

COMPARATIVE FORENSIC TAPE EXAMINATIONS: INTRODUCING BETTER DISCRIMINATION CAPABILITIES

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Introduction

The FBI Laboratory continues to develop capabilities to allow for better discrimination between submitted tape evidence items. Through a combination of internal and collaborative research efforts as well as maintaining communication channels with industry contacts, efforts to improve characterization and differentiation of the various types of tape submitted to the FBI Laboratory have led to new and/or improved technical procedures for electrical, duct, and packaging tapes. The following discussion will highlight these successes as well as the challenges that forensic tape samples present when determining if a used piece of tape could have originated from a partial roll of tape, or other rolls manufactured in the same manner.

The FBI Laboratory's Chemistry Unit is equipped to analyze and compare a variety of pressure sensitive adhesive (PSA) tapes that are received as part of a criminal investigation. The request for assistance most often originates from an FBI field office, a local or state law enforcement agency, or laboratory that is not equipped to analyze tapes or adhesives. PSAs with a paper backing (i.e., masking tape) or common office tape are not accepted in the Chemistry Unit as their physical properties are better evaluated by a different set of forensic analysts in the Lab. When a (partial) roll of duct tape is not recovered, the Chemistry Unit attempts to source the received tape pieces to a known manufacturer, distributor, and/or point of sale unit in the event that a roll can be recovered or an investigative lead generated/corroborated using this information. Only duct tapes contain sufficient physical and chemical properties and variation in these properties to allow for sourcing examinations to be pursued.

Physical examinations of PSA tapes in the FBI Laboratory include assessment and documentation of features associated with all three components of the tape: the polymeric film backing, the adhesive, and in duct tapes, the fabric reinforcement (1-5). These examinations can be conducted prior to or concurrent with physical fit (i.e., end match) assessments (6-7). A physical realignment of torn edges of two or more tape pieces is the strongest conclusion that can be reported, second only to an elimination of a partial tape roll as the source of physically consistent tape pieces.

Duct Tapes

Duct tapes have numerous features that can be leveraged to determine everything from the grade of tape to possible manufacturer, or distributor depending upon the features available for comparison to known rolls. Comparatively speaking, tapes can be forensically differentiated based on film color, film thickness, film construction (i.e., blown vs calendered, layer structure, orientation), scrim count, yarn construction (weave), fiber features (e.g., fluorescence), and/or adhesive color. The FBI Laboratory has published a study of over 80 duct tape samples in which it was reported that the discrimination power by

visual and microscopical examinations alone is 99.6%. These physical characteristics are heavily leveraged in determining if two or more tape pieces could have originated from a given roll of duct tape. Features that are often unrealized by the general public in purchasing a roll of tape can be readily understood by jurors in court testimony when the features are described and compared for the purposes of explaining how tapes are associated and discriminated in casework.

Once physical examinations are completed, chemical analysis is conducted on the adhesive, film backing layers, and yarn fibers using sample sizes on the order of microgram to milligram samples and single fibers of the scrim yarn. Instrumental analysis techniques include Fourier transform infrared spectroscopy (FTIR) (8) and scanning electron microscopy with energy dispersive X-ray spectroscopy (SEM/EDS) on the adhesive and film components (9-11). X-ray diffraction spectrometry (XRD) is conducted on intact duct tape pieces (2). On occasion, pyrolysis gas chromatography with mass spectral detection (Py-GC/MS) may also be utilized to characterize the adhesive portion of a duct tape (10, 12).

Visual and microscopical examinations

The following parameters are examined and documented in each case: backing film and adhesive colors; film surface features; film layer structure; tape width; fabric characteristics of weave/knit pattern; yarn description (e.g., twisted); yarn composition (e.g., synthetic or cotton); yarn fluorescence; fiber birefringence; scrim count. Some of these features are more readily determined and can be used orthogonally with initial instrumental examinations on a subset of tape pieces in instances where numerous (i.e., hundreds of) tape pieces or fragments are all received as a single unit for comparison to other tapes. Observed differences in any of these characteristics that can be attributed to true manufacturing differences would be sufficient cause to report that the duct tapes being compared are not from the same source.

A “source” is generally defined as a partial roll of known duct tape. However, it is understood that numerous rolls originate from each jumbo roll and that jumbo rolls of the same product type and manufacture can also produce indistinguishable rolls. Therefore, in the absence of a physical fit, reporting of associations between tape pieces and/or tape pieces to a roll are caveated to document that the concurrence is a classification of like products rather than an identification to a single roll.

For the backing and adhesive color, the observations are conducted with the unaided eye. Surface features of the backings can also be observed unaided as well as with a stereomicroscope (~5x-20x). To determine the number of layers that comprise the film, thin cross-sections are taken using a straight edge scalpel blade so that the layer structure can be viewed with transmission light microscopy (5).

