This is to certify that the
thesis entitled

The Development of an FT-IR Spectral Library of Tape
Adhesives

presented by

Kaleo Conley Kaluhiokalani

has been accepted towards fulfillment
of the requirements for the

Master of Science degree in Criminal Justice with a Specialization in Forensic Science

Jay Siegel

Major Professor's Signature

6/7/04

Date

THE DEVELOPMENT OF AN FT-IR SPECTRAL LIBRARY OF TAPE ADHESIVES

By

Kaleo Conley Kaluhiokalani

A THESIS

Submitted to
Michigan State University
in partial fulfillment of the requirements
for the degree of

MASTER OF SCIENCE

School of Criminal Justice

2004

ABSTRACT

THE DEVELOPMENT OF AN FT-IR SPECTRAL LIBRARY
OF TAPE ADHESIVES

By

Kaleo Conley Kaluhiokalani

The adhesives in tape possess unique chemical characteristics that can be distinguished by Fourier Transform Infrared (FT-IR) spectrophotometry. In this study, the FT-IR spectra of fifty adhesive tapes were collected and compiled into a spectral library. It was determined that a diamond anvil cell was the most effective sampling accessory for tape adhesives because it produced high quality spectra with minimal sample preparation. A test of the spectral library showed that individual tape adhesive samples could be classified despite visual similarities in many of the spectral patterns.

To my wife Sina, my son Talanoa, and my parents, Norman and LaVerne.
Thank you for teaching me the importance of education.

ACKNOWLEDGEMENTS

I would like to thank my professor Dr. Jay Siegel for being a great teacher, mentor, and advisor of forensic science. It is an honor and a privilege to have studied forensic science under the guidance and leadership of one of the leading scholars in the field of forensic science. I would also like to thank Dr. Melvin Yokoyama and his wife Ana for providing support throughout my career at Michigan State University. I would also like to acknowledge Jenny Smith, from the Missouri State Highway Patrol Crime Laboratory, Ed Bender, from the Bureau of Alcohol Tobacco and Firearms, John "JJ" Johnston, pro bono Technical Consultant for the Pressure Sensitive Adhesive Tape Council of the USA and pioneer in the tape industry, and Claire Chun and Tracy Tanaka of the Honolulu Police Department Trace Evidence Unit. Each of these people provided valuable information, insight, enthusiasm, and encouragement throughout this project.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

Introduction

The identification of physical evidence can provide valuable information in the search for a suspect, or provide a link between evidence found in possession of a suspect and evidence recovered from a crime scene. The idea of associating criminal to crime scene through physical evidence interpretation derives from the Locard Exchange Principle. According to this principle, whenever a criminal comes into contact with an object or a person at a crime scene, a transfer of evidence occurs. When a crime is committed, the perpetrator deposits telltale information or clues unique to himself onto objects he encounters at the scene, while at the same time information from objects at the crime scene are transferred to the perpetrator. By identifying the source of physical evidence recovered from the crime scene as originating from the suspect, or evidence found in possession of the suspect as originating from the crime scene, investigators can demonstrate that a cross transfer of evidence has taken place. If such an exchange can be proven in court, the suspect can almost certainly be connected to the scene of the crime.

In order to identify and characterize physical evidence, investigators often rely on the skill and knowledge of experts such as forensic scientists. Forensic scientists employ generally accepted scientific methods and techniques when making comparisons and identifications. They make detailed comparisons of the many features of the evidence by studying the physical and chemical characteristics of each item. Based on the results of these tests, forensic scientists are able to determine whether or not the evidence at the scene could have originated from the suspect or vice versa.

Adhesive tape is an example of a type of physical evidence that forensic analysts are sometimes asked to examine. Tape is used by criminals to bind and gag victims, to package and conceal illegal items like drugs or money (1), it is found on questioned documents (2), and used in the construction of improvised explosive devices (3). To some, the evidentiary value of tape may not seem to be very powerful because it is commonly encountered. However, this can be misleading. When analyzed properly in a well-developed classification scheme, tape can provide ample information to link a suspect to a crime scene (4).

Tape is defined as a pressure sensitive adhesive. A pressure sensitive adhesive is an adhesive applied to a backing (5). The analysis of tape differs from the analysis of conventional adhesives because tape consists of two distinct components, the backing and the adhesive. These two components are very different in construction and composition and can provide forensic analysts with two excellent sources of discriminatory information that can be used in the differentiation of tape samples.

The backing portion of the tape is its most recognizable feature. Tape manufacturers design tape to allow the backing to perform a specific task. The more specific the task, the more specialized the backing material becomes. As a result, backings are constructed from a wide variety of materials. Several examples of backing materials are cellophane for an invisible look, fabric for strength and ease in tearing, and PVC for insulating bare electrical wires. Each type of backing material bears distinct physical and chemical properties that can be examined when making tape comparisons.

In addition to the backing, the adhesive part of the tape can also provide meaningful information when differentiating tape samples. The adhesive part of the tape is not as specific in its design like the backing. Often manufacturers design the adhesive simply to ensure that the tape backing stays affixed to whatever surface it is attached to. Despite being less specific in its the design, the chemical components that make up the adhesive portion of tape are much more complex, and as a result exhibit more variability than the materials found in the backing (6) (7).

Like conventional adhesives, tape adhesives are complex mixtures that contain a variety of natural and synthetic materials in their fillers, binders, and tackifiers. The complexities of the tape adhesive formulas contribute greatly to the variability of the tape. The more variable a product is, the more significant it becomes as evidence.

Another factor that contributes to the variability of tape is that the adhesive formulas are constantly changing. Tape manufacturers frequently change the components in the adhesives to cut production costs. Tape manufacturers can profit by replacing an expensive ingredient with one that is less expensive yet comparable in performance. Also, primary vendors, suppliers, and distributors of major adhesive ingredients change as well. These factors complicate the adhesive production process and in turn increase the variability of the tape.

When analyzing tape, forensic analysts use a number of established analytical methods and procedures. Pyrolysis gas chromatography, gas chromatography/mass spectrometry, gel permeation, and x-ray diffraction, are just a few methods used to characterize the various properties of tape adhesives.

One method often used to characterize tape adhesives is infrared (IR) spectrophotometry (8). With IR spectrophotometry, an infrared spectrum is produced by the absorption of certain frequencies of radiation by the molecules in a sample. IR spectrophotometry is useful in identifying the organic materials in the sample. No two substances that absorb IR radiation do so at the same frequencies. As a result, each substance produces a unique IR spectrum that that can be used to differentiate one substance from another.

Most IR spectrophotometers are interfaced to a computer that is capable of storing hundreds of digitized spectra in a database or spectral library. A spectral library is a searchable compilation of spectra collected under carefully controlled, optimized conditions (9). Through a search algorithm, the spectrum of an unknown substance can quickly be compared against hundreds of spectra contained in the library (10). The results of a spectral library search can often lead to the classification or even the identification of an unknown substance.

Spectral libraries are widely used by forensic analysts to assist in the identification of certain unknown substances. Specialized spectral libraries are already in use for the characterization and identification of substances such as illicit drugs, paints, fibers, and accelerants. Presently, there are no spectral libraries available commercially for the characterization and identification of adhesive and tape products, and yet there is a need for such a system (3).

Suppose an unknown adhesive residue is recovered from a crime scene and it is uncertain whether the adhesive residue is a superglue, epoxy, rubber cement, or adhesive tape residue. A comprehensive spectral library would be most useful in cases like this. The spectral library could be used to classify the type of adhesive residue recovered. Moreover, if the residue happened to be a tape adhesive, a spectral library created specifically for tape adhesives could be searched to further classify the tape adhesive into a subclass such as office tape, electrical tape, packaging tape, etc.

A spectral library of tape adhesives can provide an efficient system for classifying unknown adhesive substances. Forensic analysts who encounter tape evidence can use the results from this reference collection as part of an overall classification scheme to better characterize unknown adhesives. This project is designed to develop a method for building an FT-IR spectral library of tape adhesives. It is part of an ongoing project to build a comprehensive adhesive database.

Adhesive History

Adhesives have been used for thousands of years. The earliest recorded use of adhesives was discovered in a 3300 year old carving from Thebes. The carving depicts workers gluing a piece of veneer to a plank of Sycamore wood. Ancient Egyptians used natural substances such as gum Arabic from the acacia tree, egg, and plant resins to make glues. The ancient Romans used substances like beeswax and pine tar to seal up the hulls of their sailing vessels. For thousands of years it was understood that certain substances were useful for holding materials together by surface attachment or adhesion (11). It is this ability to attach things together that has made adhesives such a versatile product in today's society.

