Pacific Northwest
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PNNL-35433

Performance Assessment of Field-Portable Instruments and Assays for Fentanyl and Fentanyl-Related Compounds

Test Report

September 2023

Ashley Bradley
Kai-For Mo
Angela Melville
Danielle Saunders
Sonja Jensen
Vivian Lin
Jennifer Mobberley
Charles Doll
Daniel Orton
Alejandro Heredia-Langner
Rich Ozanich

U.S. DEPARTMENT OF ENERGY

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Pacific Northwest National Laboratory
Richland, Washington 99354

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Acronyms and Abbreviations

ASTM	ASTM International (formerly American Society for Testing and Materials)
ATR	Attenuated Total Reflectance
CDC	Center of Disease Control and Prevention
CL	Confidence Level
CRM	Certified Reference Material
DHS S&T	Department of Homeland Security Science and Technology Directorate
GC/MS	gas chromatograph(y)/mass spectrometer(y)
FTIR	Fourier transform infrared spectrometer
HCl	hydrochloride
HPMS	high-pressure mass spectrometer
IMS	ion mobility spectrometer
LCB	Lower Confidence Bound
LC/MS	liquid chromatograph(y)/mass spectrometer(y)
PNNL	Pacific Northwest National Laboratory
mg	milligram
mL	milliliter
MPA	mobile phase A
MPB	mobile phase B
ng	nanogram
POD	Probability of Detection
RFI	Request for Information
RSD	relative standard deviation
SAVER	System Assessment and Validation for Emergency Responders
SERS	surface enhanced Raman spectroscopy
TM	test module
µg	microgram
v/v	volume by volume ratio
W/W%	weight by weight percentage

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1.0 Executive Summary

This project addressed the growing issue with synthetic opioids entering the United States by providing an understanding of the baseline performance of equipment and assays using a wide range of fentanyl-related compounds and mixtures as test samples that reflect real-world mixture compositions seen in seizures. This contributes to the goal of improving the performance of field detection systems that use spectral library matching, colorimetric assays, and immunoassays to identify synthetic opioids. The outcomes of this project benefit first responders and front-line personnel by providing the knowledge needed to adapt and optimize current protocols for existing deployed handheld devices (e.g., combining different technology class detection capabilities when feasible) and to inform future procurements of equipment to improve the safety of both first responders and the public.

The U.S. Department of Homeland Security (DHS) Science and Technology Directorate (S&T) announced a multiphase study in early 2021 to understand current performance of and improve detection of field chemical detection instruments. Following development of ASTM International standards for testing of field chemical detection equipment for synthetic opioids, DHS S&T, in partnership with Pacific Northwest National Laboratory (PNNL), funded a library expansion and performance assessment of field portable detection equipment, colorimetric assays, and immunoassays that are commonly used by first responders. PNNL conducted systematic evaluation following the ASTM standard specification that defined sample composition (ASTM E3243-21)[1] and test method (ASTM E3290-21)[2] that provided testing guidance for each technology class.

As it is difficult for many vendors to access and work with opioids and other controlled substances, the library expansion leveraged PNNL's illicit drug testing and evaluation capabilities. During this phase, fourteen mass-based (GC/MS, HPMS, IMS) and optical spectroscopy (FTIR and Raman) instruments underwent a library build effort where up to 50 compounds including fentanyl, fentanyl analogs, and other emerging synthetic compounds were scanned by each vendor's instrument so they could be added to their on-board libraries for all their instruments. The updated reference libraries will be available at no cost to tribal, local, state, or federal agencies who own these instruments upon vendor request.

For the performance assessment phase, compounds were organized into test modules (TMs) per the ASTM standards. TMs included 14 different analogs of pure fentanyls (TM1); fentanyl compounds in "real world" mixtures of potentially interfering compounds such as cutting agents, semi-synthetic opioids, and other drugs (TM2 and TM3); commonly encountered drugs and substances not containing fentanyl (TM4, which served as a false positive check); and precursor compounds and compounds commonly associated with the synthesis of fentanyl related compounds (TM5).

This assessment focused on the ability to detect/alarm for fentanyls at pure, 10%, and 1% percent composition in powder mixtures. Analysis included both trace (≤ 1 µg total sample) and bulk (> 1 µg and < 10 mg total sample) testing of fentanyl compounds. Direct readout results were recorded for each sample and summarized as a pass or a fail. Vendors for mass-based and optical instruments were given the option to participate in reachback assessment to compare direct readout results obtained by PNNL and their expert analysis of the data, commonly referred to as vendor reachback. Vendor reachback results required a one-hour turnaround time. Based on the ASTM International standard, the minimum performance criteria

were established to correspond to a lower confidence bound of 0.85 at 80% confidence level for the detection probability of target compounds in each test module. This equates to requiring instrument or assay to pass at least 28 of 31 test samples. In this assessment, 14 unique samples comprised each test module, and each test sample was tested 2-3 times resulting in a total number of 31 tests[†].

The results of our performance testing indicated that multiple field detection technologies are needed to confidently detect the presence of fentanyls in unknown samples, especially in mixtures and at fentanyl concentrations at or below 10%. While mass-based and optical instruments readily detected pure fentanyl compounds, the mass-based instruments performed better with mixtures where fentanyls were at 10% of the total sample composition or below at trace amounts (≤ 1 ug total sample) in mixtures. Conversely, spectroscopy systems on the whole outperformed mass-based instruments for fentanyl precursor detection (TM5) and had lower false positive alarms in samples that did not include fentanyl analogs (TM4). Colorimetric assay performance was poor for both pure fentanyl samples and mixtures. Immunoassays performed better at detecting fentanyls in TM2 and TM3 10% and 1% mixtures than at detecting pure fentanyls. However, no colorimetric assays or immunoassays could reliably detect trace amounts (≤ 1 µg total sample) of fentanyl analogs, but many were close to achieving minimum acceptable performance criteria.

No product tested in any technology class was able to meet minimum performance criteria for 1% fentanyl mixtures using trace amounts (≤ 1 µg) of sample, which is representative of what might be expected from swabs and swipes from surfaces such as door handles, baggies, and suspects, when only a barely visible or invisible level of contamination is present. A summary of findings for the 17 instruments and eight assays tested is given in Figure 1 below.

[†] TM4 for the Griffin G510 was tested at 14 samples, one time only due to time limitations.

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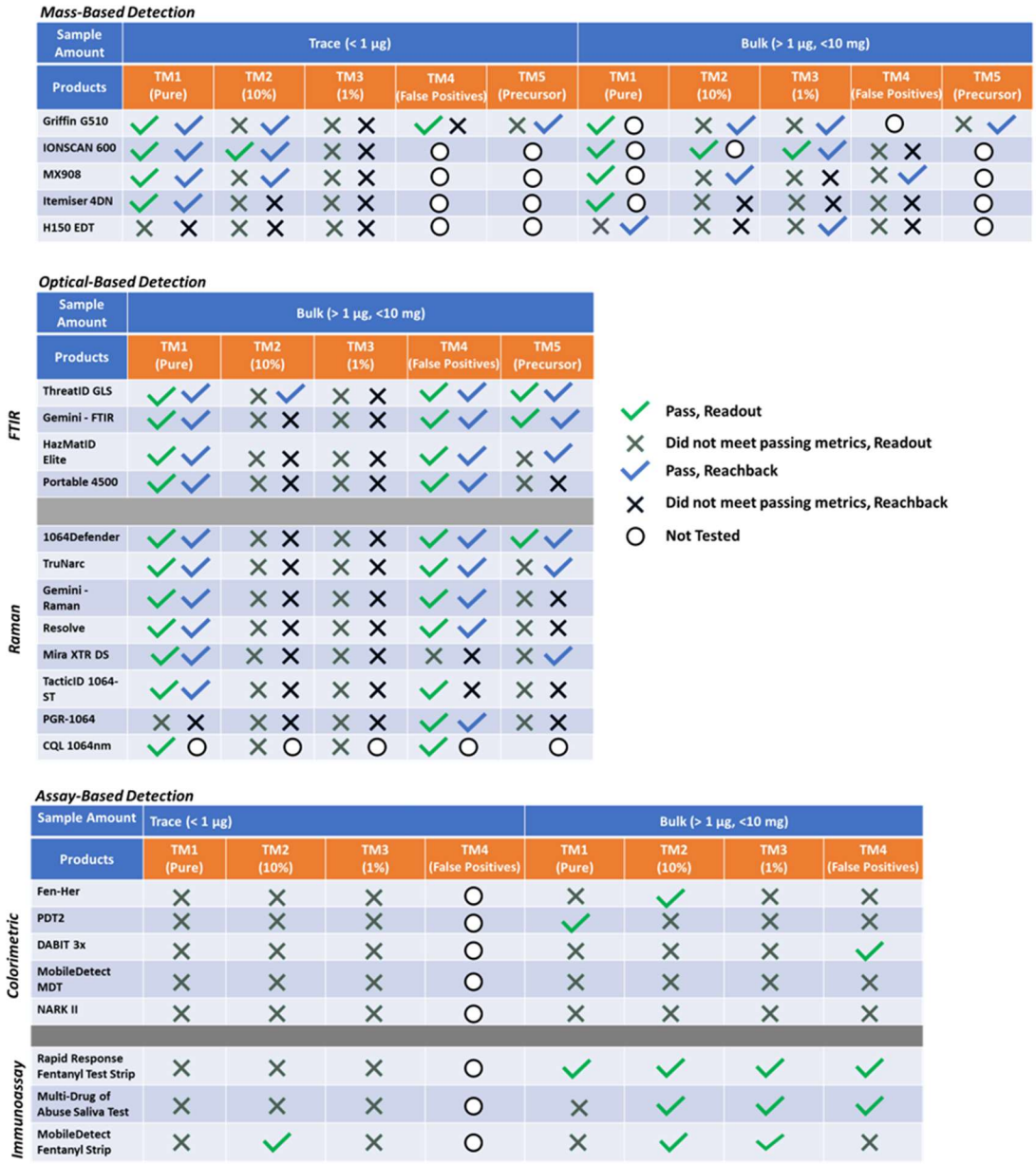

Figure 1. Summary of Results for Performance Assessments Based on ASTM Standard Specification (ASTM E3243-21)

The performance assessment results are organized by technology type and sample amount tested. Based on the ASTM international standard, the minimum passing metric corresponds to a lower confidence bound of 0.85 probability of detection at 80% confidence level for the detection of target compounds in each test module (checkmarks). Anything below this did not meet the minimum metric (cross mark). Direct readout results that passed are in green and reachback results that passed (if applicable) are in blue. For mass-based instruments, if any TM passed trace amounts (≤ 1 µg total sample), it was assumed that bulk amounts would pass so, in general, no direct readout measurements were taken and no reachback was performed (see section 4.1.1 for details). The Griffin G510 was measured at trace amounts for TM4 with a total of 14 samples, while the other mass-based systems and assays were tested at bulk amounts with 31 tests for the TM. For the assays, no reachback was performed as no electronic data was generated.

2.0 Background

Since the 1990s, the United States has been in an opioid crisis caused first by the overprescription of opioid pain relievers, and now largely caused by the ever-increasing abuse and overdose deaths due to illicit synthetic opioids, such as fentanyl and its analogs. According to the Centers for Disease Control and Prevention (CDC), from 2013- 2019 the rates of death involving synthetic opioids increased 1,040%[3], and these trends show no sign of abating. Synthetic opioids have a much higher potency than other opioid classes leading them to become highly abused substances. While these substances can be obtained through medical prescriptions, the illicit manufacturing of synthetic opioids by clandestine laboratories and distribution is a growing concern. The opioid epidemic has impacted first responders who regularly encounter the compounds, placing them at risk for incidental and potentially unknown exposure to potent synthetic opioids. Methods for detecting and distinguishing opioids are crucial considering that visual inspection cannot differentiate analogs and lethal doses can be smaller than a grain of table salt if inhaled or ingested. It is of critical importance that a responder understands the limitations of the opioid detection technology they use and whether it can be employed for confident identification of unknown substances encountered in the field.

Following the July 2021 publication of ASTM International standards for testing field chemical detection equipment for synthetic opioids[4], the DHS S&T funded a library expansion and performance assessment effort as described below.

Respondents to a Request for Information were first selected by S&T based on a variety of factors, including frequency of use by first responders, such as hazardous materials teams and law enforcement. After vendor selection, S&T entered into cooperative research and development agreements (CRADAs) with each vendor. The CRADA between S&T and each vendor stipulated that expansion of on-board libraries of illicit substances would be offered by S&T, but vendors had to agree to update their libraries for current and future users at no cost, as well as submit to testing to the ASTM standards. In this arrangement, both vendors and end-user benefit from S&T's efforts to improve emergency response, safety, and detection technology.

PNNL conducted testing following ASTM standard specification (ASTM E3243-21)[1] and test method (ASTM E3290-21)[2] that defined sample types and testing guidance, respectively.

Seventeen instruments and eight assays were tested:

- One gas chromatograph/mass spectrometer (GC/MS),
- One high pressure mass spectrometer (HPMS),
- Three ion-mobility spectrometers (IMS),
- Three Fourier-Transform Infrared (FTIR) spectrometers,
- One dual FTIR/Raman spectrometer,
- Eight Raman spectrometers,
- Three immunoassays,
- Five colorimetric assays.

Prior to the performance assessment phase, mass-based and optical spectroscopy instrument vendors participating in this project underwent a library build effort where up to 50 compounds,

including novel psychoactive substances such as fentanyl, fentanyl analogs and other emerging synthetic compounds, were measured (Appendix A). Compounds not already in the instrument library were measured at PNNL on an instrument provided by the vendor unless requested otherwise. These updated reference libraries are available upon request at no cost to first responders and other end users of these instruments.

Vendors were expected to include these compounds in their libraries prior to returning the instrument to PNNL for the performance assessment, as this library build-out included the fentanyl analogs that comprise the test samples prescribed in the ASTM standard specification.

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3.0 Experimental

3.1 ASTM Standards

The ASTM standard test method E3290−21[2] (available for a fee from ASTM) was used as guidance for sample preparation and data collection best practices for each class of detection technology. Analysis with mass-based instruments and assays included both trace and bulk testing. Trace amounts/levels are defined as ≤ 1 µg and bulk amounts/levels are defined as > 1 µg and < 10 mg of the total test sample or mixture.

Samples were first measured in trace amounts. If the direct readout result passed in trace, bulk amounts were not tested. However, if the readout result failed at trace amounts, bulk amounts were measured until there was either a passing result or the maximum concentration suggested by the vendor was reached.

Vendors were briefed on the performance test sample compositions and testing approach prior to PNNL receiving the instrument. Prior to testing, the vendor conducted a virtual training session.

Many, but not all, vendors offer 24/7/365 reachback support for a fee. Reachback support is a service that allows end users to contact the vendors' subject matter experts for interpretation of instrument direct readout spectra and likely presence of dangerous compounds. In this performance assessment, sample scans from the instrument were sent to the product vendor's designated contact for analysis and the response recorded. Subject matter experts at PNNL analyzed all collected data prior to sending for reachback to ensure the data did not show evidence of operator error or instrument malfunction. While not all instruments offer 24/7/365 reachback technical support, all vendors were given the option to participate in that portion of the performance assessment since reachback often improves identification/detection. The assays were excluded from reachback analysis along with the Rigkau CQL 1064nm (due to scheduling/time limitations).

ASTM Standard Specification E3243−21[1] defines the test modules used for assessing instrument and assay performance, including detailed descriptions of the compounds and compositions specified. Test module 1 (TM1) required a yes/no response of whether a "fentanyl or fentanyl-related compound" was present with pure compounds (≥ 95% concentration by mass). Test modules 2 and 3 (TM2 and TM3) required an indication if a fentanyl or fentanyl-related compound was present within mixtures containing 10% and 1% of the target fentanyl compounds by mass, respectively. For all TM1-3 test samples, correct identification of the compound was not required to pass.

The intent of test module 4 (TM4) was to ensure the instrument or assay did not generate a false-positive result when no fentanyl or fentanyl-related compounds were present. The sample was considered to have a passing result whether the compounds present were correctly identified or not, as long as no fentanyl or fentanyl related compound was indicated as being present.

Unlike TMs 1-4, test module 5 (TM5) required a correct identification of precursor compounds and compounds commonly associated with the synthesis of fentanyl related compounds.

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Due to the limitation and primary focus of end-users of certain technologies, colorimetric assays and immunoassays were not included in TM5 testing as they do not typically provide specific chemical identification of compounds such as those in TM5. Vendors of HPMS and IMS instruments were given the option to opt out of TM5, which they all chose to do as their products were not specifically designed to identify these types of compounds (though HPMS and IMS are technically capable of doing so). It was expected that the GC/MS, FTIR and Raman instruments would be able to detect and identify TM5 compounds and those vendors were not given the option to opt out of TM5 testing.

Direct readout results were recorded for each sample/concentration and summarized as a pass or fail in Appendix E. Results are summarized in Section 4.1. Vendors were given the option to participate in a reachback assessment to compare direct readout results obtained from PNNL testing and the vendor analysis of the data. Vendors participating in this assessment were provided reachback files and the requirement to return results within one hour of receipt. For samples that required testing of both trace and bulk concentrations, files of each were sent for reachback analysis. Files were sent at random (e.g., not in sequential order or necessarily from the same TM). The number of files returned, and the date/time data were sent was coordinated between PNNL and the vendor support team. If vendors failed to respond one hour after receipt of data, the sample was counted as a failed result. Reachback statistical results are summarized in Section 4.0 with the pass/fail analysis in Appendix E.

The testing methodology employed in this work consists of conducting 31 pass/fail tests in each TM. This allows demonstration that the minimum performance criterion of 0.85 lower confidence bound (LCB) on probability of detection at 80% confidence level (CL) can be achieved even with up to three failed results per TM. With four or more failed results, it is not possible to meet the minimum performance requirements in 31 tests, and testing for the TM can cease.

For cases where reachback was available, testing continued, even with four or more failed results recorded for the instrument, as long as fewer than four failed reachback results were recorded. Any direct readout results recorded after four direct failures were not included in the statistical analysis for that metric.

3.2 Statistical Approach to Assess Performance

Some instruments or detectors that produce binary outcomes, such as 0/1, detected/not detected, can be characterized by their statistical performance. The statistical measure of interest is the probability that the instrument will perform as expected during a given test. An estimate of performance can be calculated as the number of times the instrument performs correctly (detects a substance when present, for example), divided by the total number of tests performed, $\hat{p} = x/N$, where $\hat{p}$ is the estimate of the probability of detection, x is the number of tests where the instrument performed correctly, and N is the total number of tests performed. The calculation of $\hat{p}$ alone may not be sufficient to characterize performance of the instrument or system because the same value of $\hat{p}$ can be obtained under widely different sets of experimental results, and the value of $\hat{p}$ does not reflect the total number of tests performed.

An LCB on the value of $\hat{p}$ provides a better picture of performance. An LCB represents a lower limit at a certain confidence level on the probability of detection, $\hat{p}$, and it reflects the quantity and quality of results obtained during testing. The LCB allows users or regulatory agencies to prescribe a minimum level of performance that needs to be demonstrated by manufacturers or service providers, since the *estimated* probability of detection will always be at least as high as the LCB at the confidence level given using the experimental results found.

In this work, following the methodology in the ASTM standards, an LCB of 0.85 with an 80% confidence level (CL) represents the minimum acceptable performance level, ensuring that $\hat{p} \geq 0.85$. Notice, however, that $\hat{p}$ is only an *empirical estimate* of the true probability of detection (POD), which is unknown. The testing methodology used in this work ensures only that a sufficiently large number of experiments is conducted to state if a lower limit on the POD estimate, $\hat{p}$, is high enough given the observed results. The confidence level expresses the percent of times the true POD is at least as high as the LCB calculated if the experiments are conducted many times in independent trials and the modelling assumptions are satisfied. The 0.85 LCB with 80% CL were selected as minimum acceptable values as a compromise between testing for a sufficiently high level of performance using a practical number of experiments.

A testing scenario that meets or exceeds the minimum level of performance consists of conducting 31 experiments and observing three or fewer failures, where a failure occurs, for example, when an instrument fails to detect a target substance when it is present. Within a TM, compounds were assumed to be equivalent for purposes of calculating probability of detection. For this reason, 31 experiments were conducted spanning every compound within a TM two to three times, assuming results to be applicable to the set as a whole. Due to time constraints, TM4 for the Griffin G510 was only tested with 14 experiments, with each compound tested once. In this case, only one failure was allowed to reach the minimum level of performance. The ASTM standard E3243-21 provides details on the order and number of tests that should be conducted for the compounds in each TM.

The total number of tests that need to be conducted for each TM (31 in most cases) is sufficient to determine whether the minimum LCB/CL performance can be achieved, while still allowing for three or fewer failures to occur. Different numbers of total tests may also be used that allow for determining if the minimum 0.85/80% LCB/CL can be met. Table 1 summarizes the number of tests that need to be performed as a function of LCB/CL and the number of failed tests.

Table 1. Number of tests that need to be conducted as a function of LCB/Confidence level and number of failed tests. Most testing was conducted using 31 test samples for each test module (note that testing ceased for a test module once four failures were obtained, as the minimum acceptable performance criteria of 0.85/80% is not attainable with more than three failures in 31 tests).

Number of Failed Tests	LCB/Confidence Level		
	0.95/95%	0.90/90%	0.85/80%
0	47	14	4
1	79	31	14
2	107	44	23
3	134	57	31

Table 1 shows that the minimum performance level of 0.85/80% LCB/CL can be obtained by performing 4 tests when observing no failures, 14 tests and up to a single failed result, 23 tests and two or fewer failed results, or 31 tests and three or fewer failed results. This table was used as the basis for choosing to perform 31 or 14 tests for each TM. While it is possible to achieve the minimum LCB/CL of 0.85/80% with only four tests, it is advisable to perform as many tests as practical. A larger number of tests allows for a more accurate estimate of POD (resulting in an LCB closer to the estimated POD), while providing some flexibility in the number of failed

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results that can be tolerated while still meeting the LCB/CL requirements. Table 1 also shows that a large number of tests and very few failed results are needed to achieve high LCB/CL levels.

