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ARTIFICIAL AGING AND ITS EFFECT ON ADHESIVES AND BACKINGS OF OFFICE AND PACKAGING TAPES

presented by

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has been accepted towards fulfillment of the requirements for the

degree in

Master of Science **Criminal Justice**

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FTIR SPECTROPHOTOMETRY OF OFFICE AND PACKAGING TAPES, THAT HAVE BEEN SUBJECTED TO ACCELERATED AGING BY INCANDESCENT AND UV LIGHT

Bу

Chadwyck Lynn Douglass

A THESIS

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

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ABSTRACT

FTIR SPECTROPHOTOMETRY OF OFFICE AND PACKAGING TAPES, THAT HAVE BEEN SUBJECTED TO ACCELERATED AGING BY INCANDESCENT AND UV LIGHT

By

Chadwyck Lynn Douglass

Fourier transform infrared spectrophotometry (FTIR) may be used to aid in the analysis of adhesives and backings from tape samples. Backings and adhesives from artificially aged and naturally aged packaging and office tape were analyzed. The spectra of aged and unaged backings and adhesives were compared to each other to determine if changes were present. Results show that ultraviolet light and incandescent light cause changes in some backings and adhesives when artificially aged. To Jennifer, Liam and Jack. Thank you for all of your insight, love, support and kindness during this process.

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TABLE OF CONTENTS

LIST OF TABLESvi
LIST OF FIGURES
INTRODUCTION
CLASSIFICATION AND MANUFACTURING OF ADHESIVE TAPES
REVIEW OF LITERATURE
MATERIALS AND METHODS14
RESULTS AND DISCUSSION
CONCLUSION
APPENDECES APPENDIX A Unaged Tape Backing and Adhesive FT-IR Spectra
REFERENCES90

LIST OF TABLES

Table 1 – IR Changes with Age for 51 Packaging and Office Tapes	15
Table 2 – Physical description of 51 Packaging and Office Tapes	16
Table 3 – Weather Conditions for Outside Aging of Tapes	19
Table 4 – IR Changes in Backings Related to Time Aged with a 100-W bulb	28
Table 5 – IR Changes in Adhesives Related to Time Aged with a 100-W bulb	28
Table 6 – IR Changes in Adhesives Related to Time Aged with a UV light	29

LIST OF FIGURES

-	A schematic drawing of the FT-IR used in this study with major parts labeled
Figure 2 - A	A schematic drawing of an ATR crystal with the IR beam reflecting through the crystal and into the sample6
Figure 3 –	A sample spectrum of an unaged adhesive considered unchanged when compared to the aged adhesive
Figure 4 –	A sample spectrum of an aged adhesive considered unchanged when compared to the unaged adhesive
Figure 5 –	A sample spectrum of an unaged adhesive considered changed when compared to the aged adhesive
Figure 6 –	A sample spectrum of an aged adhesive considered changed when compared to the unaged adhesive
Figure 7 –	Sample spectrum of an office tape adhesive with broad peaks between 3000 cm ⁻¹ and 2800 cm ⁻¹ and peaks around 1730 cm ⁻¹ , 1450 cm ⁻¹ and 1375 cm ⁻¹ 24
Figure 8 –	Sample spectrum of a packaging tape adhesive with broad peaks between 3000 cm ⁻¹ and 2800 cm ⁻¹ and peaks around 1730 cm ⁻¹ , 1450 cm ⁻¹ and 1375 cm ⁻¹ 25
Figure 9 –	Sample spectrum of an office tape backing with large, broad peaks between 3000 cm ⁻¹ and 2800 cm ⁻¹ 25
	- Sample spectrum of an office tape backing with smaller, less broad peaks between 3000 cm ⁻¹ and 2800 cm ⁻¹
	- Sample spectrum of a packing tape backing with broad peaks between 3000 cm ⁻¹ and 2800 cm ⁻¹ and peaks around 1450 cm ⁻¹ and 1375 cm ⁻¹
-	- Sample spectrum of a packing tape backing with well defined peaks between 3000 cm ⁻¹ and 2800 cm ⁻¹ and one peak around 1450 cm ⁻¹ 27

Introduction

Various types of tape can be used in the commission of a crime. These tapes may be new or many years old. In this study the adhesive and backing of two types of tape were artificially aged and analyzed to determine if their infrared spectra changed as they aged. By aging the tapes, seeing if there are changes in their spectra and trying to determine what the changes came from may allow investigators to include or exclude people from their list of potential suspects based on the tapes to which they have access. Also, if a piece of tape on an item recovered has been off the roll for a time it could show different characteristics than the tape that is still on the roll. Thus the investigator and forensic scientist should not eliminate the tape still on the roll from being the taped used based on IR spectra alone.

In the process of investigating a crime physical evidence can be a major key in identifying a suspect and solving the crime that was committed. Forensic scientists first analyze the evidence using scientific techniques and methods that are generally accepted. They then draw their conclusions based on their interpretation of the physical evidence as well as their education, training and experience.

Packaging tape and clear tape, also referred to as adhesive tape or "Scotch" tape can be physical evidence. These tapes are common products found in many homes and businesses across the country. The uses of these tapes are numerous and include sealing packages to be mailed, mending papers or wrapping presents. Illegal uses include wrapping bundles of drugs and making improvised explosive devices. Because these tapes are so widely available it would be helpful if the forensic scientist could determine how both the adhesive on and the backing of these tapes age with time. If the tapes age,

defined here as changing either chemically or physically due to environmental conditions such as exposure to light or moisture, this may allow for the tape in question, once analyzed, to be linked to a particular roll of tape and that roll in turn to a suspect. To most people tape may not seem like valuable evidence, however, when analyzed properly tape can provide enough information to link the suspect to a crime.

Tape is classified as a pressure sensitive adhesive, which is an adhesive that is applied to a backing (1). The addition of the backing makes tape different from most traditional adhesives and permits two items to be analyzed as opposed to just the adhesive. Some pressure sensitive adhesive products include labels, miscellaneous products like Post-It Notes, and of course, tapes. Pressure sensitive adhesives normally adhere firmly to many different surfaces with the need for only finger or hand pressure. Also, the adhesive does not require a solvent, water or heat to become activated or have a strong hold. The adhesive and backing, when analyzed in combination, may allow forensic scientists to differentiate tape samples from one another.

Of the two constituent parts of the tape, the adhesive exhibits more variability than the material that makes up the backing (2). The adhesive, like traditional adhesives, is composed of binders, tackifiers and fillers, which contain a variety of natural and synthetic materials. Typically the backing of the tape is composed of paper, cloth or plastic. Until recently an important component in the backing of tape was cellophane. This is because cellophane has exceptional strength but can still be easily torn and is also very transparent. However, recently cellophane is being replaced by cellulose acetate, which exhibits many of the same properties as cellulose, but also resists ultraviolet light, greases, oils and moisture (2).

Previous studies have described the development of an infrared spectral library of tape adhesives, techniques to sample the adhesive of the tape and the evidentiary value of tape (2, 3). In these studies the backings were not always analyzed but when they were they and the adhesives were analyzed to determine if their manufacturer could be identified. A previous study showed that backings were too similar to differentiate a manufacturer for certain, but the adhesive contained enough variation to be able to establish a link between two tapes or a tape and a manufacturer (4). However, there have been no reported studies to determine how certain adhesive tapes change with the passage of time; doing such a study would allow for a comparison to be made between known and unknown aged tapes as well as determining what evidential value aged tape has if it is found at a crime scene.

This study is an attempt to determine if the infrared characteristics of tapes change as the tape ages. This can help to establish whether or not a particular roll of tape taken from a suspect's house could have been used in the crime that is being investigated. The method of collection and analysis used in this study was non-destructive, so if needed the sample could be reanalyzed by either FT-IR or some other analytical method such as pyrolysis gas chromatography. The sample preparation time and amount of the sample used were kept to a minimum, as is needed in the day-to-day operation of a forensic laboratory. The tapes that were chosen in this study were various types of clear office tapes and packaging tapes that originated from several manufacturers, including different brands from each manufacturer.

Analytical techniques used by the forensic scientist to compare the components of tape include pyrolysis gas chromatography and infrared (IR) spectrophotometry. The

predominant type of IR spectroscopy used today is Fourier transform IR (FTIR) spectroscopy. Some advantages of this type of spectroscopy over the previously used dispersive IR spectroscopy are that it takes less time to obtain a spectrum and the energy that reaches the sample is greater. The radiation from the IR source is sampled simultaneously, which is why this method is quicker than the dispersive method. In order to obtain a spectrum the sample being analyzed is placed in or on the sample holder and is irradiated by the IR radiation. The samples in this study were placed on the sample holder and compressed using a diamond compression cell attenuated total reflectance (ATR) accessory, which flattened the sample into a thin layer across the sampling area of the accessory. The most often used method for comparison of tape adhesives and backings is infrared spectrophotometry. This is primarily because of its short sample preparation time, the time it takes for the instrument to collect a spectrum and the fact that it is a non-destructive technique.

While the idea of IR radiation being absorbed by a sample and detected by a detector may seem straight forward, in reality the radiation passes through many steps to reach the sample and then the detector. A schematic of an infrared spectrophotometer is seen in figure 1. The IR radiation is produced at a start-point, called a source and proceeds through five steps prior to reaching the sample. After the radiation leaves the source it bounces off a mirror that directs it to a beam splitter. This beam splitter, as the name implies, splits the radiation into two groups, sending part of the radiation to a fixed mirror and the other part to a moving mirror. Once the radiation reaches these two mirrors it is bounced back to the beam splitter, allowed to pass through it and onto the next set of mirrors. Because one mirror is fixed and one is moving this can cause either

constructive or destructive interference in the wavelength of the radiation being transmitted. The radiation from constructive interference is transmitted to the sample and then on to the detector. However, radiation from destructive interference never reaches the detector because the two wavelengths are out of phase with each other, therefore cancel each other out.

After passing though the beam splitter the radiation will bounce off two mirrors prior to reaching the sample compartment. Depending on the sample accessory being used the radiation at this point may bounce off more mirrors prior to reaching the sample, as is the case with the ATR used in this study, or go directly to the sample and then the detector after bouncing off the detector mirror if using a potassium bromide pellet.

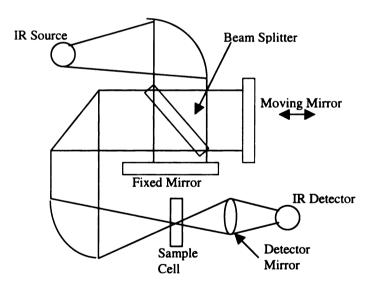


Figure 1. A schematic drawing of the FT-IR used in this study with major parts labeled.

As shown in figure 2 the ZnSe focusing element of the ATR crystal allows the IR beam to be focused on and penetrate the sample numerous times, in this case nine times, by reflecting off the internal part of the crystal before being detected. This multiple

reflection technique produces a spectrum that has less background noise and interference than that of a crystal with fewer penetrations into the sample.

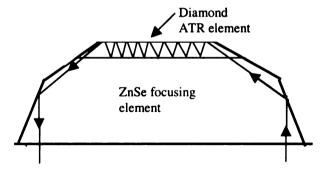


Figure 2. A schematic drawing of an ATR crystal with the IR beam reflecting through the crystal and into the sample.

An infrared spectrophotometer aids the forensic scientist in identifying the adhesive or backing of the tape by being able to differentiate one substance from another. Different items will vary in the amount of IR radiation the sample being analyzed, either the adhesive or backing, absorbs and the frequency at which the molecules in the sample vibrate at various wavelengths. Differentiation can occur because no two substances absorb IR radiation at the same frequency, therefore each substance produces a unique IR spectrum. In this study a UV light source and a 100-watt incandescent light bulb were used to artificially age both the backing and adhesive sides of 27 office tapes and 24 packaging tapes.

Classification and Manufacturing of Adhesive Tapes

Adhesives can be divided into two general categories: natural and synthetic. Natural adhesives include any adhesive made from animal or plant sources. Synthetic adhesives are adhesives that are made from any other source.

Natural adhesives include glues made from albumen, casein, shellac, beeswax, fish byproducts, etc. These natural adhesives do not bond as strongly as synthetic adhesives and therefore are used in applications where a minimal amount of stress is placed on the adhesive. Some of the uses for these adhesives are the bonding of light materials such as paper, foil and light wood.

Examples of synthetic adhesives are man-made polymers and other manufactured adhesives. Synthetic adhesives can be further subdivided into four categories depending on the properties of the chemicals in the adhesive. Those categories are thermoplastic, thermosetting, elastomeric, and alloys. Thermoplastic adhesives are generally made up of alcohol, polyvinyl, polyvinyl acetate and some acrylics. The thermoplastic adhesives do not cross-link upon curing, thus allowing these adhesives to be re-softened if necessary. Thermosetting adhesives cross-link upon curing and therefore cannot be re-softened once the adhesive is totally cured. Some examples of thermosetting adhesives are epoxies, polyesters and cyanoacrylates, which is better known as "super glue." Elastomeric adhesives possess properties such as elongation, strength and flexibility properties and are tough. This subdivision of the synthetic adhesive category includes items such as silicones, styrene-butadiene rubbers and

neoprene. Alloys encompass adhesives made from a combination of any of the other three previously mentioned synthetic adhesive subdivisions.

While the above system of classification is recognized by many researchers working with adhesives, there is no universal system. Other classifications such as physical form, chemical composition, or means of application for classifying adhesives while others classify tapes by either their construction or their use, are also used. Tapes that are classified by their construction are almost exclusively differentiated by what materials make up the backing such as cloth, paper and PVC. Tapes that are classified by their use are differentiated by the purpose of the tape. Some examples of these include office tapes, packaging tapes, painter's tape and duct tapes.

Tape backings also fall into different categories. Backings are made from materials such as plastic, paper, PVC and cloth. The two most popular materials for backings are cellophane and polypropylene. Cellophane is a form of regenerated cellulose and is an important type of backing. It is a transparent, thermosetting film and has excellent strength and clarity yet can be torn easily. However, this backing is being replaced in some applications by cellulose acetate because of its superior resistance to moisture, oils, greases and ultraviolet light (2). Cellulose acetate is used primarily in clear office tapes, while packaging tapes use primarily polypropylene for their backing. This is because polypropylene has a greater strength and is more resistant to abrasion than cellulose acetate.

Along with these polymers, adhesive tape backings may also contain plasticizers, stabilizers, pigments, extenders and other additives. On the side of the backing that does not contain the adhesive a release coating is typically applied (5). This coating, as the

name implies, allows the adhesive to release from the backing so that as much of the adhesive as possible can be used to adhere to the surface on which the tape is being placed. The release layer also facilitates unwinding of the tape and prevents splitting of the adhesive layer.

Tape is generally produced in the same manner no matter what its final intended use. There are two phases to the production of tape: coating and slitting. The coating phase is the application of the adhesive to the backing and the slitting phase is the process of cutting the tape into rolls. Prior to these two phases of producing the final tape product, there are steps that need to be taken in order to get the backing and adhesive prepared for application of one to the other.

The first step in the coating process is preparing the adhesive. At the start of the process the adhesive is in a concentrated bulk state and must be thinned down so it is less viscous and can be applied to the backing. There are three methods of making the adhesive less viscous: water based coating, hot melt coating and solvent coating. Water based coating and solvent coating are very similar because both us a solvent – either water or some other solvent – to reduce the bulk adhesive, which is in liquid form, to the proper consistency. Once the desired consistency is reached for the adhesive it is coated onto the backing and then both the adhesive and backing go through an oven that is heated. This heating process evaporates off the solvent and the final product remains. The hot melt process is different because the bulk adhesive is a solid instead of a liquid. Once the adhesive has reached the desired consistency it is coated onto the backing just as previously described. Because there is no solvent to evaporate, the tape does not pass through an oven, so once cooled it is the finished product.

The backing of the tape is prepared on the side opposite the adhesive so when the roll of tape is formed it does not stick to itself. The backing has a release coating applied to it which is often made of silicone (5). Along with making the tape easier to unwind the release coating allows the slitting process to be more efficient.

Once the coating process is complete the tape is on a large roll that is ready to be cut into smaller rolls. These smaller rolls can vary in length and width by changing settings on where the cutting blades are placed in relation to the blade next to it. In order to achieve the final product the tape is unwound off the large roll and onto a machine that has cutting blades. As the backing passes over the blades they cut the tape into the desired widths and lengths. Most of the tapes marketed today are produced in this general way.

While there are similarities in the manufacturing process different manufacturers make the backing of the tape from many different materials. Some examples that are used in backings are paper, cloth and cellulose. The backings of tape can be clear, colored or have design on them and their properties are selected by the manufacturer to allow the tape to function for a specific purpose. For example, electrical tape is made of a plastic or PVC backing that is designed to insulate electrical wires, while masking tape uses a backing that has a moisture resistant coating.

While adhesive composition is more variable than backing composition, it is still critical to the performance of the tape, since without the adhesive the tape would not serve its purpose. There are two types of adhesives used in pressure sensitive tapes: rubber or resins and acrylics. The rubber and resins are relatively inexpensive to produce

since they come from rubber trees and the oil industry, respectively, but do not perform as well as the acrylics, particularly in relation to exposure to light, heat or aging.

Acrylics are synthetic polymers and have excellent aging characteristics and very strong tack. In addition, acrylics also are resistant to hydrolysis and have good ultraviolet stability. Acrylics are more expensive to manufacture, but when high standards of performance are needed the manufactures turn to acrylic rather than rubber or resin adhesives (5).

The components that make up the adhesive in tape are continuously being updated and changed by the manufacturers (6). Factors such as the need to reduce the cost, improve the quality or availability of products are reasons for such changes.