Width measurements are taken with a ruler to the nearest 0.5 mm. To best visualize the fabric, the adhesive is removed with a suitable solvent such as hexane. The weave/knit pattern and general yarn description are both observed by stereomicroscopy. Yarn fluorescence is initially observed under long wave UV light ($\lambda = 366$ nm) using a handheld light. Fiber analysis also includes a more comprehensive analysis of fluorescence using a polarized light microscope with dedicated fluorescence cubes over a series of wavelengths.

The scrim count is measured using a ruler, counted per square inch, and recorded as number of warp yarns / number of weft yarns. Scrim counts of +/-1 are generally acceptable in the manufacturing of duct tape products (1).

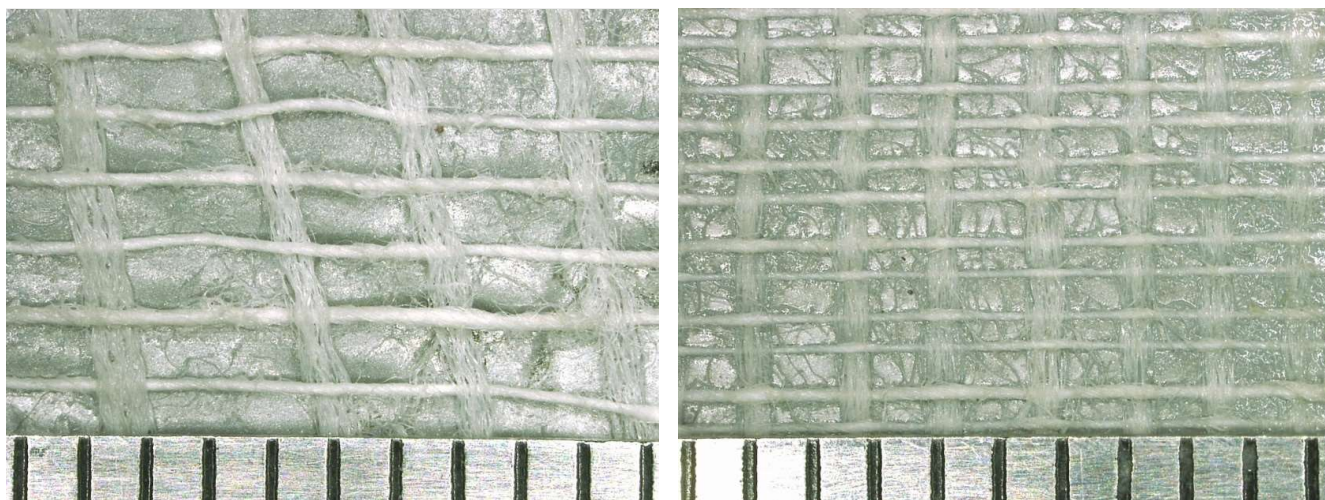


Figure 1. Side-by-side comparison of the scrim in two consumer grade duct tape products. Adhesive has been removed by dropwise addition of solvent blotted away with a laboratory paper wipe. (Scale = mm)

FTIR

FTIR analysis of the adhesive is conducted by smearing a thin film of the adhesive on a single diamond window using a microscope attachment equipped with a MCT/A detector ($4000\text{-}650\text{cm}^{-1}$), coupled to a FTIR spectrometer. The spectra and background are each collected using an approximate aperture size of 100×100 microns and 128 scans at 4 cm^{-1} resolution. Data collection and processing are conducted using the instrument's software package.

Analysis of the film backing requires removal of the adhesive with hexane (or another inert solvent) and manually cutting away the scrim using a scalpel and tweezers. Then, each side of the film is analyzed using an attenuated total reflectance (ATR) accessory attached to the IR. The spectra and background are each collected using 32 scans at 4 cm^{-1} resolution.

SEM/EDS

The adhesive is sampled using a solid metal probe to tease up a portion of the material into a ball that is smeared into a thin film on a pyrolytic carbon planchet. For analysis of the film, a small sample ($\sim 5 \times 5$ mm) of the tape is cut with a clean scalpel blade and affixed to the same carbon planchet using its own adhesive. A thin layer of carbon paint can be applied to the edges of the film to mitigate charging in the SEM before the planchet is carbon coated by vacuum evaporation for the same reason. Elemental analysis is then conducted under high vacuum with a tungsten filament as the source. The detector is operated with a dead time of approximately 30% and live count time of 50-200 s. The working distance is approximately 12 -15 mm, the take-off angle is approximately 30° , and the accelerating voltage is generally set at 25 kV.

