Application Note

Forensics

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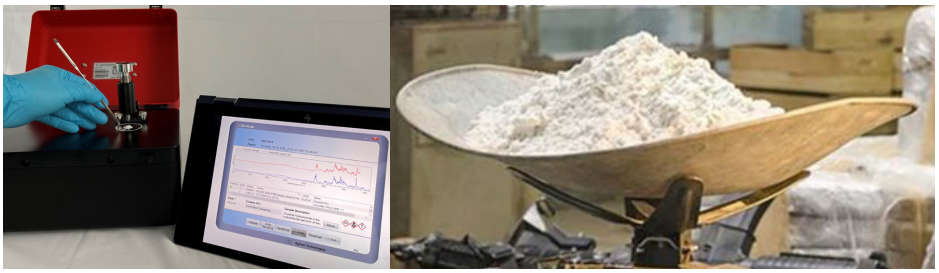
Identification of Narcotics and Related Substances with Portable FTIR ATR Analyzers

Fast results using the Agilent 4500a and 5500a FTIR forensics analyzers



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Abstract

Fourier transform infrared (FTIR) is a technique that uses infrared light to probe a sample where the resultant spectra is highly specific to the chemical and structural composition, and thus the substance can be identified with confidence. Specifically, attenuated total reflectance (ATR) sampling interfaces using diamond as the internal reflecting element have risen to the fore due to their inherent toughness and ease of use over traditional IR techniques. The Agilent 4500a and 5500a FTIR forensics analyzers are two ATR FTIR spectrometers that utilize a diamond ATR sampling interface and are designed for at-field or in-field use. The increasing prevalence of illegal drugs in all markets globally requires an instrumental technique that is simple to operate, reliable, with high sensitivity and selectivity, and can be operated inside or outside the lab. This application note describes the use of the portable 4500a and the transportable 5500a FTIR forensics analyzers for narcotic detection, and demonstrates some new features in Agilent MicroLab 5.7 software. Many conventional street narcotics and new psychoactive substances (NPS) were positively identified, along with a common cutting agent. Moreover, new features in MicroLab are shown that can warn nonchemists to identify the presence or absence of such controlled substances. Finally, the selectivity of FTIR ATR is demonstrated with three closely related analogs of an NPS.

Introduction

The world's population has grown by about 10% in the last decade (2010 to 2020), while the number of narcotic users has increased by 22% in the same period.[1] The prevalence of the five major classes of narcotic (cannabis, opioids, amphetamine-type stimulants, cocaine, and NPS) has risen. To help combat the illicit increased supply, an analyzer or instrument that can positively identify suspicious substances in the field with high specificity and sensitivity is needed. This would aid and alleviate the strain on forensics departments and law enforcement around the world, and could even help fast track prosecutions where appropriate.

The 4500a and 5500a FTIR forensics analyzers provide portable and transportable capabilities. These capabilities are coupled with a dedicated diamond ATR sampling interface, which enables a "vibrational molecular fingerprint" of the substance to be collected and compared to library data, resulting in a ranked display of potential matches. The diamond ATR sampling interface is well suited to a wide variety of material states and forms, and provides universal coverage for all narcotic types, combined with minimal or no sample pretreatment. Figure 1 shows some highlights of the 4500a and 5500a FTIR forensics analyzers and supplied software.

FTIR spectroscopy has a long and proven heritage of identifying substances, from early Nujol mull-based transmission legacy libraries to more current and comprehensive ATR libraries. For narcotic analysis the 4500a and 5500a can be paired with the ATR Forensics Library for Mobile FTIR and the TICTAC FTIR ATR drugs library. The ATR Forensics Library contains a diverse variety of compounds, mixtures, conventional narcotics, white powders, explosives, and fibers, whereas the TICTAC FTIR ATR drugs library contains conventional narcotics and NPS, plus analogs, common precursors, and cutting agents.

For library searching, MicroLab software provides a wide variety of algorithms with extensive search and display options plus reporting choices. Custom library creation and a commercial library are both available, as well as ad hoc spectral entry addition to custom user-created libraries.

