Application Note

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Identification of Narcotics and Cutting Agents in Mixtures with the Agilent FTIR Forensics Analyzer Package

Identifying primary and residual compounds in Agilent MicroLab PC software

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Abstract

Street drugs are often diluted or cut by a dealer before bagging and selling, primarily to increase profit, with the level and type of cutting agent varying widely. In this application note, various common street narcotics and their cutting agents were correctly identified using the Agilent 4500a and 5500a FTIR forensics analyzers. When dilution is mild, the primary identified compound matches the narcotic, and the residual identified compound matches the cutting agent. Alternatively, when the cutting agent has been added at higher quantities, the primary identified compound matches the cutting agent, and the residual identified compound matches the narcotic. Both scenarios are demonstrated with the application of the residual analysis function. Also demonstrated is the detection of a ternary mixture.

Introduction

Fourier transform infrared (FTIR) spectroscopy is a proven, powerful technology for the analysis of illegal substances and potentially hazardous compounds. The ability to rapidly measure the molecular fingerprint of a substance and successfully match it to libraries of known compounds depends on the selectivity and sensitivity of the technique. Few spectroscopic techniques have the sensitivity and ease-of-use to be practical; FTIR is one such instrumental technique, where the identity of a suspicious powder or liquid can be determined in less than 1 minute, commonly with little or no sample preparation required. Drugs that are pure will yield hit quality index (HQI) values near 1 (where 1 = 100% algorithmic match; often this is called HQI% or match%) compared to the reference compounds. The reference compounds within the TICTAC ATR FTIR drugs library are all high-purity compounds that are synthesized, purified, and batch tested with secondary confirmation. The identification and library match of high-purity narcotics is a simple task, as when the HQI value is high there can be high confidence in results. However, street drugs are often diluted ("cut") by the supplier or the dealer, either deliberately to make more profit, or inadvertently from poor chemical process controls. Deliberately diluted compounds are commonly called cutting agents. Poor chemical manufacturing practices may lead to the presence of impurities in the base drug, where the impurity is usually a related compound present in the plant source, i.e., another active compound in the poppy or coca plant. Some common cutting agents are listed in Figure 1.

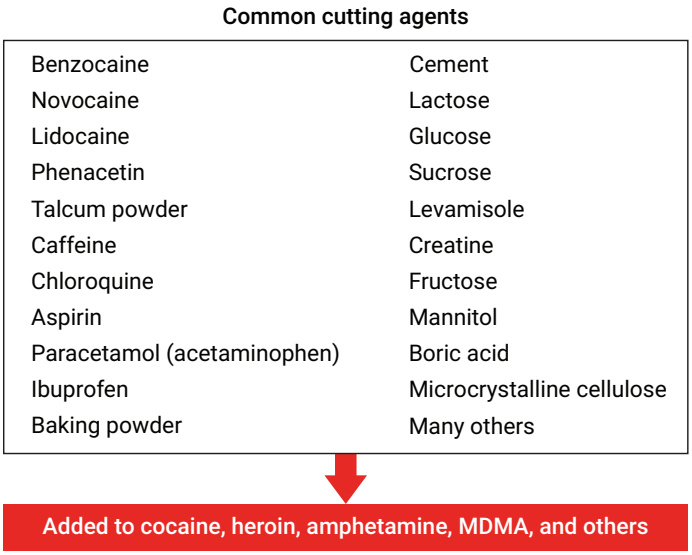


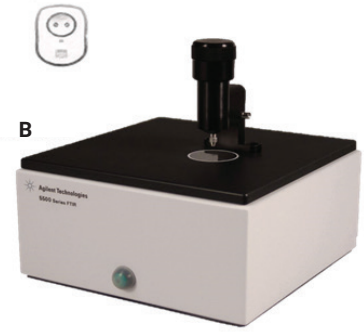
Figure 1. Some common cutting agents and the drugs they are incorporated into.

