WHITE PAPER

Illicit Drug Trafficking and Border Control with Mira DS: Safe Fentanyl Identification

As with heroin and cocaine, the vast majority of fentanyl found in the United States is made by foreign producers and illegally transported to ports and across US borders. Policing national borders is crucial to prevent illicit substances from entering the country. Fentanyl, a synthetic opioid much more potent than morphine, is an especially harmful drug and can lead to death even in miniscule amounts. Increasingly, it is mixed with other drugs to create a more intense effect with less cost to the producer. Fentanyl overdose incidents are increasing, including greater potential to exposure for law enforcement during narcotics field testing.

Border control and customs agents already rely on portable chemical identification systems, such as handheld Raman devices. These are rapid and accurate methods for illicit drug ID that can be easily operated in the field with minimal user training. This white paper describes on-site detection of both bulk fentanyl samples (Raman testing) and fentanyl as a trace component in mixtures (ID Kit Illicit Material trace detection analysis) with Mira DS through quality data collection from samples containing fentanyl and fentanyl-related compounds.

Metrohm
Raman

FENTANYL AT THE BORDER

The worldwide opioid epidemic is intensified by the abuse of synthetic opioids, including fentanyl and its analogues. Originally used as a highly effective surgical analgesic, fentanyl abuse can be attributed to both its availability and enhanced potency over morphine and heroin for achieving the sense of euphoria sought by drug users [**1**]. Fentanyl is also highly toxic, straining public health resources in affected communities with a substantial increase in drug-related hospitalizations and overdose deaths [**2**]. This results in additional burden on First Responders and drug law enforcement entities.

The bulk of fentanyl recovered in the United States is synthesized in China and transported by air and water to designated ports of entry [**3**]. While China has initiated efforts to stem the illegal flow of fentanyl overseas, Mexican drug cartels sponsor illegal drug manufacturing facilities and direct large-scale shipments across the US-Mexican border. This is demonstrated by the ~60-fold increase in the bulk weight of fentanyl confiscated by the US Border Patrol between years of 2015–2020 [**4**]. Moreover, less than 15% of total annual US fentanyl confiscation occurs at the border, indicating that the vast majority of the drug is successfully delivered for processing and distribution [**5**].

FENTANYL ID WITH HANDHELD RAMAN

Ideally, portable drug identification methods should be compact, rugged, easy-to-use and accurate for success in a range of different test environments.

Fentanyl testing poses some very specific challenges for Raman analysis:

- Some pure fentanyl analogues may fluoresce and overwhelm instrument sensors.
- Fentanyl is most commonly found as a trace component in a complex sample.
- Excipients used as fillers in complex samples can either dominate the Raman signal or may themselves fluoresce.
- Traditionally, complex laboratory separation methods are required before identification of trace analytes is possible.

Metrohm's **Mira DS handheld Raman spectrometer** and **ID Kit** with test strips are ideal solutions for identification of fentanyl in the field. ID of pure fentanyl in bulk amounts with handheld Raman is a simple point-and-shoot exercise that yields results in seconds. When fentanyl is present in a mixture, as is typical of street samples, ID Kit can be used for positive identification. Trace ID requires a quick and easy sample preparation, followed by analysis with Mira DS for fast onsite results.

STANDARDS FOR FENTANYL TESTING

This white paper demonstrates conformance with government test standards such as ASTM (American Society for Testing and Materials) for positive ID of bulk and trace fentanyl and its analogues [**6**]. Testing standards provide rigorous guidance in establishing minimum criteria required for the qualitative identification of illicit substances. Defined assays and instrument performance assessments validate practices for optimal Raman identification of fentanyl in the presence of cutting agents and chemical interferents (e.g. fluorescent additives) that can confound identification.

METHODOLOGY

– BULK AND MIXTURES METHODS

For pure reagents, 5 mg of powder in a glass vial was measured directly through glass with the Long Working Distance (LWD) Smart Tip. For mixtures, stock solutions were prepared at 5 mg/mL by dissolving solids in water or methanol. To simulate street samples, stock solutions of fentanyl, other illicit drugs, and common cutting agents were mixed in recommended proportions with fentanyl. These solutions were evaporated in glass vials and the dried residues were analyzed with Mira DS and LWD. All acquisition parameters were automatically optimized by Smart Acquire, which determines laser power, integration time, and spectral averaging for high quality, instant results.

– ID KIT METHODS

Measurements were collected for pure fentanyl analogues and for mixtures containing fentanyl in combination with excipients and other drugs using ID Kit test strips. Pure samples were prepared through direct application of stock solutions onto ID Kit test strips. Each test strip was placed into the SERS-A Smart Tip on Mira DS for measurement. Fentanyl was extracted from mixtures by shaking 2–3 mg of mixed samples with ethyl acetate from ID Kit, then placing 2–3 drops of this solution onto an ID Kit test strip. Use of the SERS-A Smart Tip on Mira DS activates ID Kit OP Smart Acquire routines, which also automatically optimize acquisition parameters.

RESULTS AND DISCUSSION

– RAMAN SPECTRA OF FENTANYL AND ANALOGUES

Figure 1 shows stacked Raman spectra acquired for pure samples of *cis*- and *trans*- isomers of 3-methyl fentanyl and two chemically related analogues. While the spectra are visually similar, their peak positions and intensities are sufficiently different for effective discrimination.