- Easy to use, graphically driven, minimal training required for successful operation; the 4500a has a self-contained four-hour battery
- Little-to-no sample preparation
- Produces highly reproducible spectra
- Detailed spectral "pattern", perfect for library matching
- Fast results with an analysis scan time of less than 30 seconds; the quoted 3 minutes includes cleaning the crystal before and after sample analysis and a review of the data
- Can search commercial and custom user-created libraries
- Suitable for a wide variety of substance states or forms: liquids, crystals, powders, pastes, gels, films, tablets, flours, plant cuttings, soils, and so on

Figure 1. Features of the Agilent 4500a (left) and 5500a (right) FTIR forensics analyzers. The footprints of the 4500a and 5500a are 22 × 29 cm (equivalent to a piece of A4 paper) and 20 × 20 cm respectively, and they weigh in at 6.8 and 3.6 kg, respectively.

Experimental

In this application note, the Agilent 4500a and 5500a FTIR forensics analyzers equipped with single-bounce ATRs were used to identify a variety of conventional and new psychoactive substances. The method was also performed to demonstrate new metadata features in Agilent MicroLab 5.7 software in conjunction with the TICTAC FTIR ATR drugs library and the ATR Forensics Library for Mobile FTIR. The 5500a is optically identical to and uses the same software as the 4500a, and therefore would be expected to yield identical search results. A qualitative library search method with the following conditions were used on both the 4500a and 5550a: sample and background scans were set at 50 with a resolution of 4 cm^{-1} and zero-fill factor of 2, taking 22 seconds for data acquisition of each substance. When including (A) cleaning the crystal before and after data acquisition stages, (B) transferring the sample onto the crystal, (C) engaging the clamp, (D) inputting the details, (E) performing the library search, and (F) presenting the ranked results, the workflow duration per sample was approximately two to three minutes.

Results and discussion

Onsite analysis by portable FTIR ATR is useful for the identification of:

- Probable cause
- Samples for seizure
- Forensically significant samples
- Unlabeled or suspiciously labeled products/materials
- Hazard and risk assessments

To combat the increase in illegal drugs, FTIR is a fast, reliable, sensitive, and highly selective spectral technique that is well suited to sample identification. However, the system operation should be designed for nonscientists with features that assist first responders and law enforcement.

MicroLab software features pictorial guidance with highly tailorable search criteria combined with some new display features and options. The method, libraries, and 4500a/5500a instrument collectively represent an efficient analyzer that performs a narcotic library search with minimal fuss, and displays clear, actionable results. Both systems are optically completely sealed and safe to use for prolonged periods as necessary. No special setup or in-depth user training is required for the instrument's narcotic workflow. Nevertheless, the appropriate precautions and PPE for suspected narcotics should be adhered to.

Six simple steps to a library search result

Although diminutive in size when compared to lab-based instruments, the performances of the 4500a or 5500a are on par or exceed traditional standard lab FTIR instruments. A reliable, field-proven, and durable optical engine tuned to yield both high spectral sensitivity, selectivity, and long-term stability is coupled with easy-to-use software and a dedicated diamond ATR sampling interface. Together this enables a sample preparation-free analysis that yields consistent results. The software is method driven with individual steps that are graphically supported, and sample turnaround times in a few minutes at most. Figure 2 shows the library search process for a street-confiscated sample of heroin. The process is graphically guided in six simple steps from initial method selection and initiation to the library hit quality index (HQI) results, which are ranked from best to worst. The minimum threshold hit quality and the number of hits to be shown are both fully tailorable, as is the choice of search algorithm. The most important stage during the unknown sample analysis is the live-view assessment of the sample-to-diamond contact; if it is low, the user can reseat the sample, adjust the clamp, or add more sample. Typically, the end user should aim for >0.2 Abs peak maximum, and the simply guided, software-led process in Figure 2 takes less than two minutes from start to finish.

