

**Optimization of Microspectrophotometric Analysis for the Differentiation of  
Pressure Sensitive Tapes**

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THESIS

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## LIST OF ABBREVIATIONS

(ATR) FTIR	Attenuated Total Reflectance Fourier-Transform Infrared
CCD	Charged Coupled Device
EA IRMS	Elemental Analysis Isotope Ratio Mass-Spectrometry
FTIR	Fourier-Transform Infrared
LA/ICP/MS	Laser Ablation Inductively Coupled Plasma Mass Spectrometry
LDPE	Low Density Polypropylene
MSP	Microspectrophotometry
NIST	National Institute of Standards and Technology
PDA	Photodiode Array
PLM	Polarized Light Microscopy
PVC	Polyvinyl Chloride
py-GC/MS	Pyrolysis Gas Chromatography Mass-Spectrometry
SEM-EDX	Scanning Electron Microscopy Energy Dispersive Spectroscopy
UV-Vis	Ultraviolet-Visible
XRD	X-Ray Powder Diffraction
XRF	X-Ray Fluorescence

## SUMMARY

Four different types of colored pressure sensitive tapes—duct tape, electrical tape, packaging tape, and masking tape—were analyzed using visible microspectrophotometry configured for transmission. Sample preparation of these tapes was optimized for microspectrophotometric analysis with consideration to sample thickness, adhesive removal, and feasibility of sample handling. Duct and electrical tapes were best prepared by adhering the tape to a piece of cling wrap, fixing the sample in epoxy, and microtoming the sample into cross sections with thicknesses between 5  $\mu\text{m}$  and 50  $\mu\text{m}$  such that a single sample's thickness varied less than 15  $\mu\text{m}$ . Packaging tapes were best prepared by adhering the tape to a slide for direct-through analysis without cross sectioning. Masking tapes were best prepared by hand cross sectioning the tape sample. Four rolls of each type of tape, varying in color and brand, were prepared using these preparation methods respectively and analyzed for intra- and inter-roll variation as detected by visible microspectrophotometry. The beginning, middle, and end of each roll were compared and found to have no appreciable variation as observed through microspectrophotometry. The beginning of each roll was compared to the beginning of a roll of the same type, style, and manufacture purchased 6 months later and no appreciable variation was observed through microspectrophotometry. Comparisons of tape samples of the same color (e.g. blue) that differed in type or manufacturer did show appreciable differences as observed through microspectrophotometry. Duct tapes, electrical tapes, packaging tapes, and masking tapes were found to be amenable for visible microspectrophotometric analysis. This analysis was found to have the capacity to provide information indicative of class characteristics.



## I. INTRODUCTION

### A. Contextual Background

Pressure sensitive tapes—colloquially referred to as tapes—are the combination of, minimally, a flexible backing and an adhesive that is capable of temporarily bonding to a surface as a result of light pressure.<sup>1</sup> These kinds of tapes are common household items that are readily available for purchase and have been found to be useful by criminals committing nefarious acts. Tapes may be used criminally as, but aren't limited in use to, bodily restraints, the construction of improvised explosive devices, and in the packaging of illicit materials. Current literature indicates that tapes have some degree of inter-roll variability between styles and manufacturers but are consistent throughout single rolls and manufactured jumbo rolls.<sup>2-17</sup> As such, tapes have the potential to serve as useful forensic evidence. As of 2012, more than 100 forensic laboratories across the world reported performing analyses on tapes. Forensically relevant tape, that is tapes most commonly processed by these forensic laboratories, include duct tape, electrical tape, packaging tape, and masking tape respectively.<sup>18</sup>

Forensic tape samples are primarily analyzed in an attempt to provide possible answers to two questions: what the tape is and to whom the tape belongs. The 'what' of the tape is determined by analysis of class characteristics that provide insight into its manufacture and distribution. The 'who' of the tape is determined by analysis of class and individualizing characteristics that provide insight as to what specific roll of tape the sample originated from. Current forensic analysis of tapes varies depending upon the specificities of the sample and questions being asked, but most commonly includes physical end matching, macro- and microscopic observation of physical characteristics,

observation of optical properties by polarized light microscopy (PLM), characterization by Fourier-transform infrared (FTIR) spectroscopic analysis and/or pyrolysis gas chromatography mass-spectrometry (py-GC/MS), and elemental analysis via scanning electron microscopy with energy dispersive spectroscopy (SEM-EDX).<sup>19-24</sup> Less common supplemental analysis may include microspectrophotometry (MSP), Raman spectroscopy, elemental analysis isotope ratio mass spectrometry (EA IRMS), X-ray fluorescence (XRF), laser ablation inductively coupled plasma mass spectrometry (LA/ICP/MS), and/or X-ray powder diffraction (XRD).<sup>1,18,19</sup> These methods may be used individually or in tandem to increase discrimination and identification of a sample until sufficient information is gained and a conclusion is reached.

While the majority of the supplemental analysis methods provide information that is complimentary to that of the commonly used methods, microspectrophotometry is unique in the information it provides: microspectrophotometry measures a material's absorbance of electromagnetic radiation in the ultraviolet and/or visible ranges.<sup>25,26</sup> That is, microspectrophotometry has the capacity to provide quantitative color analysis of a sample. Color assessment through unaided human vision is highly subjective and limited while instrumental color assessment through a microspectrophotometer has the capacity to provide objective discriminatory results that are preferred, if not expected, in United States' courtrooms.<sup>27,28</sup> Additional proponents of this type of spectroscopic analysis are that microspectrophotometry is a non- to semi-destructive technique that utilizes an instrument already considered to be integral to the analysis of other trace evidentiary samples such as fibers and paints.<sup>29,30</sup> It can be expected that forensic laboratories with trace evidence sections have access to microspectrophotometers and analysts trained to

use them and render conclusions based on microspectrophotometric data. It can also be expected that conclusions made with consideration to microspectrophotometric results have already been presented in and accepted by United States' courtrooms. Since it is the goal of a forensic scientist to analyze and render conclusions about evidence that may aid in legal proceedings, given sufficient sample availability and consideration to the idea that the more information extracted from a sample the stronger a conclusion about that sample may be, the addition of microspectrophotometry to the common tape analysis workflow is apropos.

#### **B. Statement of Problem**

No published guidelines for the use of microspectrophotometric analysis of tapes exists and minimal published research on microspectrophotometric analysis of tapes is available. The Tape Subgroup of the Scientific Working Group for Materials Analysis through the American Society of Trace Evidence Examiners has briefly mentioned the use of microspectrophotometry in the analysis of tapes.<sup>22</sup> A few forensic laboratories that perform tape analysis have also reported using microspectrophotometry in their analysis of tape evidence.<sup>18,31</sup> At the time this research began, a single study exploring the discriminatory power of various analytical techniques on office and packaging tapes mentioned the inclusion of microspectrophotometry in the analysis of packaging tapes.<sup>32</sup> Prior to the conclusion of this research, an additional study that performed more in depth analysis of microspectrophotometry of blue and red tape backings was published.<sup>33</sup> The lack of available information alongside the recent publication highlight the timeliness and necessity of research into microspectrophotometric analysis of tapes.

While there is an abundance of thorough and in-depth research on the use of a microspectrophotometer for evaluation of evidentiary items such as fibers, there is a significant gap in knowledge regarding microspectrophotometry's potential use in the analysis of pressure sensitive tapes. Tapes are different than fibers in their structure and composition; the available microspectrophotometric research done on fibers is not intrinsically applicable to tapes. Therefore, at present, there is a need for foundational research on the application of microspectrophotometry on the analysis of tapes. Specific and urgent needs include determination of what types of tape samples are amenable to microspectrophotometric analysis, optimization of sample preparation, determination of the discriminatory power of this technique in regard to class and individualizing information with respect to intra- and inter-roll variation, and consideration of potential effects on orthogonal analysis and sample consumption. Addressing these needs and publishing on them is prudent for the analysis of tapes by microspectrophotometry.

### **C. Purpose of Study**

This study aims to provide fundamental information pertaining to the analysis of pressure sensitive tapes by microspectrophotometry. Specifically, this study will address three foundational topics in regard to the microspectrophotometric analysis of duct tapes, electrical tapes, packaging tapes, and masking tapes. The research conducted during this study will determine the best sample preparation methods of the aforementioned tapes for analysis by microspectrophotometry with consideration towards sample thickness, adhesive removal, and feasibility of sample handling. This study will assess for which of the forensically relevant tape samples and in what scenarios microspectrophotometry may

serve as worthwhile analytical step. This study will also address the intra- and inter-roll variation of tapes as observed by microspectrophotometric analysis. The findings of this research will promote successful analysis of tape samples in crime laboratories as well as serve as a foundational basis for future research studies.

#### **D. Research Hypotheses**

It is hypothesized that pressure sensitive tapes will be amenable for analysis by microspectrophotometry. Specifically, the microspectrophotometric analysis of prepared duct tape, electrical tape, packaging tape, and masking tape samples will provide useful data that has potential to aid in forensic investigations. The resultant collected data and information is hypothesized to be indicative of class characteristics. That is, microspectrophotometric data will provide information that is consistent throughout a single roll and throughout rolls of the same type, style, and manufacture, but different between rolls of unique type, style, and manufacture.

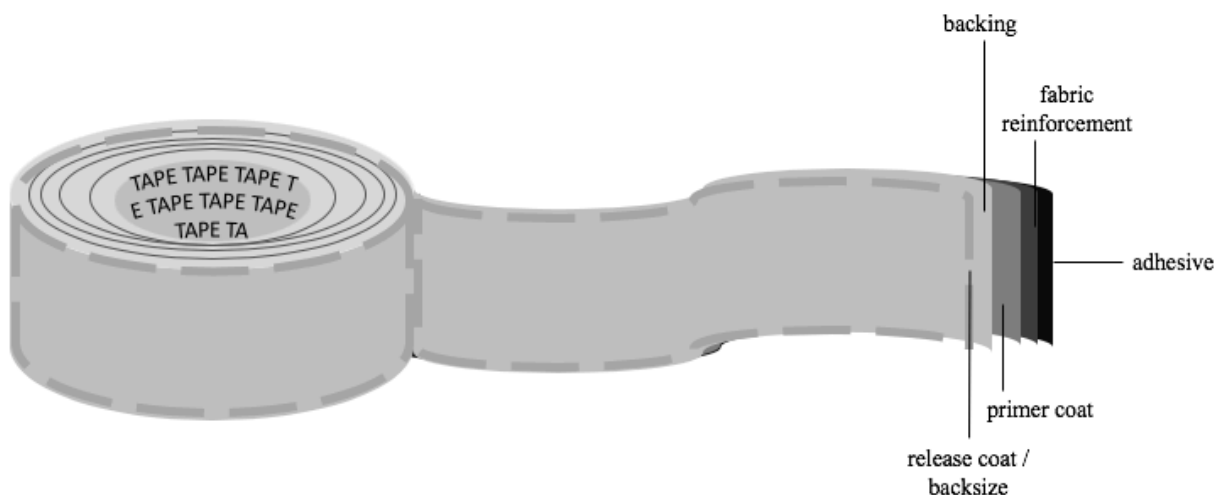
## II. LITERATURE REVIEW AND RELATED BACKGROUND INFORMATION

### A. Pressure Sensitive Tapes

Different types of tapes, that is tapes with different end uses, are highly variable. Tapes with the same end use will also vary over different manufacturers, styles, and time. These variations are the result of differences in production and materials due to natural progression as well as market trends and costs. The forensic analysis of tape relies on careful detection of these variations. Tapes are manufactured in layers through a series of milling and mixing processes that culminate in the combination of layers through coating and calendaring. The end result is a large jumbo roll tape product that is cut, or slit, into the smaller recognizable tape rolls available for commercial purchase.<sup>34</sup> The layered nature of tape further adds to the amount of variation that may be possible.

Tapes can be the culmination of many layers including, but not limited to, a release coat, backsize, the tape backing, a primer coat, fabric reinforcement, and the adhesive. See Diagram 1 displaying this layered structure below.

Diagram 1.) General Tape Layer Structure



The release coat, backsize, and primer coat are not always present in tapes and when they are present, they are relatively thin and do not contribute an appreciable amount to the analysis.<sup>1</sup> The backing, fabric reinforcement of duct tapes, and adhesive are the most commonly analyzed and consequently researched components of tapes. Generally, the backing layer of a tape is a polymer that serves to provide support for the adhesive. Other components such as colorants, fillers, cross-linkers, plasticizers, stabilizers, and fire retardants may be added to the backing to impart desirable qualities or reduce production costs.<sup>1</sup> The adhesive layer of a tape is the combination of an elastomer and resin tackifier that is used to hold objects together by surface contact.<sup>19</sup> The strength of an adhesive may be measured by peel adhesion, cohesion, and/or tack. Peel adhesion is the strength of a bond between the tape and the substrate, cohesion is the internal strength of the adhesive, and tack is the ability of the tape to create an immediate bond with a substrate.<sup>35</sup> Fillers, colorants, extenders, and cross linkers may also be present in the adhesive layer. All of these layers vary depending on the type and end use of the tape: The four tapes included in this research, duct, electrical, packaging, and masking tape vary in their use, construction, research, and analysis. It is therefore important to consider each tape type individually.

### 1. Duct Tapes

Duct tape, also referred to as poly coated cloth tape, originated in The United States of America in the 1930s where it was adapted for military use in World War II as a waterproof ammunition container seal.<sup>36</sup> Today, duct tape is a popular commercial product that is widely used across the world. Duct tapes most commonly consist of three layers: a low-density polyethylene (LDPE) backing, a fabric reinforcement, and a thick

natural rubber-based adhesive that may also be blended with a synthetic rubber. Since LDPE is produced under high pressures and temperatures that cause uncontrollable branching of the macromolecules, the backing layer of duct tapes is flexible enough that plasticizers are not necessary.<sup>1,37</sup> The backing is commonly colored with aluminum powder to impart a silver color, but color may also be imparted through other colorants or screen printing of patterned designs. The fabric reinforcement of duct tape, known as scrim, consists of loosely woven fabric that allows for easy tearing across the tape. Scrim is typically embedded in the adhesive layer and is often comprised of cotton, polyester, glass filament, or nylon fibers.<sup>1</sup> Given the prolific production and use of duct tape, all three layers may be analyzed in an effort to discriminate between samples.

Experts in the field recommend that, when possible, the analysis of duct tapes should start with physical end matching. After which, analysis should include physical characterization, elemental analysis of both the backing and the adhesive layers, FTIR spectroscopic analysis of both the backing and adhesive layers, and PLM based analysis of the adhesive and scrim layers to compliment the information gained through instrumental analysis.<sup>1,20-23,36</sup> Available research on duct tape analyses reflect the usefulness of these methods.

Despite being the first analytical step, pattern-based evidence, including physical matching as a means of individualization, has faced criticisms for its lack of foundational studies and statistical support.<sup>28</sup> However, a 2011 study performed by Frederic Tulleners and Jerome Braun addressed physical end matching of duct tapes and proposed a statistical evaluation of error rates and accuracy. It was shown that both torn and cut duct tape samples could be accurately matched from 98.58 to 100.00 percent and from 98.15



to 99.83 percent respectively.<sup>38</sup> While this study highlights the strength of physical matches, these kinds of matches are not always possible. Research into other means of duct tape analysis, therefore, must be available and recognized.

Historically, tape evidence analysis was based on case examples and discussion between laboratories. More formalized studies into the usefulness and power of different analytical techniques were eventually published. In 1984 at a symposium on the analysis and identification of polymers, J. D. Benson showed how physical characteristics, FTIR, solubility, and emission spectroscopy could be used to analyze duct tape samples. Of the twelve samples he analyzed, he found that physical characteristics could be used to distinguish the majority of the tapes from one another and that instrumental methods could be used to differentiate the remaining samples.<sup>14</sup> At the same symposium, T. L. Jenkins Jr. highlighted the power of X-Ray spectrometry in the analysis of duct tape. He found that most duct tapes analyzed could be identified by the presence and concentration of certain elements. This study also noted that these observations were consistent throughout a single roll of tape.<sup>11</sup>

Later, in 1998, Jenny Smith assessed the variability of duct tapes from different manufacturers as well as the same manufacturer through physical characteristics, FTIR, and EDX. She concluded that the combined use of all these techniques could help discriminate different duct tapes and that duct tapes produced from the same manufacturer under the same labeled style could be variable. This emphasized the value of duct tape as forensic evidence since the characteristics observed could point to a smaller class of groupings.<sup>15</sup>

In 2000, Rena Merrill and Edward Bartick addressed the advantages and disadvantages for various ATR accessories. It was previously accepted that ATR was the preferred method for FTIR analysis, but advances in instrumentation had not been formally addressed. This research not only looked at duct tapes, but also included electrical tapes, packaging tapes, and office tapes.<sup>9</sup> In 2007, the FBI modified their standard operating procedures to reflect research conducted by Andria Hobbs et. Al. that found that it was useful to conduct microscopic examinations of duct tape cross sections. This cross-sectional analysis allowed for observation of the layered structure of the tape. Moreover, it was concluded that ATR analysis of both sides of the backing allowed for more discriminating results than analysis of just one backing side.<sup>10</sup>

Expanding upon previous research, in 2012 Andria Mehlretter and Maureen Bradley addressed the discriminating power of physical characteristic analysis, FTIR, XRD, and SEM-EDX. They found that physical characteristics, including backing color, discriminated 99.6 percent of possible comparisons. In this study, subjective interpretation of color based on visual examination was performed. Beyond this, they found that subsequent instrumental analyses further discriminated samples to 99.8 percent.<sup>12</sup> In 2015, Andria Mehlretter et. Al. performed an extensive study assessing the intra- and inter-roll variation of duct tape and what analyses would be suitable after accounting for such variation. She found that traits such as scrim count, width, thickness, and adhesive composition were continuous throughout a single roll of duct tape. She also observed that warp yarn offset was variable throughout a single roll of duct tape and that width varied between different rolls cut from the same jumbo roll.<sup>13</sup>

None of the available research on duct tapes address the use of microspectrophotometry. These publications do however outline important trends and conclusions about duct tape analysis as a whole. In multiple studies, it was found that physical characteristics, including backing color, provided some of the most discriminating conclusions about the tape. These studies did not employ the use of a microspectrophotometer, but the addition of this instrument would provide stronger color-based conclusions than those which have already been found to be useful. This could preclude the need for further analysis, saving laboratories time and money. Additionally, microspectrophotometry is non- to semi-destructive so, if necessary, subsequent analyses would still be possible. Research into microspectrophotometric analysis of duct tapes would add a novel contribution to presently available body of research on the analysis of duct tapes.

## 2. Electrical Tapes

Electrical tapes have a very specific end use: they are designed for use on electrical components. As such, their properties serve to increase insulation and corrosion resistance. The main layers of electrical tape include a plasticized poly vinyl chloride (PVC) backing and a natural rubber based adhesive.<sup>1,4</sup> The backing will commonly include fillers, fire retardants, colorants, and stabilizing compounds. Without the plasticizer, the PVC backing would be inflexible and useless as a tape backing. Unfortunately, the added plasticizer can mask the detection of other components as well as migrate into the adhesive layer where it may also make detection of other components more difficult.<sup>1,4,37</sup> Despite this, electrical tapes have been successfully analyzed as useful forensic evidence.

Experts in the field recommend that after a physical match is attempted, analysis should include physical characterization, elemental analysis of both the backing and the adhesive layers, FTIR spectroscopic analysis of both the backing and adhesive layers, and py-GC/MS.<sup>1,20-24</sup> Research on electrical tape analyses reflect the usefulness of these methods.

As with duct tapes, electrical tape evidence analysis was historically based on case examples and discussion between laboratories. More formalized studies that addressed the usefulness and power of different analytical techniques were eventually published. At the previously mentioned 1984 symposium on the analysis and identification of polymers, Thomas Kee presented a classification scheme to differentiate electrical tape samples based on elements found to be present through XRF.<sup>4</sup> Here, Raymond Keto also analyzed three rolls of the same style of electrical tape from six different brands using stereomicroscopy, FTIR, and XRF. He found that using these methods, tapes from different brands could be distinguished and that tapes from the same brands were consistent with one another.<sup>8</sup> Later in 1988, Evan Williams and T. O. Muson showed how py-GC/MS could be used to distinguish different rolls of electrical tape and that these results were also consistent throughout a single roll of tape.<sup>7</sup>

As previously mentioned, in 2000 Rena Merrill and Edward Bartick addressed the advantages and disadvantages for various ATR accessories. Their research looked at electrical tapes, duct tapes, packaging tapes, and office tapes.<sup>9</sup> In 2007, John Goodpaster et. Al. performed an extensive study that showed the discriminating power of SEM-EDX in the analysis of electrical tapes.<sup>5</sup> This was quickly followed by a 2009 study by Goodpaster et. Al. that found ATR was more discriminating than SEM-EDX.

Specifically, they found that ATR analysis of the electrical tape adhesive was most discriminating.<sup>6</sup>

In 2011, Andria Mehlretter et. Al. explored the discriminatory power of individual techniques and the combination of techniques including stereomicroscopy, FTIR, py-GC/MS, and SEM-EDX. This study focused on the adhesive layer of electrical tapes while another 2011 study by Mehlretter et. Al. addressed the same techniques but for the backing layer of the electrical tapes. Both studies showed that the combination of all of these techniques allowed for the most discriminating analysis of electrical tapes.<sup>2,3</sup> These studies were also the first published studies that formally addressed the discriminatory power of physical characterization of electrical tapes.

Similarly to duct tapes, none of these studies include the analysis of electrical tapes by microspectrophotometry. There is, however, the recurrent conclusion that a combination of methods increases the level of sample discrimination that is possible. Most of these studies also emphasized the analysis of black electrical tape but did not address the usefulness of current techniques' discriminating power for colored electrical tapes. It is not unreasonable to assume that the color of a colored electrical tape could be a useful observation, especially when considering the value previously seen for the physical characterization of tapes. Microspectrophotometry could therefore serve as a helpful analysis method for colored electrical tapes. Microspectrophotometry is non- to semi-destructive so subsequent analyses would still be possible. Research into microspectrophotometric analysis of electrical tapes would serve as a novel addition to the current body of research available on the analysis of electrical tapes.

### 3. Packaging Tapes

Prevalent for their use in shipping, packaging tapes are most commonly composed of two layers: a polypropylene backing and an acrylic based adhesive. Packaging tapes are frequently clear or brown colored. The brown coloring may be the result of iron oxides or titanium dioxide added to the backing or adhesive layer.<sup>1</sup> Packaging tape backings are oriented polymers, meaning, when they are produced the film is stretched out in one or two directions and slowly cooled. This slow cooling allows previously unordered polymer chains to align themselves in an ordered fashion related to the direction of stretch. A single direction of stretch forms a monoaxially oriented polymer while two directions of stretch forms a biaxially oriented polymer. This film orientation can be determined by PLM. Microscopy may also be used to detect subtle differences in thickness by retardation colors under cross polarized light. Other observations such as the presence of additives and artifacts in the tape surface can also be made using PLM. After analysis by PLM, it is recommended that the backing and adhesive of packaging tapes are analyzed via FTIR.<sup>1</sup>

Compared to duct tapes and electrical tapes, little research has been published on the analysis of packaging tapes. Other than the previously mentioned 2011 study by Rena Merrill and Edward Bartick that addressed the usefulness of various ATR accessories, only one relevant study was found in which packaging tapes were analyzed.<sup>9</sup> This research, a 2001 study by Philip Maynard et. Al., uniquely includes instrumental analysis of packaging tapes by microspectrophotometry. Specifically, packaging and clear office tapes were analyzed by various techniques including visible microspectrophotometry, ultraviolet spectroscopy, FTIR, and Py-GC/MS to assess to discriminatory power of the

techniques. For the visible microspectrophotometric analysis, small cuttings of tape samples were placed on glass slides and data was collected in reflectance mode from 380 nm to 750 nm. Samples were collected from three locations on a roll of tape and fifty scans were collected per sample. They concluded that microspectrophotometric analysis of packaging tapes provided the same level of discrimination as visual assessment of color. They did, however, note that microspectrophotometry does offer an objective measure of color that has the potential to be included in database searches.<sup>16</sup> While this study did address microspectrophotometry of packaging tapes, an expansion upon their conclusions and further development of the sample preparation of packaging tapes for microspectrophotometric analysis is still necessary.

#### 4. Masking Tapes

Masking tapes are designed to mask off surface areas during painting. As such, they are designed to have a low level of tackiness that does not damage the substrate to which they are applied and they are designed with barrier characteristics that repel moisture and paint solvents. The main components of masking tapes most commonly include a saturated crepe paper backing, a backsizing that smooths the surface of the backing, and a low tack rubber based adhesive.<sup>1</sup> Very little relevant research into the analysis of masking tapes has been published, but it is thought that physical characterization of a masking tape such as color and dimensions might be useful.<sup>1</sup> A 1984 study by Robert Blackledge emphasized the usefulness of fluorescence spectroscopy in the analysis of masking tapes. This study also highlighted the use of visual analysis, infrared spectroscopy, and py-GC/MS.<sup>17</sup>

There is an obvious lack of published research on masking tapes. There has been an indication in existing literature on the importance of a masking tape's color for analysis. As such, microspectrophotometric analysis of masking tapes has a potential to be useful and contribute to the body of research on the forensic analysis of masking tapes.

**Note:** Prior to the completion of this research, a study on the microspectrophotometric analysis of pressure sensitive tape backings was published. Susan Gross and Joshua Jorstad analyzed red and blue duct, electrical, and miscellaneous tapes. Specifically, 20  $\mu\text{m}$  cross sections were mounted in Permout<sup>®</sup> on a glass slide with a coverslip. Each sample was comprised of at least five cross sections and each sample was scanned fifty times. A collection range from 400 nm to 850 nm was used. All blind comparisons of microspectrophotometric data were found to be correct and it was concluded that the discriminatory power of microspectrophotometry alone ranged from 50 to 91 percent. The addition of microspectrophotometry to physical and ATR analysis served to increase the discriminatory power of the analyses.<sup>33</sup> This study emphasized that there is still a need for analysis of additional sample sets, additional colors, achromatic colors, and inter-roll variation. The research conducted for the purposes of this study addresses all of those points and expands upon and optimizes the process of tape sample preparation for microspectrophotometric analysis. Had Gross and Jorstad's work been published prior to the conception of this research, it would have only served to support the focus of this project and speak to the relevancy of this research topic in the field of forensic science.



## **B. Class and Individualizing Characteristics**

As a result of the manufacturing process and properties intrinsic to tapes, the analysis of tapes most often result in class characteristic observations. Class characteristics are traits produced by a controlled process that allow for sets of groupings of like objects.<sup>39</sup> At present, the only individualizing feature that has been documented in the analysis of any kind of tape has been physical end matching.<sup>38</sup> Individualizing characteristics are unique properties produced by uncontrolled processes that may individualize items to a common source.<sup>39</sup> Consideration to if an analysis is providing class or individualizing information is necessary when rendering conclusions about forensic evidence. This is especially true for tape evidence because the amount and length of material present in a single roll of tape has the potential to create variability within a single sample. This intra-roll variability in relation to inter-roll variability must be considered when conclusions are made as to the class or individualizing power of an analysis.

Ultimately, the goal of any analysis on tapes is dependent upon the questions being asked. Depending on the specificities of the sample and the case, the goal is most often to render a conclusion as to if an unknown sample could have originated from a certain source. In the context of tape evidence, this could involve a piece of tape found at a crime scene and a roll of tape found in a suspect's home. In leu of a physical end match, analyses are performed to see if the samples share class characteristics. If any disagreement is found the samples can be excluded as a possible match. If no disagreements are found the samples can be concluded to belong to the same class of items, meaning there is a potential that they could have originated from the same source.

In this way, an increased ability to discriminate between samples increases an analyst's ability to avoid type II errors. A type II error is an error in which there is a failure to reject a false hypothesis: In the context of comparisons, a type II error is a failure to exclude a sample that is not a true match. If there is no possibility for a direct comparison, tape evidence may be used to provide investigative leads. In this context, a piece of tape found at a crime scene may be analyzed for class characteristics that provide insight as to when the tape was manufactured, where it was manufactured, where it is sold, etc. In both of these instances, class characteristics have the potential to provide valuable information for a forensic science-based investigation.

### **C. Microspectrophotometry**

#### **1. Human Perception of Color**

Colorants, like dyes and pigments are used by the polymer industry to impart desirable aesthetic qualities to materials such as tapes.<sup>41</sup> A colorant is seen as colored based on the absorbance and transmittance or reflectance of specific wavelengths of light. When white light, light comprised of all wavelengths within the visible spectrum at equal intensities, interacts with a colored object the wavelengths of light which are not absorbed by the object are detected by the human eye.<sup>25,26</sup> Normal human eyesight is sensitive to light between roughly 400 nm to 800 nm and processes visual stimuli as a combination of brightness and color.<sup>41</sup> This is the range of light considered to be the visible region of the electromagnetic spectrum. The interpretation and communication of color as seen through human eyesight is highly subjective: Not only are specific hues,

tints, tones, and shades difficult to verbally describe, but conditions such as genetics, age, and environment may also affect how color is perceived.

Trace evidence comparisons are most commonly performed using comparison microscopes in which there are limits to the amount of lighting conditions available. In this way, it is possible for observed colors to be deemed similar when they are actually different. Simultaneous contrast, the phenomena in which color is perceived based on context, exemplifies the subjective nature of color vision. An example of simultaneous contrast is metamerism: Colors that appear the same under certain lighting conditions but appear different under other lighting conditions.<sup>25-27,43</sup> In the context of tapes, samples deemed similar after an initial physical characterization will be further analyzed by instrumental methods. While this is not inherently a problem, the use of a more discriminating method of color assessment at this stage in analysis could prevent the detection of erroneous similarities and save time, money, and sample consumption. Color detection through an instrument also serves to quantify color allowing for easier communication of results between laboratories and in court rooms. Unlike human vision, instrumental assessment of color allows for objective, repeatable, quantitative, and standardize-able observations that are necessary for proper forensic characterization of tapes.<sup>40</sup>

## 2. Instrumental Design

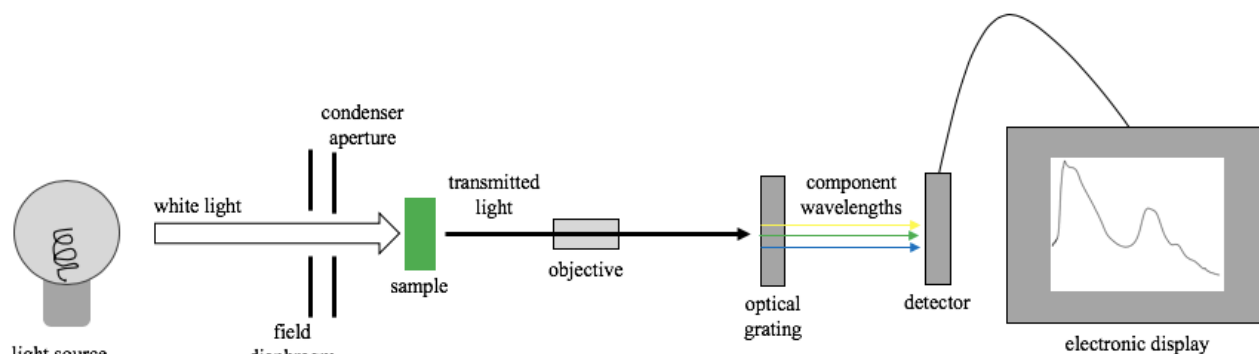
Spectrophotometry and microspectrophotometry are techniques in which a material's absorbance of electromagnetic radiation in the ultraviolet and/or visible ranges is measured.<sup>25,26,43</sup> A microspectrophotometer is a spectrophotometer that has been integrated with a microscope for the purpose of analyzing microscopic samples. The first

forensic use of microspectrophotometers was described in 1959 by H. Amsler for the analysis of fibers and microspectrophotometers had been used prior to this for the analysis of biological and non-biological samples.<sup>26</sup> Major advancements were made in microspectrophotometry in the 1990s with both the ability to pair the instrument with computers and the development of advanced detectors which allowed for the collection of high quality spectra in much shorter amounts of time than previously possible. Today, there are many different microspectrophotometer systems available and capable of producing reliable spectra.<sup>26</sup>

There are two different styles of microspectrophotometer: multi-channel and single-channel scanning.<sup>26</sup> A multi-channel microspectrophotometer, such as that used in this study, is comprised of a light source, a microscope, a dispersive or polychromator system, and a detector.<sup>26</sup> See Diagram 2 displaying this system design on the following page. In this style of system, light travels from the light source and through the microscope where it is focused onto the sample. The microscope serves to not only hold the sample in a defined position, but also transport and focus light onto and through the sample. The light then interacts with the sample and depending upon the properties of the sample specific wavelengths of light are absorbed or transmitted. The light that exits the sample is gathered by the objective and travels to the dispersive system. This system, consisting of an optical grating, separates the light into its component wavelengths whose intensities are picked up and recorded by the detector.<sup>43</sup> The detector may be a photodiode array (PDA) or Charged Coupled Device (CCD).<sup>26</sup> PDAs are unable to accumulate signals of specific wavelengths while CCDs, such as that present in the

instrument used for this study, can. After signal detection, information is communicated through a computer system as spectral data.

Diagram 2.) General Multi-Channel Microspectrophotometer Design



In contrast, a single-channel scanning microspectrophotometer is comprised of a light source, a monochromator, a microscope, and a photomultiplier.<sup>26</sup> In this style of system, light travels from the light source and through the monochromator which allows only specific spectral bands of light through at a time. Over the course of approximately a minute, different spectral bands are focused onto the sample by the microscope. After interacting with the sample, the resulting light is amplified and transformed into electrical energy by a photomultiplier tube. The electrical energy can then be communicated through a computer system as spectral data.

### 3. Theory of Use and Analysis

There are many factors in the instrumental design and use of a microspectrophotometer that have an influence on results obtained during analysis. To collect quality spectra, it is imperative to consider the impact of different conditions on analysis and to keep all conditions consistent throughout analysis. Conditions that may impact results include instrumental setup and parameters as well as sample preparation.

The ideality of these conditions may vary depending on the specificities of the samples and goals of analysis.

Instrumental setup and parameters that should be controlled and considered include bulb type, microscope setup, integration time, resolution factor, and instrumental aperture size. The two major bulb types used in microspectrophotometry include tungsten halogen bulbs and xenon bulbs. A xenon bulb provides a higher intensity output throughout the entire UV-Vis range.<sup>44</sup> Neither bulb is inherently better than the other and bulb type is often instrument or need dependent. The microscope setup should be performed with consideration to Köhler illumination.<sup>26,44</sup> Köhler illumination is a means of microscope setup that allows for maximum control over contrast, resolution, and depth of field as well as even illumination over the field of view.<sup>45</sup> Within the context of microspectrophotometry and this setup, the field diaphragm and condenser aperture both affect the amount of light allowed to pass through the sample. This light can be maximized or minimized depending upon the desired intensity of the spectrum.<sup>44</sup> The integration time used affects how long sample collection takes, this should be set based on the instrument and sample being analyzed. The resolution factor is a whole number value that describes the number of scans taken and averaged on either side of a specific data point. A higher resolution factor will result in smoother data, while a lower resolution factor provides the rawest data. The instrument aperture describes the size of the area being sampled and should be set to the largest size that does not extend beyond the sample.<sup>44</sup>

Sample preparations that should be controlled and considered include the slide and coverslip composition, the mounting media, and the sample itself. Common slide and

coverslip materials include quartz and glass. Both materials have been found to provide minimally different emission spectra across the visible wavelengths.<sup>44</sup> Quartz has been found to perform better in the UV region, but is also more expensive than traditional glass slides. Three mounting media are commonly used for microspectrophotometric sample preparation: distilled water, xylene, and glycerin. In general, it is recommended that the mounting media used have a refractive index that is similar to the material being analyzed.<sup>44</sup> Other media such as refractive index liquids and Permount<sup>®</sup> have also been used.<sup>33,44</sup> Sample-solvent interactions should also be considered with regard to sample degradation, bleaching, and downstream analysis. Sample effects observed in fibers include pleochroism and should be considered with respect to the sample being analyzed.<sup>42,44</sup> Pleochroism is a change in absorption color that corresponds with a change in the vibrational direction of light.<sup>45</sup> The most important aspect of all of these factors is that they remain consistent throughout a single experiment or are otherwise the same when samples are compared to one another.

Other factors such as ambient temperature, bulb degradation, and bulb replacement cannot be controlled. These factors would not affect typical forensic analysis because they are stable in the short term.<sup>44</sup> Research involving microspectrophotometry cannot be performed in a short enough time span to completely avoid the influence of these factors. Efforts can be taken to minimize and track these impacts. Daily colorimetric and photometric calibrations that check the location and intensity of spectral bands are not only important in the detection of any unexpected instrumental problems, but also in the tracking of bulb degradation over time. When a bulb has become overly degraded or has otherwise reached the end of its life, reference scans collected as part of

daily calibrations can be used to realign bulbs such that the position allows for as similar a spectrum to previous bulbs as possible. The collection of dark and reference scans during calibration and regularly throughout use minimize the impact of environmental changes such as temperature.<sup>44</sup> Even with these difficult to control variables there is an abundance of research using microspectrophotometers available which provide insight into its validity, limitations, and applications.<sup>25,26,42,44</sup>

The number of samples and corresponding scans collected as well as how resultant data is analyzed is another key component to microspectrophotometric analysis of forensic samples. There are no formal guidelines or specifying the number of spectra that should be collected for any number of samples.<sup>26</sup> However, in terms of fibers, it has been recommended that data is collected from five to ten fibers.<sup>29</sup> It has also been suggested that a minimum of 5 spectra should be collected from each fiber.<sup>46</sup> Accounting for both suggestions, it can be assumed that the collection of 50 spectra may serve as an adequate representation of a sample. This number of spectra was used in the two previously mentioned studies relating to microspectrophotometric analysis of tapes and is currently used as the standard at the McCrone Research Institute.<sup>16,33</sup> The number of spectra collected should always be decided with consideration towards the variability of the samples in question. When multiple spectrum are collected, they are typically averaged and interpreted visually based on experience and expertise.<sup>26,29,30,33,44</sup> Various means of numerical based comparisons have been performed such as comparisons of absorption at peak and band maxima.<sup>26</sup> There is a push for the development of statistical approaches for the interpretation of microspectrophotometric data, but at this time no published standards are available.



The impact of the instrument on the sample must also be considered. Microspectrophotometry is generally considered to be a non-destructive analysis method.<sup>26,29,30</sup> However, a study has documented the occurrence of photobleaching of fiber samples.<sup>47</sup> Photobleaching occurs when there is color loss of a sample. In the context of microspectrophotometry, the ultraviolet light that focuses on the sample causes the sample to lighten in color. This phenomenon should be considered, and care should be taken to avoid sample degradation when working with a microspectrophotometer. That is, sample exposure to high levels of electromagnetic radiation originating from the microspectrophotometer light source should be minimized and samples should be screened for vulnerability to photobleaching prior to study. Beyond this, no other destructive qualities of microspectrophotometric analysis on samples have been documented.

### III. MATERIALS

The following instruments, tools, and consumables were used as described in the subsequent sections.

#### A. Instruments and Tools

Craic FLEX<sup>TM</sup>/508 PV<sup>TM</sup> Microspectrophotometer

- The following parameters were chosen based on instrument design and installation setup:
  - Xenon transmitted light source
  - 36x Reflecting objective
  - Instrument configured for transmission through the sample
  - Field diaphragm set to just outside the field of view
    - This position was determined during the initial instrument installation and bulb adjustments. It remains fixed throughout the course of experimentation and is only adjusted during bulb changes/adjustments in an effort to return the reference spectrum to the original conditions reached during installation.
  - Condenser aperture set to just below 0.2
    - This position was determined during the initial instrument installation and bulb adjustments. It remains fixed throughout the course of experimentation and is only adjusted during bulb changes/adjustments in an effort to return the reference spectrum to the original conditions reached during installation.
- The following parameters were designated during experimental setup and remained consistent throughout the course this project
  - Spectra collected in absorbance

- Absorbance was chosen over transmittance because it is the preferred standard for the field.<sup>29,43</sup> While the spectral data can be converted between absorbance and transmittance, absorbance values are preferred because they vary linearly with concentration while transmittance values vary logarithmically with concentration.
- Spectral range of 280-875 nm
  - This range was chosen because the focus of this project was on colored tapes, therefore the visible region (approximately 400-800 nm) was appropriate. The region was expanded slightly to allow for the collection of complete spectral bands.
- Integration time of 15 ms
  - This time was determined based on the reference spectrum's maximum peak location of 55000 counts. The "autoset optimize" feature of the microspectrophotometer was not used because this would adjust the integration time throughout the course of the project.
- Resolution factor of 0
  - This value was chosen because it provides the least adjusted data. A higher value would reduce noise but would average values on either side of the desired point, artificially smoothing the data.
- Instrument aperture of 4 (7.8  $\mu\text{m}$  x 7.8  $\mu\text{m}$ )
  - This size was chosen because it was the largest aperture size that would not extend beyond the sample.

- National Institute of Standards and Technology (NIST) Traceable microspectrophotometer standards, such as holmium, didymium, and neutral density filters provided by Craic were also used throughout the daily instrument calibration process
- Olympus BH2 Polarized Light Microscope
  - This microscope was used as a tool to measure cross sectional thickness of samples and as such the scaled 10x eyepiece was calibrated with a micrometer for the 10x, 20x, and 40x objectives.
- Leica EZ4D Stereolight Microscope
  - This microscope has a working range of 8-35x magnification.
- MAGNASONIC MGUC500 Digital Ultrasonic Jewelry & Eyeglass Cleanser
  - This sonicator produces 42,000 Hz waves in 90, 180, 280, 380, and 480 second cycles.
- American Optical Model 820 Rotary Microtome
  - This microtome was setup and used with appropriate maintenance as outlined by the user manual.<sup>48</sup>
  - A tungsten carbide microtome knife was used.
  - A blade angle of 45° was chosen based on trial and error as literature recommended.<sup>49</sup>

## **B. Pressure Sensitive Tapes**

The following tapes were commercially available in common retail stores across the Chicago area and purchased in spring of 2019:

- D1: Orange duct tape produced by Shur Tech Brands (Duck Tape) 3920-OR 151219; 1.88 in x 20 yd. Purchased from Home Depot.
- D2: Blue duct tape produced by Shur Tech Brands (Duck Tape) 3920-BL 180806; 1.88 in x 20 yd. Purchased from Home Depot.
- D3: Orange duct tape produced by Scotch 3M 31 18255; 1.88 in x 20 yd. Purchased from Target.
- D4: Blue duct tape produced by Scotch 3M 31 19004; 1.88 in x 15 yd. Purchased from Target.
- E1: Red electrical tape produced by Commercial Electric EE-100; ½ in x 20 ft-7 mil. Purchased from Home Depot.
- E2: Green electrical tape produced by Commercial Electric EE-100; ½ in x 20 ft-7 mil. Purchased from Home Depot.
- E3: Red electrical tape produced by Scotch 3M Professional Grade 35; ¾ in x 66 ft x 0.007 in. Purchased from Home Depot.
- E4: Green electrical tape produced by Scotch 3M Professional Grade 35; ¾ in x 66 ft x 0.007 in. Purchased from Home Depot.
- P1: Brown packaging tape produced by Scotch 3M 143; 1.88 in x 22.2 yd. Purchased from Walgreens.
- P2: Brown packaging tape produced by Shur Tech Brands (Duck Tape); 1.88 in x 30 yd. Purchased from Walmart.
- P3: Brown packaging tape produced by Elmer's; 1.89 in x 13.89 yd. Purchased from Walmart.

- P4: Brown packaging tape produced by Tartan 3M; 1.88 in x 54.6 yd. Purchased from Walmart
- M1: Blue masking tape produced by Scotch 3M 2090 39114144; 1.41 in x 60 yd. Purchased from Home Depot.
- M2: Brown masking tape produced by Scotch 3M 2020 01122019 200737; 0.94 in x 60.1 yd. Purchased from Home Depot.
- M3: Green masking tape produced by Shur Tech Brands (Frog Tape) 00075540G; 1.41 in x 60 yd. Purchased from Walmart.
- M4: Yellow masking tape produced by Shur Tech Brands (Frog Tape) 000755032G; 1.41 in x 60 yd.

The above tapes were repurchased from the same retailers in the fall of 2019 and given the designation of D1A, D2A, and so on. When possible, the repurchased ‘A’ tapes were confirmed to have different lot numbers than their predecessors. The tape which would have been designated at P3A was no longer available for purchase in stores or online and was therefore excluded. Additional tapes, not designated by abbreviations and primarily used in preliminary experimentation, include the following:

- Blue electrical tape produced by Commercial Electric EE-100; ½ in x 20 ft-7 mil. Purchased from Home Depot.
- Yellow electrical tape produced by Commercial Electric EE-100; ½ in x 20 ft-7 mil. Purchased from Home Depot.
- Red duct tape produced by Scotch 3M 3920-RD 180928; 1.88 in x 20 yd. Purchased from Home Depot.

- Yellow duct tape produced by Scotch 3M 3920-YL 191219; 1.88 in x 20 yd.  
Purchased from Home Depot.
- Black electrical tape produced by Commercial Electric EE-100; ½ in x 20 ft-7 mil.  
Purchased from Home Depot.
- White electrical tape produced by Commercial Electric EE-100; ½ in x 20 ft-7 mil.  
Purchased from Home Depot.
- Black duct tape produced by Scotch 3M 3960-BK 190319; 1.88 in x 60 yd. Purchased from Home Depot.
- White duct tape produced by Scotch 3M 3920-WH 190321; 1.88 in x 20 yd.  
Purchased from Home Depot.
- Silver colored duct tape produced by Scotch 3M 2900 180115; 1.88 in x 60 yd.  
Purchased from Home Depot.
- Patterned duct tape produced by Shur Tech Brands (Duck Tape) Real Tree AP-Xtra Duct Tape Camouflage Design 0007484225 31 18341; 1.88 in x 10 yd.
- Clear Packaging Tape produced by Scotch 3M 142; 1.88 in x 22.2 yd. Purchased from Walgreens.

The above tapes were also commercially available in common retail stores across the Chicago area and purchased in spring of 2019.

### **C. Other Consumables and Tools**

- Hardman Double/Bubble® Epoxy by Royal Adhesive & Sealants (# 04001)
  - Epoxy was used because the viscous mixture forms a thermoset chemical resistant solid capable of being cut by a microtome.

- This epoxy had a work time of three minutes and reached complete hardness after 24 hours.<sup>50</sup>
- The exact formula of the epoxy is unknown as it is a trade secret and unavailable to the public.<sup>51</sup>
- Silicon rubber embedding molds (standard flat rectangular silicon molds) by LADD research industries
  - 14 mm long x 5 mm wide x 3 mm deep
- Glass slides and coverslips
  - Grafo® microscope slides (# 3703-2P) with clear ground edges and clipped corners. 75 mm long x 25 mm wide x 1.0-1.2 mm thick
  - Thermo scientific microscope cover glass. 18 mm x 18 mm NO. 1.5.
- Quartz slides and coverslips
  - Craic microspectrophotometry grade quartz slides (# 0C005) with a spectral range of 200 to 2100 nm. 76 mm long x 25 mm wide x 1 mm thick
  - Craic microspectrophotometry grade quartz coverslips (# 0C015) with a spectral range of 200 to 2100 nm. 25 mm x 25 mm x 0.15 mm +/- 0.05 mm thick
- Cell culture slides (# 160005) by nunc™ Permanox®
  - 74 mm long x 25 mm wide
- Off-brand clingwrap
- Double sided tape by Scotch 3M
- Teflon coated, aluminum backed, stainless-steel single-edged razor blades by PERSONNA® GEM®
  - 3-Facet 0.009"



- Platinum coated, stainless-steel double-edged razor blades (# 72003-01) by Electron Microscopy Sciences
- Forceps (Dumont #5) by Fine Science Tools
- Paint brush (#3 Series) by WINDSOR & NEWTON
- Tungsten needles
  - Chemically sharpened
  - 99.9% 0.51 mm diameter tungsten wire (# 73800) by Electron Microscopy Sciences
  - Held in a Moria nickel plated needle holder (# 26016-12) by Fine Science Tools
- Xylenes isomers (247642-4L-CB Sigma Aldrich)  $\geq 75.0\%$
- Hexane, mixture of isomers (227064-2L Sigma Aldrich)  $\geq 99\%$
- Distilled water (store bought)

## IV. PROOF OF CONCEPT: PRELIMINARY METHODS AND RESULTS

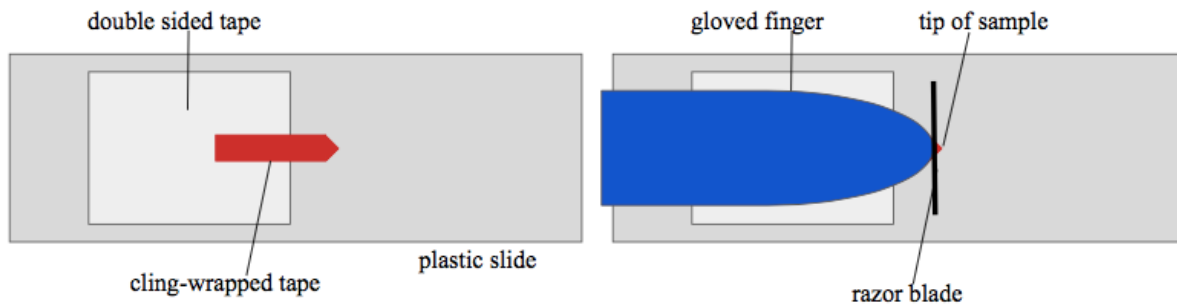
### A. Sample Preparation

#### 1. Sample Cross Sectioning

##### a. Hand Cross Sectioning

Current literature suggests that hand cross sectioning of tapes is best achieved with the aid of freezing by liquid nitrogen and that without liquid nitrogen, the thinnest cuttings that may be obtained range from 100  $\mu\text{m}$  to 200  $\mu\text{m}$ .<sup>52</sup> However, following hand-cross sectioning techniques implemented at the McCrone Research Institute, it was found that cross sections of 15  $\mu\text{m}$  to 20  $\mu\text{m}$  can be readily obtained without the use of liquid nitrogen. This method involved adhering a piece of tape to a piece of off-brand cling-wrap. The cling-wrapped tape was then cut down to a roughly 1 mm x 10 mm rectangle and adhered to a piece of double-sided tape on a plastic slide such that half the length of the rectangle was hanging off the tape and over the slide. The portion hanging over the slide was then cut to a point. Under a stereo microscope, cross sections of the tape were cut using a double-edged razor blade supported by one gloved finger in a chopping motion. See Diagram 3 showing this setup below. Once cut, the cuttings were handled with a tungsten needle.

Diagram 3.) Hand Cross Sectioning Setup



To assess the variability in hand cross sections, an experienced analyst with over fifteen years of training and an inexperienced analyst who had just learned this technique made ten cross sections of duct, electrical, packaging, and masking tapes. The inexperienced analyst repeated this process six months later to check for improvements with increased experience. Cross sectional thickness was measured with calibrated scaled oculars on a polarized light microscope. The scaled oculars were calibrated using a stage micrometer: The stage micrometer was brought into focus and aligned with the scaled oculars such that the number of ocular scale divisions were found to be equal to a number of stage micrometer divisions. The number of micrometers associated with each ocular scale division was then calculated. The tape cross sections were placed on their sides on a piece of double-sided tape on a glass slide and measured with the calibrated scaled oculars. For the complete dataset see Table 3 in appendix A. Hand cross sectioning is easier and faster than microtoming but depending upon the skill of the analyst and type of tape it may not be as controllable. The amount of variability between hand cut cross sectioned tapes is highlighted and summarized in Table 1 on the following page.

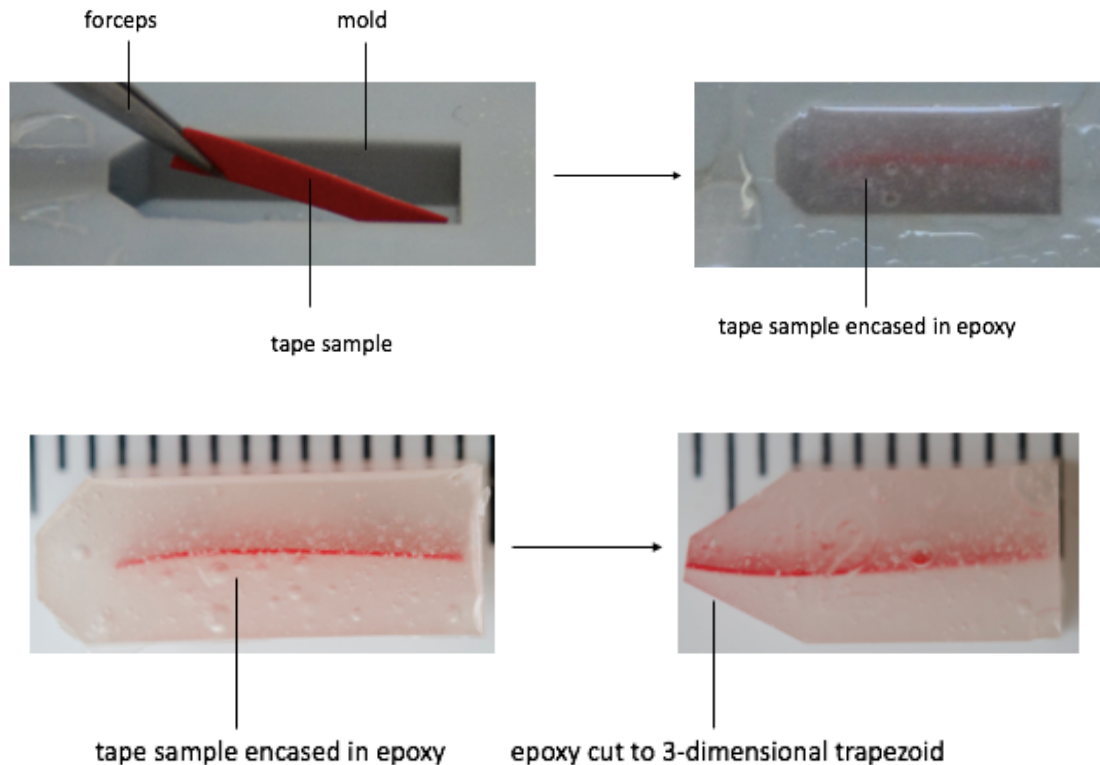
Table 1.) Hand Cross Sectioning Range of Variability Summary

		Maximum (μm)	Minimum (μm)	Range of Variation (μm)
Experienced Analyst	Duct Tape	15	35	20
	Electrical Tape	20	45	25
	Packaging Tape	15	30	15
	Masking Tape	20	45	25
Inexperienced Analyst	Duct Tape	20	70	50
	Electrical Tape	20	50	30
	Packaging Tape	20	70	50
	Masking Tape	50	100	50
Inexperienced Analyst After 6 Months	Duct Tape	20	45	25
	Electrical Tape	20	45	25
	Packaging Tape	15	45	30
	Masking Tape	30	55	25

#### b. Microtome Cross Sectioning

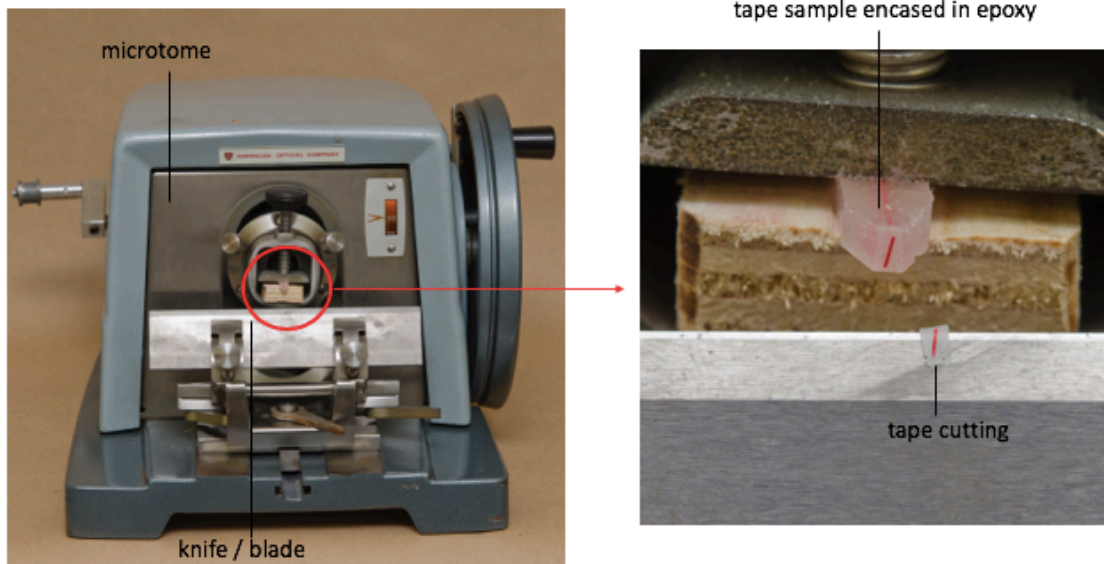
To prepare the tape samples for microtoming, roughly 1 mm x 10 mm rectangles of tape were placed vertically into an elongated 3-dimensional trapezoidal mold and encased in epoxy. See diagram 4 showing this setup on the following page. The epoxy was allowed to harden for at least 24 hours. After hardening, one side of the epoxy-molded tape sample(s) was cut into a point similar to the shape used during hand cross sectioning. See diagram 4 showing this setup on the following page.

