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Screening Controlled Substances Using the Near-infrared Fourier Transform Raman Technique

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KEYWORDS

FT-Raman/forensics/illicit drugs, controlled substances, screening techniques

INTRODUCTION

The FT-Raman technique is very sensitive to the presence of a wide range of controlled substances and has several unique attributes which are invaluable when screening seized evidence for the presence of these compounds. The method allows materials to be quickly analyzed without removing them from the sealed evidence bags in which they are collected eliminating cross contamination or sample contact. An unknown 'sealed' compound can be analyzed in less than 30 seconds by referencing a library of known standard samples. Nicolet has developed a library of over 200 compounds for this analysis.

The correct identification and analysis of controlled substances samples is essential to law enforcement departments worldwide. Typical on-site screening methods rely on wet-chemical field test kits. These test kits have significant drawbacks since they are frequently unstable and cease to give reliable results after a period of time. In addition, the field testing kits are not simple to use and can easily be cross contaminated.

The most common alternative to the field test methods is full laboratory analysis using a gas chromatograph coupled with a mass spectrometer (GC/MS).[1] While GC/MS is a thorough and widely accepted method, it does have some limitations. Samples require preparation – a potential risk for contamination of the sample and increased risk for the analyst. Samples are usually dissolved in a hexane/ethanol mixture and a typical analysis takes approximately 30 minutes, including preparation and standard running. Reliable results depend on a high level of user expertise, and while spectra generally are compound specific, there are key compounds which produce very similar GC/MS data. Finally, while small quantities of material can be analyzed, the sample is not recovered after analysis and therefore cannot be re-used if confirmational testing is required.

FT-IR has also been used to analyze drug samples.[2,3] A relatively inexpensive analysis technique, FT-IR has similar problems associated with sample handling as GC/MS. Preparation of samples for FT-IR analysis typically require dilution of the sample with KBr prior to analysis.

When evidence is collected in drug related cases, positive identification of illicit materials can often lead to out of court settlement, saving time and money. In these cases, only a lack of a suitable screening technique necessitates full and time consuming analysis by GC-MS. There is a clear need for a technique which is capable of quickly and simply identifying controlled substances without the need for direct contact with the evidence.

In a recent series of measurements made in a collaboration between Nicolet's Spectroscopy Research Group and scientists at the Mesa Police Department, Forensics Laboratory, Mesa, AZ, FT-Raman spectroscopy was demonstrated as a viable screening technique. FT-Raman – in conjunction with newly developed library searching routines - has several benefits that relate directly to the needs discussed above:

- FT-Raman quickly and easily identifies unknown samples by measuring the vibrational spectrum and comparing the results with a database of known standards. The analysis often takes less than 30 seconds, including all sample preparation time.
- FT-Raman is simple and easy to use. The software and hardware can be customized for simple push button or touch screen operation displaying the sample's ID.
- FT-Raman eliminates user contact with the sample. In tests, FT-Raman successfully identified controlled substances, even when they were presented in sealed plastic evidence bags. The presence of the bag does not significantly effect the spectral results of the analysis.
- FT-Raman is non-destructive. This is essential in a screening technique since the same sample of evidence can later be used for the full laboratory analysis if needed.
- Identification by FT-Raman is largely insensitive to user technique, sample size and granularity and the sample container (evidence bag). This makes the FT-Raman system much more powerful, reliable and easier to implement than other potential vibrational spectroscopy techniques such as FT-IR and Near IR absorption.

SAMPLE HANDLING

For this development work, 200 high quality spectra were measured from the Mesa Forensics Laboratory's set of standard samples containing controlled substances, related isomers and prescription drugs. Fourier transform techniques allow the use of near-infrared excitation at 1064 nm rather than the shorter wavelengths of laser light common in older dispersive Raman instrumentation.

Using this relatively long wavelength, almost all samples can now be analyzed by FT-Raman without the problems of fluorescence[4] associated with conventional dispersive Raman instruments. In our study, excellent quality spectra were measured from all 200 samples. These spectra were used to create a spectral database, or library, against which unknown samples can be searched and identified. Running unknowns can be a very simple process for the user, since parameter optimization, analysis of spectra and library searching can be automated to run by simply pushing an "Analyze" button.

Field evidence is normally collected and returned to the lab in sealed evidence bags. These bags are made from a heavy gauge, transparent plastic so that samples are visible, protected and not at risk of contamination. Example FT-Raman spectra of some common controlled substances are shown in Figure 1. FT-Raman analysis can be carried out simply by placing the evidence bag in the sample compartment such that the "unknown" sits in the instrument's field of view. The measurement takes only a couple of seconds, a full spectral library search happens almost instantaneously and results can be either displayed on the computer screen or printed as a standard report. Sampling, is that simple.

Sampling is simple for a couple of reasons. First, the system does not need constant alignment and is insensitive to sample position. This means that the sample bag may be placed in the instrument without complicated, time consuming alignment. Second, the plastic evidence bag has a much weaker spectrum than the materials inside the bag; therefore the drug spectrum is measured with minimal interference from the container. Figure 2 shows the FT-Raman spectra of an empty evidence bag, powdered methamphetamine and a sample of methamphetamine measured inside a sample bag. As can be seen, spectra b) and c) appear identical and the sample bag is shown to have little effect on the measured data.