Cutting agents are usually white or near-white powders that may be chemically active or inactive. The ratio of cutting agent to narcotic can vary widely depending on the dealer and supplier. Figure 1 is a small example of the variety of common cutting agents, ranging from relatively benign substances to active pharmaceuticals and substances. This application note demonstrates the identification of the primary and residual compounds in some moderately to heavily cut narcotic samples. When the cutting agent is a minor component of the sample, the primary identified compound matches the narcotic. Conversely, when the cutting agent is the major component, the residual identified compound matches the narcotic and the primary identified compound matches the cutting agent. The use of FTIR can rapidly aid the triage of suspect samples and determine the presence or absence of narcotics and cutting agents. It is not uncommon to find pure cutting agent or a combination of cutting agents masquerading as a narcotic. Whether the narcotic is a free-form powder or tablet, there is a high possibility that the material has been deliberately diluted to some degree. The residual analysis function in Agilent MicroLab PC software takes the primary identified compound or the user-selected match and subtracts it from the library hit and then performs a secondary search with the residual spectra. The function can be performed more than once, with each iteration containing less spectral information for spectral matching.

Experimental

Both a portable 4500a and a transportable 5500a FTIR forensics analyzer equipped with a single-bounce diamond were used with the ATR Forensics Library for Mobile FTIR and the TICTAC FTIR ATR drugs library. The combination of the 4500a and 5500a FTIR forensics analyzers and the libraries enables in-field and onsite analysis of narcotics and related substances. Both instruments are equally comfortable in the lab or field. The 5500a can be powered by mains power or external power supply, and the 4500a has its own internal battery. The hardware and software package comes supplied with the versatile Agilent MicroLab software suite. Each sample is scanned in approximately 20 seconds, requiring only a few milligrams of sample. In this study, the instrumental resolution selected was 4 cm^{-1} with 50 scans and a zero-fill factor of 2. Spectral collection duration was approximately 22 seconds, and sample turnaround time including (a) cleaning the crystal in readiness for the next sample, (b) sample information input, and (c) data review was approximately 2 minutes. Both instruments are shown in Figure 2.

Agilent 4500a FTIR forensics analyzer



Agilent 5500a FTIR forensics analyzer

Figure 2. (A) The Agilent 4500a FTIR forensics analyzer (fully portable mains/battery/external power bank). (B) The Agilent 5500a FTIR forensics analyzer (transportable external power bank/mains power supply). Both have detachable clamping mechanisms.

Results and discussion

A selection of commonly cut street samples were analyzed by both the 4500a and 5500a FTIR forensics analyzers. The approach was to treat the samples as unknown to ascertain the ability of the residual analysis function to detect moderately to highly diluted narcotics. Examples spanning commonly cut narcotics and different degrees of dilution with cutting agents are detailed in Table 1. The residual analysis can be used even when the narcotic is the primary compound, aiding identification of potential interferents for hyphenated chromatographic techniques such as GC/MS or LC/MS. Moreover, even when the cutting agent is the primary match, the table demonstrates the ability of the residual spectral data function to identify the narcotic in the residual match. Cocaine, heroin, and amphetamine are often sold in the street bagged or in small cling film wraps as the free-flowing powder, with FTIR analysis requiring only a fraction of a gram. The analysis is examined nondestructively, so the sample can be recovered if necessary. 3,4-Methylenedioxymethamphetamine (MDMA), however, is common in both powder and tablet forms. Table 1 is a summary of the primary and the residual identified compounds and their HQIs in a range of seized street samples, all of which were cut to varying extents. Generally, when the primary identified compound HQI% exceeds 95% there is minimal residual data left for residual analysis (as shown with the pure crystalline MDMA in Figures 8A and 8B). The residual analysis function in Agilent MicroLab PC software is accessed by clicking **Details** on the library search ranked table screen (Figure 3B), followed by clicking **Residual** on the Details screen (Figure 3C).

Table 1. Overview of residual analysis results for a range of narcotics with different degrees of deliberate dilution. Agilent MicroLab PC software library search using similarity; no exclusion regions applied. HQI% = HQI × 100. Bold type is used to indicate the narcotic in the primary and residual identified compound columns.