Diagram 4.) Epoxy Embedding Setup



The sample was then secured in the microtome and cut to a desired thickness. Thickness' ranged from 5  $\mu\text{m}$  to 50  $\mu\text{m}$ . The epoxy encased tape sample was secured with the clamp present on the microtome as well as an added piece of wood which allowed for the sample to be completely secure and free from movement. See diagram 5 showing the microtome setup on the following page. After the sample was secured, the rotary arm of the microtome was rotated such that the sample moved forward and down over the blade. The rotary arm was moved at a speed that allowed the blade to cut through the sample in a single motion. After a cut was made, the sample was collected and moved to a glass slide using a combination of forceps and a paint brush. The sample was then gently removed from the epoxy using a combination of forceps and a tungsten needle under a stereo microscope.

Diagram 5.) Microtome Setup



To ensure that the epoxy was not interfering with the tape sample's spectra, the spectra of samples molded in epoxy and microtomed to 30  $\mu\text{m}$  were compared to the spectrum of samples hand-cross sectioned to the same measured thickness. These comparisons showed no considerable differences in spectra. See Figure 1 in appendix A. Microspectrophotometric data for the epoxy itself was also collected. See Figure 2 in appendix A.

During the development of this method, it was noted that the adhesive portion of the tape samples combined with the epoxy and made microtoming with any degree of control difficult. This was especially true for the duct and masking tape samples. In order to achieve better cross sections, the adhesive portion of the tape samples was to be removed.

## 2. Adhesive Removal

The adhesive portion of tapes may cause evidentiary tape samples to be difficult to work with and handle: Tapes may come into a forensic laboratory tangled and stuck

together or adhered to other items. Forensic scientists also may want to analyze the adhesive portion of tape samples separately from the backing portion. An initial step in the analysis of tapes has commonly been the removal and/or separation of the backing and adhesive layers of the sample. This step has the potential to be congruent with or subsequent to the sample preparation for the microspectrophotometric analysis of tape samples. If tape samples are to be analyzed by microspectrophotometry directly through the sample, the adhesive portion of the tape should be removed because it will interfere with analysis by reducing the amount of light that may pass through the sample and/or by adding additional colors to the sample, potentially altering the resultant absorbance spectra. If tape samples are to be analyzed cross sectionally, adhesive removal may not be as imperative since the adhesive present would not directly interfere with analysis. In all cases, adhesive removal may be beneficial for ease of sample handling including cross sectioning. Literature suggests two common methods for adhesive removal: mechanical separation aided by freezing or heating or separation by the application of various solvents such as xylene or hexane. For the purpose of this research, an adhesive removal technique that was minimally destructive to the tape backing layer, was applicable to all four tape types being examined, and was reasonably fast and straightforward was desired.

While mechanical separation may be possible for tapes with thicker adhesive layers such as duct tapes, tapes with thinner adhesive layers such as electrical tapes are much less attune to mechanical separation. In addition to being time consuming, mechanical separation with tools such as forceps or tungsten needles may cause crushing, stretching, or piercing of the sample backing. Each of which would cause a change in sample thickness and potentially cause a change in the resultant absorbance spectra of the

sample. For these reasons, mechanical separation was initially ruled out as a means of adhesive removal for this research.

Adhesive removal aided by solvents has the potential to work on any type of tape, although, no previous assessment on solvent bleaching of tape-backing layers was found to be published. If performed via sonication, as literature recommends, this kind of adhesive removal may also serve to be fast and straightforward.<sup>1,19</sup> Two solvents, xylene and hexane, are mentioned for use in the removal of adhesive from tapes. Sonication has also been mentioned, but no specifics of the technique have been found to be reported. The effects on the tape backing layer, specific type of solvent, and sonication protocol were determined for duct, electrical, packaging, and masking tapes.

#### a. Type of Solvent

Separate cuttings of approximately 20 mm length x tape width of each tape type were sonicated for 90 seconds in 30 mL of xylene and hexane respectively. This amount of solvent was used because it was the minimum amount necessary to completely cover the tape samples in the sonicator used. An initial time of 90 seconds was chosen because it was the shortest cycle setting on the sonicator.

By visual examination, it was immediately apparent that sonication in xylene was destructive to the backing layer of the tape samples. The tape samples sonicated in xylene were lighter in color than they had been prior to sonication and the xylene liquid appeared to be colored. It was also observed that the electrical tape backing felt brittle after sonication in xylene. Xylene was therefore excluded as a possible solvent for the removal of adhesive for this research and sonication protocol determination was continued with the use of hexane.



By visual examination, sonication in hexane did not appear to have any effect on the backing layer of any of the tape samples. The tape samples sonicated in hexane appeared to be the same color prior to sonication and the hexane liquid appeared cloudy white.

#### b. Sonication Protocol

Separate cuttings of approximately 20 mm length x tape width of each type of tape were sonicated in 30 mL of hexanes for 90, 180, 280, 380, and 480 seconds respectively. After 90 seconds, the adhesive layer of the duct, electrical, masking, and packaging tapes was majorly removed as determined by feel of sample tackiness with gloved hands. However, some of the samples felt lightly tacky on the originally non-adhesive side of the tape. Length of sonication did not aid in the removal of this tacky feeling, but a second 90 second sonication wash in 30 mL of fresh hexane was found to remove any remaining adhesive, as determined by feel of sample tackiness on both sides of the tape backings.

Through this process, it was observed that the duct tape samples had a tendency to curl inwards and if they were allowed to curl adhesive removal was not achieved in two 90 second cycles. Curling could be prevented by gentle handling with gloved hands or forceps. The removal of the adhesive layer from the duct tape did not guarantee the removal of the scrim layer, but without the adhesive the scrim could be easily pulled off of the backing with gloved hands or forceps.

It was determined that the best means of adhesive removal for duct, electrical, masking, and packaging tapes was two consecutive 90 second cycles of sonication in approximately 30 mL of hexane (replaced in between sonication cycles). However, while

the backing of the samples appeared visually unaffected by this treatment, microspectrophotometric comparisons of samples subjected to and not subjected to this treatment gave an indication that some bleaching effects were occurring. See Figure 3 in appendix A. As such, it was concluded that the application of solvents to tapes in an effort to remove the adhesive layer should not be performed on tapes intended for microspectrophotometric analysis and another means of adhesive removal should be performed.

### c. Physical Barrier

Similar to techniques used for hand cross sectioning, tape samples were adhered to cling wrap to cover the adhesive without removing it. This physical barrier made the sample easy to handle and work with without physically damaging the sample.

Additionally, all tape types prepared in this manner and molded in epoxy per previously described methods could be microtomed with a fair degree of control. This was determined by preparing a duct, electrical, packaging, and masking tape sample as previously described and making ten cuts at four different thickness': 50  $\mu\text{m}$ , 30  $\mu\text{m}$ , 15  $\mu\text{m}$ , and 5  $\mu\text{m}$ . These cuttings were moved with a combination of forceps, tungsten needles, and paint brushes and measured using a calibrated scaled ocular in the same way that the hand cut cross sections were measured. These cuttings were largely found to be equal to the expected thickness dictated by the microtome, but some cuttings did vary throughout the length of the cut. These differences in width were all  $\pm 5 \mu\text{m}$ . Spot checks of microtomed section thicknesses were also performed throughout the entirety of this research anytime a change in microtome thickness or a change in sample being cut

was made. All spot checks resulted in cuts that were within  $\pm 5 \mu\text{m}$  of the expected thickness dictated by the microtome.

## **B. Microspectrophotometric Analysis**

Prior to any sample analysis, a daily instrumental calibration was performed as recommended by the manufacturer. This calibration was performed after the instrument had a 30-minute warm up period. The process started with an internal instrumental calibration called ‘auto calibration transmission’ by the Lambda Scan software. A quartz slide with a sharpie marker cross drawn on it was brought into focus and moved to a clear portion of the slide. Dark and reference spectra were collected followed by colorimetric checks of holmium and didymium and photometric checks of neutral density filters. If the holmium and didymium bands were within a tolerable level of the expected positions and if the photometric bands were within a tolerable level of the expected intensity the instrument passed its internal calibration. The tolerable level of wavelength position, per the manufacturer, is 3.0 nm for holmium and didymium. Tolerable levels of absorbance intensity, per the manufacturer, are 0.026, 0.047, and 0.095 for the lowest to highest photometric intensities respectively.

After this calibration, a second check of the dark, reference, holmium standard, and didymium standard was performed. The purpose of this second check was to monitor absorbance band intensity and location under normal experimental conditions. This check used a tape cross section mounted with distilled water on a glass slide with a glass coverslip. The tape sample was brought into focus and then moved to a clear portion of the slide. Dark and reference spectra were collected followed by holmium and didymium

spectra. These collected spectra were then compared to previous calibrations to ensure no band shift of intensity or wavelength was occurring and to monitor possible bulb degradation. If any issues were noticed during either calibration the problems were troubleshoot and addressed accordingly.

Throughout the entirety of this research, bulb changes were performed due to a combination of natural bulb degradation and limitations to the lifespan of the bulb. No other major changes to the instrument occurred and the influence of the bulb changes were minimized with careful bulb alignment and installation. To further minimize the impact of possible long-term stability changes, an effort was made to collect complete datasets in a single day and minimize the amount of time in between collection of datasets which would be directly compared to one another.

#### 1. Slide Type and Collection Range

At this time, distilled water was chosen as the mounting medium because of its low potential to interfere with the sample in regard to bleaching and downstream analyses as well as its standard use at the McCrone Research Institute. The next experimental condition addressed was the slide and coverslip composition. Two materials, glass and quartz, are common for microspectrophotometric sample preparation. Absorbance spectra were collected for holmium and didymium through a quartz slide and coverslip, a glass slide and coverslip, and a glass slide and coverslip with an ultraviolet filter. A tape cross section was mounted in distilled water on the respective slide, covered with the corresponding coverslip, and moved to a clear section of the slide. Five scans were collected. Since there was little to no variance in the collected five scans additional scans were not necessary. See Figure 4 and Figure 5 in appendix A.

No difference in absorbance in the visible spectrum was observed between quartz and glass slides and coverslips. Outside of the visible spectrum, on the lower end, there was some difference in absorption between quartz and glass slides and coverslips. The addition of the ultraviolet light filter did affect the absorbance spectrum in the visible region. Since there would be no added benefit of using the more expensive and labor-intensive quartz slides and coverslips it was decided that glass slides and coverslips would be used for the remainder of the experiment. Since the ultraviolet light filter did affect results, it was decided that the filter would only be used in between the collection of spectra to help reduce possible photobleaching.

## 2. Dark and Reference Collection Frequency

After the use of distilled water and glass slides was established, the next experimental condition addressed was the frequency for which dark and reference scans would be collected. Dark and reference scans are collected in variable frequency ranging from collection prior to each scan or collected needed as experimental conditions change. Manufacturer recommendations are to collect a new dark and reference spectrum when moving to a new field of view. Internal procedures for collection frequency at the McCrone Research Institute call for one set of dark and reference scans to be collected for each fiber scanned as determined by internal proof of concept studies.

Absorbance spectra were collected for 15  $\mu\text{m}$  cross sections of blue electrical tape with one dark and one reference spectrum collected per cutting, one dark and one reference spectrum collected total, and one dark and one reference spectrum collected per field of view. For each condition a total of fifty scans were collected over ten cross sections. See Figure 6 in appendix A. All three conditions result in indistinguishable

spectra. Since there was no observed benefit of increasing the dark and reference scan collection frequency beyond the frequency with which they were currently being collected at the McCrone Research Institute, it was decided that a set of dark and reference scans would be collected for each cross section of tape.

### 3. Preliminary Tape Thickness Ranges

At the time of this data collection, no information had been published in regard to what thickness of tapes would provide useable microspectrophotometric data. As such, a spectrum was collected directly through an array of different colored duct, electrical, packaging, and masking tapes without their adhesives removed. See Figures 7 through 13 in appendix A. When spectra were collected directly through the duct and electrical tape samples the instrument was over saturated as seen as noise in the spectral bands. The packing tape sample's colored backing was translucent and thinner than 50  $\mu\text{m}$  without being cross sectioned: spectrum collected directly through this sample did not oversaturate the instrument and provided non-noisy data. The masking tape samples did not oversaturate the instrument when the spectra were collected directly through the samples.

Initially tapes being analyzed directly through the samples were stuck to glass slides without the presence of a coverslip or distilled water. It was observed that without the presence of distilled water and a coverslip the duct tape backings melted during exposure to the xenon light. In order to avoid melting tape samples and keep consistency between samples analyzed as cross sections and samples analyzed directly through, samples analyzed directly through the tape were also mounted in distilled water under a glass coverslip.

The same array of duct, electrical, and masking tapes were cross sectioned to 50  $\mu\text{m}$ , 40  $\mu\text{m}$ , 30  $\mu\text{m}$ , 20  $\mu\text{m}$ , 10  $\mu\text{m}$ , and 5  $\mu\text{m}$ . The packaging tape was not cross sectioned because it was assumed that thinner sections would also provide non-noisy, albeit lower absorbance, spectra. The masking tapes were not cross sectioned to 10  $\mu\text{m}$  and 5  $\mu\text{m}$  because of their fragility which made removing the tape cuttings from the epoxy difficult. Five spectra were collected at each of the cross-sectional thicknesses. See Figures 12 through 17 in appendix A. All cross sectioned samples provided non-noisy data which did not immediately appear to oversaturate the instrument. This collection was not intended to serve as a comprehensive analysis of the tape samples, but rather served to provide insight as to what thicknesses may be appropriate for further microspectrophotometric analysis.

#### 4. Photobleaching Check

The samples selected to be studied further included four rolls of duct tape, four rolls of electrical tape, four rolls of packaging tape, and four rolls of masking tape. These rolls were given the nomenclature of D1, D2, D3, D4, E1, E2, and so on. Prior to the final selection of these tape rolls, they were checked for susceptibility to photobleaching. A 15  $\mu\text{m}$  cross section of each tape was exposed to the full intensity of the microspectrophotometric light for ten minutes. During a typical spectrum collection, a sample is exposed to the microspectrophotometric light source for less than two minutes. During this exposure, spectra were collected at approximately every minute through the TimePro feature of the Lambda Scan software. See Figure 18 through 33 in appendix A. The samples all showed minimal photobleaching over the allotted time. To ensure the repeatability of these results, the process was repeated for samples D1, E1, P1, and M1

four additional times. No differences in photobleaching activity were observed. These samples were therefore found to be not overly susceptible to photobleaching and were appropriate for further microspectrophotometric analysis.

## 5. Achromatic and Patterned Samples

Achromatic and patterned tapes provide data that appears minimally useful in the visible region of the electromagnetic spectrum. Tapes that are clear, silver-colored, black, white, or contain printed designs were not found to provide useful data for the purposes of this study and were therefore excluded. These tapes were prepared and analyzed using previously described methods at a 30  $\mu\text{m}$  microtomed thickness. Clear tapes, such as clear packaging tape, do not absorb or transmit any wavelengths of light in the visible spectrum and as such only produce near-baseline data. See Figure 34 in appendix A. Black, white, and silver-colored tapes such as duct tapes and electrical tapes, are primarily colored by additives such as carbon black, titanium dioxide, and aluminum powder over any other colorants. See Figure 35 through 39 in appendix A. Tapes that contain designs, such as patterned duct tapes, are the printed result of a thin layer of many different colored dots and are not indicative of the color of the backing of the tape itself. See Figure 40 in appendix A. Further analysis of these non-colored tapes was beyond the scope of this project, but this does not preclude these kinds of tape's potential to provide any useful microspectrophotometric data.



## V. EXPERIMENTAL METHODS

### A. Optimization of Sample Preparation

The conditions for this research were decided based on available published information, internal procedures used at the McCrone Research Institute, and data observed during the proof of concept portion of this study. These conditions were chosen because they were thought to be the most suitable for microspectrophotometry of tapes, but their use in this study does not preclude the use of other conditions for similar work.

It was found that cross sectioning the samples was necessary for the collection of microspectrophotometric data relating to duct and electrical tapes. While microspectrophotometric data free of noise could be obtained without cross sectioning packaging and masking tapes, cross sectioning these samples could be necessary in some instances and may allow for the collection of better microspectrophotometric data. Cross sections of useful thicknesses were successfully cut using both hand cross sectioning and microtoming. Microtoming allowed for more controlled cross sections compared to the more variable hand cut cross sections, but cross sectioning by hand is faster and easier than microtoming. At this time, it was not known what range of thickness variation could be detected by a microspectrophotometer. If the amount of variability that contributes a noticeable difference to microspectrophotometric analysis of tapes is less than that which occurs through hand cross sectioning of tapes, hand cross sectioning may be a preferred sample preparation method to microtoming. If the amount of variability that contributes a noticeable difference to microspectrophotometric analysis of tapes is more than that which occurs through hand cross sectioning of tapes, microtoming may be a preferred sample preparation method to hand cross sectioning. If microtoming is found to be

preferable, then the exact thickness or range of thicknesses that a tape sample should be cut to must also be decided.

To answer these final questions related to sample preparation of tapes for microspectrophotometric analysis, the previously mentioned four different rolls of duct tapes, electrical tapes, packaging tapes, and masking tapes were analyzed. When possible, each tape was microtomed to four different thickness: 50  $\mu\text{m}$ , 30  $\mu\text{m}$ , 15  $\mu\text{m}$ , and 5  $\mu\text{m}$ . These thicknesses were chosen because they allow for assessment of the variability of 10  $\mu\text{m}$ , 15  $\mu\text{m}$ , 20  $\mu\text{m}$ , 25  $\mu\text{m}$ , 35  $\mu\text{m}$ , and 45  $\mu\text{m}$ . Ten cross sections of each thickness were prepared as previously described, and five spectra were collected from each cross section to total fifty spectra per sample. See Figure 41 through 56 in appendix B.

#### **B. Intra- and Inter-Roll Variation**

Based on the results observed, duct and electrical tapes were prepared to 30  $\mu\text{m}$  microtomed cross sections, packaging tapes were analyzed directly through the samples, and masking tapes were prepared by hand cross sectioning.

Samples from the approximate beginning, middle, and end of each roll of tape were prepared. The beginning of a secondary tape roll of the same type, style, and manufacture were also prepared. Fifty spectra were collected for each sample similarly to the process previously described. See Figures 57 through 72 in appendix C and Figure 73 through 87 in appendix D.

## VI. EXPERIMENTAL RESULTS

### A. Optimization of Sample Preparation

Visual Assessments of the microspectrophotometric data was performed based on current recommended standards in the field for fibers and paint.<sup>29,30</sup> All observations were made with consideration to the visible region of the electromagnetic spectrum (400-800 nm). When performing visual assessments of microspectrophotometric data, fifty spectra were averaged and overlaid and the position of band maxima and minima, width, and intensity were compared. The standard deviation of the fifty spectra was calculated and doubled to represent the spread of the data, where +/- two standard deviations is expected to cover 95 percent of sample variation. A spectral inclusion, or spectra found to be indistinguishable, occurred when the curve shape and absorbance values of one sample were found to be within the range of variation of another sample. See Figure 1 in appendix A for an example of spectra found to be indistinguishable. Conversely, a spectral exclusion, or spectra found to be distinguishable, occurred when the curve shape and absorbance values of one sample were found to be outside of the range of variation of another sample. See Figure 3 in appendix A for an example of spectra found to be distinguishable. There is also the possibility of an inconclusive result which occurs when there are no clear points of comparison in either spectra. See Figure 34 in appendix A for an example of a spectrum with no clear points of comparison.

Prior to any comparisons a spectrum must be assessed for its quality or acceptability. A high quality or acceptable spectra is one which falls within the dynamic range of the instrument. There are no exact ranges which constitute the dynamic range of the instrument, however, internal communication with the manufacturer of the

microspectrophotometer used in this study suggest that absorbance intensities below 1.5 are more accurate. This was also reflected in the internal photometric calibration of the instrument which called for a higher tolerance threshold at higher absorbances compared to lower absorbances. Per the manufacturer, at an absorbance intensity of 1.0 the instrument accepts variation of 0.095, at an absorbance intensity of 0.5 the instrument accepts variation of 0.047, and at an absorbance intensity of 0.1 the instrument accepts variation of 0.026. Despite these possible instrumental limitations, there have been publications that report microspectrophotometric spectral results with absorbance intensities beyond 1.5 by the manufacturer and by other scientists. As such, it may be desirable to have spectral data with absorbances below 1.5, but spectral data with absorbances beyond this may still be acceptable if there are no other indications of instrumental problems such as noise.

Other observations may include spectral alterations such as band flattening and band fusion. Band flattening is the leveling out of band maxima and can be observed in Figure 45 in appendix B by the comparison of band maxima between 480 and 580 nm. Band fusion is the combining of separate bands and may be compared to a loss of resolution between bands. This is exemplified in Figure 42 in appendix B by the comparison of band maxima between 480 and 780 nm.

#### 1. Duct Tape

The absorbance spectra generated for D1, D2, D3, and D4 were assessed visually as described above. See Figure 41 through 44 in appendix B. All thicknesses assessed (50  $\mu\text{m}$ , 30  $\mu\text{m}$ , 15  $\mu\text{m}$ , and 5  $\mu\text{m}$ ) provide absorbances within the dynamic range of the instrument with regard to lack of noise. However, at all thickness for both the Shur Tech

and Scotch 3M blue duct tapes some absorbance intensities beyond 1.5 did occur. At thicknesses of 50  $\mu\text{m}$  for both the Shur Tech and Scotch 3M orange duct tapes absorbance intensities beyond 1.5 occurred, but thinner section's absorbances were below 1.5. For all duct tape samples, the wavelength location of band maxima, minima, and width were unaffected by differences in sample thickness.

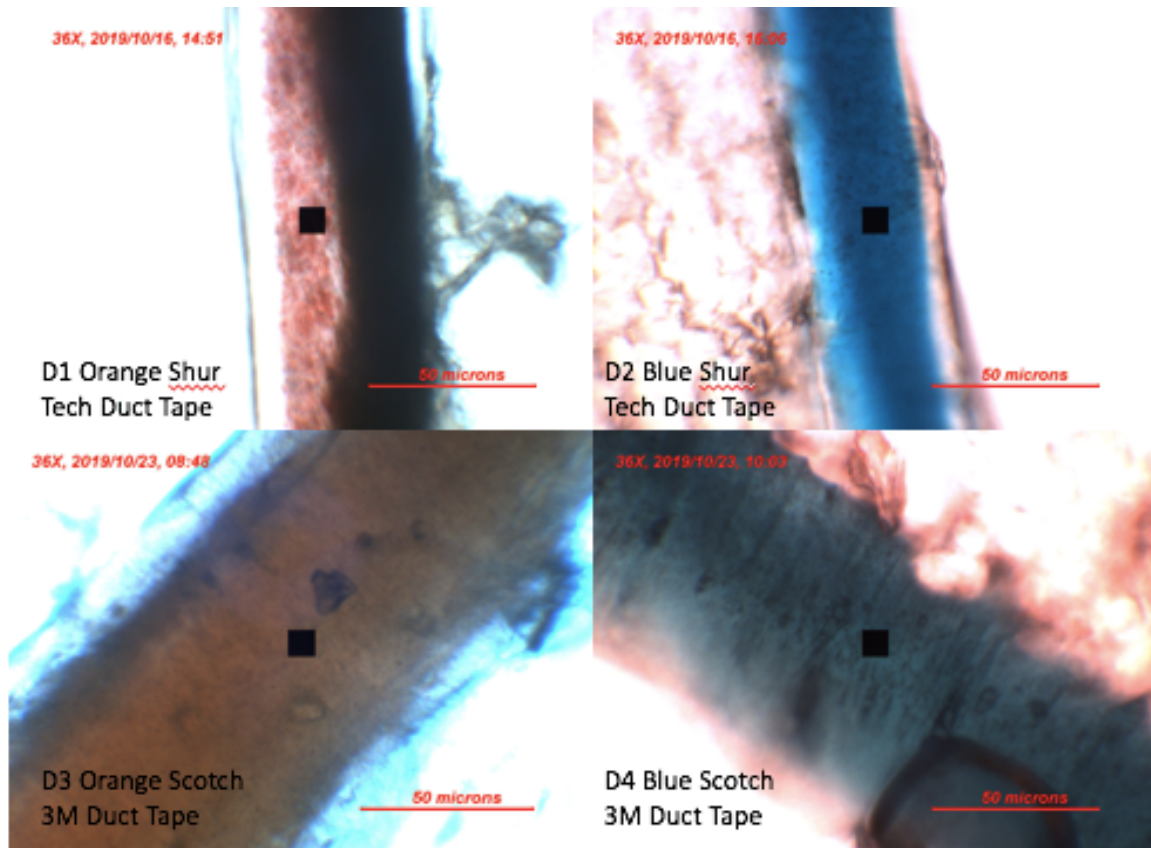
Some spectral alterations with change in sample thickness were observed in the duct tape samples. Band flattening at thicknesses below 15  $\mu\text{m}$  were observed in the orange Scotch 3M duct tape (D3). Band fusion, or a loss of band resolution, was observed in thicker sections of blue Shur Tech duct tape (D2), this occurred most strongly at 50  $\mu\text{m}$  with complete band fusion.

Sample thickness affected absorbance intensity for all duct tape samples. All duct tape samples show some degree of overlap into another thickness' range of variability. That is, when comparing the 5  $\mu\text{m}$  and 50  $\mu\text{m}$  cuttings of same duct tape samples, a difference in thickness of 45  $\mu\text{m}$  across samples could be expected to have some spectral overlap. However, in some samples, this amount of sample thickness difference would result in a spectral exclusion based on previously defined parameters. Additionally, a difference of 45  $\mu\text{m}$  across a single sample would greatly increase the sample's absorbance spectrum variation. In the orange Shur Tech duct tape (D1) a comparison between samples cut to 15  $\mu\text{m}$  and 30  $\mu\text{m}$  would result in a spectral exclusion. In the blue Shur Tech duct tape (D2) a comparison between samples cut to 15  $\mu\text{m}$  and 50  $\mu\text{m}$  would result in a spectral exclusion. In the orange Scotch 3M duct tape (D3) and the blue Scotch 3M duct tape (D4) a comparison between samples cut to 5  $\mu\text{m}$  and 30  $\mu\text{m}$  would result in a spectral exclusion.

The above observations indicate that, for duct tapes, samples cross sectioned to thicknesses between 5  $\mu\text{m}$  and 50  $\mu\text{m}$  have the potential to provide quality spectral data and that cross sections of duct tape samples should not vary by more than 15  $\mu\text{m}$  thickness. Meaning, if two duct tape samples are being compared, they should not vary in thickness by more than 15  $\mu\text{m}$ .

During the collection of each spectrum images of the sample were also collected through the microspectrophotometer. Representative images of the duct tape samples microtomed to 30  $\mu\text{m}$  can be seen in Diagram 6 below.

Diagram 6.) Duct Tape Images



## 2. Electrical Tape

The absorbance spectra generated for E1, E2, E3, and E4 were assessed visually as previously described. See Figures 45 through 48 in appendix B. All thicknesses assessed (50  $\mu\text{m}$ , 30  $\mu\text{m}$ , 15  $\mu\text{m}$ , and 5  $\mu\text{m}$ ) provide absorbances within the dynamic range of the instrument with regard to lack of noise. However, for the red Commercial Electric (E1), green Commercial Electric (E2), and red Scotch 3M (E3) electrical tapes thicknesses of 50  $\mu\text{m}$  resulted in average absorbance intensities above 1.5 and thicknesses of 30  $\mu\text{m}$  resulted in average absorbance intensities below 1.5 but a range of variation above 1.5. The green Scotch 3M (E4) electrical tape had average absorbance intensities below 1.5 for both thicknesses of 50  $\mu\text{m}$  and 30  $\mu\text{m}$  but a range of variation above 1.5 for both thicknesses. For all electrical tape samples, the wavelength location of band maxima, minima, and width were unaffected by differences in sample thickness.

Some spectral alterations with change in sample thickness were observed in the electrical tape samples. Band flattening at thicknesses at and below 15  $\mu\text{m}$  were observed in all electrical tape samples.

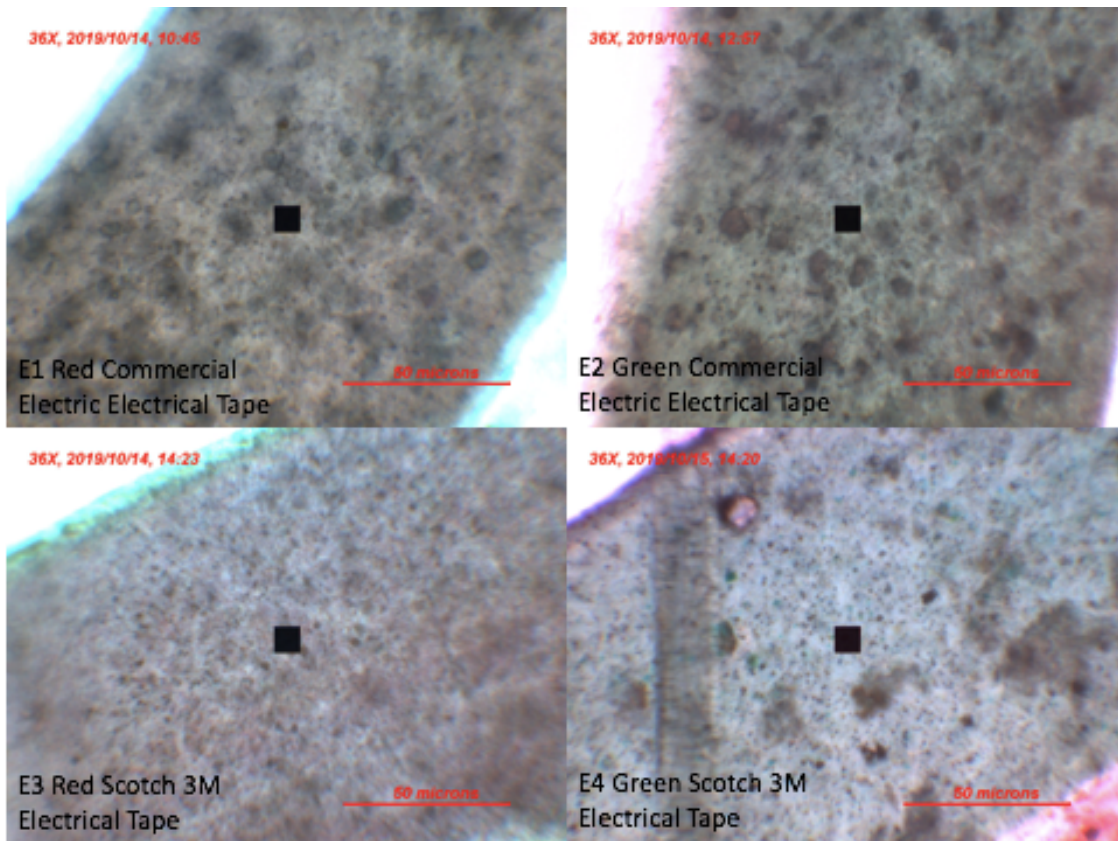
Sample thickness affected absorbance intensity for all electrical tape samples. Electrical tapes that varied in thickness by greater than 35  $\mu\text{m}$  showed no overlap. That is, when comparing the same electrical tape samples, a difference in thickness of 25  $\mu\text{m}$  across samples could be expected to have some similar spectral overlap. However, in some samples, this amount of sample thickness difference would result in a spectral exclusion based on previously defined parameters. Additionally, a difference of 25  $\mu\text{m}$  across a single sample would greatly increase the sample's absorbance spectrum

variation. For all the electrical tape samples, a comparison between samples cut to 15  $\mu\text{m}$  and 30  $\mu\text{m}$  would result in a spectral exclusion.

The above observations indicate that, for electrical tapes, samples cross sectioned to thicknesses between 5  $\mu\text{m}$  and 50  $\mu\text{m}$  have the potential to provide quality spectral data and that cross sections of electrical tape samples should not vary by more than 15  $\mu\text{m}$  thickness. Meaning, if two electrical tape samples are being compared, they should not vary in thickness by more than 15  $\mu\text{m}$ .

During the collection of each spectrum images of the sample were also collected through the microspectrophotometer. Representative images of the electrical tape samples microtomed to 30  $\mu\text{m}$  can be seen in Diagram 7 below.

Diagram 7. Electrical Tape Images





### 3. Packaging Tape

Preliminary microspectrophotometric analysis of packaging tapes indicated that their translucence would allow for direct through analysis to provide quality microspectrophotometric data. Analysis directly through a tape is influenced by the adhesive present and for this reason, cross sectional analysis was still performed.

The absorbance spectra generated for P1, P2, P3, and P4 were assessed visually as previously described. See Figure 49 through 52 in appendix B. All thicknesses assessed (50  $\mu\text{m}$ , 30  $\mu\text{m}$ , 15  $\mu\text{m}$ , and 5  $\mu\text{m}$ ) provide absorbances within the dynamic range of the instrument with regard to lack of noise. The brown Scotch 3M packaging tape (P1) and the brown Tartan packaging tape (P4) were colored based on the presence of colorant in the tape backing layer while the brown Shur Tech packaging tape (P2) and the brown Elmer's packaing tape (P3) were colored based on the presence of colorant in the tape adhesive layer. The physical barrier used in the sample preparation contained the adhesive layer of the tape at all thicknesses, but sample handling that kept the adhesive layer intact at 15  $\mu\text{m}$  and 5  $\mu\text{m}$  could not be reliably achieved. As such, samples P2 and P3 were only analyzed at thickness of 50  $\mu\text{m}$  and 30  $\mu\text{m}$ . For the brown Scotch 3M packaging tape (P1), thicknesses of 50  $\mu\text{m}$  and 30  $\mu\text{m}$  resulted in absorbance intensities above 1.5. For the brown Shur Tech packaging tape (P2), both thicknesses of 50  $\mu\text{m}$  and 30  $\mu\text{m}$  resulted in absorbance intensities above 1.5. For the brown Elmer's packaging tape (P3), both thicknesses of 50  $\mu\text{m}$  and 30  $\mu\text{m}$  resulted in average intensities below 1.5 but a range of variation above 1.5. For brown Tartan packaging tape (P4) a thickness of 50  $\mu\text{m}$  resulted in an average intensity above 1.5 and thicknesses of 30  $\mu\text{m}$  and 15  $\mu\text{m}$  resulted in average intensities below 1.5 but a range of variation above 1.5. For all

packaging tape samples, the wavelength location of band maxima, minima, and width were unaffected by differences in sample thickness, but there were very few clear points of comparison present in these spectra.

Some spectral alterations with change in sample thickness were observed in the packaging tape samples. Band flattening at thicknesses at and below 15  $\mu\text{m}$  were observed in both the brown Scotch 3M packaging tape (P1) and the brown Tartan packaging tape (P4) .

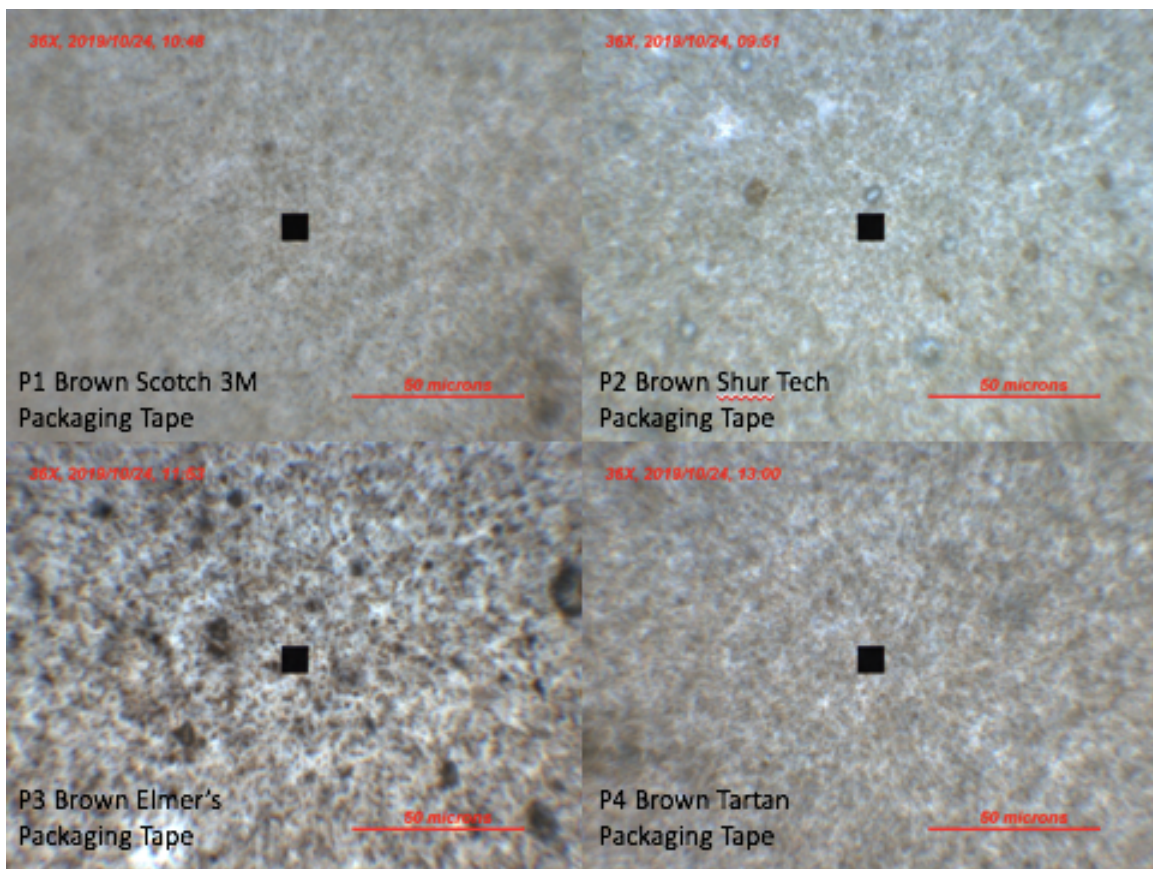
Sample thickness affected absorbance intensity for the brown Scotch 3M packaging tape (P1) and the brown Tartan packaging tape (P4), but the sample thickness did not appreciably affect the absorbance intensity for the brown Shur Tech packaging tape (P2) and the brown Elmer's packaging tape (P3). Packaging tape samples P1 and P4 did not overlap into another thickness' range of variability when they varied by more than 25  $\mu\text{m}$ . That is, when comparing the same packaging tape samples, a difference in thickness of 20  $\mu\text{m}$  across samples could be expected to have some similar spectral overlap. However, in some samples, this amount of sample thickness difference would result in a spectral exclusion based on previously defined parameters. Additionally, a difference of 20  $\mu\text{m}$  across a single sample would greatly increase the sample's absorbance spectrum variation. In both P1 and P4 a comparison between samples cut to 5  $\mu\text{m}$  and 15  $\mu\text{m}$  would result in a spectral exclusion.

Since packaging tapes are translucent, when possible, microspectrophotometric data should be collected directly through the sample. If this is not possible, the above observations indicate that, for packaging tapes, samples cross sectioned to thicknesses between 5  $\mu\text{m}$  and 50  $\mu\text{m}$  have the potential to provide quality spectral data and that cross

sections of packaging tape samples should not vary by more than 10  $\mu\text{m}$  thickness. Meaning, if two packaging tape samples are being compared, they should not vary in thickness by more than 10  $\mu\text{m}$ .

During the collection of each spectrum images of the sample were also collected through the microspectrophotometer. Representative images directly through the packaging tape samples can be seen in Diagram 8 below.

Diagram 8.) Packaging Tape Images



#### 4. Masking Tape

The absorbance spectra generated for M1, M2, M3, and M4 were assessed visually as previously described. See Figure 53 through 56 in appendix B. All thicknesses

assessed (50  $\mu\text{m}$ , 30  $\mu\text{m}$ , 15  $\mu\text{m}$ , and 5  $\mu\text{m}$ ) provide absorbances within the dynamic range of the instrument with regard to lack of noise. Previously, masking tape samples were not prepared to thickness less than 20  $\mu\text{m}$  because they were too fragile to remove from the epoxy. It was found that if they were cut out of the epoxy with a razor blade these thinner sections could be reliably handled. Despite this, due to its color similarity to the epoxy and its fragility, the brown Scotch 3M masking tape (M2) could only reliably be prepared at thicknesses of 50  $\mu\text{m}$  and 30  $\mu\text{m}$ . Additionally, due to its light color and its fragility, the yellow Shur Tech masking tape (M4) could only reliably be prepared at thicknesses of 50  $\mu\text{m}$ , 30  $\mu\text{m}$ , and 15  $\mu\text{m}$ . For the blue Scotch 3M masking tape (M1) a thickness of 50  $\mu\text{m}$  resulted in absorbance intensities above 1.5. For the brown Scotch 3M masking tape (M2) a thickness of 50  $\mu\text{m}$  resulted in an average absorbance intensity below 1.5 but a range of variation above 1.5. For the green Shur Tech masking tape (M3) thicknesses of 50  $\mu\text{m}$ , 30  $\mu\text{m}$ , and 15  $\mu\text{m}$  resulted in an average absorbance intensity below 1.5 but a range of variation above 1.5. For the yellow Shur Tech masking tape (M4) no thicknesses resulted in absorbance intensities above 1.5. For all masking tape samples, the wavelength location of band maxima, minima, and width were unaffected by differences in sample thickness.

Some spectral alterations with change in sample thickness were observed in the masking tape samples. Band flattening at thicknesses at and below 15  $\mu\text{m}$  were observed in M1 and M3.

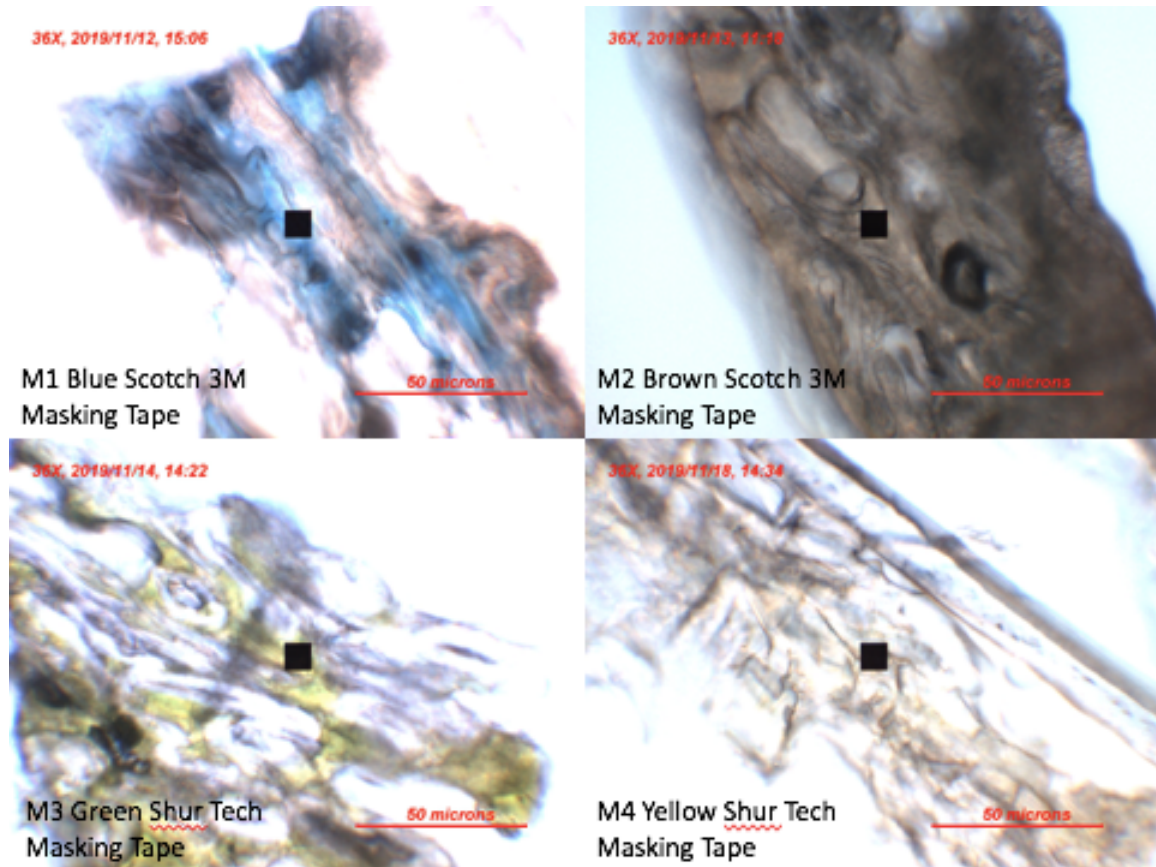
Sample thickness affected absorbance intensity for all masking tape samples. All masking tape samples show some degree of overlap into another thickness' range of variability. That is, when comparing the same masking tape samples, a difference in

thickness of 45  $\mu\text{m}$  across samples could be expected to have some spectral overlap. However, in some samples, this amount of sample thickness difference would result in a spectral exclusion based on previously defined parameters. Additionally, a difference of 45  $\mu\text{m}$  across a single sample would greatly increase the sample's absorbance spectrum variation. For the blue Scotch 3M masking tape (M1) a comparison between samples cut to 15  $\mu\text{m}$  and 50  $\mu\text{m}$  would result in a spectral exclusion. For the brown Scotch 3M masking tape (M2) no spectral exclusion would be made given the available spectral data, but there were very few clear points of comparison present in these spectra. For the green Shur Tech masking tape (M3) a comparison between samples cut to 5  $\mu\text{m}$  and 50  $\mu\text{m}$  would result in a spectral exclusion.

The above observations indicate that, for masking tapes, samples cross sectioned to thicknesses between 5  $\mu\text{m}$  and 50  $\mu\text{m}$  have the potential to provide quality spectral data and that cross sections of masking tape samples should not vary by more than 35  $\mu\text{m}$  thickness. Meaning, if two masking tape samples are being compared, they should not vary in thickness by more than 35  $\mu\text{m}$ .

During the collection of each spectrum images of the sample were also collected through the microspectrophotometer. Representative images of the masking tape samples microtomed to 30  $\mu\text{m}$  can be seen in Diagram 9 on the following page.

## Diagram 9.) Masking Tape Images



### 5. Optimized Sample Preparation

Given the range of variation found in hand cross sectioning and the difference in sample thickness found to be detectable by microspectrophotometry, it was determined that the best means of preparing duct and electrical tapes is through microtoming, the best means of preparing packaging tapes is directly through when possible (or microtoming), and the best means of preparing masking tapes is hand cross sectioning. These conclusions are dependent upon the analyst's hand cross sectioning ability as well as the availability of the materials necessary to perform either method. These conclusions may also be dependent upon the microspectrophotometer instrument itself. The best sample

preparation methods for duct, electrical, packaging, and masking tapes are summarized in Table 2 below.

Table 2.) Best Methods of Sample Preparation

	Duct Tape	Electrical Tape	Packaging Tape	Masking Tape
Range of Useful Thickness	5-50 $\mu\text{m}$ excluding directly through	5-50 $\mu\text{m}$ excluding directly through	5-50 $\mu\text{m}$ including directly through (if colorant is in the adhesive, sections thinner than 30 $\mu\text{m}$ may not be possible)	5-50 $\mu\text{m}$ including directly through (if lightly colored, sections thinner than 30 $\mu\text{m}$ may not be possible)
Range of Acceptable Thickness Variation	15 $\mu\text{m}$	15 $\mu\text{m}$	10 $\mu\text{m}$	35 $\mu\text{m}$
Experienced Analyst Range of Thickness Variation	20 $\mu\text{m}$	25 $\mu\text{m}$	15 $\mu\text{m}$	25 $\mu\text{m}$
Inexperienced Analyst Range of Thickness Variation	50 $\mu\text{m}$	30 $\mu\text{m}$	50 $\mu\text{m}$	50 $\mu\text{m}$
Inexperienced Analyst After 6 Months Range of Thickness Variation	25 $\mu\text{m}$	25 $\mu\text{m}$	30 $\mu\text{m}$	25 $\mu\text{m}$
Best Method of Sample Preparation	Microtome	Microtome	Direct Through when possible, if not Microtome	Hand Cross Sectioning

When possible (i.e. for masking tapes), hand cross sectioning was preferred over microtoming because of its ease and speed, but in these cases if hand cross sectioning was not possible, microtoming could be used interchangeably. If microtoming was not

possible for samples which were recommended to be prepared by microtome, samples could be cross sectioned by hand, measured, and selectively chosen based on desired thickness and range of variability. Samples recommended to be analyzed directly through should be analyzed in this way only when there is no concern for the sample to have been affected (e.g. crushed, stretched, etc.) in such a way that its thickness was changed beyond that of the acceptable range of variability. If this occurred, these samples may also be prepared by cross sectioning.

For samples recommended to be prepared by microtome the sample preparation method is as follows: The tape sample should be adhered to a piece of off-brand cling wrap and cut to an appropriate size to fit into an epoxy mold. The sample should then be mounted in epoxy and allowed to set completely. The mounted sample should then be cut to a narrowly pointed 3-dimensional trapezoid and cut via microtome. Cuttings can be moved using a combination of paint brushes, forceps, and tungsten needles. Using a stereo microscope, these cuttings can be mounted on a glass slide with distilled water and a glass coverslip.

While all thicknesses between 5  $\mu\text{m}$  and 50  $\mu\text{m}$  resulted in quality spectra, microtoming called for a choice of a specific thickness. Thicknesses at and below 15  $\mu\text{m}$  were observed to have resulted in some band flattening and were more difficult to handle than thicker sections. Thicknesses of 50  $\mu\text{m}$  were observed to have resulted in some band fusion and were consistently above an absorbance intensity of 1.5. A thickness of 30  $\mu\text{m}$  was often at or below an absorbance intensity of 1.5, was often free of spectral alterations, and was reasonable to handle. As such, sections were microtomed to thicknesses of 30  $\mu\text{m}$ .



For samples recommended to be prepared by hand cross sectioning, the sample preparation method is as follows: The tape sample should be adhered to a piece of off-brand cling wrap and cut to a roughly 1 mm x 10 mm sharply pointed trapezoid and stuck to a piece of double-sided tape on a plastic slide with the pointed end hanging off the tape and over the slide. Using a stereo microscope, cross sections of the tape can be cut using a double-edged razor blade supported by one gloved finger in a chopping motion. The cuttings can be handled with a tungsten needle and mounted on a glass slide with distilled water and a glass coverslip.

For samples recommended to be analyzed directly through the sample, the preparation method is as follows: The tape sample should not just be stuck directly to a slide but should be mounted on a glass slide with distilled water and a glass coverslip.

## **B. Intra-Roll Variation**

### **1. Duct Tape**

Using 30  $\mu\text{m}$  microtomed cross sections, the beginning, middle, and end of D1, D2, D3, and D4 were analyzed. The absorbance spectra generated for D1, D2, D3, and D4 were assessed visually. See Figure 57 through 60 in appendix C. For all duct tape samples, the position of band maxima and minima, width, and intensity were similar throughout all positions in the roll of tape. There was not an appreciable amount of intra-roll variation that can be observed through microspectrophotometry to a point of making a spectral exclusion.

## 2. Electrical Tape

Using 30  $\mu\text{m}$  microtomed cross sections, the beginning, middle, and end of E1, E2, E3, and E4 were analyzed. The absorbance spectra generated for E1, E2, E3, and E4 were assessed visually. See Figure 61 through 64 in appendix C. For all electrical tape samples, the position of band maxima and minima, width, and intensity were similar throughout all positions in the roll of tape. There was not an appreciable amount of intra-roll variation that can be observed through microspectrophotometry to a point of making a spectral exclusion.

## 3. Packaging Tape

The beginning, middle, and end of P1, P2, P3, and P4 were analyzed directly through the samples. The absorbance spectra generated for P1, P2, P3, and P4 were assessed visually. See Figure 65 through 68 in appendix C. For all packaging tape samples, the position of band maxima and minima, width, and intensity were similar throughout all positions in the roll of tape. There was not an appreciable amount of intra-roll variation that can be observed through microspectrophotometry to a point of making a spectral exclusion. However, in all packaging tape samples there are very few points of comparison an inconclusive result may be appropriate.

## 4. Masking Tape

Using hand cut cross sections, the beginning, middle, and end of M1, M2, M3, and M4 were analyzed. The absorbance spectra generated for M1, M2, M3, and M4 were assessed visually. See Figure 69 through 72 in appendix C. For all masking tape samples, the position of band maxima and minima, width, and intensity were similar throughout all positions in the roll of tape. There was not an appreciable amount of intra-roll variation

that can be observed through microspectrophotometry to a point of making a spectral exclusion.

### **C. Inter-Roll Variation**

#### **1. Duct Tape**

Using 30  $\mu\text{m}$  microtomed cross sections, the beginning of D1, D2, D3, and D4 were compared with the beginning of D1A, D2A, D3A, and D4A respectively. The absorbance spectra generated for D1 and D1A, D2 and D2A, D3 and D3A, and D4 and D4A were assessed visually. See Figure 73 through 76 in appendix D. For all duct tape samples, the position of band maxima and minima, width, and intensity were similar through the different rolls of the same tape. For D2, D3, and D4 there was not an appreciable amount of inter-roll variation that can be observed through microspectrophotometry to a point of making a spectral exclusion. D1 does have some appreciable amount of inter-roll variation that can be observed through microspectrophotometry to a point of making a spectral exclusion. However, there is some amount of overlap that would allow for a spectral inclusion.

#### **2. Electrical Tape**

Using 30  $\mu\text{m}$  microtomed cross sections, the beginning, middle of E1, E2, E3, and E4 were compared with the beginning of E1A, E2A, E3A, and E4A respectively. The absorbance spectra generated for E1 and E1A, E2 and E2A, E3 and E3A, and E4 and E4A were assessed visually. See Figure 77 through 80 in appendix D. For all electrical tape samples, the position of band maxima and minima, width, and intensity were similar through the different rolls of the same tape. There was not an appreciable amount of

inter-roll variation that can be observed through microspectrophotometry to a point of making a spectral exclusion.

### 3. Packaging Tape

The beginning of P1, P2, and P4 was compared to the beginning of P1A, P2A, and P4A directly through the samples respectively. The tape which would have been P3A was no longer available for purchase and comparisons between P3 and P3A were not possible. The absorbance spectra generated for P1 and P1A, P2 and P2A, and P4 and P4A were assessed visually. See Figure 81 through 84 in appendix D. For all packaging tape samples, the position of band maxima and minima, width, and intensity were similar through the different rolls of the same tape. For P1, P2, and P3 there was not an appreciable amount of inter-roll variation that can be observed through microspectrophotometry to a point of making a spectral exclusion. P4 does have some appreciable amount of inter-roll variation that can be observed through microspectrophotometry to a point of making a spectral exclusion. However, in all packaging tape samples there are very few points of comparison an inconclusive result may be appropriate.

### 4. Masking Tape

Using hand cut cross sections, the beginning of M1, M2, M3, and M4 were compared with the beginning of M1A, M2A, M3A, and M4A. The absorbance spectra generated for M1 and M1A, M2 and M2A, M3 and M3A, and M4 and M4A were assessed visually. See Figure 85 through 87 in appendix D. For all masking tape samples, the position of band maxima and minima, width, and intensity were similar through the different rolls of the same tape. There is not an appreciable amount of inter-roll variation

that can be observed through microspectrophotometry to a point of making a spectral exclusion.