Up until the early part of the twentieth century most of the available adhesive products came from natural sources. Natural adhesives are primarily composed of animal or vegetable matter. Adhesives of animal origin are derived from animal offal such as hide, bone, blood, fish byproducts, milk byproducts, and insect exudates (shellac). Adhesives made from vegetable products are derived from sources such as starch, dextrin, rosin, and natural rubber.

Despite their abundance and ease of use, natural adhesives lack the moisture resistance and strength properties that would make them useful at the industrial level. For instance, casein glues that were used during World War I to construct the wooden mainframes of aircraft suffered from limited resistance to moisture and were extremely susceptible to mold growth. Deficiencies such as these limited the use of natural adhesives to light construction and were not reliable for jobs that required more stress resistance.

By the early 1920's adhesive manufacturers began experimenting with new adhesive components such as synthetic materials, in the attempt to improve the quality and strength properties of their products. During this expansive period, many new adhesive products were developed. Some of these products were so innovative that several are still being manufactured today (12).

Masking tape is one of the products developed during that time period. Masking tape is the first tape designed for industrial use. Prior to the development of masking tape, tapes were generally used only in the medical profession. 3M researcher Richard Drew is credited with inventing masking tape. It was developed to assist automobile painters who were seeking better solutions to paint cars in two tones. The painters needed a good adhesive product that could "mask" the topcoat of a newly painted car so that a different color could be painted over it, producing a two-toned look with a clean distinct demarcation between the two colored layers. In addition, the tape needed to contain a light amount of tack so that it could be removed easily without damaging the topcoat layer of paint to which it was affixed. The tape also had to provide enough water resistance in its backing to keep the second color layer from seeping through to the first. It took Drew nearly ten years of work to find the right type of adhesive compound and backing material to satisfy the needs of the painters (13).

With the success of masking tape, consumers began to realize other potential uses for tape. The increased popularity of masking tape prompted 3M to begin development of other specialized pressure sensitive tapes such as decorator's tape, electrical tape, and filament tape for more specific tasks (13).

One of the most popular tapes that resulted from this tape revolution was cellophane tape. By the late 1920's masking tape was becoming a popular product. However it still suffered from several storage problems, one of which was that the adhesive on the masking tape roll would become sticky and ooze from the roll at temperatures above room temperature. In trying to resolve this problem it was proposed that a newly-developed, colorless packaging paper called cellophane be used to package the masking tape roll. Cellophane was light, colorless, and moisture resistant, properties that would be attractive to consumers and also prevent the adhesive from leaking out of the packaging. While working with cellophane as a solution to his packaging problems, the innovative Drew realized that cellophane would make an excellent backing material for a new tape. After about a year Drew finalized the development of what he called "Scotch brand cellophane tape".

Scotch tape was put on the market in 1930, which also happened to be the first year of the depression, which for many other products would have been disastrous. Despite the inopportune timing for the introduction of this item, sales of cellophane tape did surprisingly well. The American consumer, who had been forced to become more thrift conscious during the depression, found that the colorless cellophane tape was extremely useful for repairing numerous items. Consumers reportedly used the tape to mend items such as pages of books, window curtains, and sheet music. Farmers used it for patching cracked turkey eggs, and women used it for repairing torn fingernails. The uses for this tape were innumerable. Consumers enjoyed its ease of use, invisible look, and its sturdiness. Scotch brand cellophane tape quickly became the most widely used packaging item replacing string, staples, and rubber bands (13).

Today tape remains one of the most versatile and widely used adhesive products. Tape can be found in nearly every store that sells general goods. There are a great variety of tapes. Tapes are found in different sizes, widths, colors, and are designed for different purposes. The volume, variety, and complexity of tape make it an excellent product for scientific characterization.

Classification of Adhesives

Adhesives are generally classified into two broad categories, natural and synthetic. Natural adhesives refer to adhesives that derive from animal or vegetable sources, while synthetic adhesives refer to all other adhesives.

Natural adhesives contain plant or animal materials as their main component. These include glues and pastes made from casein, blood, albumin, hide, bone, fish byproducts, starch, resin, shellac, etc. Natural adhesives do not bond as well as synthetic adhesives, and as a result, are used in simple applications that do not require too much stress on the glues. Natural adhesives are generally used for bonding light materials such as paper, paperboard, foil, and lightwoods.

Synthetic adhesives refer to all other adhesives not composed of natural substances. These include adhesives that contain man-made polymers and other manufactured additives. Synthetic adhesives are further subdivided into the following four categories based on individual chemical properties; thermoplastic adhesives, thermosetting adhesives, elastomerics, and alloys.

Thermoplastic adhesives are generally composed of polyvinyl acetate, polyvinyl alcohol, and some acrylics. Thermoplastic adhesives characteristically do not cross link upon curing. Thermoplastic adhesives are applied by heating or by way of a solvent. This property allows these adhesives to be re-softened if necessary. Examples of thermoplastic adhesives include polyvinyl acetate, polyvinyl alcohol, and some acrylics.

Thermosetting adhesives, on the other hand, cure opposite of thermoplastic adhesives. Thermosetting adhesives cross link upon curing and cannot be reheated or re-softened after the initial cure. Thermosetting adhesives are densely cross-linked and cure at either room temperature or elevated temperatures. These products come as a one or two part system. Thermosetting adhesives include epoxies, cyanoacrylates, and polyesters.

Elastomeric adhesives are characteristically tough and possess elongation, strength, and flexibility properties. Elastomerics include silicones, neoprene and styrene-butadiene rubbers.

Alloys are adhesives that are made from a combination of any of the three adhesive classes mentioned previously.

Although the classification system mentioned above is followed by most researchers who study adhesives, there is no consensus on a universal system for adhesive classification. Other proposed adhesive classification systems use characteristics such as chemical composition, function, physical form, or means of application, as criteria for classifying adhesives. All the systems mentioned are acceptable methods for classifying adhesives, but without a standardized classification system, a single adhesive can be classified in more than one way under different systems.

Tapes are classified as pressure sensitive adhesives, which is a subcategory of elastomerics or alloys depending on the composition of its adhesive (14). A pressure sensitive adhesive is any adhesive applied to some backing (5). Examples of pressure sensitive adhesive products include tapes, labels, and miscellaneous products like the Post-It Notes. Pressure sensitive adhesives characteristically adhere firmly to a variety of dissimilar surfaces without the need for more than finger or hand pressure. They require no activation by water, solvent, or heat in order to exert a strong adhesive holding force (14) (15).

Besides being classified as pressure sensitive adhesives, tapes can also be classified according to their construction or by their use. Tapes that are classified by construction are differentiated almost exclusively by the materials that make up the backing. Examples would be fabric tapes, paper tapes, film tapes, PVC, etc. Tapes that are classified according to their use are differentiated by the specialized function the tape was designed to perform. Examples include office tapes, packaging tapes, electrical tape, etc (8).

Manufacture and Design

All varieties of tape are generally produced in the same process. Tape production occurs in two general phases, coating and slitting. Coating refers to the application of the adhesive onto the backing or carrier material, and slitting refers to the process of cutting the tape into small manageable sized rolls.

The first step in the coating process is preparing the adhesive. The bulk adhesive used on the tape is first thinned down and made into a less viscous liquid. Manufacturers use one of three methods to achieve an appropriate consistency. The methods used are solvent coating, water-based coating, or hot melt coating.

The solvent coating and water based coating processes are similar. Both use a liquid to thin down the bulk adhesive. For solvent coating, a solvent is used to thin down the bulk adhesive, while with water-based coating, water is used. Once the newly mixed adhesive has achieved the proper consistency it is coated onto the carrier and then passed through a heated oven where the solvent or water is evaporated off, leaving the final product.

The hot melt coating process is different because no solvents are added to thin down the adhesive. In the hot melt coating process, heat is used to melt the solid adhesive into the desired consistency. Once the adhesive has reached the appropriate viscosity, it is then coated onto the carrier or backing. The coated tape is then cooled to give the finished product.

To prevent the tape from sticking to itself and to reduce unwind tension, the backing or carrier of the tape is treated with a release coating often made of silicon (14).

Without a release coating many tapes would be impossible to use. The release coating makes the tape easier to unwind and also makes the slitting process much more efficient.

At the completion of the coating process the newly coated tape is contained on a large "jumbo" roll and prepared for slitting. During this process the "jumbo" roll of tape is cut into smaller, more manageable rolls of varying widths and lengths. The "jumbo" roll is unwound onto a machine that contains a number of razor blades. The distance between the razor blades determines the width of the final product. The backing passes over these blades cutting the tape into narrower widths and these are then wound onto cores to give the appropriate length for the finished rolls.

Majority of the tapes on the market today are produced in the general manner explained previously. Despite the similarities in the manufacturing process, it is the variety of materials used in its construction that give each tape product its unique characteristics.