RESULTS

A library of 200 controlled drug compounds, related isomers and some common pharmaceutical has been successfully constructed to analyze unknown evidence samples which are suspected of containing controlled substances.

Figure 3 shows a typical FT-Raman spectrum measured from a seized street sample during an arrest. The sample is measured in the sealed polymer evidence bag in which it was transported to the lab and the entire identification process took less than 10 seconds. This unknown sample was identified by measuring the spectrum and searching this data against the library database.

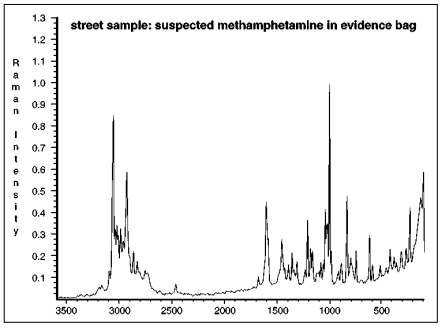

Figure 3: Seized street sample measured in the evidence bag

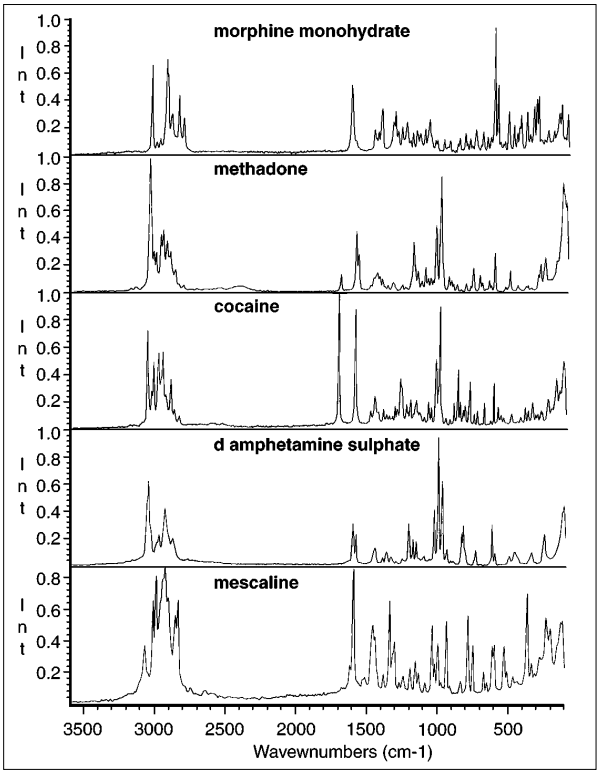

Figure 1: FT-Raman spectra of common controlled drug compounds

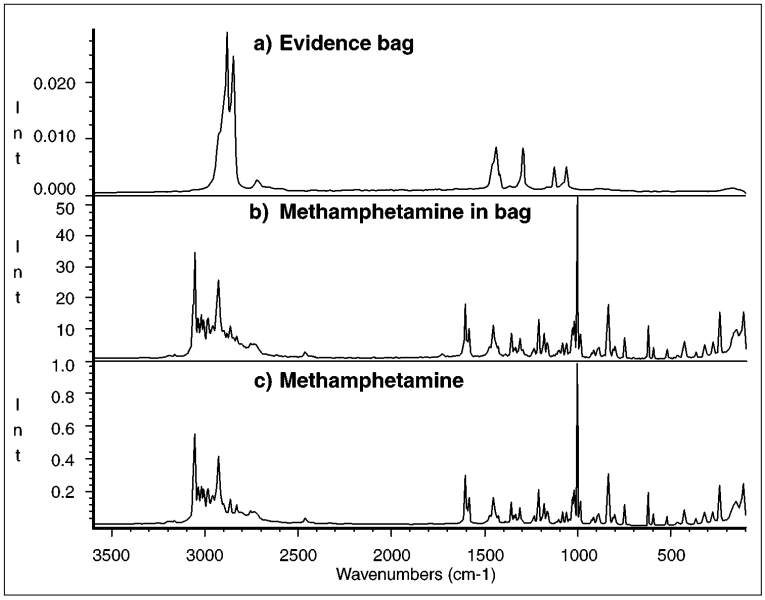

Figure 2: FT-Raman spectra of a) empty evidence bag, b) methamphetamine measured in the evidence bag, and c) methamphetamine

Collection details are 8 cm^{-1} resolution, 4 seconds collection time, autosave of results and correlation search algorithm. Results are displayed either as a spectral comparison with the best matches from the library (Figure 4a) or as a match number which is used to determine how close the match is to the library spectrum[5]. The match numbers for this unknown are given in Figure 4b. While all of the top matches from the library are similar compounds, the first selection is a significantly better match than the others. Minor differences which can be seen between the unknown spectrum and the methamphetamine library standard are attributed to the evidence bag and to the impurities and additives in the street sample. These minor changes have a minimal effect on the ability of the search routine to correctly identify the unknown.