#### **D. Additional Data**

##### **1. Single Sample Variation**

Using 30  $\mu\text{m}$  microtomed cross sections, a single sample was compared to itself: Fifty spectra were collected over ten cross sections and compared to fifty spectra collected over a separate set of ten cross sections from the beginning of the same roll of tape. These spectra were collected immediately after one another. See Figure 88 in appendix E. These averaged spectra highlight the amount of similarity and variation that may be expected in identical samples.

##### **2. Different Analyst's Hand Cross Section Variation**

The same experienced analyst and inexperienced analyst with over six months of experience (as previously mentioned) prepared ten hand cut cross sections from the same roll of tape. Fifty spectra were collected over these cross sections respectively and compared to each other. See Figure 89 in appendix E. These averaged spectra highlight the amount of variation that may occur as a result of differences in average thickness and range of thickness variability that occur as a result of different analysts' cross sections. The more experienced analyst's hand cross sections have a tendency to be thinner and more similar in thickness while the less experienced analyst's hand cross sections have a tendency to be thicker and more variable in thickness. This is reflected by a difference in the spread of data and by differences in absorbance intensity between the two sample sets. These results indicate that if hand cross sectioning is being performed, hand cross

sections should only be compared across the same analysts or across analysts of equal skill.

### 3. Comparisons of Same Colored Tapes

Tapes of the same color were prepared as previously found appropriate and compared. The two orange duct tapes were compared to each other, the two blue duct tapes were compared to each other as well as to the blue masking tape, the two red electrical tapes were compared to each other, the two green electrical tapes were compared to each other as well as to the green masking tape, and the four brown packaging tapes were compared to each other as well as to the brown masking tape. See Figures 90 through 94 in appendix E. The orange, blue, red, and green tapes could all be readily distinguished from one another through the combination of differences in the positions of band maxima and minima, band widths, and band intensities. The brown tapes, which had little activity in the visible spectral range could not all be readily distinguished through visual examination of the spectra.

## VII. DISCUSSION

This study provided fundamental information pertaining to the analysis of pressure sensitive tapes by microspectrophotometry. Specifically, this study determined what sample preparation methods were appropriate and optimal for the analysis of duct tapes, electrical tapes, packaging tapes, and masking tapes by microspectrophotometry. This study also addressed the intra- and inter-roll variation that could be detected by microspectrophotometric analysis of duct tapes, electrical tapes, packaging tapes, and masking tapes. This research shows that microspectrophotometric analysis of tape samples is possible, the sample and instrumental preparations which optimize analysis, and what kind of information can be obtained. Through this, this study has presented in what ways microspectrophotometry may serve as a worthwhile analytical step. All of this helps to promote successful analysis of tape samples in crime laboratories as well as serving as a foundational basis for future research studies.

All four examined types of tape were found to be amenable for and capable of providing useful microspectrophotometric data that has the potential to aid in the forensic analysis of tapes. Unless otherwise stated, no means of sample preparation is inherently wrong, but the best practice for sample preparation found in this study is as follows: Duct tapes and electrical tapes were best prepared by adhering the tape to a piece of cling wrap, fixing the sample in epoxy, and microtoming the sample to cross sections with thicknesses between 5  $\mu\text{m}$  and 50  $\mu\text{m}$  such that a single sample's thickness varied less than 15  $\mu\text{m}$ . Packaging tapes were best prepared by adhering the tape to a slide for direct-through analysis without cross sectioning. Masking tapes were best prepared by hand cross sectioning the tape sample. These methods are not contrary to methods used in

previous studies on the microspectrophotometric analysis of tapes. A previous study that included microspectrophotometry of packaging tapes did so using reflectance directly on packaging tape samples adhered on a glass slide without mounting media or a coverslip.<sup>32</sup> The newly proposed sample preparation method expands upon the previously published method by using transmission through the sample mounted on a glass slide with distilled water and a glass coverslip. A recent study on the microspectrophotometric analysis of tape backings prepared their samples by first mounting them in paraffin wax and microtoming them to 20  $\mu\text{m}$  and then mounting them on a glass slide with Permount<sup>®</sup> and a glass coverslip.<sup>33</sup> This study was published after the completion of the data gathering phase of this research and could not be considered during sample preparations. While the exact methods vary between studies, the use of a microtome and the thickness used in the published study falls within what was found to be acceptable for all tape samples in this research. The newly proposed sample preparation methods expand upon the recently published methods by further detailing important factors to consider when preparing different types of tape for microspectrophotometric analysis as well expanding upon the acceptable means of sample preparation. The development and detailed exploration of these suggested sample preparation methods may serve as the foundation for future microspectrophotometric analyses of tape samples.

All four examined types of tape were found to have no or minimally appreciable intra- and inter-roll variation through rolls of consistent type, style, and manufacture as detectable by microspectrophotometry. Appreciable differences were observed in rolls of the same color (e.g. blue). These conclusions show how microspectrophotometry of tapes provides class characteristic information. As emphasized as an important need by the



recently published study on the microspectrophotometric analysis of tape backings, this research addressed the observable intra- and inter-roll variation of tapes as well as provided analysis on additional sample sets.<sup>33</sup> In doing so, this research has provided fundamental information about the conclusions that can be rendered when analyzing tapes by microspectrophotometry.

In a practical sense, the information found in this research suggests that the implementation of microspectrophotometry in the analysis of tapes may be useful when colored tapes are being analyzed and are found to be visually the same color. Microspectrophotometry could either add an objective observation of color similarity without consuming the sample prior to further instrumental analysis, or microspectrophotometry could detect color differences not picked up by visual examination and eliminate the need for further instrumental analysis. The use of microspectrophotometry in the analysis of tapes could also increase the ease in which color could be communicated between laboratories and in courts. Moreover, the implementation of microspectrophotometry in the analysis of tapes could serve as a useful addition to investigative databases. This research provides foundational information that laboratories could use prior to implementing microspectrophotometry in the analysis scheme of tape samples.

While visual examination is necessary and useful for the interpretation of microspectrophotometric data, there has been a push for more statistical means of spectral analysis. There are no current standards or generally accepted methods. The expansion upon how microspectrophotometric data is analyzed is an area that could greatly impact this type of research and future studies. A statistical approach to data

analysis could be especially useful for tapes with very few points of comparison such as brown tapes and achromatic tapes. Future work should therefore include more in-depth analysis of achromatic and brown colored samples, but this type of analysis must also be paired with an expansion of the statistical means of spectral analysis. Masking tapes were found to have more spectral variation than other types of tapes, this could be a result of their natural layered composition and further research into the appropriate analysis of masking tapes especially with consideration into the number of spectra collected would also be useful. There has also been mention of the dynamic range of the instrument with regard to absorbance intensities above 1.5. Work should be done to test the success of microspectrophotometric instruments above this intensity. Finally, research on microspectrophotometry of tapes could also be expanded through the analysis of more types of tapes, more samples of the same types of tapes, analysis of less pristine samples, and analysis of environmentally degraded samples.

#### A. **Conclusion**

Pressure sensitive tapes including duct tape, electrical tape, packaging tape, and masking tape are amenable for analysis by microspectrophotometry. The best method for preparing tape samples for analysis by microspectrophotometry is dependent upon the type of tape, the range of thickness variability detectable by the microspectrophotometry instrument, and the range of thickness variability achievable by cross sectioning technique. For the conditions of this study, duct and electrical tapes are best prepared through microtoming to a thickness between 5  $\mu\text{m}$  and 50  $\mu\text{m}$ , packaging tapes are best analyzed directly through the tape, and masking tapes are best prepared through hand

cross sectioning. Microspectrophotometric data obtained from these tapes samples provides information that is consistent throughout a single roll and throughout rolls of the same type, style, and manufacture, but different between rolls of unique type, style, and manufacture: Microspectrophotometric analysis of pressure sensitive tapes provides information indicative of class characteristics of a tape. The microspectrophotometric analysis of these tape samples provides useful data that may aid in forensic investigations.

## Appendices

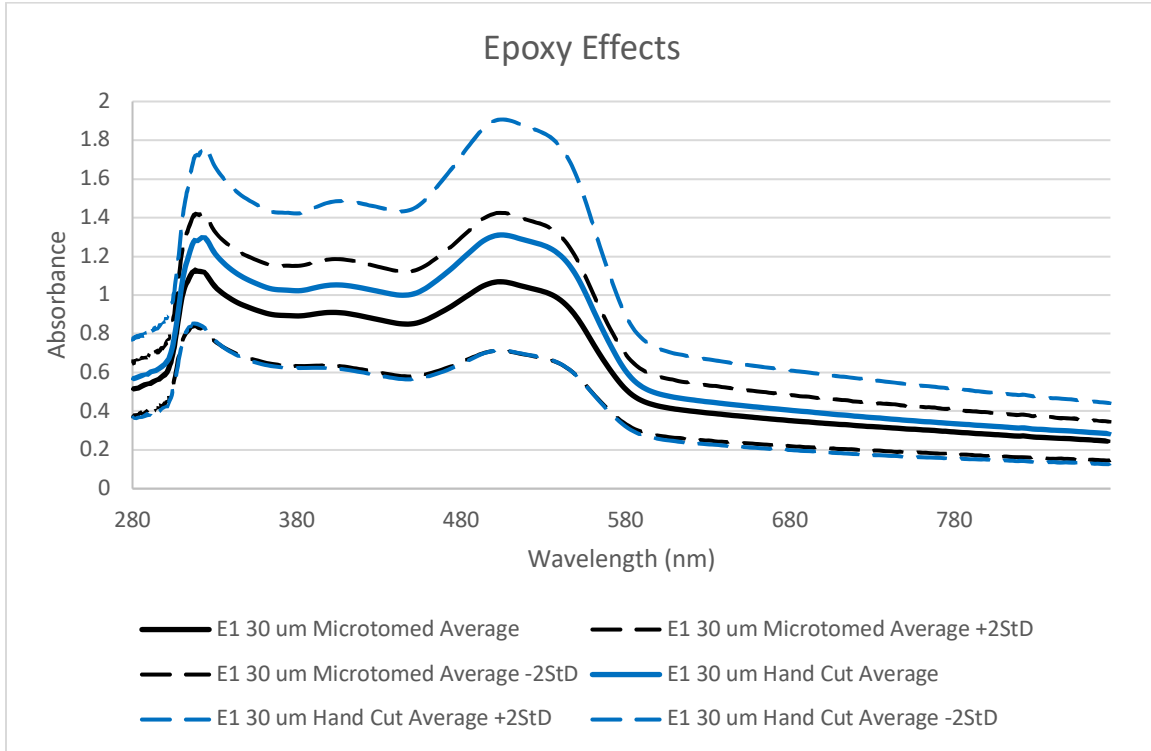
### Appendix A

Table 3.) Hand Cross Sectioning Range of Variation

Packaging Tapes				Masking Tapes		
Experienced Analyst	Inexperienced Analyst	Inexperienced Analyst After 6 Months		Experienced Analyst	Inexperienced Analyst	Inexperienced Analyst After 6 Months
15	20	15		20	50	30
15	20	20		30	60	35
15	20	20		30	60	35
20	30	20		30	60	40
20	30	20		30	60	40
20	40	35		35	70	40
20	40	35		35	70	40
20	40	35		40	70	45
20	50	40		45	80	50
30	70	45		45	100	55
15	50	30		25	50	25

	Duct Tapes				Electrical Tapes		
	Experienced Analyst	Inexperienced Analyst	Inexperienced Analyst After 6 Months	Experienced Analyst	Inexperienced Analyst	Inexperienced Analyst After 6 Months	
Thickness (μm)	15	20	20	20	20	20	
	15	30	20	20	30	20	
	15	30	20	20	30	20	
	20	30	30	25	30	30	
	20	40	35	30	30	30	
	20	40	35	35	30	30	
	25	50	40	35	30	30	
	35	60	40	35	40	40	
	35	60	45	35	40	40	
	35	70	45	45	50	45	
Range of Variation (μm)	20	50	25	25	30	25	

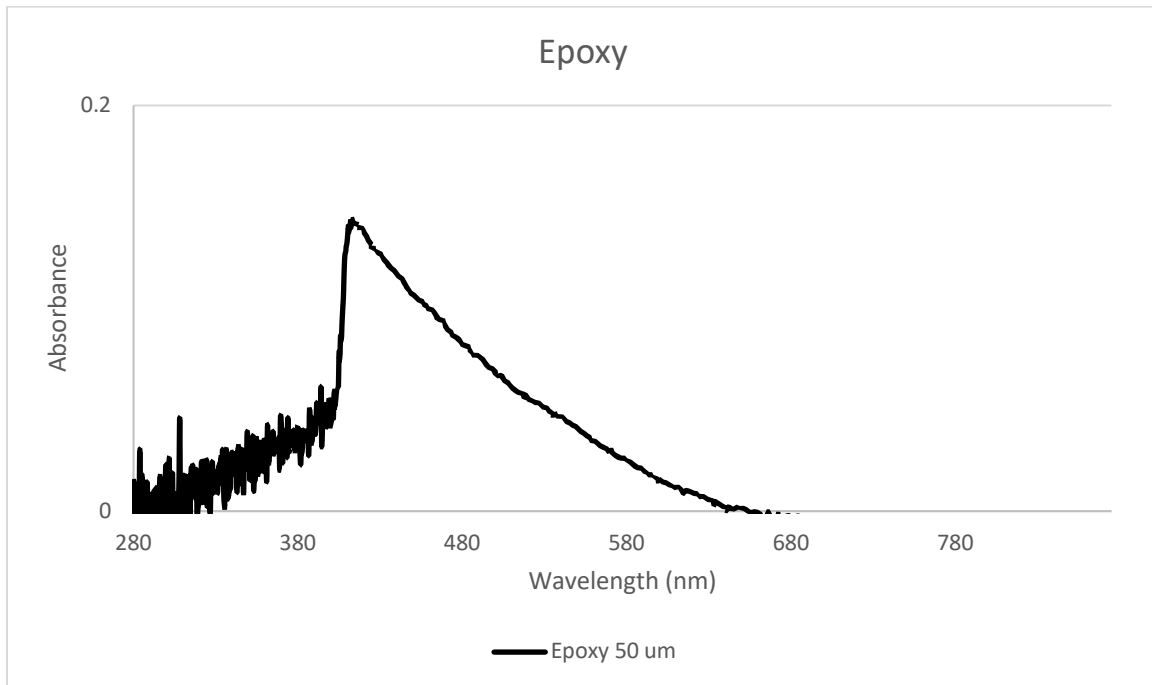
Figure 1.) Epoxy Effects



The above figure compares the average absorbance spectra of a red electrical tape molded in epoxy and microtomed to 30  $\mu\text{m}$  and the same red electrical tape not molded in epoxy and hand cross sectioned to 30  $\mu\text{m}$  from 280 to 875 nm. A solid line indicates the average of fifty scans (ten cross sections scanned at five different locations respectively) and a pair of dashed lines indicates plus or minus two standard deviations from the mean.

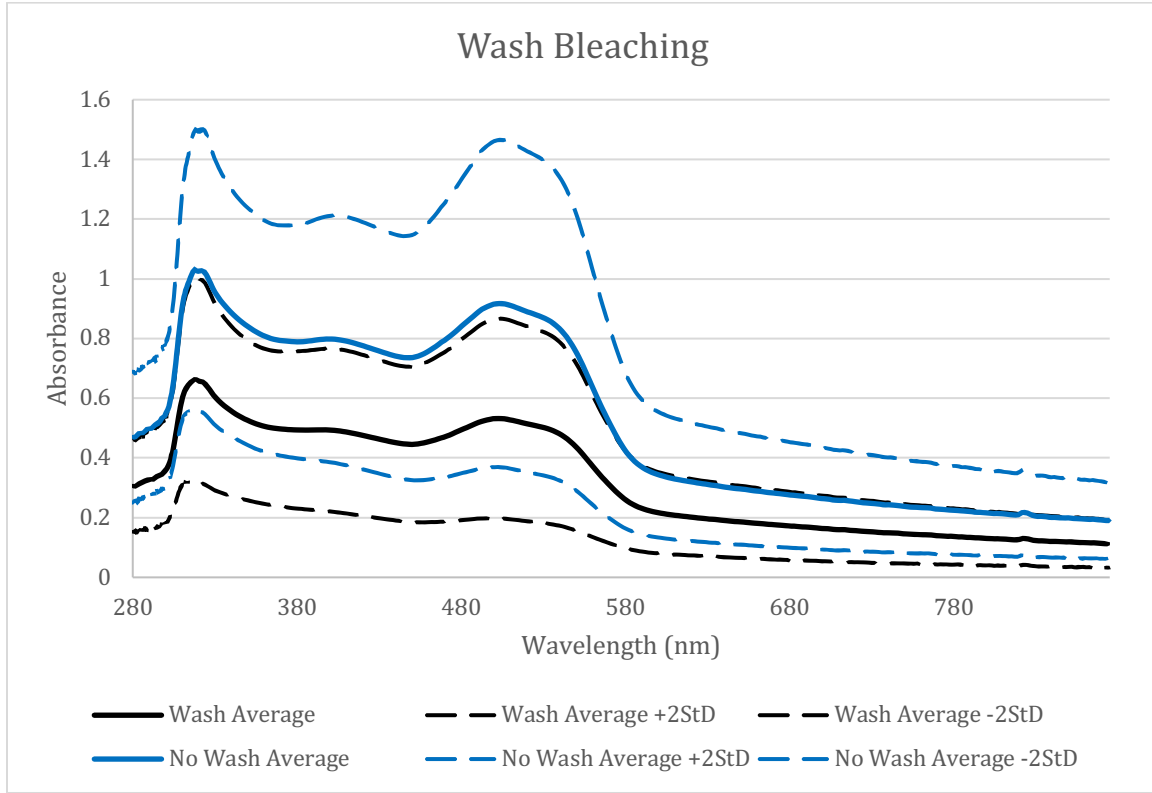
Visual comparison of the two spectral averages show no considerable differences with consideration to the variation indicated by the range of standard deviations.

Figure 2.) Epoxy



The above figure shows the absorbance spectrum of Hardman Double/Bubble® Epoxy from 280 to 875 nm.

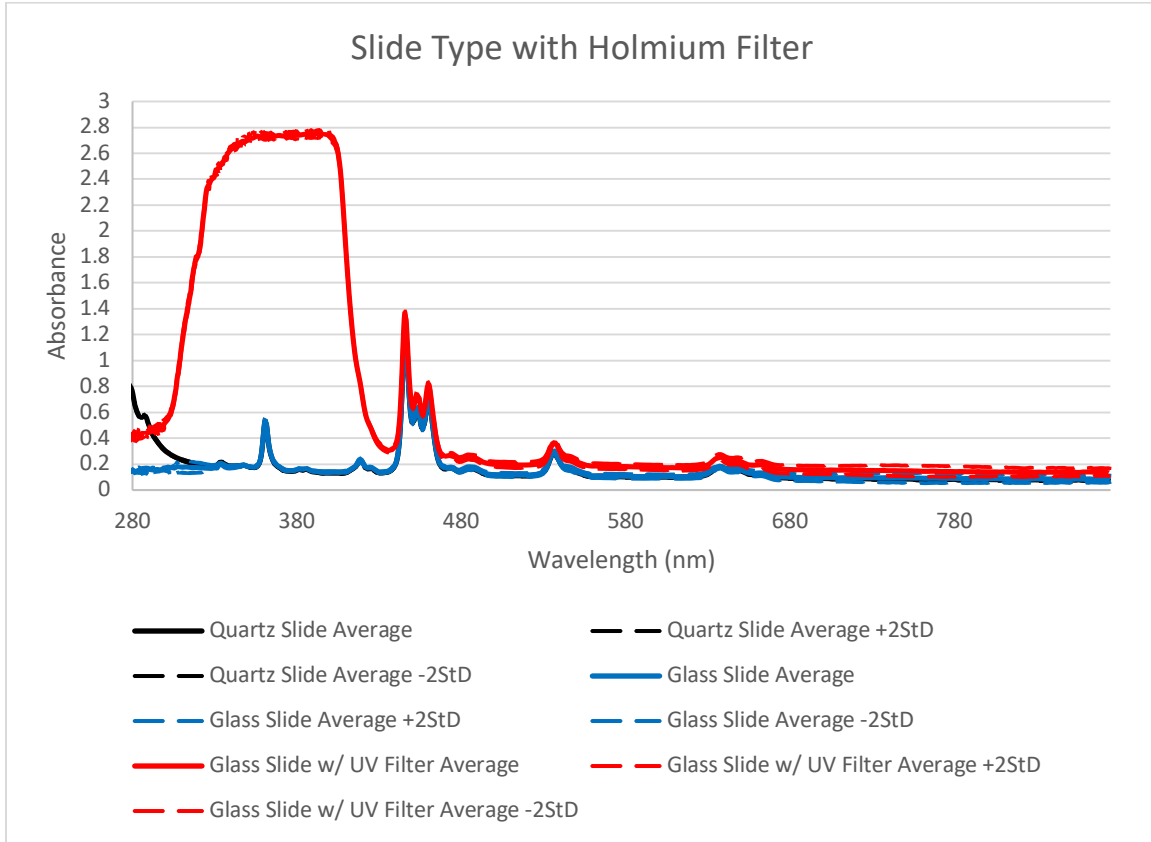
Figure 3.) Wash Bleaching



The above figure compares the average absorbance spectra of a red electrical tape sonicated in hexanes for two 90 second cycles and the same unwashed red electrical tape from 280 to 875 nm. A solid line indicates the average of fifty scans (ten 15  $\mu\text{m}$  cross sections scanned at five different locations respectively) and a pair of dashed lines indicates plus or minus two standard deviations from the mean. Visual comparison of the two spectral averages show considerable differences in the absorbance intensity with consideration to the variation indicated by the range of standard deviations. That is, the average absorbance spectrum of unwashed red electrical tape is just beyond the range of standard deviations for red electrical tape sonicated in hexanes. This shows that through sonication in hexanes some bleaching effects were occurring.

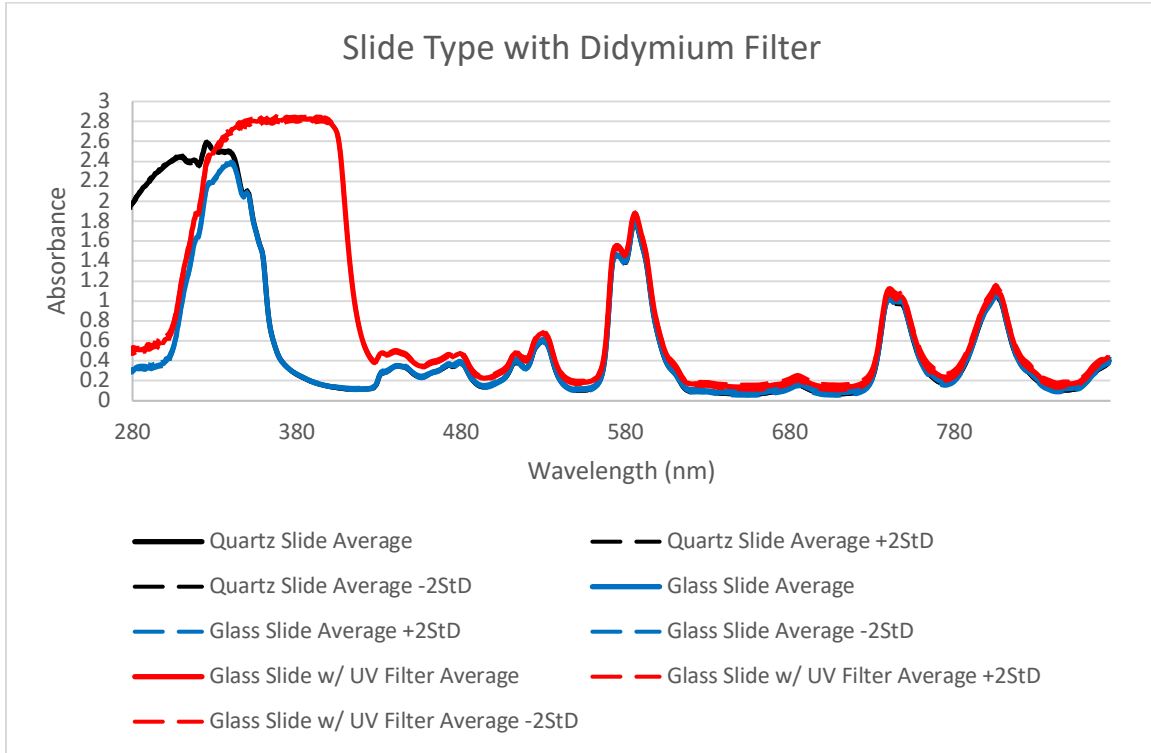


Figure 4.) Slide Type with Holmium Filter



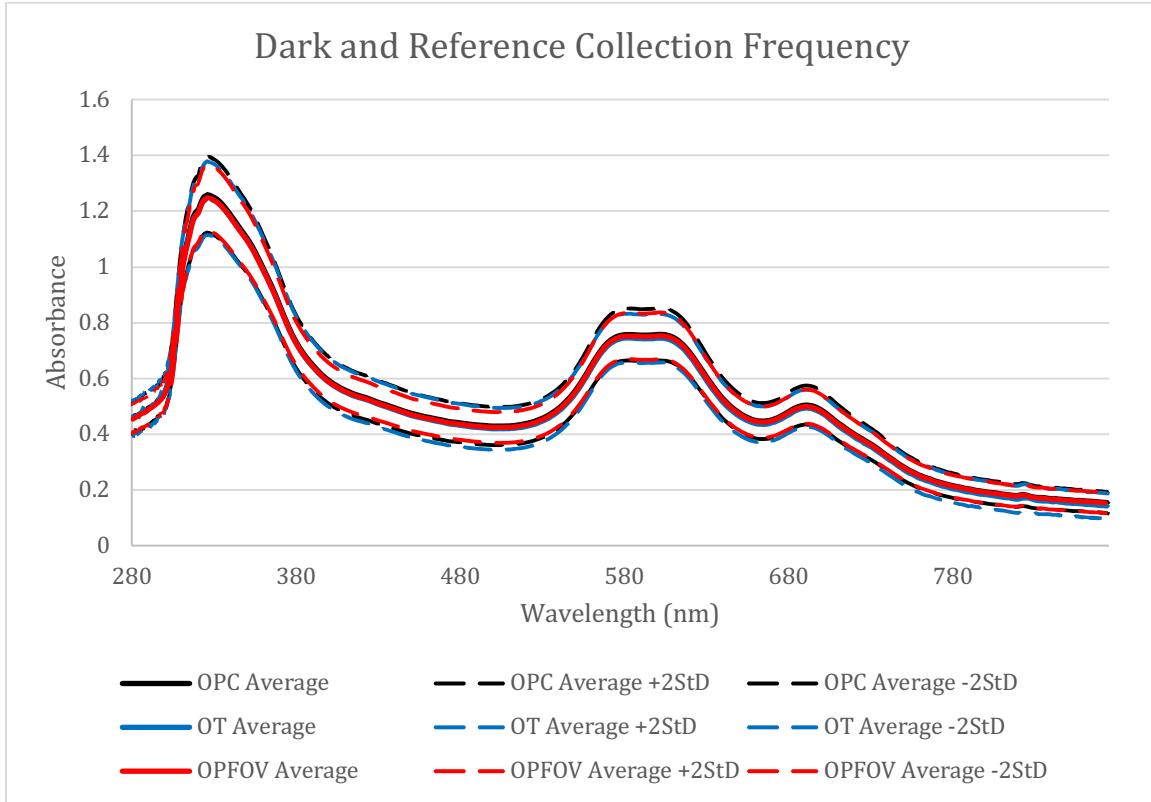
The above figure compares the average absorbance spectra of holmium through a quartz slide with water and a quartz coverslip, a glass slide with water and a glass coverslip, and a glass slide with water, a glass coverslip, and an ultraviolet filter from 280 to 875 nm. A solid line indicates the average of five scans and a pair of dashed lines indicates plus or minus two standard deviations from the mean. In the visible region (400 nm to 800 nm), a quartz slide with water and a quartz coverslip provides an indistinguishable spectrum from a glass slide with water and a glass coverslip. The addition of the ultraviolet filter does, however, affect the resultant lower visible spectrum.

Figure 5.) Slide Type with Didymium Filter



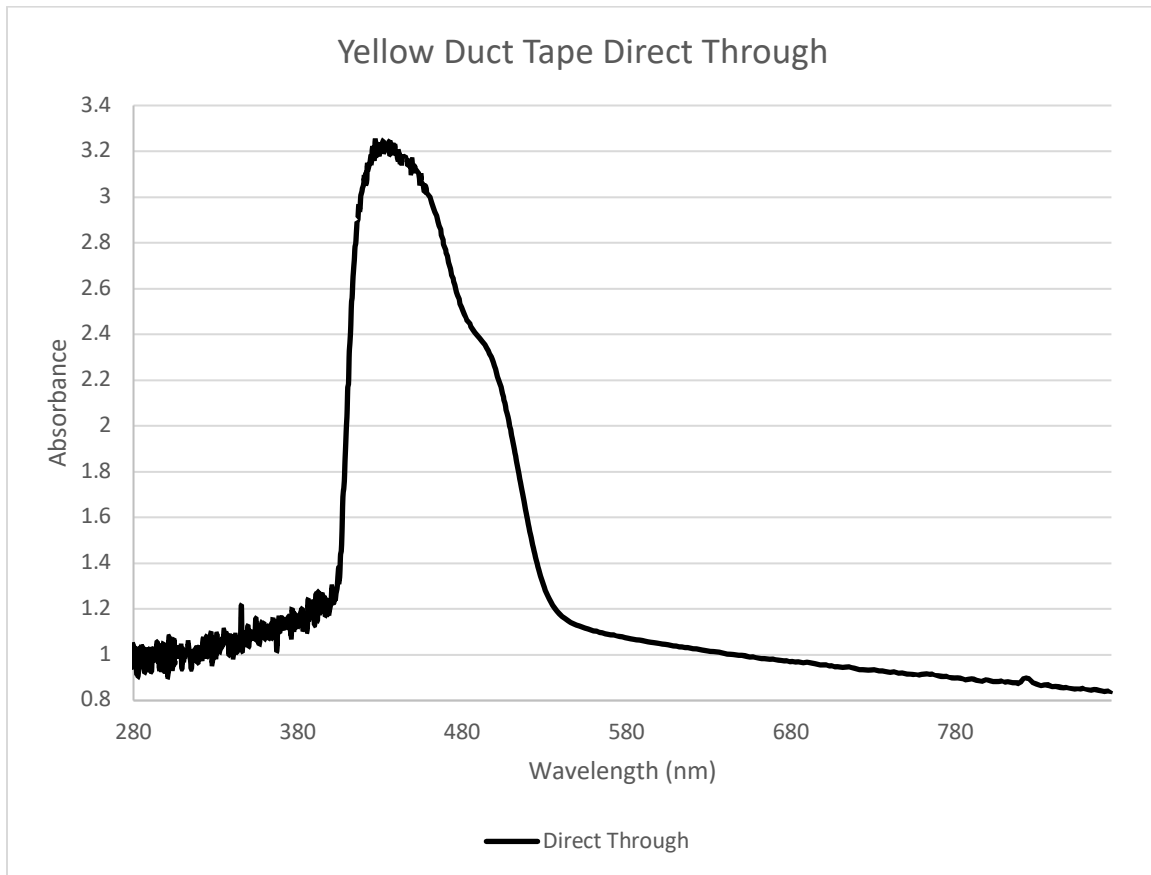
The above figure compares the average absorbance spectra of didymium through a quartz slide with water and a quartz coverslip, a glass slide with water and a glass coverslip, and a glass slide with water, a glass coverslip, and an ultraviolet filter from 280 to 875 nm. A solid line indicates the average of five scans and a pair of dashed lines indicates plus or minus two standard deviations from the mean. In the visible region (400 nm to 800 nm), a quartz slide with water and a quartz coverslip provides an indistinguishable spectrum from a glass slide with water and a glass coverslip. The addition of the ultraviolet filter does, however, affect the resultant lower visible spectrum.

Figure 6.) Dark and Reference Collection Frequency



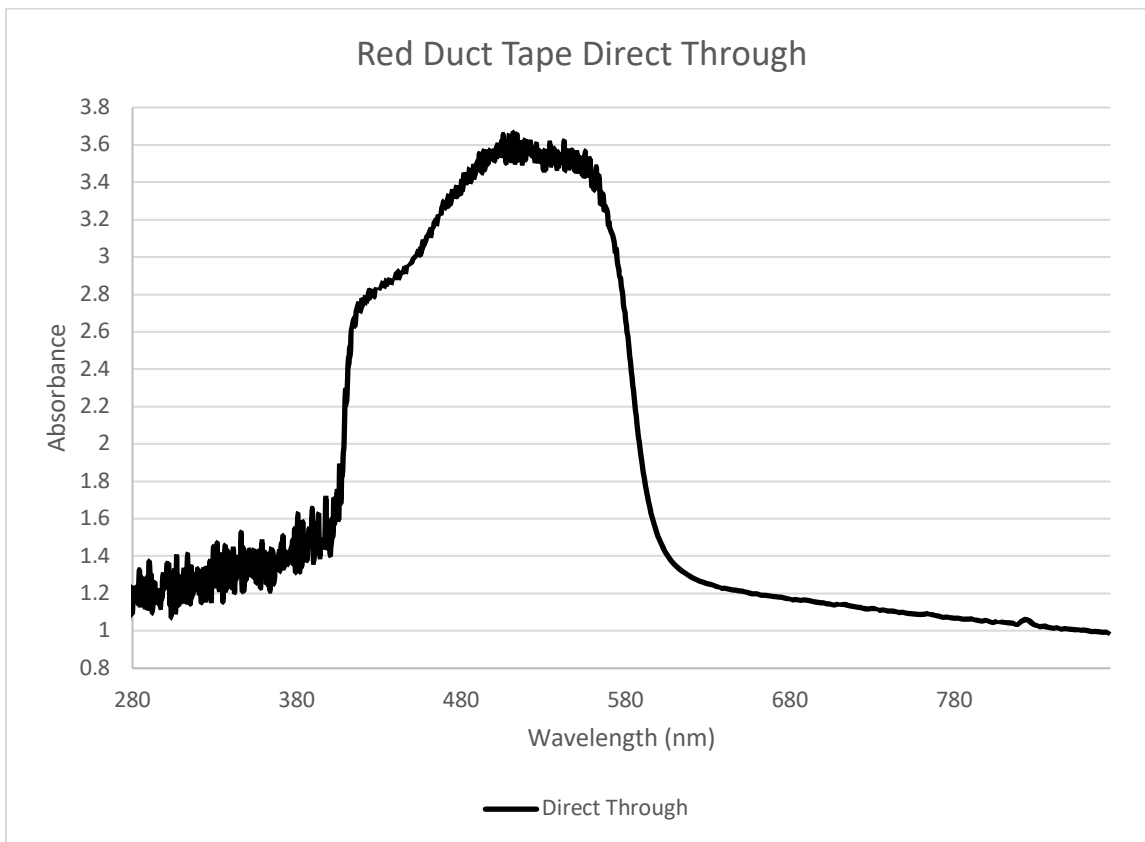
The above figure compares the average absorbance spectra of cross sections of blue electrical tape with one dark and one reference spectrum collected per cutting (OPC), one dark and one reference spectrum collected total (OT), and one dark and one reference spectrum collected per field of view (OPFOV) from 280 to 875 nm. A solid line indicates the average of fifty scans (ten 15  $\mu\text{m}$  cross sections scanned at five different locations respectively) and a pair of dashed lines indicates plus or minus two standard deviations from the mean. All three conditions result in indistinguishable spectra.

Figure 7.) Yellow Duct Tape Direct Through



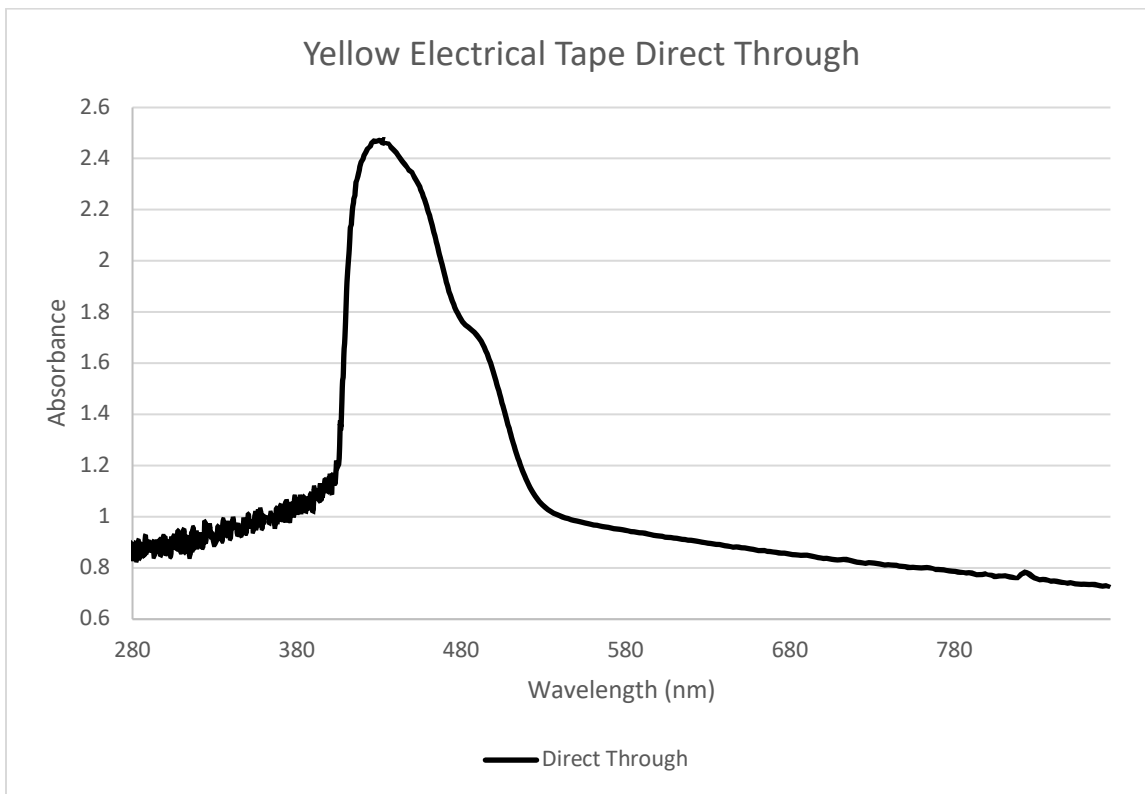
The above figure shows the absorbance spectrum directly through a piece of yellow duct tape from 280 to 875 nm. This spectrum oversaturated the instrument as indicated by noise at the band maxima.

Figure 8.) Red Duct Tape Direct Through



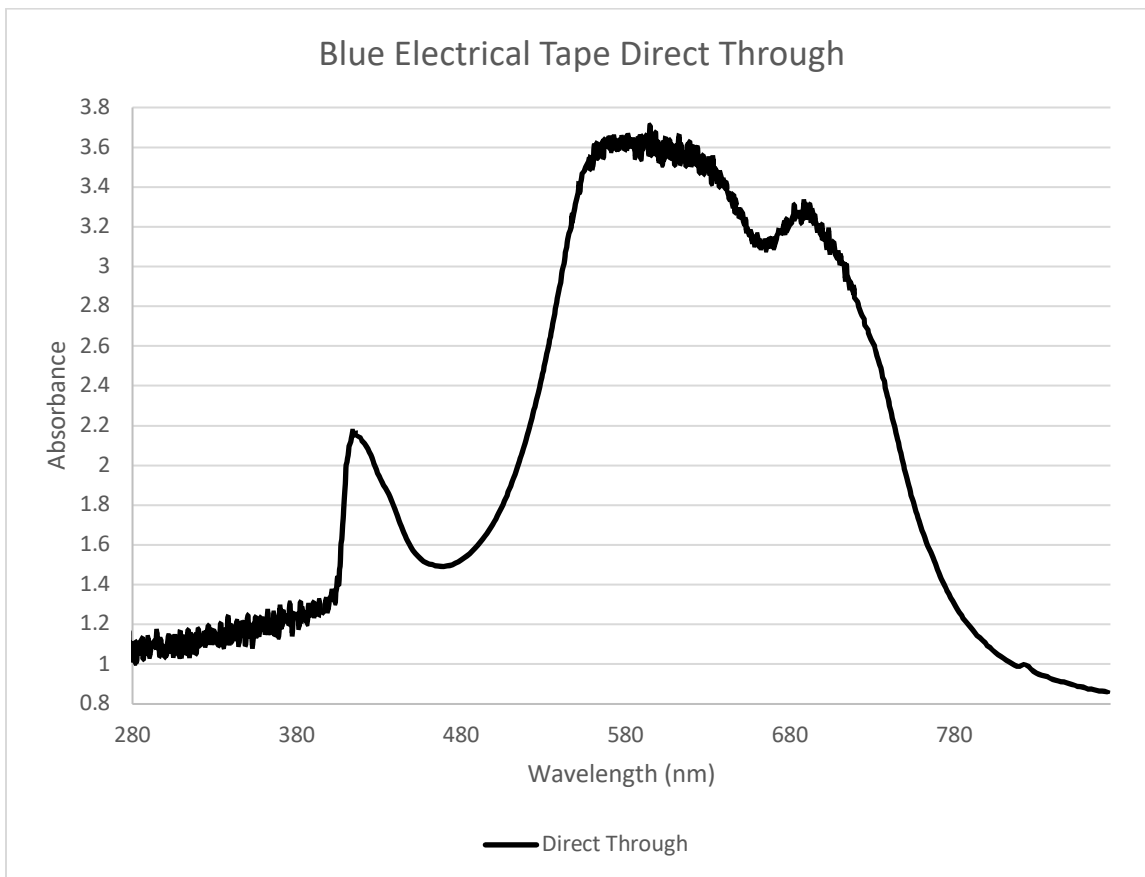
The above figure shows the absorbance spectrum directly through a piece of red duct tape from 280 to 875 nm. This spectrum oversaturated the instrument as indicated by noise throughout the band maxima.

Figure 9.) Yellow Electrical Tape Direct Through



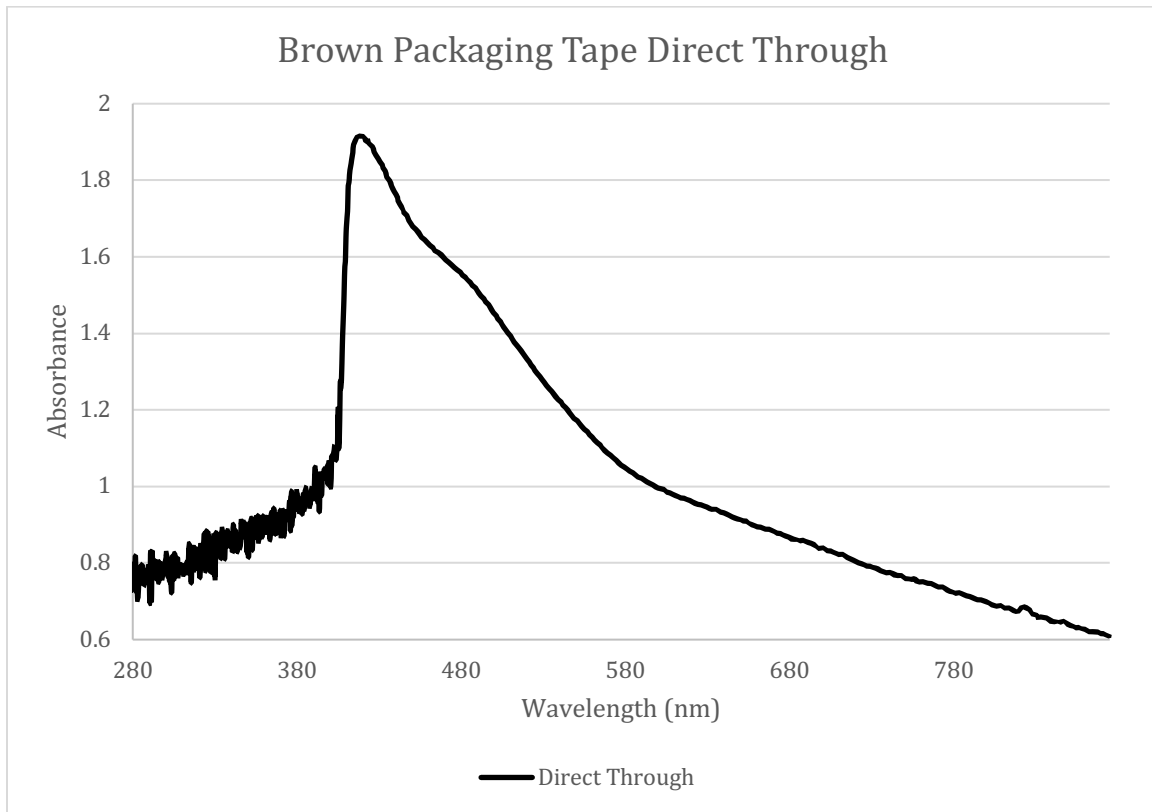
The above figure shows the absorbance spectrum directly through a piece of yellow electrical tape from 280 to 875 nm. This spectrum oversaturated the instrument as indicated by slight noise at the band maxima.

Figure 10.) Blue Electrical Tape Direct Through



The above figure shows the absorbance spectrum directly through a piece of blue electrical tape from 280 to 875 nm. This spectrum oversaturated the instrument as indicated by noise throughout the band maximums.

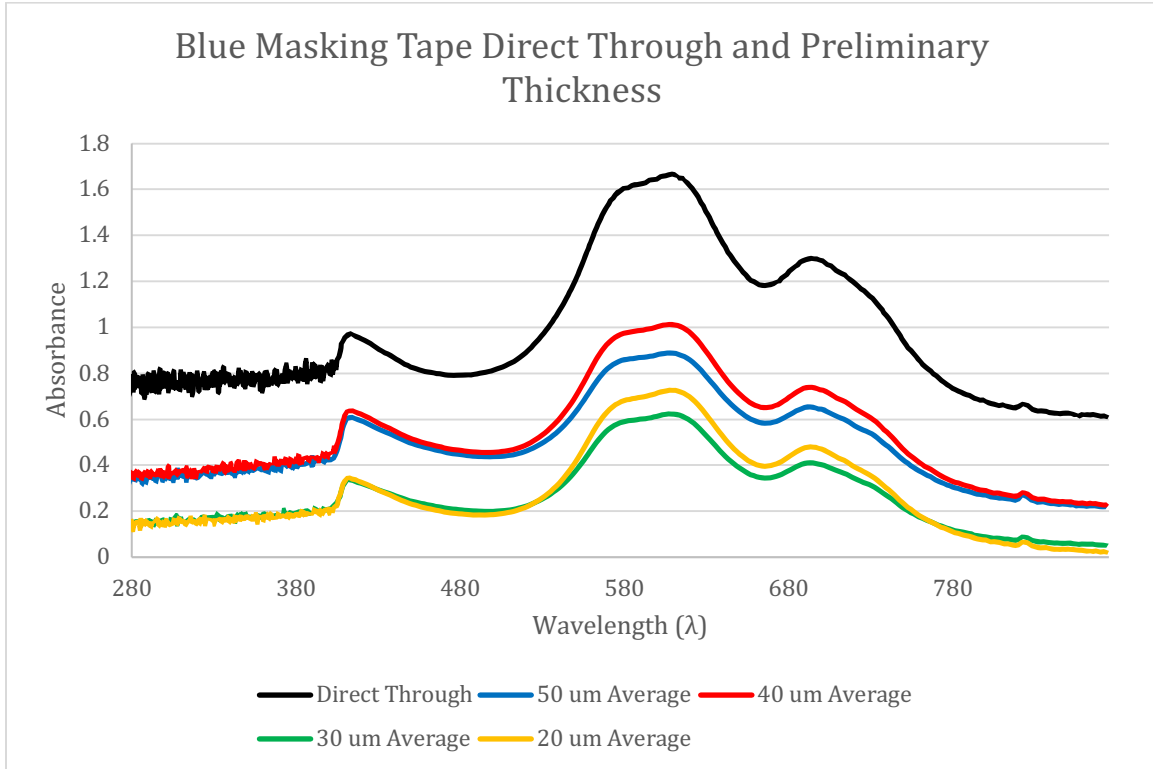
Figure 11.) Brown Packaging Tape Direct Through



The above figure shows the absorbance spectrum directly through a piece of brown packaging tape from 280 to 875 nm.

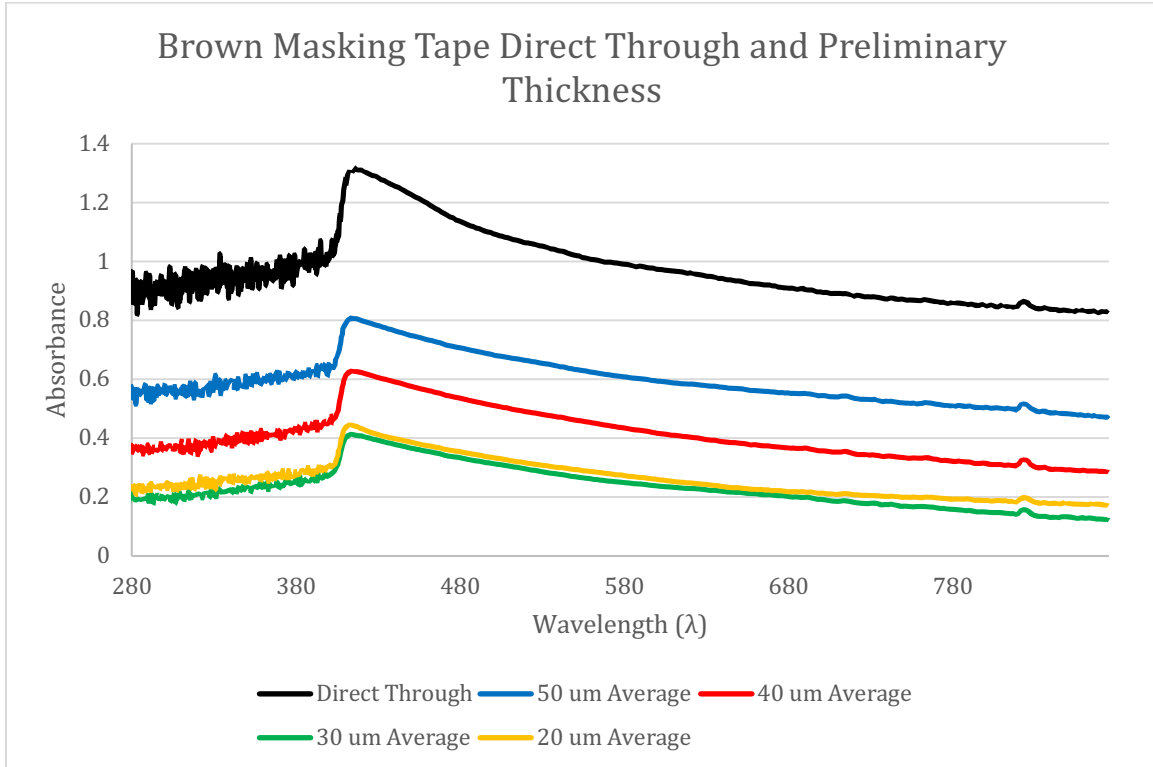


Figure 12.) Blue Masking Tape Direct Through and Preliminary Thickness



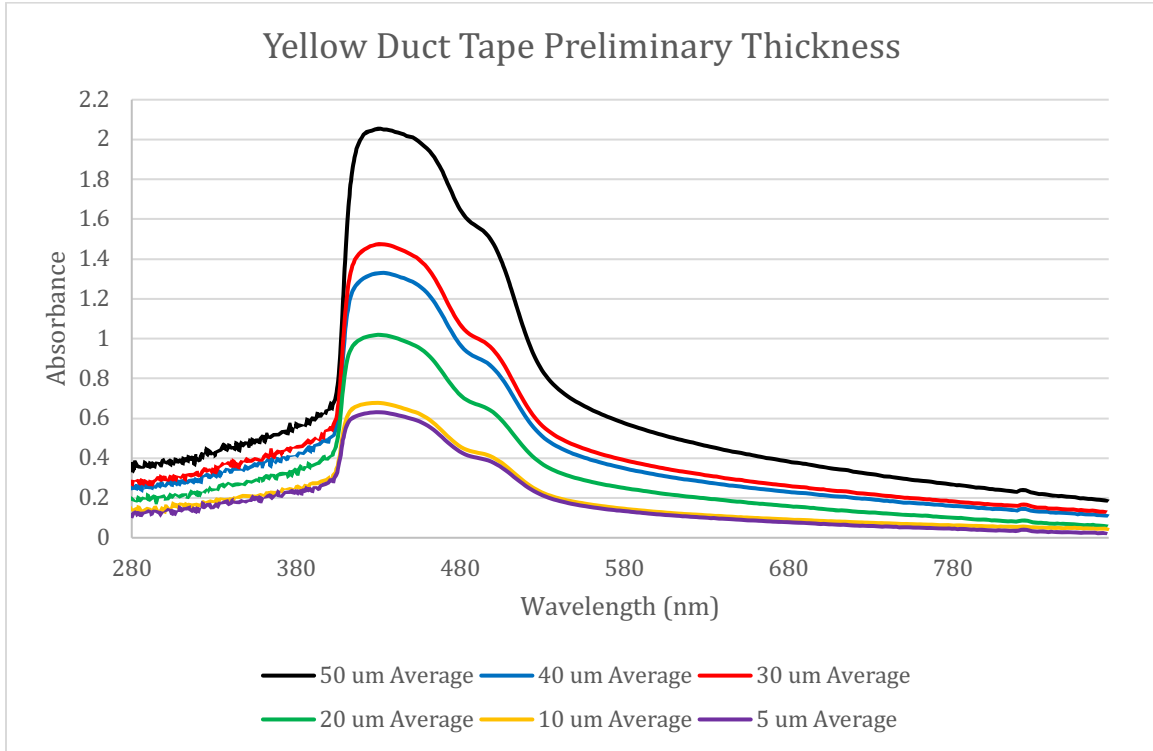
The above figure compares the absorbance spectrum directly through a piece of blue masking tape, 50  $\mu\text{m}$  microtomed cross sections, 40  $\mu\text{m}$  microtomed cross sections, 30  $\mu\text{m}$  microtomed cross sections, and 20  $\mu\text{m}$  microtomed cross sections of the same tape from 280 to 875 nm. A solid line indicates the average of five scans.

Figure 13.) Brown Masking Tape Direct Through and Preliminary Thickness



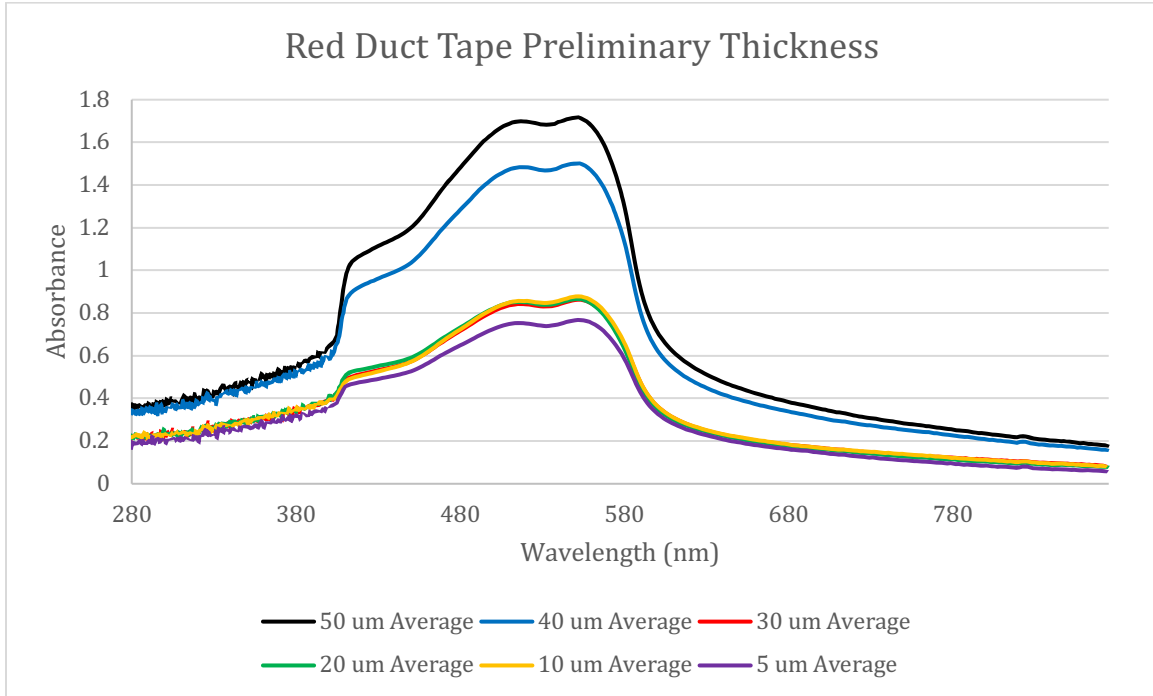
The above figure compares the absorbance spectrum directly through a piece of brown masking tape, 50  $\mu$ m microtomed cross sections, 40  $\mu$ m microtomed cross sections, 30  $\mu$ m microtomed cross sections, and 20  $\mu$ m microtomed cross sections of the same tape from 280 to 875 nm. A solid line indicates the average of five scans.