Manufacturers construct tape backings from hundreds of different materials. Materials such as paper, cloth, cellulose based films, PVC, and metal foils are commonly used in tape backing. Backings can be clear, colored, or printed. The properties of the backing material are carefully selected by the manufacturer to allow the tape to function in a specific capacity. For example, masking tape uses a backing made of paper that is impregnated with a moisture resistant coating. Electrical tape has a backing made of a plastic or PVC designed to insulate bare electrical wires. Filament tapes have backings containing fibrous material to strengthen them for heavy-duty packaging. Gaffers tape has a dull cloth backing designed with a low reflectance material for use in the stage and lighting occupations.

While the backing of the tape allows it to serve a specific function, the function of the adhesive is less specific in its design. Adhesives are generally designed to ensure that the backing material of the tape securely adheres to whatever surface it is placed on. The adhesive functions as the bond between the tape backing and the surface. A well-designed adhesive binds cohesively to the backing to which it is fixed, and binds adhesively to the surface or substrate to which it may be affixed. Despite being less specific in its design, the adhesive side is equally important to the success of the tape. A failure in the adhesive is a failure in the tape itself. If the adhesive does not adhere properly to the substrate, the backing will not be able to perform its specified task.

The two main types of adhesives used in pressure sensitive tapes are rubber/resins and acrylics. Rubber/resins come from natural or synthetic sources, with the rubber coming from trees, and the resin coming from the oil industry. Rubber/resins possess superior tack characteristics with good flexibility. Rubber/resins are relatively inexpensive, however they do not perform as well as acrylics. They do not have good resistance to heat, aging, or exposure to light.

Acrylics on the other hand, are composed entirely of synthetic polymers. Acrylics have excellent aging characteristics and strong tack. In addition, acrylics also have good ultraviolet stability and are resistant to hydrolysis. When high performance standards are required, manufacturers will often use acrylics. The drawback to acrylics however is cost; acrylics are relatively expensive polymers to produce (14).

The formulas of tape adhesives are relatively complex. In addition to the base adhesive components such as rubber/resins or acrylics, other additives such as fillers, color pigmentation, and oils are incorporated into the final product. Some even add antioxidants to stabilize the adhesive against heat and light degradation as well as to prevent oxidation (8).

The complex chemical makeup of the tape adhesives contribute greatly to the variability of the product (1) (6). Studies have found enough chemical variation in the adhesive to differentiate between different brands of tape, and were even successful in differentiating products within the same batch or lot (6). From a forensic science standpoint, the complexity of the adhesives adds probative value to tape as physical evidence. The more intricate the chemical formulation, the more likely it would be to discriminate between tape products.

Furthermore, the formulas of tape adhesives are continually changing. Manufacturers constantly upgrade the components in their adhesives (1). Some estimate that changes in tape construction, design, adhesive formula, and fabric possibly take place several times a year within a single manufacturing plant (6). Manufacturers view these frequent changes as necessary upgrades to improve the quality of the tape or to reduce costs. Other reasons for changing an adhesive formula are due to changes in the supplier, availability, or reliability. As a result of the constant changes, the chemical composition of a particular tape adhesive may be altered more than once over a period of time while the brand name remains the same (4).

A well-maintained spectral library of tape adhesives could be useful in monitoring the changes in a single product over time. This requires the spectral library to be upgraded periodically in order for it to be effective. If the spectral information is kept current, not only would it be useful in helping to identify the tape product, but it could potentially specify a timeframe in which a certain brand of tape was manufactured.

Proper maintenance of the spectral library would not only require the addition of new products to the tape library, but also require that tape products currently in the database be repurchased and re-sampled on a continual basis in order to keep the spectral information up to date, and also to track any changes that may occur within a single product over a period of time.

Review of Literature

Much of the early research into the characterization of adhesives was conducted using pyrolysis gas chromatography (PGC). PGC is a well-established analytical method that uses heat to decompose larger sample molecules into smaller fragments in an inert atmosphere. The sample molecules fragment by thermal decomposition in the injector portion of the PGC. The fragments are then introduced into a gas chromatograph (GC) where they are separated along the length of the column. The separated components are detected by a detector, and then represented graphically in a series of peaks called a pyrogram. Every substance analyzed by PGC has a unique pyrogram that can be used in its identification.

PGC was used to characterize various adhesives in studies conducted by W. Noble et al. 1974 (17) and Williams and Munson 1988 (18). Research conducted by W. Noble et al. 1974, successfully differentiated 165 of 179 adhesive samples by PGC. They were then able to classify each identifiable sample into one of twelve different categories. Williams and Munson 1988 (18) were successful in generating 26 unique pyrograms from 30 PVC tape samples. Both studies found PGC to be an effective method for the characterization of various adhesive samples.

Technological advances have since increased the sensitivity of the traditional pyrolysis gas chromatograph by interfacing it with a mass spectrometer (4). With pyrolysis gas chromatography-mass spectrometry (PyGC/MS) individual peaks in the pyrogram can be identified for greater specificity, whereas prior to PyGC/MS, sample characterization and identification was conducted through pattern matching.

The mass spectrometer is a highly sensitive instrument capable of identifying individual compounds as they elute from the GC column. As each compound enters the mass spectrometer, it is fragmented into ions by electrons. Molecules of individual compounds fragment in a predictable and reproducible manner. The fragmented ions are then separated by their mass to charge ratio and plotted on a graph called a mass spectrum. Each compound produces a unique mass spectral pattern that can be used in its identification.

The use of PyGC/MS in characterizing adhesive samples can be very effective. The only disadvantage however, is that pyrolysis is a destructive method. In order to properly analyze a sample by pyrolysis, it must be consumed. In the pyrolysis process, samples are almost instantly decomposed into gaseous products in the injector portion of the instrument. These samples can never be recovered or re-sampled once they have been pyrolyzed. Therefore, if the amount of sample is limited, other analytical methods should be considered.

Besides pyrolysis, infrared (IR) spectrophotometry has also proven to be useful for characterizing adhesive samples. IR spectrophotometry produces an infrared spectrum by the absorption of certain frequencies of radiation by sample molecules. One of the greatest attributes of IR spectrophotometry is its specificity (19). Any substance that absorbs IR radiation absorbs it at its own frequency. No two substances absorb IR radiation at the same frequencies. Therefore, the resulting IR absorption spectrum of a substance is unique, and is useful in differentiating one sample from another.

Even though adhesives are complex mixtures, and not pure compounds, it is still possible to generate a unique IR profile of the sample. Like the pyrograms that are produced in pyrolysis, an IR spectrum represents a total summation of all the compounds present in the sample. While IR spectrophotometry does not allow for the individual identification of peaks like mass spectrometry, the IR spectrum is still useful in characterizing the sample. The advantage of IR spectrophotometry is its ability to detect components that have been changed, added, or removed from an original formulation. Because adhesives are complex mixtures with variable components, the resulting IR spectrum of an adhesive sample can potentially be used to identify a specific product. The more complex the adhesive formula, the more specific the IR spectrum becomes.

Research conducted by R.O. Keto 1984 (20) and Thomas G. Kee 1984 (21) found IR spectrophotometry to be a useful method for characterizing black polyvinyl chloride (PVC) tape. Kee used IR spectrophotometry to characterize both the backing material and the adhesive of various tape samples. While in another study, Keto used IR spectrophotometry to differentiate six different brands of PVC tape. Both studies showed that an IR spectral profile could be generated from tape samples. These studies also showed that each tape sample generated a unique spectral profile that could be used to differentiate one brand of tape from another.

Although IR spectrophotometry has proven to be a reliable method for differentiating tape samples, long-established IR techniques exhibited three major disadvantages that limited their use in high throughput laboratories. First, sample preparation procedures were time consuming. Second, sample preparation procedures were destructive or required a large amount of sample. Third, long analysis times were necessary in order to obtain good quality spectra (10). Recently however, IR spectrophotometry technology has improved, and as a result, many of these problems have been resolved.

One of the most significant improvements to traditional IR spectrophotometry has been the development of Fourier transform analysis. Most laboratory IR spectrophotometers presently use the concept of Fourier transform analysis to measure the wavelengths of light at which a material will absorb the infrared spectrum. In Saferstien's textbook, *Criminalistics, an introduction to Forensic Science volume six*, the significance of the FT-IR spectrophotometry is explained (22).