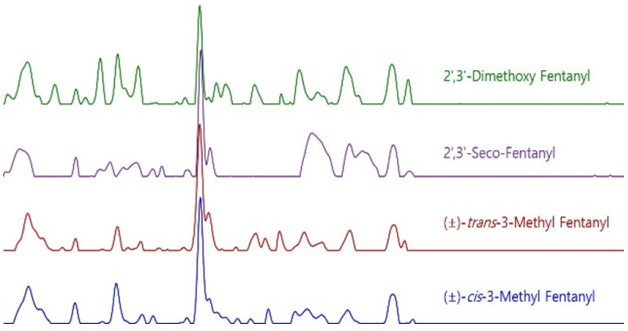

Figure 1. Raman spectra comparing different reagent-grade fentanyl analogues.

The Metrohm library development project contains **more than 230 fentanyl analogues** that can be quickly and easily discriminated by Mira DS.

– IDENTIFICATION OF FENTANYL IN MIXTURES

Fast, onsite ID of illicit drugs in complex mixtures containing excipients is one of the great challenges of forensic sample analysis. As an example of this, the Raman spectrum of 1 mg/mL fentanyl in mannitol found in **Figure 2** is dominated by the mannitol signal.

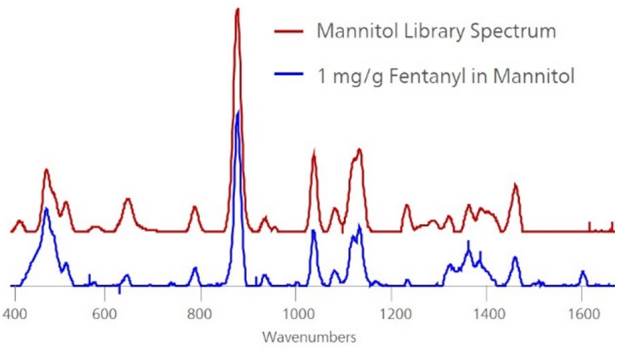

Figure 2. Comparison of Raman spectra for pure mannitol (red) and a mixture of fentanyl cut with mannitol (blue).

Recall, Raman is a bulk identification method, and therefore only the bulk component in a mixture is detected. However, SERS capabilities on Mira DS can successfully ID trace materials.

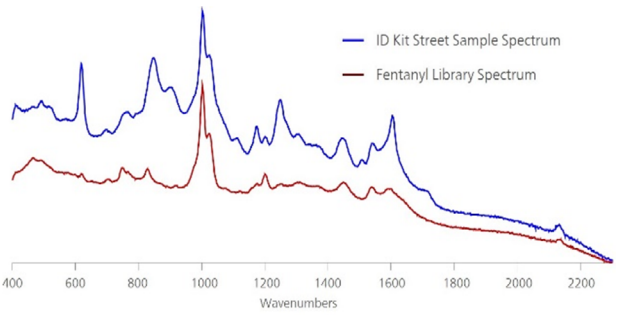

Figure 3. Overlaid ID Kit street sample (blue) and Fentanyl Library (red) spectra.

With ID Kit, fentanyl can be selectively extracted from complex mixtures, resulting in a spectrum with **improved resolution and sensitivity**.

As shown in **Figure 3**, the street sample extracted with ID Kit (blue) yields a spectrum with several peaks that agree with the Fentanyl Library Standard for ID Kit Illicit material library, resulting in a positive match for fentanyl.

– RAMAN AND ID KITS ENHANCEMENT COMPARISON

ID Kit uses a technique called Surface Enhanced Raman Scattering (SERS) to enhance the Raman signal of trace levels of fentanyl and other illicit materials. Raman and SERS spectra of confiscated drugs often express fluorescence from the target and/or the excipients which obscures spectral peaks required for positive ID [**7**].

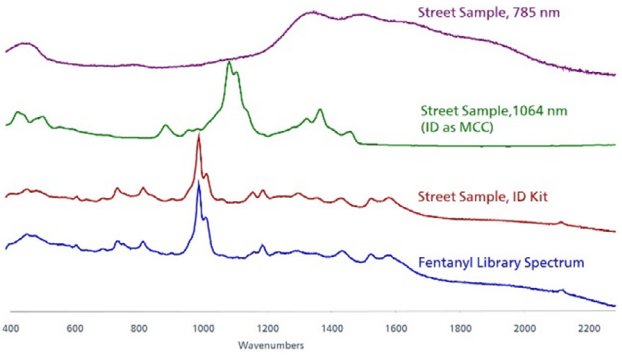

Figure 4. Overlaid 785 nm (purple), 1064 nm (green), ID Kit street sample (red), and Fentanyl Library (blue) spectra from a street drug sample containing fentanyl.

The top spectrum in **Figure 4**, collected with 785 nm Raman excitation, exhibits a broad and smooth profile characteristic of fluorescence, obscuring signature peaks. The 1064 nm excitation returned a positive ID of the primary excipient in the sample, microcrystalline cellulose (which can exhibit fluorescence at 785 nm).

A simple sample extraction with ID Kit and subsequent analysis with Mira DS reveals a result consistent with the ID Kit Illicit material library spectrum of fentanyl.

CONCLUSION

Rapid and accurate drug identification is a key requirement for border control and customs agents who guard and protect international borders. **Mira DS handheld material identification system with ID Kit from Metrohm Raman** is a compact and rugged system that is easily operated with minimal user training, even in the most demanding conditions. MIra DS and ID Kit with test strip technologies offer considerable promise for accurate, sensitive, and selective identification of fentanyl and other substances in seized drug shipments, moving forensics out of the lab and to the front line.

References

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