A more complete presentation of the statistical methodology employed here can be found in the ASTM standard (E3243-21)[1] that forms the basis for this work.

3.3 Test Samples

The components of each test sample for each test module are listed in 6.0Appendix B. In test modules 1, 4 and 5 where pure controlled substances were tested, vials containing 1 mg of compound were ordered from Cayman Chemical or Sigma Aldrich for ease of testing with mass-based systems (see Appendix C**Error! Reference source not found.**). In those cases, one mL (0.791 g) of methanol was added to the vial and dilutions were created and measured as appropriate. Material from those labeled as “large vial” (i.e., > 1 mg amounts) were used for preparing mixtures for TM2-4 and/or for pure analysis in TM1, 4, and 5 for FTIR and Raman measurements. Vendor information of the materials used for pure analysis and to create the mixtures are listed in Appendix C.

3.3.1 Mixture Component Analysis

Test mixtures were prepared in accordance with the ASTM Standard Specification E3243-21[1] test module samples. Samples range from binary mixtures up to complex, eight-component mixtures. Each 250 mg mixture was prepared in 2 mL screw top vials. All weights were recorded using a Mettler Toledo Deltarange XPR205DR balance with a 0.01/0.1 mg readability and 0.015 mg repeatability, which is within the ASTM standard stipulation to use a balance that can measure < 2 mg with <10% uncertainty.

The percentage of each compound within a mixture was calculated by its mass and further investigated by LC/MS analysis. These results are shown in Appendix D. LC/MS parameters are summarized in section 3.4.1.1. Per the ASTM Standard Test Method E3290-21[2], the relative standard deviation (RSD) of each component within a test mixture was calculated by dividing the standard deviation of the relative concentration by its mean and multiplying by 100%. For these samples, the error and RSD values were allowed to be up to 20% before a sample needed to be remade. Certain samples such as the one containing dipyrone sodium salt (sample 2.12) and some fentanyl-related compounds (samples 3.4 and 3.14) proved to be more challenging to mix in and analyze via LC/MS.

3.3.2 Sample Mixing and Division

Based on previous studies conducted at PNNL, a LabRAM I acoustic mixer (Resodyn Acoustic Mixers, Butte, MT) was used to homogenize test samples (Figure 2). Mixing parameters used 95 G for two minutes. Samples were made in 1.5 mL autosampler vials that were placed in a custom-made insert.

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Figure 2. LabRAM acoustic mixer used for homogenizing mixtures.

Immediately following mixing, the 250 mg samples were divided into ~10 mg “child” vials that were dedicated to individual technologies/instruments. Three vials were prepared for LC/MS analysis, eight vials for mass-based technologies, five vials for FTIR analysis, one for Raman analysis and two for colorimetric/immunoassays. Material was added to all vials in random order. The remaining material (if any) was placed back into the original “parent vial”.

3.4 LC/MS Verification of Test Sample Mixture Composition

Three vials of the mixed and divided sample (~10mg) for all TM2, 3 and 4 mixtures were dedicated for LC/MS analysis. These three vials were meant to represent the concentrations of the remaining sample vials.

3.4.1 Methodology

3.4.1.1 LC/MS Method Parameters

Sample analysis was performed using an Agilent 6460 Triple Quadrupole LC/MS that was equipped with an Agilent 1290 Liquid Chromatography system. Separation of controlled substances and non-target compounds was achieved using an a Phenomenex Kinetex C18 column (2.6 µm particle size, 100 x 2.1 mm). Mobile phase A (MPA) was water with 0.1% formic acid and mobile phase B (MPB) was acetonitrile with 0.1% formic acid. Samples were introduced to the instruments in 2 µL aliquots using an autosampler with mobile phase flow rate at 0.325 mL/min. The mobile phase was set to 98% MPA for the first 4 minutes, increased linearly to 95% MPB over 13 minutes and held at the same composition for 5 minutes. The mobile phase was then ramped down to 98% MPA over 2 minutes and held for another 6 minutes. The 30-minute total run time ensured the elution of analytes and equilibration of the column. The mass spectrometer was operated in positive ion mode. Nitrogen was used as the drying gas at 200 °C, and the sheath gas (nitrogen) was set at 250 °C where the flow rates of gas were 5 and 11 L/min, respectively. The skimmer and fragmentation voltages were both constant, at 65 and 135 V, respectively. Spectra were recorded over a range of 50 to 1000 m/z at a scan rate of 1.03 scans/s. All data was processed using MassHunter Workstation Software Qualitative Analysis version B.07.00.

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3.4.1.2 Preparation of Standard Calibration Curves and Test Samples

All standard calibration curve standards and test samples were prepared in a 50/50 volume per volume (v/v) water and methanol solution. Each calibration standard curve was prepared with the same composition as the test samples. For instance, the TM2 sample #1 calibration standard consisted of 10% fentanyl HCl, 40% mannitol and 50% acetaminophen. A stock solution of each calibration standard was prepared at 100 µg/mL and diluted to the desired seven concentrations for the LC/MS study. Insoluble compounds or non-detectable LC/MS compounds were replaced by the same amount of 50/50 water/methanol. For example, in TM3 sample #1, the 15% microcrystalline cellulose and 3% stearic acid constituents were replaced by 50/50 methanol/water solution. Three sample replicates (LC1, LC2, and LC3) were tested for each TM sample. Each sample was reconstituted in 50/50 (v/v) methanol/water to give a 5.5 mg/mL solution and diluted to the desired concentration (around the mid-point of the calibration standard curve). Any insoluble components of the mixture were settled on the bottom of the vial so as not to be added into the final analyte solution.

3.4.1.3 Compound Stability Considerations

Heroin was known to degrade in the methanol/water solution to give its corresponding 6-acetylmorphine (loss of one acetyl group) and even morphine (loss of two acetyl groups)[5] . To calculate the actual amount of heroin in the sample mixture, we also included the amount of these two compounds if they were present in the LC/MS data. Due to insolubility of certain non-target samples, true concentrations in solution may not be the same as those calculated by w/w prior to being dissolved. In fact, samples with a large amount of insoluble material may have slightly higher percentages of fentanyl in the 1 and 10% mixture as a result. In cases where it was available, certified reference material (CRM) dissolved in methanol was used for the standard's target compound. These samples were tested once unless there was an error with the instrument or noted otherwise.

3.4.1.4 Homogeneity of Prepared Mixtures as Determined by LC/MS

All components in each mixture test sample of each TM were quantified. Per ASTM E3290-21[1], mean concentration and relative standard deviation (RSD) were calculated for the three LC/MS replicate samples. Several cutting agents or non-fentanyl compounds were above the recommended 20% RSD as described below for each TM that contained mixtures. It should be noted that metrics to assess completeness of mixing were estimated at the time of drafting and publishing the ASTM standards. These standards would benefit from updates to metrics of completeness of mixing to match current laboratory capabilities.

- TM2: Methoxyacetyl fentanyl (26% RSD; sample 2.4) and acetyl fentanyl (38% RSD, 2.12)
- TM3: Fentanyl citrate (53% RSD; 3.4), methoxyacetyl fentanyl (32% RSD; 3.9), cyclopropyl fentanyl (35% RSD; 3.11), fentanyl citrate (37% RSD; 3.14). Heroin is present in these samples also and had high RSD, although in other mixtures with heroin RSDs are within the 20% guidance.
- TM4: Six test samples in TM4 were mixtures. %RSD values were <20% for 14 components and 20-35% for 4 components.

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Overall, mixing completeness was good given the complex nature of the samples and small test sample amounts and considering the intended use of testing non-laboratory field equipment. Inspection of the performance data does not indicate the higher %RSD values impacted outcomes (i.e., failures were not due predominantly to these sample numbers). Four of five mass-based products tested passed the above high %RSD TM2 samples and all mass-based products passed the high %RSD samples in TM3.

For TM2 (10% fentanyl mixtures), the percent composition by weight of fentanyl ranged from 8.28%-13.00%. For TM3 (1% fentanyl mixtures), the percent composition by weight of fentanyl ranged from 0.60%-1.38%. These differences were not observed to impact performance outcomes for any products and represent challenges in the preparation of these types of complex mixture samples.

3.5 Products Tested

A summary of specifications for each instrument tested is presented at the beginning of each technology subsection. More information may be found on the vendor websites and in DHS System Assessment and Validation for Emergency Responders (SAVER) market survey reports and assessment reports (if applicable).

Training sessions were coordinated with each vendor prior to operating their instrument(s) to ensure sampling techniques were followed in the same way first responders are trained. Technologies are presented in three categories: mass-based, spectroscopic-based, and assay-based. Information for each product is summarized below in Table 2.

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Table 2. List of Products Tested

Vendor	Product	Technology
Mass-Based Detection Products		
FLIR	Griffin G510	GC/MS
908 Devices	MX908	HPMS
Leidos	Portable H150E	IMS
Rapiscan	Itemiser 4DN	IMS
Smiths Detection	IONSCAN 600	IMS
Optical Detection Products		
Smiths Detection	HazMatID Elite	FTIR
Agilent	Portable 4500 FTIR	FTIR
RedWave Technology	ThreatID GLS	FTIR
Thermo Scientific	Gemini	FTIR/ Raman
Agilent	Resolve	Raman
Chemring	PGR-1064	Raman
Metrohm	Mira XTR DS	Raman
Metrohm	TacticID-1064 ST	Raman
Rigaku	CQL 1064nm	Raman
Thermo Scientific	TruNarc	Raman
Thermo Scientific	1064Defender	Raman
Assays		
DetectaChem	MobileDetect Pouch-Multi -Drug Test	Colorimetric assay
Field Forensics	Fen-Her	Colorimetric assay
Field Forensics	DABIT 3x	Colorimetric assay
Mistral Group	Fentanyl 2 PDT	Colorimetric assay
Sirchie	NARK II Fentanyl Reagent	Colorimetric assay
DetectaChem	MobileDetect Fentanyl Test Strip	Immunoassay
Confirm Biosciences	Multi-drug of abuse saliva test	Immunoassay
BTNX	Rapid Response Fentanyl Test Strip	Immunoassay

3.5.1 Gas-Chromatography/Mass-Spectrometry

The FLIR Detection Griffin G510 was the only portable GC/MS system tested for this performance assessment as no other GC/MS vendors expressed interest in participating. Assessments were conducted on all five test modules using direct injections except for the liquid precursors (i.e., propionic anhydride, 2-chloroethylbenzene, 2-bromoethylbenzene, propionyl chloride, aniline, and pyridine) in TM5 which were tested using the vapor method.

Instrument specifications and a user assessment may be found in the Field Portable Gas Chromatograph Mass Spectrometers DHS SAVER report[6] and the vendor website.

Solid samples were dissolved in methanol and serially diluted to desired concentration. Test modules 1-3 were tested in trace amounts (≤ 1 µg total sample) using direct injection. Bulk amounts (> 1 µg and < 10 mg) were used for samples that did not pass trace amounts. If a test sample passed trace, it was assumed that higher concentrations (i.e., bulk amounts of powder)

would also yield a positive result, if properly diluted, and therefore were not tested. Per vendor guidance, a 20 µg total sample concentration, which included fentanyls and other sample components, was injected for bulk concentrations (i.e., 2 µg of fentanyls were present in the test sample for TM2 samples and 0.2 µg fentanyls for TM3 samples).

TM4 samples were tested at 100 ng to assess potential false-positive results. Due to time constraints only 14 tests were conducted for the Griffin G510 instead of 31 for TM4 (see Table 1 for the number of tests that allow for testing if the minimum LCB/CL can be achieved). For this TM, it should be noted that a false-positive result for a trace amount of sample would not necessarily result in a false-positive result for a bulk amount of sample.

Vapor detection mode was utilized for the liquid samples in TM5 (samples 5.7, 5.9, 5.10, 5.12, 5.13, 5.14). In these cases, ~5 mL of the pure liquid was transferred to a 20 mL scintillation vial and the sample probe held over the opening of the vial for a few seconds.

Each new test sample was followed by a methanol blank to minimize carryover. For vapor testing, the instrument was cleared after each sample.

3.5.2 High-Pressure Mass Spectrometry and Ion-Mobility Spectrometry

The 908 Devices MX908 was the only portable high-pressure mass spectrometer (HPMS) tested. Instrument specifications can be found on the 908 Devices website or in the Explosives Trace Detectors (ETDs) Market Survey Report[7].

Three ion mobility spectrometers (IMS) were tested as part of this assessment: the Smiths Detection IONSCAN 600, the Leidos H150E, and the Rapiscan Itemiser 4DN.

Samples were dissolved in methanol and serially diluted until the desired concentration was reached. The solution was then deposited onto manufacturer-supplied coupons and allowed to dry before initiating the measurement. Blanks and calibrations were conducted as instructed during the training session. Bulk amounts were used for samples that did not pass trace amounts. If a test sample passed in trace amounts, it was assumed that higher concentrations (i.e., bulk amounts of powder) would also yield a positive result, if properly diluted, and therefore were not tested.

TM5 samples were not tested for on any of the HPMS and IMS systems.

Based on guidance from the vendors, bulk amounts of up to 10 µg of total sample were introduced into the IONSCAN 600 and Itemiser 4DN systems. 20 µg of total sample was the maximum bulk amount suggested by the 908 Devices and Leidos vendors.

TM4 was conducted at the bulk levels based on which total sample concentration resulted in a passing test for TM3. Therefore, 10 µg of sample was introduced to both the IONSCAN 600 and Itemiser 4DN instruments while 20 µg samples were used for the MX908 and H150E instruments.

3.5.3 Fourier-Transform Infrared Spectroscopy

Four Fourier-Transform Infrared (FTIR) spectrophotometers were tested: the Agilent Portable 4500, RedWave Technology ThreatID, Smiths Detection HazMatID Elite and the Thermo Scientific Gemini. The Thermo Scientific Gemini is a dual FTIR/Raman system, therefore both

FTIR and Raman were tested and are reported here. Technical specifications for the FTIR instruments can be found in the Portable Infrared Spectroscopy Chemical Detectors Assessment Report[8] or on the hyperlinks of the vendor's name. Note that not all products were available at the time of the assessment.

Sample mixtures from TM2 through TM4 were shaken to mix prior to analysis. 1-2 mg sample was placed onto the instrument's ATR crystal and then compressed with the anvil for analysis for each test module sample. In TM5, a few drops of the liquid samples were added to the ATR crystal and measured as instructed during training.

After each measurement the ATR crystal was cleaned with ethanol and wiped dry to prevent cross-contamination. Calibrations and background scans were conducted based on vendor guidance. Spectral resolution, spectral range and sample scans were not changed after testing began.

Direct readout results were recorded as instructed by vendors to account for differences in display (user interface). The methods used are briefly described below for each FTIR instrument.

Agilent Portable 4500

The top result (highest scoring match) on the direct readout was recorded for all samples. Additionally, the top result was recorded from the first three residual analysis screens. For TM2 and 3, the sample was considered a pass if the top score for the direct readout and three residuals listed a fentanyl or fentanyl-related compound. For TM4, the sample was considered a failure if a fentanyl or fentanyl analog was listed as a top match for those steps.

Redwave Technologies ThreatID

For test samples containing only one component, the compound identified in the "primary search" screen was recorded. For test sample mixtures, all listed compounds were recorded from the "automated mixture search" screen display along with an approximation of their percentage contribution to the mixture composition.

Smiths Detection HazMatID Elite

For all test samples, compounds listed on the direct readout (up to five total) were recorded. Compounds that were listed on the readout screen but were not one of the primary five identified were not used as results.

Thermo Scientific Gemini

The color bar of the screen indicated the type of match by the instrument: a green screen indicated a single compound match, a yellow-orange screen indicated a similarity match, a blue screen indicated a mixture of compounds, and a red screen indicated no matches found. All compound results and color bars were recorded for each sample. Fentanyl or a fentanyl analog in any type of match (single compound, mixture, and similarity) was considered a positive result for fentanyl. This method for recording was used for both the FTIR and Raman data inspection for direct readout.

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3.5.4 Raman Spectroscopy

Eight handheld Raman spectrometers were included in the performance assessment (one of which was the dual Gemini system). This includes the Thermo Scientific Gemini, Agilent Resolve, Chemring PGR-1064, Metrohm Mira XTR DS, Metrohm TacticID-1064 ST, Rigaku CQL 1064nm, Thermo Scientific TruNarc and Thermo Scientific 1064Defender.

The Mira XTR DS, TruNarc and Gemini have 785 nm excitation lasers while the Resolve uses an 830 nm laser and the PGR-1064, CQL 1064nm, 1064Defender and TacticID-1064 ST have 1064 nm lasers. Additional technical information for each Raman spectrometer can be found in the DHS SAVER Handheld Raman Spectrometers Market Survey Report[9] or in the hyperlinks. Note that not all products were available at the time of the SAVER reports.

Test samples were prepared by placing 9-10 mg of material from the respective TM parent vials in 2 mL Wheaton 33 low extractable borosilicate glass vials with PTFE lined screw caps for Raman analysis. Samples were shaken by hand to mix immediately prior to analysis. All measurements were collected in “point-and-shoot” mode. Ambient light was minimized by reducing the overhead lights in the direct area of testing.

Laser power, scan time and other parameters were modified per vendor guidance prior to testing. If, after parameter optimization, a scan did not complete within five minutes, the measurement was recorded as a failure (usually due to the presence of high fluorescent compounds in a mixture). Three spots at different sample locations were measured per test sample to account for differences in particles and laser optimization. If one of the three replicates indicated a positive result, that specific test module sample passed.

A calibration standard provided by the vendor was measured for each instrument as an operational check prior to measuring test samples.

Direct readout results were recorded as instructed by vendors to accommodate differences in algorithms and readout functionality. The methods are briefly described for each Raman instrument below.

Agilent Resolve

The Agilent Resolve reported a single or a mixture of compounds with highest priority on the main screen with mixture compounds also shown.

Chemring PGR-1064

The Chemring PGR 1064 was connected to a computer station and controlled via its software. All the compounds listed on the screen were recorded.

Mira XTR DS

The Mira XTR DS has two tabs for results: mixture and identification. Both tabs were reviewed, and compounds listed on both screens were recorded.

Metrohm TacticID-1064 ST

The TacticID-1064 ST displayed a single compound match if one was found in its library. If no initial match was found, a mixture analysis was performed, and all compounds listed on the mixtures screen were recorded.

Rigaku CQL 1064nm

The Rigaku CQL 1064 identified either one compound or listed the possible mixtures with estimated percentage composition for each. All compounds identified were recorded for each sample.

Thermo Scientific TruNarc

The TruNarc reports one compound on the results screen by highest priority: controlled substances first, precursors or chemicals second, and cutting agents or diluents third. Each compound reported was recorded for each sample.

Thermo Scientific 1064Defender

The color bar of the screen indicated the type of match by the instrument: a green screen indicated a single compound match, a yellow-orange screen indicated a similarity match, a blue screen indicated a mixture of compounds, and a red screen indicated no matches found. All compound results and color bars were recorded for each sample.

3.5.5 Colorimetric Assays and Immunoassays

Five colorimetric assays and three immunoassays were included in the performance assessment for TMs 1-4. The colorimetric assays include the MobileDetect Multi-Drug Test (MDT), Field Forensics Fen-Her, Field Forensics DABIT 3x, Mistral Group Presumptive Drug Test (PDT2) fentanyl reagent, and the Sirchie Nark II Fentanyl Reagent. The immunoassays include the Confirm Biosciences SalivaConfirm Saliva Drug Test, the Detectachem fentanyl test strip and the BTNX Rapid Response Fentanyl Test Strip.

Samples were dissolved in methanol for colorimetric assays and in water for immunoassays. Test samples amounts and methods of introduction to the assay were conducted per vendor guidance following discussions with them. These are briefly summarized below. For the colorimetric assays, samples were made at concentrations so that 980 ng of total sample was deposited for trace testing and 100 µg deposited for bulk analysis. Immunoassay testing was done at concentrations of 980 ng/mL for trace and 100 µg/mL for bulk. Bulk amounts were used for samples that did not pass trace amounts. If a test sample passed at trace levels, it was assumed that higher concentrations (i.e., bulk amounts of powder) would also yield a positive result, if properly diluted, and therefore were not tested.

To prevent bias in color detection of colorimetric assays, the two analysts responsible for conducting the testing independently evaluated the color response and whether that indicated a pass or fail. In cases where the two analysts disagreed over a pass/fail result or if a compound in TM1-3 failed to detect fentanyl or showed a false-positive result for fentanyl in TM4, a third researcher, not involved in the testing, was provided photographs of the assay along with the vendor comparison response chart. These results were combined to make the final pass/fail determination.

MobileDetect Multi-Drug Test (MDT)

Samples were pipetted onto each of the test pads and air dried. Once dried, the manufacturer's instructions were followed to break reagent vials and the color chart referenced to determine the response.

Field Forensics Fen-Her

For this test kit, samples were pipetted onto a glass slide and air dried. The sample kit pen was used to swab the dried area and the result recorded.

Field Forensics DABIT 3x

Based on manufacturer guidance, samples were pipetted onto a piece of Teflon and air dried. Teflon was used per test kit manufacturer's instructions due to the sticky glue used on the test pads.

Mistral Group PDT2

Samples were pipetted directly into the pouch and the color change noted.

Sirchie Nark II Fentanyl Reagent

Samples were pipetted onto a small piece of Kimwipe and air dried before placing into the pouch. The color change was recorded.