Review of Literature

In the past, two techniques were commonly to use in the analysis of tape adhesives: pyrolysis gas chromatography and infrared (IR) spectroscopy. These are reliable techniques and both can determine whether a sample could have come from a particular roll or not (7). Pyrolysis gas chromatography is a well established technique that uses heat to decompose small samples in the absence of air, into gaseous products which are then separated by a gas chromatograph.

Fourier transform infrared (FT-IR) spectrophotometry has been the most utilized method of analyzing adhesive tapes. Each substance that adsorbs IR radiation does so at its own frequencies resulting in a unique IR adsorption spectrum of each substance analyzed.

Today many forensic laboratories have IR spectrophotometers, most of which are FT-IR spectrophotometers. These spectrophotometers are much more efficient and cost significantly less than the previous IR spectrophotometers, allowing numerous laboratories to have this instrument at their disposal.

Clear pressure sensitive office tape was analyzed by M. Sakayanagi et al. using an attenuated total reflectance (ATR) accessory. He found that adhesive tapes use either a rubber-based pressure sensitive adhesive or an acrylic-based pressure sensitive adhesive (4). These tapes were found to contain enough different components that they could be differentiated by manufacturer, and furthermore, into various categories from the same manufacturer.

Maynard et al. did a study where they collected data relating to many different types of pressure sensitive adhesive tapes, including clear office tape (2). They collected spectra from 27 different clear pressure sensitive adhesive tapes and found they could be classified into 5 different categories based on variations in the adhesive each utilized. Although it has been noted that manufacturers change their formulation of adhesives over time, none of these studies noted how the tapes change over time (2).

Numerous studies have been performed to determine suitable techniques of collecting IR spectra from various adhesives and tape backings. Among the techniques tested were the use of a diamond anvil cell accessory, Potassium Bromide (KBr) pellets, diffuse reflectance infrared spectrophotometry and internal reflectance infrared spectrophotometry, also referred to as ATR (3, 8, 9, 10, 11, 12). Research indicates that both ATR and a diamond cell accessory are effective methods for obtaining spectra of these samples (3, 11, 12). This is because the sample size could be small, the sample requires little preparation and cleaning the sampling accessory is uncomplicated.

There have been no studies reported in the literature on aging of adhesive tapes. Some analogy can be drawn to studies involving the artificial aging of inks, which employ exposure to ultraviolet and visible light, using both natural and artificial sources (13, 14). One of these studies utilized UV light for artificially accelerating the aging process of the ink. In that study lines were drawn on paper to obtain samples of ink from the pen. These samples were subsequently irradiated with a UV light source that was approximately five and one half inches above the samples (13). Following the irradiation by the UV light for a set amount of time the sample was analyzed and the process repeated.

Materials and Methods

The diamond compression cell ATR method was chosen because it proved to be less time consuming than placing the adhesive or backing on a KBr pellet or using some other transmission method. This is because the amount of adhesive removed was small, sometimes hard to remove, and did not spread across surfaces easily. Also, all of the adhesive would need to be removed from the backing, which would be difficult to do without tearing the backing, to obtain a quality spectrum of the backing alone. Finally, cost and sample size would not be a factor because small samples can easily be analyzed. In this instance ATR spectra have an advantage over transmission spectra because the ATR allows for a smaller sample of the item to be used and negates the problem of removing all of the adhesive and trying to spread it on a surface.

Following the selection of the sampling technique, samples from various adhesives and backings were collected. Fifty-one individual samples of commercially available tapes were collected prior to the aging the tapes. These tape samples were obtained from hardware stores, grocery stores, convenience stores, pharmacies and office supply stores in the greater Toledo, Ohio area. A complete list of these tapes can be found in Table 1, while their physical description is in Table 2.

Two types of tape were used in this study: office tape and packaging tape. Office tapes are generally colorless and have a clear cellophane backing and all 27 office tapes in this study were colorless. In contrast, packaging tape backings often come in colors, typically tan, but some come in clear as well. Of the 24 packaging tapes in this study,

two were tan and 22 were clear. All of the packaging tapes were wider than the office tapes, since they are used to hold together larger items. Given that the two types of tape

Table 1. IR Changes with Age for 51 Packaging and Office Tapes

Sample	Common Name	Classification	Adhesive Changes	Backing Changes
1	3m Transparent Tape	Office	No	No
2	Ace Invisible Tape	Office	No	No
3	Ace Transparent Tape	Office	No	No
4	Annapolis Clear Tape	Office	No	No
5	Annapolis Invisible Tape	Office	No	No
6	Big Lots Invisible Value Tape*	Office	No	Yes, 100W bulb
7	Duck No More Scissors Tape	Office	No	No
8	Duck One Touch Tape	Office	No	No
9	Elmer's Invisible Tape	Office	No	No
10	Elmer's Ultra Clear Tape	Office	No	No
11	Highland Transparent Tape	Office	No	Yes, 100W bulb
12	LePages Transparent Tape	Office	No	Yes, 100W bulb
13	Office Max Invisible Tape	Office	No	Yes, 100W bulb
14	Office Works Invisible Tape	Office	No	Yes, 100W bulb
15	Office Works Transparent Tape	Office	No	Yes, 100W bulb
16	Rite Aid Gift Wrap It Tape	Office	Yes, 100W bulb	No
17	Rite Aid Invisible Tape*	Office	No	No
18	Scotch Gift Wrap Tape	Office	No	No
19	Scotch Glossy Tape	Office	No	No
20	Scotch Magic Tape	Office	No	No
21	Scotch MultiTask Tape	Office	No	No
22	Scotch Removable Tape*	Office	No	Yes, 100W bulb
23	Scotch Transparent Tape	Office	No	No
24	Target Invisible Tape*	Office	No	No
25	Walgreens Invisible Tape	Office	No	No
26	Walgreens Transparent Tape*	Office	No	No
27	Work.org Invisible Tape ⁺	Office	No	Yes, 100W bulb
28	3m Packaging Tape ⁺	Packaging	Yes, 100W bulb	No
29	Annapolis Mailing Tape	Packaging	No	No
30	Duck EZ Start Tape*	Packaging	No	No
31	Duck Super EZ Tape	Packaging	No	No
32	Office Depot Strapping Tape	Packaging	Yes, 100W bulb & UV	No
33	Office Max Mailing Tape	Packaging	No	No
34	Office Max Strapping Tape	Packaging	No	No
35	Office Max Tan Mailing Tape	Packaging	No	No

36	Office Works Clear Mailing Tape*	Packaging	Yes, UV	No
37	Office Works Strapping Tape	Packaging	Yes, UV	No
38	Quik Stik Packaging Tape	Packaging	Yes, UV	No
39	Quik Stik Strapping Tape	Packaging	Yes, UV	No
40	Quik Tear Packaging Tape*	Packaging	No	No
41	Scotch Clear Box Sealing Tape ⁺	Packaging	Yes, UV	No
42	Scotch Packaging Tape*	Packaging	Yes, UV	No
43	Scotch Storage Tape	Packaging	No	No
44	Scotch Strapping Tape	Packaging	No	No
45	Scotch Stretchy Tape	Packaging	Yes, UV	No
46	Scotch Super Strength Packaging Tape ⁺	Packaging	Yes, UV	No
47	Scotch Tear-by-Hand Tape	Packaging	Yes, UV	No
48	Scotch Transparent Duct Tape	Packaging	No	No
49	Target Packaging Tape ⁺	Packaging	Yes, 100W bulb & UV	No
50	USPS Packaging Tape*	Packaging	No	No
51	Walgreens High Performance Packaging Tape	Packaging	No	No
	ese tapes were aged outside for seven days ese tapes were re-aged and re-analyzed			

Table 2. Physical description of 51 Packaging and Office Tapes

Sample	Common Name	Classification	Width (in inches)	Color
1	3m Transparent Tape	Office	1/2	Clear
2	Ace Invisible Tape	Office	1/2	Clear
3	Ace Transparent Tape	Office	1/2	Clear
4	Annapolis Clear Tape	Office	3/4	Clear
5	Annapolis Invisible Tape	Office	3/4	Clear
6	Big Lots Invisible Value Tape	Office	1/2	Clear
7	Duck No More Scissors Tape	Office	3/4	Clear
8	Duck One Touch Tape	Office	3/4	Clear
9	Elmer's Invisible Tape	Office	3/4	Clear
10	Elmer's Ultra Clear Tape	Office	3/4	Clear
11	Highland Transparent Tape	Office	3/4	Clear
12	LePages Transparent Tape	Office	1/2	Clear
13	Office Max Invisible Tape	Office	3/4	Clear
14	Office Works Invisible Tape	Office	1/2	Clear
15	Office Works Transparent Tape	Office	1/2	Clear
16	Rite Aid Gift Wrap It Tape	Office	3/4	Clear
17	Rite Aid Invisible Tape	Office	1/2	Clear
18	Scotch Gift Wrap Tape	Office	3/4	Clear
19	Scotch Glossy Tape	Office	3/4	Clear
20	Scotch Magic Tape	Office	3/4	Clear
21	Scotch MultiTask Tape	Office	3/4	Clear
22	Scotch Removable Tape	Office	3/4	Clear
23	Scotch Transparent Tape	Office	3/4	Clear
24	Target Invisible Tape	Office	3/4	Clear

25	Walgreens Invisible Tape	Office	1/2	Clear
26	Walgreens Transparent Tape	Office	1/2	Clear
27	Work.org Invisible Tape	Office	3/4	Clear
28	3m Packaging Tape	Packaging	1.88	Clear
29	Annapolis Mailing Tape	Packaging	2	Clear
30	Duck EZ Start Tape	Packaging	1.88	Clear
31	Duck Super EZ Tape	Packaging	1.88	Clear
32	Office Depot Strapping Tape	Packaging	1.88	Clear
33	Office Max Mailing Tape	Packaging	1.88	Clear
34	Office Max Strapping Tape	Packaging	1.88	Clear
35	Office Max Tan Mailing Tape	Packaging	1.88	Tan
36	Office Works Clear Mailing Tape	Packaging	1.88	Clear
37	Office Works Strapping Tape	Packaging	1.88	Clear
38	Quik Stik Packaging Tape	Packaging	1.88	Clear
39	Quik Stik Strapping Tape	Packaging	1.88	Clear
40	Quik Tear Packaging Tape	Packaging	1.88	Clear
41	Scotch Clear Box Sealing Tape	Packaging	1.88	Clear
42	Scotch Packaging Tape	Packaging	1.88	Clear
43	Scotch Storage Tape	Packaging	1.88	Clear
44	Scotch Strapping Tape	Packaging	2	Clear
45	Scotch Stretchy Tape	Packaging	1.88	Clear
46	Scotch Super Strength Packaging Tape	Packaging	2	Tan
47	Scotch Tear-by-Hand Tape	Packaging	2	Clear
48	Scotch Transparent Duct Tape	Packaging	1.43	Clear
49	Target Packaging Tape	Packaging	2	Clear
50	USPS Packaging Tape	Packaging	2	Clear
51	Walgreens High Performance Packaging Tape		1.88	Clear

have different uses it was assumed that the backings would be different materials. However, the material from which the backing was made was not able to be determined when compared to both spectra obtained in this study and known spectral databases.

The Fourier transform infrared (FT-IR) spectrophotometer used to collect the infrared spectra of both the backings and adhesives was a DigiLab Excalibur Series FT-IR spectrophotometer (Randolph, Massachusetts). The spectrophotometer is equipped with a deuterated triglycine sulfate (DTGS) detector and the diamond compression cell accessory (SensIR Technologies, Danbury, CT). The software used to analyze the spectra was Resolutions Pro, also a product of DigiLab. The aging of the tapes was

accomplished using a 100W incandescent light bulb as well as a Spectroline CX-20 UV light source (Westbury, NY).

Prior to irradiation, spectra were collected from both the adhesive and backing of the unaged tape samples. Then adhesive and backing samples were irradiated under a 100W incandescent light bulb and a UV light source, set to 254nm, for 20, 40, 50, 70 and 90 hours. The backings and adhesives were irradiated separately by placing the tape on a piece of paper with the part of the tape being studied facing the light source which was raised six centimeters above the samples. During the aging and analysis process the backing was peeled off, analyzed and then replaced on the paper for further aging. An additional step to allow irradiation of the adhesive was tacking the tape down on each end with a small piece of tape to ensure the tape did not curl or wind back onto itself.

The proximity of the light source to the adhesive and backing being aged was such that all of the adhesive would be exposed to the irradiation since it is approximately 40µm thick. The thickness of the backing was not determined because its removal was not needed for analysis to occur. To determine the approximate temperature these tapes were exposed to during the artificial aging process a thermometer was placed beside the tapes, directly under the light source. Following obtaining the temperature reading, approximately 62^oC, an oven was set at that temperature and one tape was placed in an oven for 90 hours to determine if the changes observed were from light or temperature. It was determined that the changes came from the light, as the tape heated in the oven had no spectral changes.

After irradiation for the given times spectra were collected. Even though time passed between when the exposure to the irradiation ended and the last spectrum was

obtained, this delay would be negligible because of the effects of irradiation on the tape. Also, any light that might reach the samples after artificial aging would not be as intense since it would be coming from a source considerably further than six centimeters away.

Ten tapes were placed outdoors and allowed to age for a week and a whole roll of tape was analyzed in ten foot increments. Spectra of these increments were collected prior to artificial aging and the aged 90 hours to determine if changes were consistent throughout the roll. Table 3 lists the conditions for the outdoor aged tapes. The IR spectra of both the adhesive and backing of these tapes were then collected. Table 1 indicates which tapes had changes in their spectra following the artificial aging as well as their classification as either office tape or packaging tape, determined from their end use. The spectra of the unaged tapes are in Appendix A; Appendix B contains the spectra of the tapes that exhibited

Day	High Temperature (°F)	Low Temperature (°F)	Other Conditions
1	33	22	Overcast, light snow (trace)
2	36	16	Overcast
3	37	25	Overcast
4	32	26	Light freezing rain, light snow (0.5 inches)
5	29	24	Light snow (0.3 inches)
6	33	20	Partly Sunny
7	38	20	Partly Sunny

Table 3. Weather Conditions for Outside Aging of Tapes

changes, in the following order: backing changes with a 100W bulb, adhesive changes with a 100W bulb and adhesive changes with the ultraviolet light. Prior to each aged sample is an unaged sample for comparison.

The instrument was calibrated daily using a polypropylene film. Before each spectrum was collected a background spectrum was taken with the top to the instrument

open, due to the size of the diamond compression accessory. Twenty scans from 4000 cm^{-1} to 600 cm^{-1} at five kHz and a resolution of 4 were taken of the background and each tape sample.

To prepare the adhesive samples for analysis they were carefully removed from the outermost and middle part of a one-centimeter square region of the backing using a scalpel and forceps. The area the adhesive was removed from was determined by observing that there was still tackiness on the backing following the removal of some of the adhesive. Following this removal the adhesive was worked into a ball, approximately the size of the head of a pin, weighing about 0.8 mg.

The removal of the adhesive from the backing was necessary because when the adhesive was placed on the diamond compression cell, while still attached to the backing, and sampled the adhesive was too thin to obtain a good spectrum. The adhesive sample was then removed from the backing and placed on the diamond compression cell with forceps and compressed, spreading the adhesive into a thin layer over the diamond cell. Following the collection of each spectrum the adhesive was removed from the diamond cell and the compression pin and both were cleaned with chloroform to remove any adhesive residue and allowed to dry for 5 minutes.

The backing required much less preparation than the adhesive since the adhesive did not need to be removed prior to obtaining a spectrum of the backing. The irradiated backing was removed from the paper on which it was placed to be exposed to the light source, a clean part of the backing was chosen, placed on the diamond compression cell and compressed. Multiple spectra were collected from the backing at various times of

aging to obtain the most consistent results. However, due to the small area sampled by the ATR accessory the exact area was not sampled each time.

Following the collection, spectra were compared. The unaged spectra from each make and type of tape were compared to the aged spectra of that tape. Each comparison was analyzed for differences between the unaged vs. the aged spectra as well as between the spectra that had been aged for different amounts of time. After these initial comparisons another four spectra were collected from a single piece of aged tape to determine if sampling different areas of this small piece of tape had any effects on the spectrum obtained.

Comparisons of the spectra were performed on the computer screen by overlaying one spectrum on top of the other. The spectra were determined to be different when peaks increased or decreased by more than one-half of their intensity, were totally eliminated or added, or where shoulders of peaks appeared or disappeared. See figures 1 and 2 for tapes considered the same and figures 3 and 4 for tapes considered different before and after aging. Tapes in which changes were observed were noted and a random sample of five of the tapes that changed were selected and artificially aged and analyzed again; see Table 1 for these tapes.

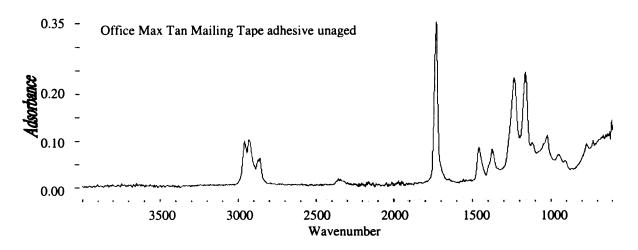


Figure 3. A sample spectrum of an unaged adhesive considered unchanged when compared to the aged adhesive.

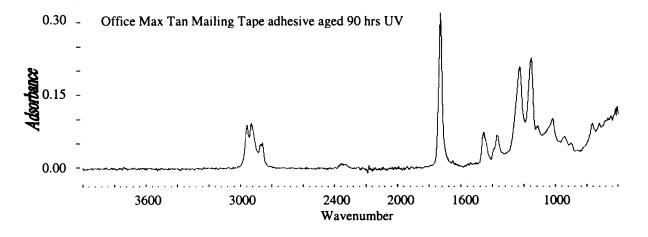


Figure 4. A sample spectrum of an aged adhesive considered unchanged when compared to the unaged adhesive.

