

Field Detection of Seven Fentanyl Analogues Using a Portable Ion Mobility Spectrometer: A New Application for Police Officers and Medical Examiners

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Abstract

Objective: Although fentanyl is a prescribed drug in many countries, its analogues are not available by prescription. These potent synthetic opioids have become a new threat to public safety with their growing epidemic abuse and addiction worldwide. Therefore, a device for rapid, in-field, and *in situ* detection and identification of fentanyl analogues is much needed for police officers, crime scene technicians, lab medical examiners as well as public-health workers.

Methods: While a direct wipe collection method for the screening of targeted fentanyl analogues by a portable device has not been reported, a portable Ion Mobility Spectrometer (IMS) was employed to assess the detection of seven fentanyl analogues in a national forensic laboratory in North America. Under a quasi-experimental design, a purposive sampling with a black box method enabled the project to be more similar to a field detection.

Results: Seven fentanyl analogous samples were provided by the national forensic lab from its street collections that are mostly commonly encountered. In less than 10 seconds, the LED screen displays the name, concentration, and strength of each targeted analogue. The portable Ion Mobility Spectrometer device was able to successfully detect six out of the seven fentanyl analogues by a simple wipe (10~50 nanograms).

Conclusions: With a data search algorithm in the device library, the results of these tests suggest that this novel portable IMS is able to address the challenges from their variety and speeds in illicit market, providing a new direction to safely detect and identify illicit fentanyl analogues for police officers in the field and medical examiners in the lab.

Keywords: Forensic research • Toxicology • Analytical chemistry • Fentanyl analogues • Real-time *in situ* portable device

Introduction

Current detection method of opioid-based fentanyl is facing several challenges: First, novel synthetic opioids of both fentanyl and non-fentanyl analogues are often found in streets in mixtures with other opioids, posing greater risk for unintended exposure in the field. Secondly, the mixtures of the novel synthetic opioid may possess similar structures, causing more false positives or negatives by available field tests. Thirdly, the mixtures appear to have varying purity and potency, containing more potent compounds. Next, these compounds may not exist in the current reference database or may possess abnormal response behaviours similar to those of other known compounds or due to the tiny amount below detection limits. Finally, fentanyl analogues are often more difficult to detect with available field-testing devices. Therefore, a new device is much needed to address the challenges from their variety and speeds in illicit market, especially for the safety of police officers and medical examiners who are working in the first lines.

One of the earlier reports described fentanyl and its analogues as synthetic opioids with several street names such as synthetic heroin or China White in Europe [1]. Fentanyl is now a prescribed drug in many countries; yet, their potent synthetic analogues are not available by

prescription but have become a new threat due to their abuse and addiction potential with overdose-related deaths in North America. In October 2017, the U.S. Department of Health and Human Services declared that the ongoing opioid crisis was a nationwide public health emergency. Later, the Centres for Disease Control and Prevention found this highly addictive class of drugs responsible for 68% of all fatal overdoses in the U.S. in 2017 alone [2]. On April 5, 2018, the U.S. Surgeon General released an advisory on the use of naloxone for not only users of opioids but also their friends and family to help curtail the overdose epidemic [3]. Therefore, fentanyl and its analogues have become a great concern to law enforcement and public health professionals as they cost tens of thousands of lives each year in the U.S. and Canada. Finally, the impact of fentanyl analogues is becoming a worldwide problem where these analogues are appearing and causing overdose deaths requiring expert bodies to assess their impact [4].

Canada has been particularly struck by the opioid crisis, especially in the province of British Columbia (B.C.). On April 14, 2016, the province of B.C. proclaimed a "Public Health Emergency" to alert policy makers, the public and the media as to the impact of synthetic opioids in local communities. In 2016, there were 668 overdose-related deaths, where fentanyl was detected; it rose to 1,228 in 2017 and 1,334 in 2018 for all of B.C. [5]. For the city of Vancouver, the largest city in B.C., the annual figures for overdose-related deaths during the same period rose from 152 to 306, and then to 337, respectively. To put this into perspective, in Vancouver in 2018, the overdose death rate for fentanyl was determined to be 53 per 100,000 [4]. Given this unprecedented public health crisis, not only across the U.S. and Canada, but also around the globe, there is a concern for the welfare of first responders, law enforcement officers and public health workers to enter, or process a scene where fentanyl or an analogue may be a suspected contaminant. The relationship of the potency between fentanyl, two fentanyl analogues, and two opiates [6] are shown in Figure 1.