Figure 14.) Yellow Duct Tape Preliminary Thickness



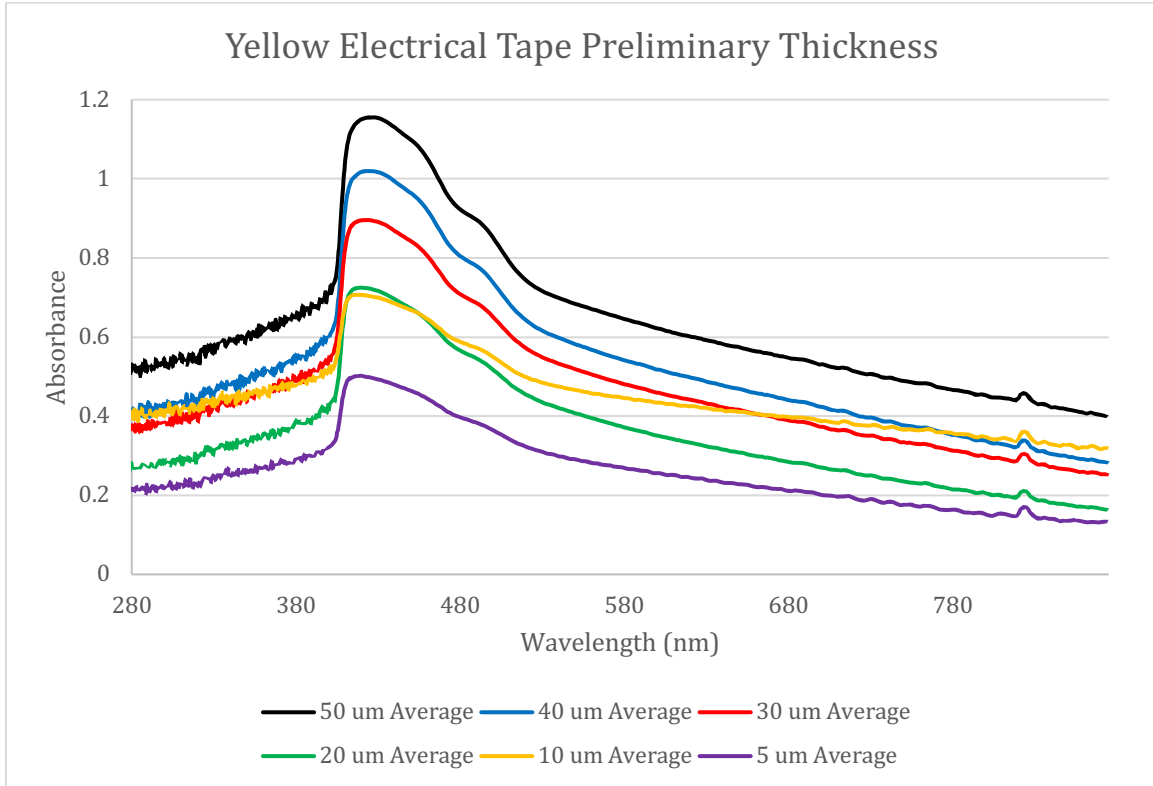
The above figure compares the average absorbance spectra of 50  $\mu\text{m}$  microtomed cross sections, 40  $\mu\text{m}$  microtomed cross sections, 30  $\mu\text{m}$  microtomed cross sections, 20  $\mu\text{m}$  microtomed cross sections, 10  $\mu\text{m}$  microtomed cross sections, and 5  $\mu\text{m}$  microtomed cross sections of yellow duct tape from 280 to 875 nm. A solid line indicates the average of five scans.

Figure 15.) Red Duct Tape Preliminary Thickness



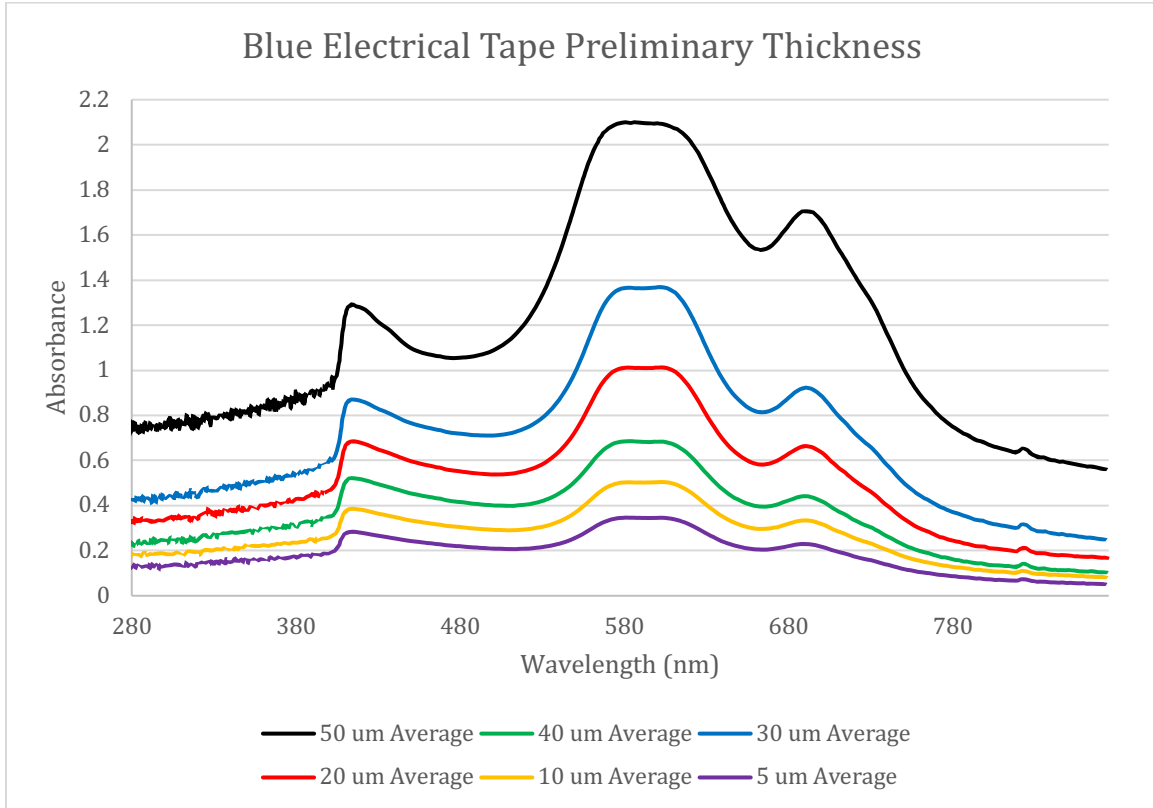
The above figure compares the average absorbance spectra of 50  $\mu\text{m}$  microtomed cross sections, 40  $\mu\text{m}$  microtomed cross sections, 30  $\mu\text{m}$  microtomed cross sections, 20  $\mu\text{m}$  microtomed cross sections, 10  $\mu\text{m}$  microtomed cross sections, and 5  $\mu\text{m}$  microtomed cross sections of red duct tape from 280 to 875 nm. A solid line indicates the average of five scans.

Figure 16.) Yellow Electrical Tape Preliminary Thickness



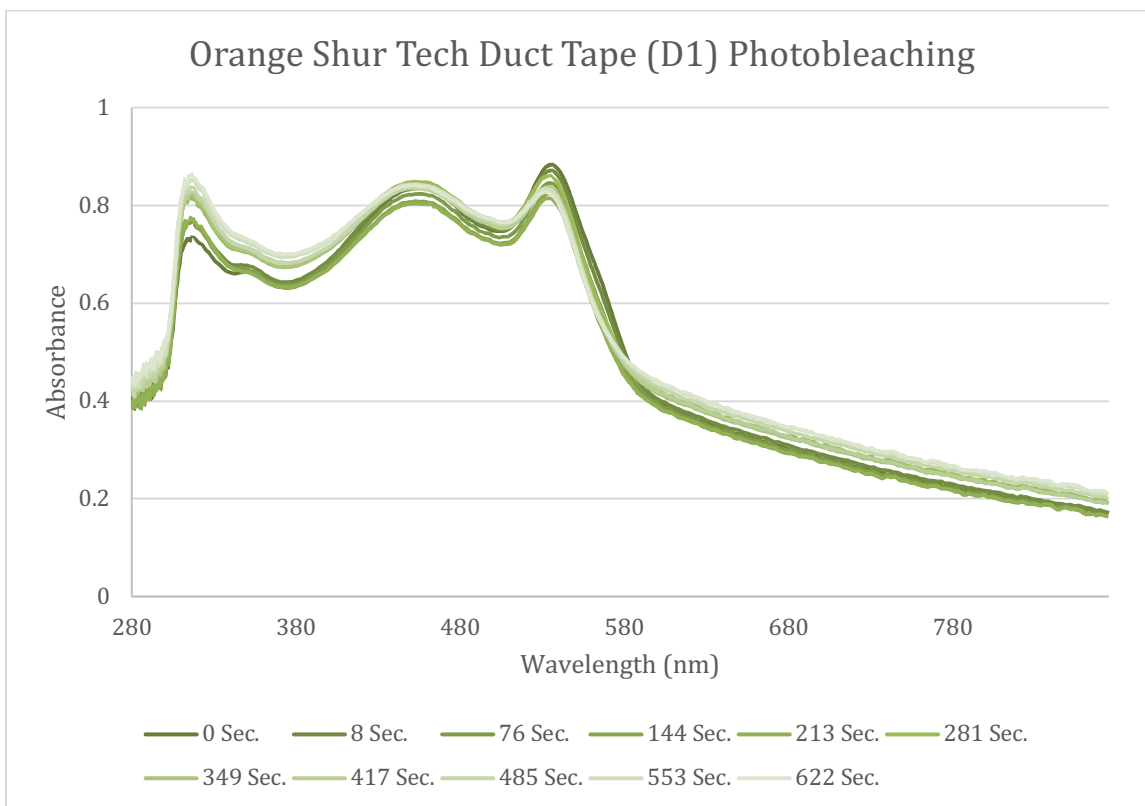
The above figure compares the average absorbance spectra of 50  $\mu\text{m}$  microtomed cross sections, 40  $\mu\text{m}$  microtomed cross sections, 30  $\mu\text{m}$  microtomed cross sections, 20  $\mu\text{m}$  microtomed cross sections, 10  $\mu\text{m}$  microtomed cross sections, and 5  $\mu\text{m}$  microtomed cross sections of yellow electrical tape from 280 to 875 nm. A solid line indicates the average of five scans.

Figure 17.) Blue Electrical Tape Preliminary Thickness



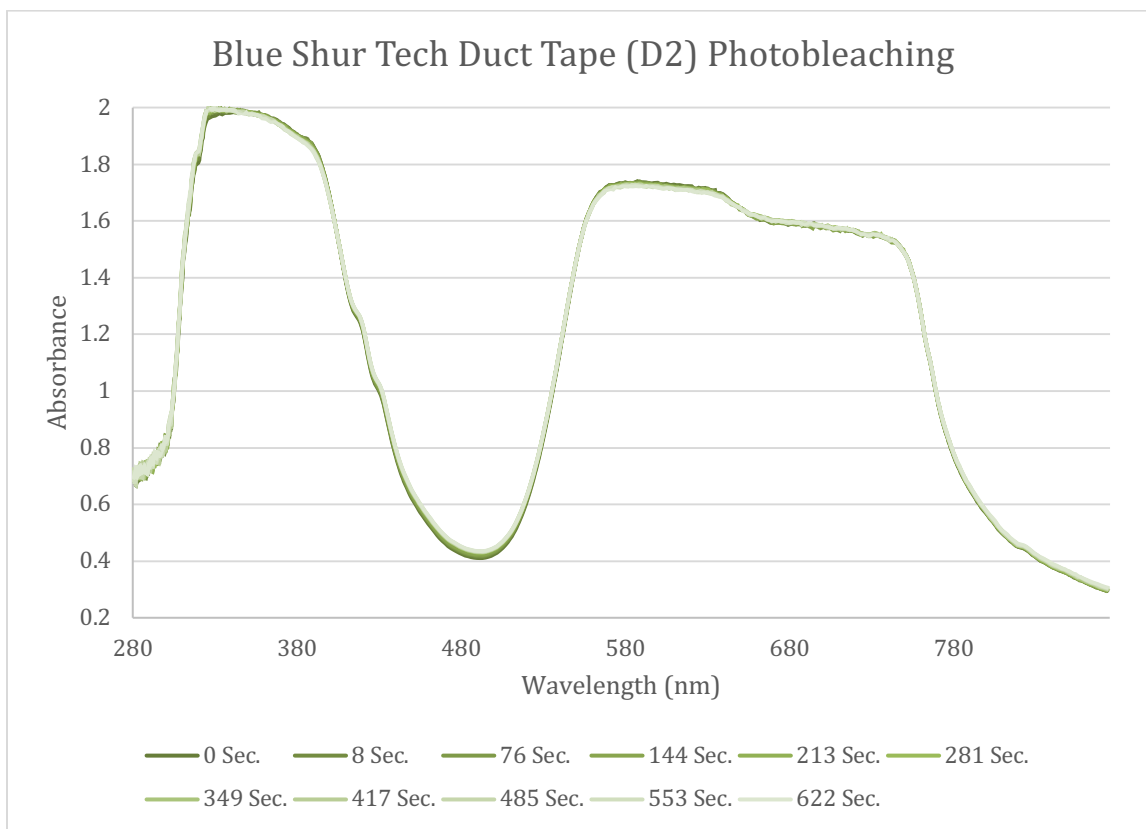
The above figure compares the average absorbance spectra of 50  $\mu\text{m}$  microtomed cross sections, 40  $\mu\text{m}$  microtomed cross sections, 30  $\mu\text{m}$  microtomed cross sections, 20  $\mu\text{m}$  microtomed cross sections, 10  $\mu\text{m}$  microtomed cross sections, and 5  $\mu\text{m}$  microtomed cross sections of blue electrical tape from 280 to 875 nm. A solid line indicates the average of five scans.

Figure 18.) Orange Shur Tech Duct Tape (D1) Photobleaching



The above figure shows the absorbance spectra of a 15  $\mu\text{m}$  microtomed cross section of sample D1 over 10 minutes of continuous bulb exposure from 280 to 875 nm. No significant photobleaching was observed.

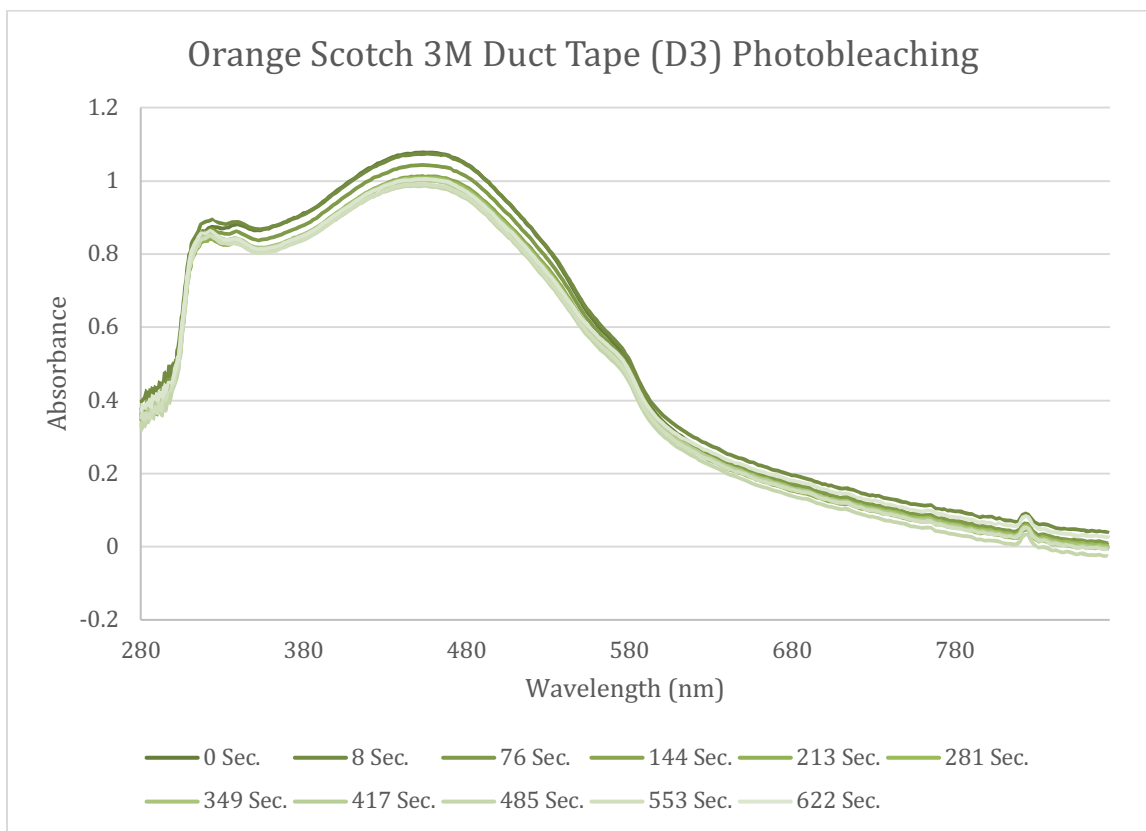
Figure 19.) Blue Shur Tech Duct Tape (D2) Photobleaching



The above figure shows the absorbance spectra of a 15  $\mu\text{m}$  microtomed cross section of sample D2 over 10 minutes of continuous bulb exposure from 280 to 875 nm. No significant photobleaching was observed.

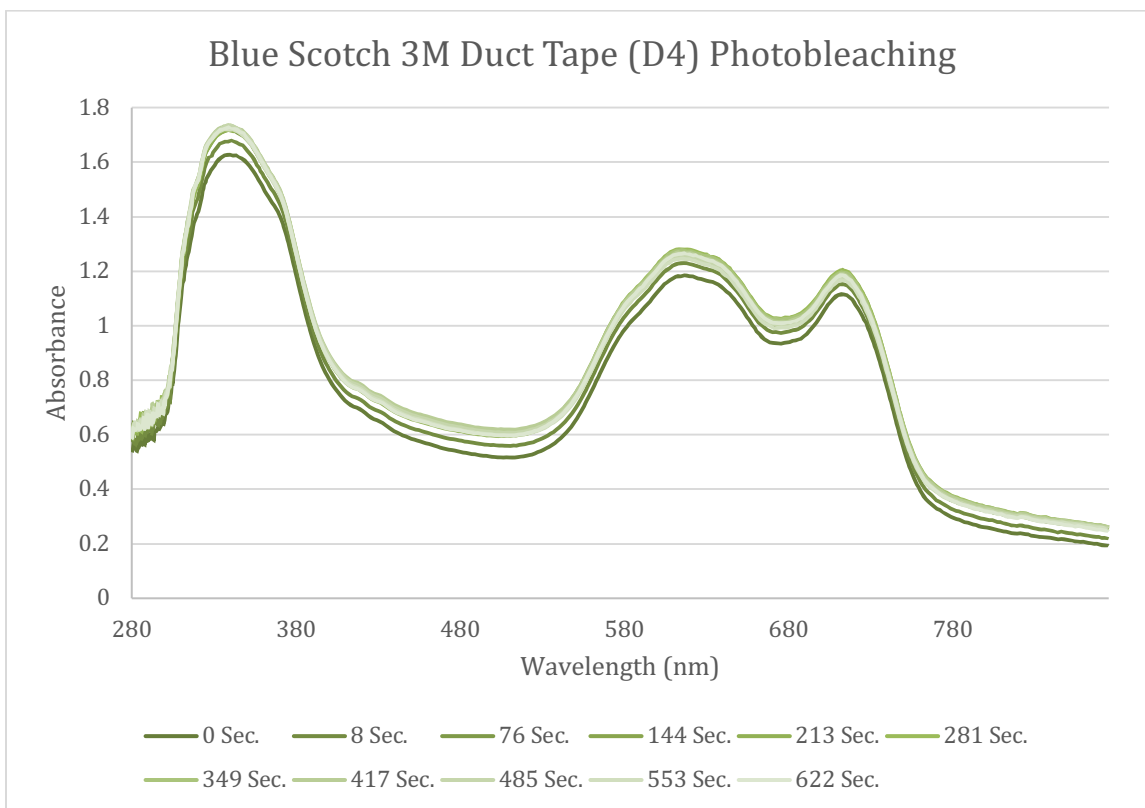


Figure 20.) Orange Scotch 3M Duct Tape (D3) Photobleaching



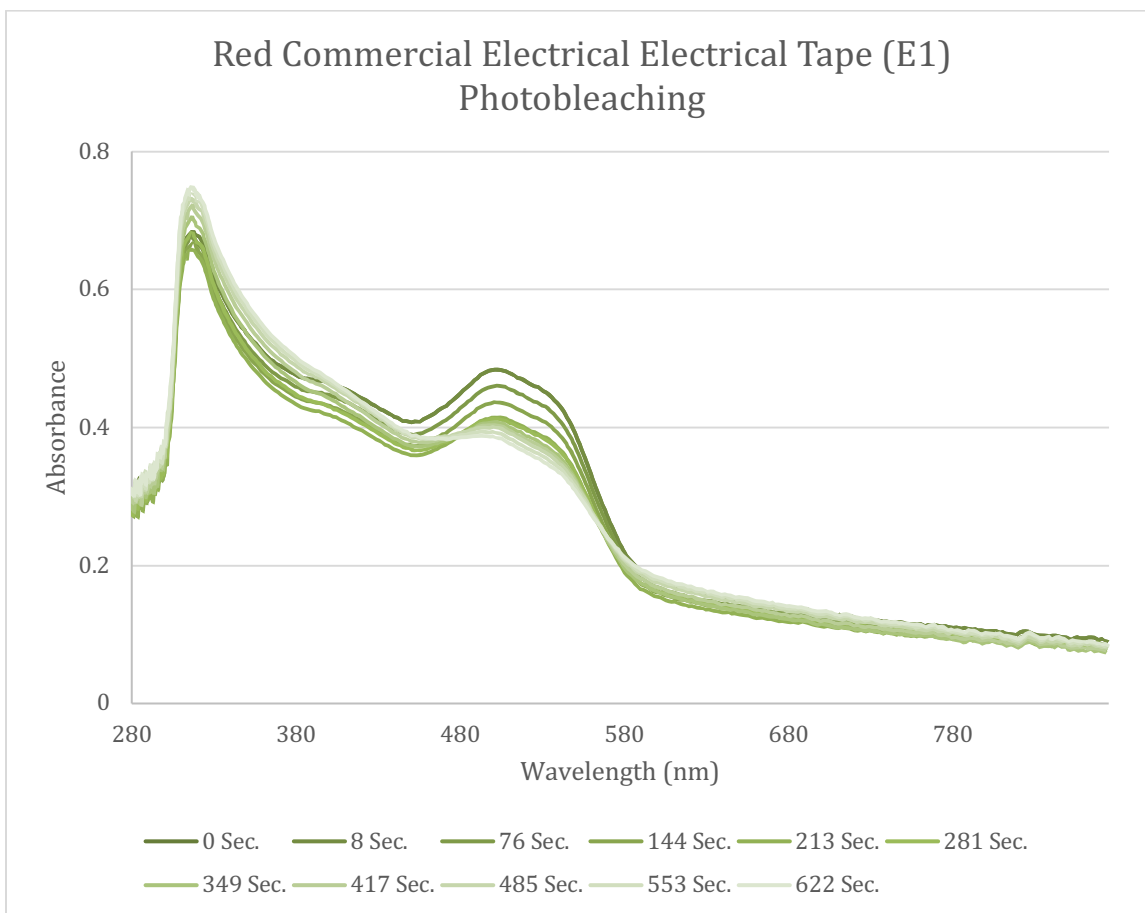
The above figure shows the absorbance spectra of a 15  $\mu\text{m}$  microtomed cross section of sample D3 over 10 minutes of continuous bulb exposure from 280 to 875 nm. No significant photobleaching was observed.

Figure 21.) Blue Scotch 3M Duct Tape (D4) Photobleaching



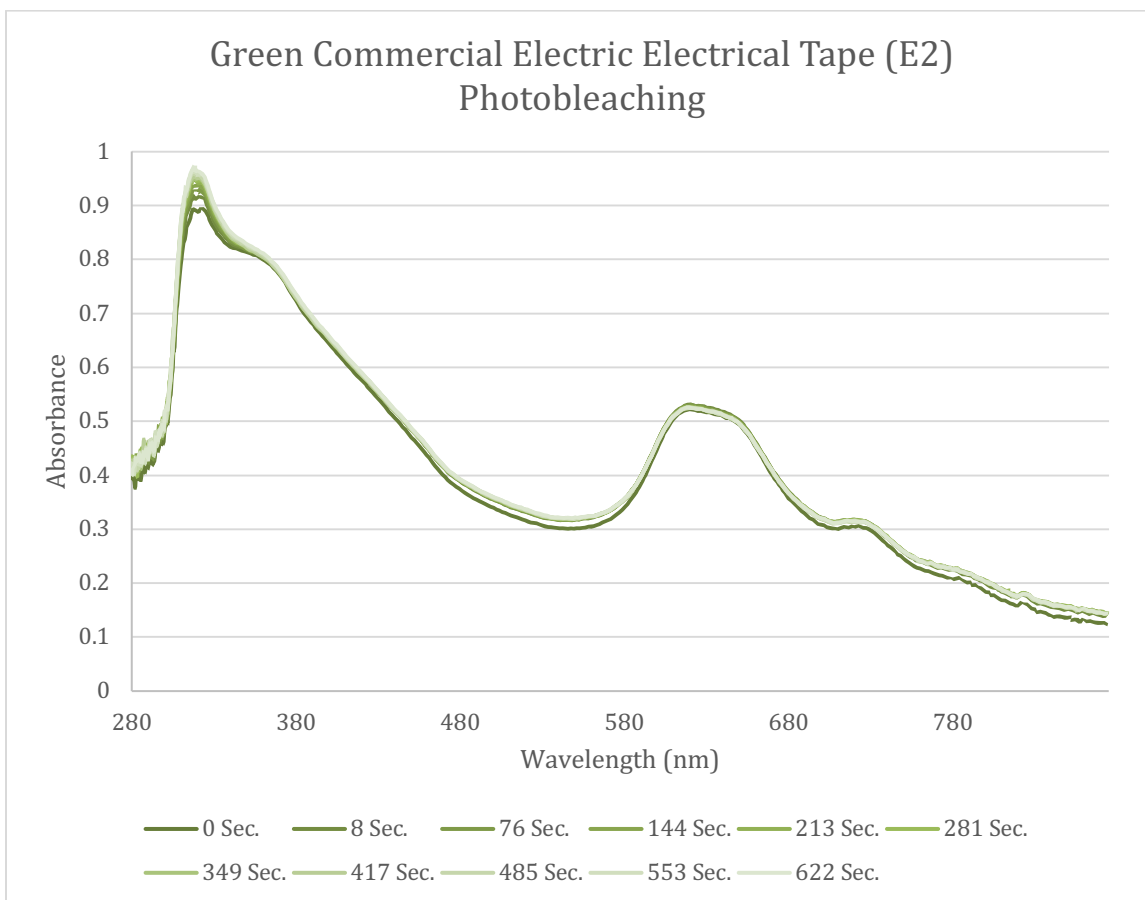
The above figure shows the absorbance spectra of a 15  $\mu\text{m}$  microtomed cross section of sample D4 over 10 minutes of continuous bulb exposure from 280 to 875 nm. No significant photobleaching was observed.

Figure 22.) Red Commercial Electric Electrical Tape (E1) Photobleaching



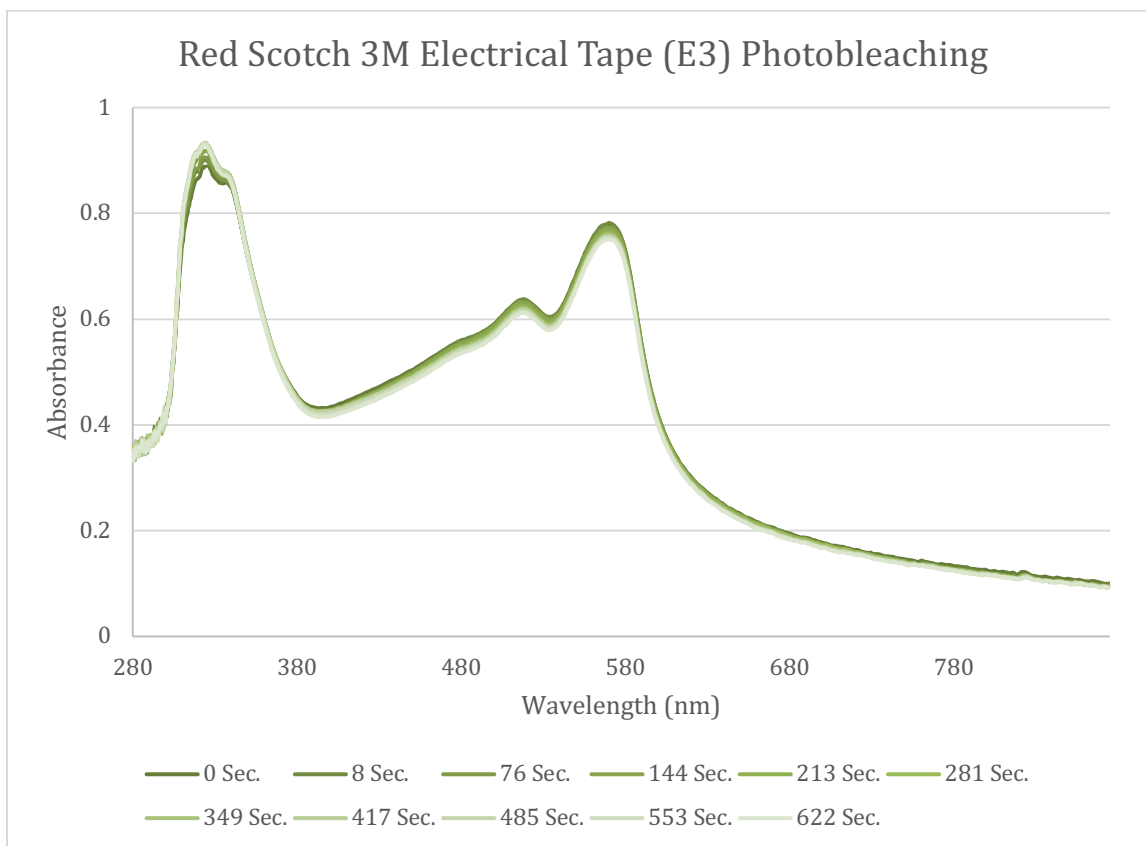
The above figure shows the absorbance spectra of a 15  $\mu\text{m}$  microtomed cross section of sample E1 over 10 minutes of continuous bulb exposure from 280 to 875 nm. No significant photobleaching was observed.

Figure 23.) Green Commercial Electric Electrical Tape (E2) Photobleaching



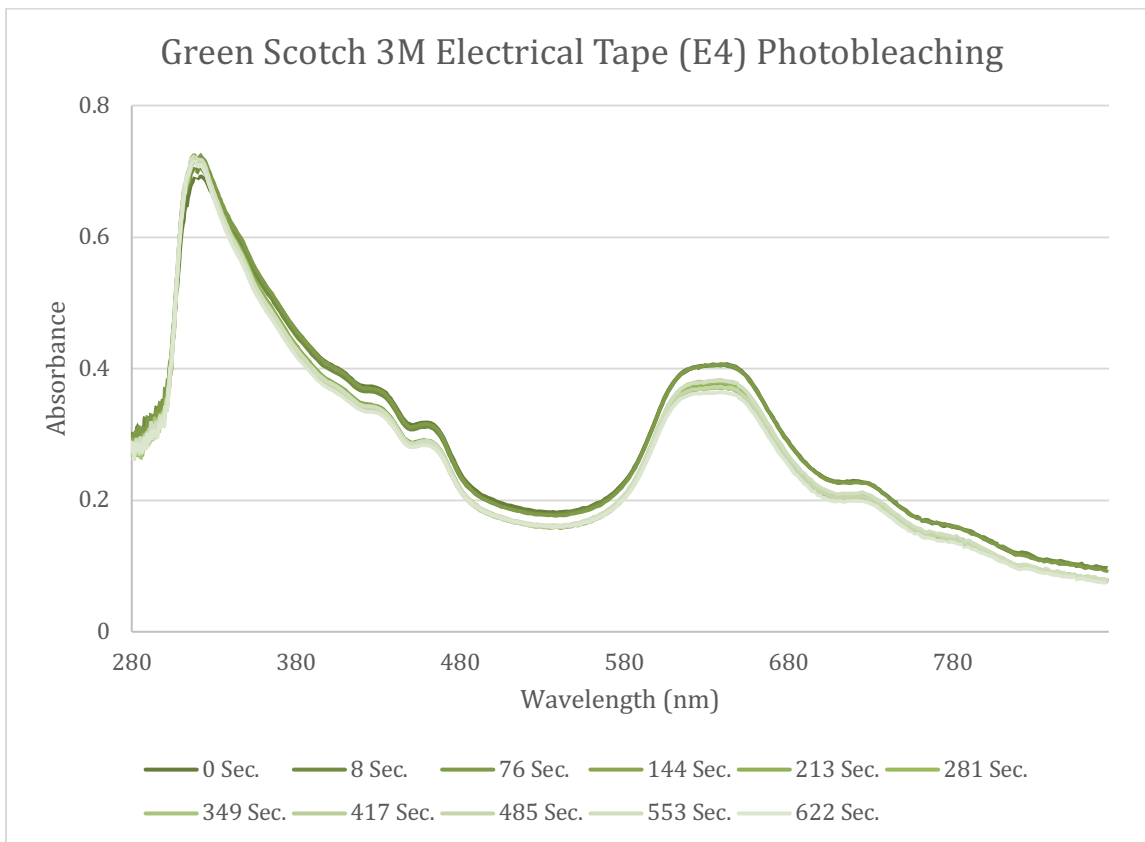
The above figure shows the absorbance spectra of a 15  $\mu\text{m}$  microtomed cross section of sample E2 over 10 minutes of continuous bulb exposure from 280 to 875 nm. No significant photobleaching was observed.

Figure 24.) Red Scotch 3M Electrical Tape (E3) Photobleaching



The above figure shows the absorbance spectra of a 15  $\mu\text{m}$  microtomed cross section of sample E3 over 10 minutes of continuous bulb exposure from 280 to 875 nm. No significant photobleaching was observed.

Figure 25.) Green Scotch 3M Electrical Tape (E4) Photobleaching



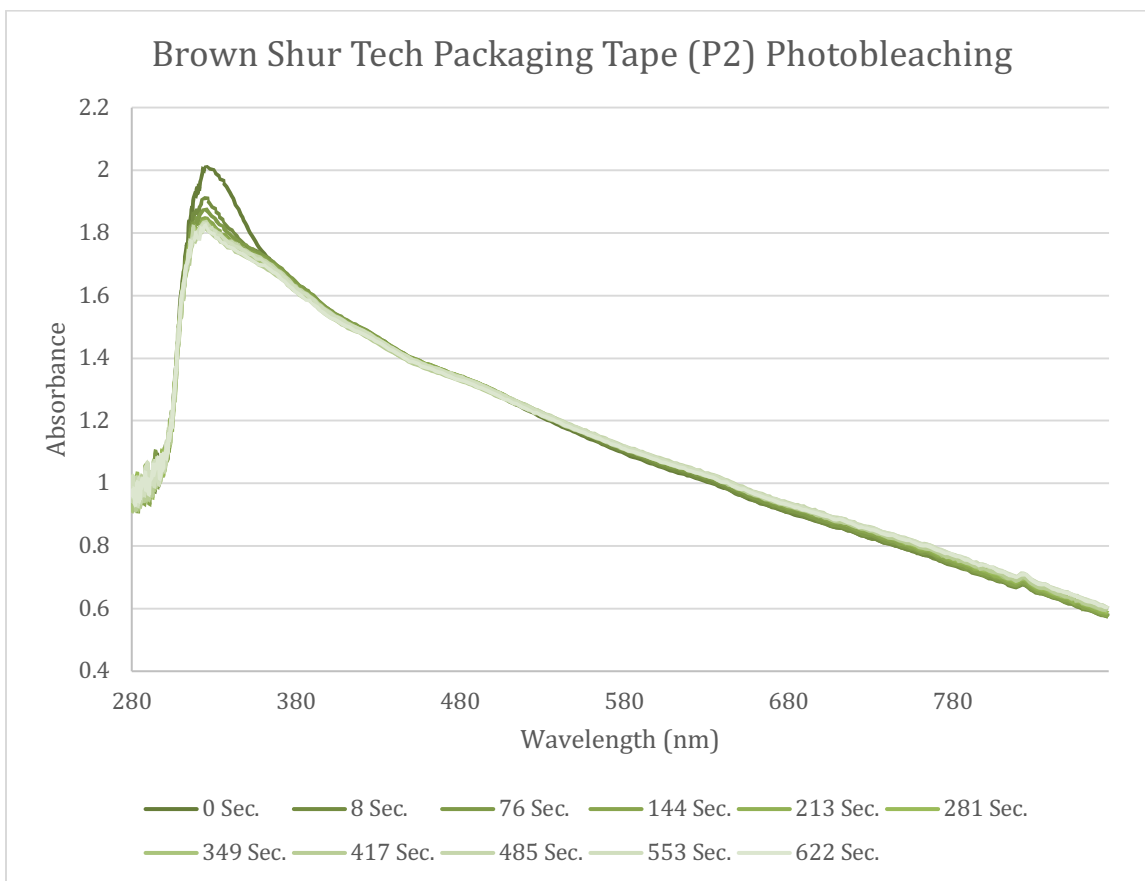
The above figure shows the absorbance spectra of a 15  $\mu\text{m}$  microtomed cross section of sample E4 over 10 minutes of continuous bulb exposure from 280 to 875 nm. No significant photobleaching was observed.

Figure 26.) Brown Scotch 3M Packaging Tape (P1) Photobleaching



The above figure shows the absorbance spectra of a 15  $\mu\text{m}$  microtomed cross section of sample P1 over 10 minutes of continuous bulb exposure from 280 to 875 nm. No significant photobleaching was observed.

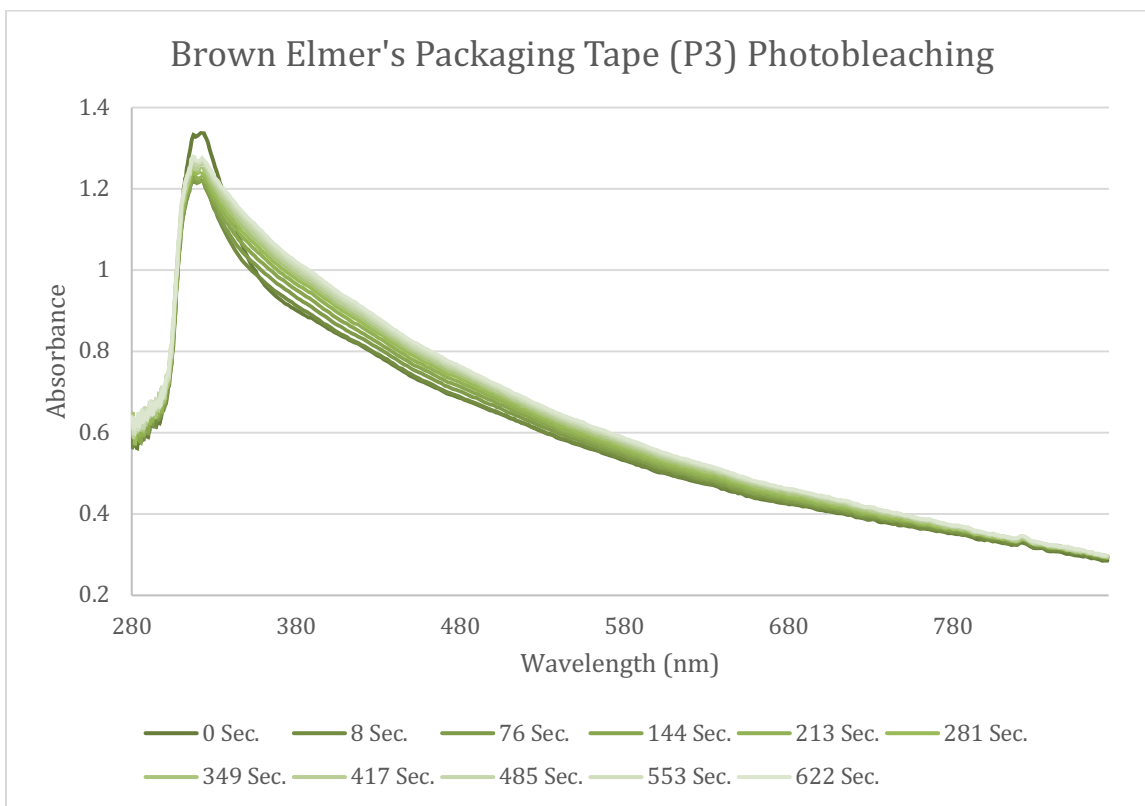
Figure 27.) Brown Shur Tech Packaging Tape (P2) Photobleaching



The above figure shows the absorbance spectra of a 15  $\mu\text{m}$  microtomed cross section of sample P2 over 10 minutes of continuous bulb exposure from 280 to 875 nm. No significant photobleaching was observed.

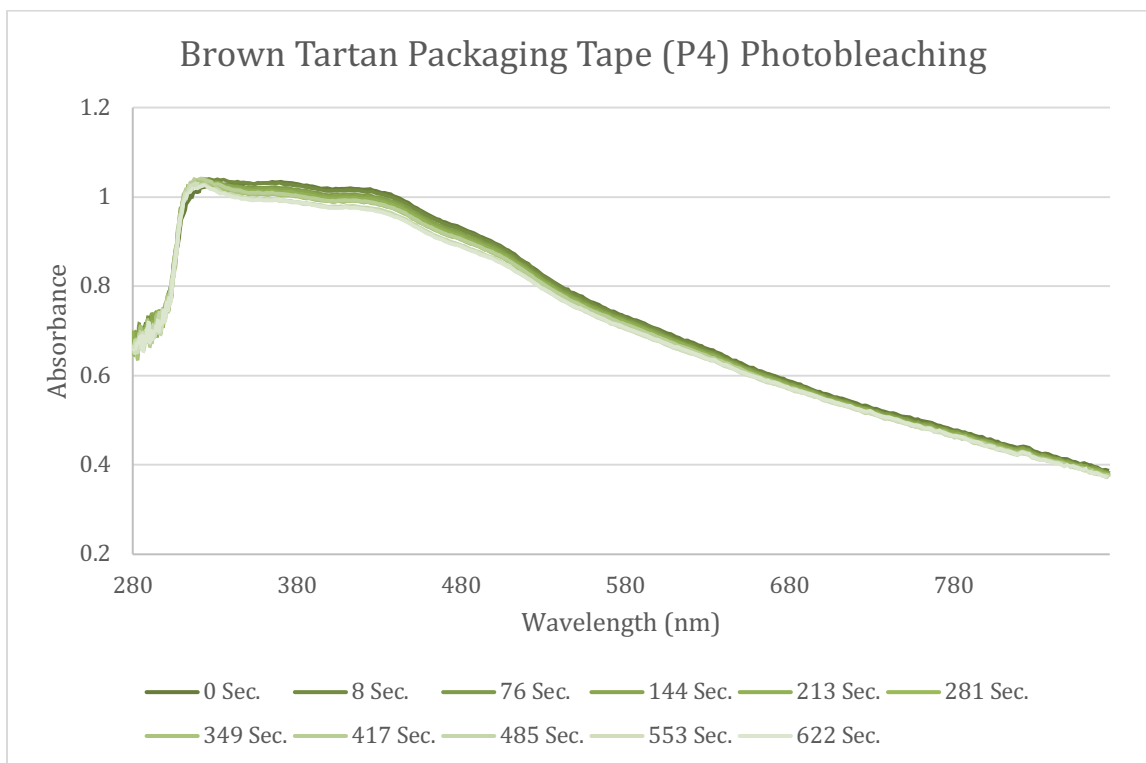


Figure 28.) Brown Elmer's Packaging Tape (P3) Photobleaching



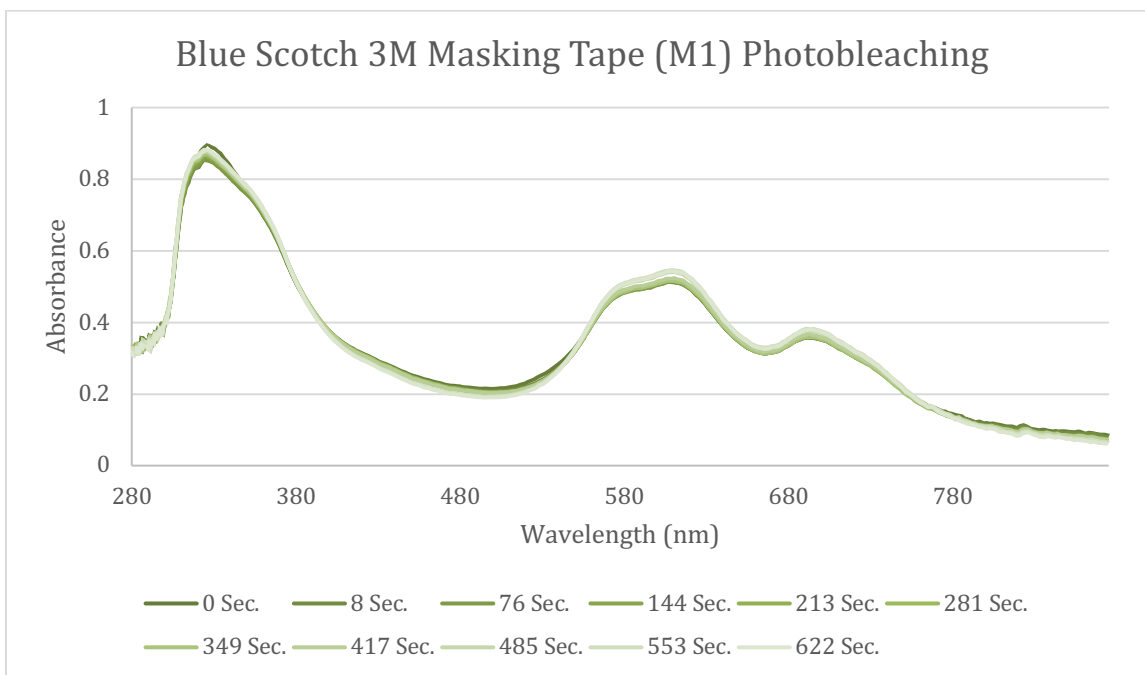
The above figure shows the absorbance spectra of a 15  $\mu\text{m}$  microtomed cross section of sample P3 over 10 minutes of continuous bulb exposure from 280 to 875 nm. No significant photobleaching was observed.

Figure 29.) Brown Tartan Packaging Tape (P4) Photobleaching



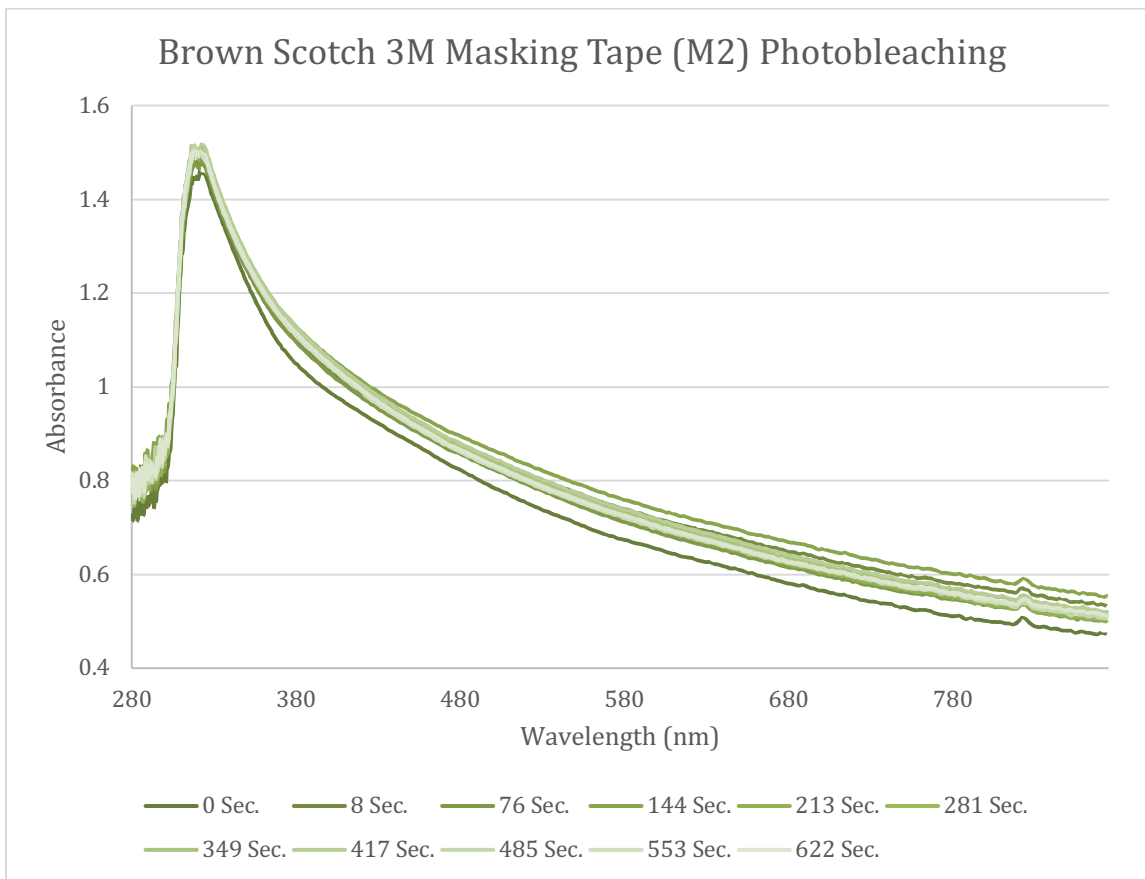
The above figure shows the absorbance spectra of a 15 µm microtomed cross section of sample P4 over 10 minutes of continuous bulb exposure from 280 to 875 nm. No significant photobleaching was observed.

Figure 30.) Blue Scotch 3M Masking Tape (M1) Photobleaching



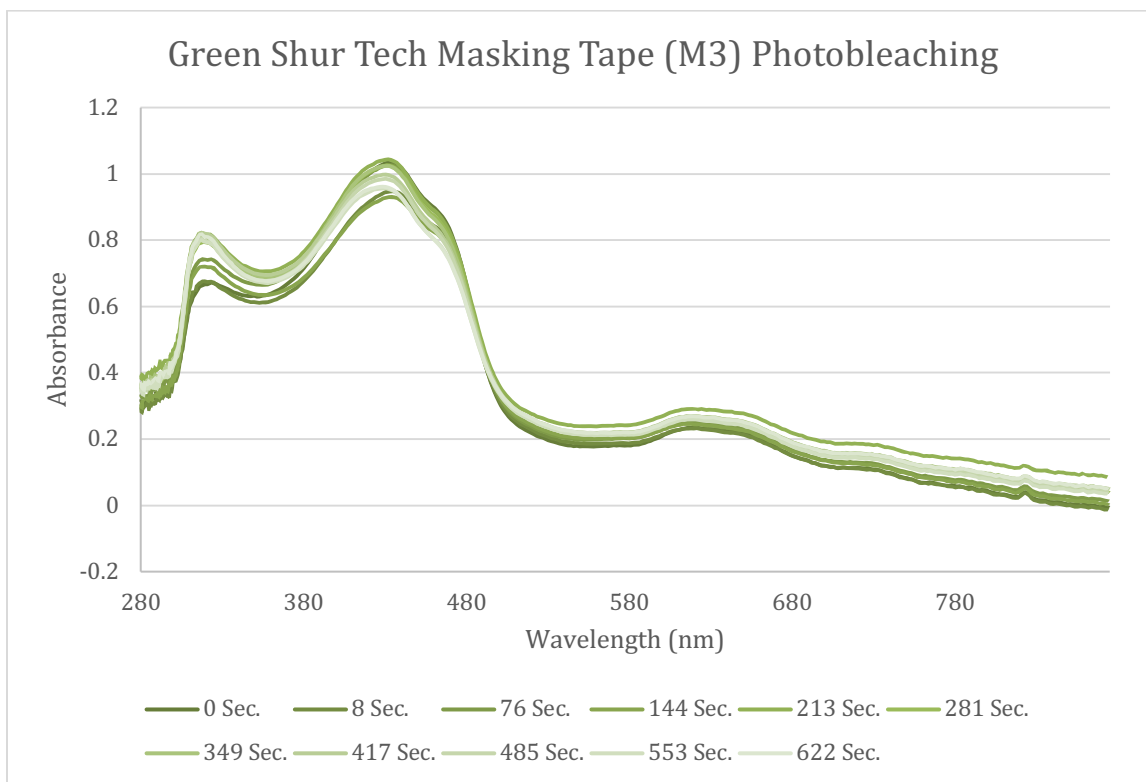
The above figure shows the absorbance spectra of a 15  $\mu\text{m}$  microtomed cross section of sample M1 over 10 minutes of continuous bulb exposure from 280 to 875 nm. No significant photobleaching was observed.

Figure 31.) Brown Scotch 3M Masking Tape (M2) Photobleaching



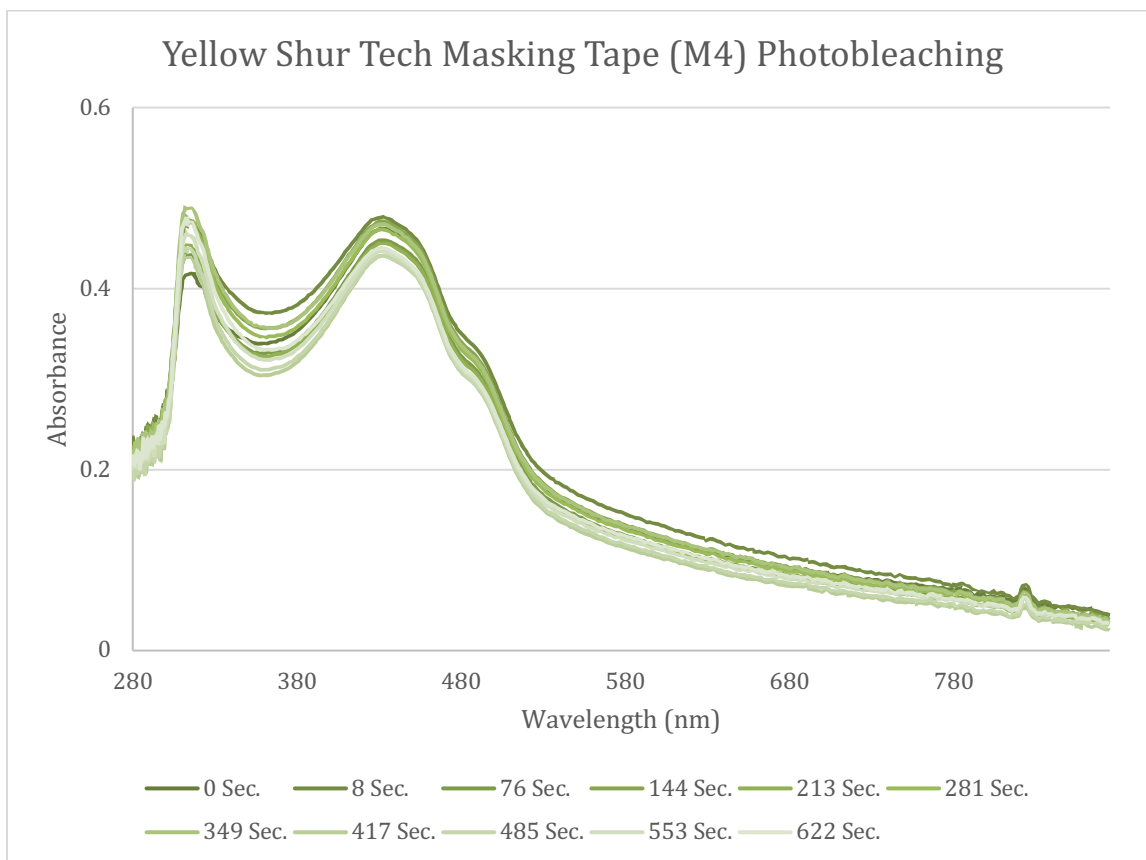
The above figure shows the absorbance spectra of a 15  $\mu\text{m}$  microtomed cross section of sample M2 over 10 minutes of continuous bulb exposure from 280 to 875 nm. No significant photobleaching was observed.