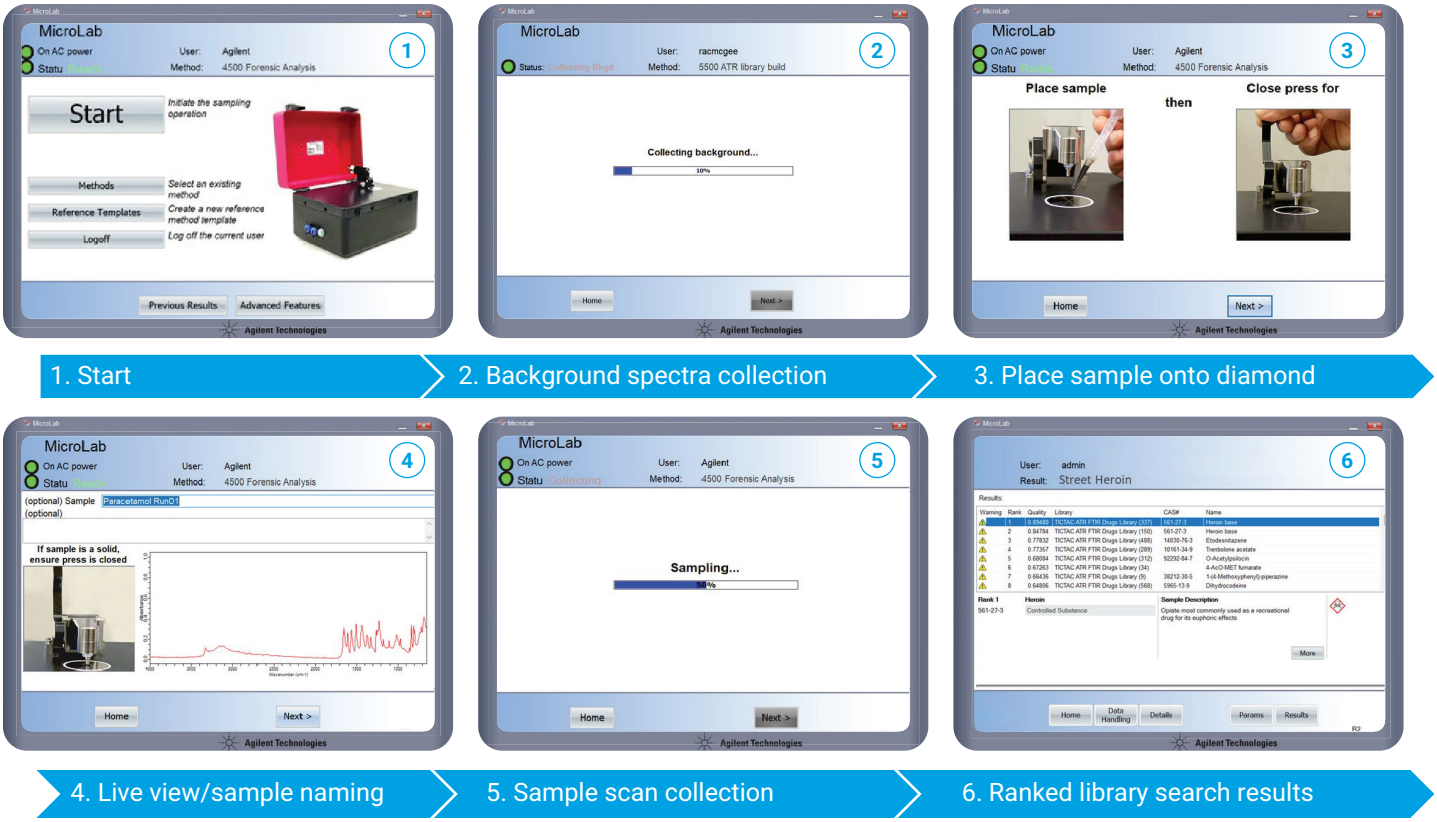

Figure 2. The six steps of a library search method (called qualitative search in Agilent MicroLab software). Results are for a street heroin sample that had an HQI of 0.89480 (a perfect match would be 1.0000).

The library search process consists of the following steps labeled in Figure 2:

1. Locating and starting the library method
2. Collecting the background (no sample on the diamond ATR interface)
3. Placing a small aliquot of sample onto the diamond and turning the clamp. (Note: the clamp is only needed for solid samples; it is not needed for liquid samples)
4. The live view and user input of the sample name and optional comment
5. Scanning the sample; once completed, the spectra is automatically searched against the chosen libraries
6. The ranked results appear from best to worst and top to bottom:
   - A yellow warning triangle appears in the first column if the related match is a narcotic and/or is hazardous (having metadata selected is required to be active).
   - Abbreviated metadata is present below the library matches; click More for the full metadata, which appears in a new window.
   - Any associated GHS hazard symbols are displayed near the bottom-right of the screen.