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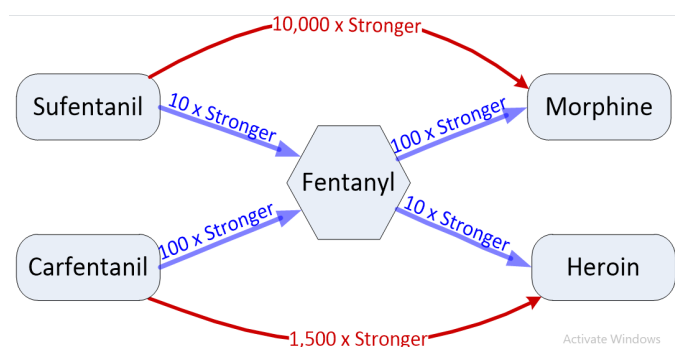


Figure 1. A comparison of potency amongst fentanyl, its analogues carfentanil and sufentanil, and the opiates morphine and heroin.

A portable device that can detect residual fentanyl analogues in a rapid and in-situ manner in the field would be a powerful tool for processing crime scenes or death related scenes in many urban settings for law enforcement and public health workers. Items such as clothing, cigarette packages, tents, deceased individuals, mails, or parcels could be all tested with such a device. Drug paraphernalia such as abandoned drug apparatus (needles or pipes) that are discarded on the street or found in an alley can often consist of complex mixtures containing fentanyl or its related analogues. These items would be amenable to analysis by this technique. Finally, pre-screening samples in the field would create efficiencies in processing items for submission to a forensic laboratory and would minimize risks to anyone handling the items as a time-saving and cost-saving measure.

Some Ion Mobility Spectrometer (IMS) techniques have been used in laboratories to determine the variability of the detection window for certain controlled substances such as fentanyl [7]. However, there is a paucity of forensic literature on the application of a portable IMS field device for fentanyl analogues analysis. There is a practical and urgent need for such a device for both in-field and laboratory environmental assessments for the presence of low-level fentanyl-related analogues.

The potency of many fentanyl analogues is unknown and may be present at very low concentrations in seized samples, e.g., several nanograms per millilitre or per gram. Therefore, the presence of these analogues may represent a significant overdose risk to individuals exposed to them. Traditional methods of analysis such as Gas Chromatography-Mass Spectrometry (GC-MS) and Liquid Chromatography-Mass Spectrometry (LC-MS) are limited due to requirements for sample preparation and analysis time. Also due to their physical size, these devices would require some type of costly mobile laboratory setup to do fieldwork. Therefore, a rapid, in-field, and *in situ* detection and identification of illicit fentanyl analogues method is much needed. The purpose of this study is to assess the use of IMS for field use and pre-screening and identification of unknown samples for seven fentanyl analogues.

Materials and Methods

In recent years, some well-known analogues or compounds within the fentanyl group such as fentanyl, alfentanil, sufentanil, or remifentanyl are used for medical anesthetic or analgesic purposes, some other compounds, in addition to fentanyl, are being abused as illicit narcotics such as carfentanil. In fact, more than twenty plus new fentanyl analogues or compounds have been found in street samples. They have posed new handling threats, as well as adding more challenges to the examination and identification process. Therefore, a new detection method, device, and technique are required to meet the new challenges.

Quasi-experimental design method

In order to represent a maximum similarity to an in-field as well as *in situ* detection and identification, this study was conducted in a national forensic laboratory in North America. Seven illicit fentanyl analogues were provided from street seized samples in the previous two years and have been identified and confirmed by the laboratory. Figure 2 shows the chemical structures of the seven fentanyl analogues used in this study with designated numbers 1, 2, 3, 4, 5, 6, and 7 and their corresponding chemical names, as listed in Table 1.

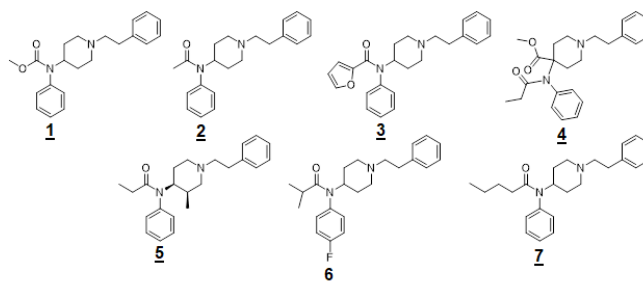


Figure 2. Chemical structures of the seven fentanyl analogues that were test in the study.