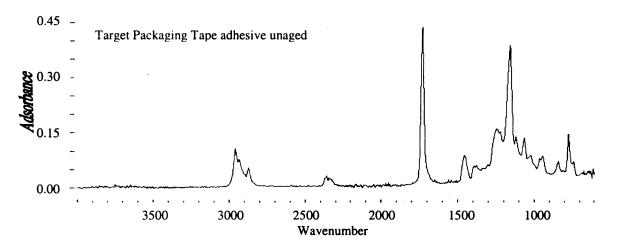


Figure 5. A sample spectrum of an unaged adhesive considered changed when compared to the aged adhesive.

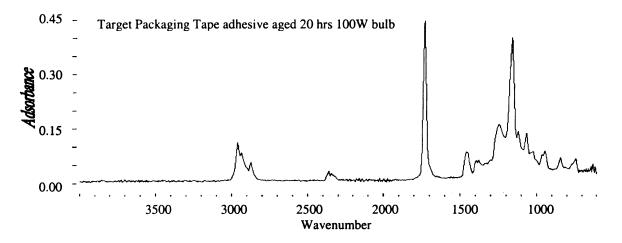


Figure 6. A sample spectrum of an aged adhesive considered to be changed between 800 cm^{-1} and 700 cm^{-1} when compared to the unaged adhesive.

Results and Discussion

Analysis and comparison of the unaged tapes showed that both the office and packaging tapes had similarities in peaks within the class being compared, no matter what brand was being analyzed. A comparison of the backing as described above yielded a similar conclusion.

As seen in Figures 5 and 6, both the office and packaging tape adhesives had a broad peak that occurs between 3000 cm⁻¹ and 2800 cm⁻¹, which occurs in all 51 of the tapes. This could be a grouping of three or four unresolved peaks which may be from the stretching of a polypropylene molecule. Another common, sharp, peak found in the adhesives, which is probably from a carbonyl group, occurred around 1730 cm⁻¹ in all 27 of the office tape spectra and in 17 out of 24 of the packaging tape spectra. Two other sharp peaks present in all of the office tape adhesive spectra as well as those of the packaging tape adhesive spectra are found around 1450 cm⁻¹ and 1375 cm⁻¹. These may also be from the stretching of carbon-hydrogen bonds in a polypropylene molecule.

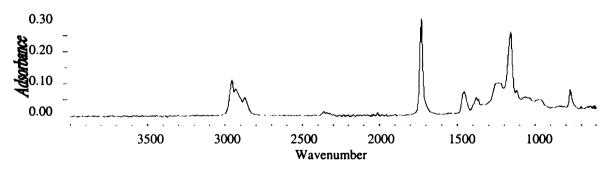


Figure 7. A sample spectrum of an office tape adhesive with broad peaks between 3000 cm^{-1} and 2800 cm^{-1} and peaks around 1730 cm^{-1} , 1450 cm^{-1} and 1375 cm^{-1} .

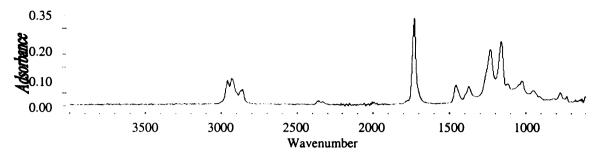


Figure 8. A sample spectrum of a packaging tape adhesive with broad peaks between 3000 cm^{-1} and 2800 cm^{-1} and peaks around 1730 cm^{-1} , 1450 cm^{-1} and 1375 cm^{-1} .

Just as office and packaging tape adhesives have similarities between them, the backings of these tapes do as well. The office tape backing spectra showed large, broad peaks between 3000 cm⁻¹ and 2800 cm⁻¹ in 21 out of 27 tapes and just as with the adhesives, these are probably from three or four different peaks that are unresolved (Figure 7). Six of the 27 office tape backings had peaks at this same range, but these were smaller and not as broad (Figure 8). There were peaks around 1730 cm⁻¹, and the group of two around 1450 cm⁻¹ and 1375 cm⁻¹, just as seen previously with the adhesives. These peaks were present in 22 and 20 out of the 27 office tape backing spectra, respectively.

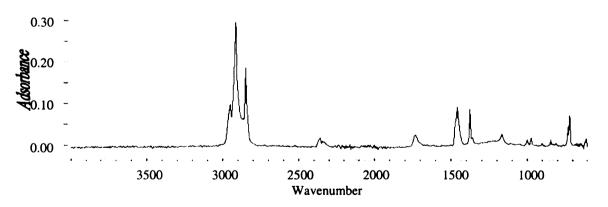


Figure 9. A sample spectrum of an office tape backing with large, broad peaks between 3000 cm^{-1} and 2800 cm^{-1} .

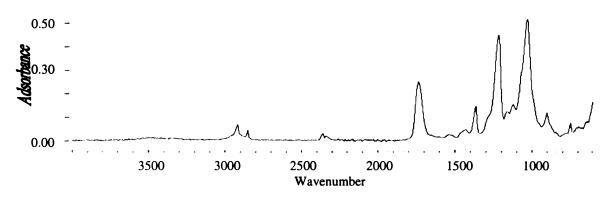


Figure 10. A sample spectrum of an office tape backing with smaller, less broad peaks between 3000 cm^{-1} and 2800 cm^{-1} .

There were two similarities in all 24 of the unaged packaging tape spectra analyzed. These were the large, broad peaks as seen in the other spectra between 3000 cm⁻¹ and 2800 cm⁻¹, and the two peaks around 1450 cm⁻¹ and 1375 cm⁻¹. There were 21 out of 24 packaging tape spectra that had three or four distinct peaks with the broad area; three out of 24 of them had two well defined peaks in this region and the two peaks around 1450 cm⁻¹ and 1375 cm⁻¹ occurred in all 24 and 20 out of the 24 packaging tape backings, respectfully (Figures 9 and 10).

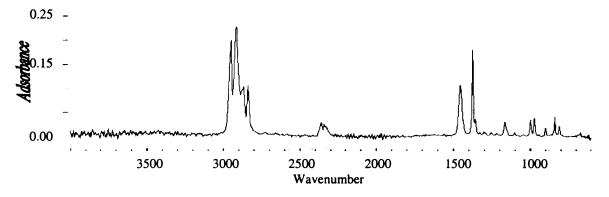


Figure 11. A sample spectrum of a packing tape backing with broad peaks between 3000 cm^{-1} and 2800 cm^{-1} and peaks around 1450 cm^{-1} and 1375 cm^{-1} .

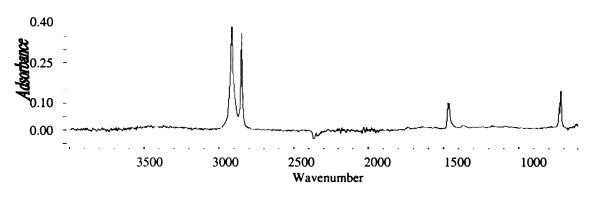


Figure 12. A sample spectrum of a packing tape backing with well defined peaks between 3000 cm^{-1} and 2800 cm^{-1} and one peak around 1450 cm^{-1} .

Even though many of the spectra were similar to each other they all varied in certain regions. All of the samples differed the most in the fingerprint region between 1400 cm^{-1} and 200 cm^{-1} (15). While this area of the spectrum contains much variation, due to numerous absorptions being present, the amount of variation is also the reason interpretation and determination of an exact molecule is difficult.

Following the artificial aging the aged and unaged spectra were compared and it was determined that the most significant changes were found in the fingerprint region in all of the backings and adhesives in which changes occurred. Neither the color nor the tackiness of the tape changed from what it was prior to the artificial aging process. In the office tapes eight out of 27 tapes had changes present in their backings and one out of 27 had a change in its adhesive when artificially aged with a 100W light bulb. No changes were seen for the backing of the packaging tape when artificially aged in this manner but it was able to be determined, based on spectral comparison to both known library databases and samples obtained in this study these backings were not cellulose acetate (16, 17). However, three of the 24 adhesives exhibited changes in their spectra. When artificially aged using ultraviolet light none of the office tapes exhibited any changes in

either their backing or adhesive spectra. While the backings of the 24 packaging tapes also showed no changed, 11 of the 24 packaging tapes had spectral changes in their adhesives using this light source. See Tables 4, 5 and 6 for these results. The ten tapes that were aged outside showed no changes in their spectra, while the tapes that were sampled in different places on the small piece and along the entire roll showed changes similar to those previously mentioned. The changes that were seen in the adhesives could be from bonds between parts of the molecules in the adhesive becoming weaker or even breaking as the tape ages.

Table 4. IR Changes in Backings Related to Time Aged with a 100-W bulb

Sample	Common Name	Classification	Spectral difference 20 vs. 0 hours	20	40	70 vs. 50 hours	70	0
Sample								
1	Big Lots Invisible Value Tape	Office	No	Yes	No	No	No	Yes
2	Highland Transparent Tape	Office	No	Yes	No	No	No	Yes
3	LePages Transparent Tape	Office	No	Yes	No	No	No	Yes
4	Office Max Invisible Tape	Office	No	Yes	No	No	No	Yes
5	Office Works Invisible Tape	Office	No	Yes	No	No	No	Yes
6	Office Works Transparent Tape	Office	No	Yes	No	No	No	Yes
7	Scotch Removable Tape	Office	No	Yes	No	No	No	Yes
8	Work.org Invisible Tape	Office	No	Yes	No	No	No	Yes

BACKING CHANGES - 100W bulb

Table 5. IR Changes in Adhesives Related to Time Aged with a 100-W bulb

Sample	Common Name	Classification	Spectral difference 20 vs. 0 hours	20	40	70 vs. 50 hours	70	0
1	Rite Aid Gift Wrap It Tape	Office	No	No	No	No	Yes	Yes
2	3m Packaging Tape	Packaging	Yes	No	No	No	No	Yes
3	Office Depot Strapping Tape	Packaging	Yes	No	No	No	No	Yes
4	Target Packaging Tape	Packaging	Yes	No	No	No	No	Yes

ADHESIVE CHANGES - 100W bulb

Table 6. IR Changes in Adhesives Related to Time Aged with a UV light

			Spectral difference 20 vs. 0	40 vs. 20	50 vs. 40	70 vs. 50	90 vs. 70	90 vs. 0
Sample	Common Name	Classification	hours	hours	hours	hours	hours	hours
1	Office Depot Strapping Tape	Packaging	Yes	No	No	No	No	Yes
2	Office Works Clear Mailing Tape	Packaging	No	No	No	No	Yes	Yes
3	Office Works Strapping Tape	Packaging	Yes	No	No	No	Yes	Yes
4	Quik Stik Packaging Tape	Packaging	No	No	Yes	Yes	No	Yes
5	Quik Stik Strapping Tape	Packaging	Yes	No	No	No	No	Yes
6	Scotch Clear Box Sealing Tape	Packaging	No	Yes	No	No	Yes	Yes
7	Scotch Packaging Tape	Packaging	No	No	No	No	Yes	Yes
8	Scotch Stretchy Tape	Packaging	No	No	No	No	Yes	Yes
9	Scotch Super Strength Packaging Tape	Packaging	No	No	No	No	Yes	Yes
10	Scotch Tear-by-Hand Tape	Packaging	Yes	No	No	Yes	No	Yes
11	Target Packaging Tape	Packaging	Yes	Yes	No	No	No	Yes

ADHESIVE CHANGES - UV

In the office tape backings many of the peaks in the fingerprint region that were present when the tapes were analyzed without aging were either significantly less in their intensities or missing all together. The peak just prior to the fingerprint region, around 1730 cm⁻¹, was still present in all but one brand of tape. However, in one brand but two different types of tape, it was significantly less intense than it was prior to the artificial aging process. Along with these changes, other changes occurred in the area where the broad bands were seen prior to aging. These changes, while occurring in all eight tapes in which the backing changed, had slightly different effects on the specific spectrum. In three of the tapes the bands were less intense and not as defined as they were prior to the artificial aging, while in the other five the peaks were more intense and more defined.

It was determined that the backing of the office tape was most likely cellulose acetate, which is usually resistant to UV light and other elements. However, since the aging of the backing occurred using a 100W incandescent light bulb this could be an explanation as to why the backing showed changes.

Even though with the 100W light bulb as an artificial aging source there were not as many office tape adhesives that changed when compared to office tape backings, some changes were noted. These occurred in one office tape adhesive as well as three packaging tape adhesives. In these four all but one of the changes were present in the previously mentioned fingerprint region of the spectrum. The lone change outside of that region dealt with the peak around 1730 cm⁻¹; it was present in the unaged spectrum and present but much less intense in the artificially aged spectrum. Three of the four spectra had changes between 800 cm⁻¹ and 700 cm⁻¹ while the other had additions and deletions of peaks between 1250 cm⁻¹ and 1025 cm⁻¹.

Just as with the adhesives mentioned above, the majority of the changes following artificial aging using the ultraviolet light source occurred in the fingerprint region as well. In this region six of the 11 tapes that had spectral changes gained broad unresolved peaks between 1350 cm⁻¹ and 925 cm⁻¹, while two tapes lost peaks in this region and two tapes exhibited similar changes that had previously been noted between 800 cm⁻¹ and 700 cm⁻¹. One final change that was observed was dealing with the previously mention peak around 1730 cm⁻¹. In this instance its intensity in two tapes diminished greatly, in one tape it intensified somewhat and in six others it was present post-artificial aging but not present prior to that.

The 12 adhesives that changed in their IR spectra were compared to spectral libraries and determined to be two different polymers, six of each. The polymers that made up most of the adhesives were most likely polyisobutylacrylate, which was determined to be on tapes such as Rite Aid Gift Wrap It Tape and 3M Packaging Tape, and poly-4-methyl-1-pentene, which was most likely on tapes such as Office Works

Mailing Tape and Scotch Clear Box Sealing Tape. Also based on spectral comparison, the changes in the 12 adhesives were most likely from partial evaporation of the solvent phenylglycidylether, which was still present in residual amounts on the adhesive after the manufacturing process (16, 17).

Adhesive on one tape, Quik Stik Packaging Tape, seemed to have erratic changes during the aging process. The original changes from unaged to aged 50 hours could be attributed solvent evaporation, whereas the changes from 50 to 70 hours of aging could be linked to the degradation of some of the bonds in the molecule after irradiation by UV light.

These results show that infrared changes due to artificial aging can occur when both an incandescent 100W light bulb and an ultraviolet light source are used. While some spectra changed others did not.

Conclusion

This study has shown that both the adhesive and backing of tapes can be characterized by infrared spectrophotometry and that aging of adhesives and backings could help determine if a questioned piece of tape could have come from a particular source. Also shown is that a forensic scientist must be careful when analyzing the infrared spectrum since changes in the tape originally used may cause that tape to be eliminated as the tape used years later. Because infrared spectroscopy measures only one chemical property of tapes, it would be sensible to employ other methods of analyses of the physical and chemical characteristics of the adhesive and backing prior to making a final conclusion in the comparison of known and unknown tapes. Maynard et al. used a well-rounded analytical scheme to analyze numerous chemical and physical characteristics of the tape (2). A scheme such as this, along with the infrared spectral data will provide the forensic scientist more information to possibly confirm the origin of the tape.

The spectra collected from the aged and unaged tape adhesives and backings indicate that the components of the tape change over time. Some spectra are similar between the different adhesives from different tapes, so caution must be used to be sure that the questioned tape is the same brand and type (i.e. Scotch Packaging Tape or Annapolis Clear Mailing Tape) as the brand and type being used for the known, unaged, sample. This may be possible if the questioned tape can be linked back to a suspect and some tape found at the residence or other place from which the item may have originated. However, if a known sample is not available it would be hard to determine the

manufacturer or type of tape from which the questioned item came based solely on IR spectra. Possible manufacturers may be able to be determined if other analytical tests are performed. A spectral library that includes both aged and unaged tapes could help narrow the options of manufacturers or types of tape that the questioned sample could be.

Other physical properties such as color and width should be compared prior to drawing a conclusion as to whether or not the tape could have come from the same source.

The collection of useful spectra from both backings and adhesives in this study confirmed the use of the diamond compression method as an effective and time saving method, just as previously cited studies had. Another advantage of this diamond compression method is that it is not necessary to use a microscope accessory to the FT-IR spectrophotometer. This will allow laboratories that have limited funds to still analyze tape adhesives and backings.

This study showed that some adhesive tapes show changes in their infrared spectra as they age. Since tapes that have aged naturally change color and lose their tack over time it would be conceivable that a change in coloration or tackiness of the tape would occur when artificially aged. However, the change is not as consistent as may have been previously thought, at least with the use of this artificial aging technique.

The diamond compression ATR method could be used as a sampling technique in future studies in which FT-IR spectrophotometers are used to look at different methods of aging tape. Since tapes that age naturally lose their tackiness and change color, some other experiments that could be done include allowing tape to age naturally in a variety of conditions, such as outside, inside and in direct sunlight. UV-visible

microspectrophotometry can also be employed to study color changes in these tapes as they age.

While one whole roll of tape was analyzed in this study, its focus was on only a small part of a whole roll. In future studies, valid conclusions could be determined about the consistency of tape should be determined by aging samples from more than one entire roll.

Another valuable study would be to purchase several rolls of tape of the same brand and type over a period of time. After doing this, these rolls could be analyzed to see how often the manufacturer makes changes to their adhesive formula and the variability between the formulas when changes are made. Also, a library of the aged adhesives could be created so it could be searched if a questioned aged sample is submitted to the laboratory, although further studies on variability would first be necessary to confirm reliability of such a database.

Studies similar to the previous three described could be done in relation to the backing of tapes as well. However, these changes may not be as prevalent because the composition of the backing varies less frequently than that of the adhesive and it seems that the backing for the most part, does not age when using this accelerated aging technique. Nonetheless, identification of the backing is important, because the more knowledge there is about a tape product, the greater the understanding of its evidentiary value. Therefore, studies of this type should be considered. If the tape changes frequently the greater the evidentiary value is and the more significant the product is to the investigation.