Confirm Biosciences SalivaConfirm Saliva Drug Test

The samples were pipetted onto the sponge end of the supplied collection sticks and then placed into the plastic collection box. Results were read and recorded after 10 minutes.

DetectaChem Fentanyl Test Strip

Strips were immersed in a sample solution at trace and bulk concentrations for ~60 seconds and interpreted within several minutes. The vial of buffer included in the kit was not used.

BTNX Rapid Response Fentanyl Test Strip

Strips were immersed in a sample solution at trace and bulk concentrations for ~10 seconds and interpreted after several minutes.

4.0 Results

4.1 Direct Readout

Results from the direct readout of the instruments and assays are summarized below. For TM1-3 a positive result indicated the presence of any fentanyl or fentanyl analog. A positive result for TM4 indicated that no fentanyl or fentanyl analogs were present. For TM5 a positive result was one that correctly identified the tested compound. Pass/Fail responses for each instrument and sample are listed and described further in Appendix E. TM5 readout and reachback reports were analyzed together, and therefore are listed in the Reachback section (4.2). Table entries shaded in red indicate cases where testing ceased, or the number of failed tests would prevent achieving the minimum acceptable performance criteria of 0.85 LCB/80% CL.

For mass-based technologies and assays where trace and bulk sample amounts were tested in TMs 1-3, samples were first tested in trace levels (≤ 1 µg) and then bulk if the trace level did not detect a fentanyl compound. If a fentanyl compound was identified at trace, the test sample was not measured at bulk since it was assumed that higher concentrations would also cause a fentanyl detection.

4.1.1 Mass-Based Instrument Performance

Statistical analyses of the direct readout results are summarized below in their given categories (mass-based instruments, optics-based instruments, and assays). More information regarding these metrics is described in 3.2. Table 1 summarizes the relationship between number of test sample failures and LCB/CL values shown in the tables below.

Results from Table 3 show that few technologies can meet the minimum POD/CL of 0.85/80% as stated in ASTM E3243-21[1] for all TMs. Four out of the five mass-based products demonstrated acceptable performance for TM1 with the HPMS exhibiting slightly lower performance based (0.85 LCB/90% CL) as compared to GC/MS and IMS. Only one IMS had acceptable performance for TM2 (and with high performance metrics). All mass-based technologies fell below minimum performance requirements for TM3 and TM4, except the GC/MS. For TM4 testing, IMS and HPMS gave higher false positives than GC/MS for non-fentanyl containing samples. The Griffin G510, MX908 and IONSCAN 600 were the only mass-based technologies tested to demonstrate acceptable performance for two of the four test modules. As a note, when a mass-based instrument was able to identify fentanyl in trace amounts (≤1 µg total sample), that sample was not measure at bulk levels, but assumed it would be a pass. These cases are shown as a green highlight in Table 3 without a LCB/CL. A more detailed discussion of the results for each TM is given below.

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Table 3. Lower confidence bound, expressed in decimal form (left value), and corresponding confidence level, shown as percentage (right value), for Test Modules 1 through 4 obtained using GC/MS, HPMS and IMS. The table identifies the specific instruments used and contains results for trace and bulk samples as applicable. The minimum acceptable performance level consists of LCB ≥ 0.85 with CL ≥ 80%. Acceptable performance, based on ASTM Standards, for each test module is highlighted in green. Table entries highlighted in red indicate cases where testing was ceased, as the number of failed tests would prevent achieving the minimum acceptable level.

Method	**GC/MS**		**HPMS**		**IMS**					
Product	Griffin G510		MX908		IONSCAN 600		H150E		Itemiser 4DN	
Test Module	Trace	Bulk	Trace	Bulk	Trace	Bulk	Trace	Bulk	Trace	Bulk
TM1 (Pure)	0.92/ 95%	[c]	0.85/ 90%	[a]	0.92/ 95%	[c]			0.92/ 95%	[c]
TM2 (10%)		[a]		[a]	0.92/ 95%	[c]				
TM3 (1%)						[a]				
TM4 (FP)	0.90/ 90% [b]	NT	NT		NT		NT		NT	

NT: Not tested. In general, false-positive (TM4) testing was done at the bulk sample amounts (> 1 µg of sample) but not at trace for these instruments. While the Griffin G510 passed TM4 at trace concentrations, it cannot be assumed that bulk concentrations would not yield false-positives for fentanyl.

[a] Overall results indicate this system is likely to achieve minimum performance criteria with bulk sample amounts though no assignment of LCB/CL was possible because of mixed test events (trace and bulk).

[b] 14/14 trace samples passed (time limitations prevented testing 31 samples)

[c] If an instrument detected fentanyl in trace amounts, it was assumed it would pass bulk.

4.1.1.1 TM1: Pure Fentanyl and Fentanyl Analogs

The mass-based instruments (GC/MS, HPMS and IMS) were typically able to detect pure fentanyls in TM1 in trace amounts (≤ 1µg).

The Griffin G510 portable GC/MS identified a fentanyl compound in all samples at trace amounts.

The MX908 HPMS identified a fentanyl compound in 29 of the 31 samples at trace amounts. The instrument was unable to identify a fentanyl-related compound at trace and bulk for two of the samples.

Of the IMS systems, the IONSCAN 600 and Itemiser 4DN were able to detect fentanyl in all 31 samples at trace levels. The H150E identified fentanyl in 11 samples before reaching four failures. Of those 11 samples, seven passed at trace levels.

4.1.1.2 TM2: 10% Fentanyl Mixtures

While the Griffin G510 was not able to achieve the minimum performance level for all 31 trace samples (no more than four failures), the five samples that failed to be detected in trace amounts were all detected in bulk amounts. Because it is assumed that if a mass-based

detection instrument can detect trace amounts, it can also detect bulk amounts of a compound (via dilution of bulk to trace), TM2 bulk had a passing result for all 31 samples.

The MX908 passed 30 of the 31 tests, with a fentanyl-related compound detected at a trace level for 22 samples. Eight of the nine samples that failed trace levels detected a fentanyl in bulk amounts. Therefore, the instrument did not pass trace levels, but the results indicate this system is likely to achieve minimum performance criteria with bulk sample amounts though no assignment of LCB/CL was possible because of mixed test events (trace and bulk).

The Itemiser 4DN detected a fentanyl-related compound in 28 of the samples (16 at trace levels). In one instance (sample 2.6 rep 2), the instrument detected a fentanyl at trace amounts, but did not detect a fentanyl in the bulk. Due to this and the three additional failures in bulk analysis, it was unable to be assumed that the instrument would pass metrics at bulk levels with the mixed test events.

The IONSCAN 600 IMS passed all tests with mixtures introduced to the instrument at trace amounts while the H150E had a passing result for 10 samples (9 of those at trace) before reaching four failures.

4.1.1.3 TM3: 1% Fentanyl Mixtures

The Griffin G510 detected fentanyl in nine of the 1% mixtures (all at bulk amounts) prior to reaching four failures. The MX908 HPMS passed eight samples with one of those at trace levels. Of the IMS systems, the Itemiser 4DN detected a fentanyl compound in 13 samples (10 at trace), the H150E 27 samples (20 at trace levels) and the IONSCAN 600 30 of the 31 samples (23 trace).It is believed that the IONSCAN 600 would be able to achieve the minimum acceptable 0.85 LCB/80% CL at bulk levels since 23 of the samples achieved a passing result at trace levels and seven of the eight samples tested at bulk identified a fentanyl within the mixtures.

4.1.1.4 TM4: False Positive Test

For TM4 the Griffin G510 was tested at trace levels (100 ng total sample) and only 14 sample tests were conducted due to time limitation. The rest of the mass-based instruments were tested to the 31 samples at bulk amounts determined from the concentration needed in TM3 to detect a fentanyl or from the vendor recommendation. These concentrations are listed in Section 3.5.2.

The Griffin G510 portable GC/MS passed every sample (14 out of 14), meaning that fentanyl was not detected in any of the TM4 samples which were all comprised of non-target compounds.

Each of the HPMS and IMS systems identified a fentanyl in four or more of the false-positive tests resulting in cessation of testing. The MX908 had an appropriate response for 14 samples before reaching the four-failure limit while the H150E passed one test, the Itemiser 4DN passed four tests, and the IONSCAN 600 12 tests.

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4.1.2 Optics-Based Instrument Performance

Table 4. Lower confidence bound, expressed in decimal form (left value), and corresponding confidence level, shown as percentage (right value), for Test Modules 1 through 4 obtained using FTIR and RAMAN. The table identifies the specific instruments used and contains results for trace and bulk samples as applicable. The minimum acceptable performance level consists of LCB≥0.85 with CL≥80%. Acceptable performance, based on ASTM Standards, for each test module is highlighted in green. Table entries shaded in red indicate cases where testing was ceased, as the number of failed tests would prevent achieving the minimum acceptable level.

FTIR				
Product	HazMatID Elite	Portable 4500	ThreatID	Gemini FTIR
Test Module				
TM1 (Pure)	0.92/95%	0.92/95%	0.92/95%	0.92/95%
TM2 (10%)				
TM3 (1%)				
TM4 (FP)	0.92/95%	0.92/95%	0.90/90%	0.92/95%

	RAMAN							
Product	Gemini Raman	Resolve	PGR-1064	Mira XTR DS	TacticID-1064 ST	CQL 1064 nm	TruNarc	1064 Defender
Test Module								
TM1 (Pure)	0.92/95%	0.92/95%		0.85/90%	0.92/95%	0.92/95%	0.90/90%	0.92/95%
TM2 (10%)								
TM3 (1%)								
TM4 (FP)	0.92/95%	0.92/95%	0.92/95%		0.85/90%	0.91/95%	0.92/95%	0.92/95%

Each of the FTIR instruments and seven of the eight Raman instruments passed TM1 at or above the minimum performance level (Table 4). This was also the case for TM4. However, none of the optical instruments passed TM2 or TM3 where fentanyls comprised only 10% and 1% of the sample mixtures, respectively.

4.1.2.1 TM1: Pure Fentanyl and Fentanyl Analogs

The optical instruments performed well for TM1, with all FTIR instruments and the majority of Raman instruments performing at 0.92/95%. All but one Raman instrument performed at the acceptable performance level of LCB ≥ 0.85 with CL ≥ 80%.

Specifically, five of the Raman instruments were able to detect a fentanyl-related compound in all samples: the Gemini, Resolve, TacticID-1064 ST, CQL 1064 nm and the 1064Defender. The

PGR-1064 had a passing result for 18 samples, the Mira XTR DS 29 samples and the TruNarc 30 samples.

Likewise, all FTIR instruments (HazMatID Elite, Portable 4500, Threat ID GLS and Gemini) were able to detect a fentanyl compound in each of the 31 samples in TM1.

4.1.2.2 TM2: 10% Fentanyl Mixtures

All optical instruments reached the four-failure limit for TM2 samples with no major differences between FTIR and Raman performance.

Two portable FTIR instruments did not detect fentanyl in any of the mixtures before reaching four failures: the HazmatID Elite and the Gemini. The Portable 4500 indicated a fentanyl-related compound was present in ten samples via. direct readout prior to reaching four failures. The Threat ID GLS passed two test samples before reaching four failures.

Of the Raman instruments, the Mira XTR DS and TruNarc identified a fentanyl compound in four of the sample mixtures before reaching the four-failure limit. The Resolve, TacticID-1064 ST and CQL 1064nm each passed three tests before reaching four failures. The Gemini, PGR-1064, and 1064Defender each alarmed for a fentanyl in one sample before reaching four failures.

4.1.2.3 TM3: 1% Fentanyl Mixtures

It is important to note that of the samples in TM3, four (3.1, 3.4, 3.7, 3.8, and 3.10) were colored and exhibited higher fluorescence than other samples from TMs 1-5. For those samples, when possible, the laser intensity was decreased when collecting a spectrum.

Each of the FTIR and Raman instruments failed the first four samples except for the Mira XTR DS. The Mira XTR DS indicated the presence of a fentanyl-related compound in one sample before reaching the four-failure limit.

4.1.2.4 TM4: False Positive Test

All the FTIR instruments and the majority of Raman instruments had high scores in this TM with very few erroneous matches to fentanyl across the technologies.

Three out of the four FTIR systems passed all 31 tests of TM4. The Threat ID GLS falsely identified fentanyl in only one of the samples, for a score of 30 out of 31.

Five of the Raman systems passed each of the 31 tests including the Gemini, Resolve, PGR-1064, TruNarc and the 1064Defender. The TacticID-1064 ST falsely identified a fentanyl-compound in two of the tests. The Mira XTR DS passed ten of the false-positive samples before reaching four failures by incorrectly identifying fentanyl in fentanyl-free samples. The Rigaku CQL 1064nm passed all 27 of the samples that were included for direct readout. Due to instrument malfunction, four of the samples were unable to be tested.

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4.1.3 Assay-Based Performance

All five colorimetric assays failed to detect trace amounts (≤ 1 µg) of fentanyl in each of the four test modules (Table 5). Three different assays were able to detect bulk amounts (> 1 µg < 10 mg) of 10% and 1% fentanyl mixtures, but a different product was the only one to not generate high false positives (TM4).

Only one immunoassay was able to detect trace amounts of fentanyl across TM1-3. The MobileDetect Fentanyl Test Strip indicated the presence of fentanyl in all 31 tests at trace amounts in TM2. Immunoassays significantly outperformed colorimetric assays for TM2-TM4 samples with two products able to pass all three TMs. However, no immunoassays had acceptable performance with pure fentanyls. Two colorimetric assays passed TM1 but failed the other three test modules.

Table 5. Lower confidence bound, expressed in decimal form and corresponding confidence level, shown as percentage, for Test Modules 1 through 4 obtained using a variety of assays. The table identifies the specific instruments used and contains results for trace and bulk samples as applicable. The minimum acceptable performance level consists of LCB ≥ 0.85 with CL ≥ 80%. Acceptable performance for each test module is highlighted in green. Table entries shaded in red indicate cases where testing was ceased, as the number of failed tests would prevent achieving the minimum acceptable level.

Method	**Colorimetric assays**									
Product	MobileDetect MDT		Fen-Her		DABIT 3x		PDT2		NARK II	
Test Module	Trace	Bulk	Trace	Bulk	Trace	Bulk	Trace	Bulk	Trace	Bulk
TM1 (Pure)								0.85/ 90%		
TM2 (10%)				0.87/ 95%						
TM3 (1%)										
TM4 (FP)	NT		NT		NT	0.92/ 95%	NT		NT	

Method	**Immunoassays**					
Product	Multi-Drug of Abuse Saliva Test		MobileDetect Fentanyl Strip		Rapid Response Fentanyl Test Strip	
	Trace	Bulk	Trace	Bulk	Trace	Bulk
Test Module						
TM1 (Pure)						[a]
TM2 (10%)		0.85/90%	0.92/95%	[b]		0.88/85%
TM3 (1%)		0.87/95%		0.91/95%		0.90/95%
TM4 (FP)	NT	0.85/90%	NT		NT	0.85/90%

NT: Not tested. False-positive (TM4) testing was done at the bulk levels that were necessary for trying to pass TM3 for these assays.

[a] Overall results indicate this assay is likely to achieve minimum performance criteria with bulk sample amounts though no assignment of LCB/CL was possible because of mixed test events (trace and bulk).

[b] If an assay detected fentanyl in trace concentration, it was assumed it would pass bulk. In this case, bulk concentrations were not measured unless a trace concentration failed.

4.1.3.1 TM1: Pure Fentanyl and Fentanyl Analogs

The colorimetric and immunoassays were inconsistent in indicating the presence of fentanyl in the pure samples with performance ranging from near zero detections to 0.85/90% LCB/CL.

The PDT2 colorimetric assays passed 29 out of the 31 tests for the pure fentanyl compounds at bulk levels. The MobileDetect MDT, Fen-Her and NARKII colorimetric assays indicated the presence of a fentanyl in 24 samples before reaching the four-failure limit. The DABIT 3x assay failed the first four samples causing testing to cease.

The Rapid Response Fentanyl Test Strip passed 27 out of 31 trace samples, missing the 0.85 LCB/80% CL limit, but passed two out of four bulk samples tested. It was assumed that the samples that passed in trace amounts would also pass bulk and therefore would achieve the LCB/CL acceptable criteria. The other immunoassays were unable to reach the minimum criteria at trace or bulk levels.

4.1.3.2 TM2: 10% Fentanyl Mixtures

Colorimetric and immunoassays also performed inconsistently with TM2 samples. Immunoassays had higher scores than the majority of the colorimetric assays.

The Fen-Her colorimetric assay indicated the presence of a fentanyl compound in 30 of the 31 tests. The NARK II passed 16 samples, the MobileDetect MDT six samples, and the PDT2 four samples before reaching the four-failure limit. The DABIT 3x assay had a negative result for fentanyl with the first four mixtures, causing testing to cease.

The three immunoassays each indicated the presence of a fentanyl compound in all 31 samples. The Multi-Drug of Abuse Saliva Test indicated the presence of fentanyl in 22 samples at trace levels and nine at bulk. The MobileDetect Fentanyl Strip passed all samples at trace levels. For the Rapid Response Fentanyl Test Strip, 23/27 trace samples passed. The eight samples (four failed and four untested) all passed in bulk. LCB/CL values shown in Table 5 are for just bulk testing results.

4.1.3.3 TM3: 1% Fentanyl Mixtures

The colorimetric assays performed poorly with TM3 1% fentanyl test mixtures while the immunoassays had a high performance of 0.85/90% LCB/CL or higher.

The NARK II colorimetric assay suggested the presence of a fentanyl compound in four of the test mixtures before reaching the four-failure limit. The PDT2 passed 3 tests before failing out of the TM while the MobileDetect MDT and Fen-Her each had a positive result for one sample. The DABIT 3x assay had a negative result for fentanyl with the first four mixtures, causing testing to cease.

Of the immunoassays, the Saliva Drug Test indicated the presence of a fentanyl compound in 30 of the 31 samples all at bulk amounts. The other immunoassays passed all 31 tests.

The MobileDetect Fentanyl Strip passed 5/17 trace samples. The other 26 (failed and untested samples) all passed at the bulk level. LCB/CL values shown are for just bulk testing results. The Rapid Response Fentanyl Test Strip indicated the presence of fentanyl in 7/18 trace samples. The other 24 (failed and untested samples) all passed at bulk amounts.

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4.1.3.4 TM4: False Positive Test

The LCB/CL for the colorimetric assays ranged from 0.51/80% to 0.92/95%. The DABIT 3x correctly did not indicate fentanyl in any of the 31 fentanyl-free samples for a score of 31 out of 31. The PDT2 passed seven samples, the NARK II 12 samples, the MobileDetect MDT 21 samples and the Fen-Her 25 samples before failing a total of four samples and causing testing to cease.

Of the immunoassays, two products had an LCB/CL of 0.85/90% while one did not pass the TM performance limits with an LCB/CL of 0.65/80%. The Saliva Drug Test and the Rapid Response Fentanyl Test Strip passed 29 of the 31 false-positive tests. The DetectaChem Fentanyl Test Strip passed 12 samples before reaching the four-failure limit.

4.2 Reachback

Products that provide 24/7/365 reachback support were included in the reachback assessment, which is a paid service that customers use to get rapid expert guidance interpreting their Raman or FTIR spectrometer results, often during an active response situation. Vendors who do not provide 24/7/365 reachback support were given the option to participate in reachback analysis under the parameters listed in section 3.1. Due to time constraints as the project reached its endpoint, reachback for the Rigaku CQL 1064 nm was not able to be assessed, though it is a feature offered by the vendor.

Trace and bulk (if applicable) sample scans were sent to mass-based vendors regardless of whether they passed or failed readout.

For Raman systems, spectra from three different sampling locations were sent to reachback and identified as the same sample by PNNL. If reachback correctly identified one of the three spots, it was considered a pass.

Statistical analysis of the reachback performance is shown in the tables below. Pass/Fail responses are listed and described further in Appendix E.

Table 6. Lower confidence bound, expressed in decimal form and corresponding confidence level, shown as percentage, for Reachback results for Test Modules 1 through 4 obtained using GC/MS, HPMS and IMS. The table identifies the specific instruments used and contains results for trace and bulk samples as applicable. The minimum acceptable performance level consists of LCB ≥ 0.85 with CL ≥ 80%. Acceptable performance for each test module is highlighted in green and those that were unable to achieve the minimum acceptable criteria in red.

Method	**GC/MS**				**HPMS**		**IMS**			
Product	Griffin G510		MX908		IONSCAN 600		H150E		Itemiser 4DN	
Test Module	Trace	Bulk	Trace	Bulk	Trace	Bulk	Trace	Bulk	Trace	Bulk
TM1 (Pure)	0.92/ 95%	NT[a]	0.92/ 95%	[b]	0.92/ 95%	NT[a]		0.93 /90%	0.92/ 95%	NT[a]
TM2 (10%)	0.90/ 90%	0.88/ 80%	0.94/ 90%	0.85/ 90%	0.92/ 95%	NT[a]				
TM3 (1%)		0.85/ 90%				[c]		[c]		
TM4 (FP)		NT	NT	0.92/ 95%	NT		NT		NT	

NT: Not tested.
[a] All readout results were in trace concentrations, therefore bulk reachback was not applicable.
[b] If an instrument failed to detect fentanyl in trace concentration, then the bulk concentration of that sample was measured and sent for reachback though no assignment of LCB/CL was possible due to low sample numbers.
[c] Overall results indicate this system is likely to achieve minimum performance criteria with bulk sample amounts though no assignment of LCB/CL was possible because of mixed test events (trace and bulk).