Figure 32.) Green Shur Tech Masking Tape (M3) Photobleaching



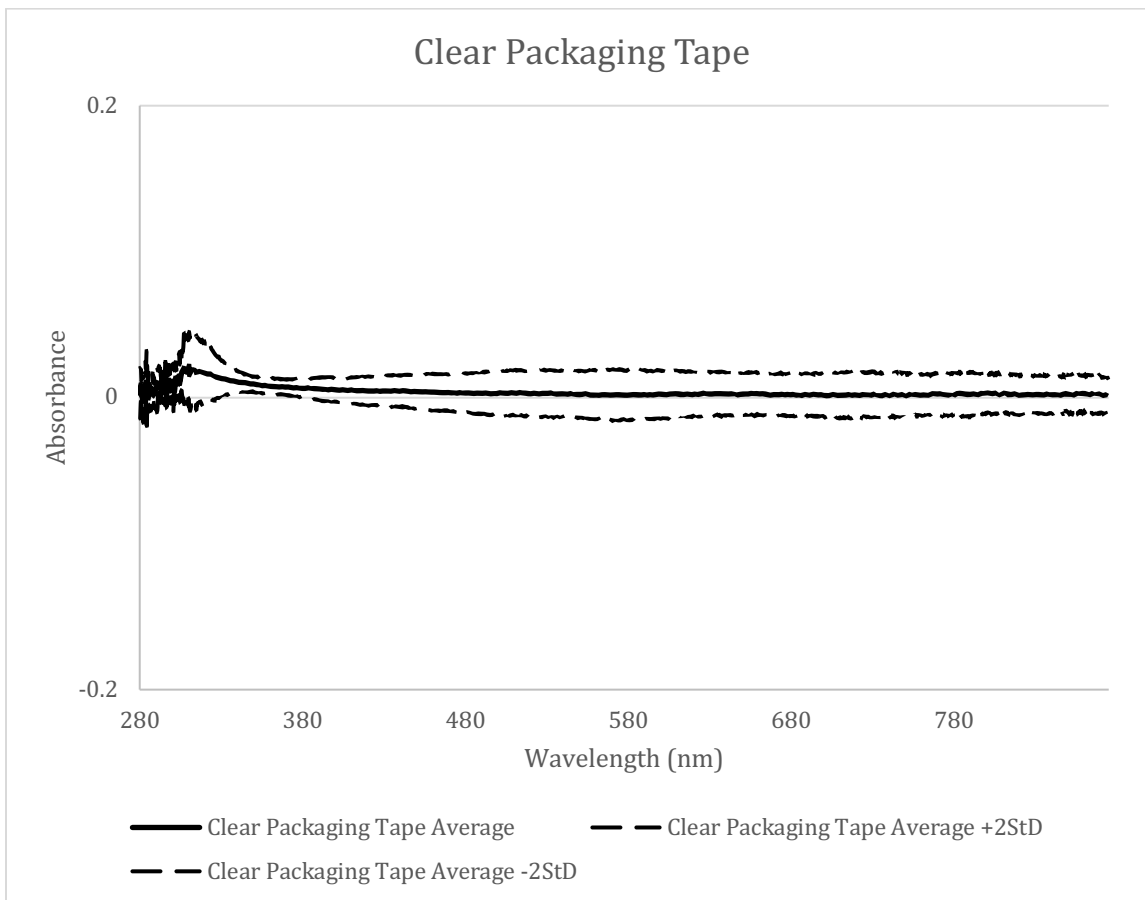
The above figure shows the absorbance spectra of a 15  $\mu\text{m}$  microtomed cross section of sample M3 over 10 minutes of continuous bulb exposure from 280 to 875 nm. No significant photobleaching was observed.

Figure 33.) Yellow Shur Tech Masking Tape (M4) Photobleaching



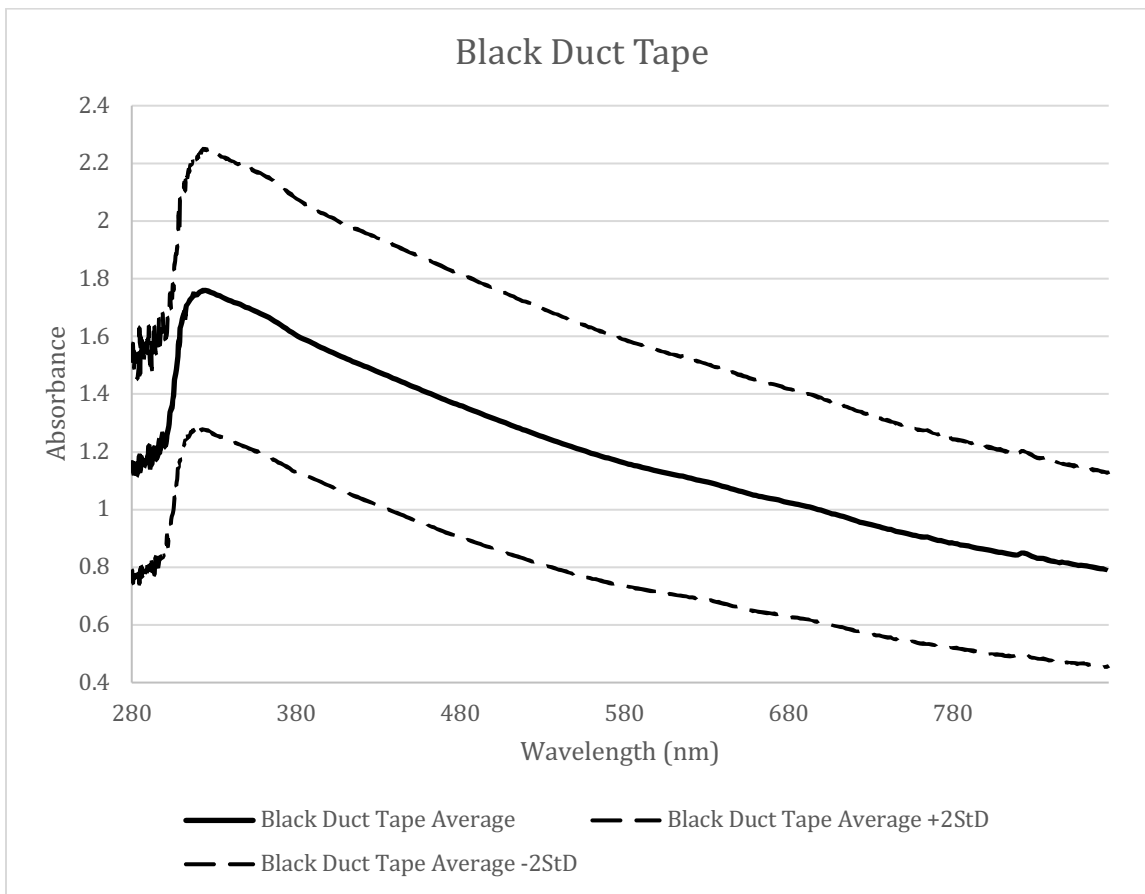
The above figure shows the absorbance spectra of a 15  $\mu\text{m}$  microtomed cross section of sample M4 over 10 minutes of continuous bulb exposure from 280 to 875 nm. No significant photobleaching was observed.

Figure 34.) Clear Packaging Tape



The above figure shows the average absorbance spectra through a 30  $\mu\text{m}$  microtomed cross section of clear packaging tape from 280 to 875 nm. A solid line indicates the average of ten scans and a pair of dashed lines indicates plus or minus two standard deviations from the mean.

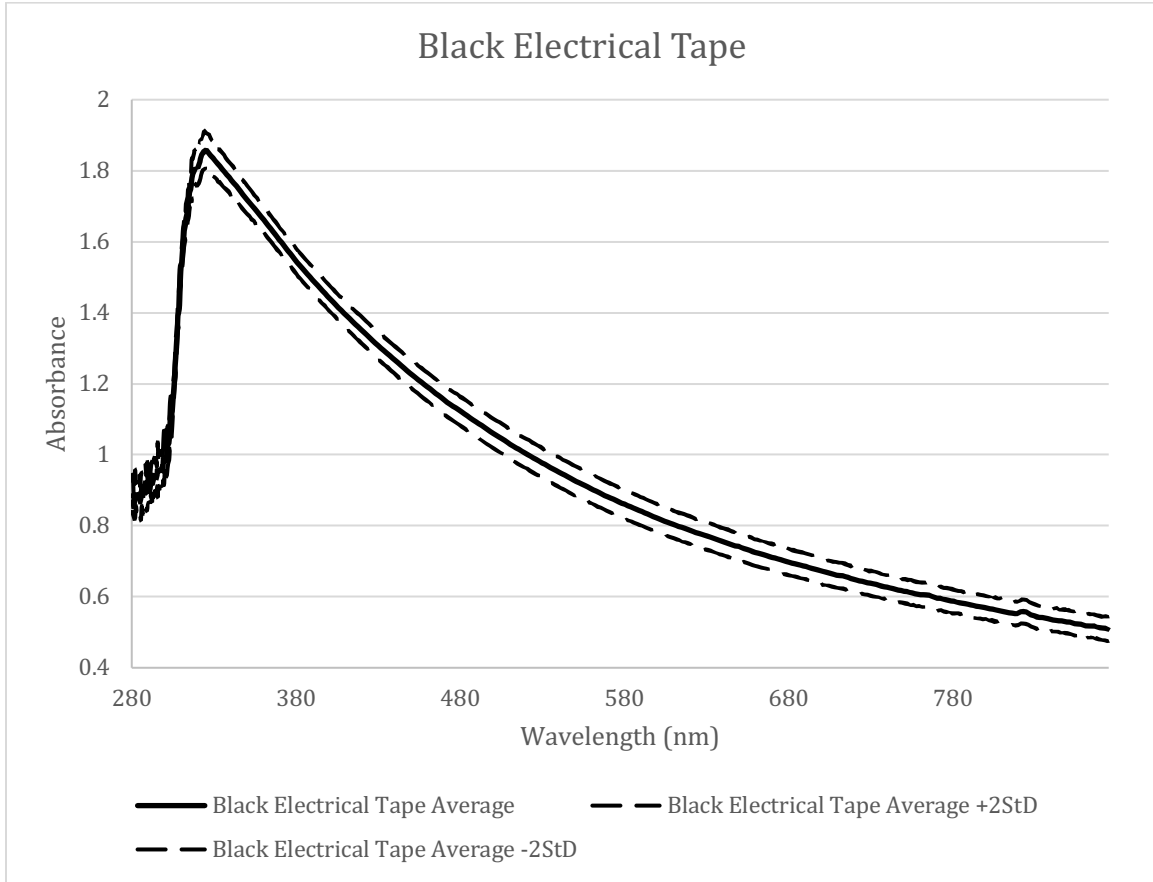
Figure 35.) Black Duct Tape



The above figure shows the average absorbance spectra through a 30  $\mu\text{m}$  microtomed cross section of black duct tape from 280 to 875 nm. A solid line indicates the average of ten scans and a pair of dashed lines indicates plus or minus two standard deviations from the mean.

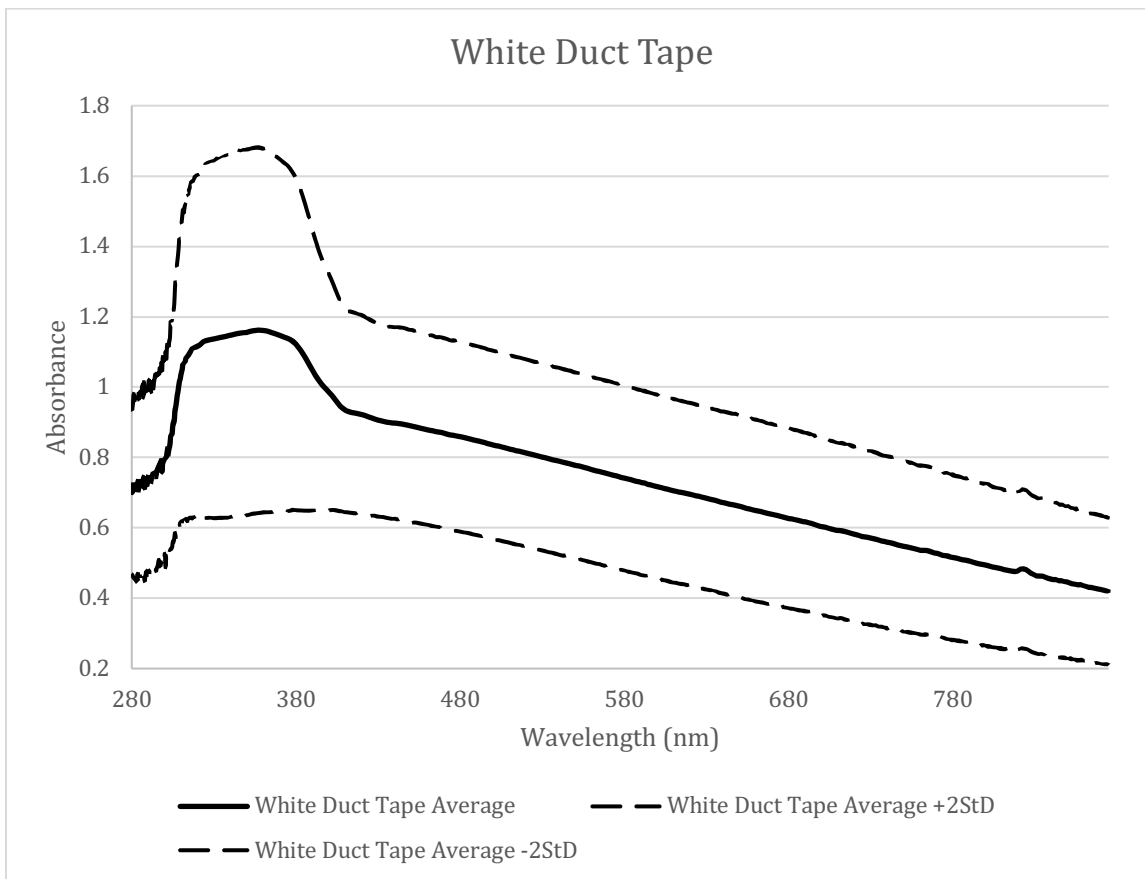


Figure 36.) Black Electrical Tape



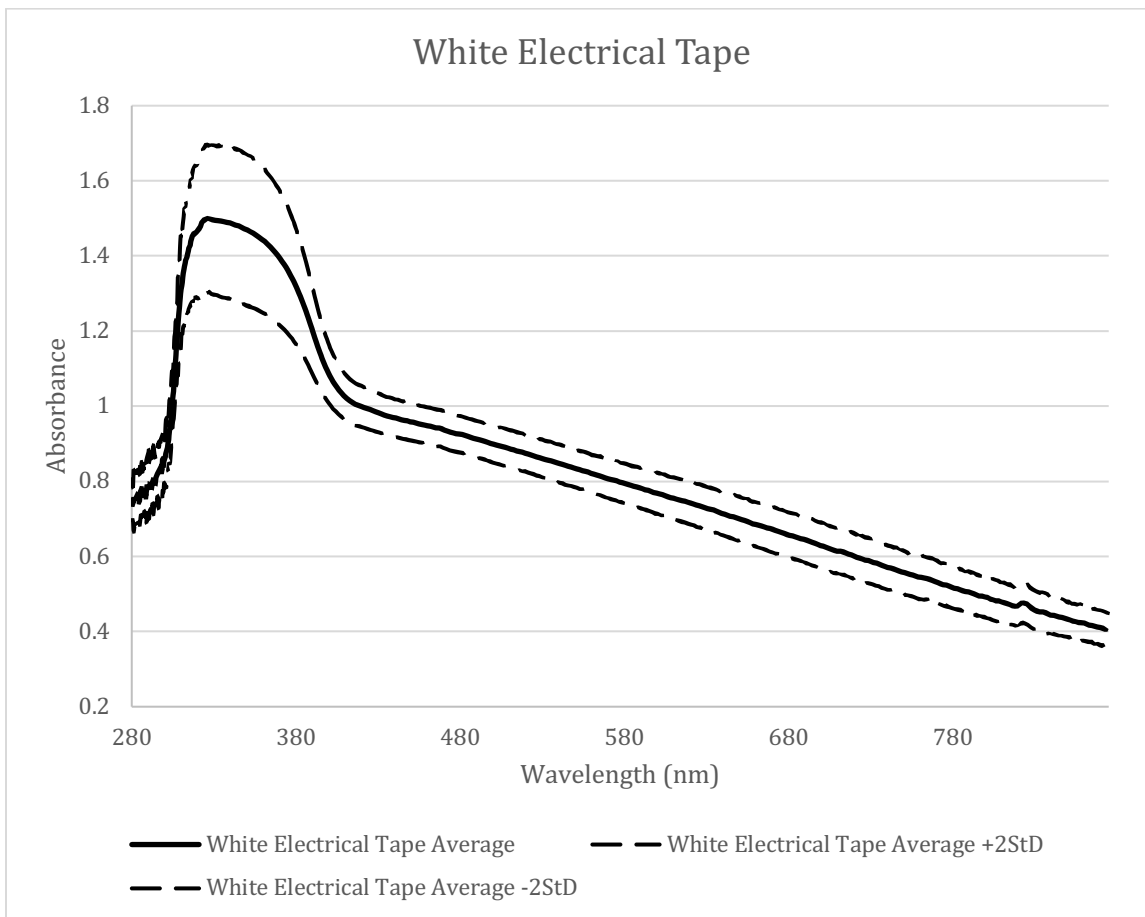
The above figure shows the average absorbance spectra through a 30  $\mu\text{m}$  microtomed cross section of black electrical tape from 280 to 875 nm. A solid line indicates the average of ten scans and a pair of dashed lines indicates plus or minus two standard deviations from the mean.

Figure 37.) White Duct Tape



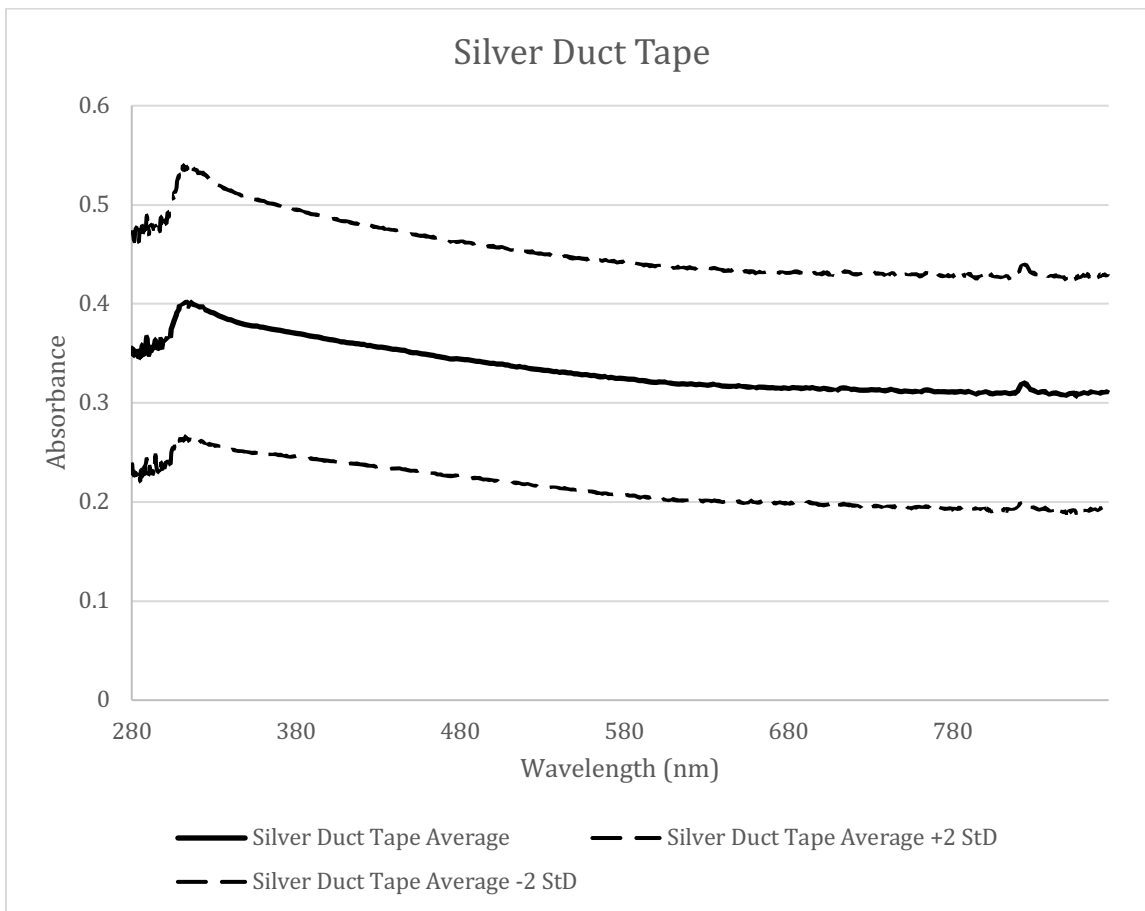
The above figure shows the average absorbance spectra through a 30  $\mu\text{m}$  microtomed cross section of white duct tape from 280 to 875 nm. A solid line indicates the average of ten scans and a pair of dashed lines indicates plus or minus two standard deviations from the mean.

Figure 38.) White Electrical Tape



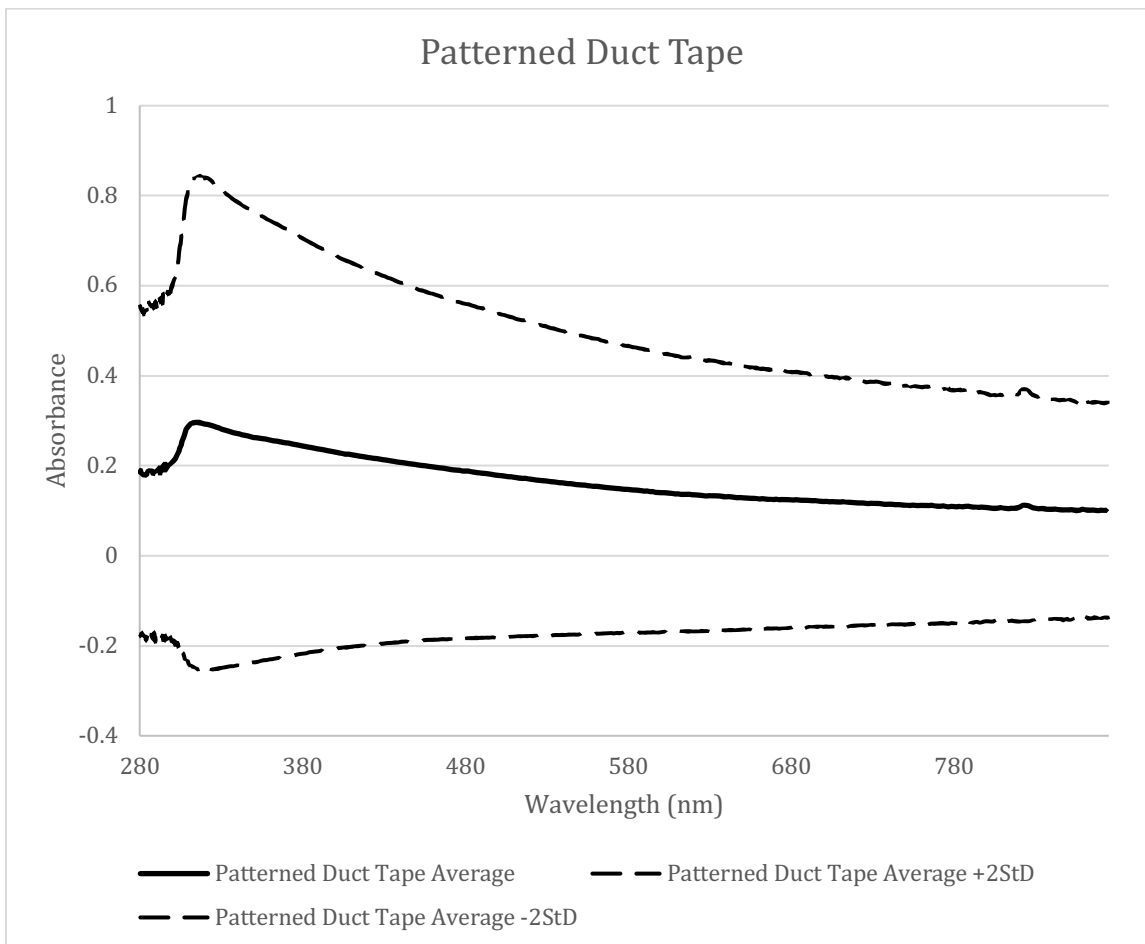
The above figure shows the average absorbance spectra through a 30  $\mu\text{m}$  microtomed cross section of white electrical tape from 280 to 875 nm. A solid line indicates the average of ten scans and a pair of dashed lines indicates plus or minus two standard deviations from the mean.

Figure 39.) Silver Duct Tape



The above figure shows the average absorbance spectra through a 30  $\mu\text{m}$  microtomed cross section of silver-colored duct tape from 280 to 875 nm. A solid line indicates the average of ten scans and a pair of dashed lines indicates plus or minus two standard deviations from the mean.

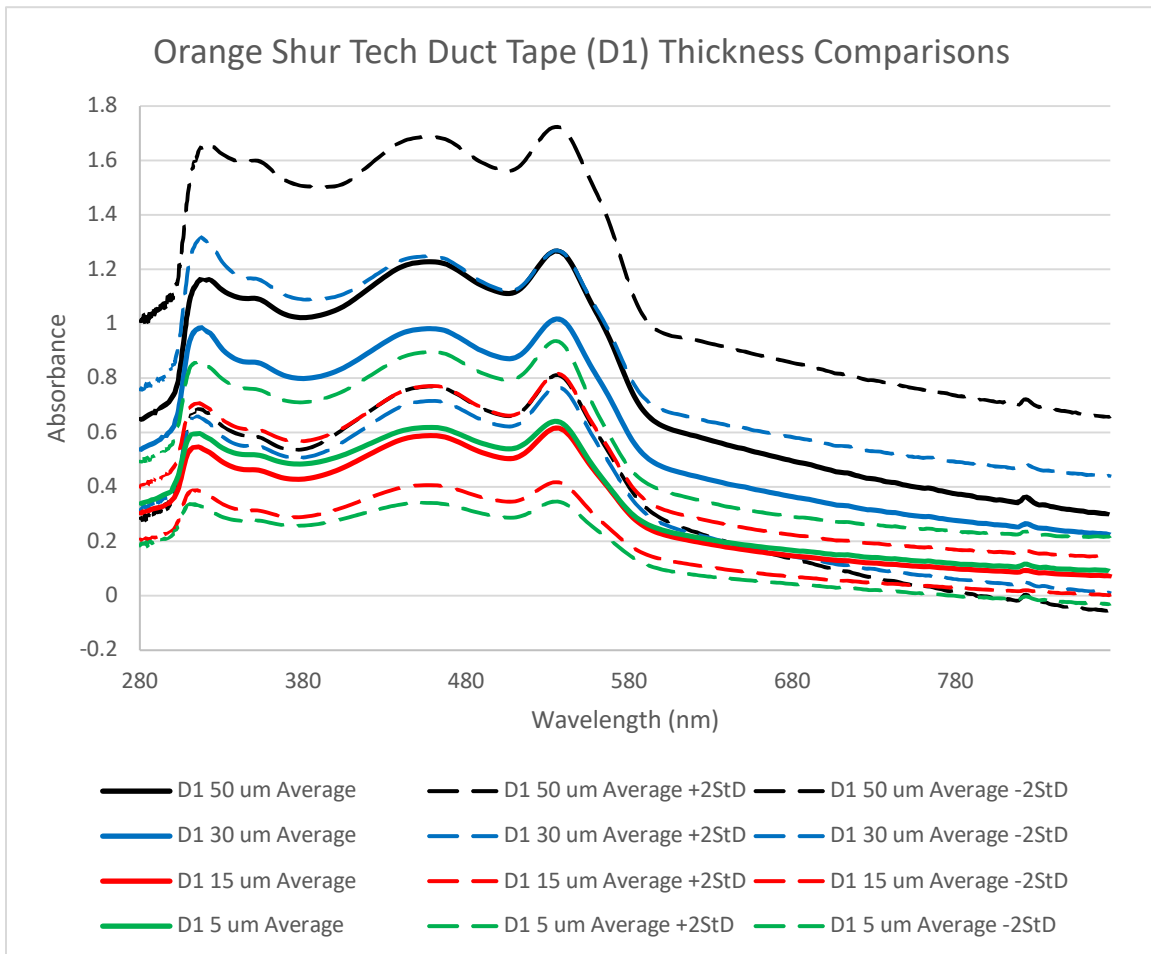
Figure 40.) Patterned Duct Tape



The above figure shows the average absorbance spectra through a 30  $\mu\text{m}$  microtomed cross section of patterned duct tape from 280 to 875 nm. A solid line indicates the average of ten scans and a pair of dashed lines indicates plus or minus two standard deviations from the mean.

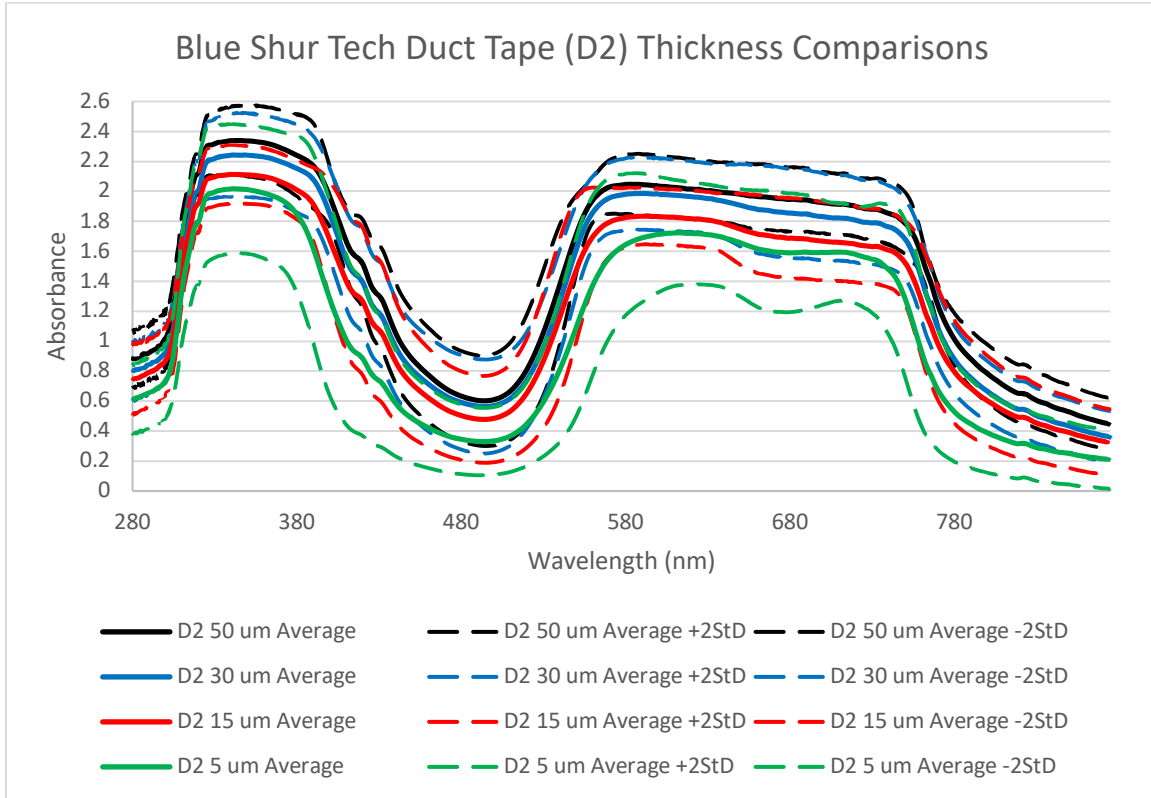
## Appendix B

Figure 41.) Orange Shur Tech Duct Tape (D1) Thickness Comparisons



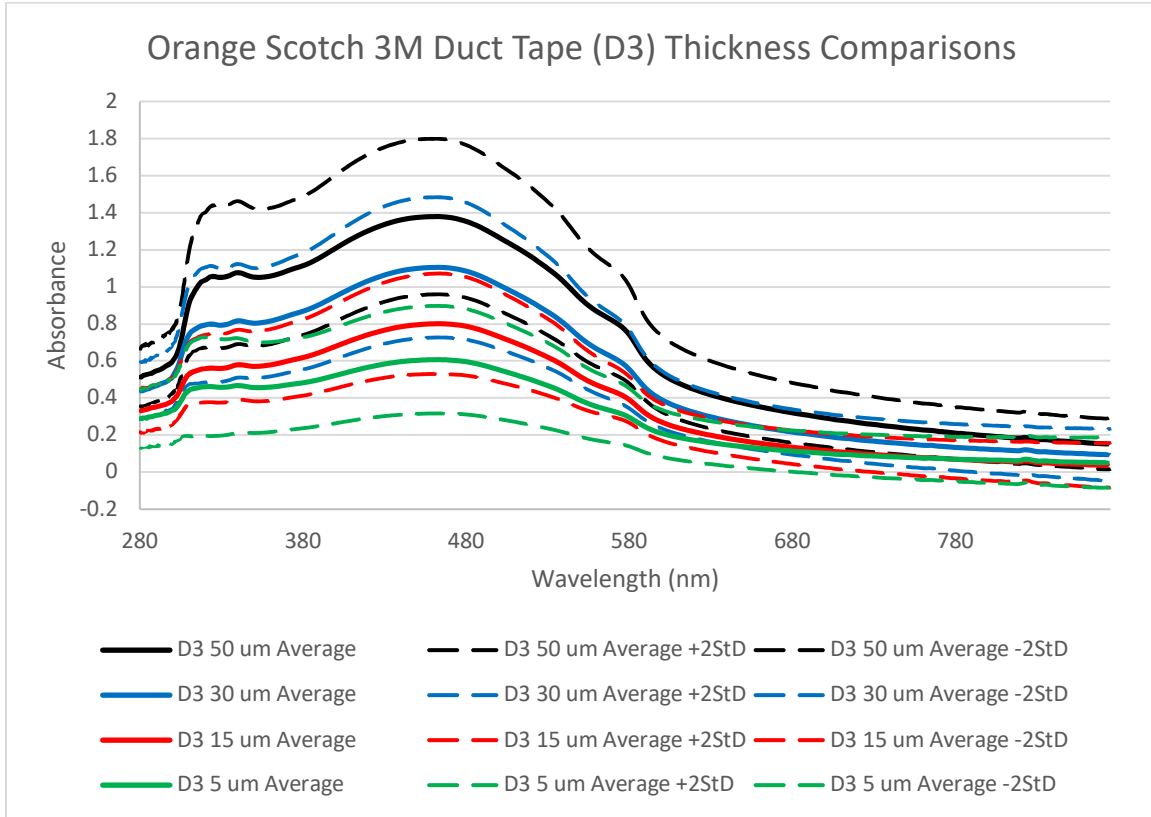
The above figure compares the average absorbance spectra through 50  $\mu\text{m}$  microtomed cross sections, 30  $\mu\text{m}$  microtomed cross sections, 15  $\mu\text{m}$  microtomed cross sections, and 5  $\mu\text{m}$  microtomed cross sections of orange Shur Tech duct tape (D1) from 280 to 875 nm. A solid line indicates the average of fifty scans and a pair of dashed lines indicates plus or minus two standard deviations from the mean.

Figure 42.) Blue Shur Tech Duct Tape (D2) Thickness Comparisons



The above figure compares the average absorbance spectra through 50 µm microtomed cross sections, 30 µm microtomed cross sections, 15 µm microtomed cross sections, and 5 µm microtomed cross sections of blue Shur Tech duct tape (D2) from 280 to 875 nm. A solid line indicates the average of fifty scans and a pair of dashed lines indicates plus or minus two standard deviations from the mean.

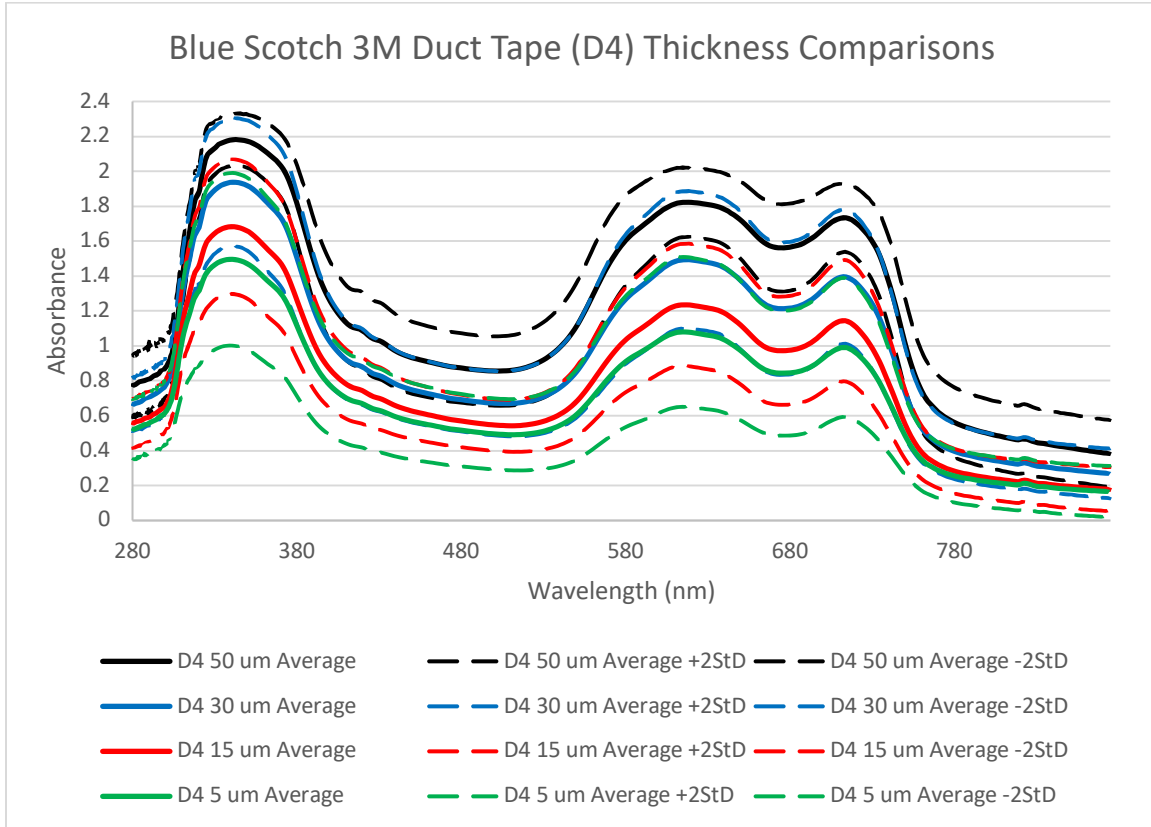
Figure 43.) Orange Scotch 3M Duct Tape (D3) Thickness Comparisons



The above figure compares the average absorbance spectra through 50  $\mu\text{m}$  microtomed cross sections, 30  $\mu\text{m}$  microtomed cross sections, 15  $\mu\text{m}$  microtomed cross sections, and 5  $\mu\text{m}$  microtomed cross sections of orange Scotch 3M duct tape (D3) from 280 to 875 nm. A solid line indicates the average of fifty scans and a pair of dashed lines indicates plus or minus two standard deviations from the mean.

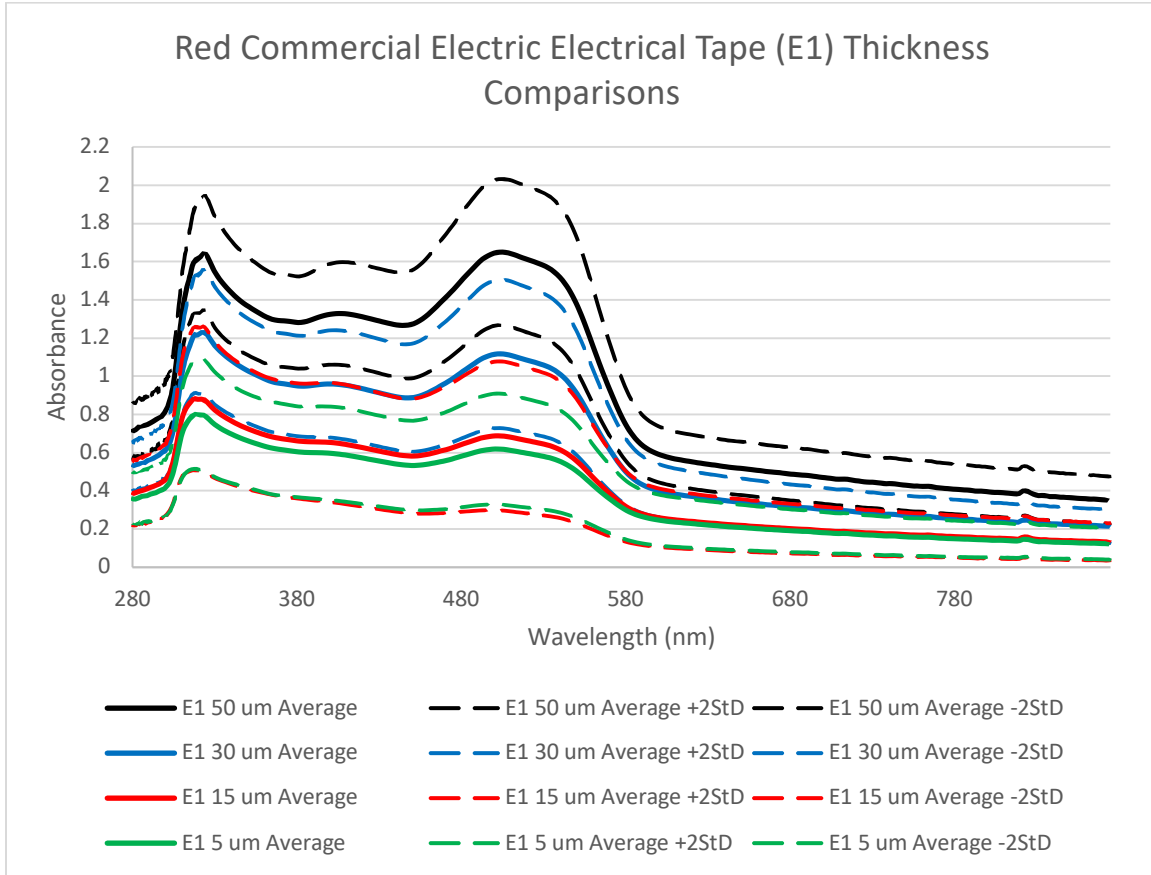


Figure 44.) Blue Scotch 3M Duct Tape (D4) Thickness Comparisons



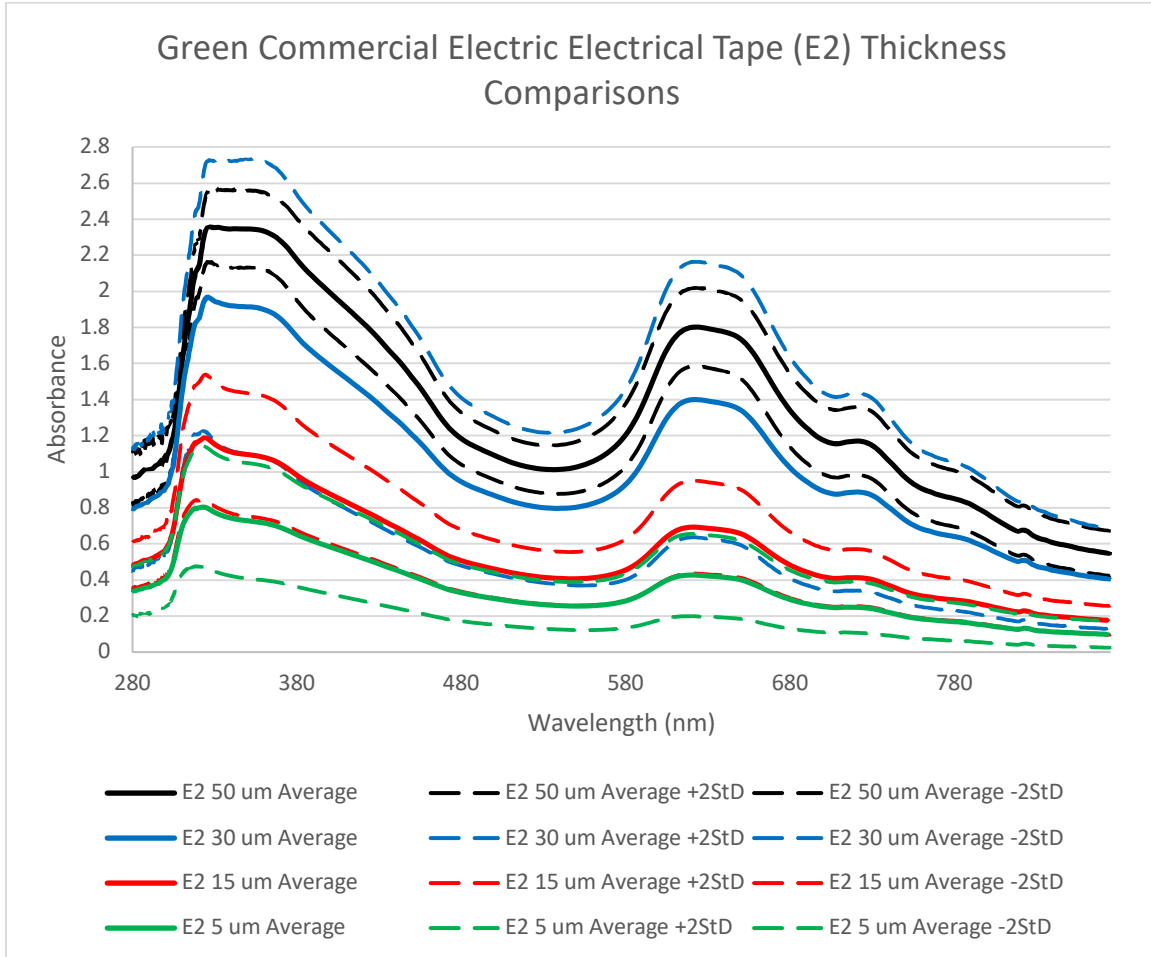
The above figure compares the average absorbance spectra through 50 µm microtomed cross sections, 30 µm microtomed cross sections, 15 µm microtomed cross sections, and 5 µm microtomed cross sections of blue Scotch 3M duct tape (D4) from 280 to 875 nm. A solid line indicates the average of fifty scans and a pair of dashed lines indicates plus or minus two standard deviations from the mean.

Figure 45.) Red Commercial Electric Electrical Tape (E1) Thickness Comparisons



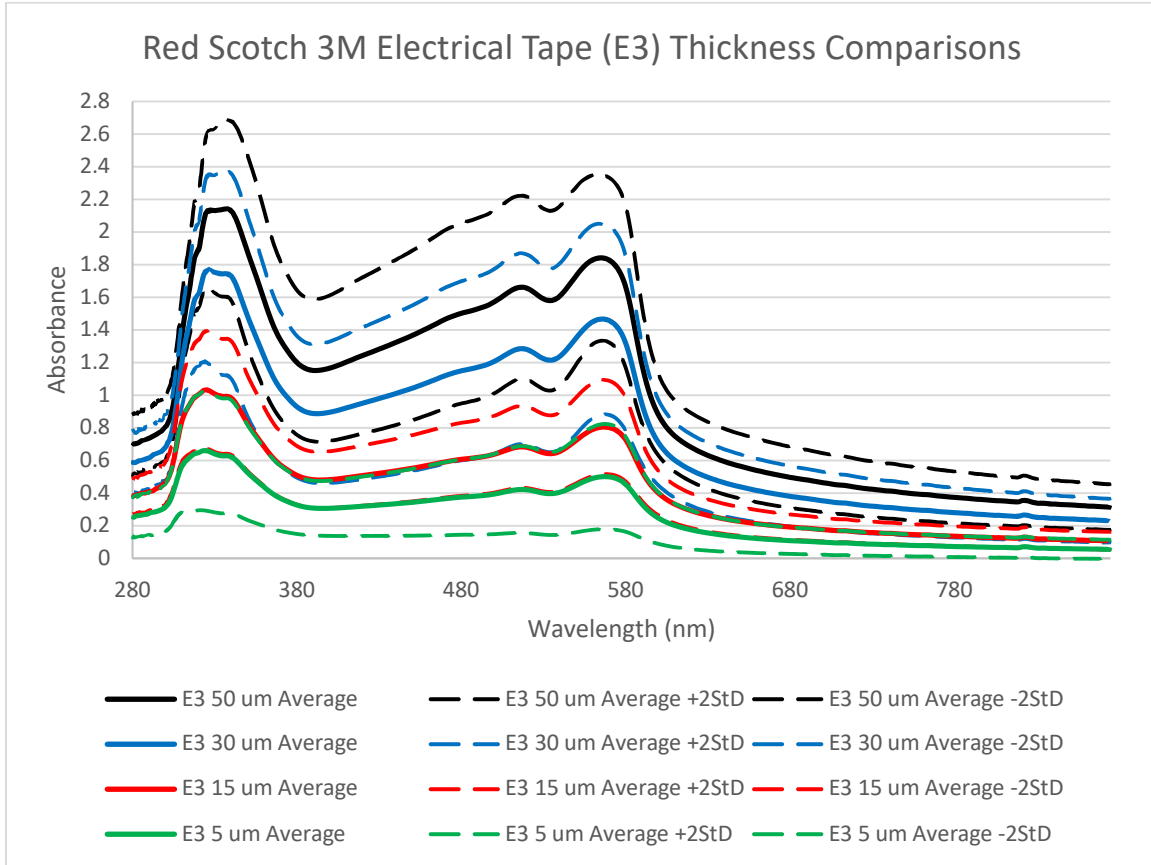
The above figure compares the average absorbance spectra through 50  $\mu\text{m}$  microtomed cross sections, 30  $\mu\text{m}$  microtomed cross sections, 15  $\mu\text{m}$  microtomed cross sections, and 5  $\mu\text{m}$  microtomed cross sections of red commercial electric electrical tape (E1) from 280 to 875 nm. A solid line indicates the average of fifty scans and a pair of dashed lines indicates plus or minus two standard deviations from the mean.

Figure 46.) Green Commercial Electric Electrical Tape (E2) Thickness Comparisons



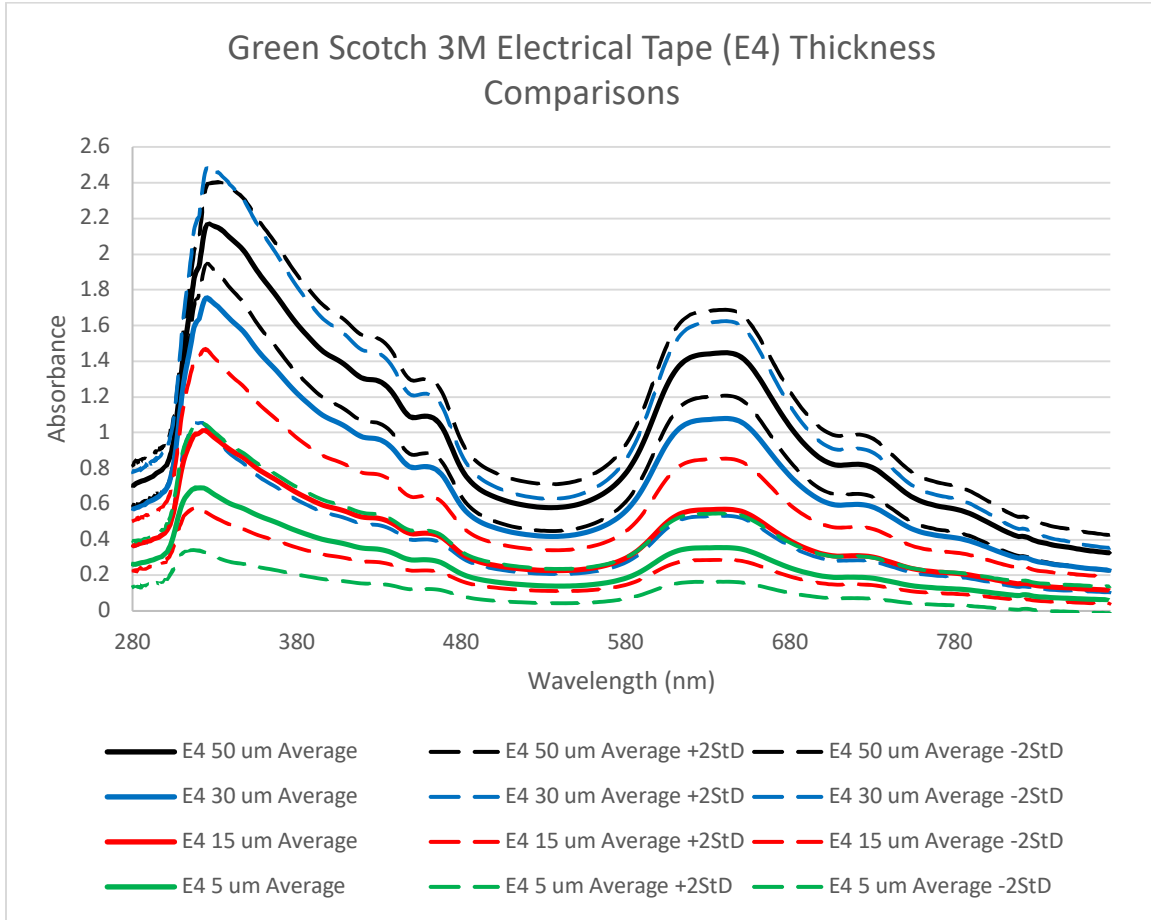
The above figure compares the average absorbance spectra through 50 μm microtomed cross sections, 30 μm microtomed cross sections, 15 μm microtomed cross sections, and 5 μm microtomed cross sections of green commercial electric electrical tape (E2) from 280 to 875 nm. A solid line indicates the average of fifty scans and a pair of dashed lines indicates plus or minus two standard deviations from the mean.