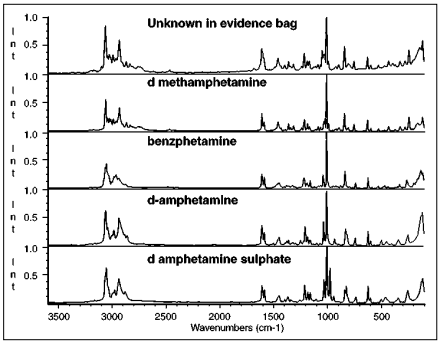

Figure 4a: Top selections from a library search of the unknown

Index	Match	Compound Name	Library
90	82.62	methamphetamine	FT-Raman Forensic Library
18	65.39	benzphetamine	FT-Raman Forensic Library
15	64.02	d-amphetamine	FT-Raman Forensic Library
14	63.29	d-amphetamine sulfate	FT-Raman Forensic Library
10	62.21	aspartame	FT-Raman Forensic Library
102	61.89	alpha methyl fentanyl	FT-Raman Forensic Library

Figure 4b: Match numbers from the library search of an unknown in evidence bag, Total analysis time 30 seconds

Since FT-Raman is a non-destructive technique, the same sample can be analyzed again using the most intensive GC/MS laboratory analysis. Example data for this analysis is shown in Figure 5a – the GC/MS spectrum of methamphetamine. For illustration, Figure 5b shows the GC/MS spectrum of phentermine. GC/MS spectral similarities between methamphetamine and phentermine make them difficult to distinguish. The same compounds are shown in Figure 6 measured by FT-Raman. These spectra are easily distinguishable.

For the increasing sample load of many forensic laboratories, FT-Raman provides quick and easy sample screening. The more complex GC/MS need only be used when a more indepth analysis is required.

CONCLUSION

Near-infrared FT-Raman is an ideal screening tool for the analysis of illegal street drugs. Advantages include:

- Fast analysis – usually less than 30 seconds
- No sample handling – data can be collected without opening the evidence bag
- Non destructive analysis – the same sample can be used for confirmational tests
- Easy operation with no instrument alignment
- Efficient, accurate library searching

In addition, FT-Raman easily differentiates between pairs of similar compounds such as cocaine and pseudo cocaine, or methamphetamine and phentermine which are traditionally difficult with other techniques.

ACKNOWLEDGMENTS

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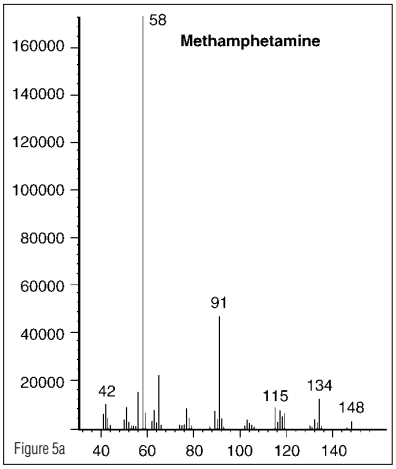
REFERENCES

1. "Forensic Mass Spectrometry, 2nd Edn.", J. Yinon, CRC Press
2. "Forensic Science Handbook, Vol.II", R. Saferstein, Prentice Hall (1988)
3. R.J. Warren, P.P. Begosh, J.E. Zarembo, *Journal of the AOAC*, 54(5), 1179 (1971)
4. Hirschfeld and Chase, *Appl. Spec*
5. Nicolet technical note TN-9695, "Improving Search Results Using High Resolution Libraries"



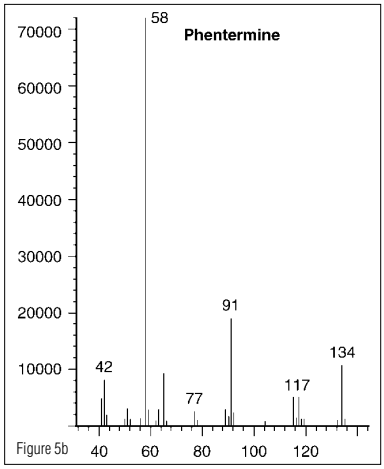


Figure 5: GC/MS data measured from a) methamphetamine and b) phentermine

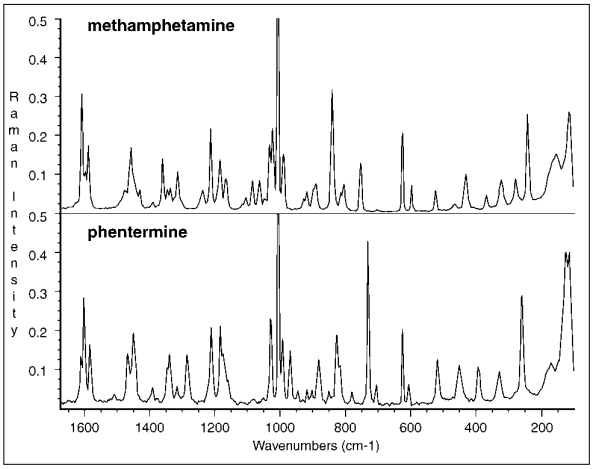


Figure 6: FT-Raman spectra of a) methamphetamine and b) phentermine

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