one week resulted in the recovery of approximately 40 ng of NPPA. In comparison, exposure to 5 mg of pure NPPA for 48 hours yielded about 3.6 µg of NPPA on the coated filter paper.

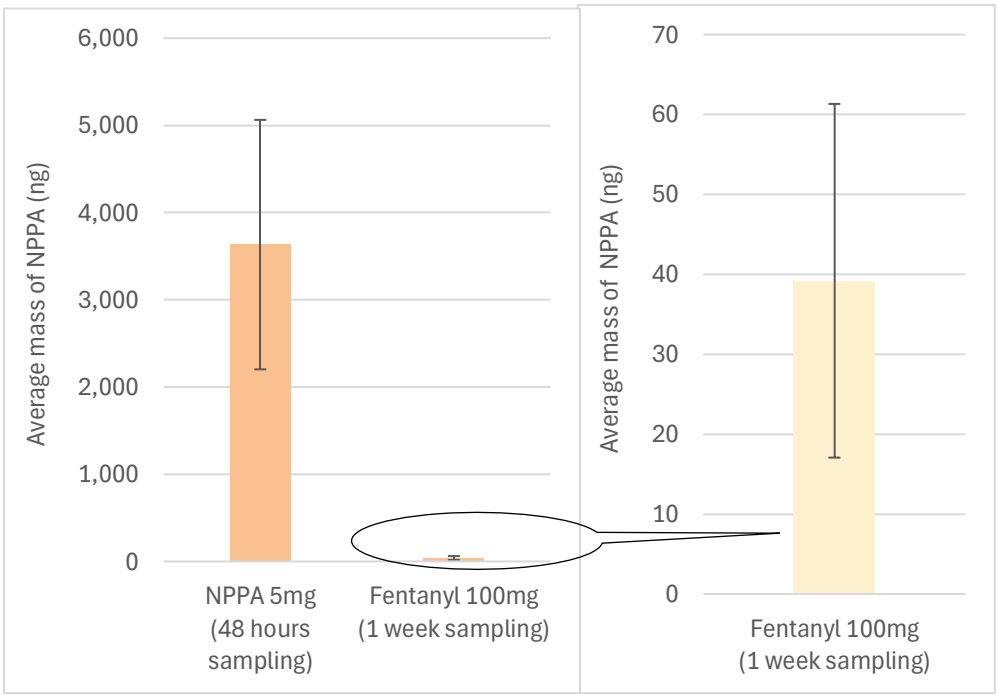


Figure 5: The average mass of NPPA collected from one-quarter of the coated filter paper in ng, using 100mg Fentanyl and 5mg NPPA.

Table 3 presents data provided by the Kentucky State Police (KSP) for ten seized fentanyl tablet samples, each identified by markings such as M, 30, Y, 21, and R039. These samples were collected between January 19, 2024, and April 5, 2024, and exhibited a wide range in weight, from 1.4561 g to 105.70 g (for two tablets combined). EGMEA-coated filter papers were used to headspace sample each of these ten seized fentanyl samples for a week.

Table 3: Sample descriptions of Kentucky State Police seized fentanyl samples

NAME	OFFENSE DATE	AMOUNT	WEIGHT	MARKINGS
KSP 1	3/7/2024	2 tablets	12.5850g	M,30
KSP 2	1/19/2024	2 tablets	1.4561g	M,30
KSP 3	2/29/2024	2 tablets	1.9000g	M,30
KSP 4	2/21/2024	2 tablets	41.4233g	M,30
KSP 5	4/1/2024	2 tablets	4.9657g	Y,21
KSP 6	3/5/2024	2 tablets	3.1654g	M,30
KSP 7	2/6/2024	2 tablets	15.9314g	M,30
KSP 8	4/5/2024	2 tablets	3.6575g	R039
KSP 9	2/6/2024	2 tablets	3.3836g	M,30
KSP 10	2/21/2024	2 tablets	105.7032 g	M,30

NPPA was successfully recovered from every sample. Figure 6 presents the average mass of NPPA that was recovered from each KSP fentanyl sample, compared to the amount that was recovered from a reference-grade 100 mg fentanyl sample at FIU. All samples were collected using EGMEA-coated filter paper. The bar graph provides a comparative analysis of NPPA recovery, demonstrating the sampling method's effectiveness across different samples.