Table 1. The analysis results of 7 fentanyl analogues using the RTA-180P IMS.

Analogue 0.047		0.047	0.047	0.047	0.047	0.047	0.047	0.047	0.047
No.	Fentanyl Analogues	Result at the First Wipe	Time (Sec.)	Possible Reasons	Result at the Second Wipe	Retest Time (Sec.)	Peak Shown	Concentration Shown	Ground Truth Confirmed
1	Methoxyacetyl fentanyl	No	~10	Not in the Original Library	Yes	~5	Yes	Yes	Yes
2	Acetylfentanyl	No	~10	Too Small Input or Similar Peaks	No	~10	N/A	N/A	No
3	Furanylfentanyl	Yes	~8	N/A	N/A	N/A	Yes	Yes	Yes
4	Carfentanil	No	~10	Not in the Original Library	Yes	~5	Yes	Yes	Yes
5	3-methylfentanyl	Yes	~8	N/A	N/A	N/A	Yes	Yes	Yes
6	para-Fluoroisobutryl fentanyl	No	~10	Not in the Original Library	Yes	~4	Yes	Yes	Yes
7	Valeryl fentanyl	Yes	~7	N/A	N/A	~5	Yes	Yes	Yes

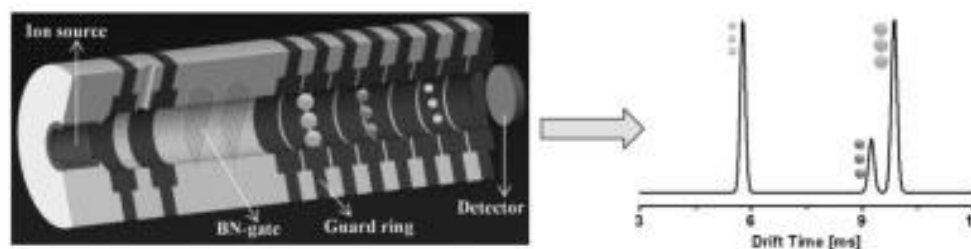


Figure 3. The schematic diagram of the IMS working principle (provided with courtesy from the manufacturer).

Due to limitations of time and resources, a quasi-experimental design was adopted for this study since its design is an empirical method estimating the causal impact of a treatment or an intervention on its targeted samples [8]. The design consists of three main components. First, a quasi-experimental research does not require preliminary random sampling (assignments) for treatment or control. Instead, a quasi-experimental design typically allows the research team to select targeted samples based on the availability of the samples within a certain criterion. In this study, the seven fentanyl analogues chosen as purposive or targeted samples were those most frequently encountered by law enforcement and public health agencies based on its record in the metropolitan area of the hosting laboratory. Secondly, the research is designed as a black box study (a blind test) where the identities of the seven samples are already known to the laboratory, but unknown to the author and his assistant (the instrument operator), thus making each sample test of the seven fentanyl analogues a validation test on the accuracy level of a novel device, the IMS. Further, the test is considered a failure if the device does not show the name of the tested analogue known to the laboratory. Thirdly, due to the potent nature of the fentanyl analogues and concerns of potential exposure of the frontline workers, the sensitivity level of the sample input was chosen at a nanogram level as a selection criterion. Each sampling size (estimated between 10 and 50 nanograms) was purposely controlled by a tiny wipe of each fentanyl analogue. Finally, there are three main types of quasi-experimental designs, each with different focuses, strengths, weaknesses, and applications, namely, pre-test versus post-test, propensity score matching, and instrumental functions [8]. This study focused on the instrumental functions of a portable IMS by testing the level of performance efficiency or technology capacities, namely, accuracy and sensitivity.