Figure 47.) Red Scotch 3M Electrical Tape (E3) Thickness Comparisons



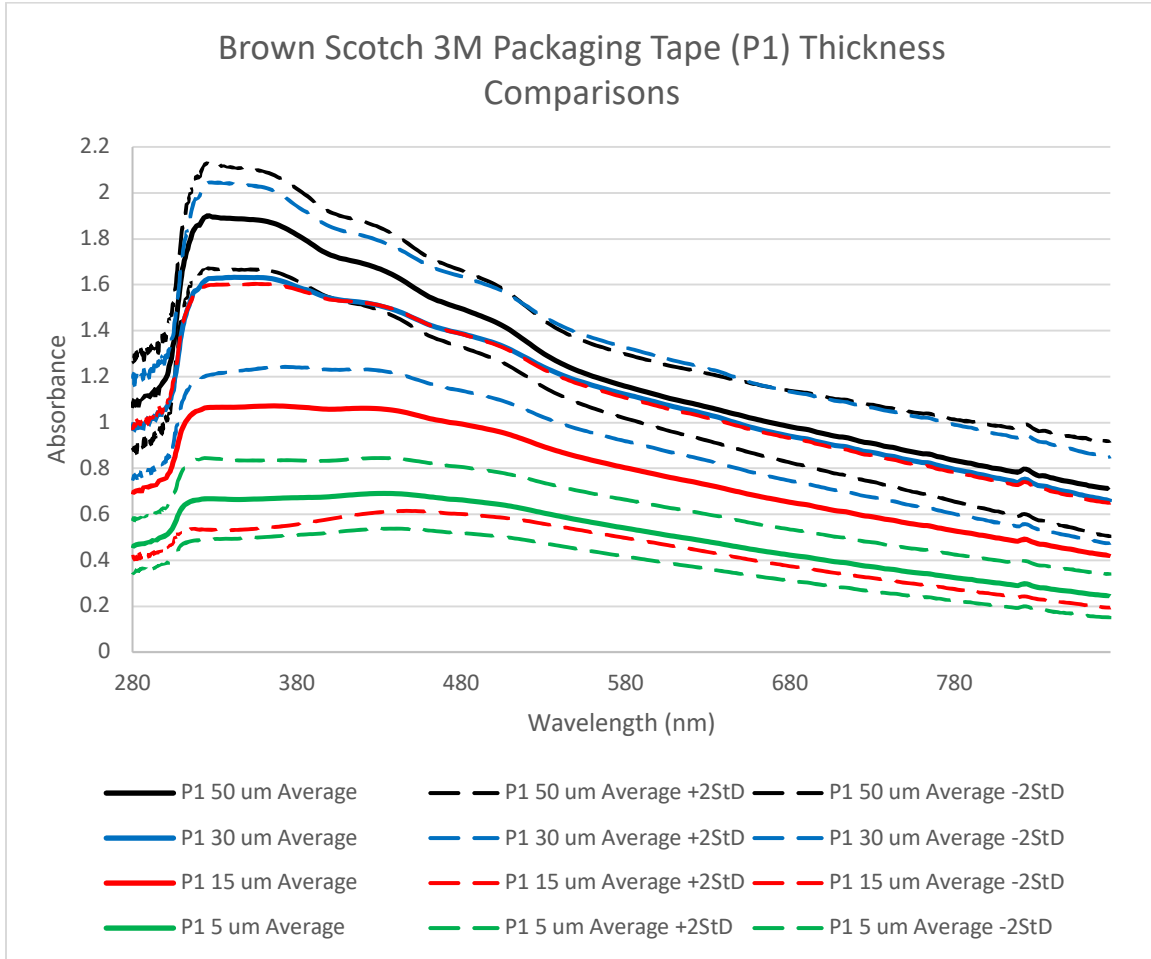
The above figure compares the average absorbance spectra through 50 µm microtomed cross sections, 30 µm microtomed cross sections, 15 µm microtomed cross sections, and 5 µm microtomed cross sections of red Scotch 3M electrical tape (E3) from 280 to 875 nm. A solid line indicates the average of fifty scans and a pair of dashed lines indicates plus or minus two standard deviations from the mean.

Figure 48.) Green Scotch 3M Electrical Tape (E4) Thickness Comparisons



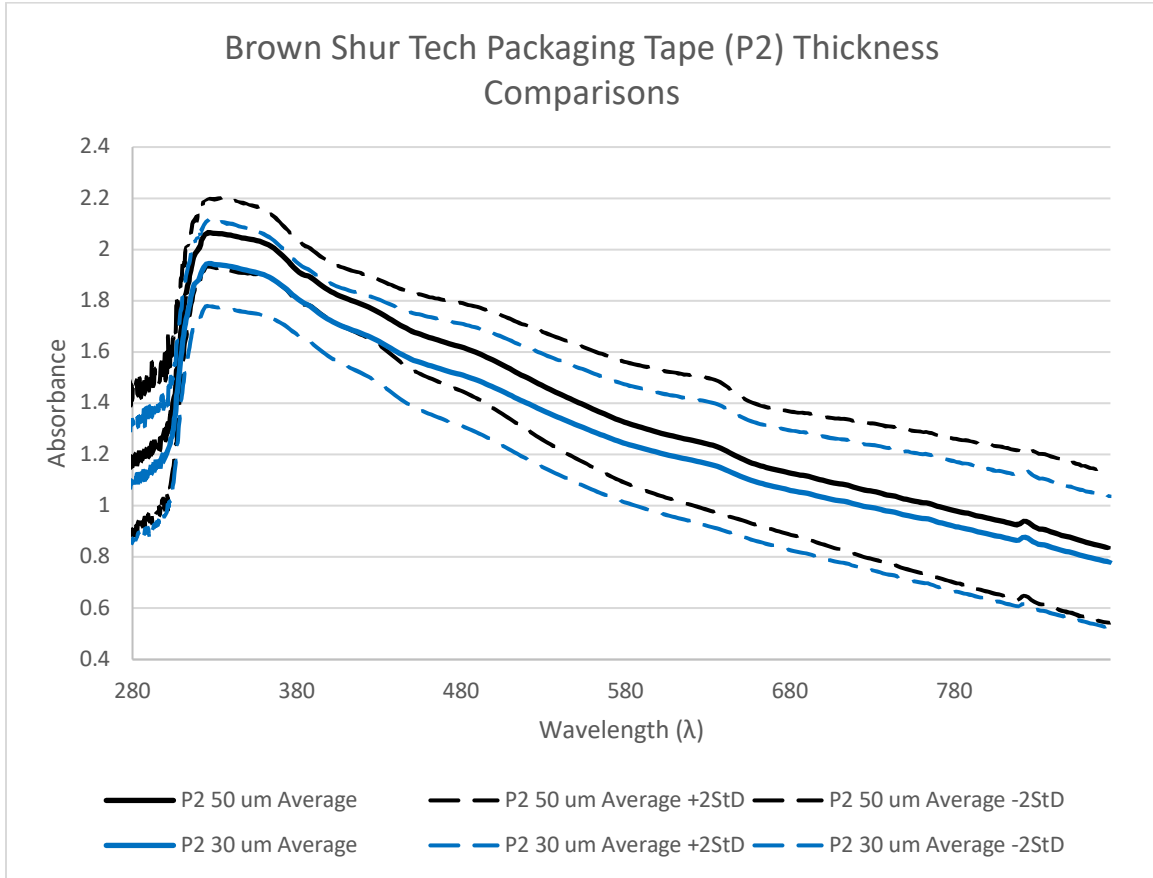
The above figure compares the average absorbance spectra through 50 µm microtomed cross sections, 30 µm microtomed cross sections, 15 µm microtomed cross sections, and 5 µm microtomed cross sections of green Scotch 3M electrical tape (E4) from 280 to 875 nm. A solid line indicates the average of fifty scans and a pair of dashed lines indicates plus or minus two standard deviations from the mean.

Figure 49.) Brown Scotch 3M Packaging Tape (P1) Thickness Comparisons



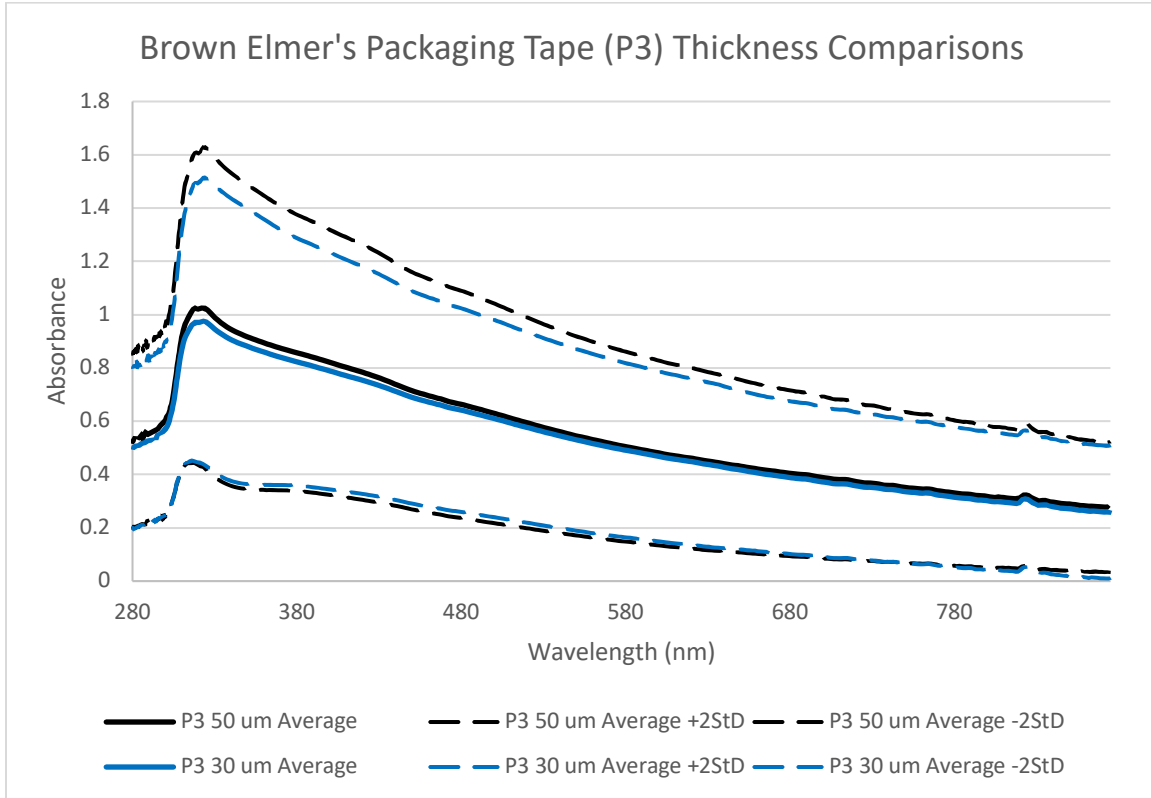
The above figure compares the average absorbance spectra through 50  $\mu\text{m}$  microtomed cross sections, 30  $\mu\text{m}$  microtomed cross sections, 15  $\mu\text{m}$  microtomed cross sections, and 5  $\mu\text{m}$  microtomed cross sections of brown Scotch 3M packaging tape (P1) from 280 to 875 nm. A solid line indicates the average of fifty scans and a pair of dashed lines indicates plus or minus two standard deviations from the mean.

Figure 50.) Brown Shur Tech Packaging Tape (P2) Thickness Comparisons



The above figure compares the average absorbance spectra through 50 μm microtomed cross sections and 30 μm microtomed cross sections of brown Shur Tech packaging tape (P2) from 280 to 875 nm. A solid line indicates the average of fifty scans and a pair of dashed lines indicates plus or minus two standard deviations from the mean.

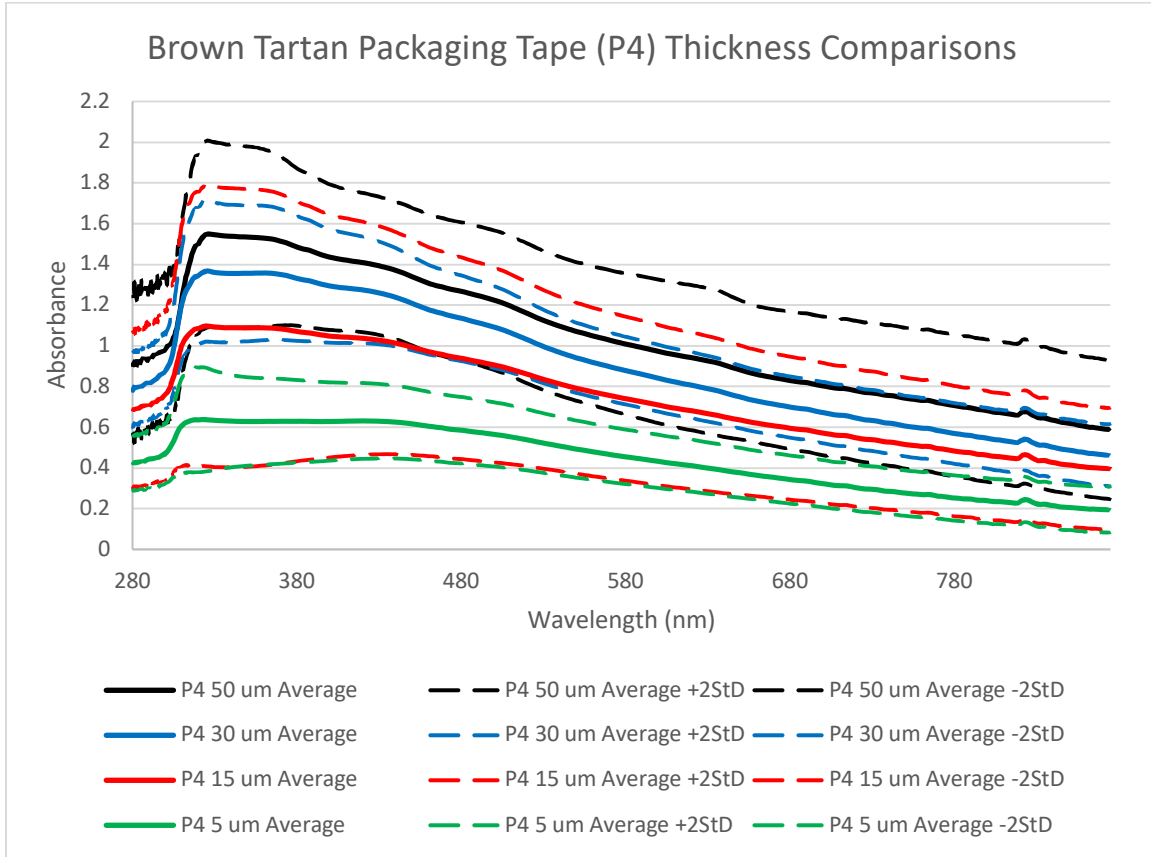
Figure 51.) Brown Elmer's Packaging Tape (P3) Thickness Comparisons



The above figure compares the average absorbance spectra through 50 µm microtomed cross sections and 30 µm microtomed cross sections of brown Elmer's packaging tape (P3) from 280 to 875 nm. A solid line indicates the average of fifty scans and a pair of dashed lines indicates plus or minus two standard deviations from the mean.

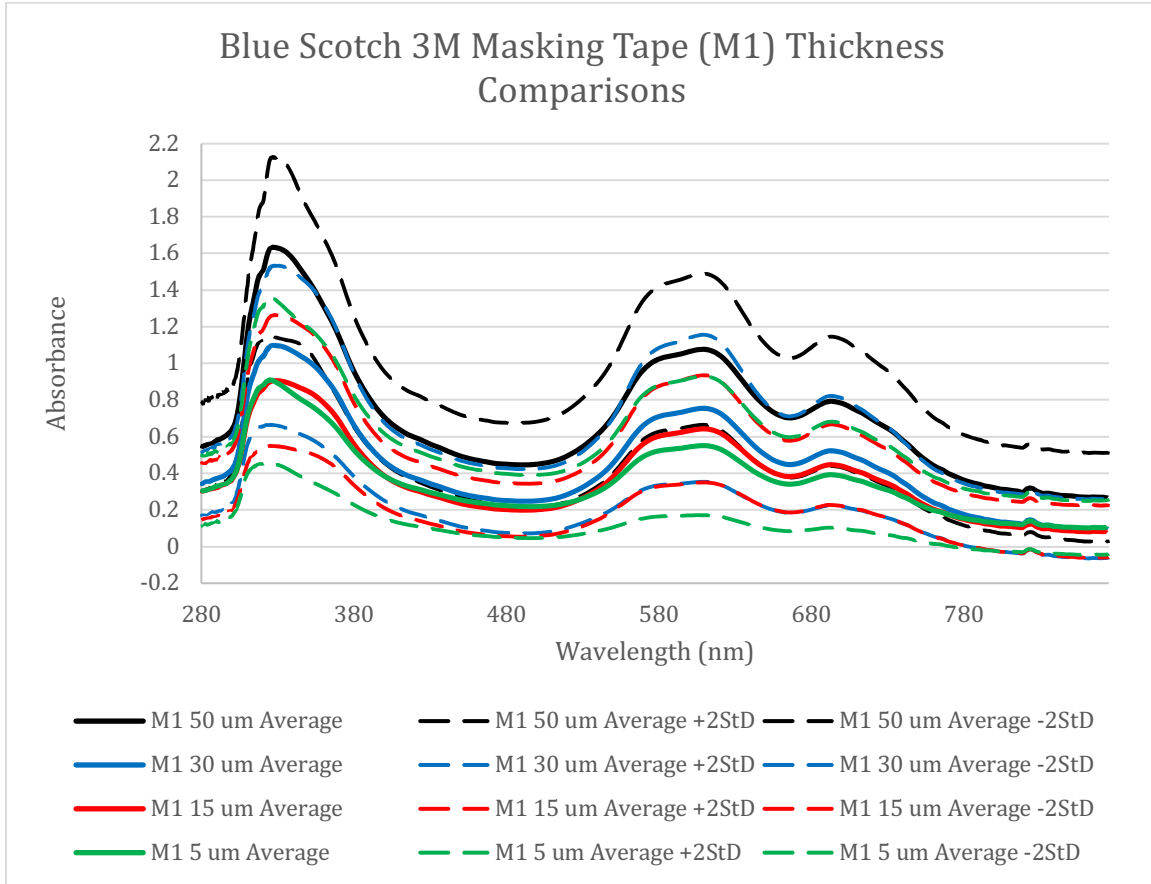


Figure 52.) Brown Tartan Packaging Tape (P4) Thickness Comparisons



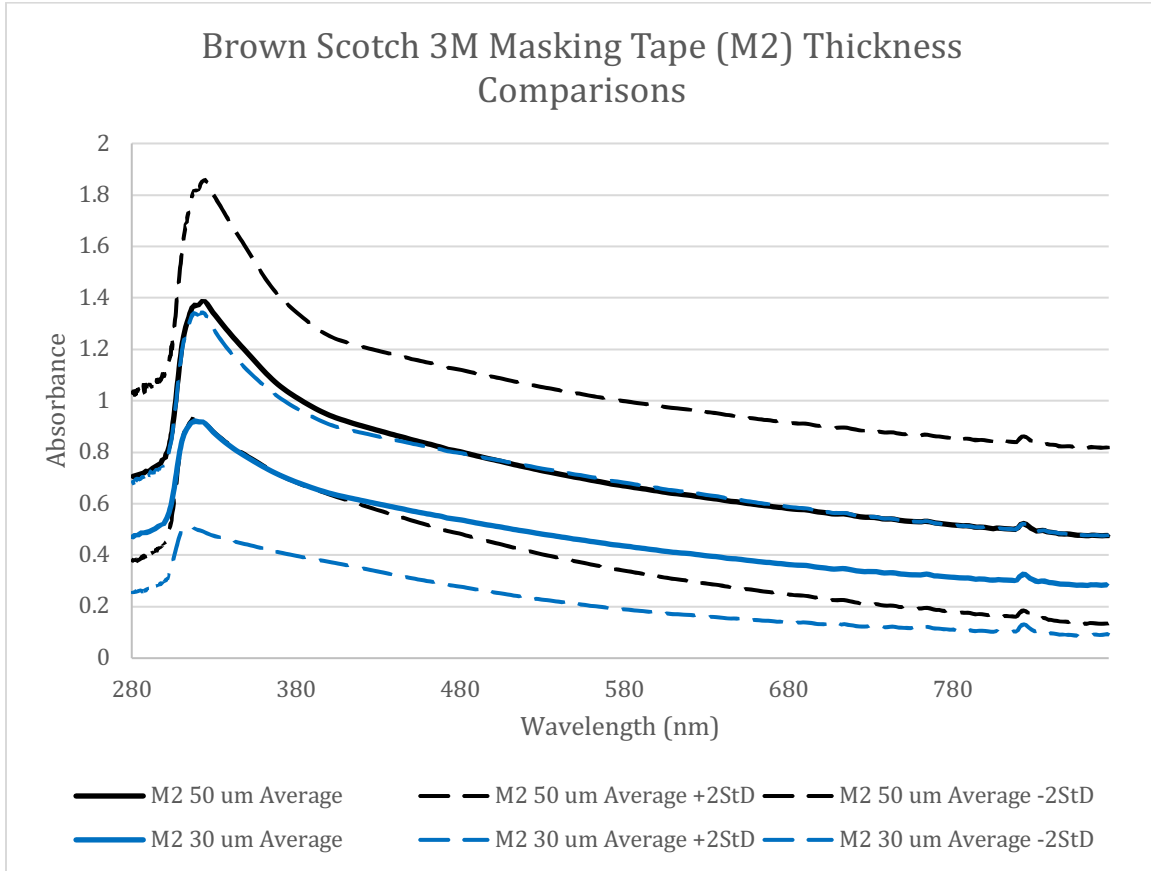
The above figure compares the average absorbance spectra through 50 μm microtomed cross sections, 30 μm microtomed cross sections, 15 μm microtomed cross sections, and 5 μm microtomed cross sections of brown Tartan packaging tape (P4) from 280 to 875 nm. A solid line indicates the average of fifty scans and a pair of dashed lines indicates plus or minus two standard deviations from the mean.

Figure 53.) Blue Scotch 3M Masking Tape (M1) Thickness Comparisons



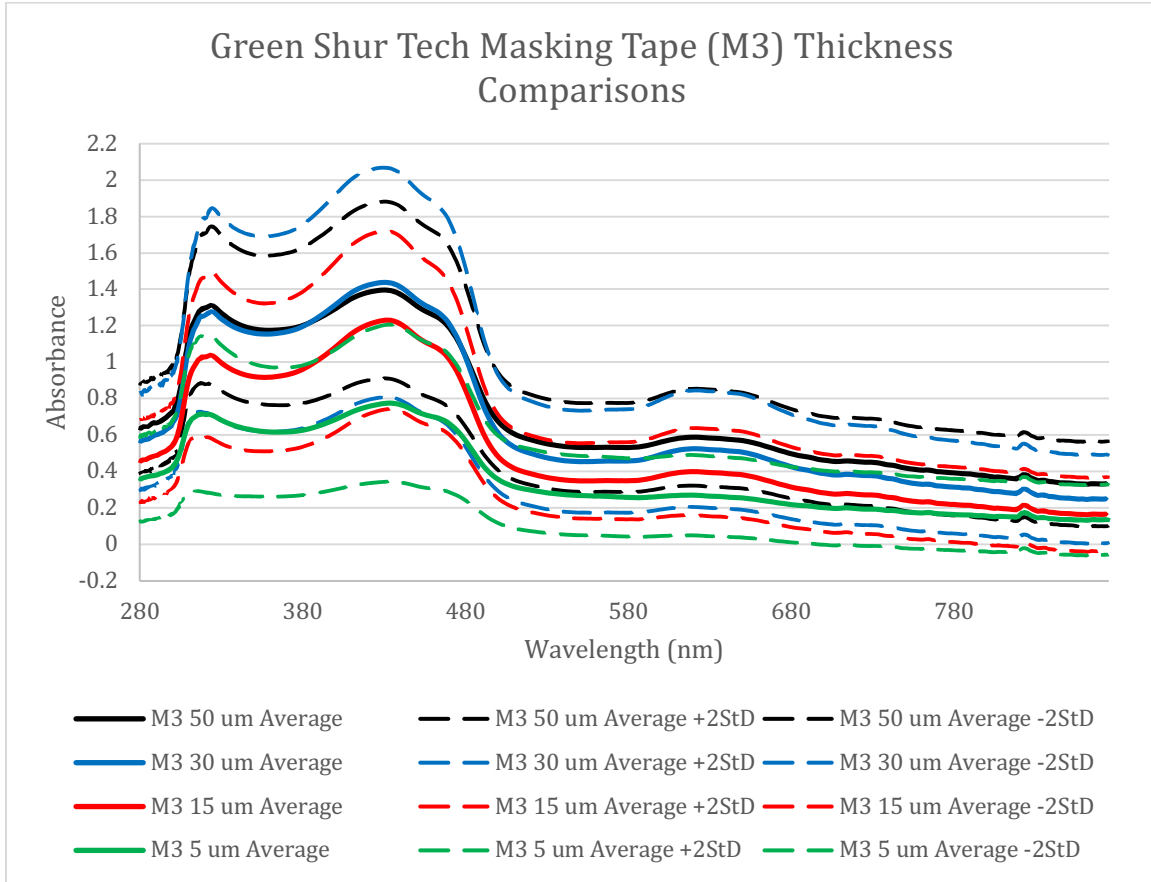
The above figure compares the average absorbance spectra through 50  $\mu\text{m}$  microtomed cross sections, 30  $\mu\text{m}$  microtomed cross sections, 15  $\mu\text{m}$  microtomed cross sections, and 5  $\mu\text{m}$  microtomed cross sections of blue Scotch 3M masking tape (M1) from 280 to 875 nm. A solid line indicates the average of fifty scans and a pair of dashed lines indicates plus or minus two standard deviations from the mean.

Figure 54.) Brown Scotch 3M Masking Tape (M2) Thickness Comparisons



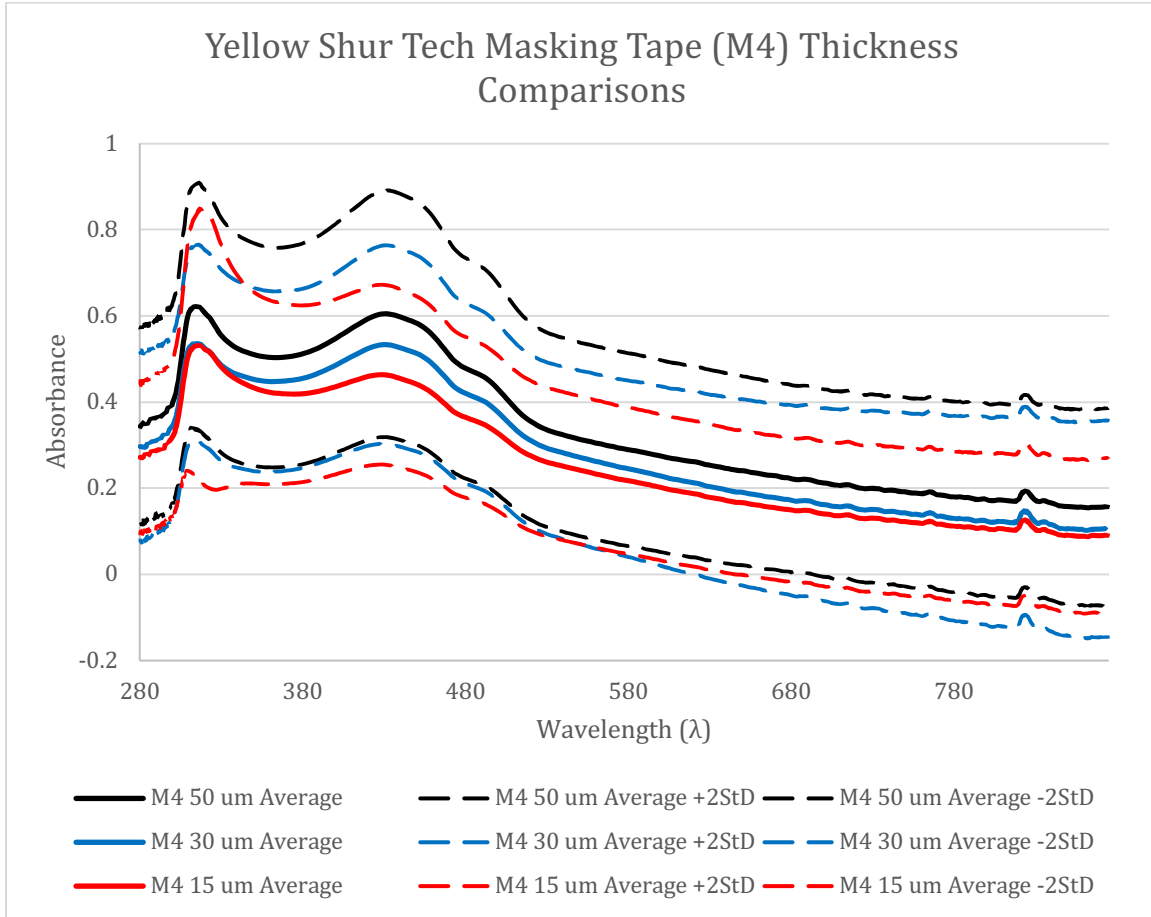
The above figure compares the average absorbance spectra through 50  $\mu\text{m}$  microtomed cross sections and 30  $\mu\text{m}$  microtomed cross sections of brown Scotch 3M masking tape (M2) from 280 to 875 nm. A solid line indicates the average of fifty scans and a pair of dashed lines indicates plus or minus two standard deviations from the mean.

Figure 55.) Green Shur Tech Masking Tape (M3) Thickness Comparisons



The above figure compares the average absorbance spectra through 50  $\mu\text{m}$  microtomed cross sections, 30  $\mu\text{m}$  microtomed cross sections, 15  $\mu\text{m}$  microtomed cross sections, and 5  $\mu\text{m}$  microtomed cross sections of green Shur Tech masking tape (M3) from 280 to 875 nm. A solid line indicates the average of fifty scans and a pair of dashed lines indicates plus or minus two standard deviations from the mean.

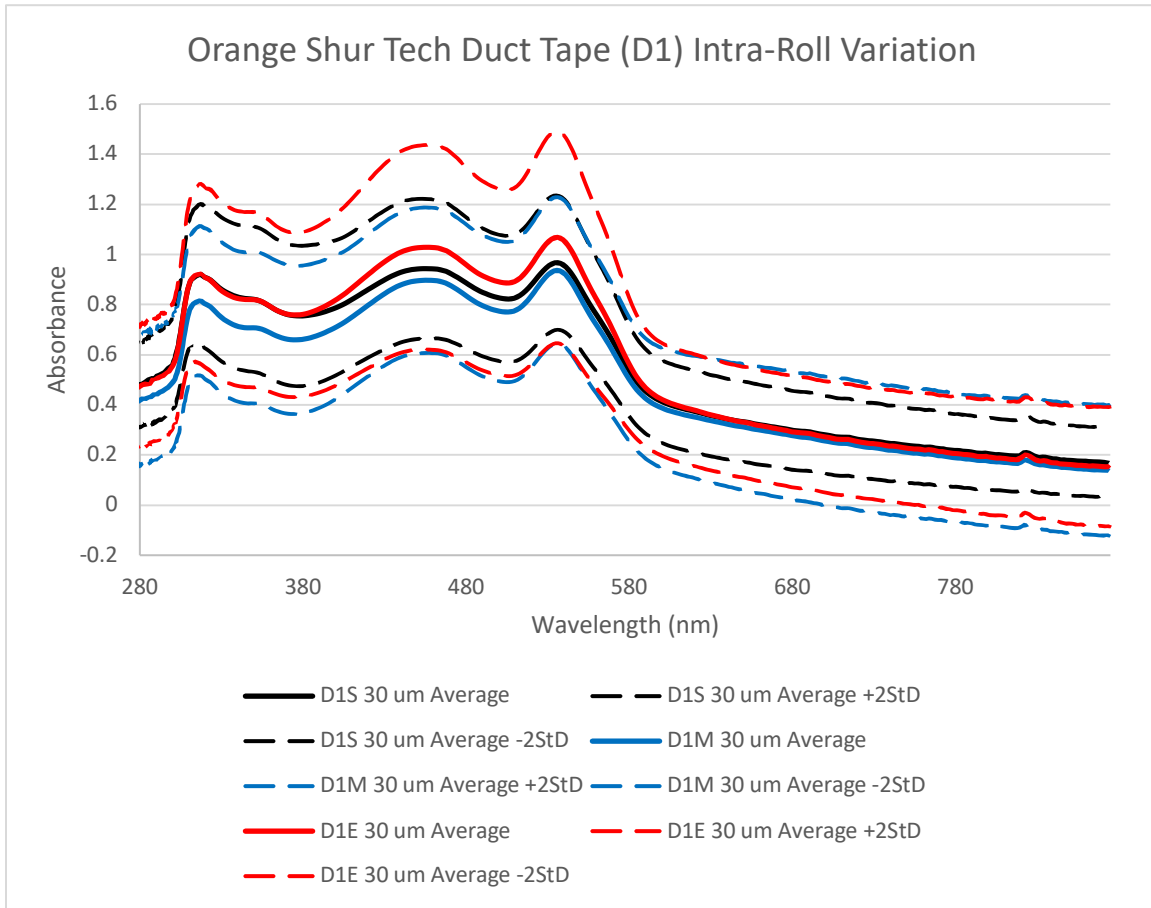
Figure 56.) Yellow Shur Tech Masking Tape (M4) Thickness Comparisons



The above figure compares the average absorbance spectra through 50 μm microtomed cross sections, 30 μm microtomed cross sections, and 15 μm microtomed cross sections of yellow Shur Tech masking tape (M4) from 280 to 875 nm. A solid line indicates the average of fifty scans and a pair of dashed lines indicates plus or minus two standard deviations from the mean.

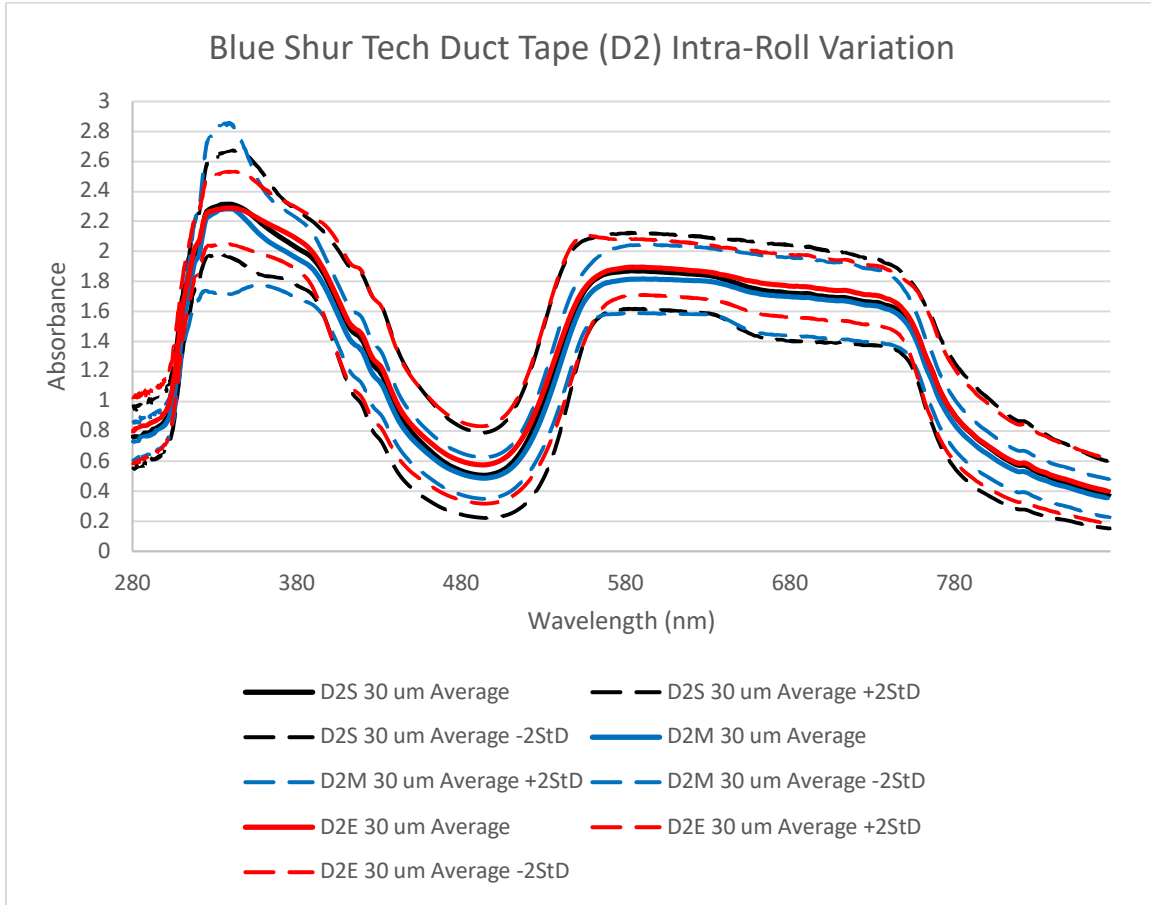
## Appendix C

Figure 57.) Orange Shur Tech Duct Tape (D1) Intra-Roll Variation



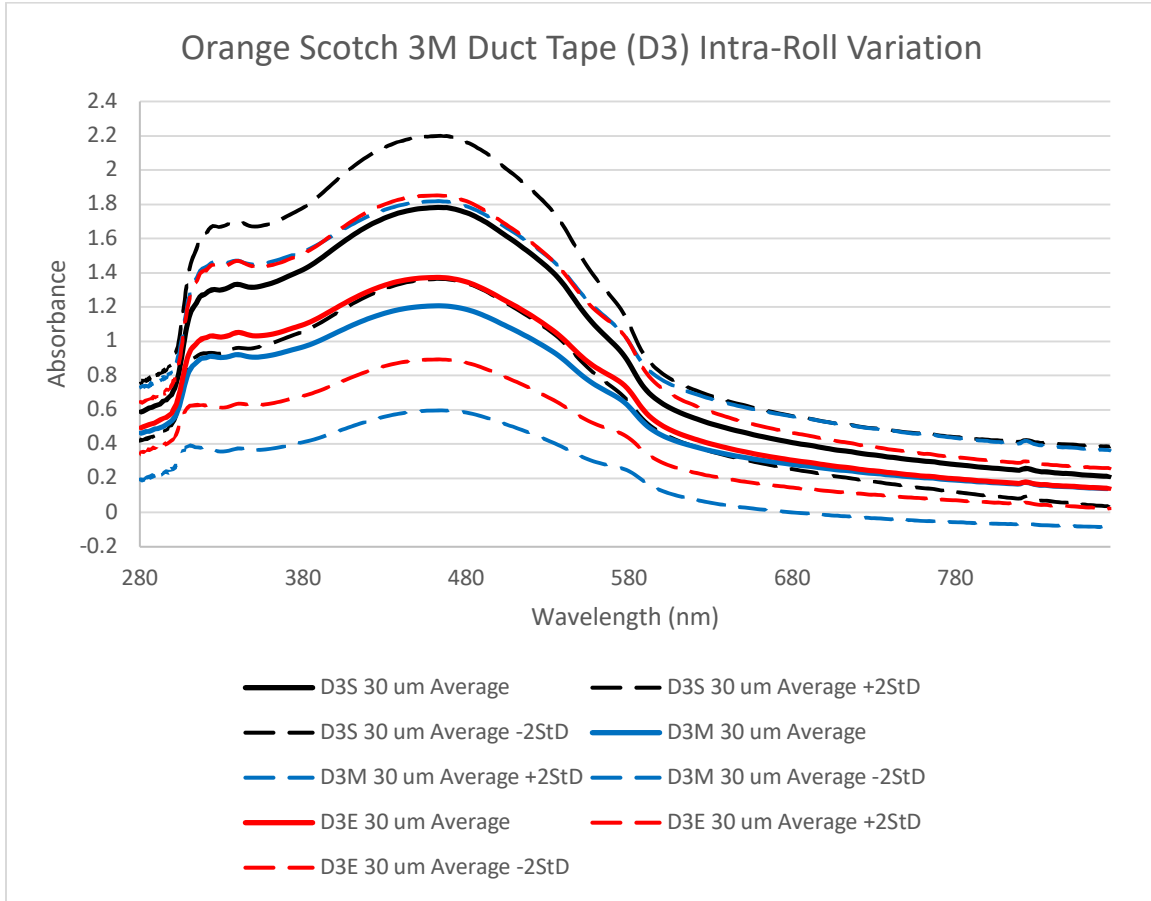
The above figure compares the average absorbance spectra through 30  $\mu\text{m}$  microtomed cross sections at the approximate beginning (S), middle (M), and end (E) of the roll of orange Shur Tech duct tape (D1) from 280 to 875 nm. A solid line indicates the average of fifty scans and a pair of dashed lines indicates plus or minus two standard deviations from the mean.

Figure 58.) Blue Shur Tech Duct Tape (D2) Intra-Roll Variation



The above figure compares the average absorbance spectra through 30  $\mu\text{m}$  microtomed cross sections at the approximate beginning (S), middle (M), and end (E) of the roll of blue Shur Tech duct tape (D2) from 280 to 875 nm. A solid line indicates the average of fifty scans and a pair of dashed lines indicates plus or minus two standard deviations from the mean.

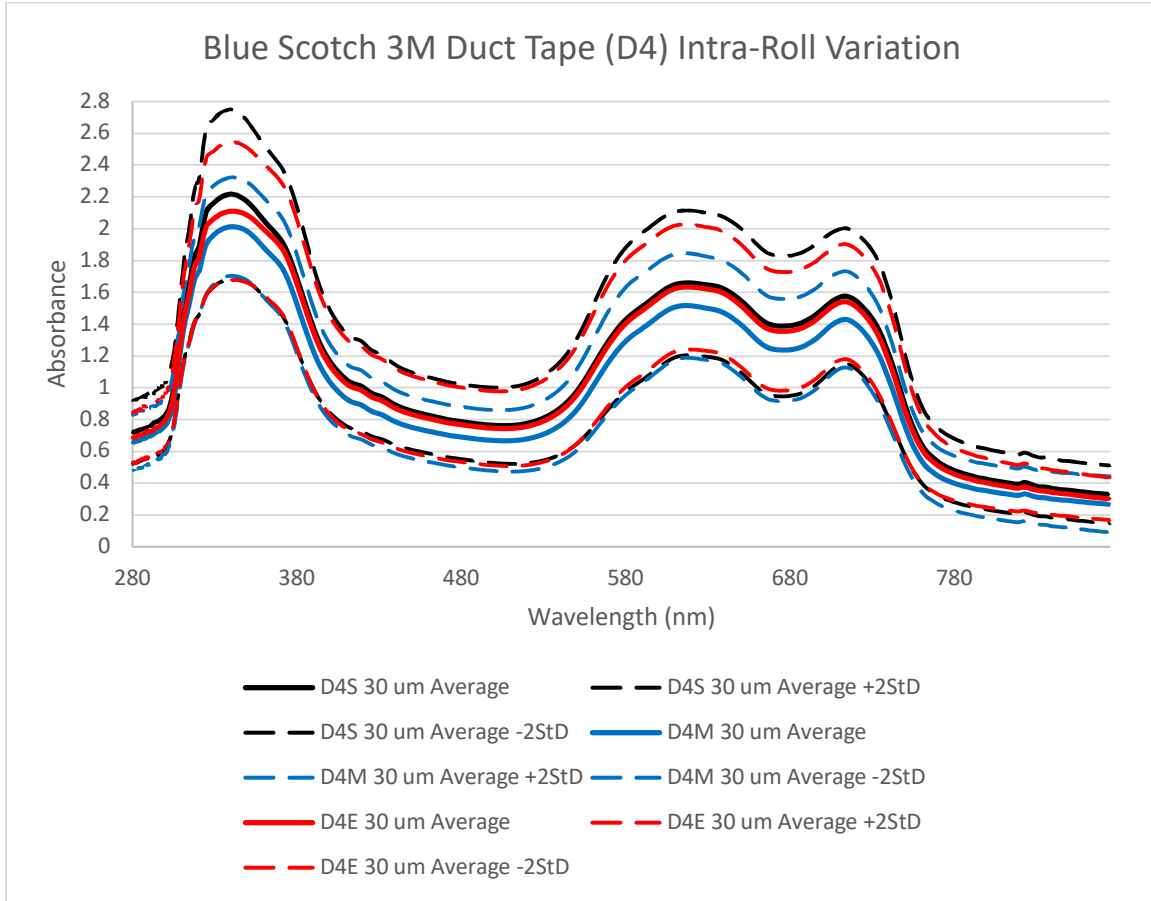
Figure 59.) Orange Scotch 3M Duct Tape (D3) Intra-Roll Variation



The above figure compares the average absorbance spectra through 30 μm microtomed cross sections at the approximate beginning (S), middle (M), and end (E) of the roll of orange Scotch 3M duct tape (D3) from 280 to 875 nm. A solid line indicates the average of fifty scans and a pair of dashed lines indicates plus or minus two standard deviations from the mean.

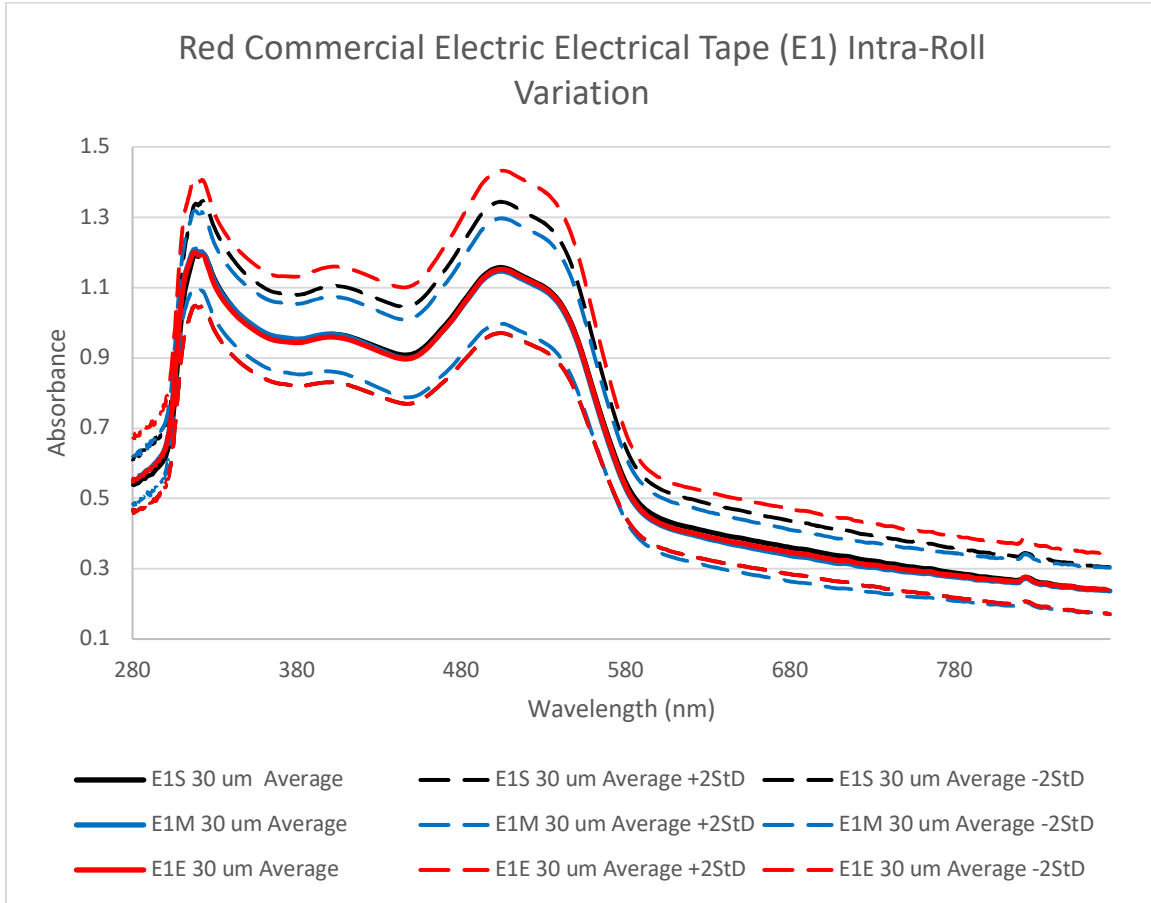


Figure 60.) Blue Scotch 3M Duct Tape (D4) Intra-Roll Variation



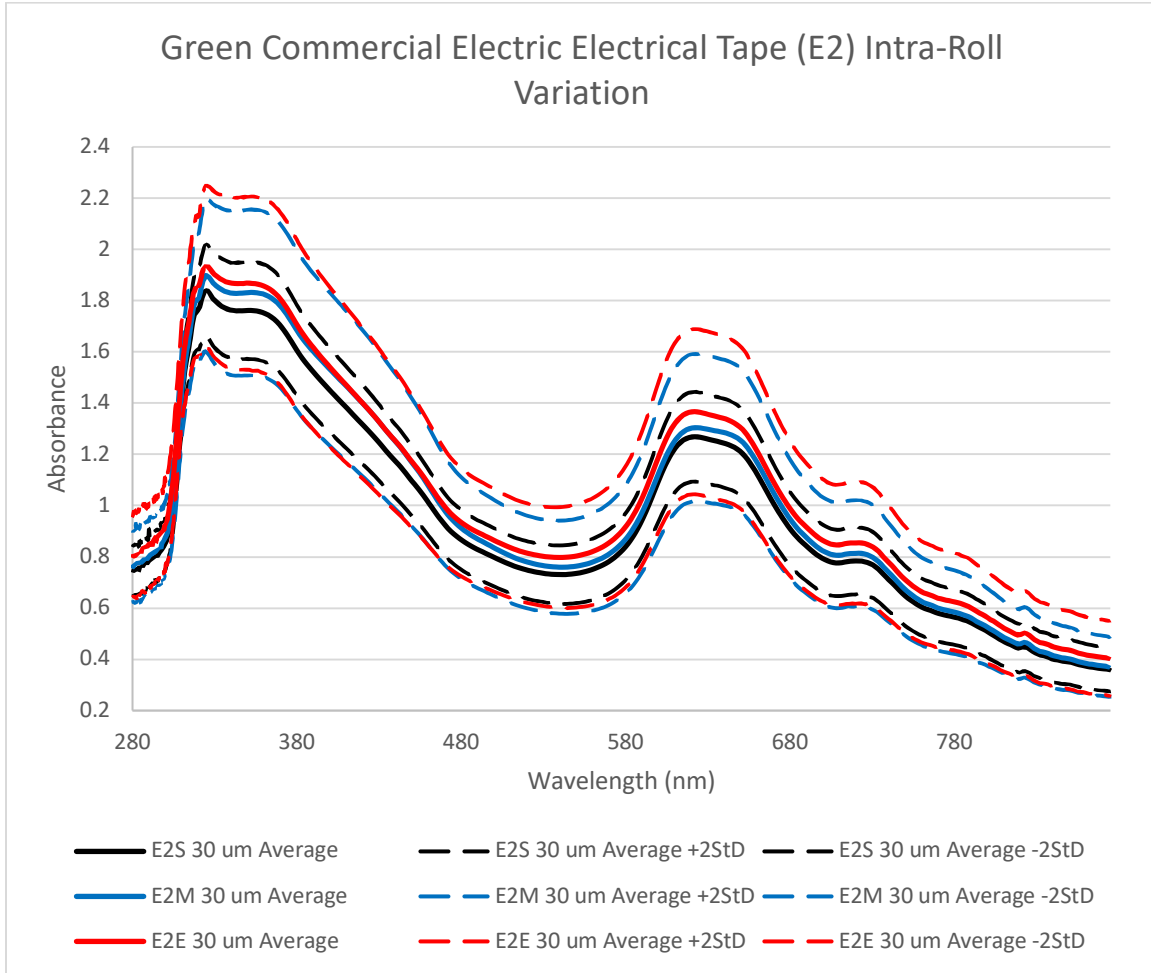
The above figure compares the average absorbance spectra through 30  $\mu\text{m}$  microtomed cross sections at the approximate beginning (S), middle (M), and end (E) of the roll of blue Scotch 3M duct tape (D4) from 280 to 875 nm. A solid line indicates the average of fifty scans and a pair of dashed lines indicates plus or minus two standard deviations from the mean.

Figure 61.) Red Commercial Electric Electrical Tape (E1) Intra-Roll Variation



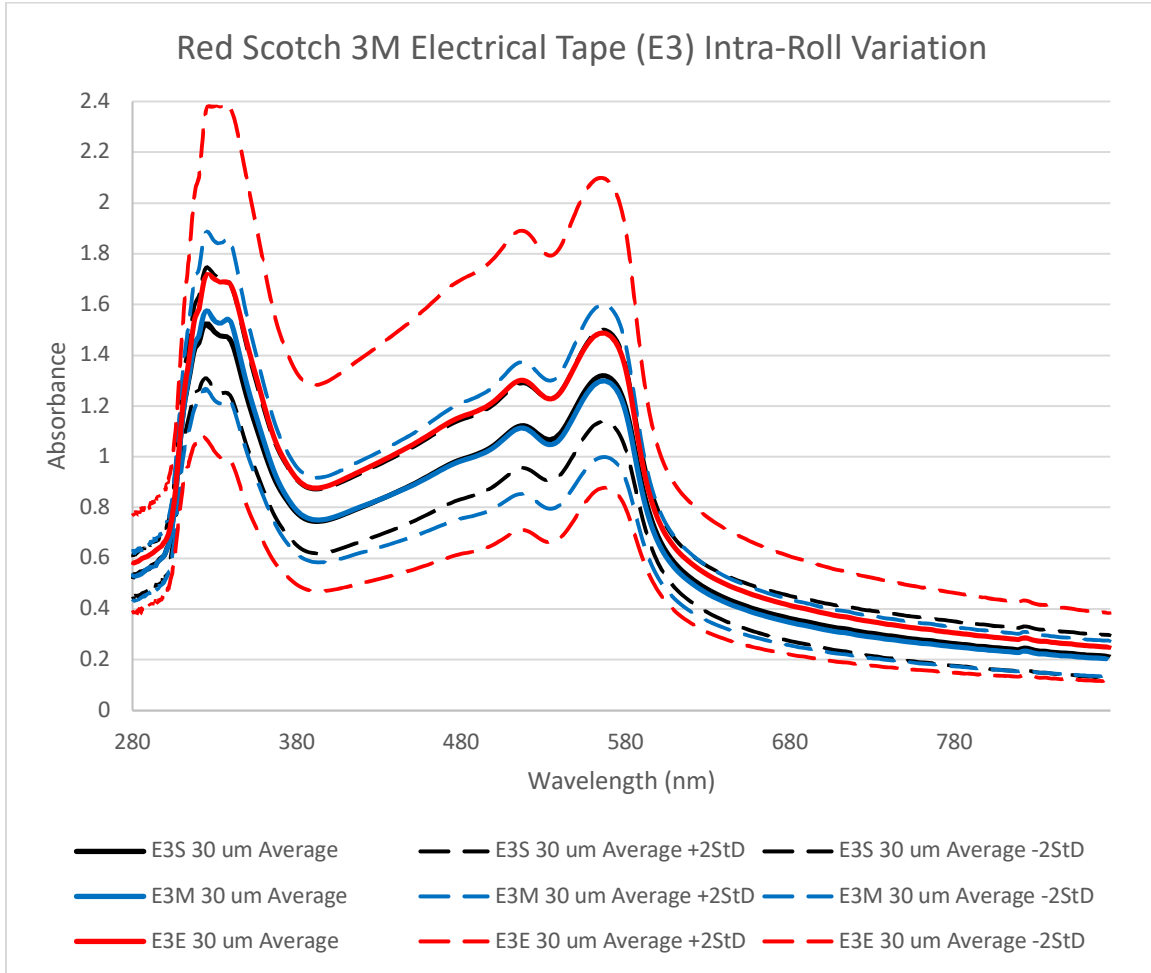
The above figure compares the average absorbance spectra through 30  $\mu\text{m}$  microtomed cross sections at the approximate beginning (S), middle (M), and end (E) of the roll of red Commercial Electric electrical tape (E1) from 280 to 875 nm. A solid line indicates the average of fifty scans and a pair of dashed lines indicates plus or minus two standard deviations from the mean.

