

Application News

No. FTIR-2003

Spectrophotometric Analysis

Illicit Drugs and Homemade Explosives Identification using FTIR Microscopy

■ Introduction

Infrared microscopy, also referred to as FTIR microscopy, is ideal for identification of trace evidence in forensics and characterizing small particles in wide varieties of other fields. Combining the accurate material and chemical identification of FTIR spectroscopy with the ability to focus directly on micro-sized particles, makes this an indispensable technique. Use of this technique for comparative identification of paint and fibers has been well documented^[1,2]. In addition to these conventional uses, FTIR microscopy allows separation of materials of interest spatially as well as spectroscopically. By visualizing small differences in shape, color and or texture under the microscope, different materials of interest can be measured separately, even if they exist in a bulk mixture. Spatially separating and measuring mixtures of powdered materials is especially useful in the areas of both drug enforcement and explosive analysis.

■ Instrumentation

The SurveyIR compact microscope from CziTek Redwave Technologies coupled with Shimadzu's compact IRSpirit FTIR spectrophotometer provide a field-deplorable system for high-sensitive measurements. In addition, a portable system has the added advantages of reducing the chain of custody and possibly reducing the sample backlog in the main lab. The SurveyIR, as seen in Figure 1, attaches to Shimadzu IRSpirit spectrophotometer using the sample compartment interface. The microscope is also compatible with all existing Shimadzu FTIR spectrophotometers.



Figure 1: SurveyIR Accessory with IRSpirit FTIR

This system, through an advanced optical design, utilizes the room temperature deuterated triglycine sulfate (DTGS) detector found in the Shimadzu FTIR spectrophotometers. DTGS detectors don't require additional cooling and have a larger spectral range compared to mercury cadmium telluride (MCT) detectors, making them easy to use and allowing more details in the IR spectrum of sampled materials.

SurveyIR Microscope Accessory Advantages

- Fits any Shimadzu FTIR model
- No alignment
- No maintenance
- Simple user controls
- View-through, clip-on diamond ATR
- Transmission/Reflection/Oblique illumination modes
- Transmission/Reflection/ATR IR collection modes
- Large, 1900 μm field of view
- 5 MP Digital camera with 2X optical magnification resulting in 0.7 $\mu\text{m}/\text{pixel}$ at the sample plane
- Affordable, compact design

■ Drug Analysis

FTIR spectroscopy allows for fast and efficient identification of most illicit drug substances. Most seized drugs are comprised of the illegal drug and cutting agents. The cutting agents both hinder and help investigators to make identification of the illegal substance more difficult, especially if they mask the infrared signature of the drug. On the positive side, many police departments have found success in tracing the distribution chain of drugs through the combination of cutting agents found in seizures. This can help lead investigators back to the larger distributors, providing more and bigger convictions. In the end, it's important to identify both the drug and the cutting agent used.

As an example, a sample of white powder seized from a crime scene was identified using the microscope accessory. A small portion of the sample was placed onto an infrared reflective (low-e) microscope slide and rolled flat. Rolling the sample helps to separate the particles and flattens them, allowing the user to isolate and measure specific sample particles for infrared spectra that are easily searched against commercially available libraries, which can easily be added to the LabSolutions IR software. An image of the sample collected with transmitted light illumination on the SurveyIR is shown in Figure 2.

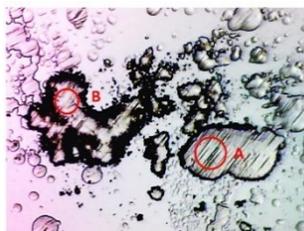


Figure 1. Sample image with sampling locations A and B.

Two different sample morphologies can be seen in the image. The spot on the right of the slide has an oblong shape and thinner edge (1). The spot on the left of the slide is irregular with a thicker edge (2). The system was able to measure each of these spots individually by using an aperture to focus only on the specific sample. The locations of the measurements are shown with the red circles, labeled A and B. The infrared spectrum measured from sample spot A was searched against a commercially available library of infrared spectra; the results are shown in Figure 3.

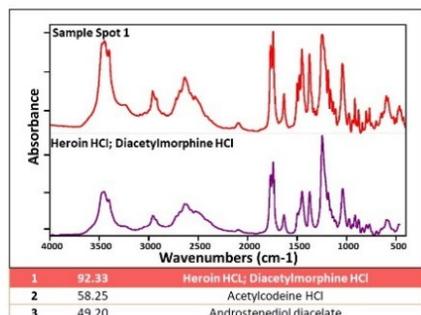


Figure 2: Library search results of sample spot A indicating diacetylmorphine HCl.