Figure	Street Sample	Primary Identified Compound HQI%	Residual Identified Compound HQI%
3	Cocaine A	**Cocaine base 80.96%**	Phenacetin 90.58%
4	Cocaine B	**Cocaine HCl 87.46%**	Sodium bicarbonate 88.31%
5	Heroin A	Paracetamol 80.03%	**Heroin 80.72%**
6	Heroin B	**Heroin:**caffeine:paracetamol (**1**:1:1, **w/w) 87.43%**	
7	Amphetamine	Lactose BP 84.36	**Amphetamine sulfate 77.66%**
8A, 8B	MDMA (pure)	**MDMA 98.04%**	NA
8A, 8C, 8D	MDMA (cut)	Lactose BP 72.78%	**MDMA 86.47%**

NA = not applicable

Figure 3. Agilent MicroLab PC software view for a library search of a street sample of cocaine (sample A). Displayed are: (A) cocaine sample A FTIR ATR spectrum; (B) ranked library search results for the primary identified compound–cocaine base (HQI% = 80.96%); (C) Details screen for the primary identified compound; and (D) ranked results of the residual analysis, showing phenacetin as the residual identified compound (HQI% = 90.58%).

Street cocaine samples cut with two different cutting agents

Two examples of street cocaine (samples A and B) that have been cut with an unknown amount of phenacetin and baking powder respectively are shown in Figures 3 and 4. The base form of cocaine can be distinguished from the hydrochloride acid salt form of cocaine (cocaine HCl). The ability to identify the cutting agent may be useful for a forensic department to allocate the most fitting extraction regime if a secondary hyphenated chromatographic analysis technique is necessary. Since phenacetin was withdrawn from use in the 1970s, the results suggest that cocaine A was deliberately cut by the supplier/manufacturer. The residual analysis of street cocaine B indicated the presence of sodium bicarbonate cutting agent, suggesting a dealer-level cutting of the cocaine HCl. When the addition of cutting agent to a narcotic increases, at some point the primary identified compound may match the cutting agent rather than the narcotic itself. Since the primary match for street cocaine samples A and B are cocaine base and cocaine HCl respectively, the use of the residual here is to identify the cutting agent, where the type of cutting agent may interfere with chromatographic techniques. The high selectivity of FTIR ATR has distinguished between the base and hydrochloride salt forms of cocaine, even though both contain cutting agents.

Figure 4. Agilent MicroLab PC software view for a library search of a street sample of cocaine (sample B). Displayed are: (A) cocaine sample B FTIR ATR spectrum; (B) ranked library search results for the primary identified compound–cocaine HCl (HQI% = 87.46%); (C) Details screen for the primary identified compound; and (D) ranked results of the residual analysis, indicating that sodium bicarbonate is the cutting agent (HQI% = 88.31%).

Street heroin–moderate to high cutting agent content

Frequently the cutting agent dominates the drug sample and this is particularly true for street heroin, which, according to the UNODC, contains on average only 10 to 20% heroin[1], also known as diacetylmorphine. Figures 5 and 6 show two such street heroin samples (A and B). Street heroin sample A has been mainly cut with paracetamol, whereas street heroin sample B has been cut with both paracetamol and caffeine. The residual analysis of heroin sample A demonstrates that heroin is the residual identified compound, whereas for heroin sample B, residual analysis is not necessary as the primary identified compound is a ternary mixture of paracetamol, caffeine, and heroin, at a 1:1:1 weight ratio (see Figure 6C). For conventional narcotics, some common cutting agent combinations are present in the ATR Forensics Library for Mobile FTIR. Residual analysis by selecting paracetamol as the primary identified compound using the TextFind function yields heroin, as does selecting caffeine (not shown).