The IMS device

The detection of the seven fentanyl analogues in this study was carried out using RTA-180P IMS. The device utilizes a combination of the atmosphere pressure photoionization with the auxiliary chemical assisted ion-molecular reaction for targeted sample ionization. A sampling cloth is used to wipe the testing surface of an area that is suspected of having been in contact with fentanyl analogues. A thermal desorption technique forms a gas-phase sample molecule when the sample collecting cloth is inserted into the sample inlet slit. The desorption temperature is set at 200°C to efficiently desorb and volatilize the fentanyl analogues from the solid/liquid phase on the sample cloth to the gaseous phase. A carrier gas of ambient air transports the sample molecules through the thermal desorption inlet, where they mix with acetone that is used as a reagent gas in the chemical ionization process within the photon ionization region of the IMS. The vacuum ultraviolet photons from a krypton lamp ionize the acetone molecules to form precursor ions. The sample molecules will then undergo a series of charge transfer ion-molecular reactions with these precursor ions to form charged sample ions.

The ion drifting time interval in the IMS drifting tube is set to 20 milliseconds. The ion drift tube was configured to detect positive ions in the Narcotics Detecting Mode at a temperature of 105°C. The sample ions enter the ion mobility tube through a periodically opened ion gate as ion pulses. They arrive at the Faraday receiving disk that converts the charges of the ions to a current signal allowing the device to create an ion mobility spectrum for review. The IMS spectrum data is then averaged by multiple

scans to improve the signal to noise ratio. A total of 20 scan averages form a single IMS spectrum for analysis. A detection algorithm is used to analyze each spectrum by comparing the unknown compound peak positions against those in the database. The mass to charge ratio and the charge transfer or collision cross sections in the atmosphere vary among different sample ions. Accordingly, ion mobility spectrometry uses this migration time difference to identify the target sample molecules. Therefore, by measuring the ion mobility spectra of the ions by its characteristic migration time, the device can then compare it to a stored standard library in the instrument to search for a match. For selected compounds, a detection threshold of minimum peak intensity count is used to trigger an alarm using a unique internal calibration. The threshold settings on the instrument can be adjusted to one of three settings by the user based on their particular situation. Figure 3 shows the schematic diagram of the IMS working principle.

Testing procedures

Since access to the resources of a forensic laboratory was limited by time and legal regulations on the handling of controlled compounds, this study employed a ground truth approach similar to a black box study design for a blinding observation. While the IMS device in use contains basic spectral data for fentanyl analogues in its existing library, the seven fentanyl analogues had already been identified and confirmed from previous examinations by the hosting laboratory. However, they were unknown to the IMS device, the author, and the operator. To assess the capabilities of the IMS device to identify fentanyl analogues, the following four criteria were examined: (1) Is the IMS device able to accurately identify the particular fentanyl analogue with its existing spectrum library (identification capacity)? (2) Is the IMS able to detect a nanogram amount of the fentanyl analogues (sensitivity capacity)? (3) Is the IMS able to add a new analogue spectrum and re-identify it if the first test fails (re-programmability capacity)? (4) Is the device able to perform the detection in a rapid, in-field, and *in situ* manner (performance capacity)? Safety precautions were carefully reviewed and exercised before and during laboratory testing. The following are the steps for the quasi-experimental design study.

Sampling procedure

Step One—Purposive Sampling: Seven fentanyl analogues in powder form were selected out of the total 21 fentanyl analogues from the laboratory's seized samples. Although these seven analogues were non-randomly chosen, they were the most commonly encountered fentanyl analogues submitted by either law enforcement or public health agencies within the previous two years, thus qualifying a targeting sampling method.

Step Two—Sub-random Sampling: While the preliminary non-random samples ($n=7$) were adopted due to the limits of availability and handling illicit drug regulations, each sample analogue in the secondary sampling ($n=7$) was randomly selected to reduce the threat of a lack of internal validity. In this way, the quasi-experimental design can reduce a possible sampling bias and render the test results to be proximately comparable at a baseline. Since one of the focuses of the quasi-experimental study is to test the instrument functions via its sensitivity level, each sample input was equally controlled with the same procedural steps.

Detection procedure

Step Three—Sample Collecting: To maximize a scenario of an in-field

and *in situ* sample collection by direct wiping without any sample preparation, the author, the lab assistant, and the device operator started the testing by wearing the protective gear required by the laboratory. First, a needle was dipped into a targeted sample analogue and gently touched to a fixed point on a piece of blank paper on the table. Second, the targeted analogue on the blank paper was gently touched with a cloth meta-aramid wipe. This resembles a real-world wipe of an in-field and *in situ* nature that provides a non-intrusive, non-invasive, and non-destructive sample collection. Finally, following the required safety protocol by a well-ventilated fume hood, the wipe was tapped three times for a nanogram level of sample input. Based on the previous testing with other narcotics, this measure allows for the input into the device around 10–50 nanogram of the sample input.