> The Fourier transform infrared spectrophotometer (FT-IR) is a low-cost, dependable infrared spectrophotometer. At the heart of the instrument is the Michelson interferometer. The interferometer uses a beam splitting prism and two mirrors, one moveable the other stationary, to direct light toward the sample. As the wavelengths pass through the sample and reach a detector, they are all measured simultaneously. A mathematical operation, the Fourier transform method is then used to decode the measured signals and record the wavelength data. These Fourier calculations are rapidly carried out by a computer. In a matter of seconds a computer operated FT-IR instrument can produce an infrared absorption pattern compatible to one generated by a prism instrument.

The advantages of FT-IR spectrophotometry are similar to that of PGC and PyGC/MS in that there is little sample preparation and that only a small amount of sample is needed to produce good results. One significant difference is that FT-IR analysis is not destructive. Samples analyzed by FT-IR are not consumed in the analysis process and as a result, may be preserved for future testing.

Prior to the development of the FT-IR spectrophotometer, sample preparation using older IR dispersive instruments were cumbersome. Early IR sampling methods, many of which are still utilized, include pelletization with KBr and cast films on the surface of KBr discs (4). These sampling methods were time consuming and required skill in order to produce reliable spectra. In addition, the intractable nature of most types of evidence such as fibers, paints, plastics, rubbers, etc. made it difficult or impossible to sample using traditional sampling techniques like KBr pellets (19). With the development of the FT-IR spectrophotometer, researchers are now able to utilize sampling accessories that were previously unavailable. The newer IR sampling techniques include diffuse reflectance, internal reflectance, attenuated total reflectance, the infrared microscope, and the diamond anvil cell.

A number of studies have explored the use of various IR sampling accessories for characterizing various adhesive samples. Posulnsny and Daugherty 1998 (23) used diffuse reflectance infrared spectrophotometry (DRIFTS) to analyze the adhesives on the back of stamps. Lennard and Mazzella 1991 (20) found DRIFTS to be effective in the analysis of adhesives recovered on questioned documents. Bartick et al. 1994 (16) used internal reflectance infrared spectrophotometry (IRS) to generate spectra from an assortment of products including adhesives. More recently, studies by Merrill and Bartick 1999 (7) and 2000 (25) found attenuated total reflectance (ATR) spectrophotometry to be an effective technique for tape analysis. Each of these studies demonstrates the possibility of characterizing various adhesive samples with FT-IR spectrophotometry. They also show that an FT-IR spectrum can be obtained from a wide variety of adhesive samples provided the proper sampling method or accessory is used.

One of the most useful methods for sampling adhesives is with a diamond anvil cell sampling accessory (19). The diamond anvil cell consists of two diamond windows (anvils) each embedded in the center of a cylindrical piston used to stabilize the anvils when they are pressed together. Virtually any material can be sampled with a diamond anvil cell provided that it is not completely opaque.

The advantage the diamond anvil cell has over other FT-IR sampling accessories is that a diamond anvil cell requires only a microgram of sample to generate a sample spectrum, whereas the other methods require larger amounts of sample. Also, the diamond anvil cell eliminates the need for sample flattening. For some IR applications the sample may need to be flattened by rolling or with a glass knife, in order to be thin enough to allow the IR beam to penetrate the sample. With the diamond anvil cell, the sample is held under constant pressure between the two diamond windows. The constant pressure not only keeps the sample flat, but also keeps the sample in constant contact with the surface of the diamond windows. Samples analyzed under these conditions often produce high quality spectra.

The improved FT-IR sampling methods have made the spectral analysis of products such as adhesives, virtually routine. The rapid scan rate and high specificity of the newer instruments with its accompanying software, make the FT-IR sampling process faster, more accurate, and easier to use. As a result, the process of creating an FT-IR spectral library has also become faster and easier to develop.

Spectral libraries have been developed for comparing, identifying, and classifying various materials. Tungol et al. 1990 (10) and 1991 (26) created a spectral database of fifty-three synthetic fibers using IR microscopy. A test of the database showed that the spectral library search system was successful in identifying the class and subclass of a variety of unknown fibers with the exception of the nylon subclass.

A study by Maynard et al. 2001 (4) created a database of adhesive tapes found commonly in Australia. The database contained detailed information on various physical and chemical characteristics of each brand of tape. Physical characteristics such as surface texture, color, and perforations of the backing material were documented for each sample. Measurements were taken of the width and the thickness of each tape sample. Tape samples were also tested for fluorescence and birefringence properties. The chemical characteristics of each sample were examined using UV spectroscopy, FT-IR spectroscopy using a DRIFTS accessory, and PyGC/MS. With the results of these various physical and chemical tests, a characteristic profile of each tape was developed. The study concluded that it was possible to individually identify an unknown adhesive tape, if a comprehensive characterization scheme was employed.

Master's theses published by Kindig 1997 (9) and Borowski 2000 (27) have also shown the potential of using spectral libraries for the classification and identification of unknown adhesives. Kindig was able to produce IR spectra from a variety of adhesives including cyanoacrylate glues, hide glues, glue sticks, cements, and silicone-type adhesives using FT-IR spectrophotometry. Kindig concluded that the development of a comprehensive spectral library of commercially available adhesives was possible, and that it would be useful in the classification and identification of unknown adhesive samples.

Borowski 2000 followed up on the research of Kindig 1997 by creating a spectral database of superglues. Borowski was able to generate spectra from fifty samples of cyanoacrylate glues (superglues). Borowski found that the spectral library he created was useful in the identification of individual superglue samples.

These studies demonstrate the practicality of an FT-IR spectral library for classifying and identifying various adhesive materials. The development of an FT-IR spectral library of tape adhesives would greatly benefit many forensic analysts, especially those responsible for classifying adhesive materials. Bakowski et al. 1985 (3) clearly states, "A need exists for a library or file capable of comparing an unknown adhesive and to identify or at least classify them." The improvements in FT-IR technology have made the process of developing a specialized FT-IR spectral library of adhesives more manageable.

Methods and Procedures

Previously cited research studies reported that FT-IR analysis of tape samples were useful in the classification and identification of individual samples. These studies however never explained in detail the precise method for preparing adhesive tape samples for FT-IR analysis. As a result, preliminary tests of several different preparation methods were conducted to determine the most reliable method for this study. In one preliminary test, the adhesive from the tape was extracted using a solvent. The solvent was then evaporated onto a sodium chloride disk leaving behind the extracted adhesive residue. This method was time consuming and the spectra produced were noisy with large amounts of interference.

A second method attempted to manually extract the tape adhesive from the sample using a scalpel and a probe, and then spread the extracted adhesive as thinly as possible onto a blank KBr disk. This method produced decent quality spectra however it was often difficult to get the sample spread thinly enough over the blank KBr disk to produce consistent, high quality spectra.

The third method never really materialized. The intent of this method was to dry the adhesive into a solid, and then add the dried adhesive sample into KBr powder and press the sample into a pellet. This method failed because the adhesive never completely dried in the oven. Despite attempts at various temperatures for various lengths of time the tape sample remained consistently tacky.

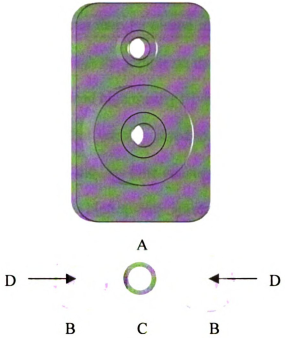

Figure 1. *Components of a Diamond Anvil Cell Sampling Accessory*
(A) Micro compression cell (B) Steel disks (C) Rubber gasket
(D) Diamond windows (anvils)

The best sample preparation method for tape adhesives was analyzing the sample in a diamond anvil cell sampling accessory (Figure 1). A diamond anvil cell consists of two finely polished diamond windows (anvils) (Figure 1 D) each embedded in the center of two steel disks (Figure 1 B). A small amount of sample is placed between the two diamond windows making a sort of sandwich with the sample. The rubber gasket (Figure 1 C) is also placed between the two disks for stability and to help ease the compression of the sample. The two disks containing the sample are then placed in the micro compression cell (Figure 1 A), and then screwed down compressing the two disks together. The tightening of the compression cell crushes the sample in between the two windows and spreads it thinly and evenly across the surface of the diamond windows. The entire micro compression cell can then be placed in the FT-IR sampling chamber. The IR beam is then able to pass through the sample on its way to the detector.

The diamond anvil cell produced high quality FT-IR spectra. The spectra had less background noise and more peaks that were resolved to the baseline. In addition, the sampling process was much simpler and less time consuming. Because of these positive results, and especially because of the high quality spectra produced, it was determined that the diamond anvil cell would be used to sample the tape adhesives in this study.

Before the diamond anvil cell sampling method was used in this study, its sampling procedure was validated. The validation of this method was done with a reproducibility study where one tape product was sampled ten times under the same parameters to ensure that the resulting spectra were consistent every time. The results of the validation showed that the diamond anvil cell tape sampling procedure could produce consistent results.