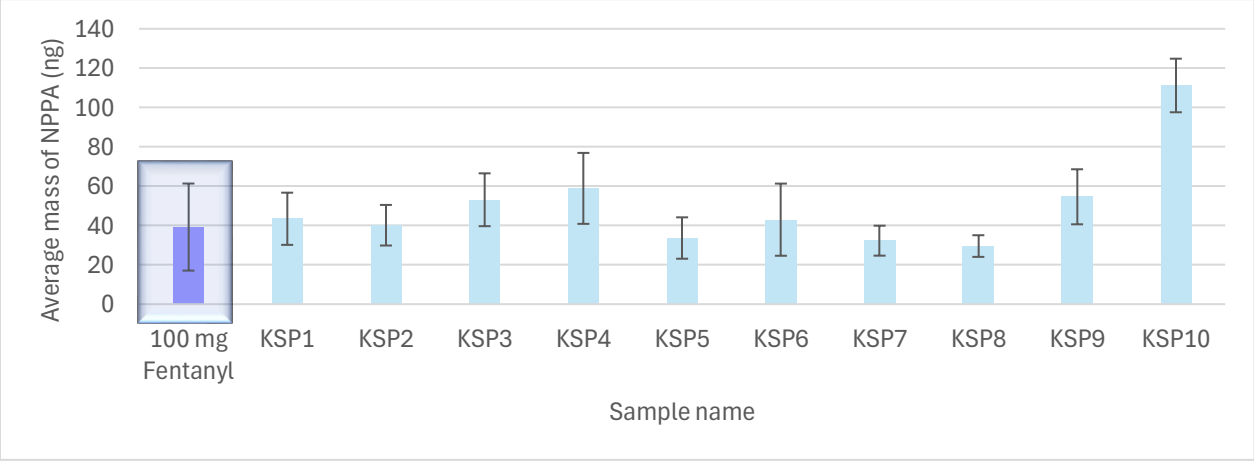


Figure 6: The average mass of NPPA collected on a quarter of the filter paper for each KSP sample compared to 1 mg of reference grade fentanyl.

Table 4 presents data provided by the Maryland State Police (MSP) for ten seized fentanyl tablet samples. EGMEA-coated filter papers were used to headspace sample each of these tablets over a one-week period.

Table 4: Sample descriptions of Maryland State Police seized fentanyl samples

Sample name	Sample content	Sample mass (g)
PSS0037	Fentanyl/Quinine/Noscapine/Acetaminophen	0.046
PSS0050	Fentanyl/Tramadol/Procaine	0.074
PSS0086	Fentanyl/ Acetyl Fentanyl	0.052
PSS0087	Heroin/Fentanyl/Acetyl fentanyl	0.052
PSS0178	Heroin/ Fentanyl/Tramadol/Meth	0.302
PSS0181	Fentanyl	0.050
PSS0198	Fentanyl/Etizolam	0.052
PSS0355	Fentanyl	0.180
PSS0381	Indication of Carfentanyl	0.056
PSS0404	Fentanyl/ p-fluorofentanyl/cocaine/ Medetomidine	0.056

NPPA was successfully recovered from every sample. Figure 7 presents the average mass of NPPA recovered from one-quarter of each MSP fentanyl sample, compared to the amount recovered from a reference-grade 100 mg fentanyl sample at FIU. To enhance data visualization, Figure 8 displays the same comparison with sample PSS0050 excluded.