Step 6 displays the HQI matches according to the match criteria with their associated HQI scores. New to the MicroLab search table are the ranking order number and the warning triangle that appears only for controlled substance matches that are present in the TICTAC FTIR ATR drugs library (i.e., actual narcotics). Also new is an abbreviated metadata section, which contains some important abbreviated information for a highlighted hit. These changes are shown in better detail in Figure 3. Clicking Details in Figure 3 yields the spectral match of the library sample with the scanned sample, together with the abbreviated HQI table and metadata. These new features are optional and were designed to visually highlight controlled substances, displaying their associated GHS symbols.

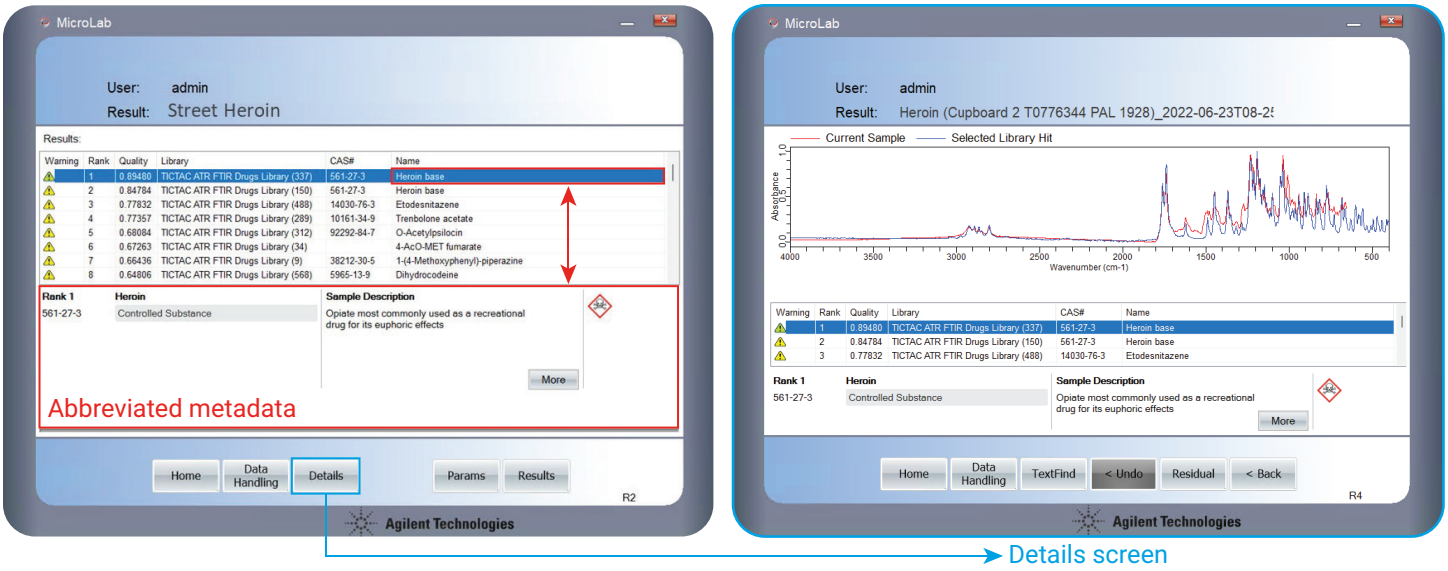

Figure 3. Left: ranked library search results screen showing the identified compound (heroin base) ranked 1, and highlighted in red is the new abbreviated metadata section. Right: Details screen of the library search showing the spectra, warning triangle, rank, and abbreviated metadata, including the associated GHS hazard symbol.

The more complete metadata is available via a new screen (Figure 4) showing comprehensive metadata fields plus a Custom Information section, and is accessed by clicking More (shown in Figure 3). The Custom Information section may be used for geographic classification differences or even to simply add handling comments or notes for the end user; it can be updated in the library management tools. The 4500a and 5500a FTIR forensics analyzers have been extensively tested for both conventional narcotics, NPS, and a wide variety of cutting agents.