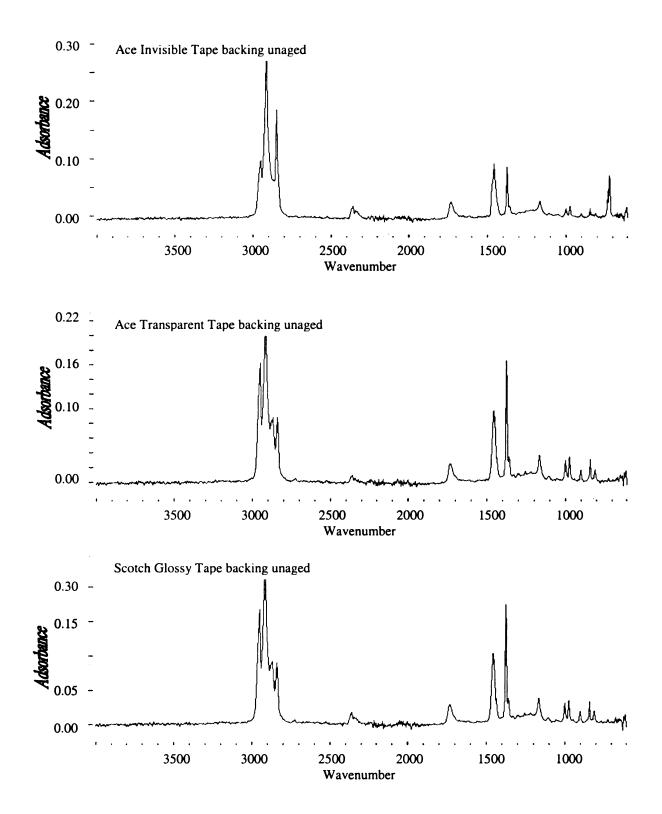
APPENDICES

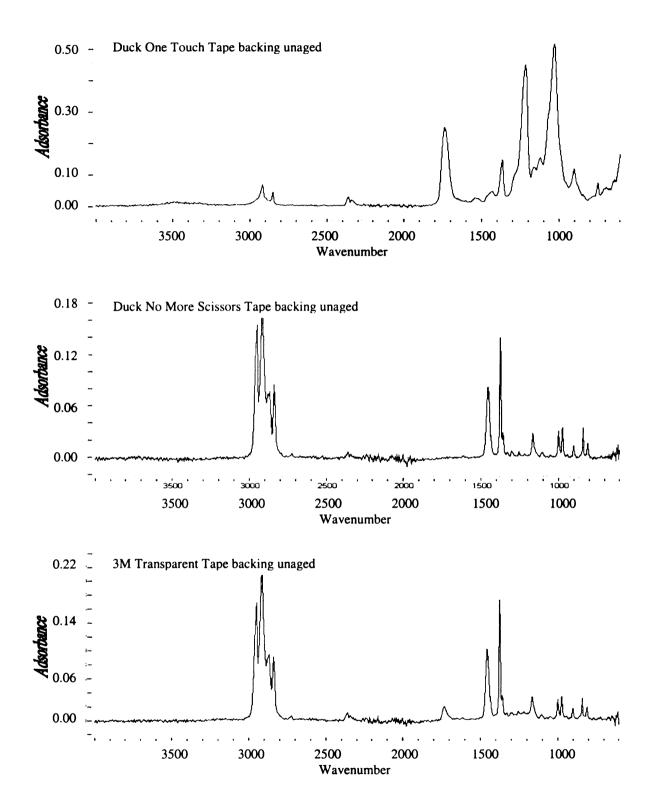
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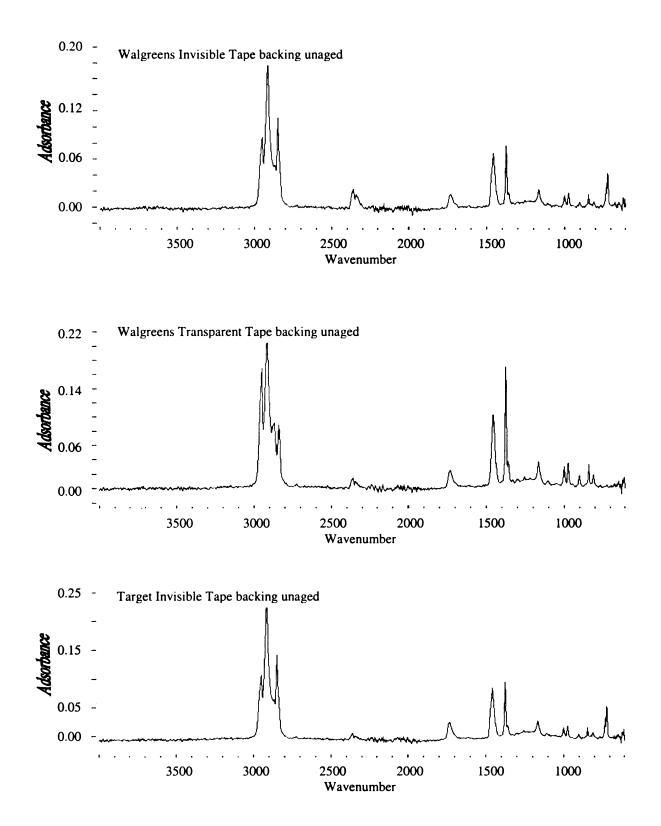
APPENDIX A

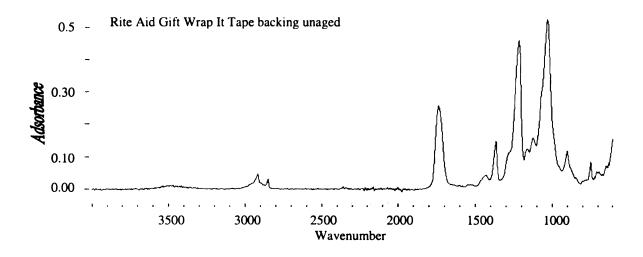
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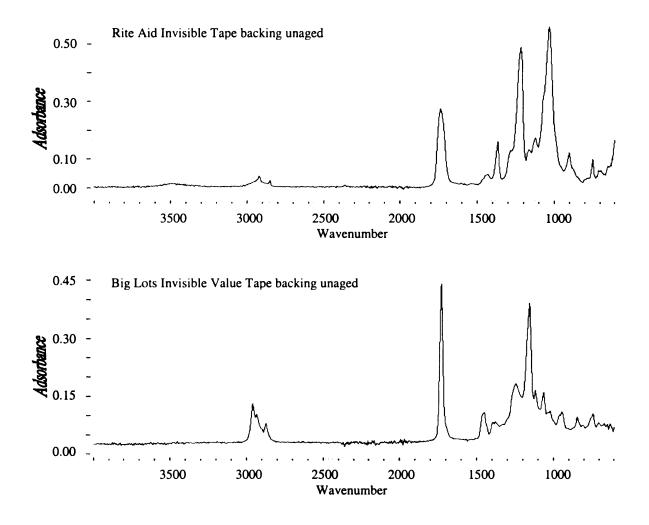
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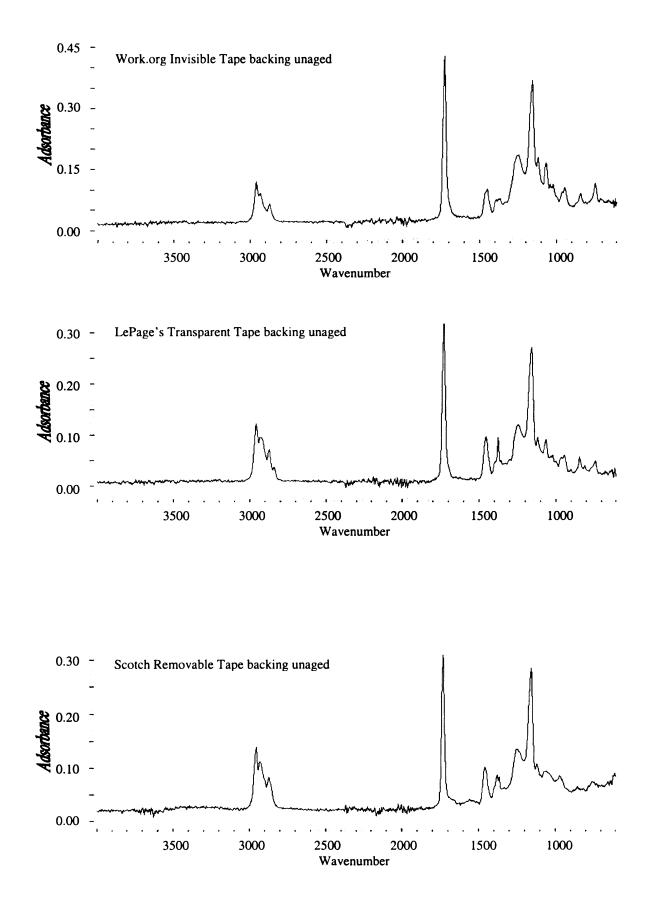