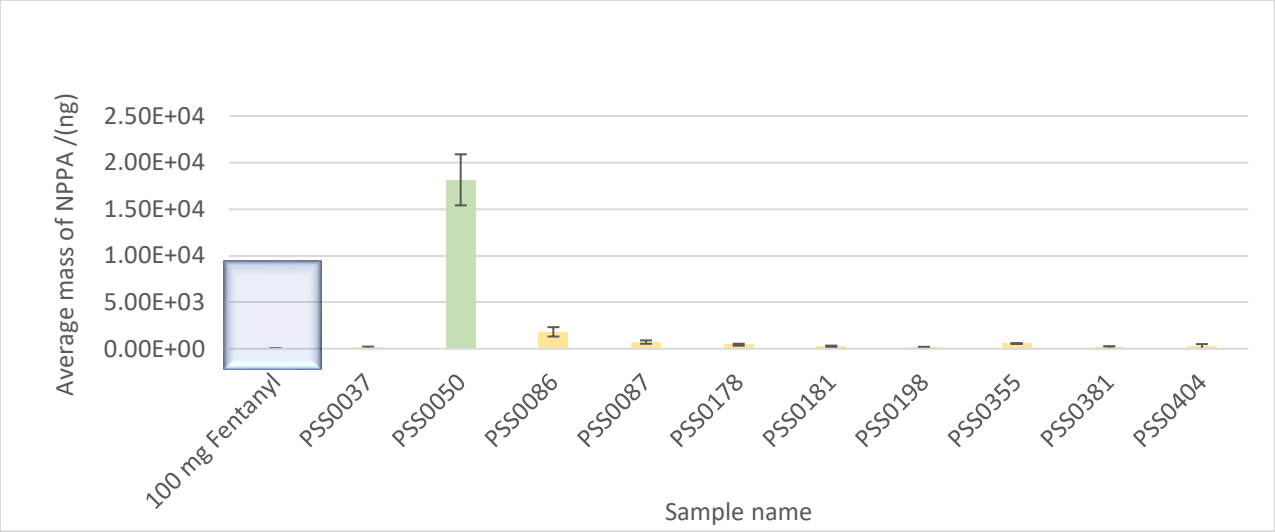


Figure 7: The average mass of NPPA collected on a quarter of the filter paper for each MSP sample compared to 100 mg of reference grade fentanyl.

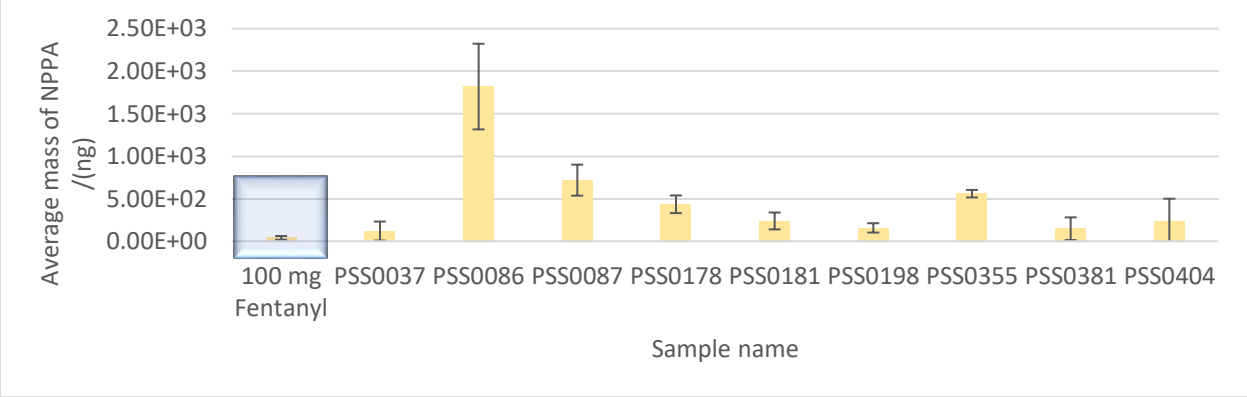


Figure 8: The average mass of NPPA collected on a quarter of the filter paper for each MSP sample, compared to 100 mg of reference grade fentanyl without PSS0050 for better comparison