The following is a brief description of each section highlighted in Figure 4:

A. Identification: contains the main identification attributes

B. Data: contains chemical and physical information

C. Compound name: first aid, firefighting, accidental release, and other information

D. Custom Information: users can add their own text to any of the fields using library management

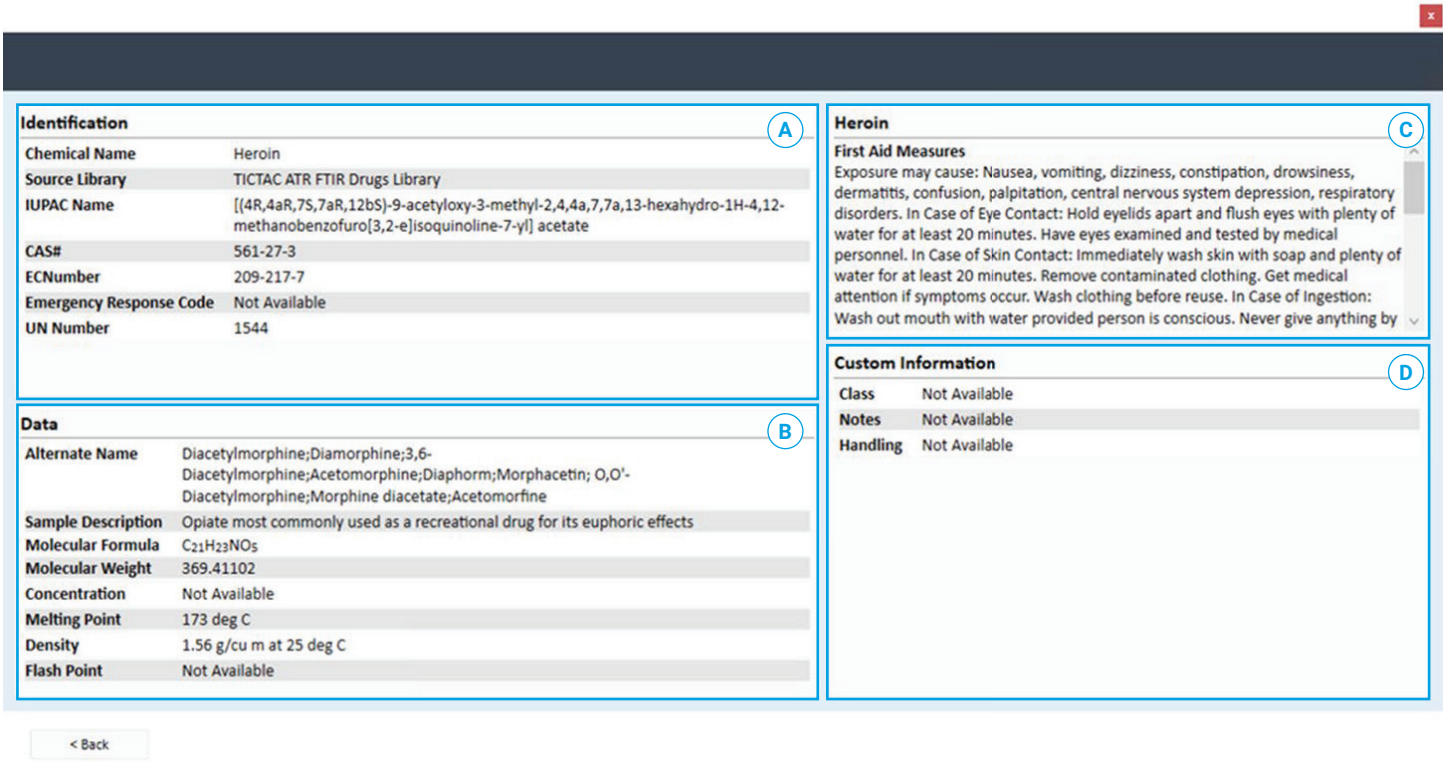

Figure 4. Metadata pop-out window containing key information for heroin, where the four main sections are shown. (A) Identification, (B) Data, (C) first aid/safety/firefighting measures, and (D) Custom Information. Clicking Back (bottom-left tab) takes the user back to either the ranked results screen or the Details screen.