Reachback significantly improved performance outcomes for GC/MS and HPMS (Table 6). Only one IMS product (H150E) improved with reachback. GC/MS passed three of the test modules (vs. only TM1 and TM4 in direct readout), though it was not able to pass TM3 using trace amounts of sample. No other products successfully detected the 1% fentanyl mixtures in TM3, although the IONSCAN 600 was just below the acceptable limits (0.85/80%).

Reachback did not have a significant impact on improving detection of 10% and 1% fentanyl mixtures in TM2 and TM3 although reachback with the ThreatID did pass TM2. Reachback significantly improved identification of pure precursor compounds in TM5 with the GC/MS (bulk samples were not tested). One additional FTIR and two additional Raman achieved acceptable performance levels (Table 7,8).

Table 7. Lower confidence bound, expressed in decimal form and corresponding confidence level, shown as percentage, for reachback results for Test Modules 1 through 4 obtained using FTIR and RAMAN. The table identifies the specific instruments used and contains results for trace and bulk samples as applicable. The minimum acceptable performance level consists of LCB ≥ 0.85 with CL ≥ 80%. Acceptable performance for each test module is highlighted in green. Table entries shaded in red indicate cases where testing ceased, or the number of failed tests would prevent achieving the minimum acceptable performance criteria.

Method	**FTIR**			
	HazMatID Elite	Portable 4500	Threat ID GLS	Gemini FTIR
Test Module				
TM1 (Pure)	0.92/95%	0.92/95%	0.92/95%	0.92/95%
TM2 (10%)			0.90/90%	
TM3 (1%)				
TM4 (FP)	0.90/90%	0.92/95%	0.92/95%	0.92/95%

Method	**RAMAN**						
	Gemini Raman	Resolve	PGR-1064	Mira XTR DS	TacticID-1064 ST	TruNarc	1064 Defender
Test Module							
TM1 (Pure)	0.92/95%	0.85/90%		0.90/90%	0.92/95%	0.90/90%	0.92/95%
TM2 (10%)							
TM3 (1%)							
TM4	0.92/95%	0.90/90%	0.90/90%			0.92/95%	0.92/95%

Table 8. Lower confidence bound, expressed in decimal form and corresponding confidence level, shown as percentage, for direct readout and reachback results for Test Module 5 obtained using GC/MS, FTIR and RAMAN. The table identifies the specific instruments used and contains results for trace and bulk samples as applicable. The minimum acceptable performance level consists of LCB ≥ 0.85 with CL ≥ 80%. Acceptable performance for each test module is highlighted in green while table entries shaded in red indicate where testing ceased, or the number of failed tests would prevent reaching the minimum acceptable limit. TM5 included precursors and compounds related to fentanyl synthesis.

Method	GC/MS	
Product	Griffin G510	
Test Module	Trace	Bulk
TM5 readout		
TM5 reachback	0.85/90%	0.87/85%

Method	FTIR				RAMAN						
Product											
Test Module	HazMat ID Elite	Portable 4500	Threat ID GLS	Gemini FTIR	Gemini Raman	Resolve	PGR-1064	Mira XTR DS	Tactic ID-1064 ST	TruNarc	1064 Defender
TM5 readout			0.92/95%	0.85/90%							0.85/90%
TM5 reachback	0.92/95%		0.92/95%	0.92/95%				0.85/80%		0.88/80%	0.92/95%

4.2.1 TM1: Pure Fentanyl and Fentanyl Analogs

Reachback provided slightly improved results for all but one mass-based system, where the improvement was significant compared to the readout response value. The H150E reachback identified a fentanyl or fentanyl-related compound in 31 samples when looking at trace and bulk responses combined (improving from 11 correct direct readout results). Reachback was able to identify the presence of fentanyl in 26 of the 31 trace samples and all 21 of the bulk scans sent.

Performance remained the same in most of the optics-based instruments since many passed all 31 samples for direct readout. The Mira XTR DS improved from 29 to 30 correct results and no difference was observed for the PGR-1064 or TruNarc direct readout vs. reachback results. The Resolve performance decreased from 0.92/95% LCB/CL in direct readout to 0.85/90%.

4.2.2 TM2: 10% Fentanyl Mixtures

Reachback analysis results for the Griffin G510, IONSCAN 600 and PGR-1064 were consistent with the number of samples that passed in the direct readout phase.

In all, reachback analysis was able to identify a fentanyl compound in each of the 31 sample mixtures for the Griffin G510, MX908 and IONSCAN 600. The MX908 reachback improved identification from 30 to 31 samples. For the remaining mass-based systems, 27 out of 29

samples passed for the H150E[‡] (an improvement from 10 positive readout responses) and zero for the Itemiser 4DN (a decrease from 28 readout results) when adding trace and bulk results.

Reachback analysis improved the HazMatID Elite performance from zero positive tests to 26, the ThreatID GLS from two to 30, the Gemini FTIR from zero to 11, the Gemini Raman from one to seven, the Mira XTR DS from four to six, the TruNarc from four to nine, and the 1064Defender from two to three positive tests.

Reachback decreased performance of the Portable 4500 from 10 to six tests and the Resolve from three positive readout results to one positive reachback response[§]. This is also the case with the TacticID-1064 where three readout results indicated the presence of fentanyl, but only one positive reachback analysis. The PGR-1064 reachback analysis resulted in the same number of positive results as the readout: one.

4.2.3 TM3: 1% Fentanyl Mixtures

For the IONSCAN 600 27/31 trace samples passed reachback but fell one failure short of the minimum performance criteria. Six of the seven bulk samples also passed reachback. Overall results indicate this system is likely to achieve minimum performance criteria with bulk sample amounts though no assignment of LCB/CL was possible because of mixed test events (trace and bulk).

27/30 trace samples passed reachback for the H150E (1 trace sample was mistakenly not sent to reachback and, if positively identified, would have reached the minimum performance criteria of 0.85/80%). 9/11 bulk samples that failed in trace amounts during direct readout testing passed reachback. Overall results indicate this system is likely to achieve minimum performance criteria with bulk sample amounts though no assignment of LCB/CL was possible because of mixed test events (trace and bulk).

For the optical instruments, reachback analysis was unable to detect a fentanyl in any of the 31 samples for all instruments other than the HazmatID Elite, ThreatID GLS and Mira XTR DS. Reachback identified fentanyl in one of the HazmatID Elite samples, two of the ThreatID GLS samples and four of the Mira XTR DS samples. Those were all improvements from the direct readout results.

4.2.4 TM4: False Positive Test

Reachback identification was not consistent in improving or decreasing performance across the mass-based systems.

Reachback analysis increased the number of passing results for the MX908 from 14 to 31 and four to 27 samples for the Itemiser 4DN. The IONSCAN 600 had 12 passing samples for both readout and reachback analysis. Reachback analysis decreased sample performance for the Griffin G510 from 14 passes to 3.[**] TM4 did not pass for reachback due to responses being sent

[‡] Two bulk samples and four trace samples were accidently omitted from being sent for reachback. Therefore, the reachback analysis for the H150E should be 27 out of 29 for bulk.
[§] Some failures can be attributed to the reachback response being over the allotted one-hour time limit.
[**] The minimum performance level of 0.85/80% can be obtained by performing 14 tests and up to a single failure; this occurred with the fourth G510 sample on reachback.

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after the allotted one-hour limit but would have passed otherwise. The H150ETD decreased from one to zero samples.

The results between readout and reachback results were typically consistent for optical instruments with most instruments remaining at the same number of samples, or increasing one to two more positive results from reachback, but there was a significant decrease for the Mira XTR DS with 10 passing readout results to five passing reachback results and 29 passing readout results for the TacticID-1064 to 19 reachback samples.

4.2.5 TM5: Pure Fentanyls and Precursors

A summary of readout vs. reachback results is shown in Table 8 with all pass/fails for each sample listed in Appendix E.

Liquid precursors and solvents (samples 5.7, 5.9, 5.10, 5.12, 5.13, 5.14) were tested using the vapor method for the Griffin G510. Samples 5.1, 5.3, 5.4 and 5.5 were correctly identified at 100 ng (trace) concentrations. Samples 5.2, 5.6 and 5.8 were measured at both trace and bulk amounts but were not correctly identified. Sample 5.7 which was measured in vapor mode was also unable to be identified which contributed to the four-failure limit when readout testing ceased. Reachback was able to identify the seven samples that were measured in bulk levels (5.3, 5.8, 5.11, 5.6 rep 2, 5.8 rep 2, 5.11 rep 2, and 5.2 rep 3). This is the cause for the asterisk in the bulk readout section Table 8 and resulted in a passing LCB/CL for bulk reachback.

Table 9.TM5 Readout vs. Reachback Summary

	# Correct Results	
Instrument	Readout	Reachback
Griffin G510	4 (all trace level)	30 (trace and bulk responses)
HazMatID Elite	7	31
Portable 4500	10	10
ThreatID GLS	31	31
Gemini- FTIR	29	31
Gemini- Raman	5	17
Mira XTR DS	15	28
TruNarc	4	28
Resolve	7	3
TacticID-1064 ST	21	9
1064Defender	29	30
PGR-1064	5	5

While reachback analysis improved performance for seven of the twelve instruments that participated in TM5 testing, performance for the Griffin G510, HazMatID Elite, Mira XTR DS and the TruNarc instruments greatly improved.

Performance was unchanged for two of the FTIR instruments and one of the Raman instruments. Identification via reachback analysis was worse than the direct readout results for two of the Raman instruments.

5.0 Conclusions

As summarized in Figure 1, the results indicate that while the optical and mass-based systems have similar performance for pure fentanyl compounds, the mass-based instruments and assays were better at measuring mixtures when fentanyls are present at lower percent concentrations (10% and 1%). Colorimetric assay performance was surprisingly poor with only three of five colorimetric assays passing a single TM out of four TMs tested for each. Immunoassay results for the 14 different pure fentanyl analogs in TM1 showed inconsistent results for replicate measurements of the same analog, contributing to the inability of any immunoassay product to meet minimum performance criteria for TM1, The GC/MS achieved performance expectations for TM1 and TM4 based on direct readout results when tested with both trace (≤ 1 µg) and bulk (up to 20 µg) sample amounts, which significantly improved with reachback, resulting in all four of the five TMs meeting minimum performance criteria. The GC/MS was the only product tested to pass all TMs at trace or bulk levels when combining readout and reachback analysis. No product tested was able to meet minimum performance criteria for TM3 using trace amounts of sample.

For pure fentanyl analogs (TM1), mass-based systems were readily able to detect compounds at trace (≤1 µg) levels. However, performance at trace amounts dropped significantly for TM2 and TM3. While one IMS passed TM2 (10% fentanyls) direct readout testing, GC/MS and HPMS required reachback to pass TM2. None of the mass-based products tested passed TM3 (1% fentanyls) at trace amounts. The GC/MS passed TM4 for readout, which was conducted at trace levels of 100 ng total sample. It is assumed all mass-based technologies and assays can detect bulk amounts if trace amounts pass because it is a simple dilution. It should be noted that the amount of sample to use for TM4 (false positive check) was determined by the maximum amount of sample used in TM3 (1% fentanyls) per vendor guidance. Therefore, most TM4 testing was done using bulk amounts. HPMS with reachback passed TM4 bulk sample testing[††].

Each of the FTIR and Raman instruments failed to pass TM2 and TM3, except for one FTIR that passed TM2 with reachback support. All four FTIR and seven of eight Raman passed TM1 and had minimal false positives for TM4. There was no correlation between performance and laser wavelength for the Raman instruments (e.g, 785nm vs. 1064 nm).

Test results for colorimetric assays were generally inconsistent for fentanyl detection across products. Most immunoassays performed better in TM2 and TM3 than in TM1. No colorimetric assays or immunoassays could reliably detect trace amounts (≤ 1 microgram) of fentanyl analogs, but many were close to achieving minimum acceptable performance criteria. One of five colorimetric assays passed TM1 with bulk amounts of sample, a single different product passed bulk TM2, none passed TM3 with bulk amounts, and a single product passed TM4 with bulk amounts. The three immunoassays tested failed TM1. One product passed trace amounts of TM2, and two others passed TM2 with bulk amounts. All three products passed TM3 with bulk amounts, but none could detect trace amounts. TM4 was passed by two of the three products.

TM5 testing was conducted with all technologies except HPMS, IMS, and assays, which are not typically used for specific precursor compound identification. GC/MS and FTIR generally performed better than Raman for identifying TM5 pure precursors. GC/MS failed TM5 testing with trace amounts of sample but passed when reachback was employed. For optical

[††] All mass-based instruments and assays were conducted at bulk levels (> 1 ug) for TM4 other than the Griffin G510 GC/MS which was tested at trace concentrations.

instruments, correct identification of pure compounds in TM5 was achieved by two of four instruments. A third FTIR vendor passed TM5 with reachback. Raman generally did not perform well for TM5 specific compound identification, with only one product passing direct readout testing. Raman reachback improved performance resulting in three of eight products passing TM5.

The results shown here indicate that multiple detection technologies are needed to confidently detect the presence of fentanyl in unknown samples, particularly if they are not pure. While many FTIR and Raman systems can readily detect pure fentanyls, identify pure precursors, and have low false alarm rates with non-fentanyls, lower levels of fentanyl require a mass-based technology or immunoassay to confidently detect.

However, none of the products tested were able to detect trace amounts of fentanyl (1%) in complex mixtures, where the total mass of fentanyl available for a test instrument or assay is in the 10's of nanogram range (e.g., amounts that may be present in a surface swab or wipe). Low percent levels of fentanyl present in many illicit drugs (in both powder and pill form) present a challenge to existing hand-held detection technology and it is important for first responders to understand the limitations of different technology classes for the types of samples and levels of target chemicals they are working with. Mass-based technologies are generally recognized as good trace detection systems, but with the test samples used in TM4, GC/MS and HPMS had very low LCB/CL outcomes (see TM4 results in Table 3). Two of the three IMS systems fell below the minimum acceptable criteria indicating that these systems have higher rates of false positive detections of opioids.

With reachback, GC/MS and HPMS were able to pass TM2 (10% fentanyl) using trace amounts. When bulk amounts of sample were tested, many additional products showed improved outcomes. For mass-based technologies, bulk sample amounts were typically in the tens of micrograms range; greater amounts of sample can lead to overloading/saturating the instrument. For FTIR, bulk amounts were a few milligrams and for Raman, approximately 10 mg samples were used.

These overall results of this performance testing indicate the capabilities of field portable products regarding opioid detection and the challenges that arise with detecting low amounts of target materials within complex mixtures. Since these compounds and/or mixtures are often encountered as unknowns, sometimes in trace amounts, it is crucial for the first responder community to understand the performance envelope of their field detection tools for detecting opioids. Data collected during the library build and performance assessment for each instrument were sent to their respective vendor contacts. A major goal of this project was to provide such data in the hopes of improving detection of opioids for these field portable products and to ultimately increase safety within the first responder community and of individuals affected by illicit opioids.

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6.0 References

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Appendix A - Phase I (Vendor Library Build-Out) Compounds

NAME	CAS#
α-Pyrrolidinohexiophenone hydrochloride (α -PHP HCl)	13415-59-3
(1-(4-fluorobenzyl)-1H-indol-3-yl)(2,2,3,3-tetramethylcyclopropyl)methanone (FUB-144)	2185863-15-2
1-(1,3-benzodioxol-5-yl)-2-(butylamino)pentan-1-one hydrochloride (N-Butylpentylone hydrochloride)	17763-10-9
1-(1,3-benzodioxol-5-yl)-2-(dimethylamino)-1-butanone, monohydrochloride (Dibutylone) (bk-DMBDB HCl)	17763-12-1
1-(1,3-benzodioxol-5-yl)-2-(ethylamino)butan-1-one hydrochloride (Eutylone HCl)	17764-18-0
1-(2H-1,3-benzodioxol-5-yl)-2-(ethylamino)pentan-1-one) monochloride (N-Ethylpentylone HCl)	17763-02-9
2-Furanyl fentanyl HCl	101365-56-4
4-Anilino-N-phenethylpiperidine (4-ANPP)	21409-26-7
4-Chloroethcathinone (4-CEC) HCl	22198-75-0
4-Chloro-α- pyrrolidinovalerophenone hydrochloride (4Cl-alpha-PVP HCl)	5537-17-7
4-Fluoroisobutyryl fentanyl (FIBF) HCl	2309383-06-8
Acetyl fentanyl HCl	117332-89-5
Acrylfentanyl HCl (HClANPP)	79279-03-1
Benzyl fentanyl HCl	5156-58-1
Butyryl fentanyl HCl	1443-52-3
Carfentanil citrate	61380-27-6
cis-tramadol HCl	36282-47-0
Cocaine HCl	53-21-4
Cyclopropyl fentanyl HCl	2306825-44-3
Despropionyl p-fluorofentanyl	122861-41-0
Fentanyl citrate	990-73-8
Fentanyl HCl	1443-54-5
Heroin HCl	1502-95-0
Methamphetamine HCl	51-57-0
Methoxyacetyl fentanyl HCl	101365-54-2
Methyl(1-(4-fluorobenzyl)-1H-indazole-3-carbonyl)-L-valinate (FUB-AMB)	1971007-92-7
Methyl(2S)-2-{[1-(5-fluoropentyl)-1H-indole-3-carbonyl]amino}-3,3-dimethylbutanoate (5F-MDMB-PICA)	1971007-88-1
Methyl-2-(1-(4-fluorobutyl)-1H-indazole-3-carboxamido)-3,3-dimethylbutanoate (4F-MDMB-BUTINACA)	N/A

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Methyl2-(1-(5-fluoropentyl)-1H-indazole-3-carboxamido)-3,3-dimethylbutanoate (5F-MDMB-PINACA)	1838134-16-9
Methylenedioxy-N-benzylcathinone (BMPD) HCl	1823274-68-5
Morphine	57-27-2
N-[1-(aminocarbonyl)-2,2-dimethylpropyl]-1-[(4-fluorophenyl)methyl]-1H-indazole-3-carboxamide (AB-FUBINACA)	1185282-01-2
N-benzyl furanyl norfentanyl HCl (2-furanylbenzylfentanyl HCl)	497240-21-8
N-methyl norfentanyl HCl	24775-71-1
Norfentanyl	1609-66-1
N-phenyl-N-[1-(2-phenylethyl)piperidin-4-yl]pentanamide hydrochloride (Valerylfentanyl HCl)	117332-91-9
phenyl fentanyl (benzoylfentanyl) HCl	N/A
trans-2-(2,4-dichlorophenyl)-N-2-(dimethylamino)cyclohexyl)-N-methylacetamide, monohydrochloride (U-48800 HCl)	N/A
trans-3,4-dichloro-N-[2-(dimethylamino)cyclohexyl]-N-methyl-benzamide hydrochloride (U-47700 HCl)	N/A
trans-3,4-dichloro-N-2-(dimethylamino)cyclohexyl)-N-isopropylbenzamide (Isopropyl U-47700)	N/A
trans-3-methyl fentanyl HCl	78995-09-2

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Appendix B - TM Sample Composition

Table B.1. Test Module 1 (TM1) Samples

Sample Number	Target Compound (≥ 95%)
1.1	Fentanyl hydrochloride (HCl)
1.2	Fentanyl citrate
1.3	2-Furanyl fentanyl HCl
1.4	Methoxyacetyl fentanyl HCl
1.5	Acetyl fentanyl HCl
1.6	Cyclopropylfentanyl HCl
1.7	4-Anilino-N-phenethylpiperidine (4-ANPP)
1.8	4-Fluoroisobutyryl fentanyl HCl (FIBF HCl)
1.9	Carfentanil citrate
1.10	Acrylfentanyl HCl
1.11	Butyryl fentanyl HCl
1.12	Benzoyl fentanyl HCl (Phenyl fentanyl HCl)
1.13	Benzyl fentanyl HCl
1.14	N-methyl norfentanyl HCl

Table B.2. Test Module 2 (TM2) Samples

Sample Number	Sample Composition (10% Fentanyl mixtures)
2.1	Fentanyl HCl (10%), Mannitol (40%), Acetaminophen (50%)
2.2	Fentanyl citrate (10%), Lactose (30%), Noscapine HCl (45%), Caffeine (15%)
2.3	2-Furanyl fentanyl HCl (10%), Inositol (15%), Dipyrone sodium salt (15%), Heroin HCl (60%)
2.4	Methoxyacetyl fentanyl HCl (10%), Lactose (40%), Diphenhydramine HCl (25%), Quinine (20%), Cocaine HCl (5%)
2.5	Fentanyl HCl (10%), Mannitol (30%), Tramadol HCl (60%)
2.6	2-Furanyl fentanyl HCl (10%), Lactose (90%)
2.7	Fentanyl HCl (10%), Lactose (20%), Mannitol (45%) Procaine HCl (25%)
2.8	Cyclopropyl fentanyl HCl (10%), Inositol (50%), Acetaminophen (40%)
2.9	Fentanyl HCl (10%), Lactose (85%), Cocaine HCl (5%)
2.10	Butyryl fentanyl HCl (10%), Mannitol (20%), Heroin HCl (60%), Tramadol HCl (10%)
2.11	Fentanyl HCl (10%), Procaine HCl (15%), Tramadol HCl (75%)
2.12	Acetyl fentanyl HCl (10%), Dipyrone sodium salt (25%), Heroin HCl (10%), Tramadol HCl (55%)
2.13	Fentanyl HCl (10%), Lactose (15%), Mannitol (30%), Diphenhydramine HCl (15%), Acetaminophen (30%)

Sample Number	Sample Composition (10% Fentanyl mixtures)
2.14	Fentanyl citrate (10%), Lactose (40%), Mannitol (20%), Acetaminophen (30%)