The KSP and MSP fentanyl samples differed by orders of magnitude in total mass; this variation does not directly correlate with the amount of NPPA recovered or the amount of fentanyl in the sample. This is because the seized samples were not pure fentanyl; each contained a mixture of other substances. For MSP samples, the co-components were identified, but the relative abundances, including that of fentanyl, were not disclosed. For KSP samples, no information was provided regarding the composition or proportion of additional substances. Therefore, total sample mass cannot be considered a reliable indicator of the amount of fentanyl or NPPA vapor availability.

Step 4: Integration of SiNW array with IMS

A 196 ppt NPPA vapor was generated using the NRL developed trace explosive sensor testbed (TESTbed) to determine preconcentration time, vapor flow rate, and desorption time. The SiNW array was attached to the TESTbed sampling port for testing. Preconcentration time was evaluated by introducing the NPPA vapor to the SiNW array for 10, 30, and 45 s. After preconcentration, a 1-Amp current was applied to the SiNW array for 20 s to desorb the preconcentrated NPPA, followed by GC/MS analysis. The optimal preconcentration time was determined to be 30 s as depicted in Figure 9. At 30 s preconcentration there is less variability in the amount of NPPA adsorbed onto the SiNW array.

Vapor flow rate was determined by using a mass flow controller to test specific flow rates. Again, a 196 ppt vapor was generated and introduced to the SNW array for 30 s at 150, 200, and 250 mL/min. A 1-Amp current was applied to the SiNW array for 20 s for desorption, simultaneously the vapor was sampled by IMS for analysis. Figure 9 shows the optimal flow rate to be 150 mL/min. Optimal desorption time was determined to be 20 s; no detection was observed at 10 s desorption and over time degradation occurred at 30 s desorption.

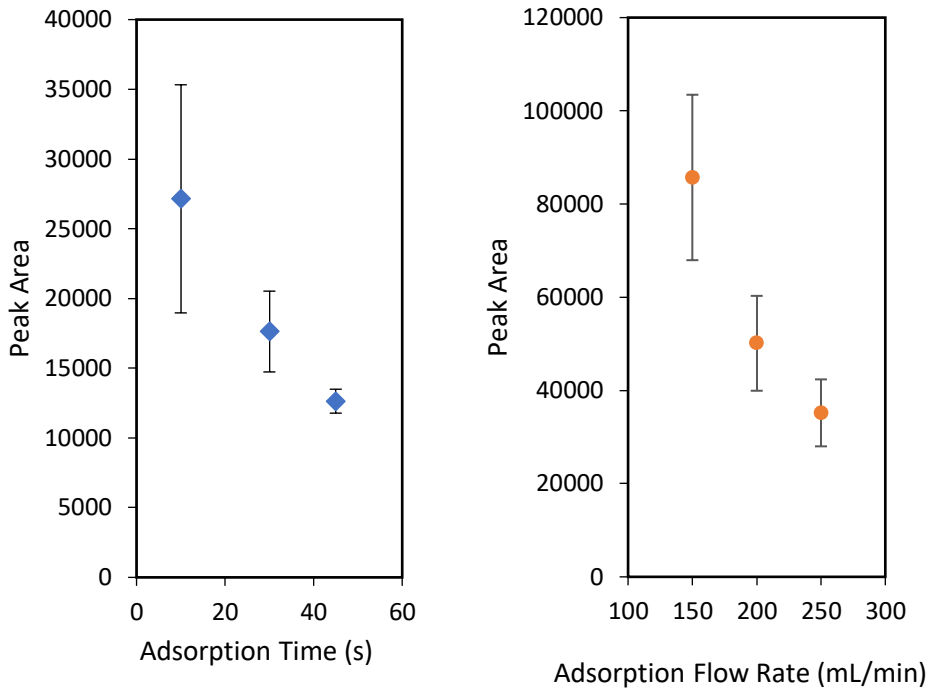


Figure 9. Determination of SiNW array optimal adsorption time and adsorption flow rate.