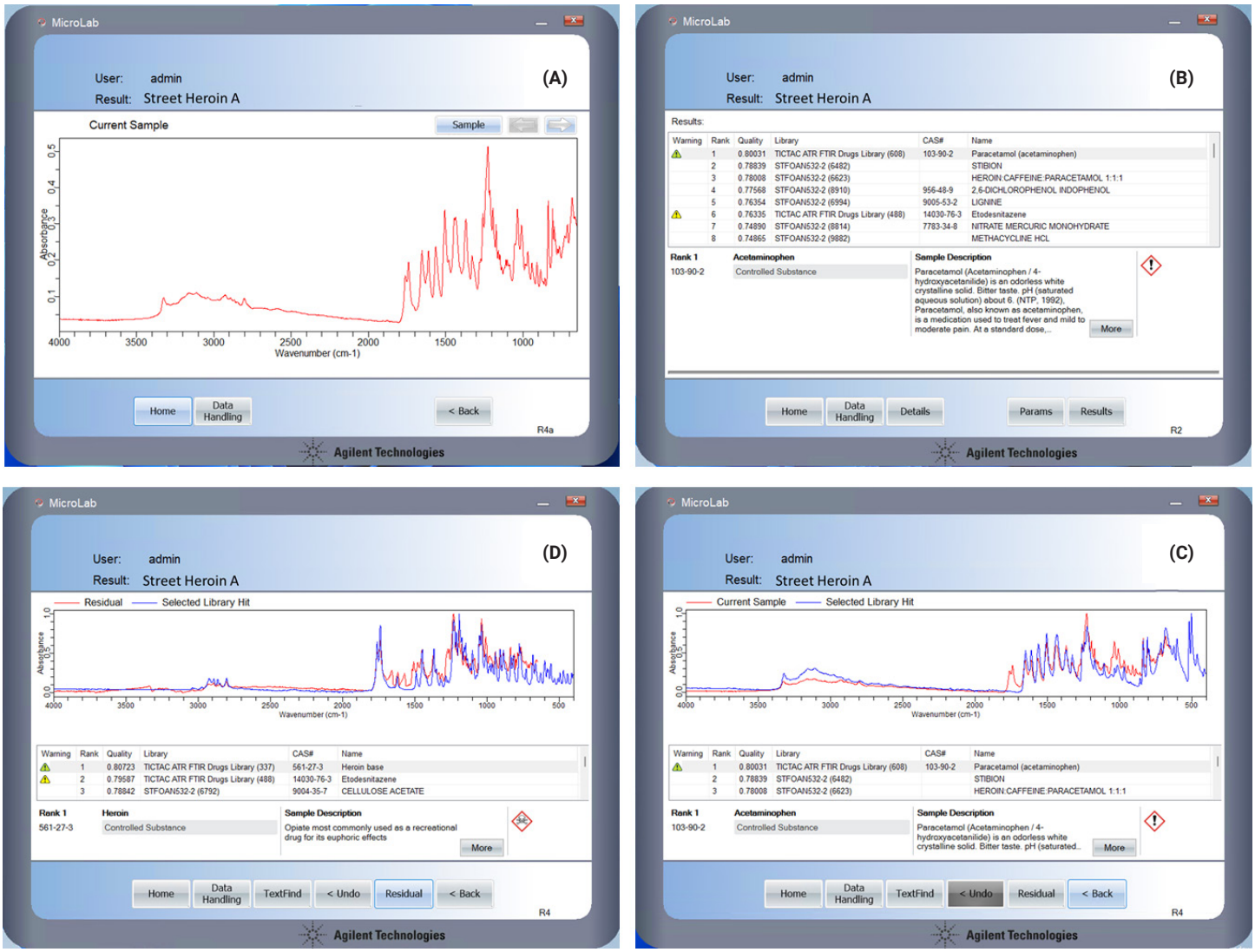


Figure 5. Agilent MicroLab PC software view for a library search of a street sample of heroin (sample A) cut with paracetamol. Displayed are: (A) heroin sample A FTIR ATR spectrum; (B) ranked library search results for the primary identified compound–paracetamol (HQI% = 80.03%); (C) Details screen for the primary identified compound; and (D) residual analysis results, showing heroin base as the residual identified compound (HQI% = 80.72%).