Table B.3. Test Module 3 (TM3) Samples

Sample Number	Sample Composition (1% Fentanyl Mixtures)
3.1	Fentanyl HCl (1%), Lactose (15%), Dipyrone (15%), Mannitol (10%), Microcrystalline cellulose (15%), Stearic acid (3%), Acetaminophen (40%), FD&C Yellow #6 (1%)
3.2	2-Furanyl fentanyl HCl (1%), Inositol (20%), Mannitol (20%), Polyethylene glycol 3350 (30%), Heroin HCl (29%)
3.3	Methoxyacetyl fentanyl HCl (1%), Lactose (10%), Noscapine HCl (30%), Tramadol HCl (40%), Heroin HCl (10%), Procaine HCl (9%)
3.4	Fentanyl citrate (1%), Inositol (50%), Dipyrone (19%), Microcrystalline cellulose (25%), Stearic acid (4%), FD&C Blue #2 (1%)
3.5	Acetyl fentanyl HCl (1%), Lactose (15%), Mannitol (25%), Inositol (30%), Xylazine (20%), Noscapine HCl (9%)
3.6	Fentanyl HCl (1%), Lactose (25%), Mannitol (25%), Heroin HCl (30%), Methamphetamine HCl (5%), Caffeine (10%), Quinine (4%)
3.7	2-furanyl fentanyl HCl (1%), Lactose (78%), Microcrystalline cellulose (15%), Palmitic acid (3%), FD&C Blue #2 (3%)
3.8	Fentanyl HCl (1%), Mannitol (32%), Hydroxypropyl cellulose (15%), Ibuprofen (50%), Ferric oxide yellow (1%), FD&C Blue #2 (1%)
3.9	Methoxyacetyl fentanyl HCl (1%), Lactose (10%), Mannitol (10%), Inositol (20%), Dipyrone sodium salt (9%), Dimethyl sulfone (30%), Heroin HCl (20%
3.10	Fentanyl HCl (1%), Lactose (14%), Mannitol (14%), Microcrystalline cellulose (20%), Acetaminophen (50%), FD&C Blue #2 (1%)
3.11	Cyclopropyl fentanyl HCl (1%), Lactose (50%), Mannitol (20%), Inositol (9%), Quinine (5%), Heroin HCl (15%)
3.12	Fentanyl HCl (1%), Lactose (64%), Sucrose (20%), Diphenhydramine HCl (10%), Heroin HCl (5%)
3.13	Acetyl fentanyl HCl (1%), Lactose (74%), Heroin HCl (25%)
3.14	Fentanyl citrate (1%), Lactose (35%), Mannitol (20%), Inositol (25%), Caffeine (14%), Nicotinamide (5%)

Table B.4. Test Module 4 (TM4) Samples

Sample Number	Sample Composition (Non-fentanyl mixtures)
4.1	Heroin HCl (85%), Noscapine HCl (7%), Morphine (8%)
4.2	Methamphetamine HCl (90%), Dimethyl sulfone (10%)
4.3	Cocaine HCl (85%), Lidocaine (5%), Benzocaine (5%), Procaine HCl (5%)
4.4	Methamphetamine HCl
4.5	Heroin HCl (40%), Lactose (50%), Caffeine (10%)
4.6	Cocaine HCl (25%), Mannitol (75%)
4.7	Heroin HCl (20%), Mannitol (30%), Caffeine (30%), Quinine (20%)
4.8	U-47700
4.9	Dipyrone sodium salt
4.10	Diphenhydramine HCl
4.11	Noscapine HCl
4.12	Cis-Tramadol HCl
4.13	Acetaminophen
4.14	Caffeine

Table B.5. Test Module 5 (TM5) Samples

Sample Number	Precursor compounds (≥ 95%)
5.1	4-ANPP
5.2	N-phenethyl-4-piperidone (NPP)
5.3	Benzyl fentanyl HCl
5.4	Norfentanyl
5.5	N-methyl norfentanyl
5.6	N-Benzyl-4-piperidone
5.7	Propionic anhydride
5.8	Piperidone
5.9	2-Chloroethylbenzene
5.10	2-Bromoethylbenzene
5.11	N-phenyl-4-piperidinamine
5.12	Propionyl chloride
5.13	Aniline (free base)
5.14	Pyridine

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Appendix C Chemicals

Compound	CAS#	Description/Manufacturer
2-Bromoethylbenzene	103-63-9	Sigma Aldrich, 98%, Item# B65780, Lot# BCCF5408
2-Chloroethylbenzene	622-24-2	Sigma Aldrich, 99%, Item# C40405, Lot# MKCPO978
4-ANPP- 1mg vial	21409-26-7	Cayman, ≥ 98%, Item# 18810, Batch #0540831-28
4-ANPP- large vial	21409-26-7	Cayman, ≥ 98%, Item# 18810, Batch# 0540831-31
4-Piperidone (HCl hydrate)	40064-34-4	Cayman, ≥ 95%, Item# 21961
Acetyl fentanyl HCl- 1mg vials	117332-89-5	Cayman, ≥ 98%, Item# ISO00128, Batch# 0497037-62
Acetyl fentanyl HCl- large vial	117332-89-5	Cayman, ≥ 98%, Item# ISO00128, Batch #0497037-63
Acryl fentanyl HCl- 1mg vial	79279-03-1	Cayman, ≥ 98%, Item#19312, Batch# 0484313-32
Acryl fentanyl HCl- large vial	79279-03-1	Cayman, ≥ 98%, Item#19312, Batch# 0484313-31
Aniline	62-53-3	Sigma Aldrich, ≥ 99.5%, Item# 242284, Lot# MKCK5587
Benzyl fentanyl HCl	5156-58-1	Cayman, ≥ 98%, Item#19883, Batch# 0563059-9
Butyryl fentanyl HCl- 1 mg vial	1443-52-3	Cayman, ≥ 98%, Item#14728, Batch# 0533221-12
Butyryl fentanyl HCl- large vial	1443-52-3	Cayman, ≥ 98%, Item#14728, Batch# 0533221-11
Carfentanil citrate	61380-27-6	TRC, Item# TRC-C183475, Batch# 10-BSR-149-1
cis-Tramadol HCl- 1mg vials	36282-47-0	Cayman, ≥ 98%, Item# 15919, Batch# 0540757-47
cis-Tramadol HCl- large vial	36282-47-0	Cayman, ≥ 98%, Item# 15919, Batch# 0540757-48
Cocaine HCl	53-21-4	Cayman, ≥ 98%, Item#22165, Batch# 0617487-12
Cyclopropyl fentanyl HCl- 1 mg	2306825-44-3	Cayman, ≥ 98%, Item# 21739, Batch# 0537408-9
Cyclopropyl fentanyl HCl- large vial	2306825-44-3	Cayman, ≥ 98%, Item# 21739, Batch# 0537408-10
D-Mannitol	69-65-8	Sigma Aldrich, ≥ 98%, Item# M4125, Lot# SLCD7105
FD&C Blue #2 (indigo carmine)	860-22-0	Sigma Aldrich, 85% dye content, Item# 131164, Lot# SHBM8289
FD&C Yellow #6 (sunset yellow FCF)	2783-94-0	Sigma Aldrich, 90% Dye content, Item# 465224, Lot# SHBL0658
Fentanyl citrate- 1 mg vial	990-73-8	Cayman, ≥ 98%, Item# 22659, Batch# 0654205-4
Fentanyl citrate- large vial	990-73-8	Cayman, ≥ 98%, Item# 22659, Batch# 0654205-6
Fentanyl HCl- 1mg vial	1443-54-5	Cayman, ≥ 98%, Item# 14719, Batch# 0530926-66
Fentanyl HCl- large vial	1443-54-5	Cayman, ≥ 98%, Item# 14719, Batch# 0530926-65
Ferric oxide yellow (pigment yellow 42)	51274-00-1	Sigma Aldrich, Item 371254
FIBF HCl- 1mg vial	2309383-06-8	Cayman, ≥ 98%, Item# 19313, Batch# 0497267-6
FIBF HCl- large vial	2309383-06-8	Cayman, ≥ 98%, Item# 19313, Batch# 0497267-7
Furanyl fentanyl HCl- 1 mg vial	101365-56-4	Cayman, ≥ 98%, Item# 18705, Batch# 0537068-36
Furanyl fentanyl HCl- large vial	101365-56-4	Cayman, ≥ 98%, Item# 18705, Batch# 0537068-35
Heroin HCl	1502-95-0	Cayman, ≥ 98%, Item# 9003076, Batch# 0559234-36
Hydroxypropyl cellulose	9004-64-2	Sigma Aldrich, 99%, 20 mesh, Item# 435007, Lot# MKCK8238
Ibuprofen	15687-27-1	Sigma Aldrich, ≥ 98%, Item# I4883, Lot# SLCD1404
Lactose	10039-26-6	Sigma Aldrich, ≥ 99.5%, Item# 61339

Lidocaine HCl	6108-05-0	Sigma Aldrich, Lot# MKCK0668
Methamphetamine HCl- 1mg vial	300-42-5	Cayman, ≥ 98%, Item# 14216, Batch# 0588059-28
Methamphetamine HCl- large vial	300-42-5	Cayman, ≥ 98%, Item# 14216, Batch# 0588059-30
Methanol	67-56-1	Sigma Aldrich, ACS reagent, ≥99.8%, Lot# SHBM4672
Methoxyacetyl fentanyl HCl- 1mg vial	101365-54-2	Cayman, ≥ 98%, Item# 20782, Batch# 0537069-20
Methoxyacetyl fentanyl HCl- large vial	101365-54-2	Cayman, ≥ 98%, Item# 20782, Batch# 0537069-19
Microcrystalline cellulose M102	9004-34-6	Sigma Aldrich, Item# 435236, Lot# MKCJ3230
Morphine	57-27-2	Cayman, ≥ 98%, Item# 15464, Batch# 0597-985-18
myo-Inositol	87-89-8	Sigma Aldrich, ≥ 99%, Item# I5125, Lot# SLCD9427
N-Benzyl-4-piperidone	3612-20-2	Cayman, ≥ 98%, Item# 21962
Nicotinamide	98-92-0	Sigma Aldrich, ≥ 99.5%, Item# 72340, Lot# BCCB6184
N-Methyl norfentanyl HCl	24775-71-1	Cayman, ≥ 98%, Item# 24446, Batch #0536396-19
Norfentanyl- large vial	1609-66-1	Cayman, ≥ 98%, Item# 15899, Batch# 0612961-2
Norfentanyl- main vial	1609-66-1	Cayman, ≥ 98%, Item# 15899, Batch# 0612961-1
Noscapine HCl	912-60-7	Cayman, Batch# 0522109-6
N-phenylpiperidin-4-amine	23056-29-3	Cayman, ≥ 98%, Item# 30898
NPP	39742-60-4	Cayman, ≥ 98%, Item# 20528
Palmitic acid	57-10-3	Sigma Aldrich, ≥ 99%, Item# P5585, Lot# SLCF9094
Phenyl fentanyl HCl- 1mg vials	2309383-16-0	Cayman, ≥ 98%, Item# 22551, Batch# 0535766-16
Phenyl fentanyl HCl- large vial	2309383-16-0	Cayman, ≥ 98%, Item# 22551, Batch# 0535766-17
Polyethylene glycol 3350	25322-68-3	Sigma Aldrich, Lot# BCCC3330
Procaine HCl	51-05-8	Sigma Aldrich, ≥ 97%, Item# P9879, Lot# SLCB0477
Propionic anhydride	123-62-6	Sigma Aldrich, ≥ 99%, Item# 240311, Lot# MKCJ9763
Propionyl chloride	79-03-8	Sigma Aldrich, 98%, Item# P51559, Lot# STBJ2165
Pyridine	110-86-1	Sigma Aldrich, ≥ 99%, Item # 360570, Lot# SHBM8719
Quinine	130-95-0	TCI, Cat# S4571-HU
Stearic acid	57-11-4	Sigma Aldrich, ≥ 98.5%, Item# S4751, Batch# BCCC3431
Sucrose	57-50-1	Sigma Aldrich, ≥ 99.5%, Item# S7903, Batch# SLCF2885
U-47700- 1 mg vials	82657-23-6	Cayman, ≥ 98%, Item# 18596, Batch# 0479029-64
U-47700- large vial	82657-23-6	Cayman, ≥ 98%, Item# 18596, Batch# 0479029-63

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Appendix D - LC/MS Measurements of Mixtures

The first percentage inside the parentheses is calculated from the quantities of each compound in the 250 mg mixture. The second percentage inside the parentheses is calculated from the quantities of each compound based on the LC/MS study. Microcrystalline cellulose and ferric oxide yellow are insoluble compounds that are separated from solution. Hydroxypropyl cellulose, dimethyl sulfone, stearic acid and palmitic acid cannot be detected by the current LC/MS method that is able to analyze the majority of mixture compounds. Only the weight by weight percentage (W/W%) of the compound in 250 mg mixture were shown in the parentheses for those compounds.

Table D.1. TM2 Mixture Composition via. W/W% and LC/MS Analysis

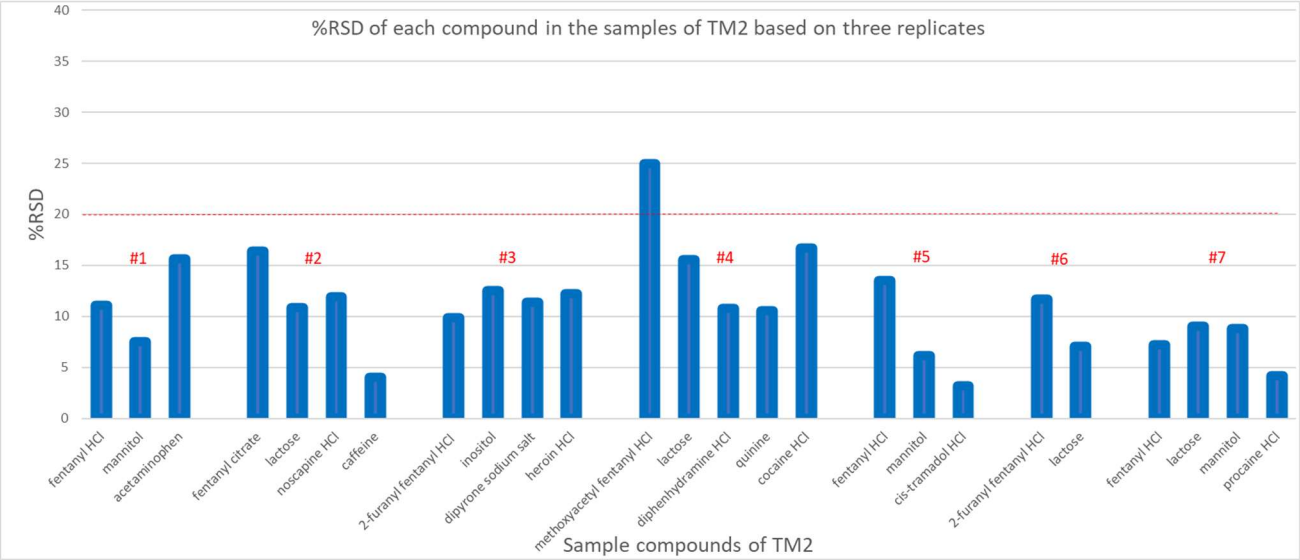
Sample number	Target compounds	Non-Target Compounds			
2.1	Fentanyl HCl (9.79% vs 9.82%)	Mannitol (40.11% vs 40.47%)	Acetaminophen (50.09% vs 49.88%)		
2.2	Fentanyl citrate (9.93% vs 10.84%)	Lactose (30.07% vs 25.96%)	Noscapine HCl (44.95% vs 49.94%)	Caffeine (15.05% vs 13.27%)	
2.3	2-furanyl fentanyl HCl (9.99% vs 10.26%)	Inositol (14.88% vs 12.15%)	Dipyrone sodium salt (15.02% vs 14.62%)	Heroin HCl (60.12% vs 62.97%)	
2.4	Methoxyacetyl fentanyl HCl (10.10% vs 12.25%)	Lactose (39.90% vs 36.73%)	Diphenhydramine HCl (25.09% vs 28.05%)	Quinine (19.92% vs 17.63%)	Cocaine HCl (4.99% vs 5.34%)
2.5	Fentanyl HCl (10.16% vs 9.00%)	Mannitol (30.01% vs 32.02%)	cis-Tramadol HCl (59.93% vs 58.97%)		
2.6	2-furanyl fentanyl HCl (10.15% vs 9.41%)	Lactose (89.85% vs 90.59%)			
2.7	Fentanyl HCl (10.02% vs 9.53%)	Lactose (19.89% vs 17.20%)	Mannitol (45.12% vs 51.30%)	Procaine HCl (24.96% vs 21.97%)	
2.8	Cyclopropyl fentanyl HCl (9.95% vs 8.28%)	Inositol (49.91% vs 51.00%)	Acetaminophen (40.14% vs 40.72%)		
2.9	Fentanyl HCl (10.04% vs 12.09%)	Lactose (84.96% vs 83.02%)	Cocaine HCl (5.01% vs 4.89%)		
2.10	Butyryl fentanyl HCl (9.98% vs 9.01%)	Mannitol (20.08% vs 19.76%)	Heroin HCl (60.00% vs 61.71%)	cis-Tramadol HCl (9.93% vs 9.53%)	
2.11	Fentanyl HCl (10.09% vs 9.80%)	Procaine HCl (15.01% vs 12.37%)	cis-Tramadol HCl (74.90% vs 77.83%)		
2.12	Acetyl fentanyl HCl (10.13% vs 9.14%)	Dipyrone sodium salt (24.79% vs 21.67%)	Heroin HCl (10.09%vs 8.13%)	cis-Tramadol HCl (54.99% vs 61.07%)	
2.13	Fentanyl HCl (9.92% vs 13.00%)	Lactose (15.01% vs 14.78%)	Mannitol (30.11% vs 29.22%)	Diphenhydramine HCl (15.10% vs 14.46%)	Acetaminophen (29.85% vs 28.54%)
2.14	Fentanyl citrate (10.07% vs 11.80%)	Lactose (39.98% vs 43.28%)	Mannitol (19.97% vs 20.48%)	Acetaminophen (29.98% vs 24.44%)	

Table D.2. TM3 Mixture Composition via. W/W% and LC/MS Analysis

Sample number	Target compounds	Non-Target Compounds			
3.1	Fentanyl HCl (1.12% vs 0.96%)	Lactose (14.98% vs 14.70%) Dipyrone sodium salt (15.02% vs 15.33%)	Microcrystalline cellulose (15%) Stearic acid (2.96%)	Acetaminophen (39.97% vs 39.96%) Mannitol (9.95% vs 10.26%)	FD&C Yellow #6 (1.00% vs 0.83%)
3.2	2-furanyl fentanyl HCl (1.01% vs 1.21%)	Inositol (20.00% vs 26.91%)	Polyethylene glycol (30.06%)	Heroin HCl (28.92% vs 17.47%)	Mannitol (20.02% vs 24.35%)
3.3	Methoxyacetyl fentanyl HCl (1.03% vs 0.60%)	Lactose (10.19% vs 9.30%) Noscapine HCl (30.02% vs 29.86%)	cis-Tramadol HCl (39.88% vs 43.81%)	Heroin HCl (9.81% vs 8.67%)	Procaine HCl (9.08% vs 7.76%)
3.4	Fentanyl citrate (1.04% vs 0.95%)	Inositol (50.02% vs 52.75%)	Dipyrone sodium salt (18.98% vs 16.76%)	Microcrystalline cellulose (25%) Stearic acid (3.99%)	FD&C Blue #2 (0.98% vs 0.54%)
3.5	Acetyl fentanyl HCl (1.05% vs 0.95%%)	Lactose (14.97% vs 11.61%) Mannitol (24.97% vs 23.70%)	Inositol (30.08% vs 38.33%)	Xylazine (19.97% vs 14.71%)	Noscapine HCl (8.96% vs 10.70%)
3.6	Fentanyl HCl (1.00% vs 1.11%)	Lactose (24.97% vs 22.06%) Mannitol (25.00% vs 27.39%)	Heroin HCl (29.92% vs 31.20%) Methamphetamine (5.10% vs 4.71%)	Caffeine (9.99% vs 10.93%)	Quinine (4.02% vs 2.61%)
3.7	2-furanyl fentanyl HCl (0.98% vs 0.95%)	Lactose (78.09% vs 78.74%%)	Microcrystalline cellulose (14.97%) Palmitic acid (2.98%)	FD&C Blue #2 (2.98% vs 2.35%)	
3.8	Fentanyl HCl (1.01% vs 0.93%)	Mannitol (31.95% vs 32.95%)	Hydroxypropyl cellulose (15.04%)	Ibuprofen (50.00% vs 49.07%)	Ferric oxide yellow (1.04%) FD&C Blue #2 (0.96% vs 0.97%)
3.9	Methoxyacetyl fentanyl HCl (1.11% vs 1.38%)	Lactose (9.93% vs 9.51%) Mannitol (9.94% vs 10.05%)	Inositol (19.98% vs 15.78%) Dipyrone sodium salt (9.02% vs 6.20%)	Dimethyl sulfone (29.78%)	Heroin HCl (20.23% vs 27.30%)
3.10	Fentanyl HCl (1.03% vs 0.72%)	Lactose (13.85% vs 14.89%) Mannitol (14.11% vs 16.24%)	Microcrystalline cellulose (20.06%)	Acetaminophen (49.97% vs 47.31%)	FD&C Blue #2 (0.98% vs 0.78%)
3.11	Cyclopropyl fentanyl HCl (1.03% vs 1.01%)	Lactose (50.01% vs 50.48%)	Mannitol (19.99% vs 14.66%)	Inositol (9.00% vs 9.20%) Quinine (4.98% vs 8.69%)	Heroin HCl (14.97% vs 15.96%)
3.12	Fentanyl HCl (1.04% vs 1.18%)	Lactose + Sucrose (84.05% vs 83.33%)	Diphenhydramine HCl (10.00% vs 11.72%)	Heroin HCl (4.91% vs 3.77%)	
3.13	Acetyl fentanyl HCl (1.04% vs 1.34%)	Lactose (73.77% vs 80.04%)	Heroin HCl (25.18% vs 18.62%)		
3.14	Fentanyl citrate (1.01% vs 1.21%)	Lactose (34.97% vs 32.83%) Mannitol (25.01% vs 24.04%)	Inositol (19.91% vs 23.97%)	Caffeine (14.11% vs 14.87%)	Nicotinamide (4.99% vs 3.09%)