Step 5: Evaluation of detection metrics

An expansive study on the NPPA LOD of the IMS without preconcentration was completed. The LOD was tested at several NPPA vapor concentrations and at five sampling times (10, 20, 30, 45, and 60 s). The results of this study are outlined in Table 5. Previous study determined the LOD of NPPA to be 984 ppt with a sampling time of 10 s. The limit of detection has drastically changed from 984 ppt to 6400 ppt. The difference in instrument LOD could be due to the age of the instrument, and has been used extensive in experiments. Further study on the change in instrument LOD are currently being explored. Additionally, a survey of other instruments is underway. Future studies will determine the improved LOD using SiNW array preconcentration.

Table 5. Baseline LOD of IMS. Detection of NPPA true-positive probability (TPP) limit of 80%, n=5.

	1600 ppt	**2000 ppt**	**2400 ppt**	**3200 ppt**	**6400 ppt**
10 sec	No	No	No	No	Yes
20 sec	No	No	No	No	Yes
30 sec	No	No	No	Yes	Yes

45 sec	No	No	Yes	Yes	Yes
60 sec	No	Yes	Yes	Yes	Yes

ARTIFACTS

Publications

Planned Manuscripts

- Jayawardana, G. D.; Fulton, A. C.; Giordano, B.C.; DeGreeff, L. E. Identification of an acrylate-based material for the collection of fentanyl vapor surrogate using quartz crystal microbalance (QCM). *Analytica Chemica Acta*, **2025**, *in progress*
- Fulton, A. C.; Jayawardana, G. D.; DeGreeff, L. E.; Giordano, B.C. Preconcentration of NPPA vaper by silicon nanowire array. *Talanta,* **2025**, *in progress*

Conferences

- Jayawardana, T.; DeGreeff, L.E; Fulton, A.C.; Giordano, B. Enhancing Field Detection of Fentanyl: A Novel Pre-Concentrator for Ion Mobility Spectrometry Using Silicon Nanowires, National Institute of Justice symposium at AAFS 2025, Baltimore, February 2025.
- Jayawardana, T.; DeGreeff, L.E; Fulton, A.C.; Giordano, B. Beyond SPME, alternative approach to fentanyl vapor sampling, Pittcon Conference and Exposition, Boston, Massachusetts, March 2025.
- Fulton, A.C.; Jayawardana, T.; Giordano, B.; DeGreeff, L.E. Detection of vaporous fentanyl using silicon nanowire array preconcentration on a handheld ion mobility spectrometer, Trace Explosives and Drug Detection Workshop 2024, Dublin, Ireland. June 2024. Trace Explosives and Drug Detection Workshop 2024, Dublin, Ireland. June 2024.
- Fulton, A.C.; Jayawardana, T.; Giordano, B.; DeGreeff, L.E. Enhanced non-contact detection of fentanyl via silicon nanowire arrays pre-concentration on a portable ion mobility spectrometer, Pittcon Conference and Exposition. February 2024

- DeGreeff, L.E.; Fulton, A.C.; Jayawardana, T.; Giordano, B.C.; Vaughan, S.R.; Forte, L.; Holness, H.; Furton, K. Non-Contact Detection of Fentanyl and Other Synthetic Drugs, AAFS Conference. February 2024.
- Jayawardana, T.; DeGreeff, L.E; Fulton, A.C.; Giordano, B. A pre-concentrating polymer coating for improved detection of Fentanyl and Fentanyl analogs, AAFS Conference. February 2024.
- Fulton, A.C,; Vaughan, S.R.; DeGreeff, L.E. Non-contact detection of fentanyl by ion mobility spectrometer, Pittcon Conference and Exposition, March 2023.