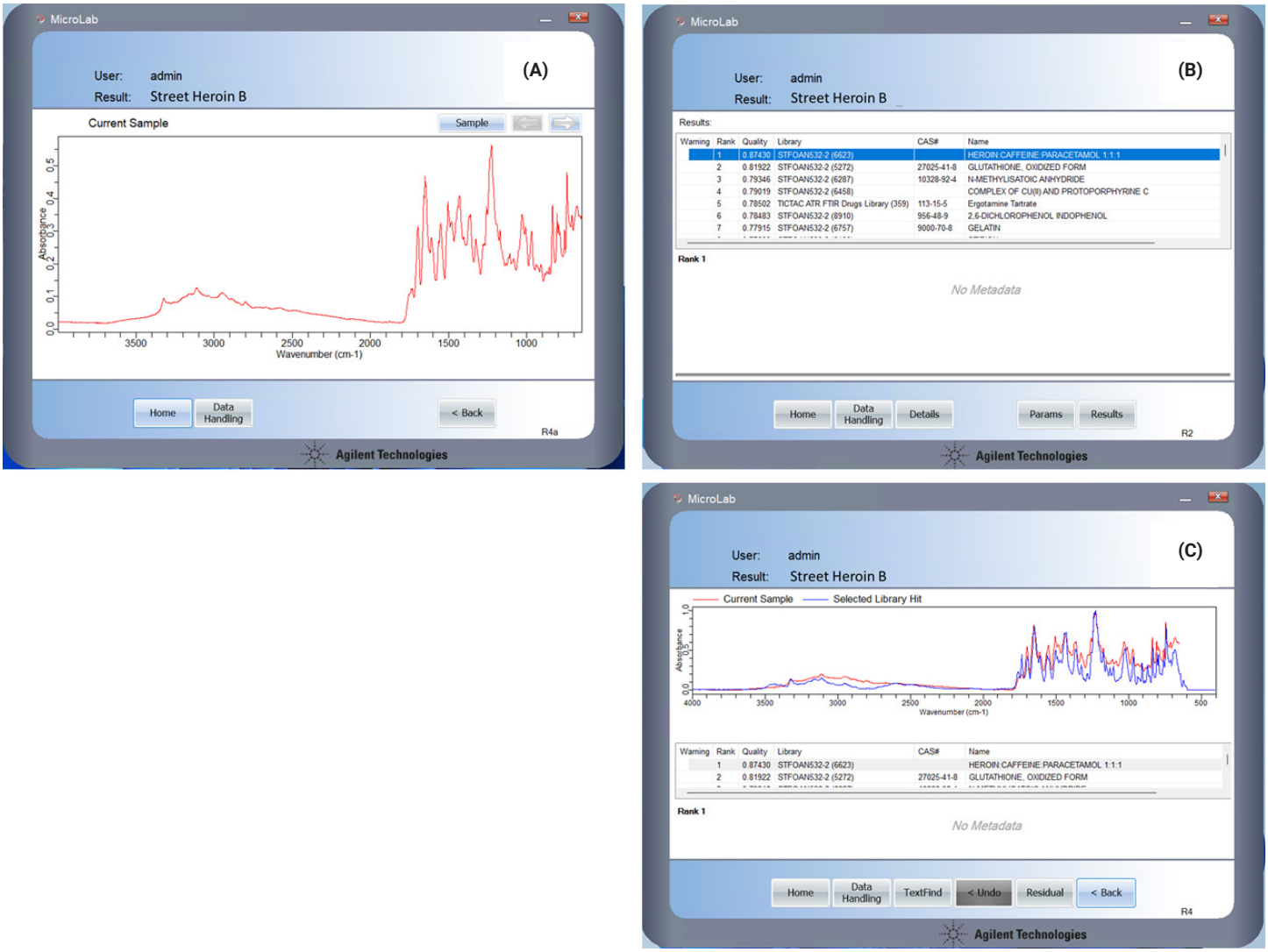


Figure 6. Agilent MicroLab PC software view for a library search of a street sample of heroin (sample B), where the primary identified compound is a 1:1:1 weight ratio of heroin, caffeine, and paracetamol. Displayed are (A) heroin sample B FTIR ATR spectrum; (B) ranked library search results; and (C) Details screen displaying the visual spectral match. **Note:** residual analysis is not required in this case with the ternary mixture directly identified.