Having settled on the best sampling method for this project, various tape samples were then collected. Fifty samples of commercially available tapes were purchased from local hardware stores, office supply stores, area supermarkets, and convenience stores in the Lansing, Michigan area. A complete listing of the tapes that were sampled can be found in Table 1.

The samples in this study are comprised of tape products from three categories of tapes: office tapes, packaging tapes, and electrical tape. Office tapes generally have a clear colorless backing often made from cellophane. This type of tape is used in office settings to attach papers, and is most often referred to as "Scotch tape." Packaging tapes have a wider, thicker backing than office tape. They are used to seal cartons and boxes and generally appear in stores with either a colorless or tan colored backing. Electrical tapes have a thick backing often made from polyvinyl chloride (PVC). They are used to insulate bare electrical wire and come in a wide variety of colors.

The instrumentation used in the tape adhesive analysis was a Nicolet Model 460 Protégé FT-IR. OMNIC 5.1 software was used for the data collection, analysis, and for the library creation. The sampling accessory used to analyze the tape adhesive was a Spectra-Tech 4x BC Sample Plan Diamond Anvil Cell.

Prior to the first run of the day, the bench was aligned and parameters set at 300 scans with a resolution of 2. An atmospheric reference spectrum was obtained in order to account for the levels of water and carbon dioxide present in the room. The atmospheric reference consisted of 300 scans run with the bench cover open. Following the atmospheric reference, the final sampling format was set to absorbance, and no corrections were made to the saved spectra.

Before each sample was analyzed, a background spectrum of dry KBr powder was taken using the diamond anvil cell sampling accessory. This was done by placing the sample in the FT-IR sampling compartment then purging it in nitrogen for five to ten minutes to clear the chamber of air containing carbon dioxide and water. Following the purging period, 300 scans were taken as the background spectrum. Once the background spectrum was obtained, the diamond anvil cell was cleaned and prepared for tape adhesive sampling.

Tape adhesive samples were then prepared for analysis. The adhesive from the tape was carefully removed from the backing using a metal probe and a scalpel. Care was taken to ensure that only the adhesive was removed from the backing. With the scalpel and probe, a small amount of adhesive was worked into a glob about the size of a pin head. The glob was then removed from the tape with forceps and placed between the two windows of the diamond anvil cell. The diamond anvil cell was then tightened, compressing the glob thinly and evenly across the windows of the diamond anvil cell, making the sample ready for analysis.

Each prepared sample was then placed into the compartment of the FT-IR and purged for about ten minutes. Following the purging period, 300 scans were taken of the sample. The computer took a ratio of the background to the sample, producing the final IR spectrum.

Once a sample spectrum was obtained it was checked to see if it met basic quality requirement standards. Each spectrum had to have no interference which meant that the baseline of the spectrum was not elevated, that peaks were resolved to the baseline, and that the spectrum was free of noise. Also the absorbance had to fall into a range between 0.5 and 2.0 in order to be considered good quality spectra.

Each spectrum that met the quality requirements was added to an internally created spectral library. The library was developed using the library manager application found in the OMNIC software. All spectra were corrected using the automatic baseline correction function before being added to the internal library. The library was set at a resolution of 2 and the spectral format was set to absorbance. The frequency limits were set at 400 cm^{-1} to 4000 cm^{-1}.

Each sample that was added to the spectral library was assigned a file name and saved. Basic product information of each tape was added to the database. Information such as brand name of the sample, sample file name, atmospheric reference file name, data collected, manufacturer, contact information for the manufacturer, UPC code, re-order number, lot number, method of collection, and active ingredient were recorded. Most of this information was taken directly from the packaging of the product.

At the completion of the sampling of all fifty samples, a test of the search capabilities of the newly-created tape adhesive library was conducted. In the test, ten adhesive samples were randomly selected from the fifty samples. Using the same sampling procedures, an FT-IR spectrum of each of the ten samples was generated; the only difference being that the test samples were run at 50 scans instead of at 300 scans. Borowski 2000 noted that fifty scans would test the minimal capabilities of the library (27). The ten test samples were then searched against the library to determine the effectiveness of the newly created spectral library. The results of the ten test samples are discussed in the Results and Discussion section of this thesis.

Table 1. Complete Listing of the Tapes sampled in the FT-IR Spectral Library

Sample	Common Name	Collected	Classification
1	Spartan Invisible Tape	4-28-00	Office Tape
2	Stockwell Invisible Tape	4-28-00	Office Tape
3	Office Max Invisible Tape	4-28-00	Office Tape
4	LePages Stick-a-Saurus See-Thru-Tape	4-21-00	Office Tape
5	Annapolis Invisible Tape	4-21-00	Office Tape
6	Annapolis Crystal Clear Tape	4-21-00	Office Tape
7	Rite Aid Invisible Tape	4-21-00	Office Tape
8	ACE Transparent Tape	4-21-00	Office Tape
9	ACE Invisible Tape	4-21-00	Office Tape
10	Let's Build-It Cellophane Tape	4-28-00	Office Tape
11	Office Select Invisible Tape	4-21-00	Office Tape
12	Office Select Transparent Tape	4-28-00	Office Tape
13	Kroger Invisible Tape	4-22-00	Office Tape
14	Rite Aid Transparent Tape	4-22-00	Office Tape
15	Kroger Transparent Tape	4-22-00	Office Tape
16	Target Invisible Tape	4-22-00	Office Tape
17	Hallmark Invisible Tape: Ruban Invisible	4-22-00	Office Tape
18	Highland Invisible Tape	4-25-00	Office Tape
19	Highland Invisible Tape: Ruban Invisible Highland	4-25-00	Office Tape
20	Scotch Utility Tape	4-25-00	Office Tape
21	Home Harvest Transparent Tape	4-25-00	Office Tape
22	Arbor Invisible Tape	4-25-00	Office Tape
23	Arbor Transparent Tape	4-26-00	Office Tape
24	ACE Carton Sealing Tape	4-28-00	Packaging Tape
25	Let's Build-It Transparent Packing Tape	4-28-00	Packaging Tape
26	Manco Super Strength Strapping Tape	4-28-00	Packaging Tape
27	Clear Package Sealing Tape	4-28-00	Packaging Tape
28	ACE Strapping Tape	4-28-00	Packaging Tape
29	Gould Quikstik Clear Polypropylene Packaging Tape	4-29-00	Packaging Tape
30	Gould Quikstart Packaging Tape	4-29-00	Packaging Tape
31	Mail Away Clear Carton Sealing Tape: Industrial Quality	5-01-00	Packaging Tape
32	Kroger Clear Sealing Tape	5-01-00	Packaging Tape
33	Mail Away Super Select Carton Sealing Tape	5-01-00	Packaging Tape
34	Manco EZ Start Frustration Free Tape	5-01-00	Packaging Tape
35	ACE Tan Carton Sealing Tape	5-01-00	Packaging Tape
36	Kroger Strapping Tape	5-01-00	Packaging Tape
37	Gould Quikstik Strapping Tape: High Tensile	5-01-00	Packaging Tape
38	Gould Quikstik Filament Strapping Tape	5-01-00	Packaging Tape
39	Gould Quikstik Self Adhesive Mailing Tape	5-01-00	Packaging Tape
40	Mail Away Fragile Tape	5-01-00	Packaging Tape
41	Let's Build-It Color Tape	5-04-00	Electrical Tape
42	Plymouth Bishop Tri Pack Vinyl Color Coding Tape	5-04-00	Electrical Tape
43	GB Electrical Tape	5-04-00	Electrical Tape
44	GAMPAK Premium Grade Electrical Tape: All Weather-Black	5-04-00	Electrical Tape
45	GAMPAK Premium Grade Electrical Tape: All Weather-Green	5-06-00	Electrical Tape
46	ACE General Purpose Vinyl Electrical Tape	5-08-00	Electrical Tape
47	ACE All Weather Vinyl Electrical Tape	5-06-00	Electrical Tape
48	ACE Extreme Weather Vinyl Electrical Tape	5-06-00	Electrical Tape
49	Manco 667 Pro Series Red Vinyl Electrical Tape	5-08-00	Electrical Tape
50	Manco 667 Pro Series Blue Vinyl Electrical Tape	5-08-00	Electrical Tape

Results and Discussion

Fifty IR spectra were obtained from various tape adhesives. Each spectrum was placed into one of three categories according to their end use. The tape samples were classified as office tape, packaging tape, or electrical tape. An example of a spectrum from each category is found in Figures 2-4.