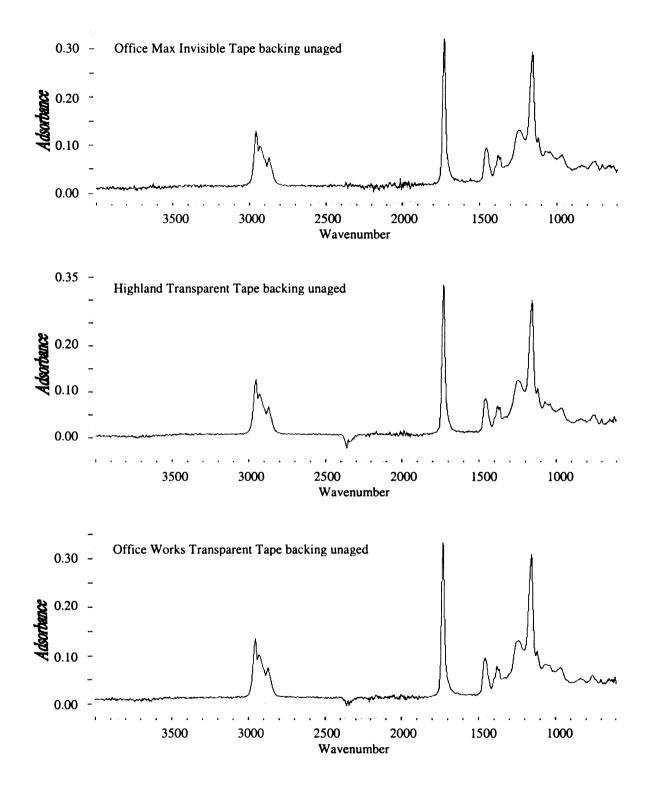


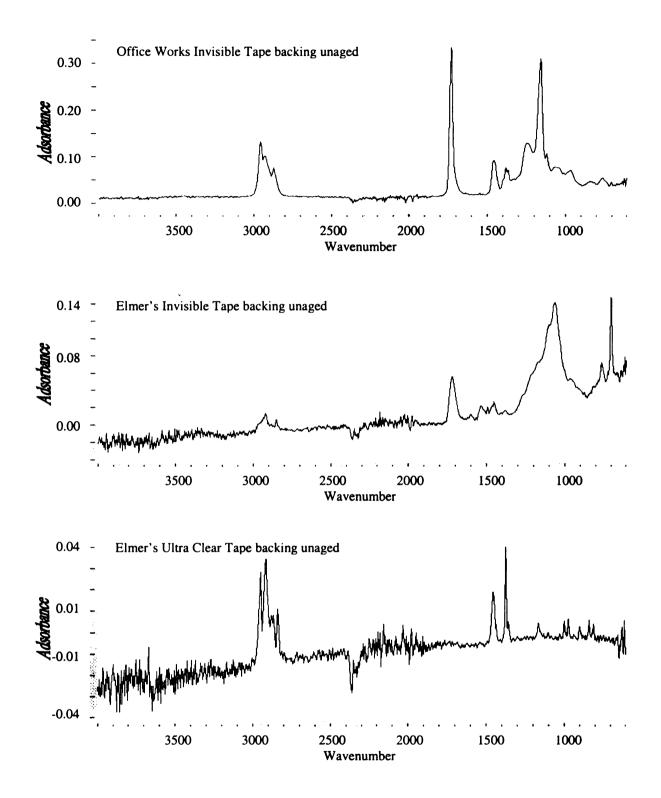


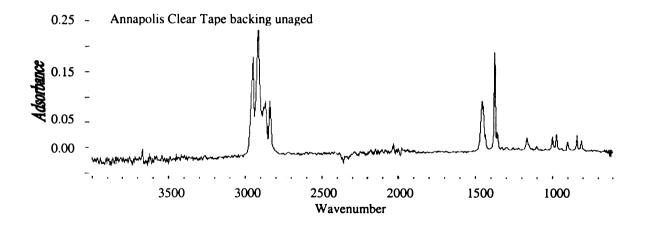


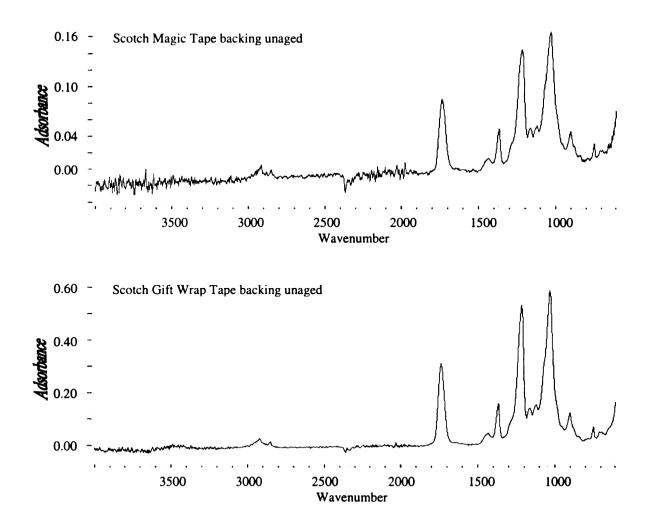


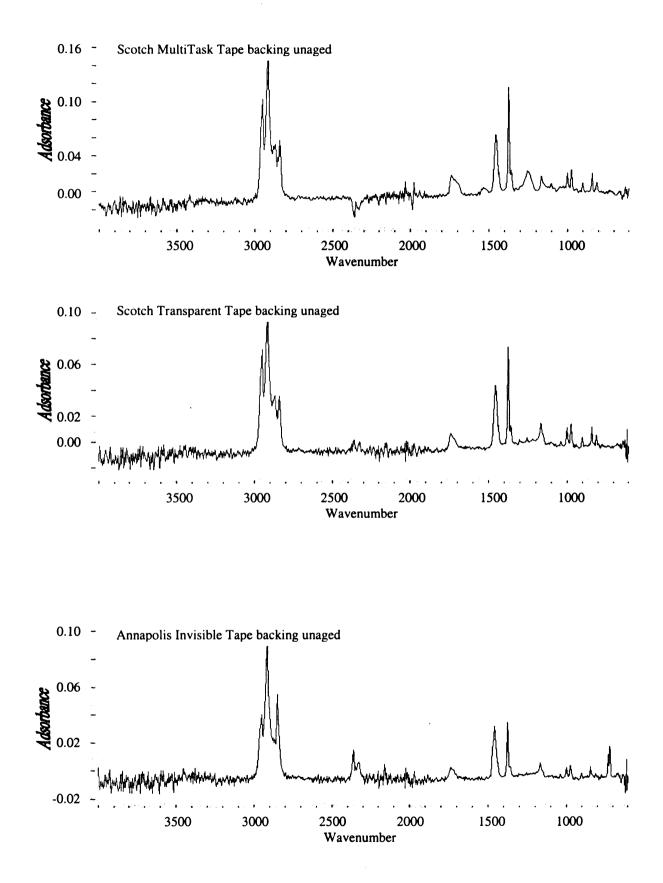


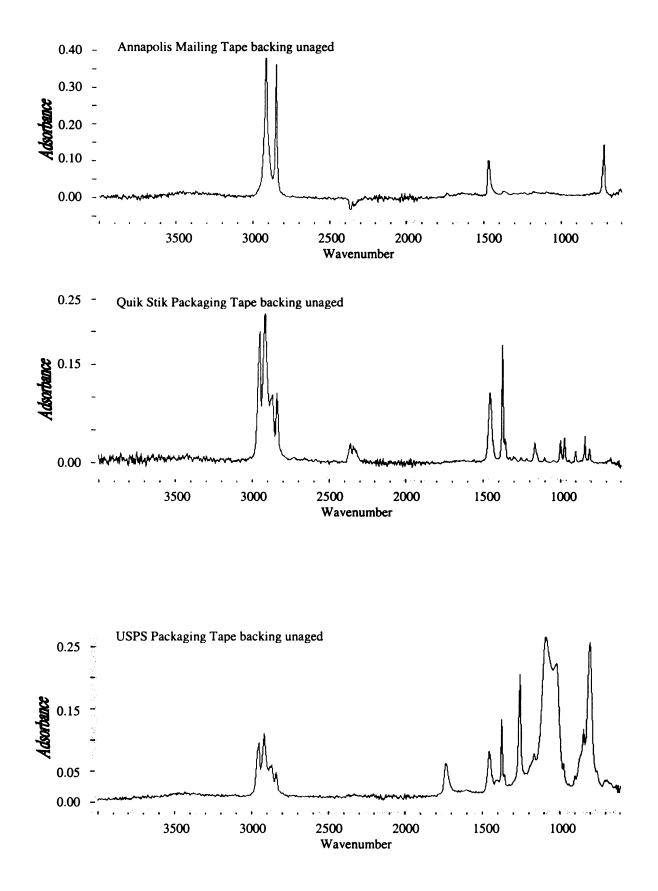


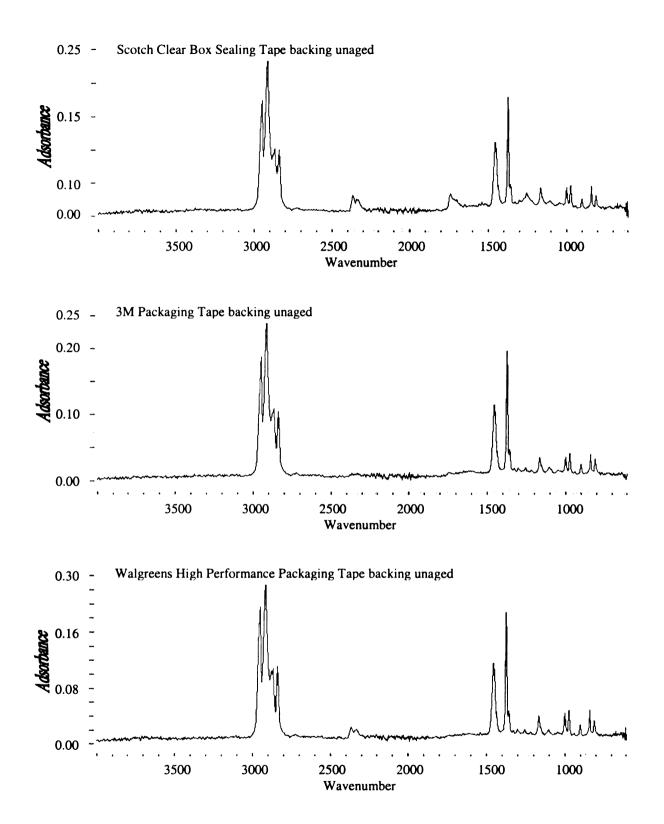


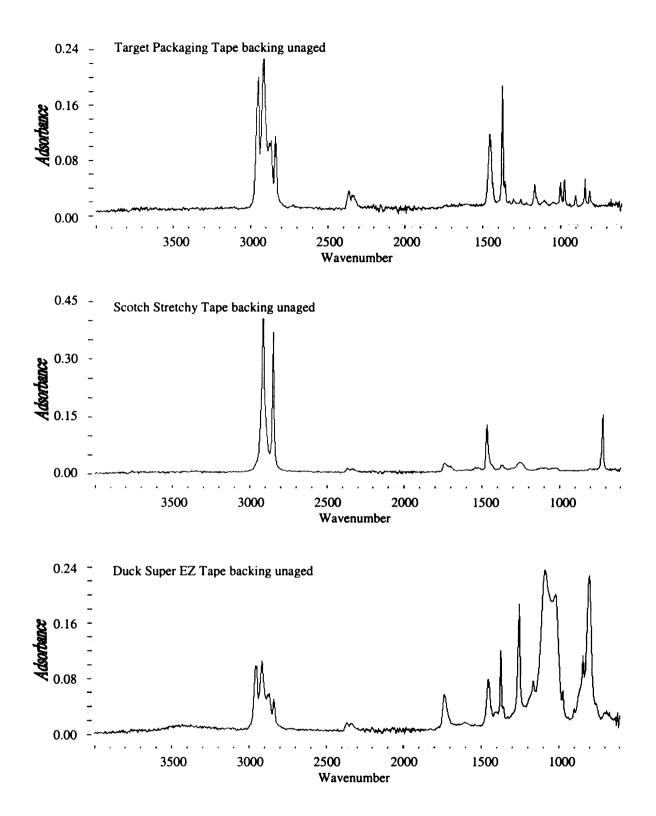


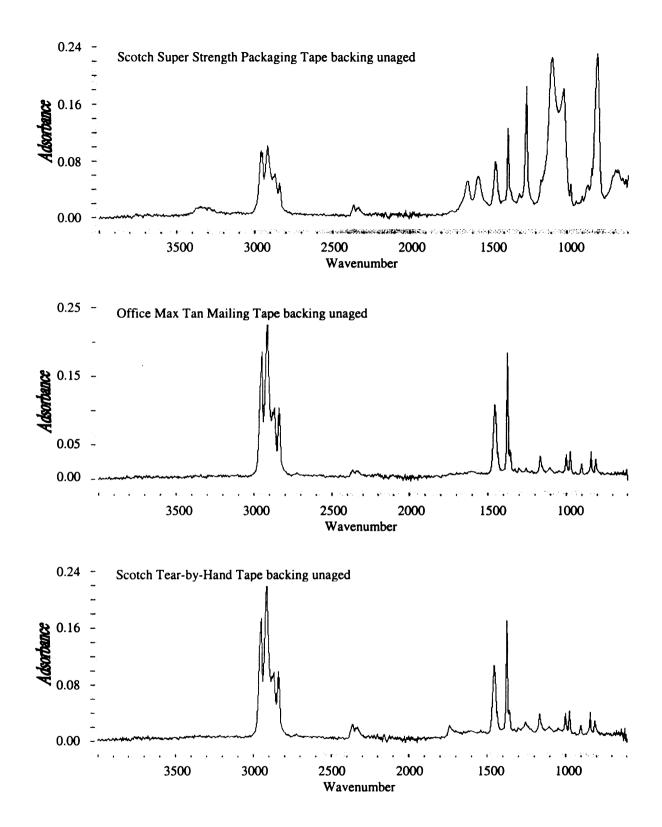


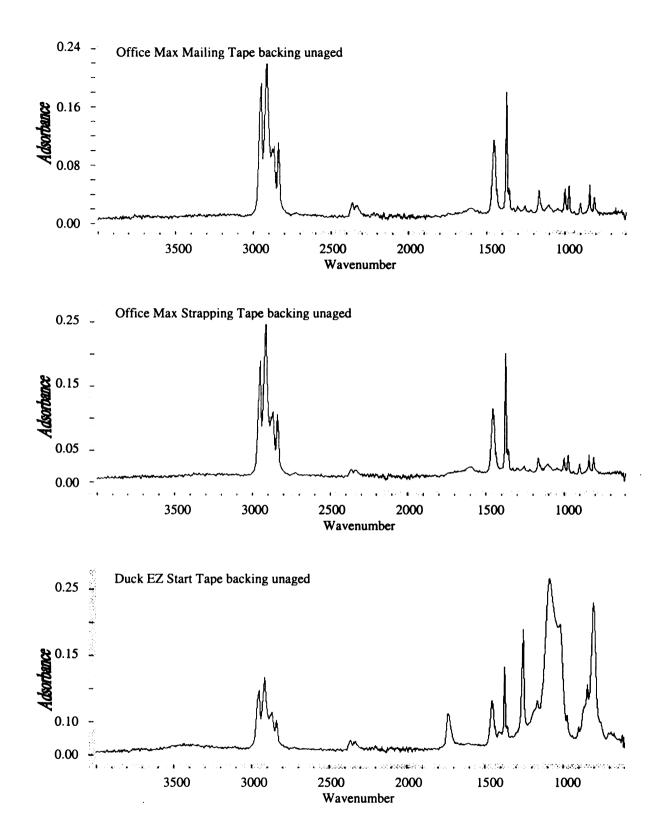


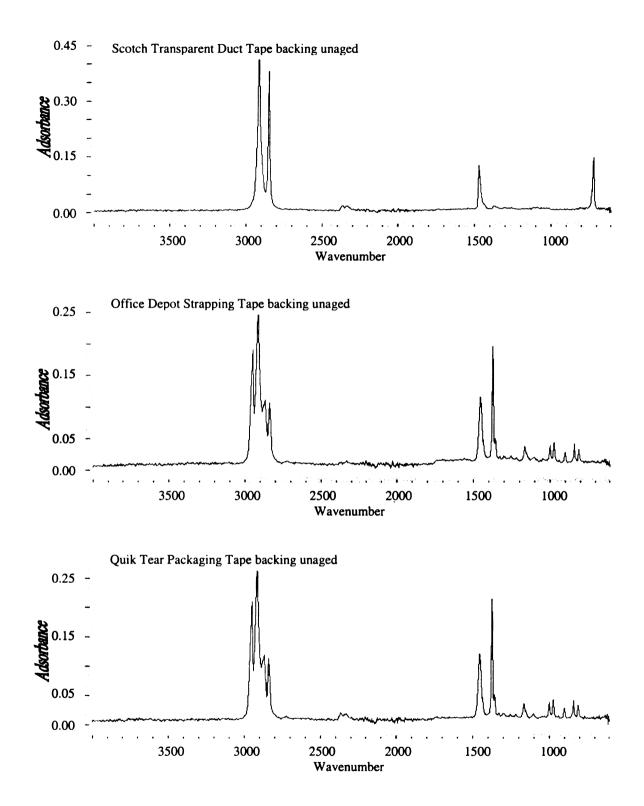