Street amphetamine–highly diluted

Another commonly diluted narcotic is amphetamine, commonly referred to as speed or whizz. The UNODC estimates the amphetamine content of street samples to be typically around 10%, but it can be as low as 1% and as high 70%.[1] When the amphetamine street sample was measured (Figure 7B and 7C), the primary identified compound matched the cutting agent lactose, with the amphetamine sulfate identified as a good residual match in terms of the HQI value and as a good visual spectral match (Figure 7D).

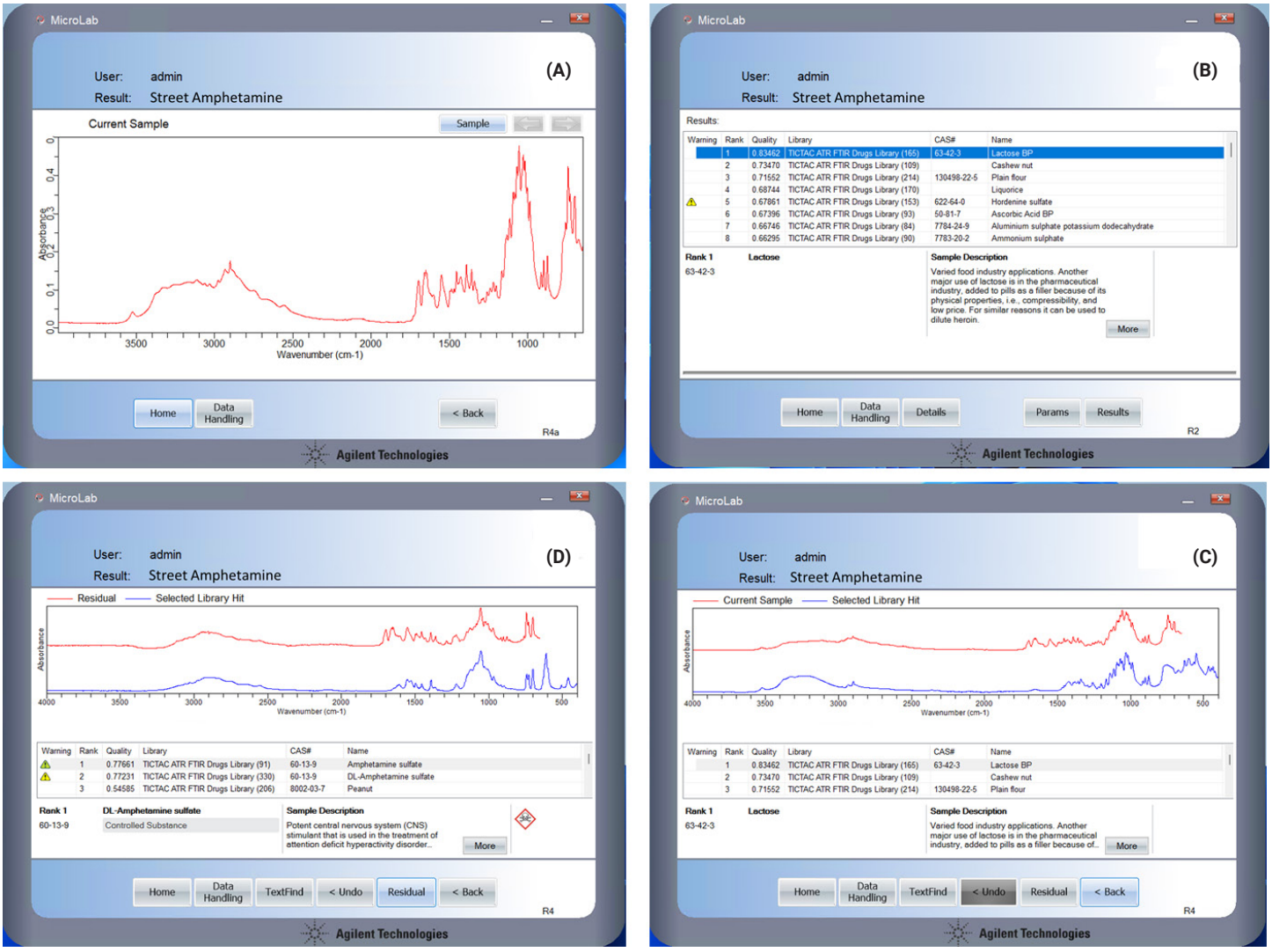


Figure 7. Agilent MicroLab PC software view for a library search of a street sample of amphetamine. Displayed are: (A) street amphetamine FTIR ATR spectrum; (B) ranked library search results for the primary identified compound–lactose BP (HQI% = 83.36%); (C) Details screen for the primary identified compound; and (D) the ranked details of the residual analysis, showing amphetamine sulfate as the residual identified compound (HQI% = 77.66%).

High-purity crystalline MDMA compared to cut and diluted MDMA

MDMA is commonly known as ecstasy, E, or molly. Molly is colloquially the street name for the free powder or free crystalline form, whereas ecstasy and E usually refer to the tablet form, where the shape, design, and color are akin to branding. In either the powder or tablet, the MDMA narcotic is either in base or crystalline salt form, and the purity can vary wildly, with added health risks inherent with dose and effect for the unwary. In Figure 8, two street samples of MDMA (samples A and B) were analyzed. Sample A is near to a perfect match to the reference spectrum, whereas sample B is highly cut with lactose and the MDMA is found in the residual analysis. The relationship between the MDMA content and the library match algorithm is empirically related to the HQI, whether the primary match or a residual match is required. If a moderate level of cutting agent is introduced to the powder or tablet, the primary identified compound will match the narcotic with a reduced library HQI value, as seen in Figures 3 and 4. As the level increases, the spectral features of the cutting agent become more prominent (Figures 5 to 8), using the residual analysis function. When a street sample is highly chemically pure, the match to the illegal drug will be close to 1 HQI (100% for the HQI%), as shown in Figure 8A (red line) and 8B for MDMA sample A.