The top search result, with a high correlation, was found to be diacetylmorphine HCl, known as heroin. The infrared spectrum measured from sample spot B was also searched against a commercially available library; the results are shown in Figure 4.

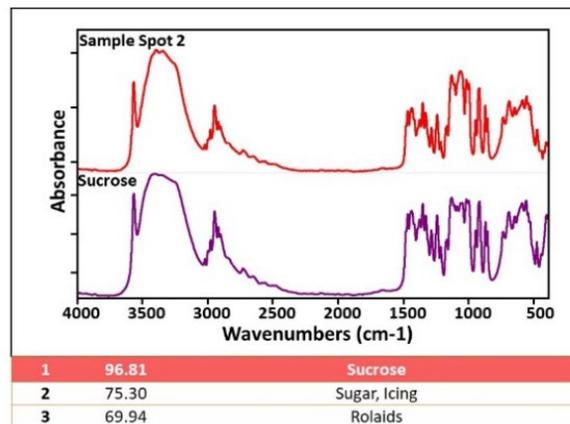


Figure 3: Library search results of sample spot B indicating sucrose.

Here, the spectrum library search results indicate sucrose, a common cutting agent, with a very high correlation value. With these results, the illegal substance has been identified despite the presence of a large amount of cutting agent. Additionally, the identification of the cutting agent gives investigators more evidence to pursue the original source. The simple sample preparation and measurement procedure took less than 30 minutes to complete and provides simple, reliable results.

■ Explosives

Identification of explosives is another area where quick accurate analysis is essential. One type of improvised explosive can be made by mixing ammonium nitrate with diesel fuel. When suspect material is found, it's important to understand if it's just fertilizer or an improvised explosive. Like the example above, separation of the mixture spatially on a microscope allows for accurate identification of most components. The following mixture of small white pellets, which appeared to be wet was found in a 55-gallon drum. The mixture was first analyzed on a handheld system and resulted in an ID of ammonium nitrate. The liquid component remained absent from initial results and the mixture was further subjected to microanalysis using the FTIR microscopy system.

The mixture was flattened using a diamond compression cell, which have the advantage of pressing the sample into a thin pathlength as well as being transparent to IR for easy transmission analysis. Viewed under the SurveyIR, both particulate and liquid portions of the sample can be observed as shown in Figure 5. Transmission spectra were measured using the IRSpirit of both components at the locations noted as 3 and 4. Infrared spectra measured of the two spots are shown in Figure 6.



Figure 4. Solid and liquid portions of the sample.

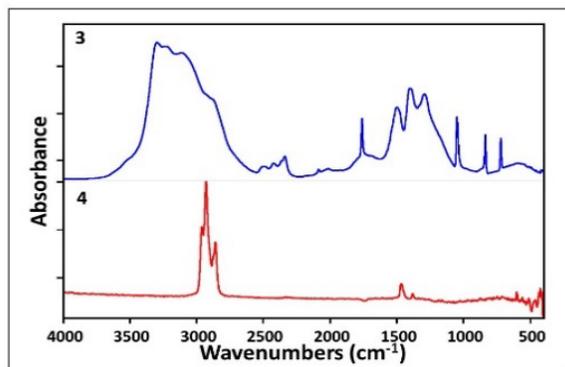


Figure 5: FTIR spectra of solid (blue) and liquid (red)

Spot 3 (Figure 6,3) had a high correlation to a commercial library spectrum of ammonium nitrate. Spot 4 (Figure 6,4) is consistent with diesel fuel or another long chain hydrocarbon. Adding diesel fuel to ammonia nitrate fertilizer makes a powerful explosive and is a well-known technique amongst those intent on causing harm. Identification of this mixture quickly and accurately was key to averting a crisis.

■ Conclusion

Material identification is key to many types of forensic analysis. In combining the identification capabilities of FTIR spectroscopy with the spatial resolution of microscopy, the IRSpirit FTIR spectrophotometer coupled with SurveyIR microscope accessory provides accurate identification of both trace samples and a wide variety of solid mixtures. In both drug and explosive analysis, the system has been shown to identify components of the mixture without resorting to complicated data analysis techniques. Furthermore, the low cost and ease of use of the system allows it to be used by any lab, or even in the field. Fast, accurate analysis is obtainable for any forensic lab.

■ Acknowledgement

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■ References

1. Czitek application note, "Micro-Spectroscopic Interrogation of Automobile Paints"
2. Czitek application note, "Evaluation of Micro-Fibers Utilizing Microspectroscopy"



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