Table D.3. TM4 Mixture Composition: W/W% vs. LC/MS Results

Sample number	Non-Target Compounds			
4.1	Heroin HCl (84.99% vs 87.34%)	Noscapine HCl (6.97% vs 6.85%)	Morphine (8.04% vs 5.82%)	
4.2	Methamphetamine HCl (89.96% vs 88.83%)	Dimethyl sulfone (10.04% vs 11.17%)		
4.3	Cocaine HCl (84.96% vs 84.28%)	Lidocaine (5.03% vs 6.30%)	Benzocaine (5.00% vs 6.37%)	Procaine HCl (5.02% vs 3.05%)
4.5	Heroin HCl (40.07% vs 42.93%)	Lactose (50.08% vs 38.99%)	Caffeine (9.85% vs 18.08%)	
4.6	Cocaine HCl (25.00% vs 19.81%)	Mannitol (75.00% vs 80.19%)		
4.7	Heroin HCl (19.99% vs 22.26%)	Mannitol (29.88% vs 21.11%)	Caffeine (30.17% vs 31.40%)	Quinine (19.95% vs 25.23%)



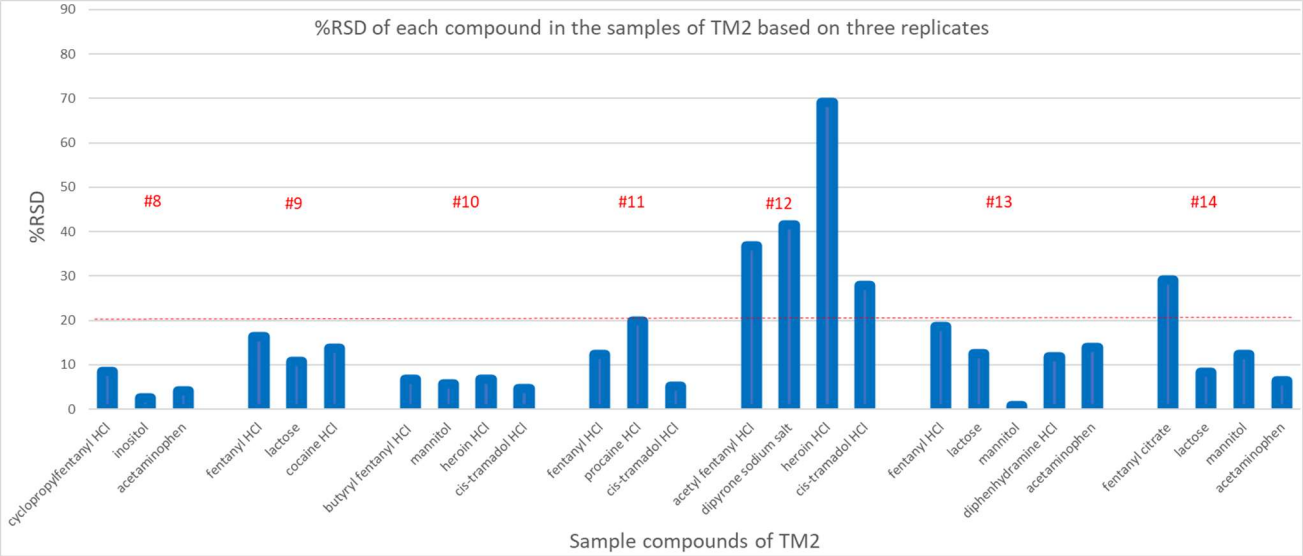
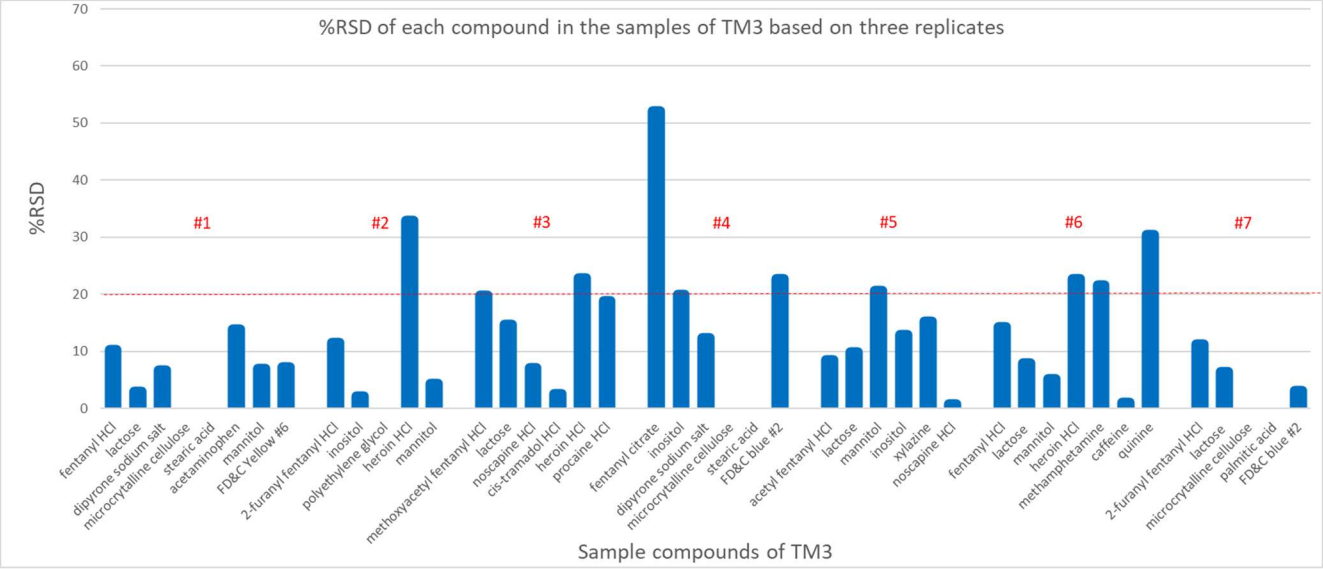


Figure D.1. %RSD of TM2 mixture samples.



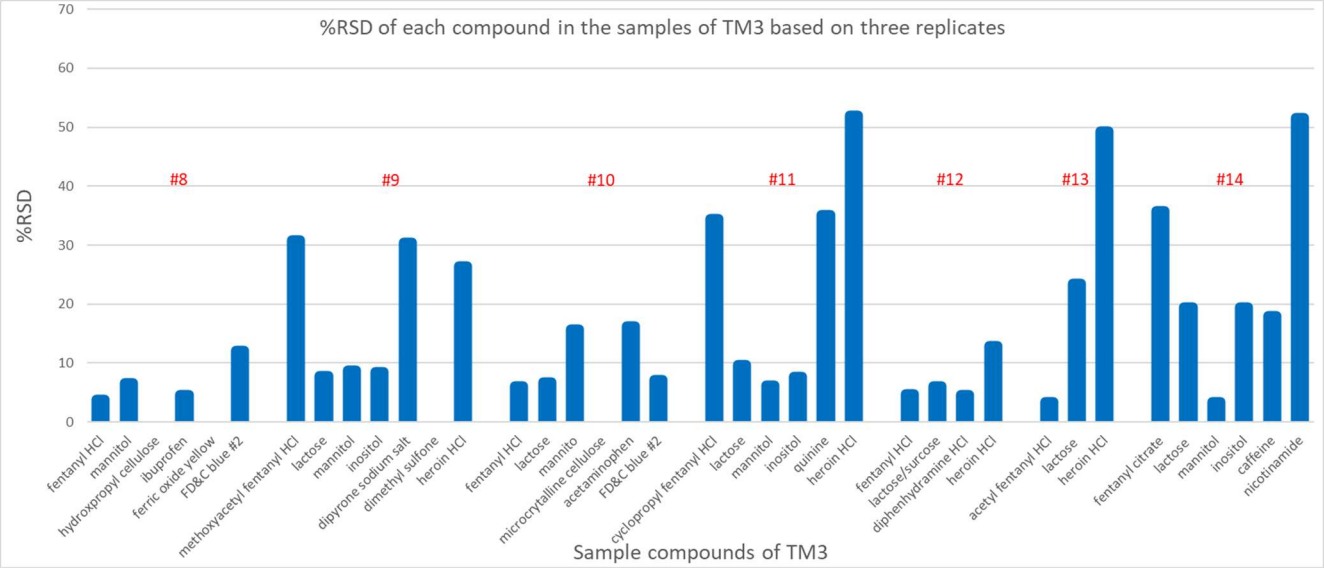


Figure D.2. %RSD of TM3 mixture samples.

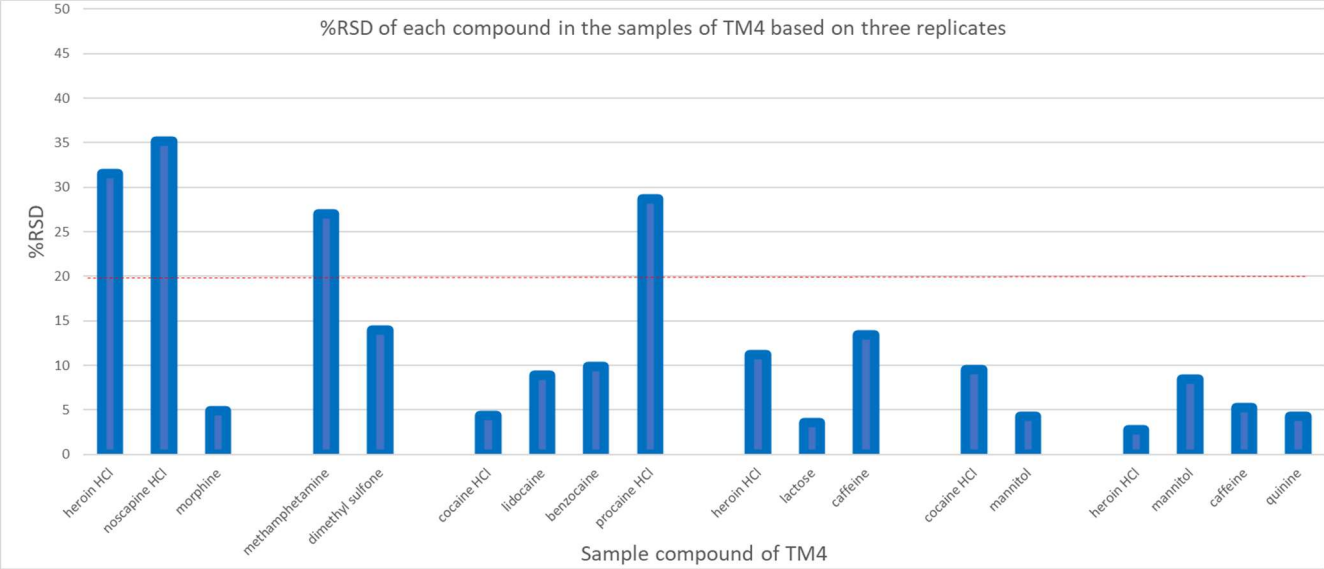


Figure D.3. %RSD of TM4 mixture samples.

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Appendix E - Direct Readout and Reachback Results

Direct Readout Results

A tabulation of direct readout results observed for applicable samples in each test module is given below. A “0” within the tables indicates that the instrument or assay did not pass that given sample. Entries of “N/A” are for instances where that specific test is not applicable (i.e.,: test was not performed due to instrument capability or previous result). Once four “0”s were reached, testing of that specific TM ceased. For TM4 there were instances where the Rigaku CQL 1064nm malfunctioned during testing; these cases are denoted with an asterisk and were not included in the statistical analysis. For mass-based technologies and assays where trace and bulk samples were tested in TMs 1-3, samples were first tested in trace amounts and then bulk amounts if the trace amounts did not produce a positive fentanyl compound detection. If a fentanyl compound was identified at trace amounts, that test sample was not measured at bulk amounts as it was assumed that bulk amounts of material would also cause a fentanyl detection when properly diluted.

Tables are separated by Test Module and by product category (mass-based, optical-based, and assays). For Test Module 4, the Griffin G510 was tested at trace concentrations with 14 test samples instead of 31. The other mass-based systems and assays were all tested at bulk concentrations with 31 test samples.

The Griffin G510 and optics-based instruments were tested in TM5 while the other mass-based systems and assays were not. However, the Rigaku CQL 1064nm was excluded from TM5 due to instrument malfunction.

Test Module 1: ≥ 95% Fentanyl and Fentanyl-Related Compounds: Direct Readout for Mass-Based Instruments

Instrument	GC/MS		HPMS		IMS					
	FLIR Detection Griffin G510		908 Devices MX908		Smiths Detection IONSCAN 600		Leidos H150E		Rapiscan Itemiser 4DN	
Sample #	Trace	Bulk	Trace	Bulk	Trace	Bulk	Trace	Bulk	Trace	Bulk
1.1	1	N/A	1	N/A	1	N/A	1	N/A	1	N/A
1.2	1	N/A	1	N/A	1	N/A	0	1	1	N/A
1.3	1	N/A	1	N/A	1	N/A	0	1	1	N/A
1.4	1	N/A	1	N/A	1	N/A	1	N/A	1	N/A
1.5	1	N/A	1	N/A	1	N/A	1	N/A	1	N/A
1.6	1	N/A	1	N/A	1	N/A	1	N/A	1	N/A
1.7	1	N/A	1	N/A	1	N/A	0	0	1	N/A
1.8	1	N/A	1	N/A	1	N/A	1	N/A	1	N/A
1.9	1	N/A	1	N/A	1	N/A	1	N/A	1	N/A
1.10	1	N/A	1	N/A	1	N/A	0	1	1	N/A
1.11	1	N/A	1	N/A	1	N/A	1	N/A	1	N/A
1.12	1	N/A	1	N/A	1	N/A	0	1	1	N/A
1.13	1	N/A	1	N/A	1	N/A	0	0	1	N/A
1.14	1	N/A	1	N/A	1	N/A	0	0	1	N/A
1.1 rep 2	1	N/A	1	N/A	1	N/A	0	0	1	N/A
1.2 rep 2	1	N/A	1	N/A	1	N/A			1	N/A
1.3 rep 2	1	N/A	1	N/A	1	N/A			1	N/A
1.4 rep 2	1	N/A	1	N/A	1	N/A			1	N/A
1.5 rep 2	1	N/A	1	N/A	1	N/A			1	N/A
1.6 rep 2	1	N/A	1	N/A	1	N/A			1	N/A
1.7 rep 2	1	N/A	1	N/A	1	N/A			1	N/A
1.8 rep 2	1	N/A	1	N/A	1	N/A			1	N/A
1.9 rep 2	1	N/A	1	N/A	1	N/A			1	N/A
1.10 rep 2	1	N/A	1	N/A	1	N/A			1	N/A
1.11 rep 2	1	N/A	1	N/A	1	N/A			1	N/A
1.12 rep 2	1	N/A	0	0	1	N/A			1	N/A
1.13 rep 2	1	N/A	1	N/A	1	N/A			1	N/A
1.14 rep 2	1	N/A	1	N/A	1	N/A			1	N/A
1.1 rep 3	1	N/A	1	N/A	1	N/A			1	N/A
1.2 rep 3	1	N/A	1	N/A	1	N/A			1	N/A
1.3 rep 3	1	N/A	0	0	1	N/A			1	N/A
Total Detections	31		29		31		7	4	31	

Test Module 1: ≥ 95% Fentanyl and Fentanyl-Related Compounds: Direct Readout for Optics-Based Instruments

Instrument	FTIR				Raman							
Sample #	Smiths Detection HazMatID Elite	Agilent Portable 4500	Redwave Technologies ThreatID	Thermo Scientific Gemini FTIR	Thermo Scientific Gemini Raman	Agilent Resolve	Chemring PGR-1064	Mira XTR DS	Metrohm TacticID-1064 ST	Rigaku CQL 1064nm	Thermo Scientific TruNarc	Thermo Scientific 1064Defender
1.1	1	1	1	1	1	1	1	1	1	1	1	1
1.2	1	1	1	1	1	1	1	1	1	1	1	1
1.3	1	1	1	1	1	1	0	1	1	1	1	1
1.4	1	1	1	1	1	1	1	1	1	1	1	1
1.5	1	1	1	1	1	1	1	1	1	1	1	1
1.6	1	1	1	1	1	1	1	0	1	1	1	1
1.7	1	1	1	1	1	1	1	1	1	1	1	1
1.8	1	1	1	1	1	1	0	1	1	1	1	1
1.9	1	1	1	1	1	1	1	1	1	1	1	1
1.10	1	1	1	1	1	1	1	1	1	1	1	1
1.11	1	1	1	1	1	1	1	1	1	1	1	1
1.12	1	1	1	1	1	1	1	1	1	1	1	1
1.13	1	1	1	1	1	1	1	1	1	1	1	1
1.14	1	1	1	1	1	1	1	1	1	1	0	1
1.1 rep 2	1	1	1	1	1	1	1	1	1	1	1	1
1.2 rep 2	1	1	1	1	1	1	1	1	1	1	1	1
1.3 rep 2	1	1	1	1	1	1	0	1	1	1	1	1
1.4 rep 2	1	1	1	1	1	1	1	1	1	1	1	1
1.5 rep 2	1	1	1	1	1	1	1	1	1	1	1	1
1.6 rep 2	1	1	1	1	1	1	1	0	1	1	1	1
1.7 rep 2	1	1	1	1	1	1	1	1	1	1	1	1
1.8 rep 2	1	1	1	1	1	1	0	1	1	1	1	1
1.9 rep 2	1	1	1	1	1	1		1	1	1	1	1
1.10 rep 2	1	1	1	1	1	1		1	1	1	1	1
1.11 rep 2	1	1	1	1	1	1		1	1	1	1	1
1.12 rep 2	1	1	1	1	1	1		1	1	1	1	1
1.13 rep 2	1	1	1	1	1	1		1	1	1	1	1
1.14 rep 2	1	1	1	1	1	1		1	1	1	1	1
1.1 rep 3	1	1	1	1	1	1		1	1	1	1	1
1.2 rep 3	1	1	1	1	1	1		1	1	1	1	1
1.3 rep 3	1	1	1	1	1	1		1	1	1	1	1
Total Detections	31	31	31	31	31	31	18	29	31	31	30	31

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Test Module 1: ≥ 95% Fentanyl and Fentanyl-Related Compounds: Direct Readout for Assays

Instrument	Assays															
Sample #	MobileDetect Multi-Drug Test		Field Forensics Fen-Her		Field Forensics DABIT 3x		Mistral Group PDT2 Fentanyl Reagent		Sirchie NARK II Fentanyl Reagent		SalivaConfirm Saliva Drug Test		Detectachem Fentanyl Test Strip		Rapid Response Fentanyl Test Strip	
	Trace	Bulk	Trace	Bulk	Trace	Bulk	Trace	Bulk	Trace	Bulk	Trace	Bulk	Trace	Bulk	Trace	Bulk
1.1	1	1	0	1	0	0	0	1	0	1	1	N/A	1	N/A	1	N/A
1.2	0	1	0	1	0	0	0	1	0	1	1	N/A	1	N/A	1	N/A
1.3	1	1	0	1	0	0	0	1	0	1	1	N/A	1	N/A	1	N/A
1.4	1	1	0	1	0	0	0	1	1	1	1	N/A	1	N/A	1	N/A
1.5	1	1		1				1	0	1	1	N/A	1	N/A	1	N/A
1.6	1	1		1				1		1	1	N/A	1	N/A	1	N/A
1.7	0	1		1				1		1	0	0	0	0	0	0
1.8	1	1		1				1		1	1	N/A	1	N/A	1	N/A
1.9	0	1		1				1		1	0	0	0	0	0	1
1.10	1	1		1				1		1	1	N/A	1	N/A	1	N/A
1.11	1	1		1				1		1	1	N/A	1	N/A	1	N/A
1.12	1	1		1				1		1	0	1	1	N/A	1	N/A
1.13	0	0		0				1		0	1	N/A	0	0	1	N/A
1.14		0		0				0		0	1	N/A	0	0	1	N/A
1.1 rep 2		1		1				1		1	1	N/A			1	N/A
1.2 rep 2		1		1				1		1	1	N/A			1	N/A
1.3 rep 2		1		1				1		1	1	N/A			1	N/A
1.4 rep 2		1		1				1		1	1	N/A			1	N/A
1.5 rep 2		1		1				1		1	1	N/A			1	N/A
1.6 rep 2		1		1				1		1	1	N/A			1	N/A
1.7 rep 2		1		1				1		1	0	0			0	0
1.8 rep 2		1		1				1		1	1	N/A			1	N/A
1.9 rep 2		1		1				1		1	0	0				1
1.10 rep 2		1		1				1		1					1	N/A
1.11 rep 2		1		1				1		1					1	N/A
1.12 rep 2		1		1				1		1					1	N/A
1.13 rep 2		0		0				1		0					1	N/A
1.14 rep 2		0		0				0		0					1	N/A
1.1 rep 3								1							1	N/A
1.2 rep 3								1							1	N/A
1.3 rep 3								1							1	N/A
Total Detections	9	24	0	24	0	0	0	29	1	24	20	1	10	0	27	2