Step Four—Sample Input: Once the cloth wipe (Length=60 millimetres, Width=20 millimetres) was inserted into the sample inlet of the device, the IMS analysis began with the push of one button and tested and re-tested continuously due to its unique automated calibration system. The automated cleaning time between samplings varied depending on the previous sample weight, ranging from 1 to 3 minutes.

Step Five—With-in seconds, the 5-inch Thin-Film-Transistor Liquid Crystal Display (TFT-LCD) provides a real-time result with a warning light signal and sound. The analysis result includes the analogue name with a graphic display (the peak and the concentration levels) on the LED touch screen display.

Results and Discussion

Currently, the most common analytical techniques employed in forensic drug laboratories are GC-MS or LC-MS. While these two types of instrumentation provide high levels of sensitivity for qualitative and quantitative results, these instrumentations also have some limitations: (1) They require a significant amount of time for sample preparation and measurement (1 hour or more) to produce their results. (2) The examination must be performed by well-trained personnel. (3) The equipment must be situated in a laboratory environment. Due to these limitations, these two types of instrumentations offer little practical value for a rapid, in-field, and *in situ* detection and identification for law enforcement and public health personnel when processing any scenes or persons that are potentially contaminated with illicit fentanyl-related analogues.

Alternatively, Raman spectroscopy has been used for fentanyl detection for evidence-submission in laboratories and sometimes used in on-site investigations [9]. However, a portable Raman device, being smaller than a laboratory-based instrument, usually emits weaker signals required for identification, often encounters fluorescent interference, and continuously provides poor results for mixed samples [9]. Further, a Raman hand-held device requires a macroscopic amount of sample, as much as tens of milligrams, and often has a cut-off of a few percent concentration in mixed fentanyl samples [10].

The results of the analysis of seven fentanyl analogues are reported in Table 1. This quasi-experimental project employed a black box design, meaning it is a validation or a ground truth confirmation test because the identities of the seven analogues were already known to the laboratory (known samples), but unknown to the author.

Out of the seven fentanyl analogues tested, the IMS device accurately identified Furanyl Fentanyl (FF), 3-Methyl Fentanyl (3-MF), and Valeryl Fentanyl (VF) with clear graphics of the peaks and concentrations on the first wipe. The results for methoxyacetylfentanyl, carfentanil, and para-fluoroisobutyrylfentanyl were positively identified following a second wipe after the spectral data for each compound was added into the library. The IMS possesses a valuable function of adaptation: the tested spectrum can be easily added into the existing library using a special data search algorithm with a mathematical formula and the updated library can then be searched immediately for repeat or future analyses. However, acetylfentanyl was the only analogue that did not trigger any alarms because the IMS device did

not correctly identify it after the two wipes. There may be three reasons that account for the missed identification. The sample wipe may not have actually touched the compound that had been deposited on the blank paper since it was visually impossible to see where the material was placed. Alternatively, another source of error could occur due to the compound falling off the wipe following the three taps and prior to being placed into the inlet of the device. However, there may exist another possibility that the acetylfentanyl produced an identical molecular ion peak with one of the fentanyl analogues in the existing library due to their fragmentation spectra [11,12]. Further examinations are needed to explore this non-identification with this compound.

The advantages of the analytical performance with the IMS device are three-fold:

(1) The device is portable and compact, which is suitable for in-field detections.

(2) Its analysis or detection time is measured in seconds by a real-time screen display.

(3) The device is very sensitive to fentanyl residue at the nanogram level providing a definitive examination for an *in situ* sampling. Most importantly, the IMS device used in this study employs a non-radioactive photoionization method to produce sample ions for detection. The portable system weighs only 4.8 kilograms with the battery and has dimensions of Length=430 millimetres × Height=190 millimetres × Width=180 millimetres. A 5-inch touch Light-Emitted Diode (LED) screen can provide a real-time detection result (a graphic table display) as well as a sound signal and colour indicator for rapid identification. The screen graphic provides a figure consisting of a column bar and a list of names that trigger the alarm. The ion mobility of each compound detected is listed in the database by the manufacturer or user-created library and in the internal memory that can be readily retrieved and read. A computer can be connected to the detector via Wi-Fi so that the results can also be downloaded for review on the computer screen or for remote communication. Finally, the Limit of Detection (LOD) is estimated to be in the ng range (10^{-9} g) with an analysis time of seconds.