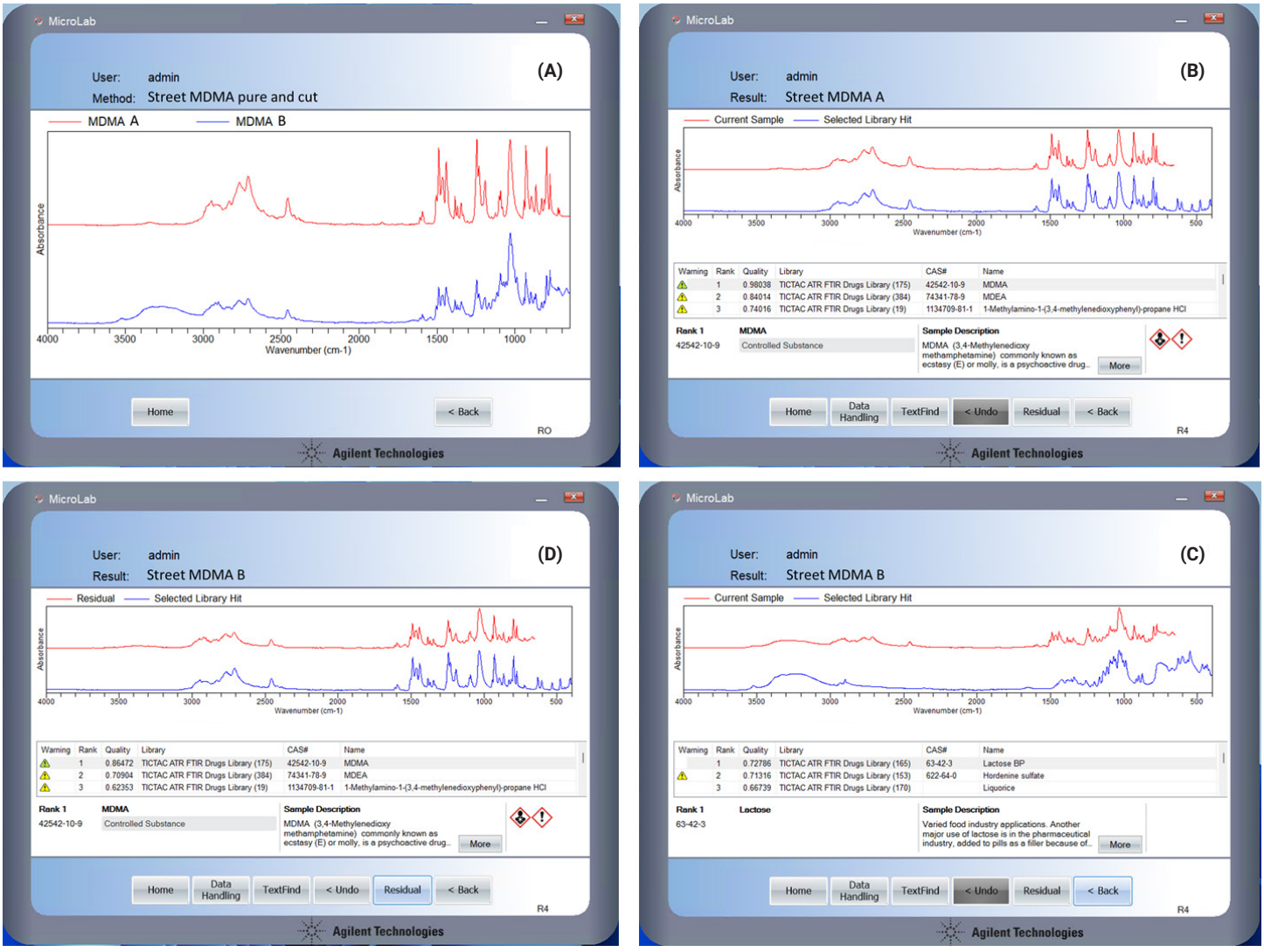


Figure 8. Agilent MicroLab PC software view for a library search of pure and cut samples of MDMA. Displayed are: (A) high-purity MDMA (sample A) FTIR ATR spectrum in red, and cut MDMA (sample B) FTIR ATR spectrum in blue; (B) Details screen for the primary identified compound in the MDMA sample A–MDMA base (HQI% = 98.04%); (C) primary identified compound in the MDMA sample B–lactose BP (HQI% = 72.79%); and (D) residual identified compound in the MDMA sample B–MDMA (HQI 86.47%).

Conclusion

The Agilent 4500a and 5500a FTIR forensics analyzers both show a high degree of selectivity and sensitivity, where the primary (main) component and residual component can both be reliably identified. We have demonstrated that, where the cutting agent or contaminant is present at low to moderate levels, the primary identified compound matches the illegal narcotic with two street cocaine samples. The residual in this case could be attributed to the cutting agent or contaminant and was also correctly identified.

We have shown that as the relative quantity of cutting agent increases, the narcotic becomes the secondary (residual) identified compound and the primary identified compound is the cutting agent. Examples of match results for extensive dilution were also presented for heroin, amphetamine, and MDMA. All the street samples had been secondarily confirmed with a gold standard methodology. The approach was to treat the samples as unknown to ascertain the ability of the residual function to identify narcotics in cut samples where the primary identified compound was no longer the main active narcotic.

The use of the residual function can be incorporated into any standard operating procedure or concept of operations guide. The 4500a FTIR forensics analyzer requires minimal user expertise, and the spectral detail contains high-density data that enables positive identification of narcotics, even if it is the second or third component in the mixture. Finally, the instrumentally easier task of identification of pure substances is demonstrated with a high-purity narcotic using the example of MDMA, which matches with a >98% HQI% to the reference lab-created MDMA. Fake drugs that are pure cutting agents have also been detected by the 4500a and 5500a. Often at a drugs bust, many pure and mixed substances are present and identifiable and the rapid triage and categorisation enables the streamlining of evidentiary processes.

Reference

1. United Nations, Office on Drugs and Crime. *Prices and Purities of Drugs*. United Nations. https://www.unodc.org/documents/data-and-analysis/WDR2021/8.1_Prices_an_purities_of_Drugs.pdf

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