Several supplementary menus are available for the user in MicroLab 5.7. The menus are available at the bottom of the screen shown in Figure 3. These menus are Home, which takes the user to the home screen, and Data Handling, which takes the user to a screen that enables (A) report generation, (B) addition of spectra to a library, and (C) exportation of spectra and reanalysis of the spectra. Often a user may want to visually check the match; this is achieved by clicking Details, shown in Figure 3. Some additional functions are available on the Details screen in Figure 3. Clicking TextFind enables the user to override the match with a chosen substance–often used to disprove a sample claim (for example, if a sample claims to be vitamin C or sugar but matches a narcotic). Clicking Undo takes the user back to the ranked HQI page. Clicking Residual subtracts the highlighted match and performs a secondary search with the residual spectral; this feature is useful for mixtures.

In Figure 5, a selection of street drugs of abuse, both a conventional and so-called new psychoactive substance, as well as a common nonnarcotic household powder, are shown, along with a cropped screenshot of their Details screen in MicroLab. All have been correctly identified with minimal user input, and the Agilent libraries contain multiple analogs of NPS and multiple mixtures of the common conventional narcotics, as well as many cutting agents, household powders, and a wide variety of chemicals. The narcotics and related samples in Figures 5A to 5F were all street-confiscated samples. No warning triangle or GHS hazard symbol appears for the sample in Figure 5F as it is a nonhazardous cutting agent.

Figure 5. Examples of street drugs and a common cutting agent. The street drug sample spectra are in red and the matching spectra with highest HQI are shown in blue for (A) cocaine hydrochloride, (B) heroin base, (C) 2C-B hydrochloride, (D) MDMA crystal, (E) N-ethylpentylone (NEP), and (F) citric acid–a common cutting agent.

Spectral selectivity of 4500a/5500a with the NPS AKB-48 and two analogs

The prevalence of NPS in recent decades has spawned the terms "legal highs" or "designer drugs", and is a huge cause for concern with new additions to the list year on year.[1] Often these drugs are chemical analogs of pre-existing drugs or compounds that are historically known to induce a biochemical effect but were not pursued. Synthetic cannabinoids, a specific class of NPS, are by far the largest-reported group. The potential harm of these synthetic cannabinoids is often underestimated due to its association with herbal cannabis, even though an NPS is often much more potent and longer acting than the drug it is attempting to replicate. Synthetic cannabinoid receptor agonists (SCRAs) cover the widest chemical structural range, as well as a variety of analogs. The TICTAC ATR FTIR drugs library contains SCRAs of many chemical classes and their analogs. To illustrate the selectivity of the 4500a or 5500a in identifying the exact structure and specific analog, Figure 6 illustrates the spectral differences between AKB-48 (APINACA), 5C-AKB-48 (5C-APINACA), and 5F-AKB-48 (5F-APINACA). All three are SCRAs, where the fluorine halogenated analog is much more potent than the base form[2], and were first recorded in South Korea in 2012.[3] In Figure 6, the three compounds share the same base structure with same number of atoms, with only a different atom at the end of the pentyl tail. For AKB-48 (Figure 6A) the highlighted group is a methyl group and its main contributions have been highlighted (2,960 and 1,450 cm^{-1}). For 5F-AKB-48 (Figure 6B), the C to F stretching vibration at ~1,000 cm^{-1} is highlighted. This compound is roughly 50 times the potency of the base AKB-48, which itself is roughly equipotent with Δ9 THC. The final analog shown in Figure 6C is the chlorinated form, which is rarer with no current metabolic data, although at the bare minimum it will likely be at least as active as the parent compound (Figure 6A). The main spectral influences of each tail grouping are separately highlighted in their respective colors per compound.