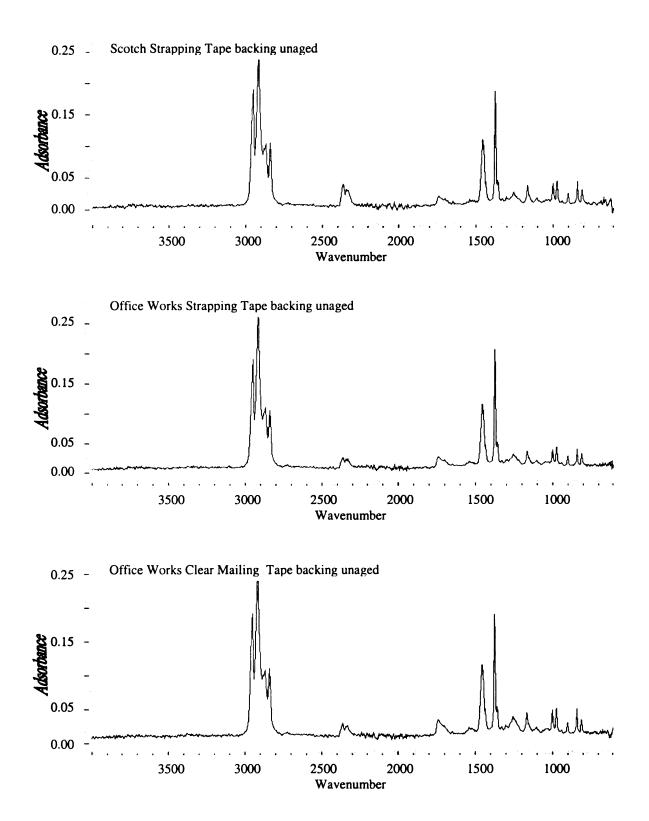


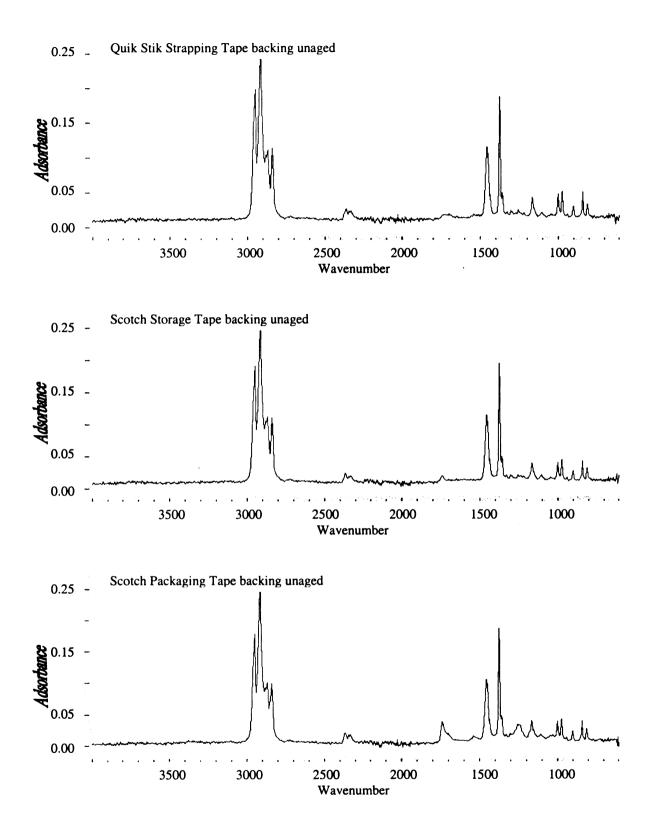


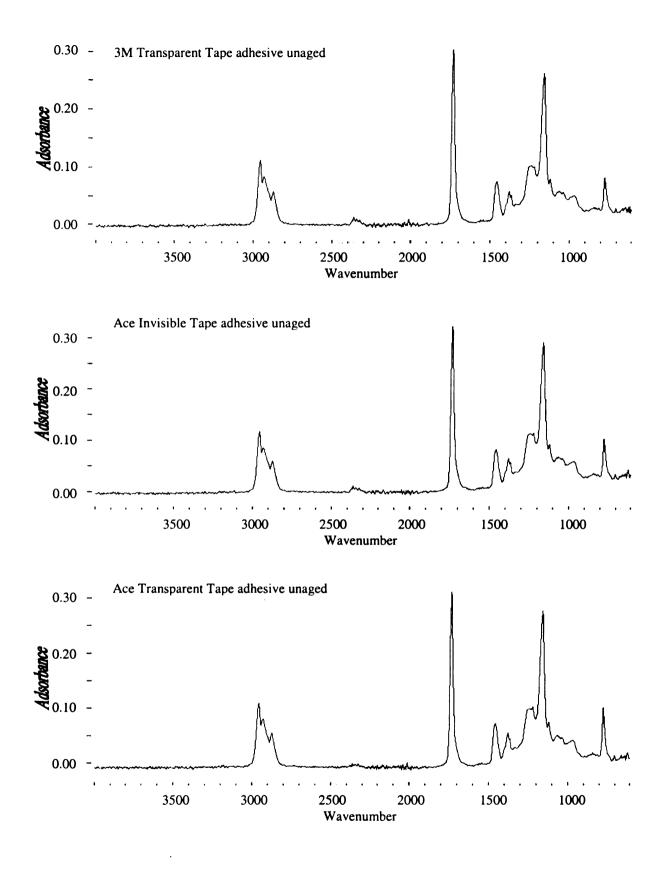


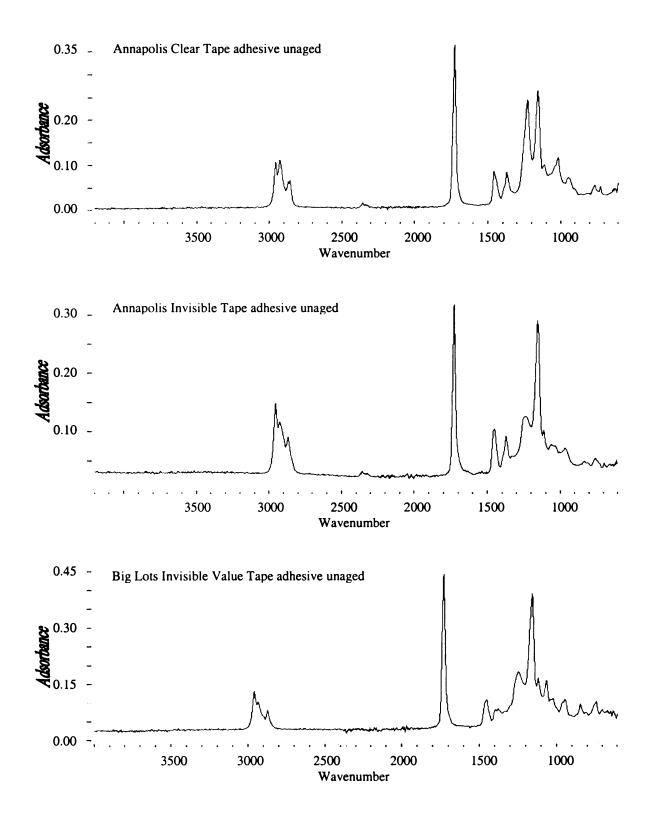




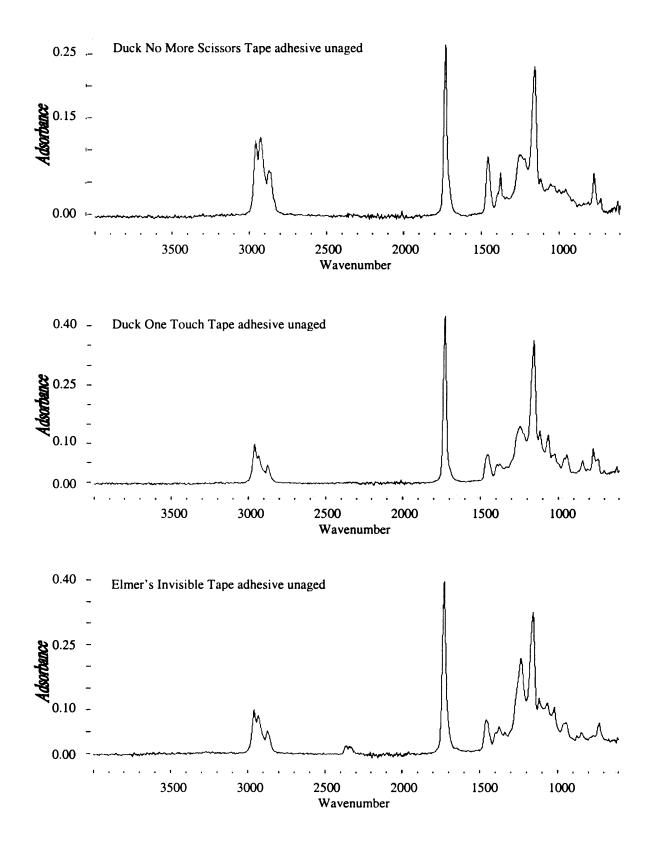


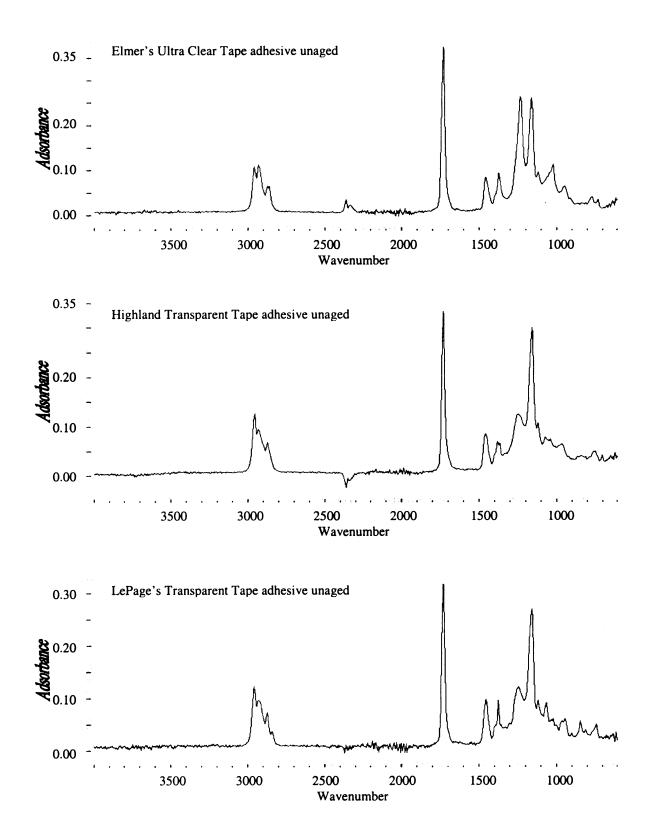


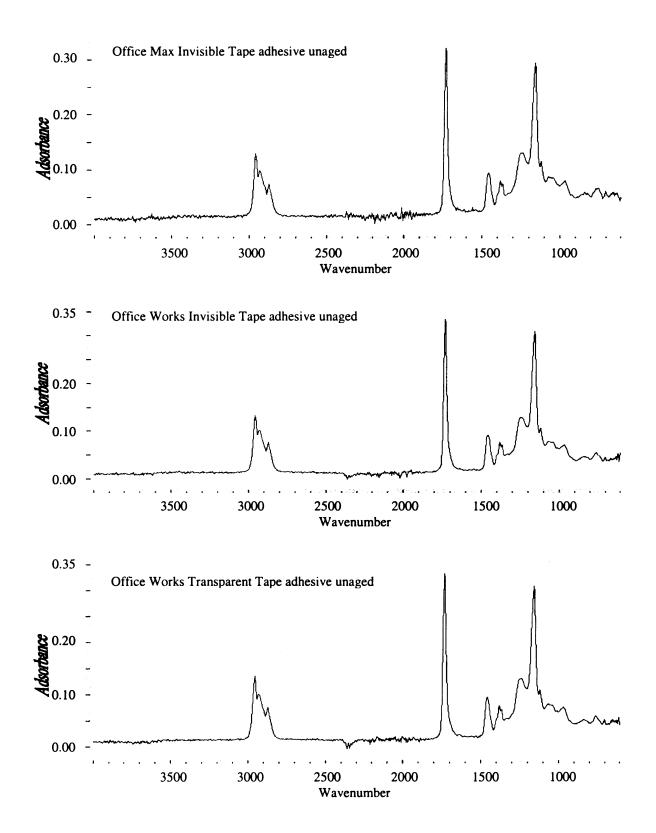


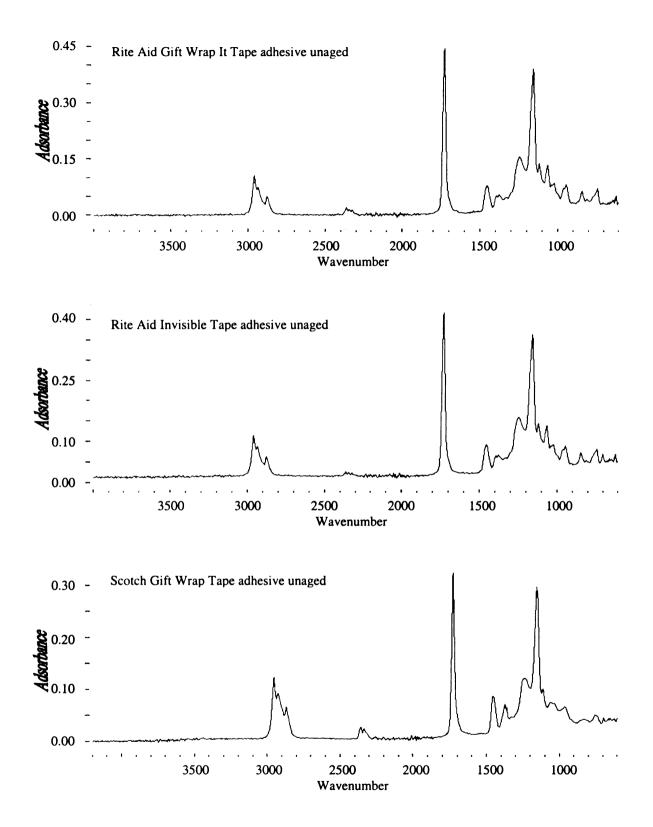


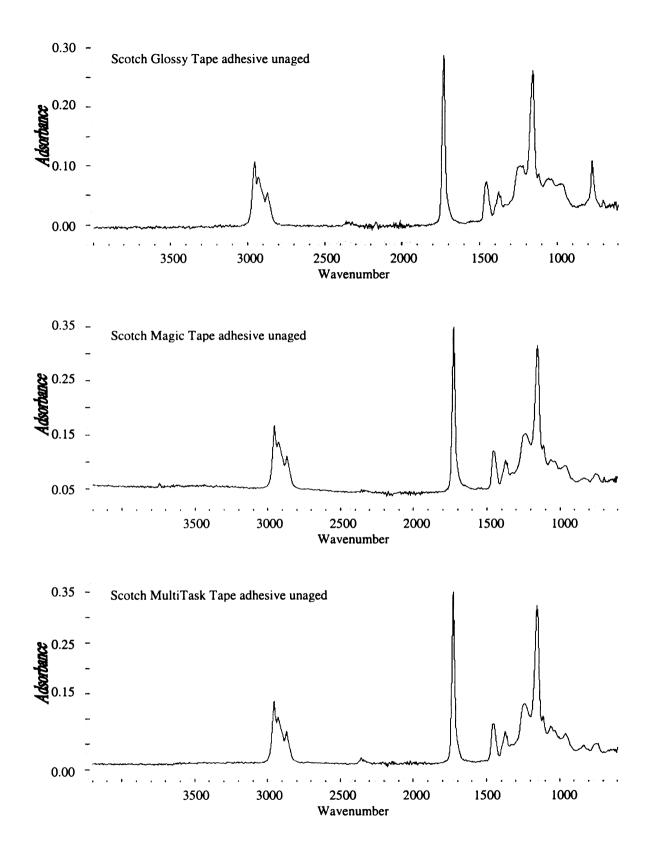
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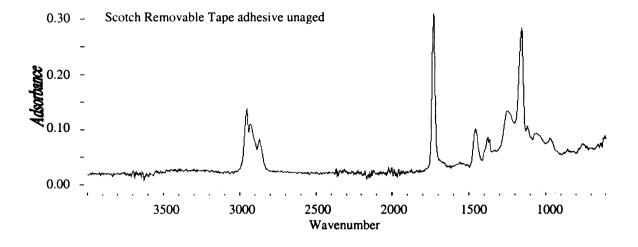