An analysis of the resulting tape adhesive spectra found that most of the spectra shared similar peak areas regardless of class or brand. The most common peak shared is a large broad peak that occurs around 3000 cm^{-1} and 2700 cm^{-1}, which could possibly be a grouping of three or more unresolved peaks. This large peak was found in all fifty sample spectra produced. Similar peaks in this region are found in the cyanoacrylate glues documented by Borowski 2000 (27) and in some superglue and cement samples documented by Kindig 1997 (9).

Another relatively common peak found in most tape adhesive spectra is a sharp peak that occurs around 1750 cm^{-1}. This peak was found in thirty-eight of the fifty tape samples. This peak occurred in all ten electrical tape spectra and was found in twenty of the twenty-three office tape spectra. This peak appeared the least in packaging tapes where it was found in only eight of the seventeen samples.

Despite several similarities, variations were observed in certain regions within each spectrum. In all samples, the region of greatest variability was the fingerprint region. The fingerprint region of the spectra is found between 1600 cm^{-1} and 700 cm^{-1}. It is a region where unique characteristics of each sample are most likely to be exhibited.

The results from the ten randomly selected test samples were promising and are displayed in Table 2. For each of the ten unknown samples the computer library search system lists the ten best matching spectra from the library. The spectrum in the database that matches closest to the unknown spectrum is listed as the number one hit, or best match, followed by the next best matches in descending order.

The samples are also assigned a match percentage, which indicates how closely each sample resembles the unknown or test sample. A perfect match would be designated as 100%. The more similarities shared between the unknown spectrum and the known sample from the library, the higher the match percentage.

The test successfully identified four of the ten samples correctly as the number one hit, or best matching spectrum. Of these four number one hits, two were office tapes and the other two were electrical tapes. Five of the remaining six samples were identified in the top five matches for their respective searches, with only one sample identified below five.

Sample number 22, Arbor Invisible Tape produced a low hit number at nine. The reason for such a low hit number is because the other tapes listed before it share similar chemical components in their adhesives, thus producing similar FT-IR spectra. The similarities between the spectra are illustrated in Table 2. under the column entitled, "% off from Actual Sample". The results show that there is less than a 1% difference between the nine samples that make up the number one through number nine hits. The similarities are so close in fact that there is a good possibility that these tapes, or their adhesive components, could have originated from the same source.

Marketing factors could possibly explain how these nine different products could have come from the same source. A common business practice among smaller tape manufacturers is to purchase raw materials or pre-made adhesives from a single source. It is also possible that several small tape retailers may purchase generic tape products from a single wholesale supplier, then package, label, and independently distribute essentially the same tape products under different company brand names.

The results from this test are listed below in Table 2. The column "Sample #" indicates the sample number as it relates to Table 1. The column "Sample Name" lists the names of each of the samples that were randomly selected to search against the library. The column "Hit #" lists the rank of the actual sample as searched by the computer. The "Match Percentage" lists how closely the actual sample from the FT-IR library matched the unknown sample. The column heading "% off from Actual Sample" lists the difference between the match percentage of actual sample and the match percentage of the number one hit.

Table 2. Library Test Results

Sample #	Sample Name	Hit #	Match Percentage	% off from Actual Sample
8	ACE Transparent Tape	1	98.13%	-
28	ACE Strapping Tape	2	98.86%	0.40%
41	Let's Build-It Color Tape	1	99.73%	-
13	Kroger Invisible Tape	4	99.50%	0.22%
43	GB Electrical Tape	1	99.70%	-
15	Kroger Invisible Tape	1	99.70%	-
5	Annapolis Invisible Tape	2	99.31%	0.03%
36	Kroger Strapping Tape	3	98.30%	1.17%
30	Gould Quikstart Packaging Tape	5	91.20%	7.53%
22	Arbor Invisible Tape	9	98.94%	0.66%
	Average	2.9	98.34%	0.99%