The sensitivity level can be adjusted to meet different concentration level criteria. The other physical features include a built-in SanDisk memory card capable of storing up to 100,000 test results and a Wi-Fi interface that can be used to send result information to a computer, connect to the main spectral library, and monitor the test within a distance of up to 10 metres. Therefore, the device can be taken into a hot-zone or an emergent environment, if needed.

While the IMS device is unable to provide quantitative results, it can be an effective qualitative tool in identifying illicit fentanyl-related analogues on evidential items, persons, or surfaces. The IMS device under the quasi-experimental study was able to identify six out of seven illicit fentanyl-related analogues with the first wipe or the second wipe after adding the compound into the library within 10 seconds or less. Practically, the IMS method does provide a viable approach as a portable unit for rapid, in-field, and *in situ* screening device.

In 1979, a series of deaths in California due to the fentanyl analogue, alpha-methyl fentanyl became the first recorded incident of a completely novel synthetic opioid, thus earning the nickname “designer drug” [13]. In the next forty years, the illicit use of fentanyl and its analogues has become a world-spread problem due to its diversion, namely, its twenty more analogues.

Drug analysis is a process of employing several analytical steps to determine the compositions of suspected controlled substances in seized drugs. Typically, most forensic drug laboratories utilize a two-step process: a screening technique followed by a spectroscopic confirmation step. A presumptive screening test typically indicates if the sample contains a controlled substance. However, such tests may produce false positives or false negatives depending upon the selectivity and sensitivity of the technique employed. Therefore, a confirmatory test is required to identify

the substance using more advanced equipment, and the result must also corroborate the result from the screening technique. Uniquely, the IMS serves both the functions of preliminary and confirmatory tests together for a definitive identification due to its advanced data search algorithm technology with the ion mobility spectrometry.

In this study, the IMS device was tested with seven fentanyl analogues. The portable device demonstrated four specific functions: (1) It was able to accurately identify three fentanyl analogues (FF, 3-MF, and VF) with its existing spectral library (identification capacity). (2) The IMS device was also able to detect the nanogram amount fentanyl and/or its analogues residue at a 10 to 50 nanograms level (sensitivity capacity). (3) The device was able to add multiple fentanyl analogue spectra and identify another analogues: methoxyacetylfentanyl, carfentanil, and para-fluoroisobutyrylfentanyl when the first tests failed (re-programmability capacity). (4) The device was able to perform the detection in a rapid, in-field, and *in situ* manner (performance capacity).

This quasi-experimental study [14] has demonstrated the four unique instrumental functions: sensitivity, identification, re-programmability, and performance capacities with a high degree of specificity. With direct sampling capacities and without the need for an extraction procedure, the IMS device was able to perform rapid, in-field, and *in situ* identifications of six fentanyl analogues at nanogram level concentrations. The traditional colour tests play an important role in a presumptive purpose and require confirmation due to cross-reactivity with other compounds/materials. Comparatively speaking, the IMS represents a distinct advantage over colour tests for field analysis of potent fentanyl analogues due to the four functions mentioned above. It is suggested that the IMS may be a new direction to help minimize the risk of exposure to illicit fentanyl analogues [15-17]. Thus, with required protection gear and fewer direct contacts with suspicious powder substances or items that might be contaminated with powder substances that may be potentially fentanyl analogue-related, police officers, crime scene technicians, laboratory examiners, and public health workers could simply and directly wipe a surface, insert the wipe into the inlet of the portable device, and receive a graphic analysis result within seconds. If the existing library does not possess the spectrum, the initial result can be added into the library.

Conclusion

In conclusion, this IMS approach can be used to greatly reduce the risk of occupational exposure by fewer contacts with the powder form of illicit fentanyl-related analogues. The IMS device claims to detect the nanogram level of illicit drugs for a safety precaution screening, and thus it can be used by law enforcement and public health personnel. Future research based on this quasi-experimental study could include studies on more illicit fentanyl analogues, in a mixed sample with other narcotics, and on fragmentation ionization. It is argued that the new device is much needed to address the challenges from their variety and speeds in illicit market, especially for the safety of police officers and medical examiners who are working in the first lines.

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