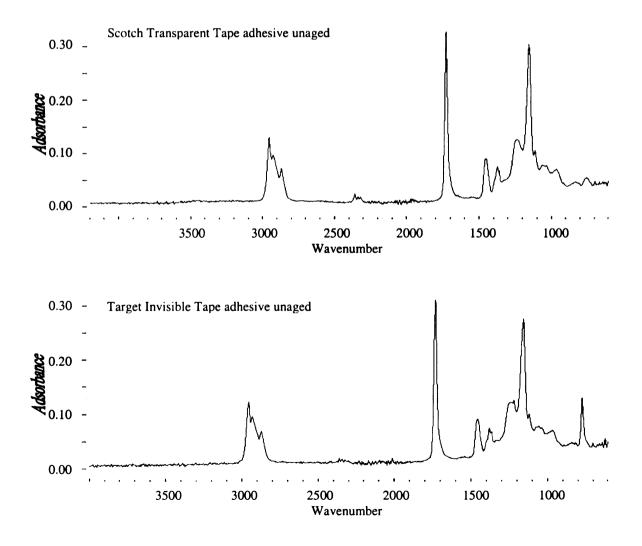


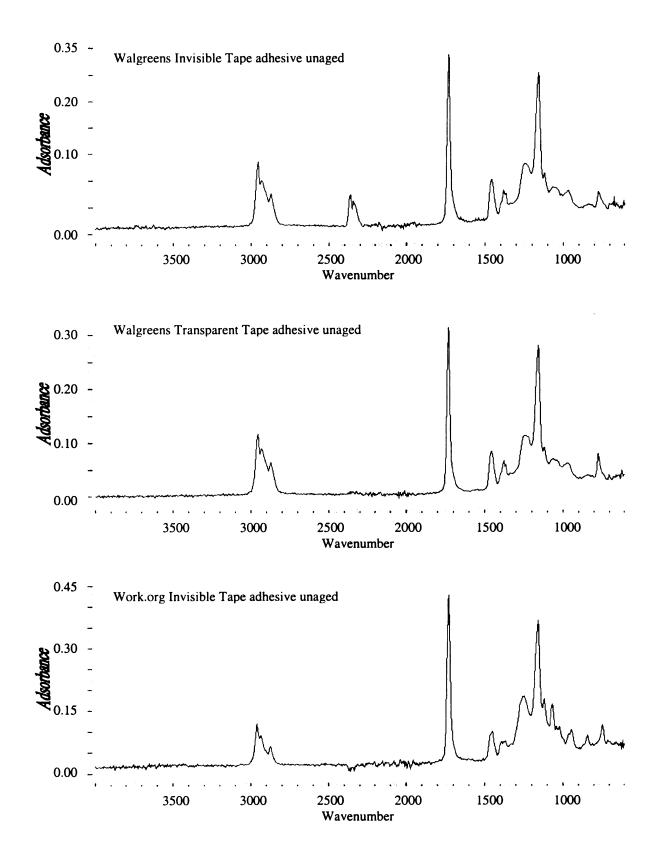


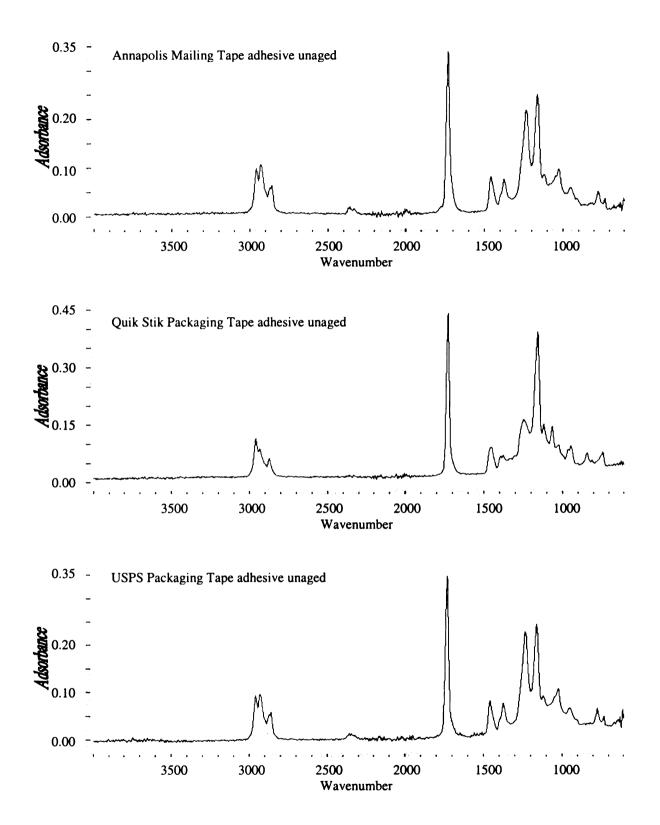


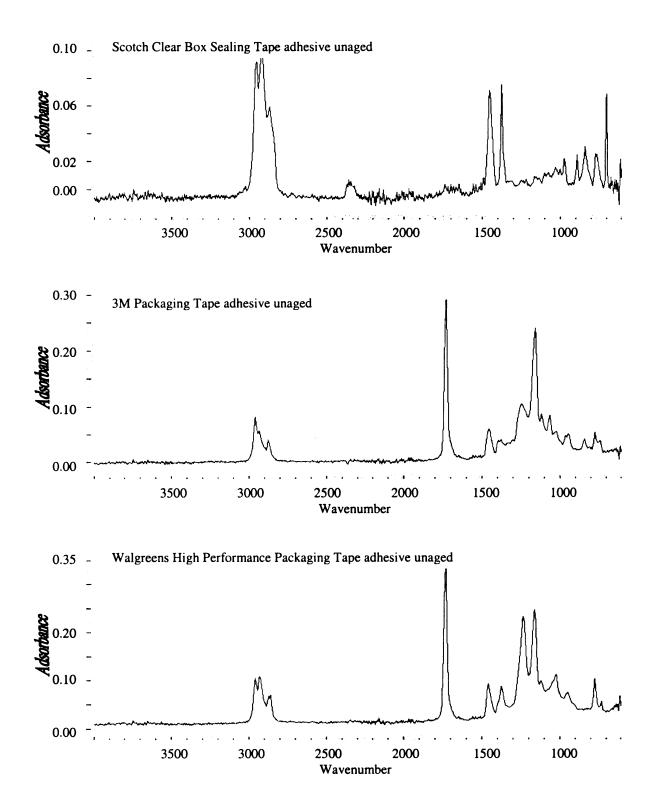


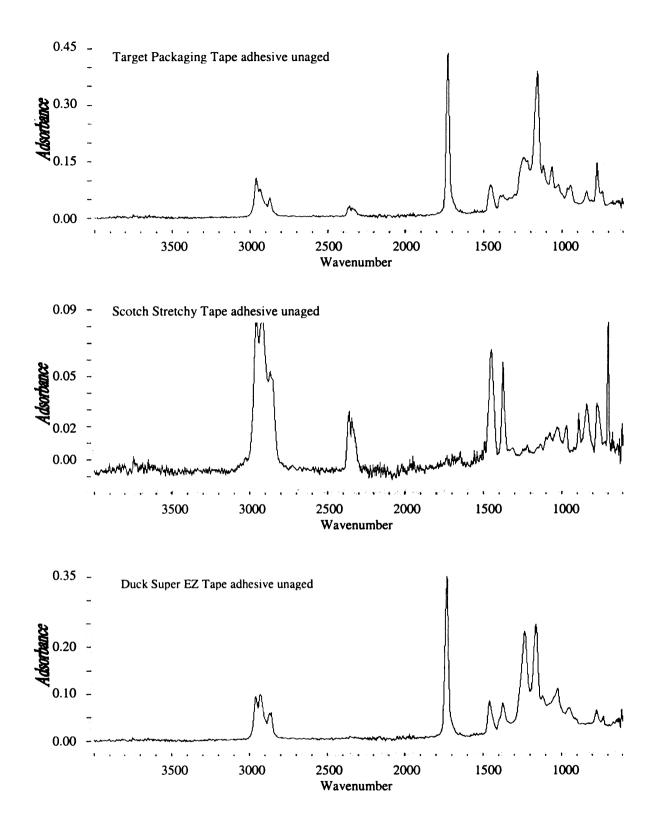


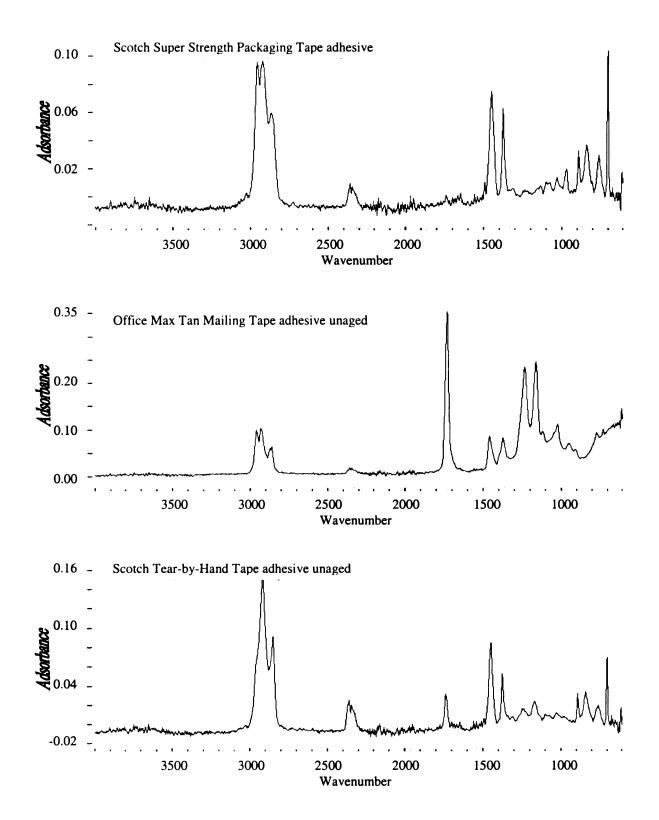


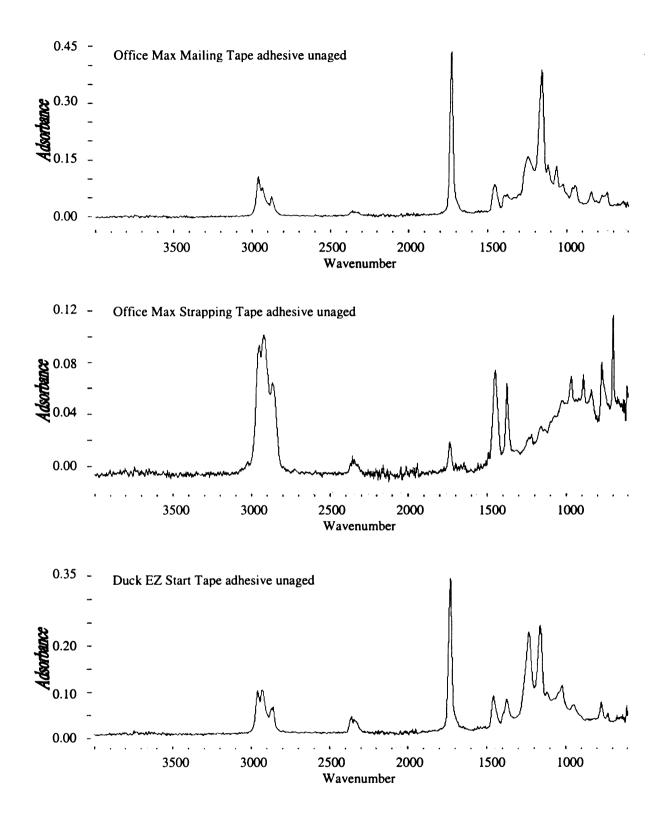


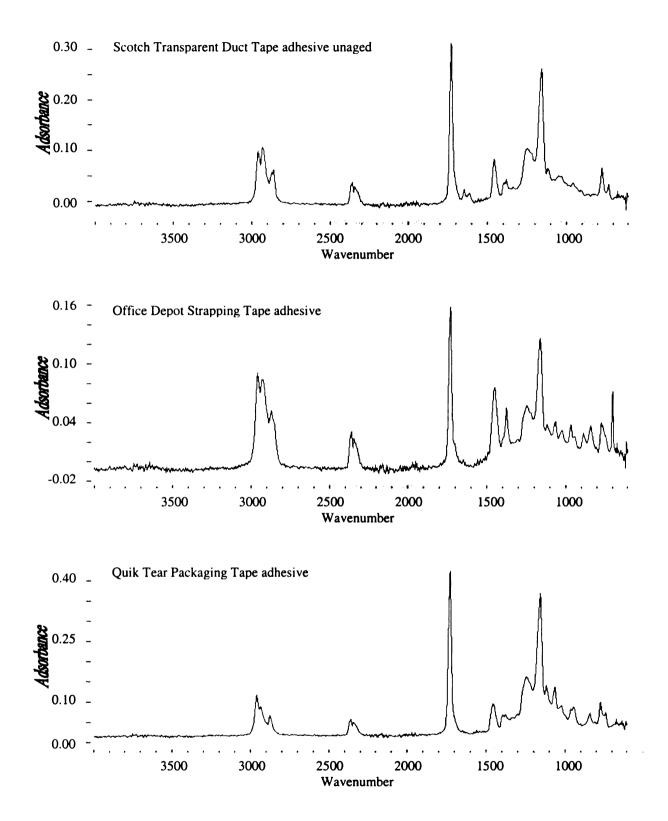


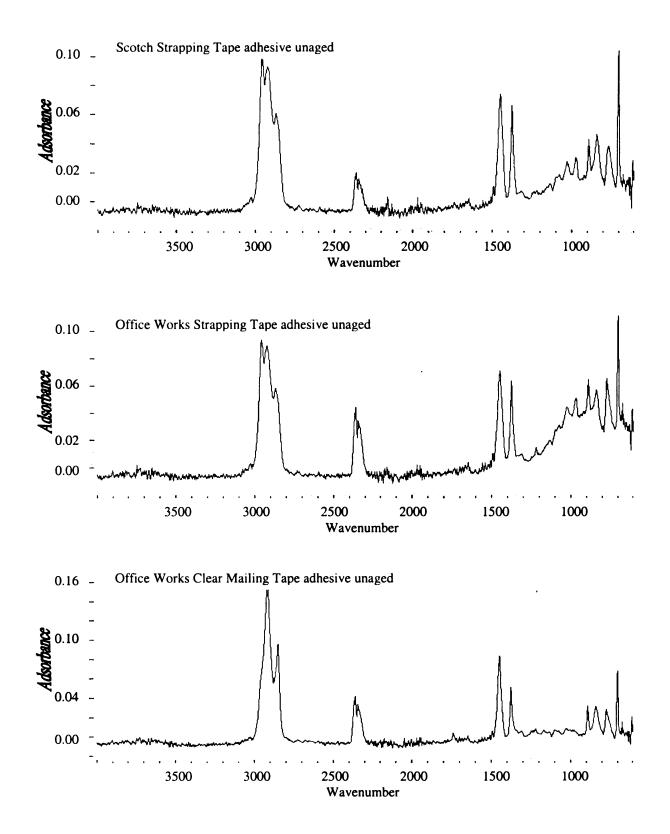


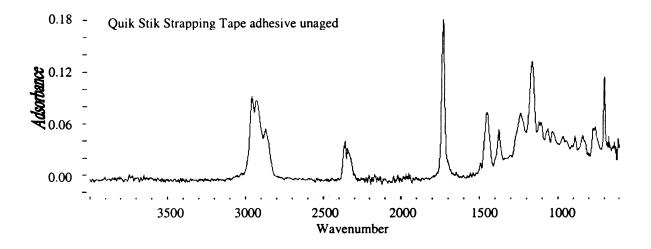


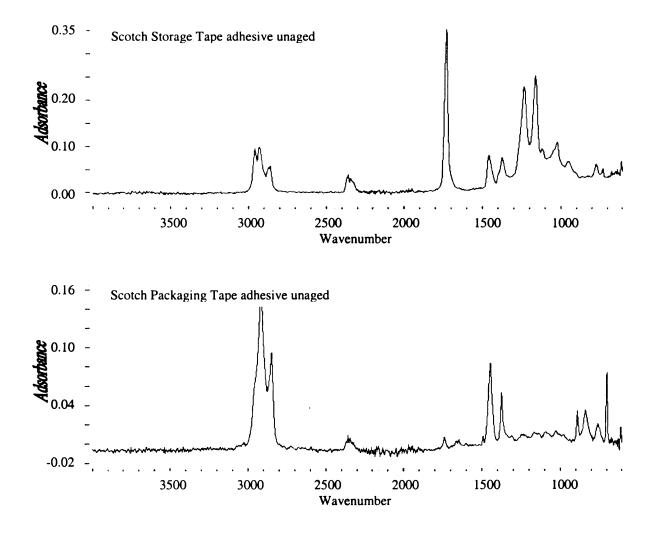








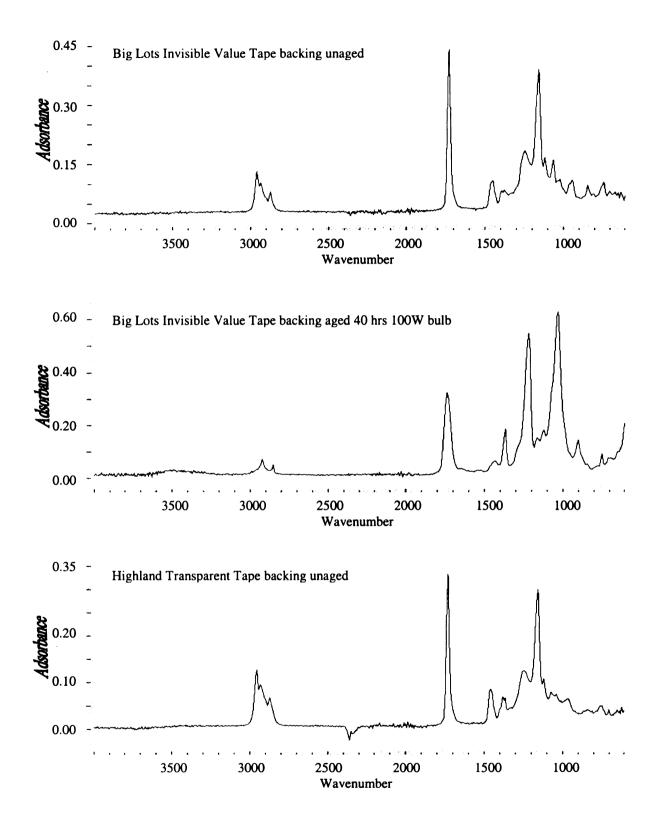


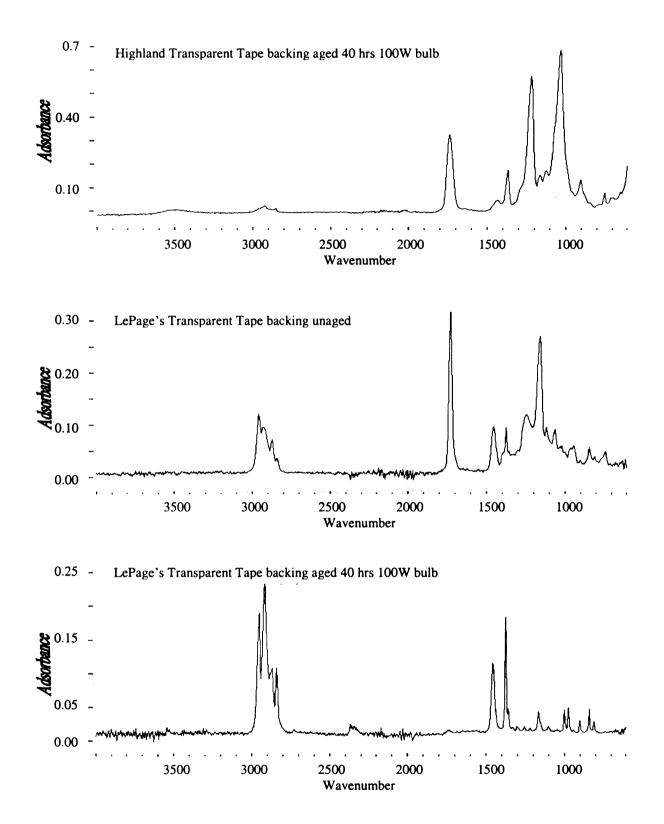


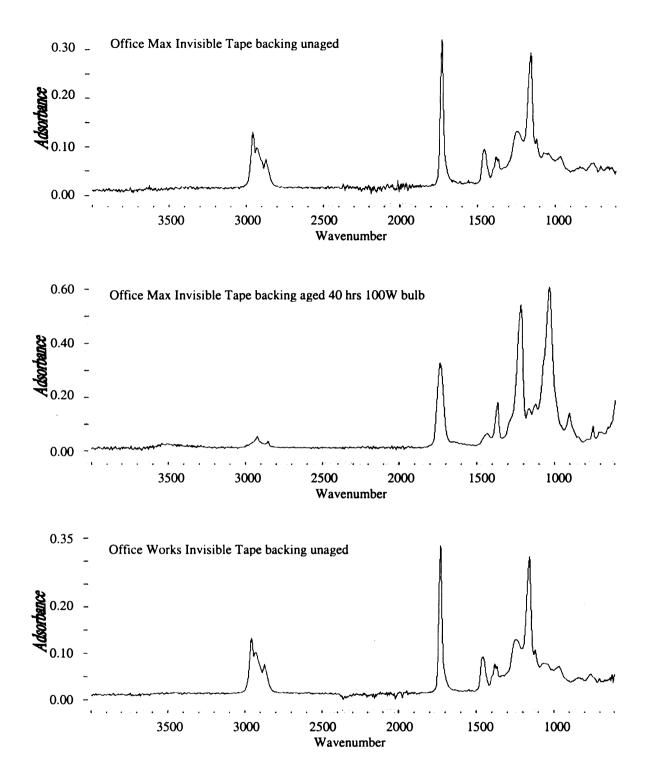
APPENDIX B

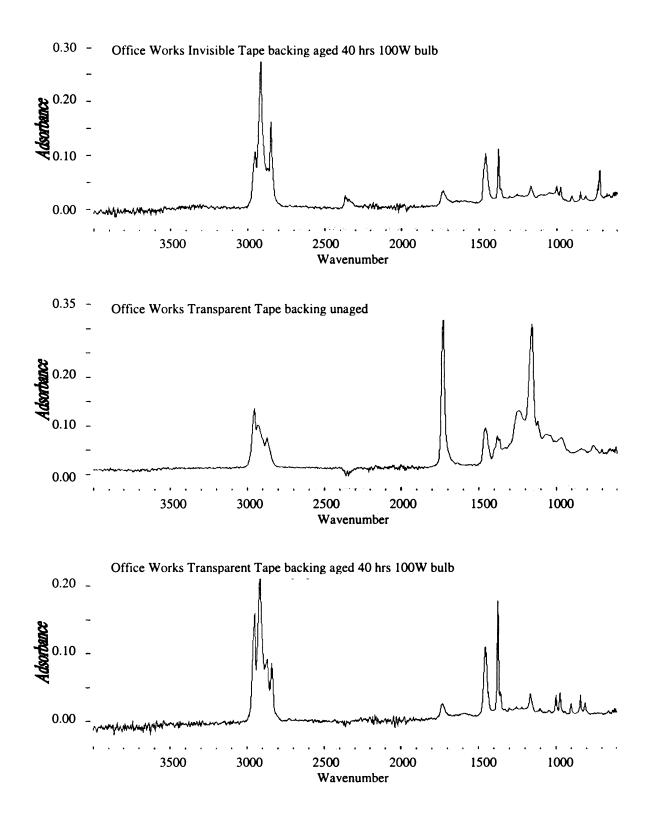
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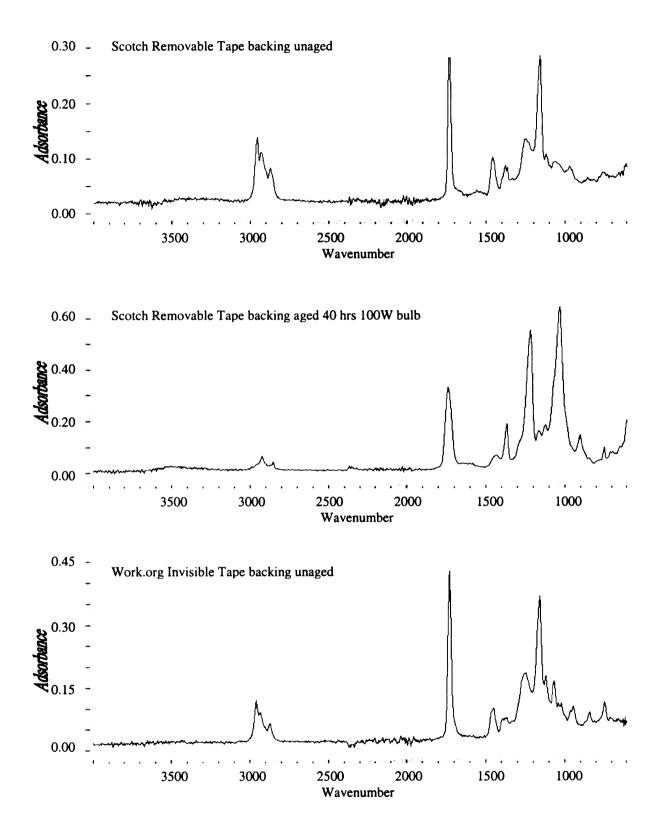
Artificially Aged Tape Backing And Adhesive FT-IR Spectra That Exhibited Changes