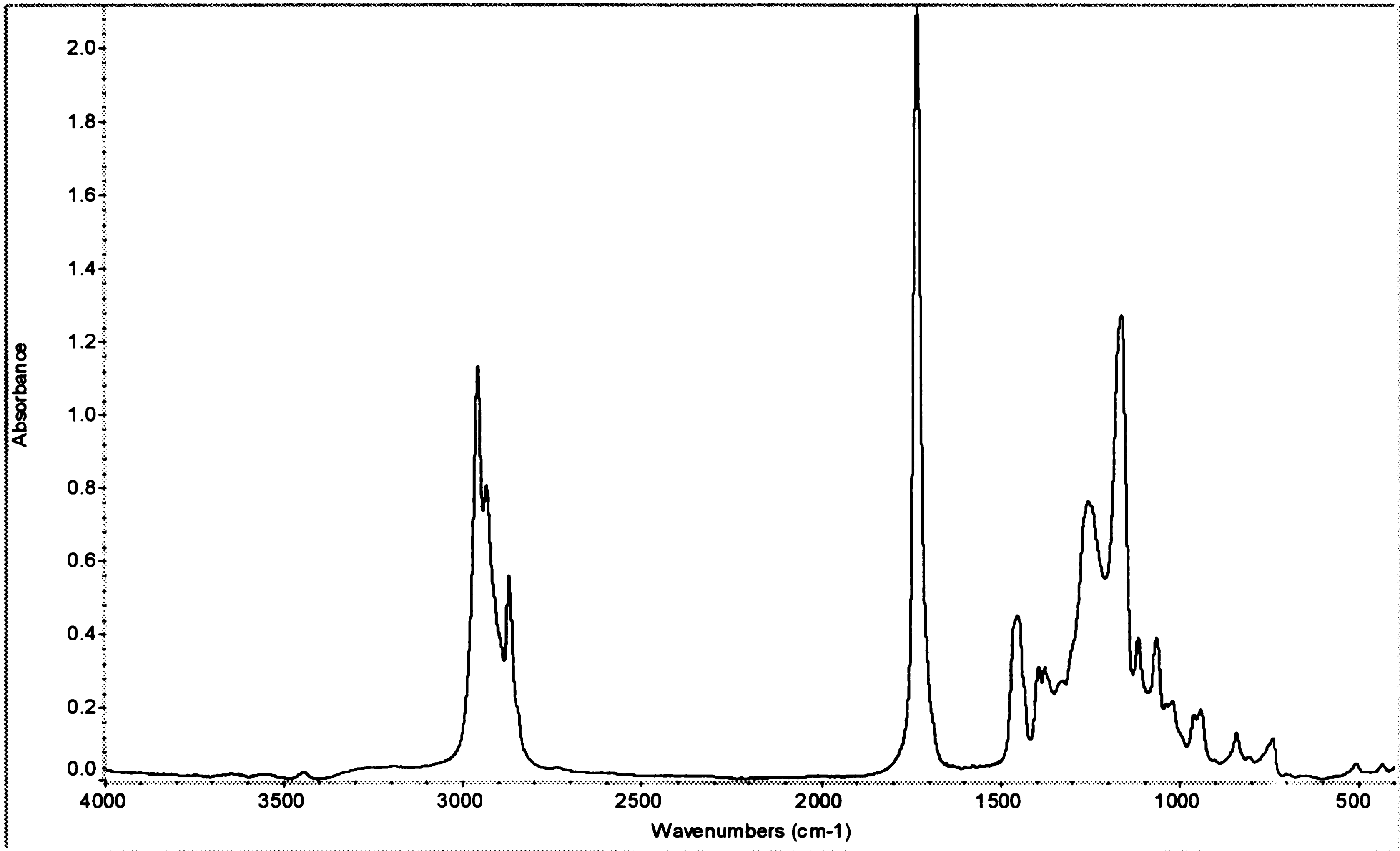

Figure 2. *FT-IR Spectrum of Arbor Invisible Office Tape*

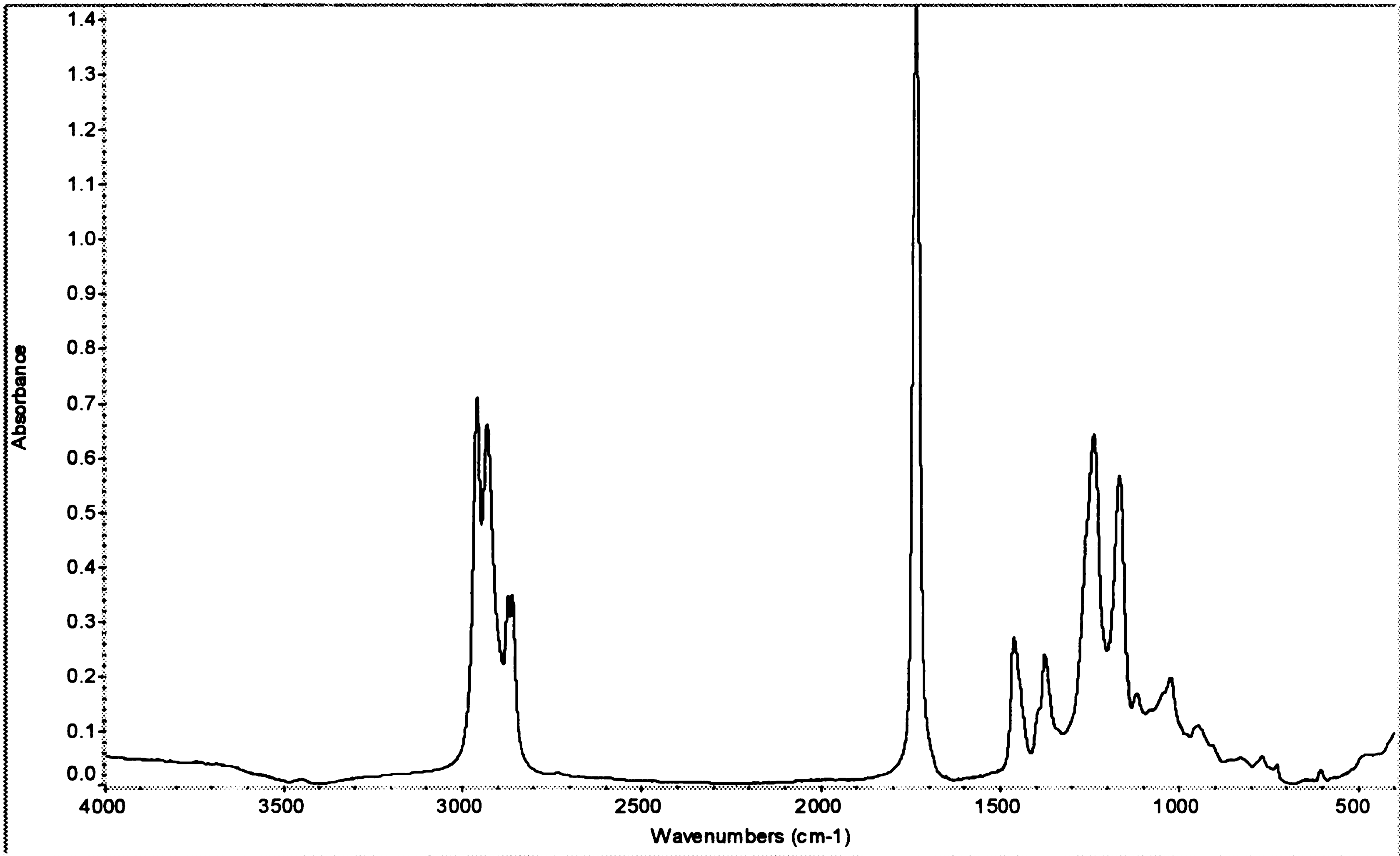

Figure 3. *FT-IR Spectrum of Gould Quikstart brand Packaging Tape*

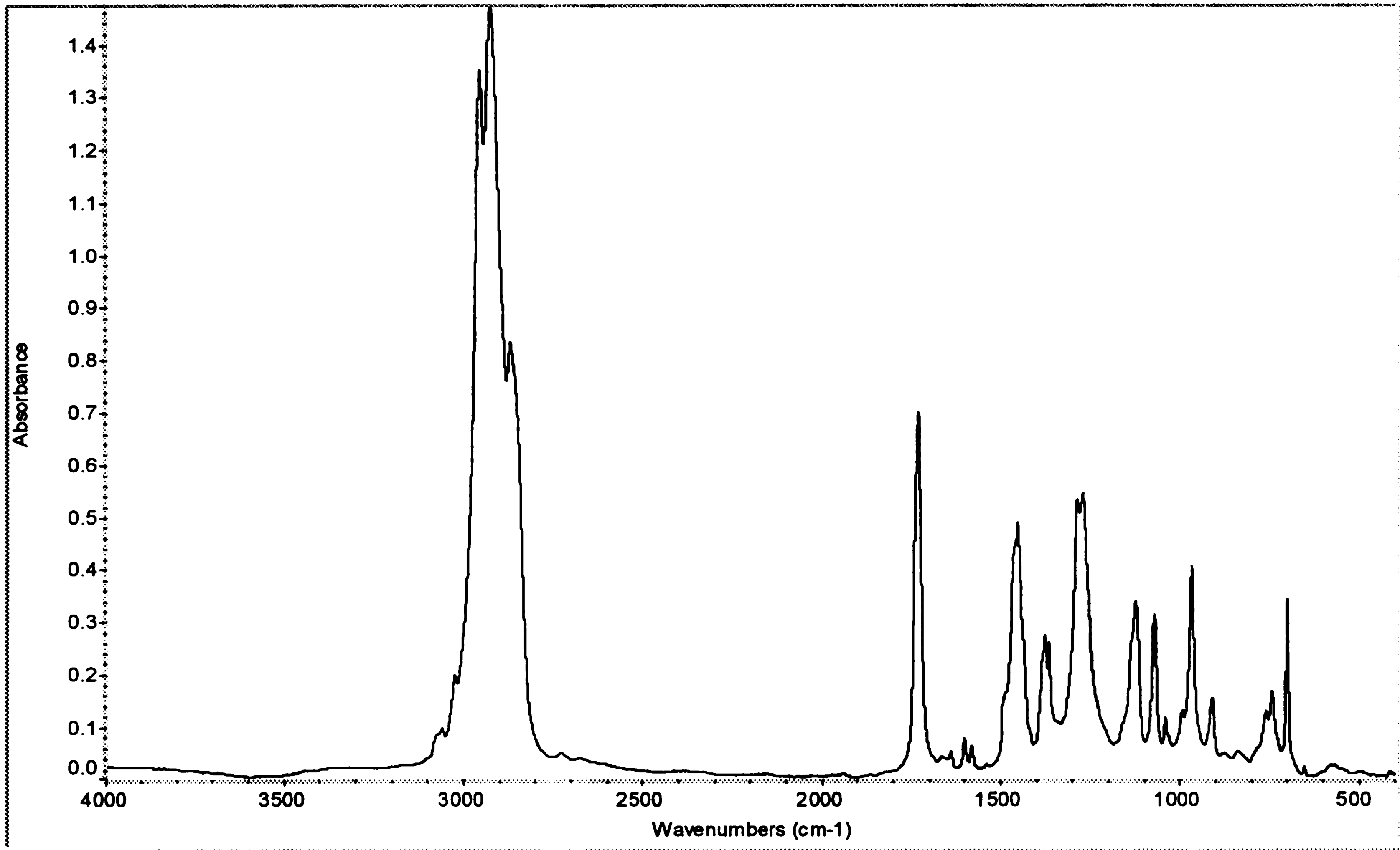

Figure 4. *FT-IR Spectrum of ACE brand All-Weather Vinyl Electrical Tape*

Conclusion

Tape is an excellent source of physical evidence. It is used by criminals to bind and gag victims, to package illicit drugs (1), it has been recovered on questioned documents (2) and is used in the construction of various improvised explosive devices (3). As a pressure sensitive adhesive, tape has a backing and an adhesive side. Both sides provide forensic analysts with a variety of physical and chemical features from which to examine. The adhesive however, offers much more discriminating value as evidence than the backing because the adhesive formulas are complex and contain many different components (16).

Through the use of an FT-IR spectrophotometer and a diamond anvil cell sampling accessory, an FT-IR spectral library of fifty tape adhesives was created. High quality FT-IR spectra were obtained from each of the of the tape adhesives sampled. The methods and procedures used were non-destructive, sample preparation time was kept to a minimum, and only a small amount of sample was used.

The test of the search function of the newly created spectral library demonstrated that there is potential for using the FT-IR spectra to at least classify tape adhesives into categories of office, packaging, or electrical tapes, and in some cases even identify individual samples. Despite similarities in peak characteristics, the FT-IR spectra of each sample exhibited enough variation to distinguish one sample from another. Even though the variations were slight, the FT-IR spectral search system was able to identify the correct spectra within its top ten matches.

The results from this test reaffirm the importance of analyst interpretation. Although the FT-IR spectrum of a sample is unique and widely used to differentiate samples, it would be unwise for any analyst to base an identification entirely on the results of a computer generated search. Other physical and chemical characteristics should be compared before reaching a final conclusion.

Although marketing factors (like the one encountered in test sample number 22) may prevent the possibility of individually identifying an unknown tape to a specific brand, it does not diminish the importance of the FT-IR spectral library. The value of the FT-IR spectral library is that it can identify an unknown to a specific class. Class identification can still be effective, especially because there are so many adhesive classes. The ability to classify an adhesive as a superglue, wood glue, epoxy, etc., can significantly narrow the possible sources, and help investigators focus on specific products.