Figure 62.) Green Commercial Electric Electrical Tape (E2) Intra-Roll Variation



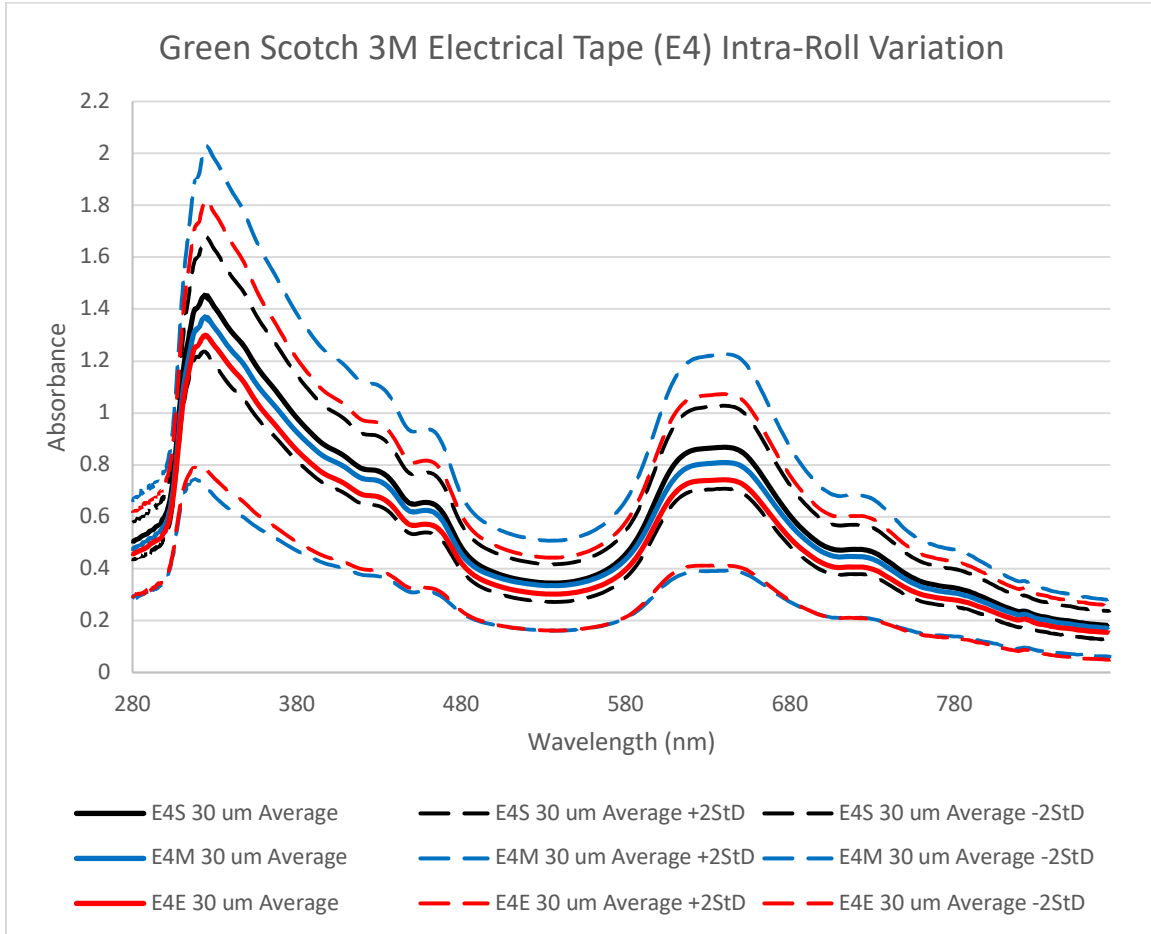
The above figure compares the average absorbance spectra through 30  $\mu\text{m}$  microtomed cross sections at the approximate beginning (S), middle (M), and end (E) of the roll of green Commercial Electric electrical tape (E2) from 280 to 875 nm. A solid line indicates the average of fifty scans and a pair of dashed lines indicates plus or minus two standard deviations from the mean.

Figure 63.) Red Scotch 3M Electrical Tape (E3) Intra-Roll Variation



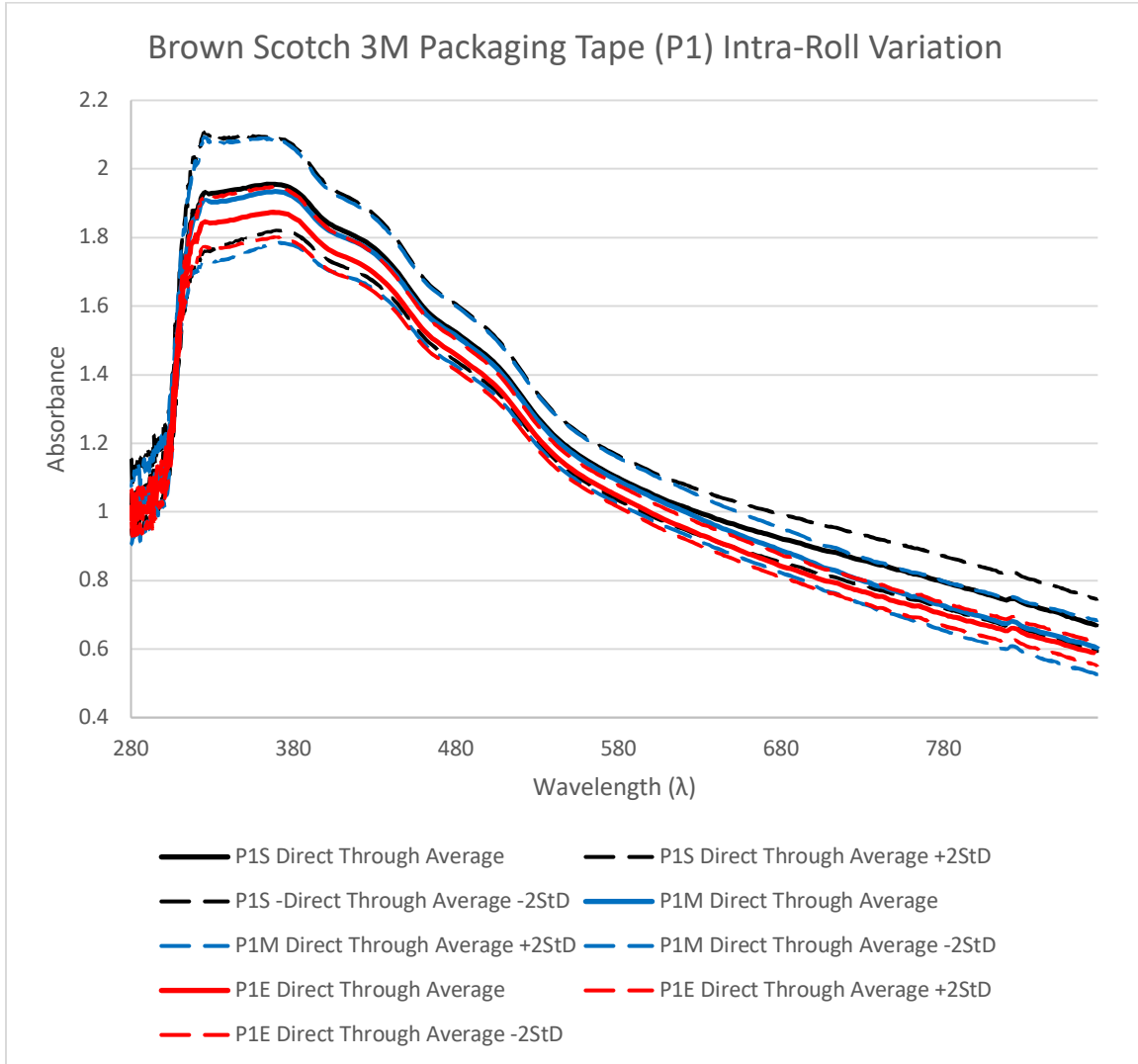
The above figure compares the average absorbance spectra through 30  $\mu\text{m}$  microtomed cross sections at the approximate beginning (S), middle (M), and end (E) of the roll of red Scotch 3M electrical tape (E3) from 280 to 875 nm. A solid line indicates the average of fifty scans and a pair of dashed lines indicates plus or minus two standard deviations from the mean.

Figure 64.) Green Scotch 3M Electrical Tape (E4) Intra-Roll Variation



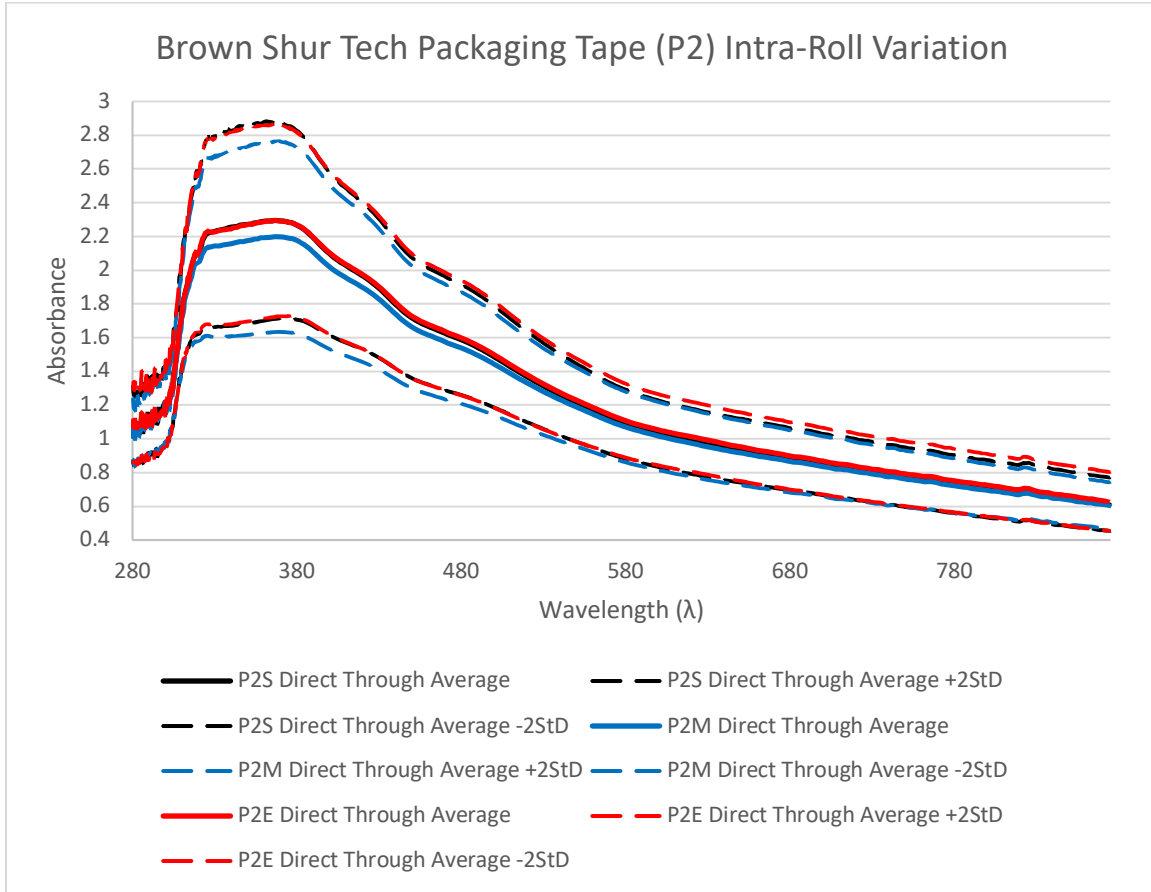
The above figure compares the average absorbance spectra through 30  $\mu\text{m}$  microtomed cross sections at the approximate beginning (S), middle (M), and end (E) of the roll of green Scotch 3M electrical tape (E4) from 280 to 875 nm. A solid line indicates the average of fifty scans and a pair of dashed lines indicates plus or minus two standard deviations from the mean.

Figure 65.) Brown Scotch 3M Packaging Tape (P1) Intra-Roll Variation



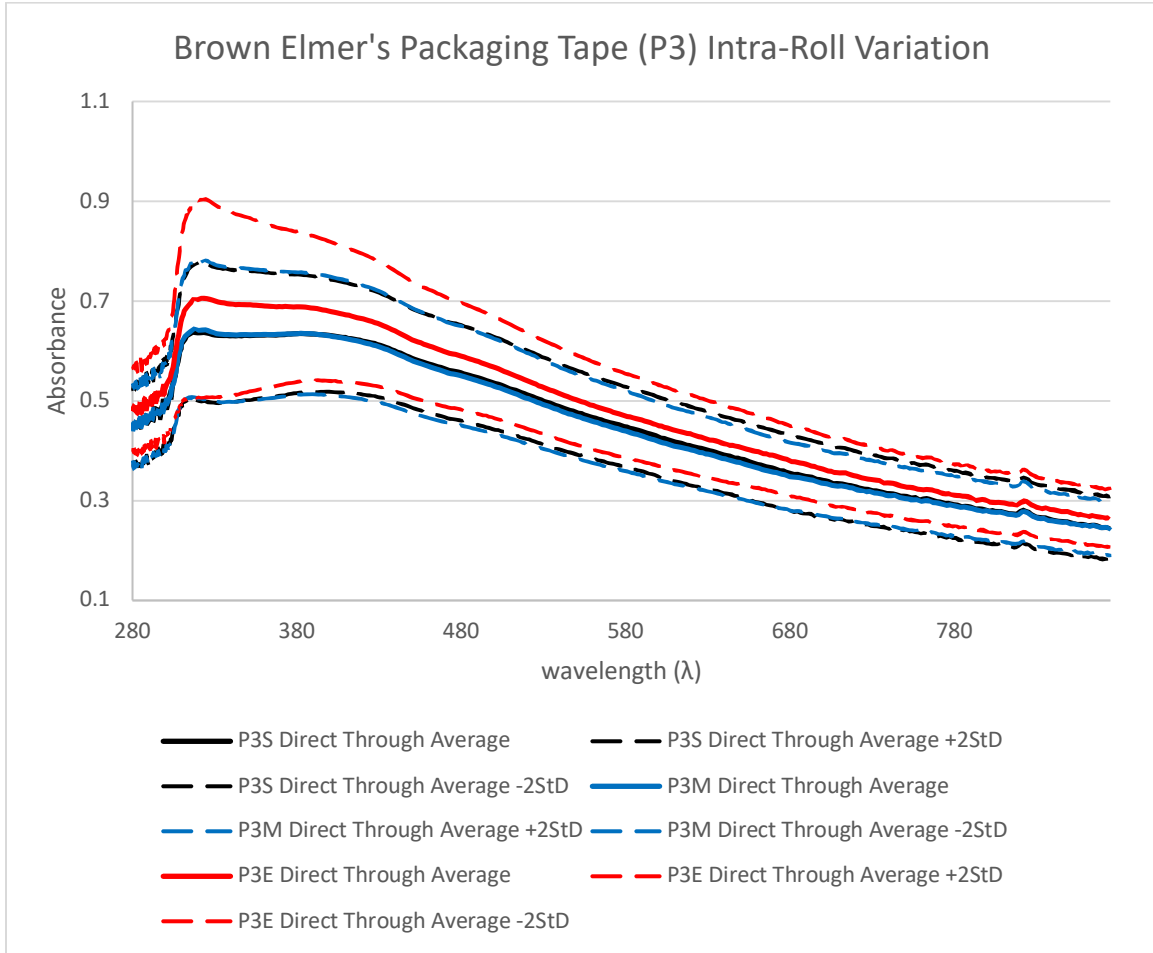
The above figure compares the average absorbance spectra directly through the approximate beginning (S), middle (M), and end (E) of the roll of brown Scotch 3M packaging tape (P1) from 280 to 875 nm. A solid line indicates the average of fifty scans and a pair of dashed lines indicates plus or minus two standard deviations from the mean.

Figure 66.) Brown Shur Tech Packaging Tape (P2) Intra-Roll Variation



The above figure compares the average absorbance spectra directly through the approximate beginning (S), middle (M), and end (E) of the roll of brown Shur Tech packaging tape (P2) from 280 to 875 nm. A solid line indicates the average of fifty scans and a pair of dashed lines indicates plus or minus two standard deviations from the mean.

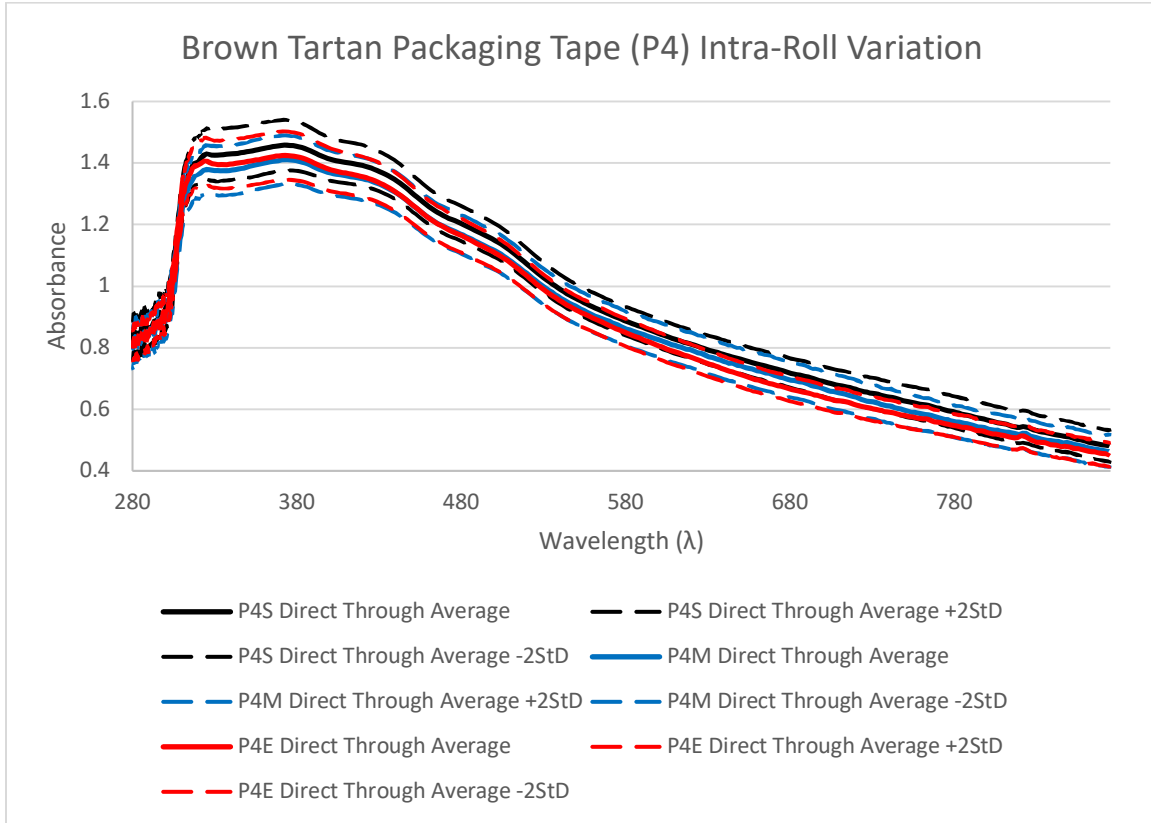
Figure 67.) Brown Elmer's Packaging Tape (P3) Intra-Roll Variation



The above figure compares the average absorbance spectra directly through the approximate beginning (S), middle (M), and end (E) of the roll of brown Elmer's packaging tape (P3) from 280 to 875 nm. A solid line indicates the average of fifty scans and a pair of dashed lines indicates plus or minus two standard deviations from the mean.

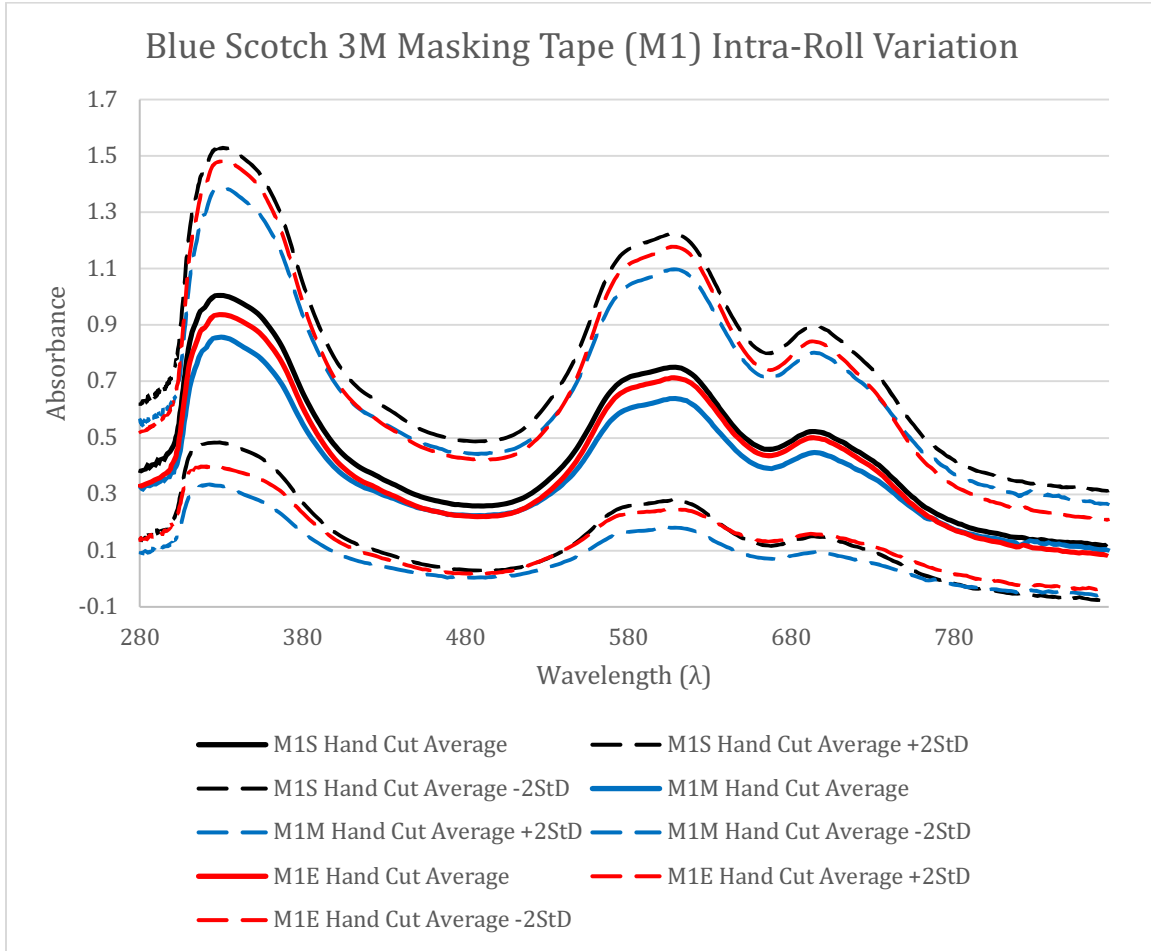


Figure 68.) Brown Tartan Packaging Tape (P4) Intra-Roll Variation



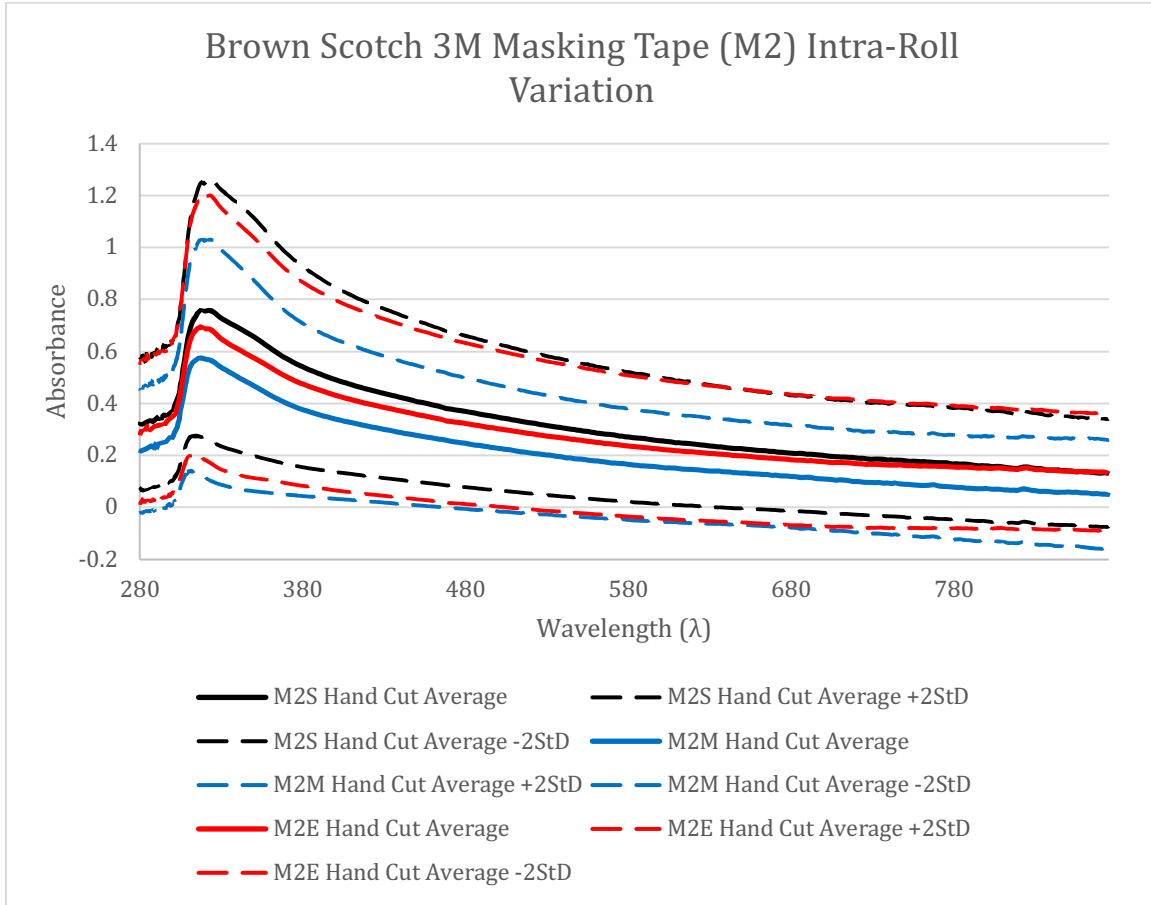
The above figure compares the average absorbance spectra directly through the approximate beginning (S), middle (M), and end (E) of the roll of brown Tartan packaging tape (P4) from 280 to 875 nm. A solid line indicates the average of fifty scans and a pair of dashed lines indicates plus or minus two standard deviations from the mean.

Figure 69.) Blue Scotch 3M Masking Tape (M1) Intra-Roll Variation



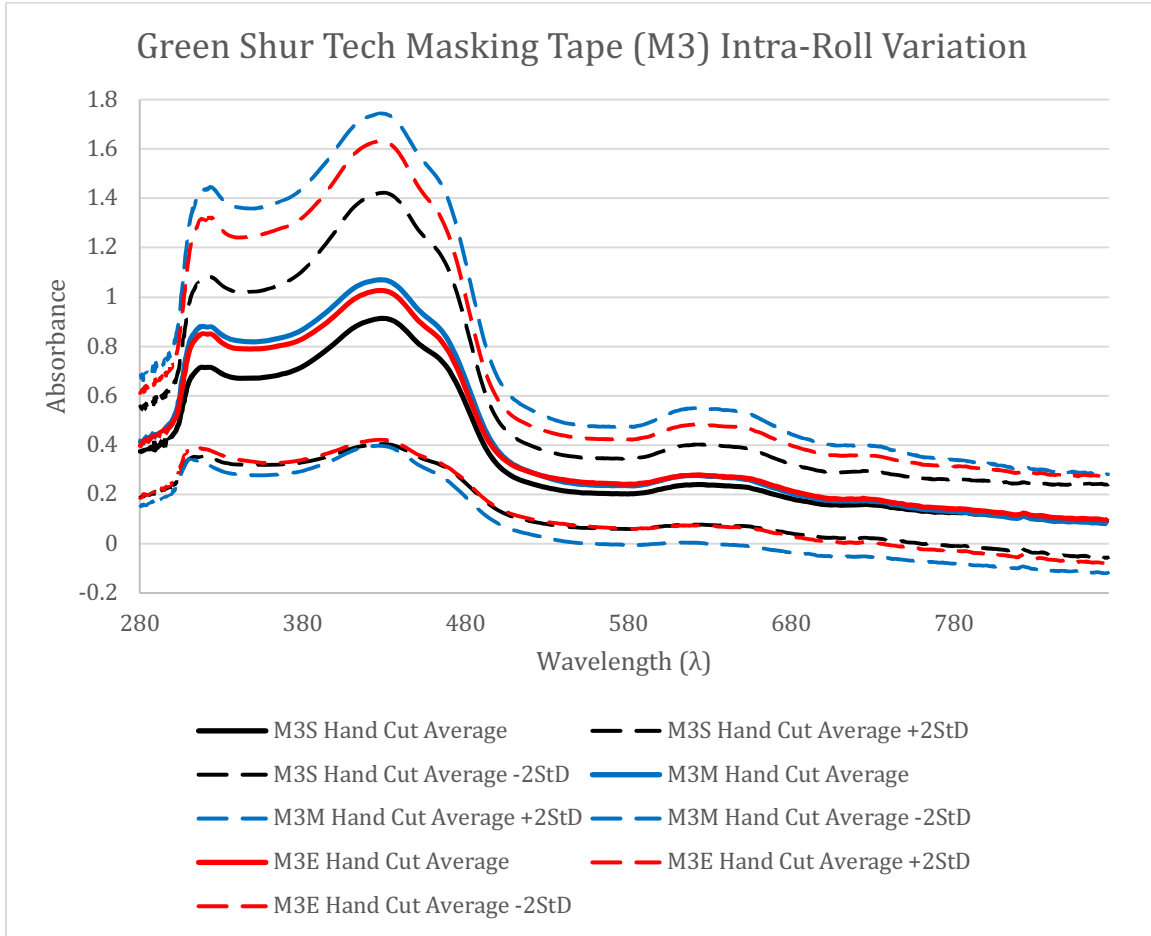
The above figure compares the average absorbance spectra through hand cut cross sections of the approximate beginning (S), middle (M), and end (E) of the roll of blue Scotch 3M masking tape (M1) from 280 to 875 nm. A solid line indicates the average of fifty scans and a pair of dashed lines indicates plus or minus two standard deviations from the mean.

Figure 70.) Brown Scotch 3M Masking Tape (M2) Intra-Roll Variation



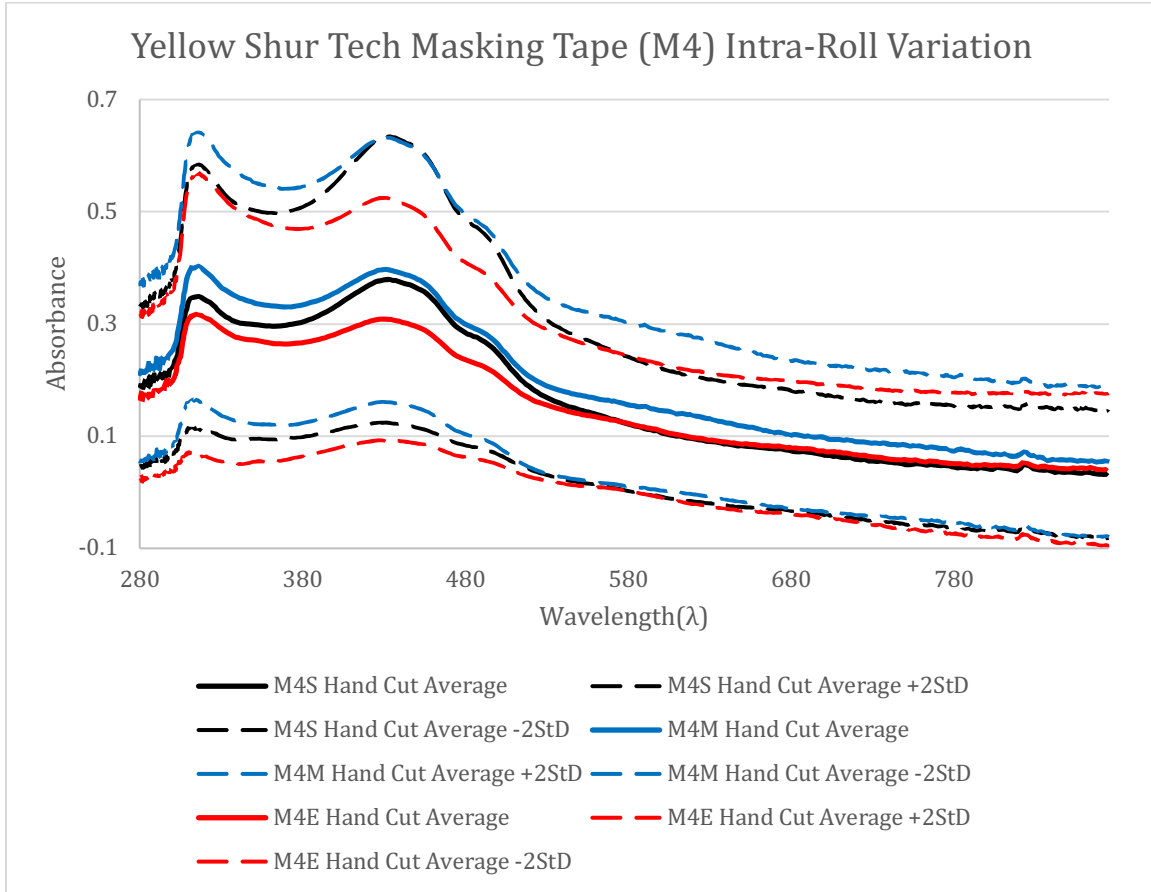
The above figure compares the average absorbance spectra through hand cut cross sections of the approximate beginning (S), middle (M), and end (E) of the roll of brown Scotch 3M masking tape (M2) from 280 to 875 nm. A solid line indicates the average of fifty scans and a pair of dashed lines indicates plus or minus two standard deviations from the mean.

Figure 71.) Green Shur Tech Masking Tape (M3) Intra-Roll Variation



The above figure compares the average absorbance spectra through hand cut cross sections of the approximate beginning (S), middle (M), and end (E) of the roll of green Shur Tech masking tape (M3) from 280 to 875 nm. A solid line indicates the average of fifty scans and a pair of dashed lines indicates plus or minus two standard deviations from the mean.

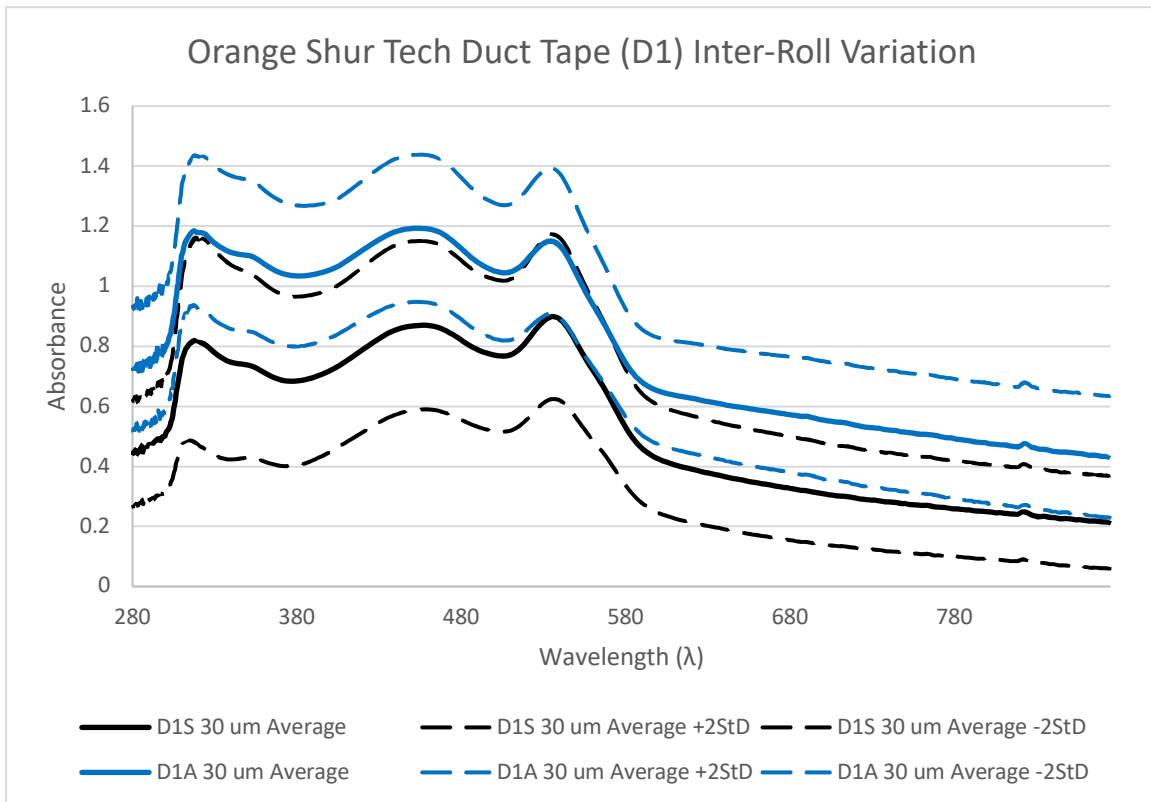
Figure 72.) Yellow Shur Tech Masking Tape (M4) Intra-Roll Variation



The above figure compares the average absorbance spectra through hand cut cross sections of the approximate beginning (S), middle (M), and end (E) of the roll of yellow Shur Tech masking tape (M4) from 280 to 875 nm. A solid line indicates the average of fifty scans and a pair of dashed lines indicates plus or minus two standard deviations from the mean.

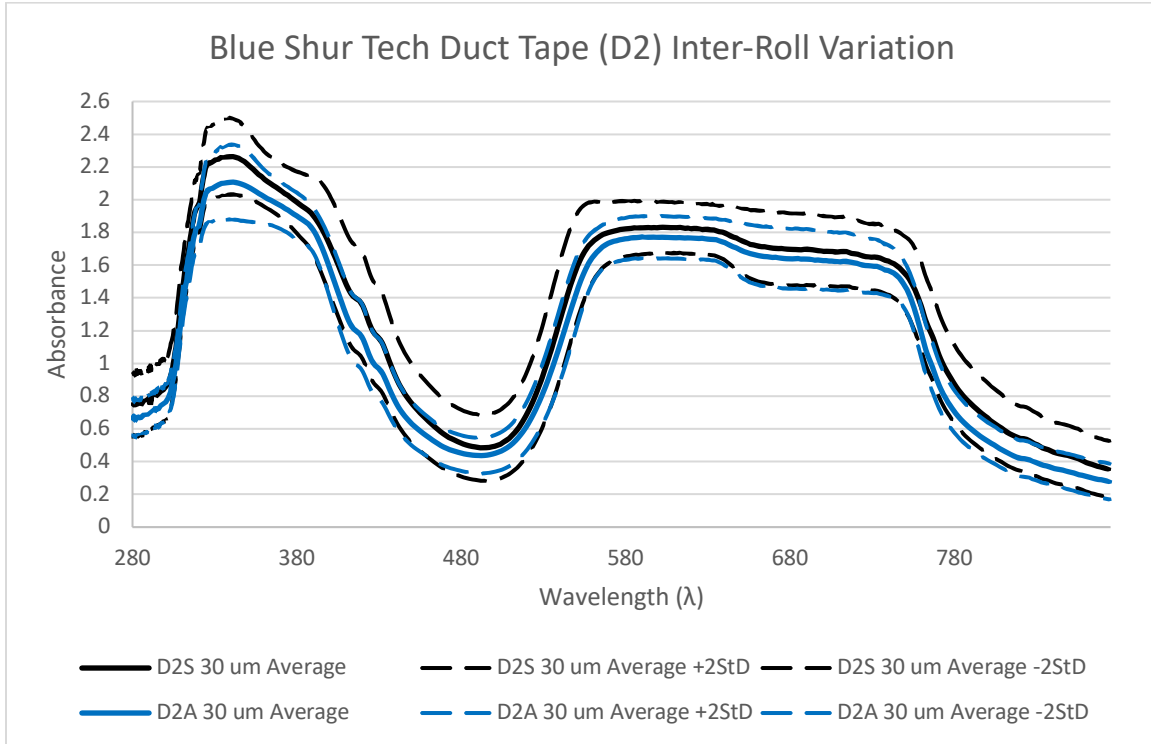
## Appendix D

Figure 73.) Orange Shur Tech Duct Tape (D1) Inter-Roll Variation



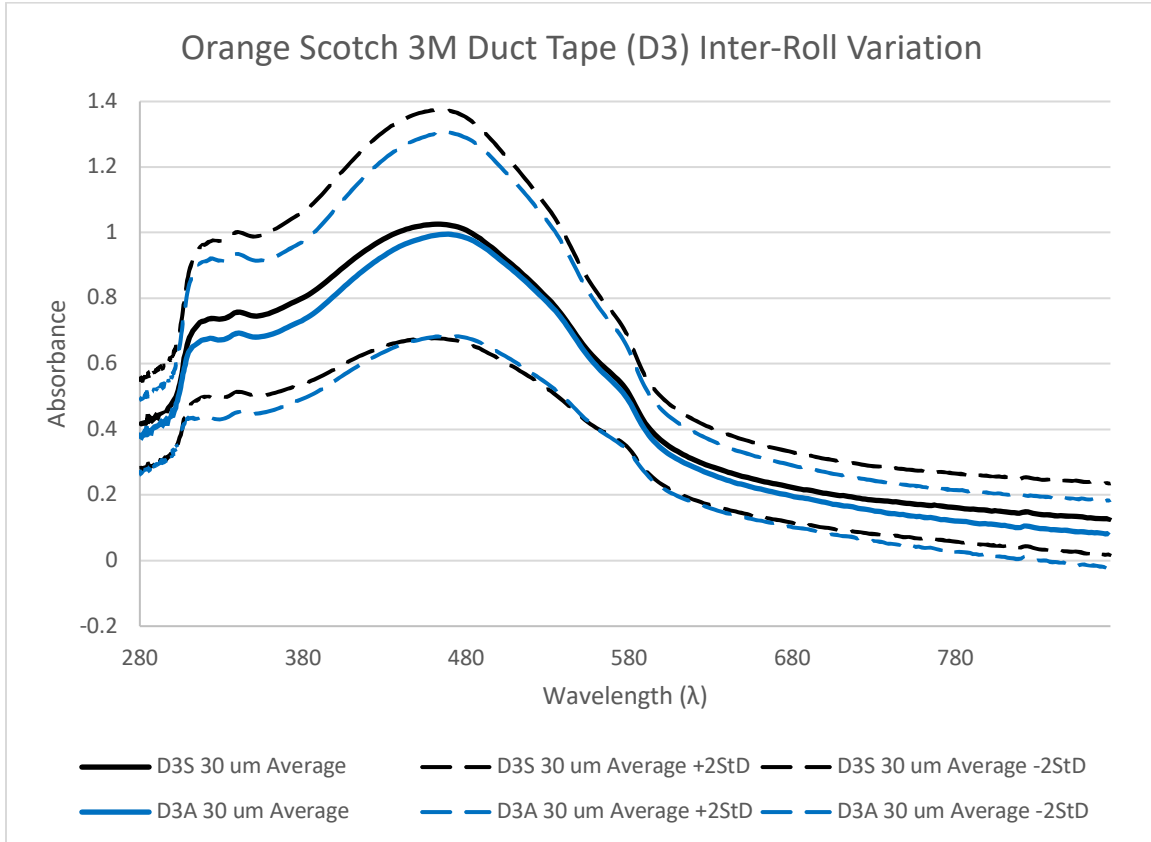
The above figure compares the average absorbance spectra through 30  $\mu\text{m}$  microtomed cross sections at the approximate beginning (S) of two different rolls of orange Shur Tech duct tape (D1 and D1A) from 280 to 875 nm. A solid line indicates the average of fifty scans and a pair of dashed lines indicates plus or minus two standard deviations from the mean.

Figure 74.) Blue Shur Tech Duct Tape (D2) Inter-Roll Variation



The above figure compares the average absorbance spectra through 30  $\mu\text{m}$  microtomed cross sections at the approximate beginning (S) of two different rolls of blue Shur Tech duct tape (D2 and D2A) from 280 to 875 nm. A solid line indicates the average of fifty scans and a pair of dashed lines indicates plus or minus two standard deviations from the mean.

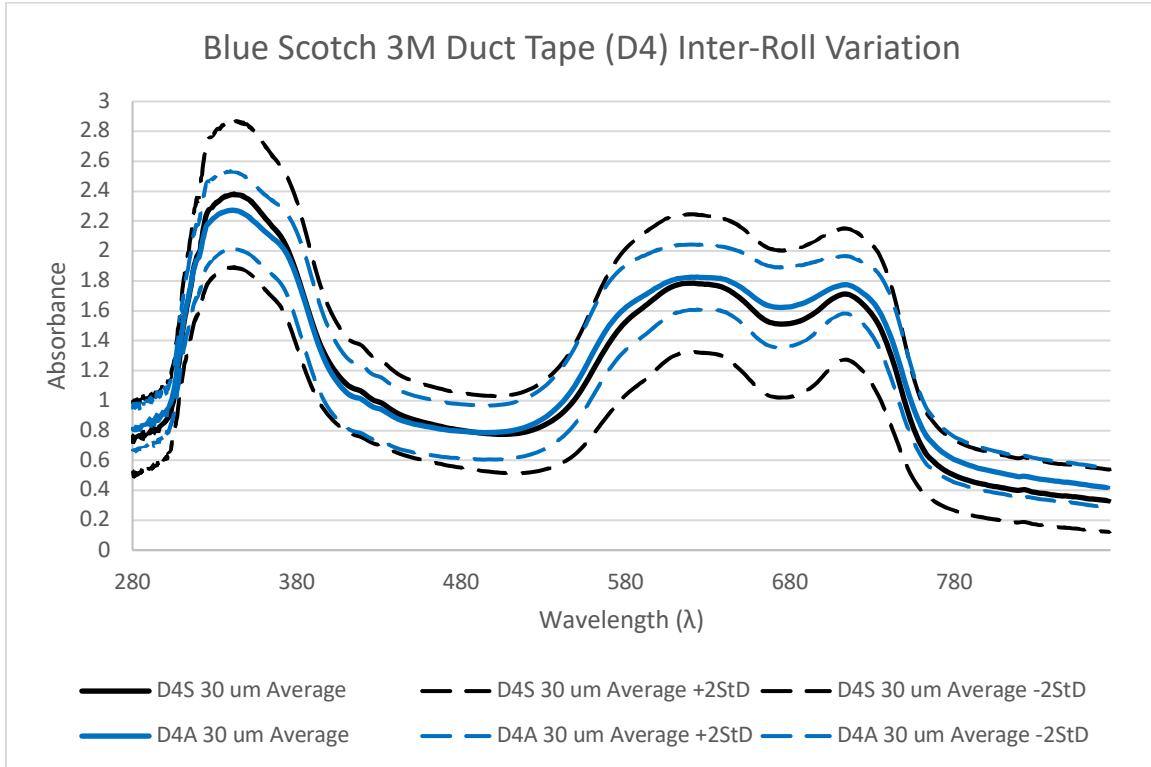
Figure 75.) Orange Scotch 3M Duct Tape (D3) Inter-Roll Variation



The above figure compares the average absorbance spectra through 30 μm microtomed cross sections at the approximate beginning (S) of two different rolls of orange Scotch 3M duct tape (D3 and D3A) from 280 to 875 nm. A solid line indicates the average of fifty scans and a pair of dashed lines indicates plus or minus two standard deviations from the mean.

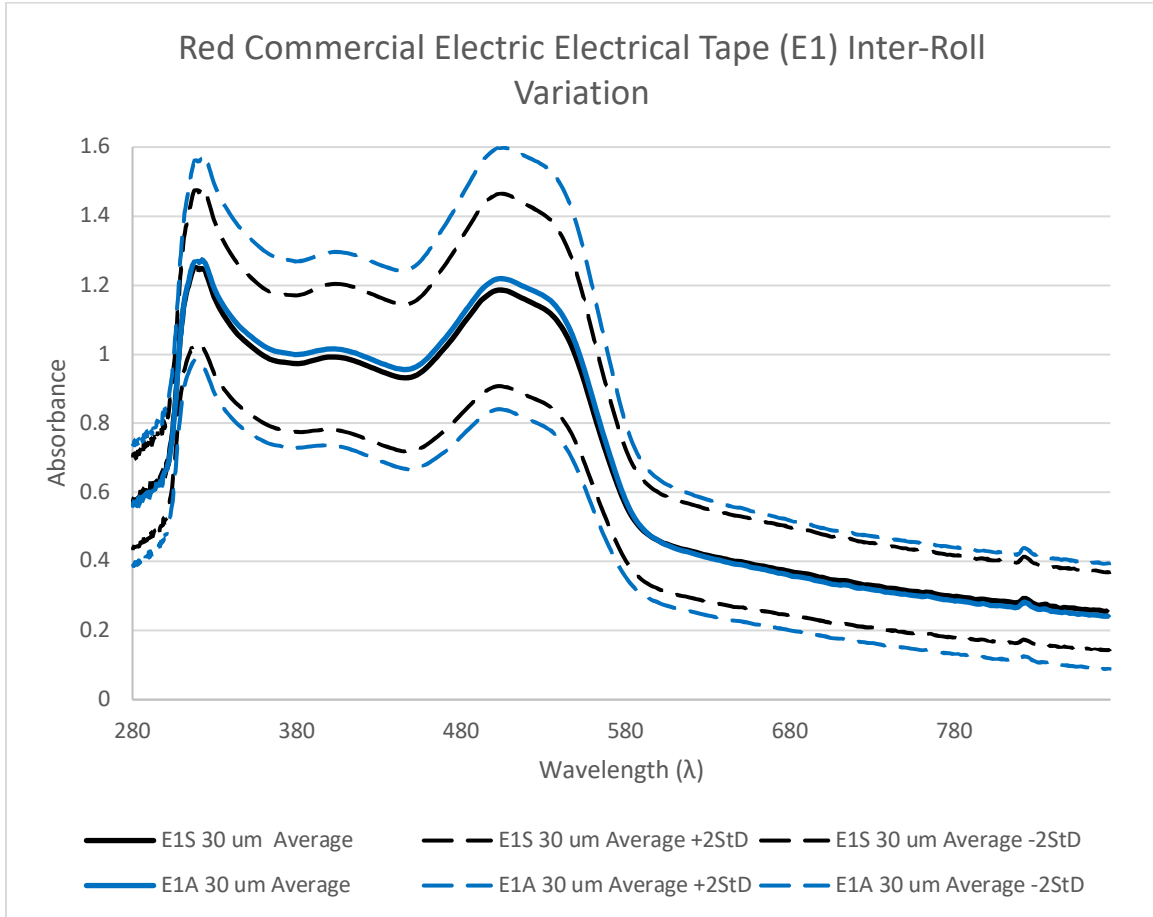


Figure 76.) Blue Scotch 3M Duct Tape (D4) Inter-Roll Variation



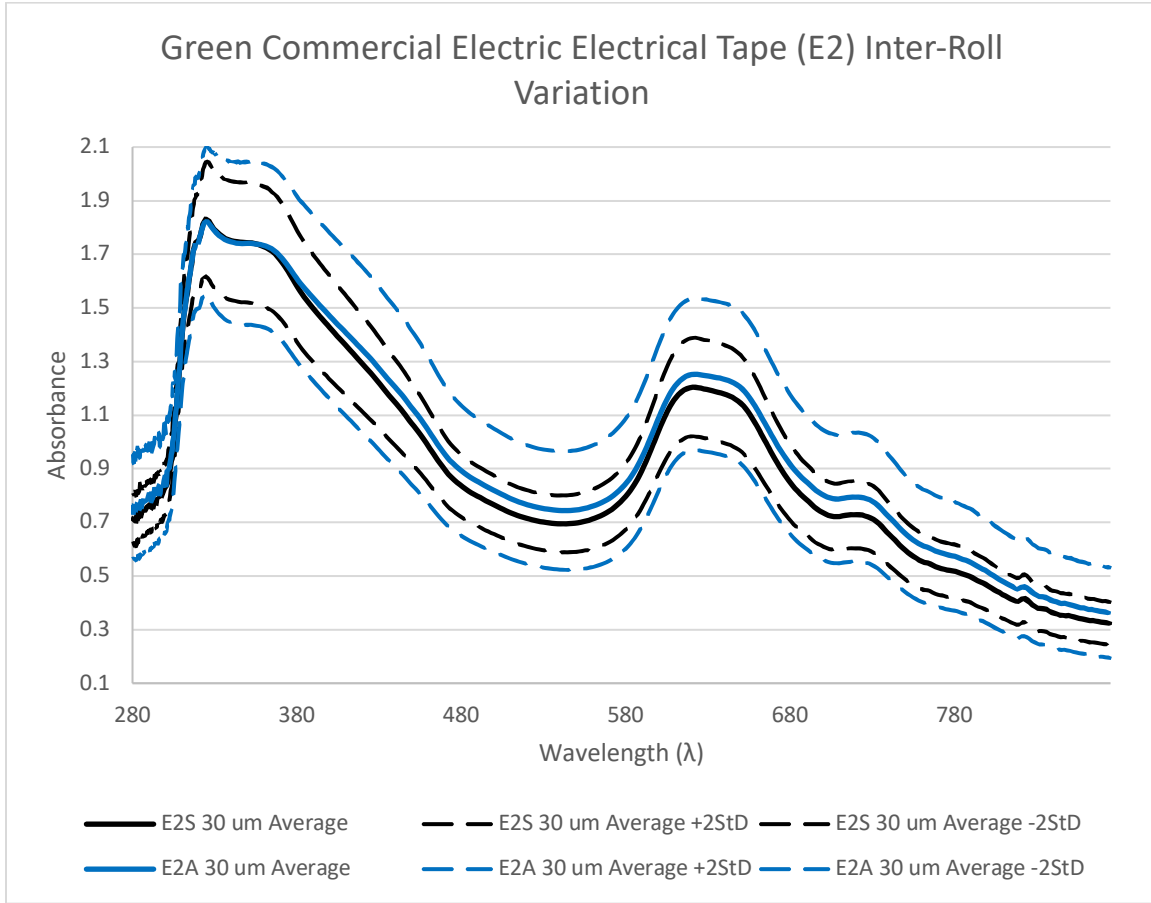
The above figure compares the average absorbance spectra through 30  $\mu\text{m}$  microtomed cross sections at the approximate beginning (S) of two different rolls of blue Scotch 3M duct tape (D4 and D4A) from 280 to 875 nm. A solid line indicates the average of fifty scans and a pair of dashed lines indicates plus or minus two standard deviations from the mean.

Figure 77.) Red Commercial Electric Electrical Tape (E1) Inter-Roll Variation



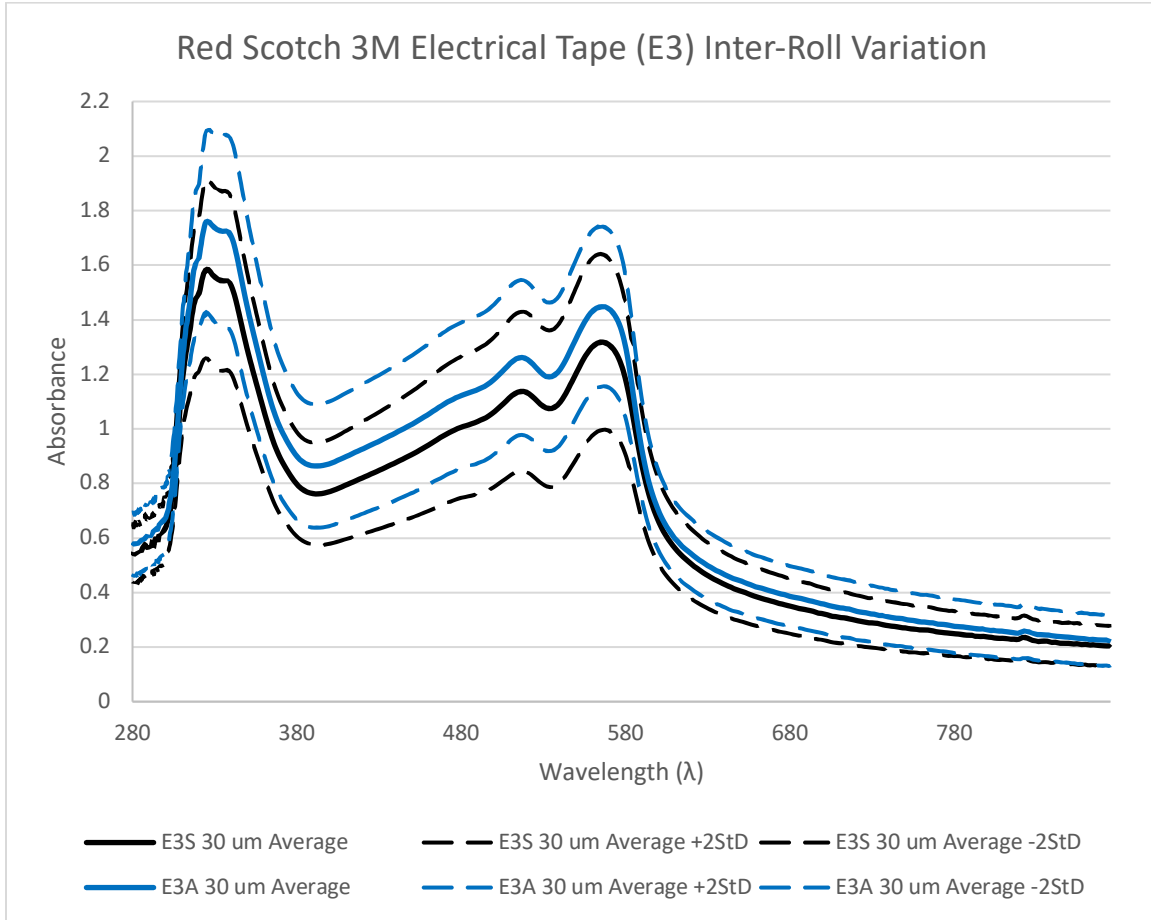
The above figure compares the average absorbance spectra through 30 μm microtomed cross sections at the approximate beginning (S) of two different rolls of red Commercial Electric electrical tape (E1 and E1A) from 280 to 875 nm. A solid line indicates the average of fifty scans and a pair of dashed lines indicates plus or minus two standard deviations from the mean.