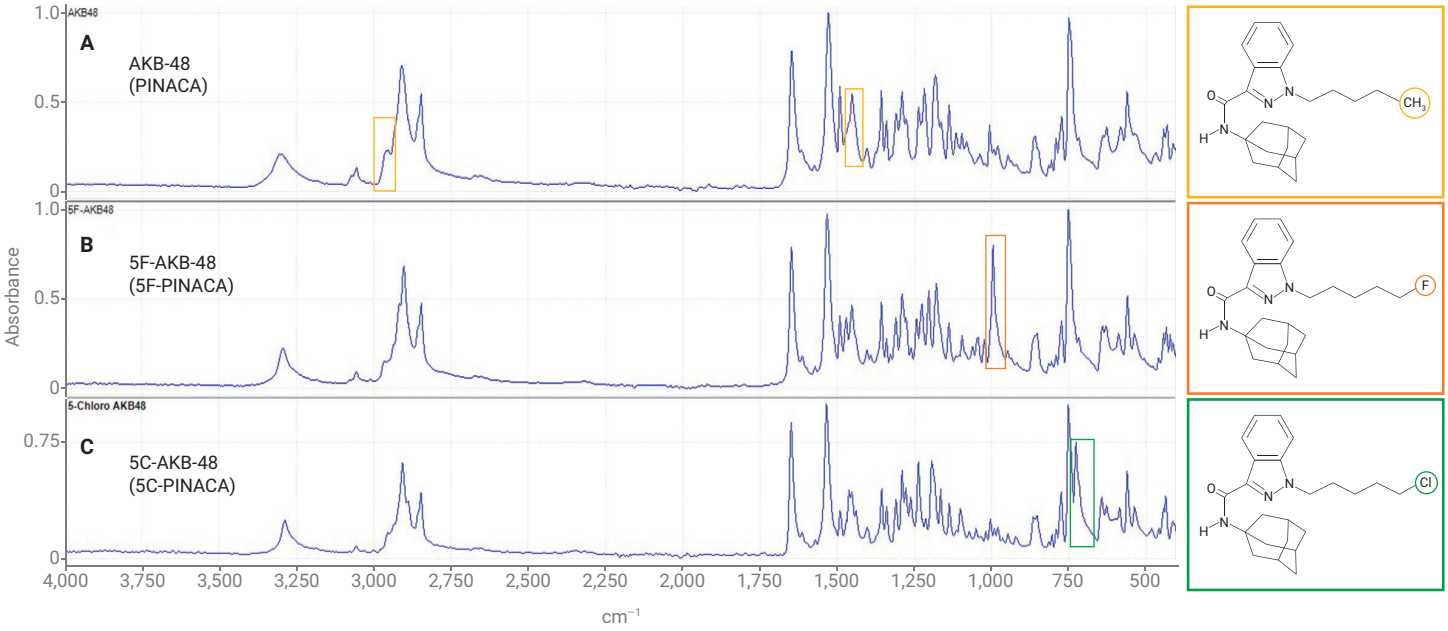

Figure 6. The Agilent 4500a FTIR forensics analyzer ATR spectra of three AKB-48 synthetic cannabinoid analogs and their main differences highlighted. (A) Base form of AKB-48: methyl hydrogens on the tail, (B) fluorinated form of AKB-48, and (C) chlorinated form of AKB-48.

Conclusion

Personnel involved in the detection and identification of narcotics and their related precursors or analogs, as well the analysis of suspicious chemicals, can now do so at the scene with high spectral data quality. They can use either the portable, battery-powered Agilent 4500a FTIR forensics analyzer, or the transportable Agilent 5500a FTIR forensics analyzer where there is mains available or a portable power pack. Both are powerful, accurate, and accomplished instruments designed for actionable spectral results, requiring minimal user engagement, with to easy-to-use software and highly tailorable operation. In this application note, examples of conventional narcotics, NPS, and cutting agents, as well as AKB-48 and analogs, have been used to demonstrate the excellent performances of the 4500a and 5500a in terms of spectral selectivity and identification of a wide variety of compounds. The optional activation of metadata, as well as the warning field and triangle with associated GHS symbol, empowers the user to exercise extra caution to suit the material examined and warns of controlled substances. The user is then alerted and can take the appropriate action, when usually many of the rarer controlled substances would not be recognizable as a narcotic simply from the library name.

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RA44770.4252314815

Printed in the USA, September 13, 2022
5994-5258EN

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