In addition to creating a spectral library of tapes adhesives, this study also found the diamond anvil cell sampling accessory to be the most effective sampling accessory for obtaining high quality FT-IR spectra from adhesive tapes. Even though Borowski 2000 (27) found this sampling accessory ineffective for producing quality spectra of superglues, it proved to be the best method for FT-IR characterization of tape adhesives.

A possible explanation as to why the diamond anvil cell did not produce quality spectra for Borowski is in the physical state of the superglue samples. The superglue samples that Borowski analyzed were hard and brittle, while the adhesive samples used in this study were soft and malleable. The hard superglue samples may not have allowed the sample to make sufficient contact with the diamond anvil windows, which would have created poor quality spectra. Fortunately, the adhesive samples in this study made good contact with the diamond anvil windows, which in turn produced good quality spectra.

Future research studies can be designed to explore the usefulness of other FT-IR sampling accessories on tape samples. Methods such as attenuated total reflection (ATR) may have the potential to provide better spectra than those generated using the diamond anvil cell. ATR offers much simpler sample preparation than the methods used in this study. For instance, a tape sample need only be laid atop an ATR sampling material, most often a crystal such as thallous bromide-iodide (KRS-5) or zinc selenide (ZnSe) to generate an FT-IR spectrum. The problem however with ATR is that it is difficult to know the depth at which the IR beam is penetrating the sample. The IR beam could possibly be penetrating into the backing material as well. In order to avoid this problem, it is important that a good background spectrum of the backing material is taken before sampling the adhesive, and then subtracted from the final spectrum. If the background spectrum is not subtracted out, its components may interfere with the final spectrum.

Another suggested research project would be to explore the variability of tape within a single roll. This could be done by taking the FT-IR spectrum of a single adhesive product at varying lengths along the roll to find out if there is variation within a roll of tape, or if the spectral profile remains consistent throughout the entire roll.

Other studies could monitor changes in a single brand of tape purchased over a period of time. The study could explore the frequency at which a single brand of tape may change its formula and the extent to which the formula changes over time. Knowing more about the variability of a tape product can help determine the evidentiary value of tape. The more frequently a tape changes the more variable the product is, and thus the easier it will be to determine significance of the product.

The results of this study have shown that tape adhesive can be characterized by FT-IR spectrophotometry using a diamond anvil cell. Also, this study has shown that an FT-IR spectral library can be useful in classifying similar tape adhesive products. It is not likely that the identification by spectral characterization alone will lead to the positive identification of a tape adhesive to the exclusion of all others. For this reason it is wise to employ a selection of methods that test the physical and chemical characteristics of the sample before a final conclusion is reached. The development of a well-rounded characterization scheme like the one found in Maynard et al. 2001 (4), where numerous physical and chemical characteristics are analyzed, will give the analyst more definitive results when classifying or identifying adhesive tape samples.

List of References

1. Snodgrass, H., *Duct Tape Analysis as Trace Evidence*, Proceedings of the International Symposium on Trace Evidence, FBI Academy, Quantico, VA. June 1991, pp. 69-73.

2. Purtell, D., and Casey, M., *Paper Tapes and Labels Encountered in Document Examination*, J. of Forensic Sci 1966; (11) 2:496-506.

3. Bakowski, N.L., Bender, E.C., Munson, T.O., *Comparison and Identification of Adhesives used in Improvised Explosive Devices by Pyrolysis-Capillary Column Gas Chromatography-Mass Spectrometry*, J. Anal. & Appl. Pyrol, 8 (1985) pp. 483-492.

4. Maynard, P., et al., *Adhesive Tape Analysis: Establishing the Evidential Value of Specific Techniques*, J. of Forensic Sci 2001; (46) 2:280-287.

5. Satas, D., *Pressure Sensitive Adhesives and Adhesive Products in the United States,* in Handbook of Pressure Sensitive Adhesive Technology, Second Edition, D. Satas ed., Van Norstrand Reinhold Co., New York, NY, 1989 pp. 1-23.

6. Smith, J., *The Forensic value of Duct Tape Comparisons,* Midwestern Association of Forensic Scientists Newsletter, Vol 27 (1) Jan 1998, pp. 28-33.

7. Merrill, R.A., and Bartick, E.G., *Advances of Infrared ATR Analysis of Duct Tape*. Presented at the 51st annual meeting of the American Academy of Forensic Sciences, Orlando FL. February 15-20, 1999.

8. Kendall, D. N., *Analytical Techniques for Identification and Characterization*, in Handbook of Pressure Sensitive Adhesive Technology, Second Edition, D. Satas ed., Van Norstrand Reinhold Co., New York, NY, 1989, pp. 204-243.

9. Kindig, J., *The Development of a FTIR Spectral Library of Commercial Adhesives*, Michigan State University, 1997, 39 pgs.

10. Tungol, M. W., Bartick, E.G., and Montaser, A., The Development of a Sectral Data Base for the Identification of Fibers by Infrared Microscopy, Applied Spectroscopy, 1990; (44) 4:543-549.

11. Skiest, I., *Introduction to Adhesives,* in Handbook of Adhesives, I. Skiest ed., Van Norstrand Reinhold Co., New York, NY, 1962, pp. 1-13.

12. Shields, J., *Introduction*, in Adhesives Handbook, J. Shields ed., CRC Press, Boca Raton, FL, 1970, pp. 1-6.

13. Huck, V., *Birth of the "Scotch" Clan*, in Brand of the Tartan: the 3M Story, V. Huck author, Appleton-Century-Crofts, Inc. New York, NY, 1955, pp. 131-138.

14. Landrock, A. H., *Adhesive Types and Their Properties and Applications*, in Adhesives Technology Handbook, Noyles Publications, Park Ridge, NJ, 1985, pp. 126-189.

15. Benedek, I., and Heymans, L.J., *Introduction*, in Pressure Sensitive Adhesives Technology, I. Benedek and L.J. Heymans eds, Marcel Dekker Inc., New York, NY, 1997, pp. 1-3.

16. Bartick, E. G., Tungol, M.W., Reffner, J.A., *A new approach to forensic analysis with infrared microscopy: internal reflection spectroscopy*, Analytica Chimica Acta, 1994, Vol 288, Iss 1-2, pp. 35-42.

17. Noble, W., Wheals, B.B. and Whitehouse, M.J., *The Characterisation of Adhesives by Pyrolysis Gas Chromatography and Infrared Specroscopy*, Forensic Science, 3 (1974) pp. 163-174.

18. Williams, E.R., and Munson, T.O., and B.S., *The Comparison of Black Polyvinylchoride (PVC) Tapes by Pyrolysis Gas Chromatography*, J. of Forensic Sci 1988, (33) 5:1163-1170.

19. Suzuki, E. M., *Forensic Applications of Infrared Spectroscopy*, in Forensic Science Handbook, Volume III, R. Saferstein ed., Prentice Hall, Englewood Cliffs, NJ, 1993, pp. 71-195.

20. Keto, R.O., *The Characterization of Black Polyvinyl Chloride Electrical Tape*, Proceedings from the International Symposium on the Analysis and Identification of Polymers, FBI Academy, Quantico, VA, July 31-Aug 2, 1984, pp. 137-143.

21. Kee, T. G., The *Characterization of PVC Adhesive Tape*, Proceedings from the International Symposium on the Analysis and Identification of Polymers, FBI Academy, Quantico, VA, July 31-Aug 2, 1984, pp. 77-85.

22. Saferstein, R., *Organic Analysis*, in Criminalistics an Introduction to Forensic Science, Sixth Edition, R. Saferstien author, Prentice Hall, Inc., Upper Saddle River, NJ, 1998, pp. 129-163.

23. Posulsny, M., and Daugherty, K.E., *Nondestructive Adhesive Analysis on Stamps by Fourier Transform Infrared Spectroscopy*, Applied Spectroscopy 1988, (42) 8:1466-1469.

24. Lennard, C.J., and Mazzella, W.D., *A simple combined technique for the analysis of toners and adhesives*, J. of Forensic Science Society 1991, (31) 3:365-361.

25. Merrill, R.A., and Bartick, E.G., *Analysis of Pressure Sensitive Adhesive Tape: Evaluation of Infrared ATR Accessory Advances*, J. of Forensic Sci 2000, (45) 1:93-98.

26. Tungol, M.W., Bartick, E.G., Montaser, A., *Spectral data base for the identification of fibers by infrared microscopy*, Spectrochimica Acta 1991, Vol 46 B, No. 11, pp. 1535E-1544E.

27. Borowski, S. G., *Development of a Comprehensive Adhesive Spectral Library – Cyanoacrylate Glues*, Michigan State University, 2000, 47 pgs.