Figure 78.) Green Commercial Electric Electrical Tape (E2) Inter-Roll Variation



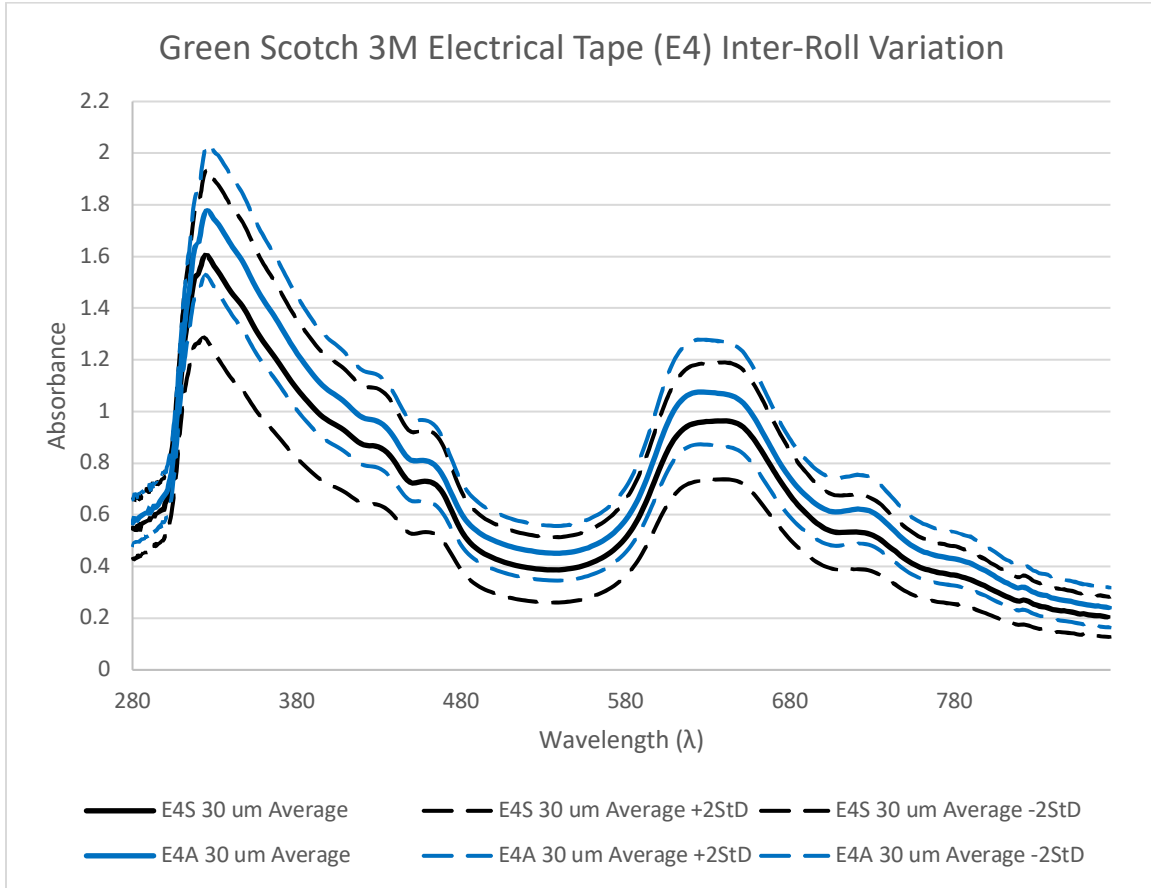
The above figure compares the average absorbance spectra through 30  $\mu\text{m}$  microtomed cross sections at the approximate beginning (S) of two different rolls of green Commercial Electric electrical tape (E2 and E2A) from 280 to 875 nm. A solid line indicates the average of fifty scans and a pair of dashed lines indicates plus or minus two standard deviations from the mean.

Figure 79.) Red Scotch 3M Electrical Tape (E3) Inter-Roll Variation



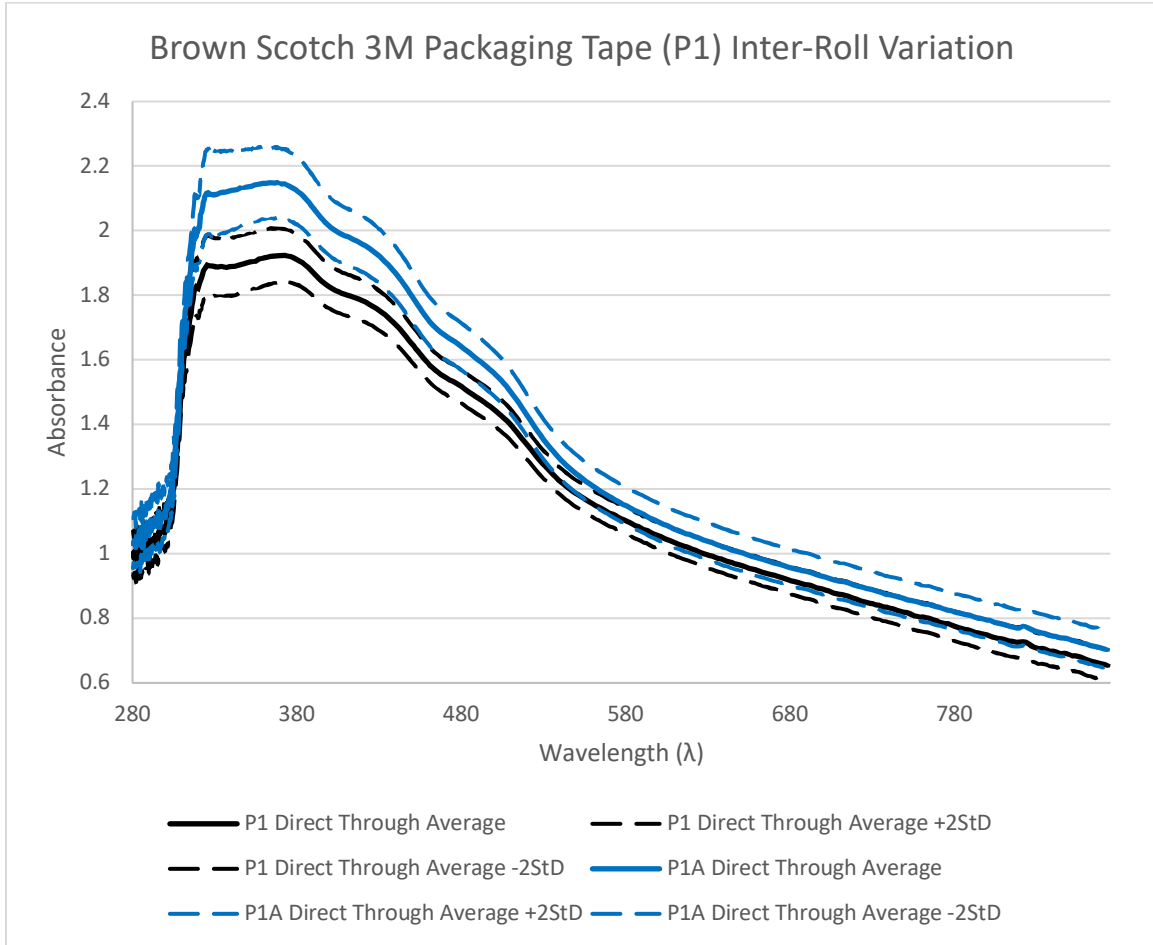
The above figure compares the average absorbance spectra through 30  $\mu\text{m}$  microtomed cross sections at the approximate beginning (S) of two different rolls of red Scotch 3M electrical tape (E3 and E3A) from 280 to 875 nm. A solid line indicates the average of fifty scans and a pair of dashed lines indicates plus or minus two standard deviations from the mean.

Figure 80.) Green Scotch 3M Electrical Tape (E4) Inter-Roll Variation



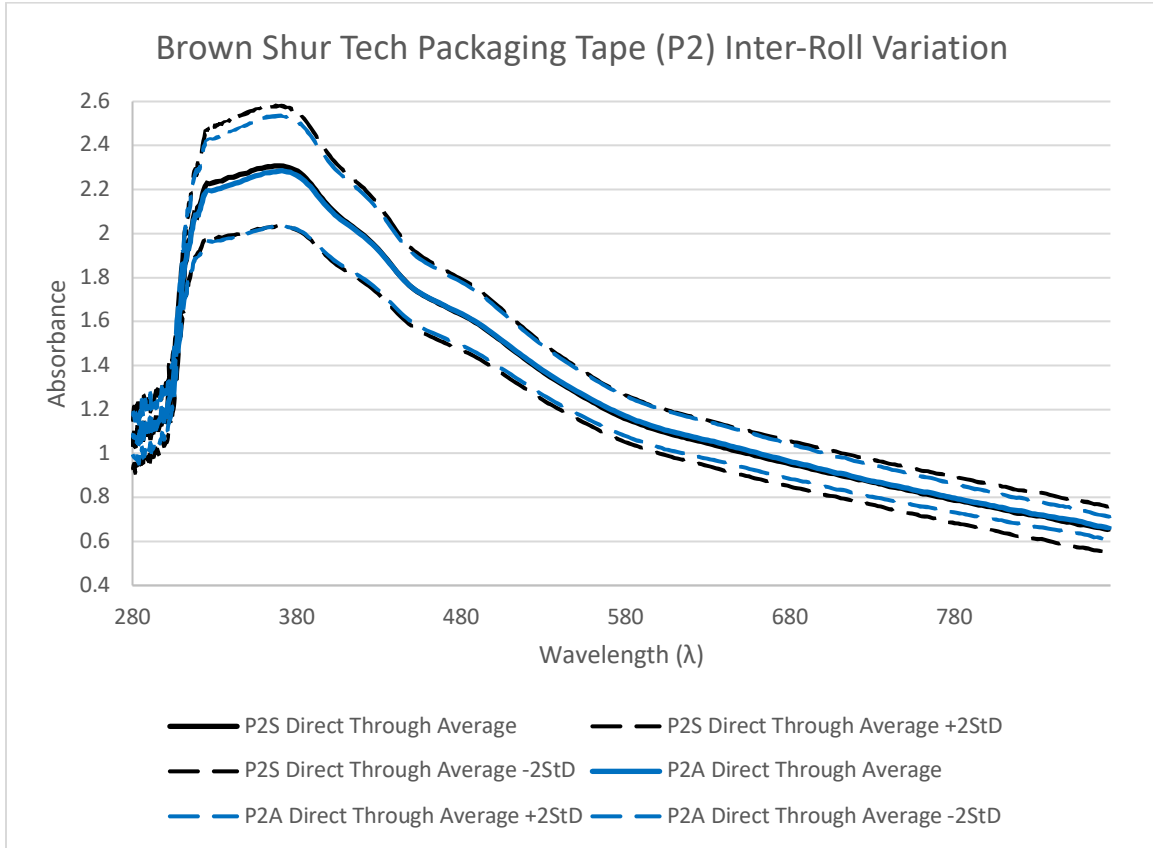
The above figure compares the average absorbance spectra through 30 μm microtomed cross sections at the approximate beginning (S) of two different rolls of green Scotch 3M electrical tape (E4 and E4A) from 280 to 875 nm. A solid line indicates the average of fifty scans and a pair of dashed lines indicates plus or minus two standard deviations from the mean.

Figure 81.) Brown Scotch 3M Packaging Tape (P1) Inter-Roll Variation



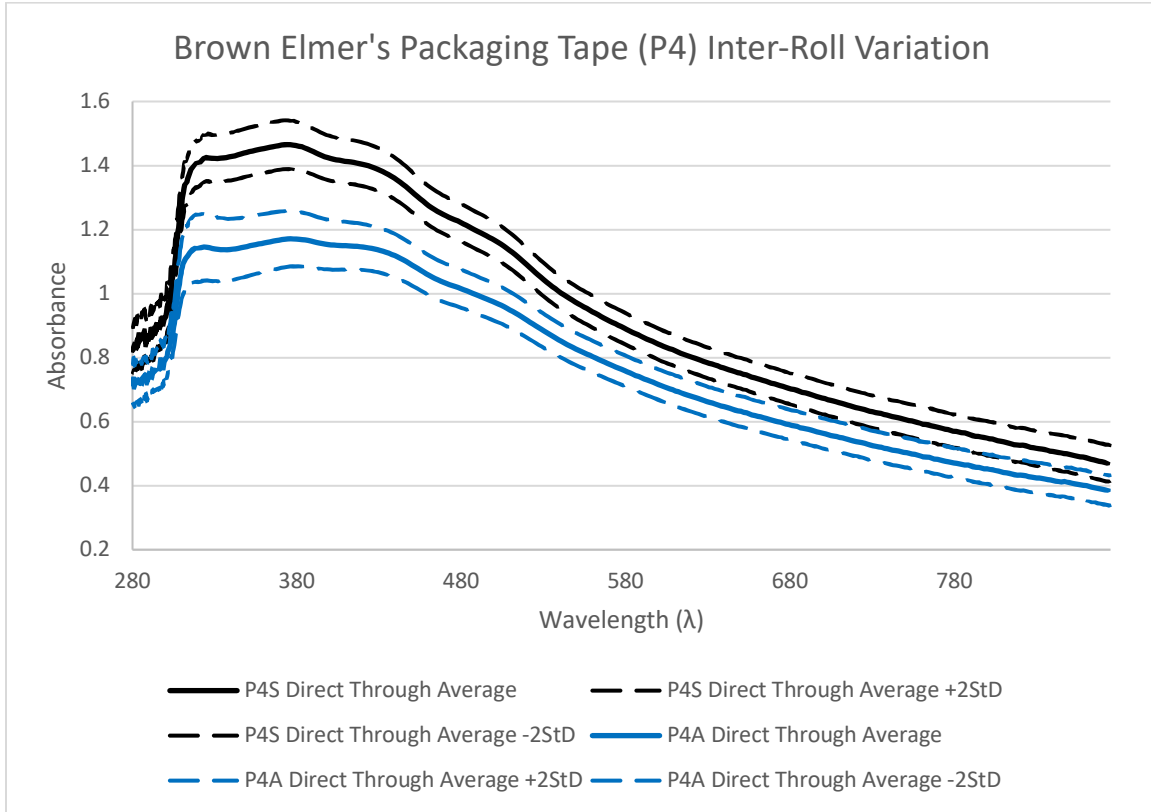
The above figure compares the average absorbance spectra directly through the approximate beginning (S) of two different rolls of brown Scotch 3M packaging tape (P1 and P1A) from 280 to 875 nm. A solid line indicates the average of fifty scans and a pair of dashed lines indicates plus or minus two standard deviations from the mean.

Figure 82.) Brown Shur Tech Packaging Tape (P2) Inter-Roll Variation



The above figure compares the average absorbance spectra directly through the approximate beginning (S) of two different rolls of brown Shur Tech packaging tape (P2 and P2A) from 280 to 875 nm. A solid line indicates the average of fifty scans and a pair of dashed lines indicates plus or minus two standard deviations from the mean.

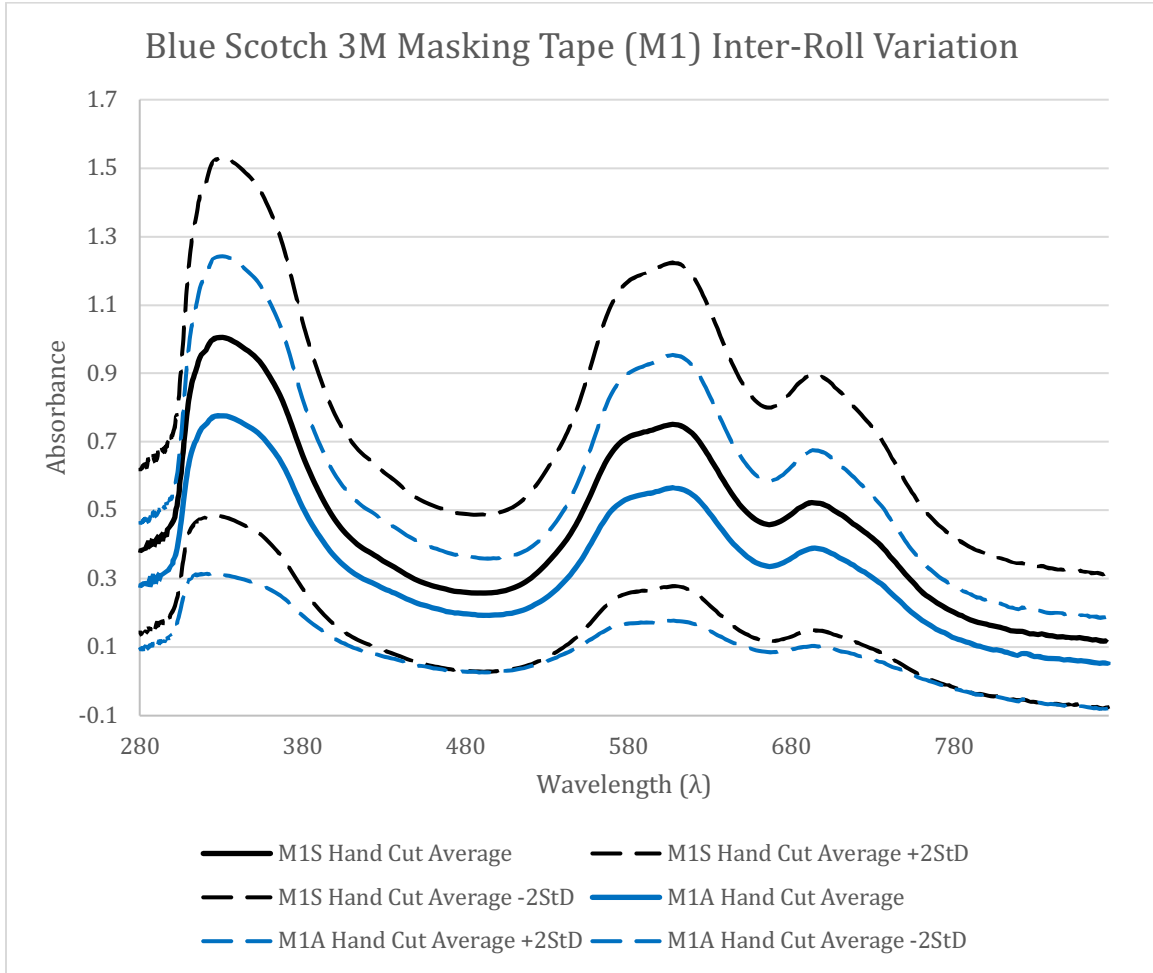
Figure 83.) Brown Elmer's Packaging Tape (P4) Inter-Roll Variation



The above figure compares the average absorbance spectra directly through the approximate beginning (S) of two different rolls of brown Elmer's packaging tape (P4 and P4A) from 280 to 875 nm. A solid line indicates the average of fifty scans and a pair of dashed lines indicates plus or minus two standard deviations from the mean.

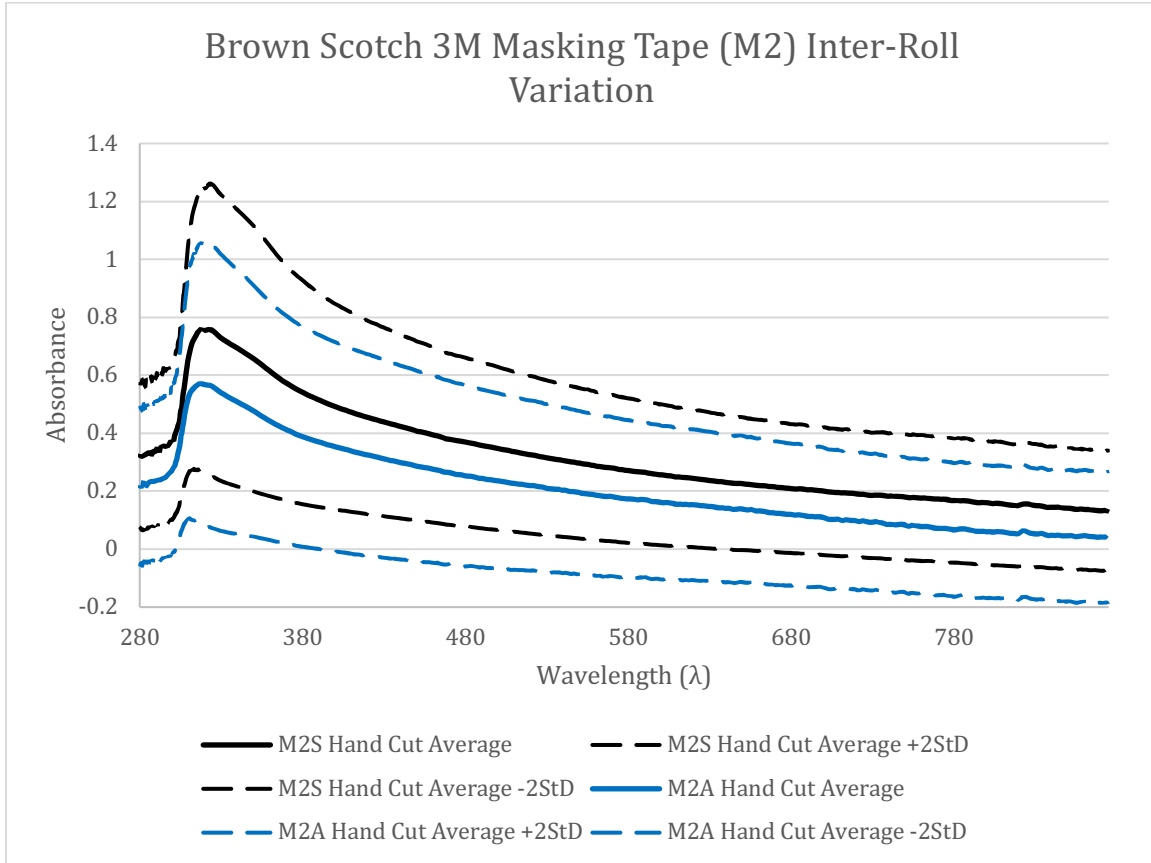


Figure 84.) Blue Scotch 3M Masking Tape (M1) Inter-Roll Variation



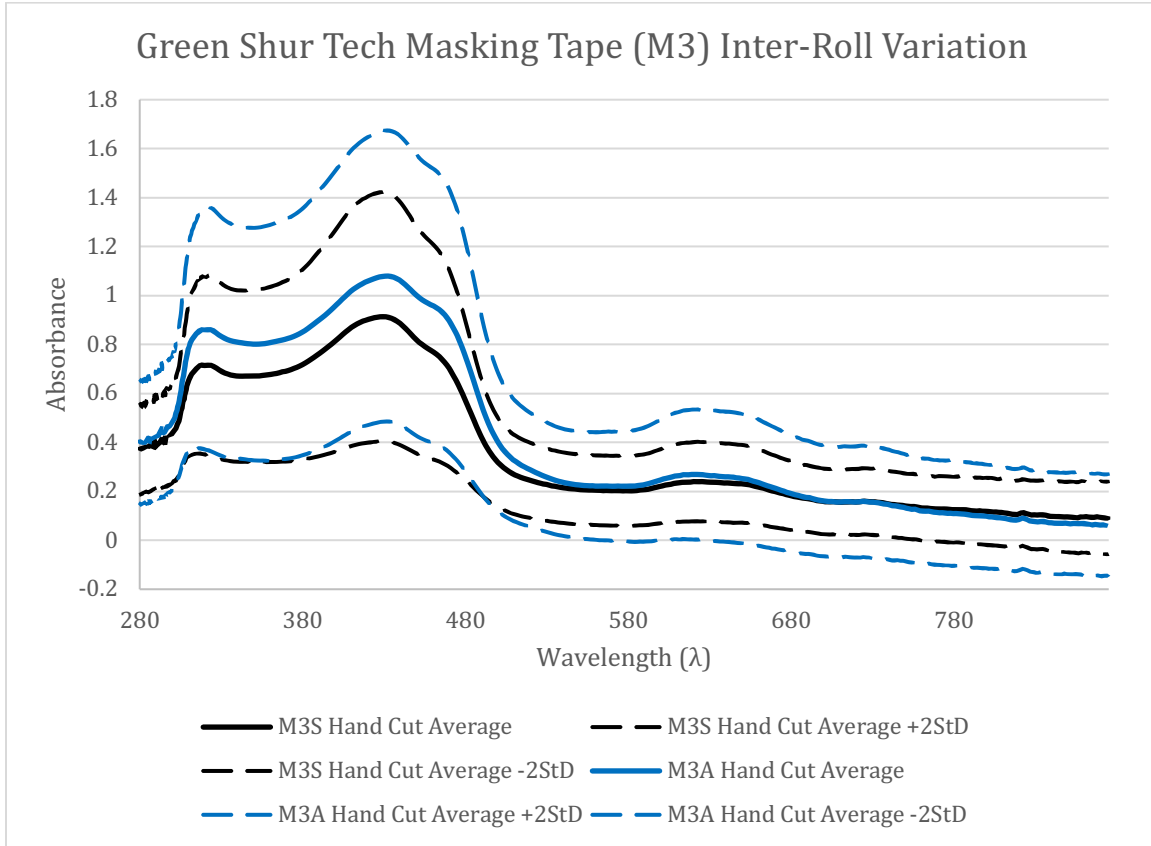
The above figure compares the average absorbance spectra through hand cut cross sections of the approximate beginning (S) of two different rolls of blue Scotch 3M masking tape (M1 and M1A) from 280 to 875 nm. A solid line indicates the average of fifty scans and a pair of dashed lines indicates plus or minus two standard deviations from the mean.

Figure 85.) Brown Scotch 3M Masking Tape (M2) Inter-Roll Variation



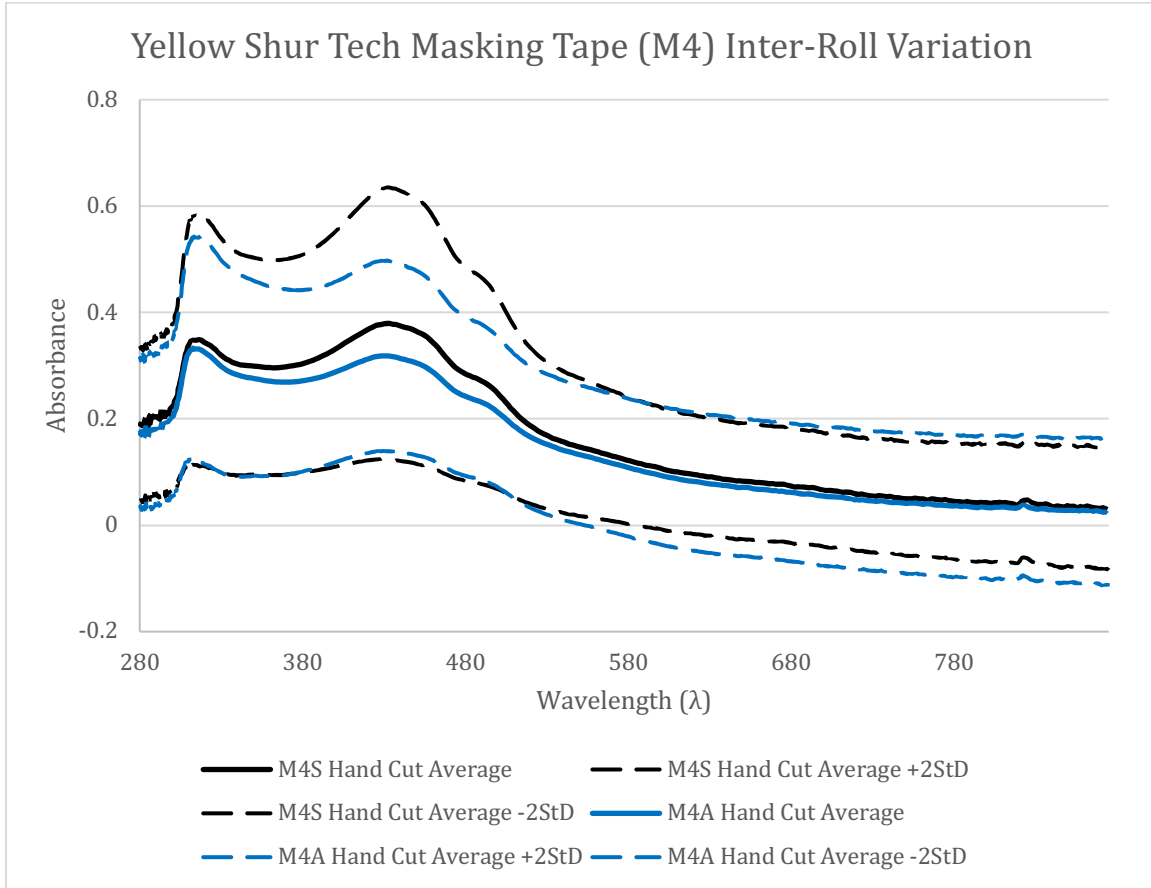
The above figure compares the average absorbance spectra through hand cut cross sections of the approximate beginning (S) of two different rolls of brown Scotch 3M masking tape (M2 and M2A) from 280 to 875 nm. A solid line indicates the average of fifty scans and a pair of dashed lines indicates plus or minus two standard deviations from the mean.

Figure 86.) Green Shur Tech Masking Tape (M3) Inter-Roll Variation



The above figure compares the average absorbance spectra through hand cut cross sections of the approximate beginning (S) of two different rolls of green Shur Tech masking tape (M3 and M3A) from 280 to 875 nm. A solid line indicates the average of fifty scans and a pair of dashed lines indicates plus or minus two standard deviations from the mean.

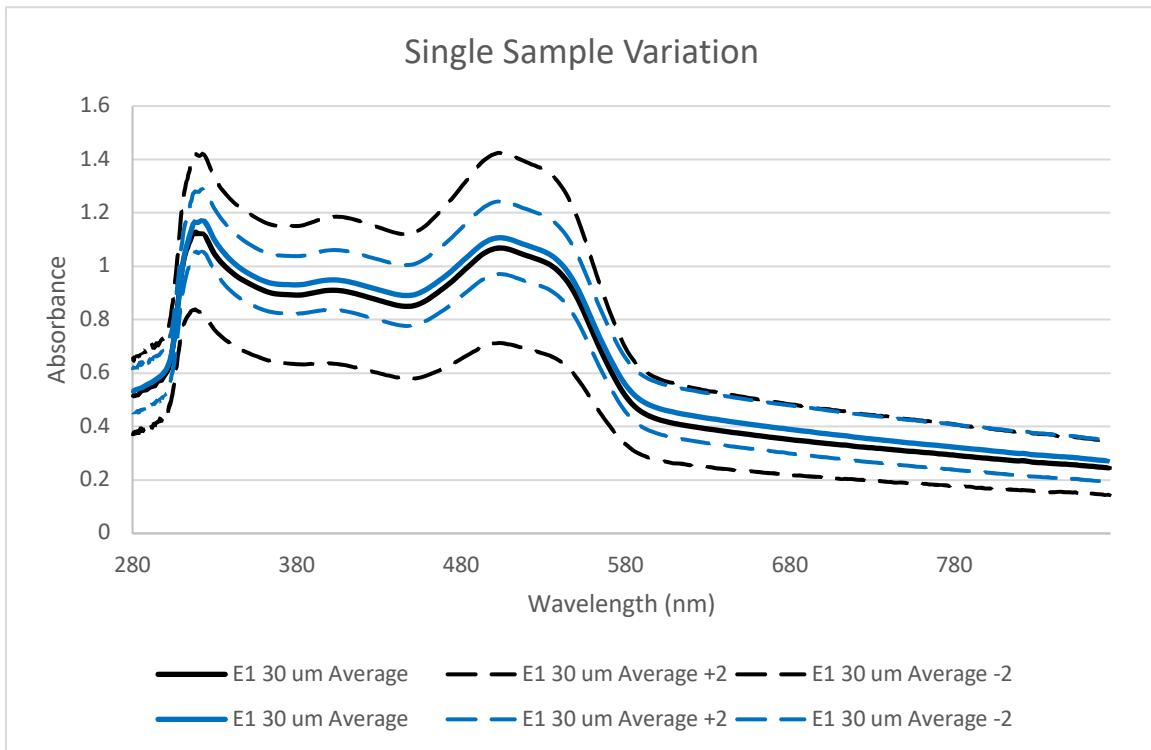
Figure 87.) Yellow Shur Tech Masking Tape (M4) Inter-Roll Variation



The above figure compares the average absorbance spectra through hand cut cross sections of the approximate beginning (S) of two different rolls of yellow Shur Tech masking tape (M4 and M4A) from 280 to 875 nm. A solid line indicates the average of fifty scans and a pair of dashed lines indicates plus or minus two standard deviations from the mean.

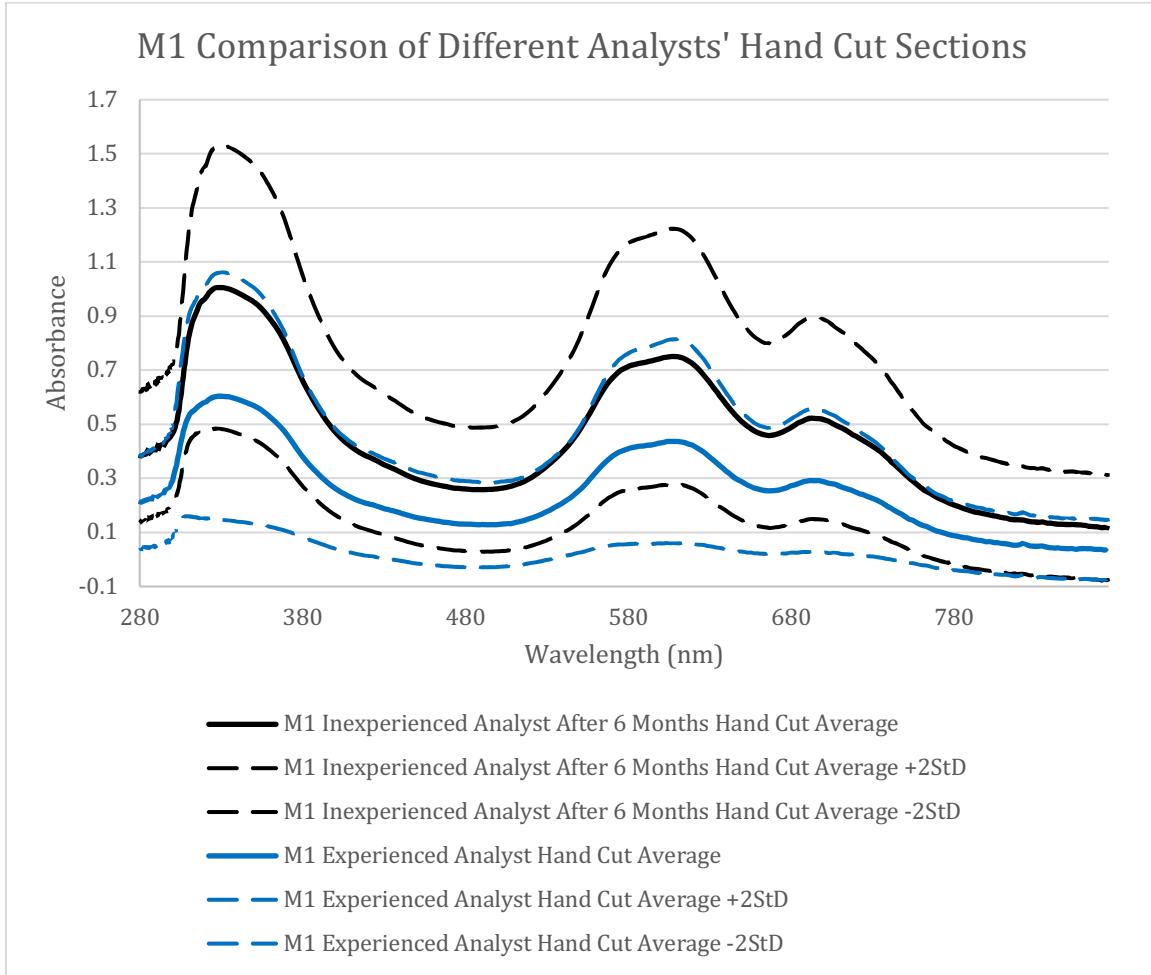
## Appendix E

Figure 88.) Single Sample Variation



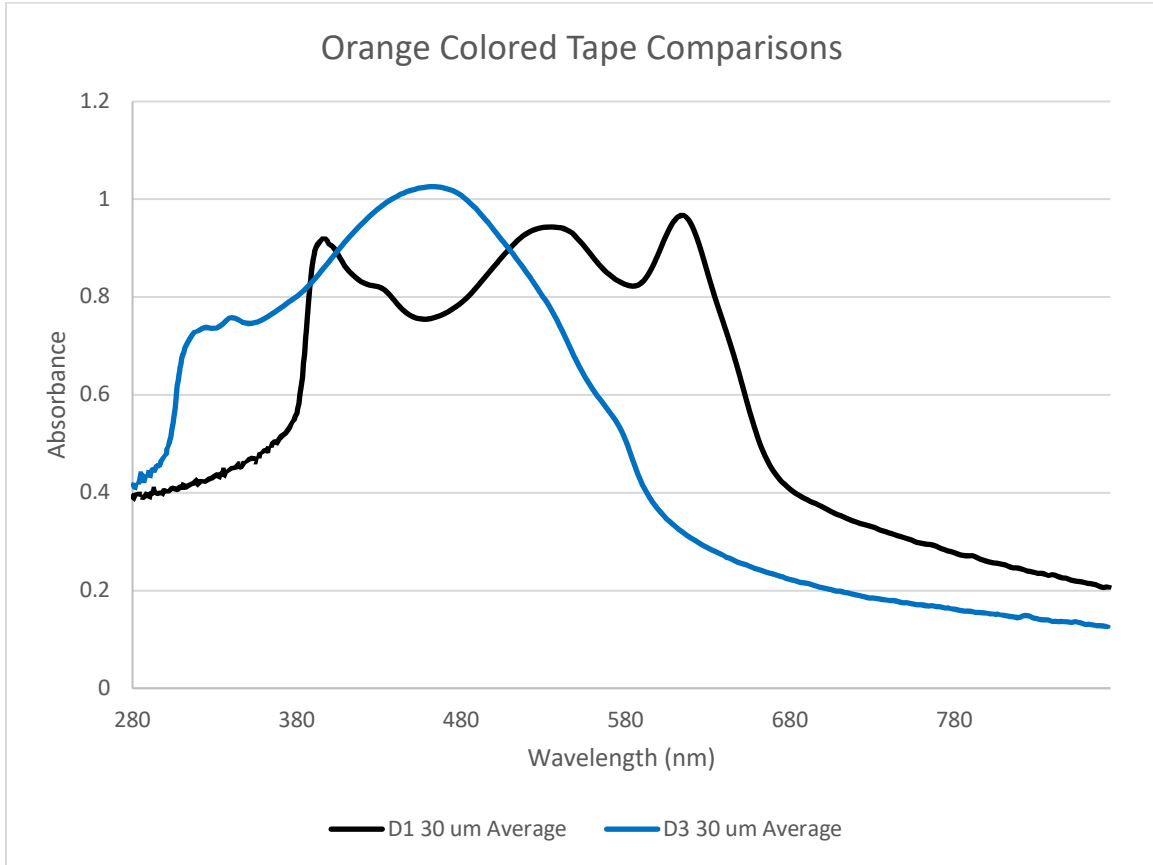
The above figure compares the average absorbance spectra through 30  $\mu\text{m}$  microtomed cross sections of the same red Commercial Electric electrical tape sample (E1) from 280 to 875 nm. A solid line indicates the average of fifty scans and a pair of dashed lines indicates plus or minus two standard deviations from the mean.

Figure 89.) M1 Comparison of Different Analysts' Hand Cut Sections



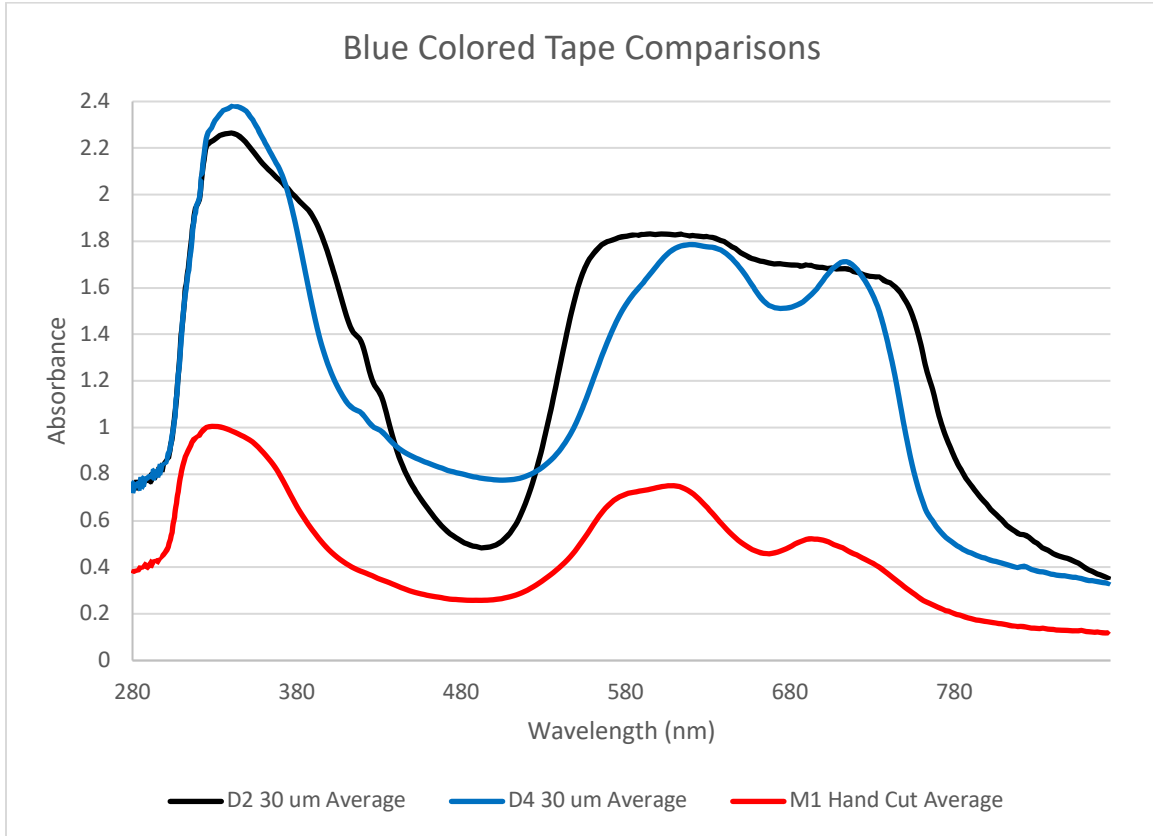
The above figure compares the average absorbance spectra through hand cut cross sections of blue Scotch 3M masking tape (M1) cut by different analysts from 280 to 875 nm. A solid line indicates the average of fifty scans and a pair of dashed lines indicates plus or minus two standard deviations from the mean.

Figure 90.) Orange Colored Tape Comparisons



The above figure compares the average absorbance spectra through 30  $\mu\text{m}$  microtomed cross sections of orange Shur Tech duct tape (D1) and orange Scotch 3M duct tape (D3) from 280 to 875 nm. A solid line indicates the average of fifty scans.

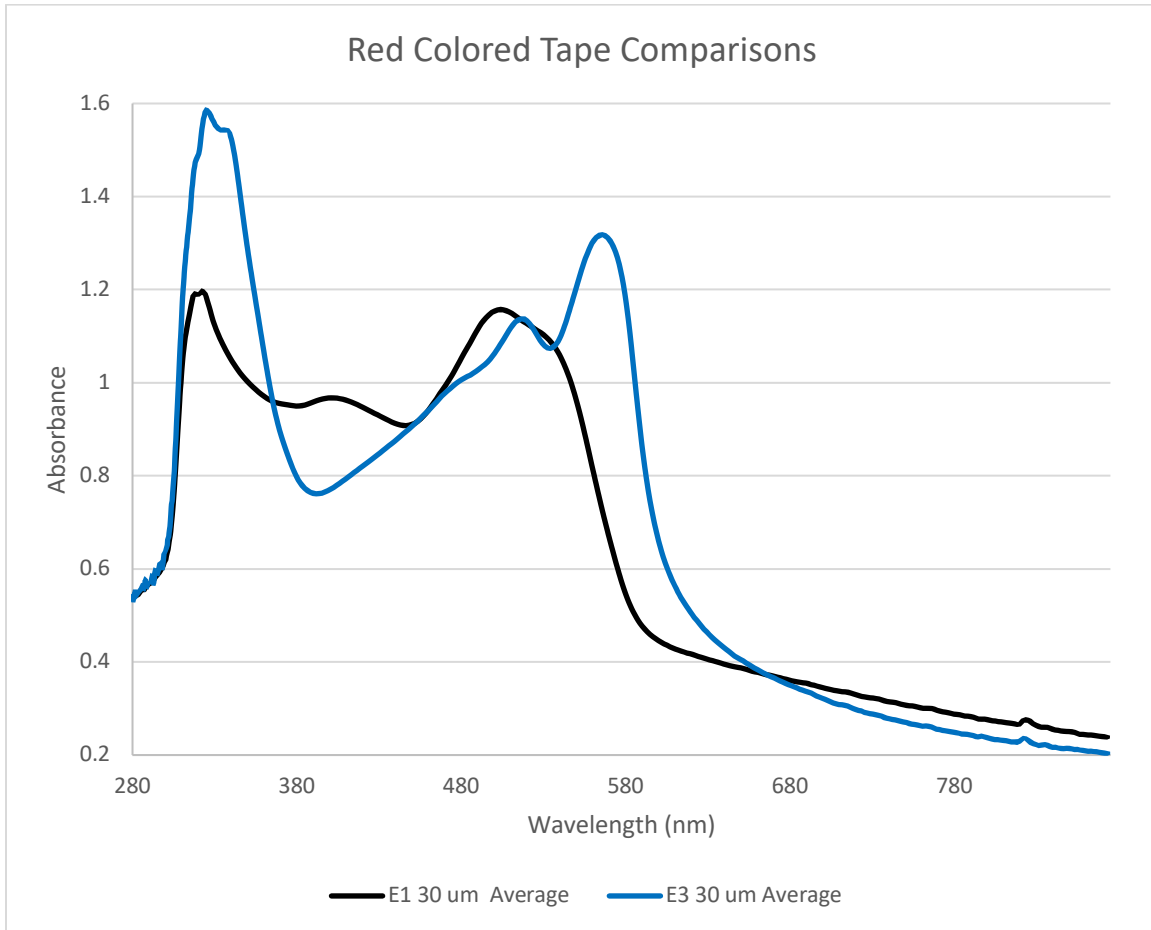
Figure 91.) Blue Colored Tape Comparisons



The above figure compares the average absorbance spectra through 30  $\mu\text{m}$  cross sections of blue Shur Tech duct tape (D2) and blue Scotch 3M duct tape (D4) as well as hand cut cross sections of blue Scotch 3M masking tape (M1) from 280 to 875 nm. A solid line indicates the average of fifty scans.

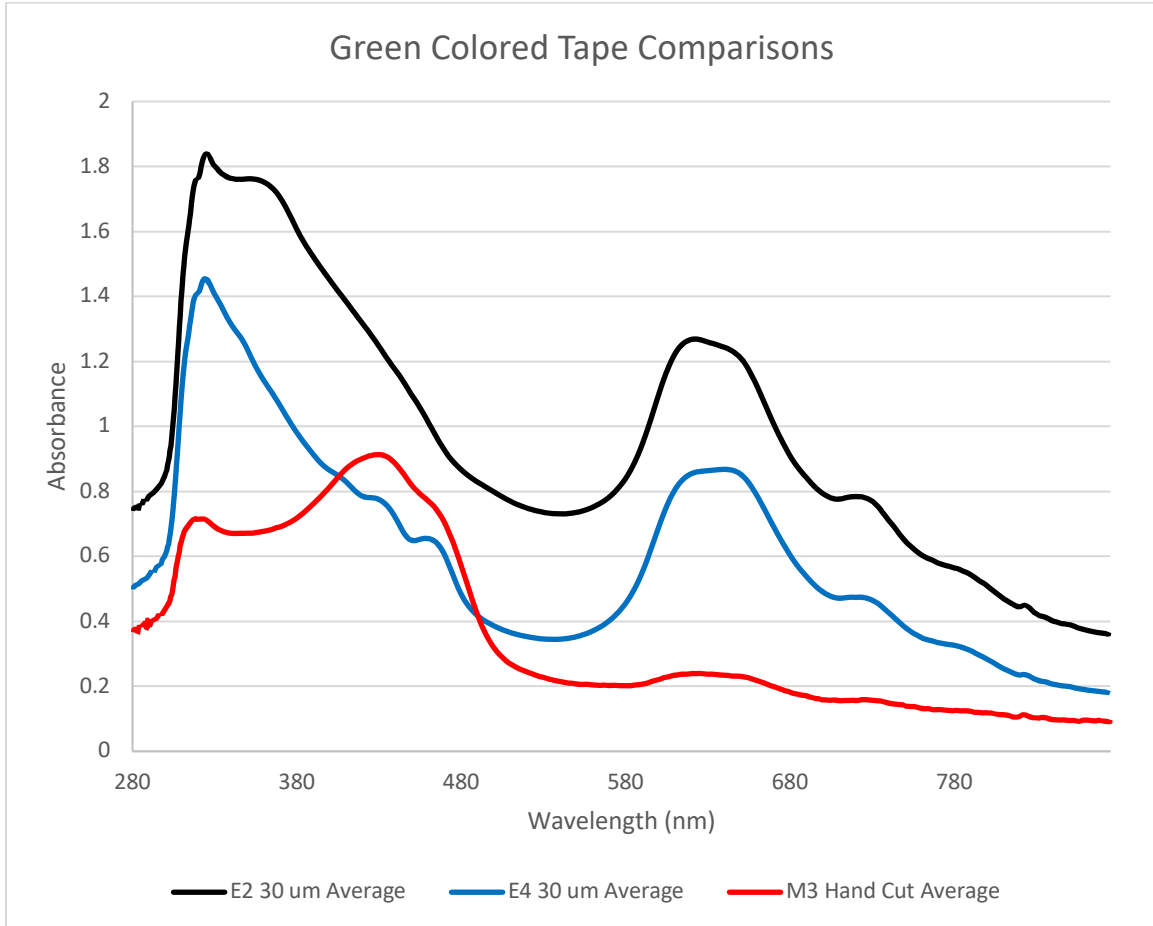


Figure 92.) Red Colored Tape Comparisons



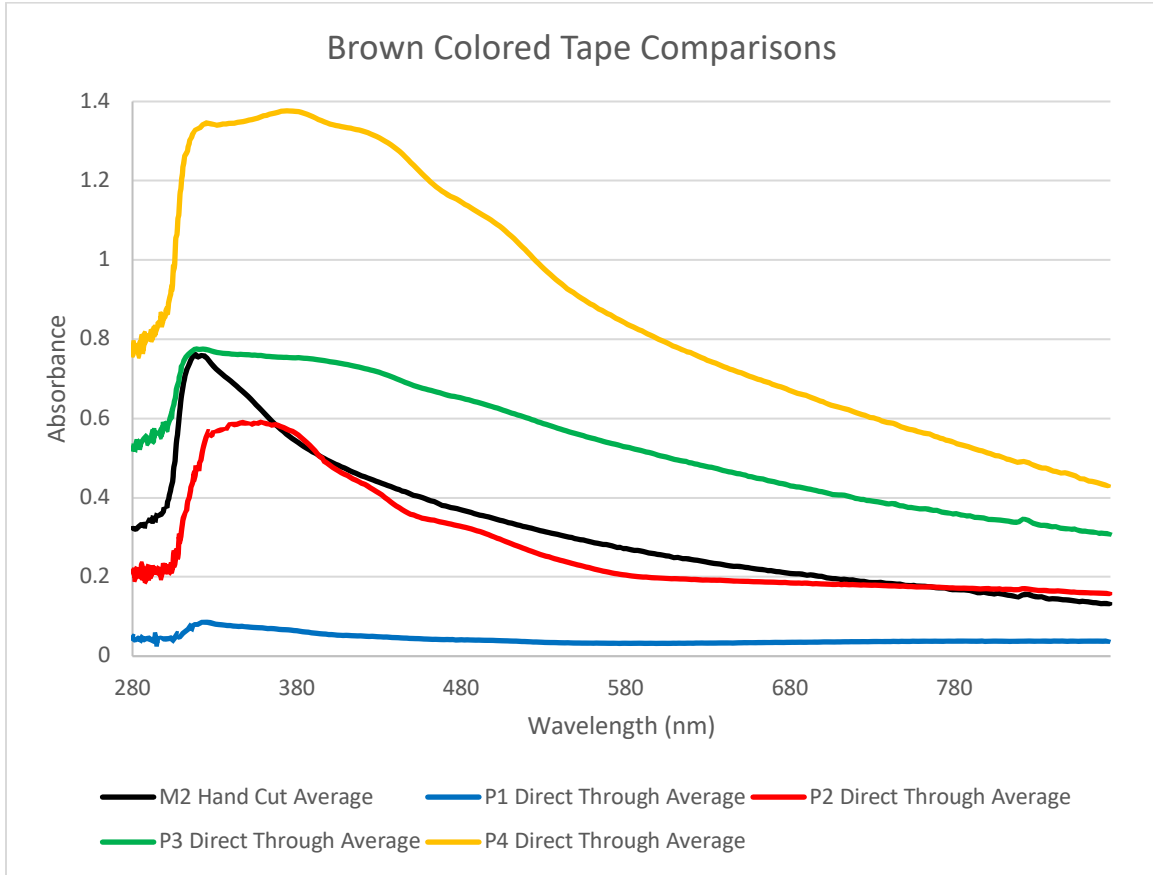
The above figure compares the average absorbance spectra through 30  $\mu\text{m}$  cross sections of red Commercial Electric electrical tape (E1) and red Scotch 3M electrical tape (E3) from 280 to 875 nm. A solid line indicates the average of fifty scans.

Figure 93.) Green Colored Tape Comparisons



The above figure compares the average absorbance spectra through 30  $\mu\text{m}$  cross sections of green Commercial Electric electrical tape (E2) and green Scotch 3M electrical tape (E4) as well as hand cut cross sections of green Shur Tech masking tape (M3) from 280 to 875 nm. A solid line indicates the average of fifty scans.

Figure 94.) Brown Colored Tape Comparisons



The above figure compares the average absorbance spectra directly through brown Scotch 3M packaging tape (P1), brown Shur Tech packaging tape (P2), brown Elmer's packaging tape (P3), and brown Tartan packaging tape (P4) as well as hand cut cross sections of brown Scotch 3M masking tape (M2) from 280 to 875 nm. A solid line indicates the average of fifty scans.

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