Test Module 2: 10% Fentanyl and Fentanyl-Related Compounds: Direct Readout for Mass-Based Instruments

Instrument	GC/MS		HPMS		IMS					
	FLIR Detection Griffin G510		908 Devices MX908		Smiths Detection IONSCAN 600		Leidos H150E		Rapiscan Itemiser 4DN	
Sample #	Trace	Bulk	Trace	Bulk	Trace	Bulk	Trace	Bulk	Trace	Bulk
2.1	1	N/A	1	N/A	1	N/A	1	N/A	1	N/A
2.2	1	N/A	1	N/A	1	N/A	0	1	1	N/A
2.3	0	1	1	N/A	1	N/A	1	N/A	1	N/A
2.4	1	N/A	0	1	1	N/A	1	N/A	0	1
2.5	1	N/A	0	1	1	N/A	0	0	0	1
2.6	0	1	1	N/A	1	N/A	1	N/A	1	N/A
2.7	1	N/A	1	N/A	1	N/A	1	N/A	0	1
2.8	1	N/A	0	1	1	N/A	1	N/A	0	1
2.9	1	N/A	1	N/A	1	N/A	1	N/A	1	N/A
2.10	1	N/A	1	N/A	1	N/A	1	N/A	0	1
2.11	1	N/A	0	0	1	N/A	0	0	0	0
2.12	1	N/A	1	N/A	1	N/A	0	0	0	1
2.13	1	N/A	1	N/A	1	N/A	1	N/A	0	1
2.14	1	N/A	1	N/A	1	N/A	0	0	1	N/A
2.1 rep2	1	N/A	1	N/A	1	N/A			1	N/A
2.2 rep2	1	N/A	1	N/A	1	N/A			1	N/A
2.3 rep2	1	N/A	1	N/A	1	N/A			1	N/A
2.4 rep2	1	N/A	0	1	1	N/A			0	1
2.5 rep2	1	N/A	0	1	1	N/A			0	0
2.6 rep2	1	N/A	0	1	1	N/A			1	0
2.7 rep2	1	N/A	1	N/A	1	N/A			0	1
2.8 rep2	1	N/A	0	1	1	N/A			1	N/A
2.9 rep2	1	N/A	1	N/A	1	N/A			1	N/A
2.10 rep2	1	N/A	1	N/A	1	N/A			0	1
2.11 rep2	0	1	1	N/A	1	N/A			0	0
2.12 rep2	1	N/A	1	N/A	1	N/A			0	1
2.13 rep2	0	1	0	1	1	N/A			0	1
2.14 rep2	1	N/A	1	N/A	1	N/A			1	N/A
2.1 rep3	0	1	1	N/A	1	N/A			1	N/A
2.2 rep3	1	N/A	1	N/A	1	N/A			1	N/A
2.3 rep3	1	N/A	1	N/A	1	N/A			1	N/A
Total Detections	26	5	22	8	31	0	9	1	16	12

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Test Module 2: 10% Fentanyl and Fentanyl-Related Compounds: Direct Readout for Optics-Based Instruments

Instrument	FTIR				Raman							
Sample #	Smiths Detection HazMatID Elite	Agilent Portable 4500	Redwave Technologies ThreatID	Thermo Scientific Gemini FTIR	Thermo Scientific Gemini Raman	Agilent Resolve	Chemring PGR-1064	Mira XTR DS	Metrohm TacticID-1064 ST	Rigaku CQL 1064nm	Thermo Scientific TruNarc	Thermo Scientific 1064Defender
2.1	0	1	1	0	0	0	0	0	0	0	0	0
2.2	0	0	0	0	0	0	0	0	0	1	1	0
2.3	0	0	0	0	1	1	0	1	1	1	1	1
2.4	0	1	0	0	0	1	1	1	1	0	1	0
2.5		1	1		0	0	0	1	0	0	0	0
2.6		0	0			1		1	1	1	1	
2.7		1				0		0	0	0	0	
2.8		1						0			0	
2.9		1										
2.10		1										
2.11		1										
2.12		1										
2.13		1										
2.14		0										
2.1 rep2												
2.2 rep2												
2.3 rep2												
2.4 rep2												
2.5 rep2												
2.6 rep2												
2.7 rep2												
2.8 rep2												
2.9 rep2												
2.10 rep2												
2.11 rep2												
2.12 rep2												
2.13 rep2												
2.14 rep2												
2.1 rep3												
2.2 rep3												
2.3 rep3												
Total Detections	0	10	2	0	1	3	1	4	3	3	4	1

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Test Module 2: 10% Fentanyl and Fentanyl-Related Compounds: Direct Readout for Assays

Instrument	Assay															
Sample #	MobileDetect Multi-Drug Test		Field Forensics Fen-Her		Field Forensics DABIT 3x		Mistral Group PDT2 Fentanyl Reagent		Sirchie NARK II Fentanyl Reagent		SalivaConfirm Saliva Drug Test		Detectachem Fentanyl Test Strip		Rapid Response Fentanyl Test Strip	
	Trace	Bulk	Trace	Bulk	Trace	Bulk	Trace	Bulk	Trace	Bulk	Trace	Bulk	Trace	Bulk	Trace	Bulk
2.1	0	0	0	1	0	0	0	0	1	1	1	N/A	1	N/A	1	N/A
2.2	0	1	0	1	0	0	0	1	0	1	1	N/A	1	N/A	1	N/A
2.3	0	0	0	1	0	0	1	1	0	1	0	1	1	N/A	1	N/A
2.4	0	1	0	1	0	0	0	1	0	1	1	N/A	1	N/A	1	N/A
2.5		1		1			0	1	0	1	1	N/A	1	N/A	1	N/A
2.6		1		1				0		0	0	1	1	N/A	0	1
2.7		1		1				0		1	1	N/A	1	N/A	1	N/A
2.8		0		1				0		0	0	1	1	N/A	0	1
2.9		1		1						0	1	N/A	1	N/A	1	N/A
2.10		0		1						1	0	1	1	N/A	0	1
2.11				1						1	1	N/A	1	N/A	0	1
2.12				1						1	1	N/A	1	N/A	1	N/A
2.13				1						1	1	N/A	1	N/A	1	N/A
2.14				1						1	1	N/A	1	N/A	1	N/A
2.1 rep2				1						1	1	N/A	1	N/A	1	N/A
2.2 rep2				1						1	1	N/A	1	N/A	1	N/A
2.3 rep2				1						1		1	1	N/A	1	N/A
2.4 rep2				1						1	1	N/A	1	N/A	1	N/A
2.5 rep2				1						1	1	N/A	1	N/A	1	N/A
2.6 rep2				1						0		1	1	N/A		1
2.7 rep2				1							1	N/A	1	N/A	1	N/A
2.8 rep2				1								1	1	N/A		1
2.9 rep2				1							1	N/A	1	N/A	1	N/A
2.10 rep2				1								1	1	N/A		1
2.11 rep2				0							1	N/A	1	N/A		1
2.12 rep2				1							1	N/A	1	N/A	1	N/A
2.13 rep2				1							1	N/A	1	N/A	1	N/A
2.14 rep2				1							1	N/A	1	N/A	1	N/A
2.1 rep3				1							1	N/A	1	N/A	1	N/A
2.2 rep3				1							1	N/A	1	N/A	1	N/A
2.3 rep3				1								1	1	N/A	1	N/A
Total Detections	0	6	0	30	0	0	1	4	1	16	22	9	31		23	8

Test Module 3: 1% Fentanyl and Fentanyl-Related Compounds: Direct Readout for Mass-Based Instruments

Instrument	GC/MS		HPMS		IMS					
	FLIR Detection Griffin G510		908 Devices MX908		Smiths Detection IONSCAN 600		Leidos H150E		Rapiscan Itemiser 4DN	
Sample #	Trace	Bulk	Trace	Bulk	Trace	Bulk	Trace	Bulk	Trace	Bulk
3.1	0	1	0	0	0	1	1	N/A	1	N/A
3.2	0	0	0	0	1	N/A	1	N/A	1	N/A
3.3	0	0	0	1	0	1	0	0	0	0
3.4	0	1	1	N/A	1	N/A	0	1	0	1
3.5	0	1	0	1	1	N/A	1	N/A	0	0
3.6	0	1	0	1	1	N/A	1	N/A	1	N/A
3.7	0	0	0	1	0	1	1	N/A	1	N/A
3.8	0	1	0	1	1	N/A	1	N/A	1	N/A
3.9	0	1	0	1	1	N/A	1	N/A	1	N/A
3.10	0	1	0	0	0	1	0	1	1	N/A
3.11	0	1	0	1	1	N/A	1	N/A	0	1
3.12	0	1	0	0	1	N/A	0	1	0	0
3.13	0	0			1	N/A	1	N/A	1	N/A
3.14					0	1	1	N/A	0	1
3.1 rep2					1	N/A	1	N/A	1	N/A
3.2 rep2					0	1	1	N/A	1	N/A
3.3 rep2					0	0	0	0	0	0
3.4 rep2					0	1	0	1		
3.5 rep2					1	N/A	1	N/A		
3.6 rep2					1	N/A	1	N/A		
3.7 rep2					1	N/A	1	N/A		
3.8 rep2					1	N/A	0	1		
3.9 rep2					1	N/A	1	N/A		
3.10 rep2					1	N/A	0	0		
3.11 rep2					1	N/A	1	N/A		
3.12 rep2					1	N/A	0	1		
3.13 rep2					1	N/A	1	N/A		
3.14 rep2					1	N/A	0	1		
3.1 rep3					1	N/A	1	N/A		
3.2 rep3					1	N/A	1	N/A		
3.3 rep3					1	N/A	0	0		
Total Detections	0	9	1	7	23	7	20	7	10	3

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Test Module 3: 1% Fentanyl and Fentanyl-Related Compounds: Direct Readout for Optics-Based Instruments

Instrument	FTIR				Raman							
Sample #	Smiths Detection HazMatID Elite	Agilent Portable 4500	Redwave Technologies ThreatID	Thermo Scientific Gemini FTIR	Thermo Scientific Gemini Raman	Agilent Resolve	Chemring PGR-1064	Mira XTR DS	Metrohm TacticID-1064 ST	Rigaku CQL 1064nm	Thermo Scientific TruNarc	Thermo Scientific 1064Defender
3.1	0	0	0	0	0	0	0	0	0	0	0	0
3.2	0	0	0	0	0	0	0	1	0	0	0	0
3.3	0	0	0	0	0	0	0	0	0	0	0	0
3.4	0	0	0	0	0	0	0	0	0	0	0	0
3.5								0				
3.6												
3.7												
3.8												
3.9												
3.10												
3.11												
3.12												
3.13												
3.14												
3.1 rep2												
3.2 rep2												
3.3 rep2												
3.4 rep2												
3.5 rep2												
3.6 rep2												
3.7 rep2												
3.8 rep2												
3.9 rep2												
3.10 rep2												
3.11 rep2												
3.12 rep2												
3.13 rep2												
3.14 rep2												
3.1 rep3												
3.2 rep3												
3.3 rep3												
Total Detections	0	0	0	0	0	0	0	1	0	0	0	0

Test Module 3: 1% Fentanyl and Fentanyl-Related Compounds: Direct Readout for Assays

Instrument	Assays															
Sample #	MobileDetect Multi-Drug Test		Field Forensics Fen-Her		Field Forensics DABIT 3x		Mistral Group PDT2 Fentanyl Reagent		Sirchie NARK II Fentanyl Reagent		SalivaConfirm Saliva Drug Test		Detectachem Fentanyl Test Strip		Rapid Response Fentanyl Test Strip	
	Trace	Bulk	Trace	Bulk	Trace	Bulk	Trace	Bulk	Trace	Bulk	Trace	Bulk	Trace	Bulk	Trace	Bulk
3.1	0	0	0	0	0	0	0	0	0	1	0	1	0	1	0	1
3.2	0	0	0	0	0	0	0	0	0	1	0	1	0	1	0	1
3.3	0	1	0	1	0	0	0	1	0	1	0	1	1	N/A	1	N/A
3.4	0	0	0	0	0	0	0	0	0	0	0	1	0	1	0	1
3.5		0		0				1		0	0	1	0	1	1	N/A
3.6								1		1	0	1	0	1	0	1
3.7								0		0	0	0	0	1	0	1
3.8										0	0	1	0	1	0	1
3.9											0	1	1	N/A	0	1
3.10											0	1	0	1	0	1
3.11											0	1	0	1	0	1
3.12											0	1	0	1	1	N/A
3.13											0	1	0	1	0	1
3.14											0	1	0	1	0	1
3.1 rep2												1		1		1
3.2 rep2												1		1		1
3.3 rep2												1	1	N/A	1	N/A
3.4 rep2												1		1		1
3.5 rep2												1		1	1	N/A
3.6 rep2												1		1		1
3.7 rep2												1		1		1
3.8 rep2												1		1		1
3.9 rep2												1	1	N/A		1
3.10 rep2												1		1		1
3.11 rep2												1		1		1
3.12 rep2												1		1	1	N/A
3.13 rep2												1		1		1
3.14 rep2												1		1		1
3.1 rep3												1		1		1
3.2 rep3												1		1	1	N/A
3.3 rep3												1	1	N/A		1
Total Detections	0	1	0	1	0	0	0	3	0	4	0	30	5	26	7	24

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Test Module 4: False-Positive Results: Direct Readout for Mass-Based and FTIR Instruments

Instrument	GC/MS	HPMS	IMS			FTIR			
Sample #	FLIR Detection Griffin G510	908 Devices MX908	Smiths Detection IONSCAN 600	Leidos H150E	Rapiscan Itemiser 4DN	Smiths Detection HazMatID Elite	Agilent Portable 4500	Redwave Technologies ThreatID	Thermo Scientific Gemini FTIR
4.1	1	1	1	0	0	1	1	1	1
4.2	1	1	0	0	1	1	1	1	1
4.3	1	0	1	1	1	1	1	1	1
4.4	1	1	1	0	1	1	1	1	1
4.5	1	1	1	0	0	1	1	1	1
4.6	1	1	1		1	1	1	1	1
4.7	1	1	0		0	1	1	1	1
4.8	1	1	1		0	1	1	1	1
4.9	1	1	1			1	1	1	1
4.10	1	1	1			1	1	1	1
4.11	1	0	0			1	1	1	1
4.12	1	1	1			1	1	1	1
4.13	1	1	1			1	1	1	1
4.14	1	1	1			1	1	1	1
4.1 rep2	N/A	1	1			1	1	1	1
4.2 rep2	N/A	1	0			1	1	1	1
4.3 rep2	N/A	0				1	1	0	1
4.4 rep2	N/A	0				1	1	1	1
4.5 rep2	N/A					1	1	1	1
4.6 rep2	N/A					1	1	1	1
4.7 rep2	N/A					1	1	1	1
4.8 rep2	N/A					1	1	1	1
4.9 rep2	N/A					1	1	1	1
4.10 rep2	N/A					1	1	1	1
4.11 rep2	N/A					1	1	1	1
4.12 rep2	N/A					1	1	1	1
4.13 rep2	N/A					1	1	1	1
4.14 rep2	N/A					1	1	1	1
4.1 rep3	N/A					1	1	1	1
4.2 rep3	N/A					1	1	1	1
4.3 rep3	N/A					1	1	1	1
Total Detections	14	14	12	1	4	31	31	30	31

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Test Module 4: False-Positive Results: Direct Readout for Raman Instruments

Instrument	Raman							
Sample #	Thermo Scientific Gemini Raman	Agilent Resolve	Chemring PGR-1064	Mira XTR DS	Metrohm TacticID-1064 ST	Rigaku CQL 1064nm	Thermo Scientific TruNarc	Thermo Scientific 1064Defender
4.1	1	1	1	1	1	1	1	1
4.2	1	1	1	1	1	1	1	1
4.3	1	1	1	1	0	1	1	1
4.4	1	1	1	1	1	1	1	1
4.5	1	1	1	1	1	1	1	1
4.6	1	1	1	0	1	*	1	1
4.7	1	1	1	1	1	*	1	1
4.8	1	1	1	1	1	1	1	1
4.9	1	1	1	1	1	1	1	1
4.10	1	1	1	1	1	1	1	1
4.11	1	1	1	0	1	1	1	1
4.12	1	1	1	0	1	1	1	1
4.13	1	1	1	1	1	1	1	1
4.14	1	1	1	0	1	1	1	1
4.1 rep2	1	1	1		1	1	1	1
4.2 rep2	1	1	1		0	1	1	1
4.3 rep2	1	1	1		1	1	1	1
4.4 rep2	1	1	1		1	1	1	1
4.5 rep2	1	1	1		1	1	1	1
4.6 rep2	1	1	1		1	*	1	1
4.7 rep2	1	1	1		1	*	1	1
4.8 rep2	1	1	1		1	1	1	1
4.9 rep2	1	1	1		1	1	1	1
4.10 rep2	1	1	1		1	1	1	1
4.11 rep2	1	1	1		1	1	1	1
4.12 rep2	1	1	1		1	1	1	1
4.13 rep2	1	1	1		1	1	1	1
4.14 rep2	1	1	1		1	1	1	1
4.1 rep3	1	1	1		1	1	1	1
4.2 rep3	1	1	1		1	1	1	1
4.3 rep3	1	1	1		1	1	1	1
Total Detections	31	31	31	10	29	27	31	31

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Test Module 4: False-Positive Results: Direct Readout for Assays at Bulk Concentrations

Instrument	Assays							
Sample #	MobileDetect Multi-Drug Test	Field Forensics Fen-Her	Field Forensics DABIT 3x	Mistral Group PDT2 Fentanyl Reagent	Sirchie NARK II Fentanyl Reagent	SalivaConfirm Saliva Drug Test	Detectachem Fentanyl Test Strip	Rapid Response Fentanyl Test Strip
4.1	1	1	1	1	1	1	1	1
4.2	1	1	1	1	0	1	0	1
4.3	1	0	1	1	1	1	1	1
4.4	1	1	1	1	0	1	0	1
4.5	1	1	1	1	1	1	1	1
4.6	1	1	1	0	1	1	1	1
4.7	1	1	1	0	1	1	1	1
4.8	1	1	1	1	1	1	1	1
4.9	1	1	1	1	1	1	1	1
4.10	0	1	1	0	1	1	1	1
4.11	0	0	1	0	1	1	1	1
4.12	1	1	1		0	0	0	0
4.13	1	1	1		1	1	1	1
4.14	1	1	1		1	1	1	1
4.1 rep2	1	1	1		1	1	1	1
4.2 rep2	1	1	1		0	1	0	1
4.3 rep2	1	1	1			1		1
4.4 rep2	1	1	1			1		1
4.5 rep2	1	1	1			1		1
4.6 rep2	1	1	1			1		1
4.7 rep2	1	1	1			1		1
4.8 rep2	1	1	1			1		1
4.9 rep2	1	1	1			1		1
4.10 rep2	0	1	1			1		1
4.11 rep2	0	0	1			1		1
4.12 rep2		1	1			0		0
4.13 rep2		1	1			1		1
4.14 rep2		1	1			1		1
4.1 rep3		0	1			1		1
4.2 rep3			1			1		1
4.3 rep3			1			1		1
Total Detections	21	25	31	7	12	29	12	29

Test Module 5: Precursor Compounds and Synthesis Related: Direct Readout Results

Instrument	GC/MS		FTIR				Raman						
Sample #	**FLIR Detection Griffin G510**		**Smiths Detection HazMatID Elite**	**Agilent Portable 4500**	**Redwave Technologies ThreatID**	**Thermo Scientific Gemini FTIR**	**Thermo Scientific Gemini Raman**	**Agilent Resolve**	**Chemring PGR-1064**	**Mira XTR DS**	**Metrohm TacticID-1064 ST**	**Thermo Scientific TruNarc**	**Thermo Scientific 1064Defender**
	Trace	**Bulk**											
5.1	1	N/A	1	1	1	1	1	1	1	1	1	1	1
5.2	0	0	0	1	1	1	0	1	1	1	1	1	1
5.3	1	N/A	1	1	1	1	1	1	0	1	1	1	1
5.4	1	N/A	1	1	1	1	0	1	1	1	1	0	1
5.5	1	N/A	1	1	1	1	1	1	0	1	1	1	1
5.6	0	0	1	0	1	1	1	0	0	1	1	0	1
5.7	0	N/A	0	1	1	1	0	1	1	1	0	0	1
5.8	0	0	1	0	1	1	1	0	1	1	1	0	1
5.9		N/A	0	1	1	1	0	0	0	1	1		0
5.10		N/A	1	1	1	1		1		1	1		1
5.11		0	0	0	1	0		0		0	0		1
5.12				1	1	1				0	1		1
5.13				1	1	1				1	1		1
5.14				0	1	1				1	1		1
5.1 rep2					1	1				1	1		1
5.2 rep2					1	1				1	1		1
5.3 rep2					1	1				1	1		1
5.4 rep2					1	1				0	1		1
5.5 rep2					1	1				0	1		1
5.6 rep2					1	1					1		1
5.7 rep2					1	1					0		1
5.8 rep2					1	1					1		1
5.9 rep2					1	1					1		0
5.10 rep2					1	1					1		1
5.11 rep2					1	0					0		1
5.12 rep2					1	1							1
5.13 rep2					1	1							1
5.14 rep2					1	1							1
5.1 rep3					1	1							1
5.2 rep3					1	1							1
5.3 rep3					1	1							1
Total Detections	4	0	7	10	31	29	5	7	5	15	21	4	29

Vendor Reachback Results

A tabulation of vendor reachback results obtained for applicable test samples within each test module is provided. Some vendors do not offer 24/7/365 reachback services, but still participated in the reachback assessment. In the tables below a “1” indicates a passing result while a “0” indicates that reachback did not provide a passing result. If vendors did not return a response within one hour of receiving data, it was recorded as a failure, even if the analysis was correct. However, in cases where responses were delayed due to email issues or file corruption, vendors were not penalized for slightly delayed results (no more than 10 minutes).