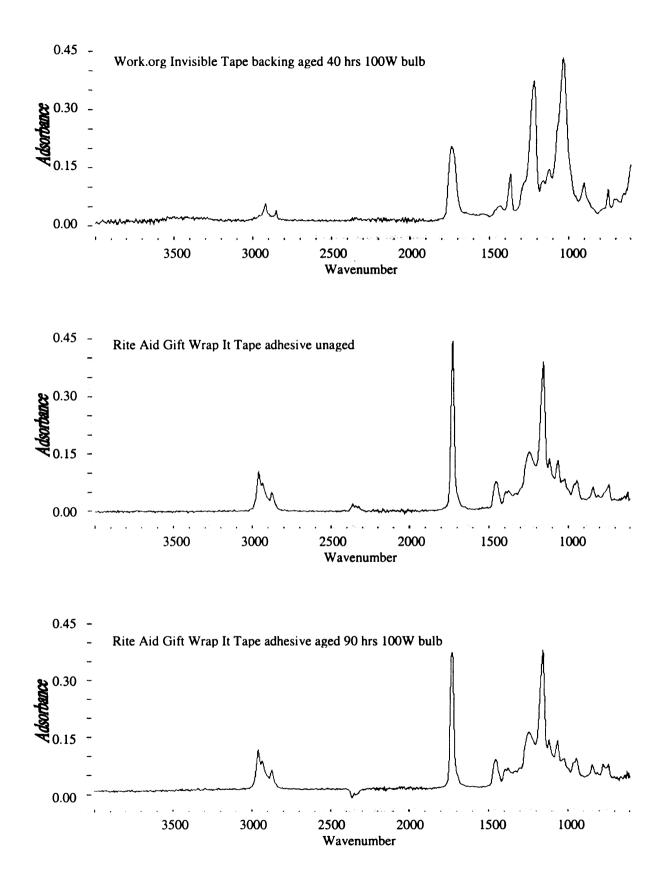


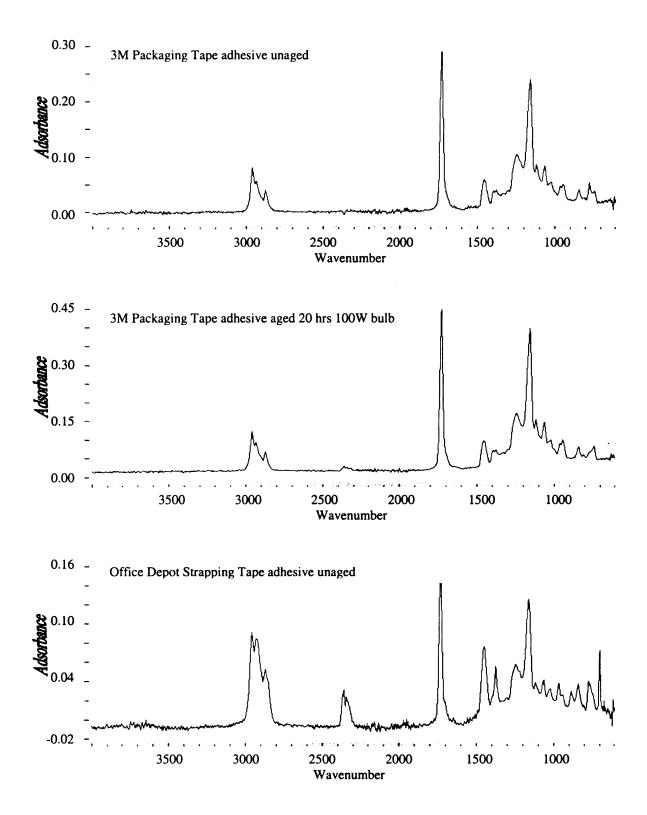


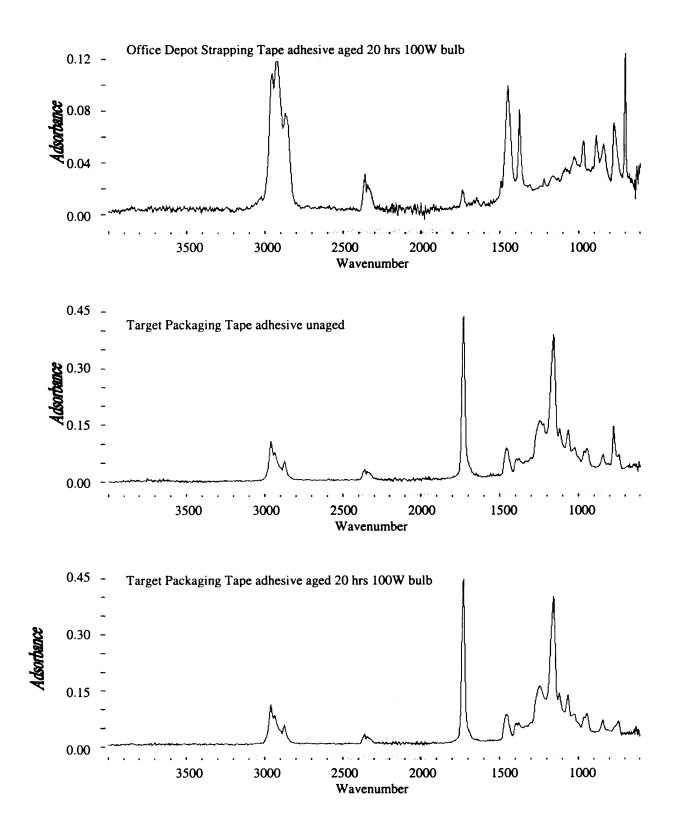


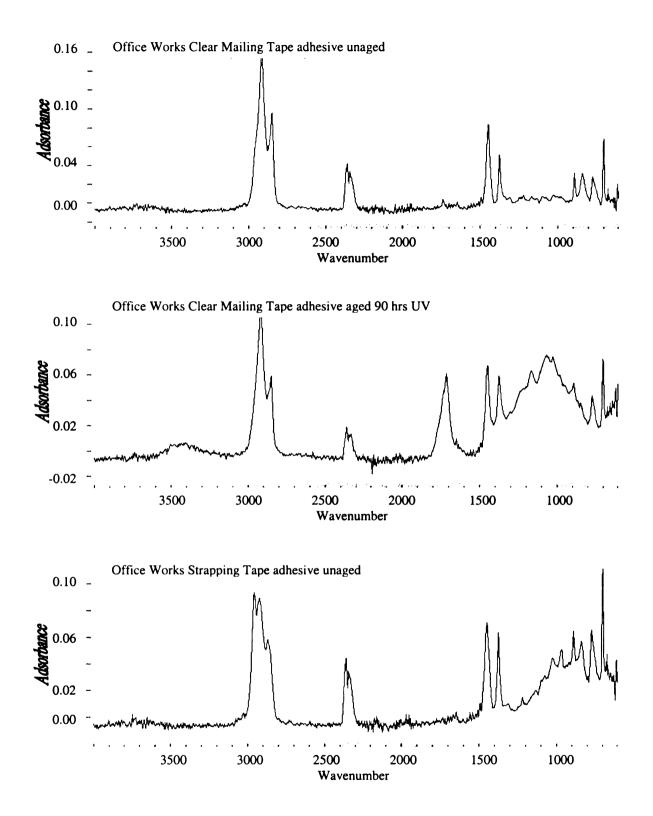


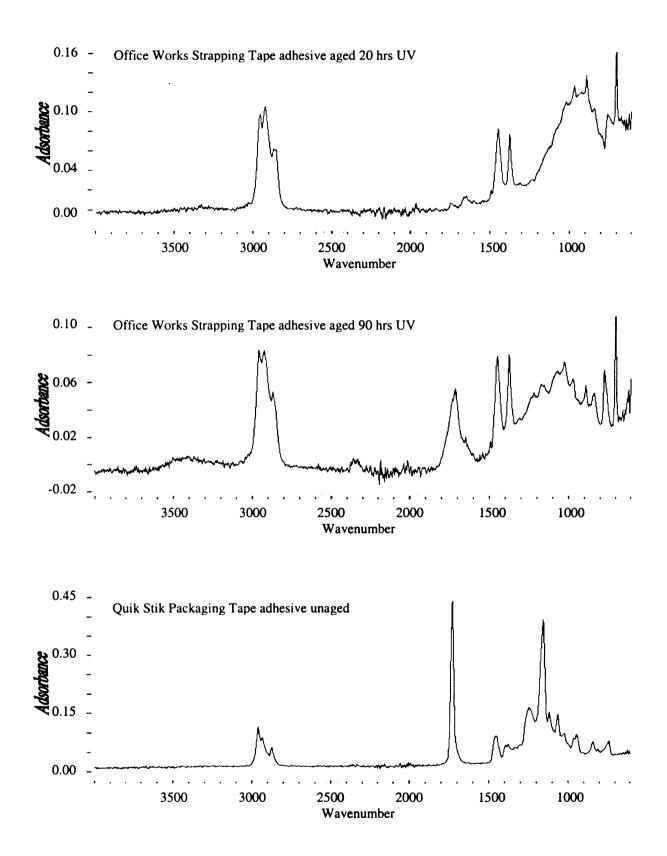


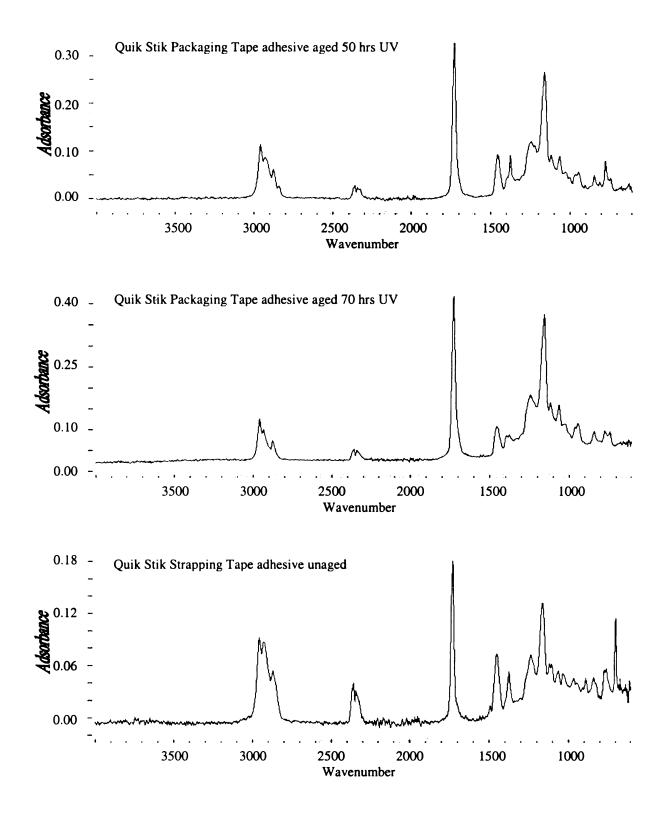


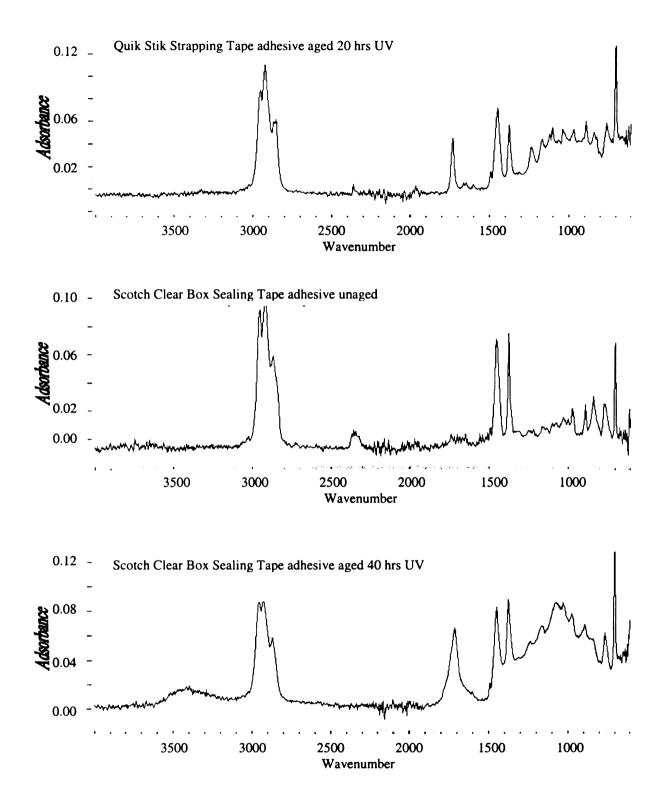


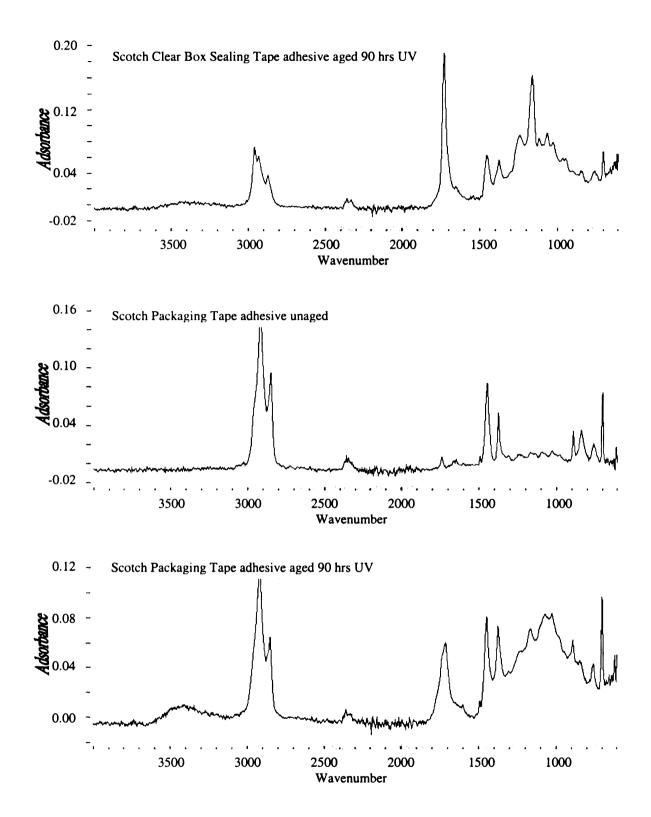


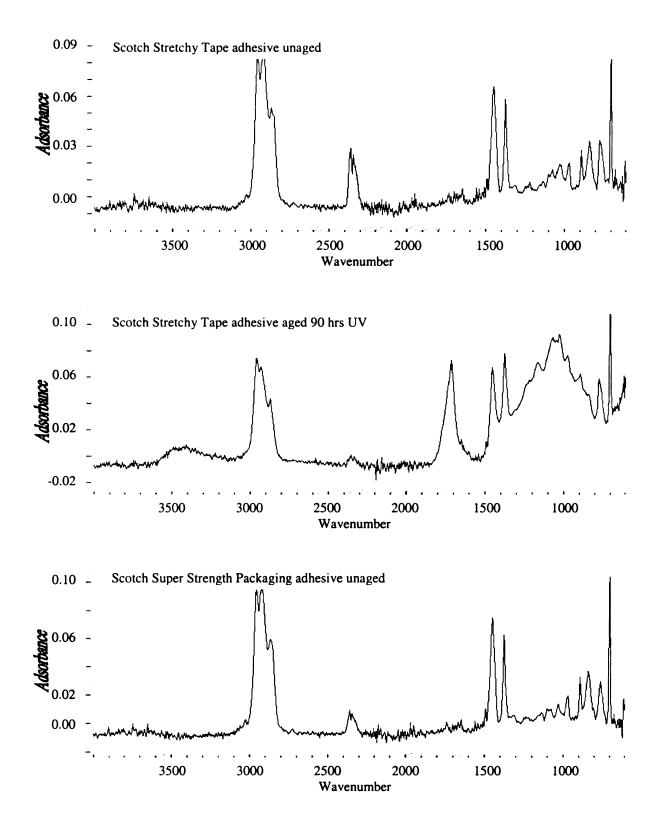


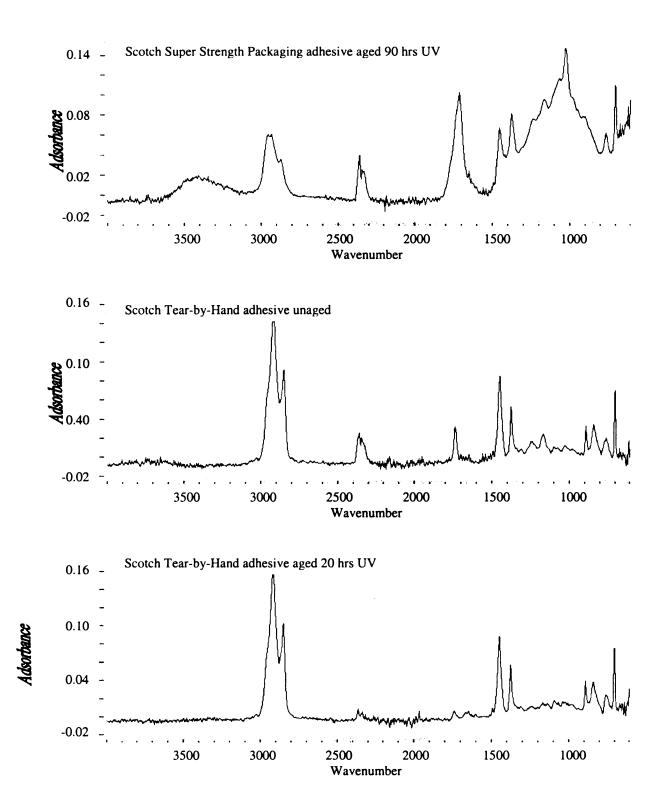


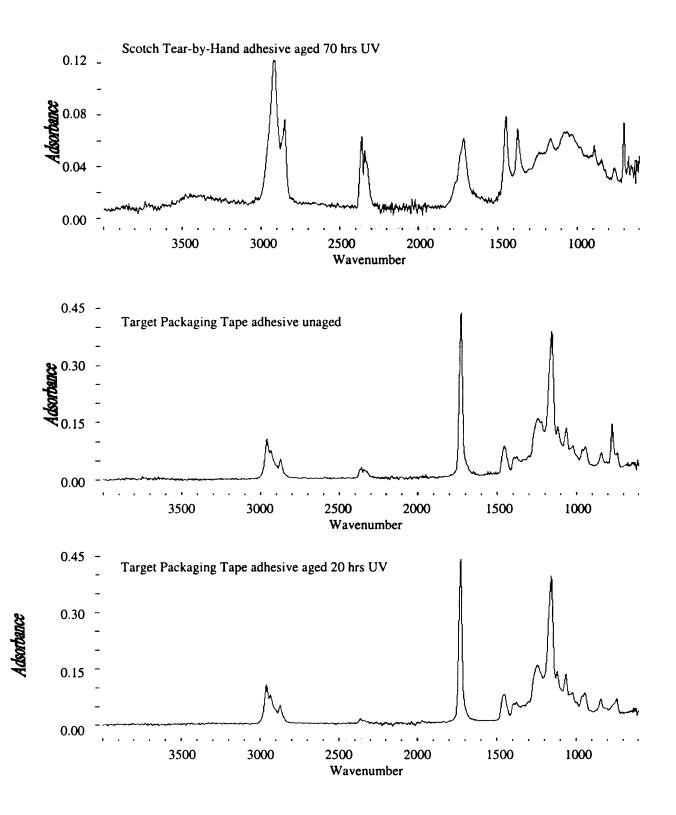




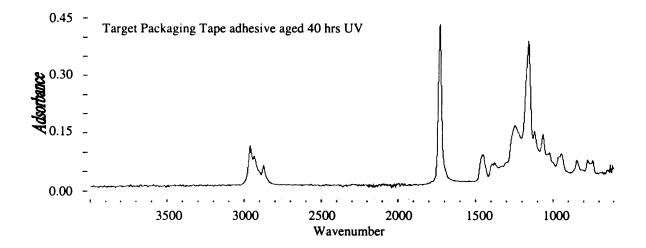












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