Sample results were sent to vendors for reachback analysis until four failures were reached. After four failures were reached, achieving an LCB/CL of 0.85/80% is not possible, even if all remaining samples successfully passed. In the case of mass-based instruments where bulk amounts were necessary for identification, this meant that four samples failed for bulk.

In certain cases, samples were sent in a randomized format and testing ceased once four failures were reached for the entire test module. This meant that data may not be presented in a consecutive order, causing gaps in the tables shown below. In some cases, an entire TM was tested at once and data analysis and reachback performed later.

The Rigaku CQL was excluded from reachback analysis due to time constraints.

Test Module 1: ≥ 95% Fentanyl and Fentanyl-Related Compounds: Reachback for Mass-Based Instruments

Instrument	GC/MS		HPMS		IMS					
	FLIR Detection Griffin G510		908 Devices MX908		Smiths Detection IONSCAN 600		Leidos H150E		Rapiscan Itemiser 4DN	
Sample #	Trace	Bulk	Trace	Bulk	Trace	Bulk	Trace	Bulk	Trace	Bulk
1.1	1	N/A	1	N/A	1	N/A	1	N/A	1	N/A
1.2	1	N/A	1	N/A	1	N/A	0	1	1	N/A
1.3	1	N/A	1	N/A	1	N/A	1	1	1	N/A
1.4	1	N/A	1	N/A	1	N/A	1	N/A	1	N/A
1.5	1	N/A	1	N/A	1	N/A	1	N/A	1	N/A
1.6	1	N/A	1	N/A	1	N/A	1	N/A	1	N/A
1.7	1	N/A	1	N/A	1	N/A	0	1	1	N/A
1.8	1	N/A	1	N/A	1	N/A	1	N/A	1	N/A
1.9	1	N/A	1	N/A	1	N/A	1	N/A	1	N/A
1.10	1	N/A	1	N/A	1	N/A	1	1	1	N/A
1.11	1	N/A	1	N/A	1	N/A	1	N/A	1	N/A
1.12	1	N/A	1	N/A	1	N/A	1	1	1	N/A
1.13	1	N/A	1	N/A	1	N/A	1	1	1	N/A
1.14	1	N/A	1	N/A	1	N/A	0	1	1	N/A
1.1 rep 2	1	N/A	1	N/A	1	N/A	0	1	1	N/A
1.2 rep 2	1	N/A	1	N/A	1	N/A	1	1	1	N/A
1.3 rep 2	1	N/A	1	N/A	1	N/A	1	1	1	N/A
1.4 rep 2	1	N/A	1	N/A	1	N/A	1	1	1	N/A
1.5 rep 2	1	N/A	1	N/A	1	N/A	1	1	1	N/A
1.6 rep 2	1	N/A	1	N/A	1	N/A	1	1	1	N/A
1.7 rep 2	1	N/A	1	N/A	1	N/A	0	1	1	N/A
1.8 rep 2	1	N/A	1	N/A	1	N/A	1	1	1	N/A
1.9 rep 2	1	N/A	1	N/A	1	N/A	1	1	1	N/A
1.10 rep 2	1	N/A	1	N/A	1	N/A	1	1	1	N/A
1.11 rep 2	1	N/A	1	N/A	1	N/A	1	1	1	N/A
1.12 rep 2	1	N/A	1	1	1	N/A	1	1	1	N/A
1.13 rep 2	1	N/A	1	N/A	1	N/A	1	1	1	N/A
1.14 rep 2	1	N/A	1	N/A	1	N/A	1	1	1	N/A
1.1 rep 3	1	N/A	1	N/A	1	N/A	1	N/A	1	N/A
1.2 rep 3	1	N/A	1	N/A	1	N/A	1	N/A	1	N/A
1.3 rep 3	1	N/A	1	1	1	N/A	1	N/A	1	N/A
Total Detections	31	0	31	2	31	0	26	21	31	0

Test Module 1: ≥ 95% Fentanyl and Fentanyl-Related Compounds: Reachback for Optics-Based Instruments

Instrument	FTIR				Raman						
Sample #	Smiths Detection HazMatID Elite	Agilent Portable 4500	Redwave Technologies ThreatID	Thermo Scientific Gemini FTIR	Thermo Scientific Gemini Raman	Agilent Resolve	Chemring PGR-1064	Mira XTR DS	Metrohm TacticID-1064 ST	Thermo Scientific TruNarc	Thermo Scientific 1064Defender
1.1	1	1	1	1	1	1	1	1	1	1	1
1.2	1	1	1	1	1	1	1	1	1	1	1
1.3	1	1	1	1	1	1	0	1	1	1	1
1.4	1	1	1	1	1	1	1	1	1	1	1
1.5	1	1	1	1	1	0	1	1	1	1	1
1.6	1	1	1	1	1	1	1	1	1	1	1
1.7	1	1	1	1	1	1	1	1	1	1	1
1.8	1	1	1	1	1	1	0	1	1	1	1
1.9	1	1	1	1	1	1	1	0	1	1	1
1.10	1	1	1	1	1	1	1	1	1	1	1
1.11	1	1	1	1	1	1	1	1	1	1	1
1.12	1	1	1	1	1	1	1	1	1	1	1
1.13	1	1	1	1	1	1	1	1	1	1	1
1.14	1	1	1	1	1	0	1	1	1	0	1
1.1 rep 2	1	1	1	1	1	1	1	1	1	1	1
1.2 rep 2	1	1	1	1	1	1	1	1	1	1	1
1.3 rep 2	1	1	1	1	1	1	0	1	1	1	1
1.4 rep 2	1	1	1	1	1	1	1	1	1	1	1
1.5 rep 2	1	1	1	1	1	1	1	1	1	1	1
1.6 rep 2	1	1	1	1	1	1	1	1	1	1	1
1.7 rep 2	1	1	1	1	1	1	1	1	1	1	1
1.8 rep 2	1	1	1	1	1	1	0	1	1	1	1
1.9 rep 2	1	1	1	1	1	1		1	1	1	1
1.10 rep 2	1	1	1	1	1	1		1	1	1	1
1.11 rep 2	1	1	1	1	1	1		1	1	1	1
1.12 rep 2	1	1	1	1	1	1		1	1	1	1
1.13 rep 2	1	1	1	1	1	1		1	1	1	1
1.14 rep 2	1	1	1	1	1	1		1	1	1	1
1.1 rep 3	1	1	1	1	1	1		1	1	1	1
1.2 rep 3	1	1	1	1	1	1		1	1	1	1
1.3 rep 3	1	1	1	1	1	1		1	1	1	1
Total Detections	31	31	31	31	31	29	18	30	31	30	31

Test Module 2: 10% Fentanyl and Fentanyl-Related Compounds: Reachback for Mass-Based Instruments

Instrument	GC/MS		HPMS		IMS					
	FLIR Detection Griffin G510		908 Devices MX908		Smiths Detection IONSCAN 600		Leidos H150E		Rapiscan Itemiser 4DN	
Sample #	Trace	Bulk	Trace	Bulk	Trace	Bulk	Trace	Bulk	Trace	Bulk
2.1	1	N/A	1	N/A	1	N/A	1	N/A		N/A
2.2	1	N/A	1	N/A	1	N/A	1	1	0	N/A
2.3	1	1	1	N/A	1	N/A	1	N/A	0	N/A
2.4	1	N/A	N/A	1	1	N/A	1	N/A	0	0
2.5	1	N/A	N/A	1	1	N/A	0	0	0	0
2.6	1	1	1	N/A	1	N/A	1	N/A		
2.7	1	N/A	1	N/A	1	N/A		N/A		
2.8	1	N/A	N/A	1	1	N/A	1	N/A		
2.9	1	N/A	1	N/A	1	N/A	1	N/A		
2.10	1	N/A	1	N/A	1	N/A	1	N/A		
2.11	1	N/A	N/A	1	1	N/A	1	0		
2.12	1	N/A	1	N/A	1	N/A	0	1		
2.13	1	N/A	1	N/A	1	N/A	1	N/A		
2.14	1	N/A	1	N/A	1	N/A	1	1		
2.1 rep2	1	N/A	1	N/A	1	N/A	1	N/A		
2.2 rep2	1	N/A	1	N/A	1	N/A	1	0		
2.3 rep2	1	N/A	1	N/A	1	N/A	1	N/A		
2.4 rep2	1	N/A	N/A	1	1	N/A	1	N/A		
2.5 rep2	1	N/A	1	1	1	N/A	0	0		
2.6 rep2	1	N/A	1	1	1	N/A	1	N/A		
2.7 rep2	1	N/A	1	N/A	1	N/A	1			
2.8 rep2	1	N/A	1	1	1	N/A	1	N/A		
2.9 rep2	1	N/A	1	N/A	1	N/A	1	N/A		
2.10 rep2	1	N/A	1	N/A	1	N/A	1	N/A		
2.11 rep2	1	1	1	N/A	1	N/A		1		
2.12 rep2	1	N/A	1	N/A	1	N/A	1			
2.13 rep2	1	1	1	1	1	N/A	1	N/A		
2.14 rep2	1	N/A	1	N/A	1	N/A	1	1		
2.1 rep3	0	1	1	N/A	1	N/A		N/A		
2.2 rep3	1	N/A	1	N/A	1	N/A	1	1		
2.3 rep3	1	N/A	1	N/A	1	N/A		1		
Total Detections	30	5	26	9	31	0	24	7	1	1

Test Module 2: 10% Fentanyl and Fentanyl-Related Compounds: Reachback for Optics-Based Instruments

Instrument	FTIR				Raman						
Sample #	Smiths Detection HazMatID Elite	Agilent Portable 4500	Redwave Technologies ThreatID	Thermo Scientific Gemini FTIR	Thermo Scientific Gemini Raman	Agilent Resolve	Chemring PGR-1064	Mira XTR DS	Metrohm TacticID-1064 ST	Thermo Scientific TruNarc	Thermo Scientific 1064Defender
2.1	1	1	1	1	1	1	0	0	0	1	0
2.2	1	0	1	0	0	0	0	1	0	1	1
2.3	1	0	0	1	1	1	0	1	1	1	1
2.4	1	1	1	0	0	0	1	1	0	1	0
2.5	1	1	1	1	1	0	0	1	0	0	0
2.6	0	0	1	1	1	1		1		1	1
2.7	1	1	1	1	1	1		1		1	0
2.8	1	1	1	1	0	1		0		1	
2.9	1	1	1	1	1	1		0		1	
2.10	1	0	1	1	1	0		0		1	
2.11	1		1	1	0					0	
2.12	1		1	1						0	
2.13	1		1	1						0	
2.14	1		1	0							
2.1 rep2	1		1								
2.2 rep2	1		1	0							
2.3 rep2	1		1								
2.4 rep2	0		1								
2.5 rep2	1		1								
2.6 rep2	0		1								
2.7 rep2	1		1								
2.8 rep2	1		1								
2.9 rep2	1		1								
2.10 rep2	1		1								
2.11 rep2	1		1								
2.12 rep2	1		1								
2.13 rep2	1		1								
2.14 rep2	1		1								
2.1 rep3	1		1								
2.2 rep3	0		1								
2.3 rep3			1								
Total Detections	26	6	30	11	7	6	1	6	1	9	3

Test Module 3: 1% Fentanyl and Fentanyl-Related Compounds: Reachback for Mass-Based Instruments

Instrument	GC/MS		HPMS		IMS					
	FLIR Detection Griffin G510		908 Devices MX908		Smiths Detection IONSCAN 600		Leidos H150E		Rapiscan Itemiser 4DN	
Sample #	Trace	Bulk	Trace	Bulk	Trace	Bulk	Trace	Bulk	Trace	Bulk
3.1	0	1	0	1	0	1	1	N/A	0	N/A
3.2	0	1	0	1	1	N/A	1	N/A		N/A
3.3	0	1	0	0	0	1	0	0	1	0
3.4	0	1	1	N/A	1	N/A		1	0	0
3.5	0	1	0	0	1	N/A	1	N/A	0	
3.6	0	1	0	1	1	N/A	1	N/A	0	N/A
3.7	0	0	0	1	1	1	1	N/A	0	N/A
3.8	0	1	0	1	1	N/A	1	N/A	0	N/A
3.9	0	1	0	0	1	N/A	1	N/A		
3.10	0	1	0	0	0	1	1	1		
3.11	0	1			1	N/A	1	N/A		
3.12	0	1			1	N/A	1	1		
3.13	0	1			1	N/A	1	N/A		
3.14	0	1			0	1	1	N/A		
3.1 rep2	0	1			1	N/A	1	N/A		
3.2 rep2	0	1			1	1	1	N/A		
3.3 rep2	1	1			1	0	0	0		
3.4 rep2	0	1			1	N/A	1	1		
3.5 rep2	0	1			1	N/A	1	N/A		
3.6 rep2	0	1			1	N/A	1	N/A		
3.7 rep2	0	1			1	N/A	1	N/A		
3.8 rep2	0	1			1	N/A	1	1		
3.9 rep2	0	1			1	N/A	1	N/A		
3.10 rep2	0	1			1	N/A	1	1		
3.11 rep2	0	1			1	N/A	1	N/A		
3.12 rep2	0	1			1	N/A	1	1		
3.13 rep2	0	0			1	N/A	1	N/A		
3.14 rep2	0	1			1	N/A	1	1		
3.1 rep3	0	1			1	N/A	1	N/A		
3.2 rep3	0	1			1	N/A	1	N/A		
3.3 rep3	1	1			1	N/A	0	1		
Total Detections	2	29	1	5	27	6	27	9	1	0

Test Module 3: 1% Fentanyl and Fentanyl-Related Compounds: Reachback for Optics-Based Instruments

Instrument	FTIR				Raman						
Sample #	Smiths Detection HazMatID Elite	Agilent Portable 4500	Redwave Technologies ThreatID	Thermo Scientific Gemini FTIR	Thermo Scientific Gemini Raman	Agilent Resolve	Chemring PGR-1064	Mira XTR DS	Metrohm TacticID-1064 ST	Thermo Scientific TruNarc	Thermo Scientific 1064Defender
3.1	1	0	1	0	0	1	0	0	0	0	0
3.2	0	0	0	0	0	0	0	1	0	0	0
3.3	0	0	0	0	0	0	0	1	0	0	0
3.4	0	0	1	0	0	0	0	0	1	0	0
3.5	0				0	0		1	0		
3.6								1			
3.7								0			
3.8								0			
3.9											
3.10											
3.11			0								
3.12											
3.13											
3.14											
3.1 rep2			1								
3.2 rep2											
3.3 rep2											
3.4 rep2											
3.5 rep2											
3.6 rep2											
3.7 rep2											
3.8 rep2											
3.9 rep2											
3.10 rep2											
3.11 rep2											
3.12 rep2											
3.13 rep2											
3.14 rep2			1								
3.1 rep3											
3.2 rep3											
3.3 rep3			0								
Total Detections	1	0	4	0	0	1	0	4	1	0	0

Test Module 4: False-Positive Results: Reachback for Mass-Based and FTIR Instruments

Instrument	GC/MS	HPMS	IMS			FTIR			
Sample #	FLIR Detection Griffin G510, Trace	908 Devices MX908, Bulk	Smiths Detection IONSCAN 600, Bulk	Leidos H150E, Bulk	Rapiscan Itemiser 4DN, Bulk	Smiths Detection HazMatID Elite	Agilent Portable 4500	Redwave Technologies ThreatID	Thermo Scientific Gemini FTIR
4.1	1	1	1	0	0	1	1	1	1
4.2	1	1	0	0	1	1	1	1	1
4.3	1	1	1	0	1	1	1	1	1
4.4	0	1	1	0	1	1	1	1	1
4.5		1	1		0	1	1	1	1
4.6		1	1		1	1	1	1	1
4.7		1	0		1	1	1	1	1
4.8		1	1		1	1	1	1	1
4.9		1	1		1	1	1	1	1
4.10		1	1		1	1	1	1	1
4.11		1	0		1	1	1	1	1
4.12		1	1		1	1	1	1	1
4.13		1	1		1	1	1	1	1
4.14		1	1		1	0	1	1	1
4.1 rep2		1	1		1	1	1	1	1
4.2 rep2		1	0		1	1	1	1	1
4.3 rep2		1			1	1	1	1	1
4.4 rep2		1			1	1	1	1	1
4.5 rep2		1			1	1	1	1	1
4.6 rep2		1			1	1	1	1	1
4.7 rep2		1			0	1	1	1	1
4.8 rep2		1			1	1	1	1	1
4.9 rep2		1			1	1	1	1	1
4.10 rep2		1			1	1	1	1	1
4.11 rep2		1			1	1	1	1	1
4.12 rep2		1			1	1	1	1	1
4.13 rep2		1			1	1	1	1	1
4.14 rep2		1			1	1	1	1	1
4.1 rep3		1			0	1	1	1	1
4.2 rep3		1			1	1	1	1	1
4.3 rep3		1			1	1	1	1	1
Total Detections	11	31	12	0	27	30	31	31	31

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Test Module 4: False-Positive Results, Reachback: Reachback for Raman Instruments

Instrument	Raman						
Sample #	Thermo Scientific Gemini Raman	Agilent Resolve	Chemring PGR-1064	Mira XTR DS	Metrohm TacticID-1064 ST	Thermo Scientific TruNarc	Thermo Scientific 1064Defender
4.1	1	1	1	1	1	1	1
4.2	1	1	1	1	0	1	1
4.3	1	1	1	0	1	1	1
4.4	1	1	1	1	1	1	1
4.5	1	1	1	1	1	1	1
4.6	1	1	1	0	1	1	1
4.7	1	1	0	0	1	1	1
4.8	1	1	1	1	1	1	1
4.9	1	1	1	0	1	1	1
4.10	1	1	1		1	1	1
4.11	1	1	1		1	1	1
4.12	1	1	1		0	1	1
4.13	1	1	1		1	1	1
4.14	1	1	1		1	1	1
4.1 rep2	1	1	1		1	1	1
4.2 rep2	1	0	1		0	1	1
4.3 rep2	1	1	1		1	1	1
4.4 rep2	1	1	1		1	1	1
4.5 rep2	1	1	1		1	1	1
4.6 rep2	1	1	1		1	1	1
4.7 rep2	1	1	1		1	1	1
4.8 rep2	1	1	1		1	1	1
4.9 rep2	1	1	1		0	1	1
4.10 rep2	1	1	1			1	1
4.11 rep2	1	1	1			1	1
4.12 rep2	1	1	1			1	1
4.13 rep2	1	1	1			1	1
4.14 rep2	1	1	1			1	1
4.1 rep3	1	1	1			1	1
4.2 rep3	1	1	1			1	1
4.3 rep3	1	1	1			1	1
Total Detections	31	30	30	5	19	31	31

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Test Module 5: Precursor Compounds and Synthesis Related, Reachback

Instrument	GC/MS		FTIR				Raman						
	FLIR Detection Griffin G510		Smiths Detection HazmatID Elite	Agilent Portable 4500	Redwave Technologies ThreatID	Thermo Scientific Gemini FTIR	Thermo Scientific Gemini Raman	Agilent Resolve	Chemring PGR-1064	Mira XTR DS	Metrohm TacticID-1064 ST	Thermo Scientific TruNarc	Thermo Scientific 1064Defender
Sample #	Trace	Bulk											
5.1	1	N/A	1	1	1	1	1		1	1	1	1	1
5.2	1	N/A	1	1	1	1	1	1	1	1	0	1	1
5.3	0	1	1	1	1	1	1	0	0	1	1	1	1
5.4	1	N/A	1	1	1	1	0		1	1	1	0	0
5.5	1	N/A	1	1	1	1	1	1	0	1	1	1	1
5.6	1	N/A	1	0	1	1	1	1	0	1	0	0	1
5.7	1	N/A	1	1	1	1	0	0	1	1	0	1	1
5.8	1	1	1	0	1	1	1	0	1	1	1	1	1
5.9	1	N/A	1	1	1	1	1	0	0	1	1	1	1
5.10	1	N/A	1	1	1	1	1			1	1	1	1
5.11	1	1	1	0	1	1	1			0	1	1	1
5.12	1	N/A	1	1	1	1	1			1	1	1	1
5.13	1	N/A	1	1	1	1	1			1	0	1	1
5.14	1	N/A	1	0	1	1	1			1		1	1
5.1 rep2	1	N/A	1		1	1	1			1		1	1
5.2 rep2	1	N/A	1		1	1	1			1		1	1
5.3 rep2	1	N/A	1		1	1	1			1		1	1
5.4 rep2	0	N/A	1		1	1	0			1		0	1
5.5 rep2	1	N/A	1		1	1	1			0		1	1
5.6 rep2	1	1	1		1	1	1			1		1	1
5.7 rep2	1	N/A	1		1	1	0			1		1	1
5.8 rep2	1	1	1		1	1				1		1	1
5.9 rep2	1	N/A	1		1	1				1		1	1
5.10 rep2	1	N/A	1		1	1				1		1	1
5.11 rep2	1	1	1		1	1				0		1	1
5.12 rep2	1	N/A	1		1	1				1		1	1
5.13 rep2	1	N/A	1		1	1				1		1	1
5.14 rep2	1	N/A	1		1	1				1		1	1
5.1 rep3	1	N/A	1		1	1				1		1	1
5.2 rep3	1	1	1		1	1				1		1	1
5.3 rep3	1	N/A	1		1	1				1		1	1
Total Detections	29	7	31	10	31	31	17	3	5	28	9	28	30

Pacific Northwest National Laboratory

902 Battelle Boulevard
P.O. Box 999
Richland, WA 99354

1-888-375-PNNL (7665)

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