XRD

A portion of the intact tape is cut with a clean scalpel blade to roughly the size of a low background silicon wafer ($\sim \frac{1}{2}$ "- 1" diameter) that is placed onto a rotating stage within the XRD. An X-ray diffractometer is employed using $\text{Cu K}\alpha$ radiation, operated at $\sim 45\text{kV}$ and $\sim 40 \text{ mA}$, scanning continuously between 8 and $80^\circ 2\theta$ with a step size of $0.0170^\circ 2\theta$, and a 10 mm beam mask. Total analysis time is approximately 8 minutes.

Py-GC/MS

The adhesive sample size is approximated to 50 micrograms as needed to ensure an adequate signal response. Samples are placed in the center of a metal cup that is loaded into an autosampler with blank cups run on either side of each sample. The autosampler is coupled to a gas chromatograph. Working conditions for the pyrolysis unit include a ramping step in which the initial temperature is set at 300 °C and ramped to 600 °C, and held at that temperature for 0.2 min, with an interface temperature of 321 °C. The column is a HP-5MS (40 m X 250 µm X 0.25 µm). The carrier gas is helium at a purity of 99.99% set at a constant flow and an initial flow rate of 0.7 mL/min. The GC is held at an initial temperature of 50 °C for 2 minutes, ramped at a rate of 13 °C /min to 325 °C, and held for 15 minutes. The inlet is operated at 300 °C in 50:1 split mode and the transfer line temperature is set to 300 °C. Analytes are detected on a single quadrupole mass selective detector with a dedicated electron impact ionization (EI) source run in full scan mode at a scan range of 34-650 m/z. The source temperature is 230 °C and the quadrupole temperature is 150 °C.

Electrical Tapes (i.e., Vinyl Tapes, Black Plastic Tapes)

Electrical tapes have far fewer physical features available to evaluate and compare to known rolls. For this reason, forensic tape examiners do not attempt to source these tapes to a manufacturer, distributor, or point of sale. These tapes can be forensically differentiated based on film width and/or adhesive color. Film thickness is less critically evaluated for discrimination purposes given its propensity to stretch and deform. Film features such as gloss or topography (e.g., crepe, orange peel, smooth appearance) can be noted but generally are not considered sufficient to conclusively discriminate compared tapes on their own.

The FBI Laboratory has published several studies of over 90 black-colored electrical tape samples in which it was reported that the discrimination power by visual and microscopical examinations alone is 64% based on film characteristics and 53% for adhesive color (13, 14).

Once physical examinations are completed, chemical analysis is conducted on the adhesive and film backing using sample sizes on the order of micrograms for FTIR, SEM/EDS, and PyGC/MS. X-ray fluorescence spectroscopy (XRF) has recently been incorporated into the examination scheme for the film backings (15) based on research conducted by colleagues at West Virginia University who ran experiments on FBI Lab instrumentation to help validate the method and demonstrate its improved discrimination capabilities vs SEM/EDS on PVC electrical tape films (16).

Published discrimination power results for these techniques on the same 90 electrical tapes described above are as follows: 83% for films and 67% for adhesives using IR spectroscopy; 81% for films and 83% for adhesives using PyGC/MS; 87% for films and 17% for adhesives using SEM/EDS; and 97% for films using XRF (13-16). The reported results for adhesives analysis by SEM/EDS have limited its use for this electrical tape component in more recent years, particularly for tapes received from an exploded improvised explosive device (IED) given the elemental contamination adhered to these adhesives from the explosives and associate environmental debris (e.g., dirt, rocks, metal, biological matter.)

Visual and microscopical examinations

The following parameters are examined and documented in each case: backing film and adhesive colors, film surface features, and tape width, with thickness (e.g., film, overall) as optional depending upon tape condition and other factors more specific to a given case.

FTIR, SEM/EDS, and PyGC/MS are carried out as stated for duct tape samples with respect to film and adhesive sample preparation and instrumental conditions. For XRF analysis, the adhesive is removed with solvent and the film is placed on a strip of carbon tape on a carbon planchet. The sample is then introduced to the XRF under vacuum conditions. The X-Ray tube uses a rhodium (Rh) target and the tube voltage and current are set at 40 kV/300 μ A, respectively. The acquisition time is set for 60 live seconds or more and a minimum number of 3 replicates are collected per sample using different areas of the sample to collect the data.

Packaging tapes

Less frequently than the two tape types already discussed, the FBI Laboratory also receives a variety of packaging tapes for analysis and comparison. As with electrical tapes, the number of physical and chemical features available for discrimination are limited and sourcing individual pieces to a manufacturer or distributor is not a current capability. While some discrimination is possible via FTIR and/or PyGC/MS, physical and optical characteristics provide the most variability for evaluation and comparison between tape pieces and to partially used tape rolls.

Visual and microscopical examinations

The physical characteristics most readily available for discrimination are film and adhesive colors differences. There have also been cases in which some items are fiber reinforced, either with yarn or glass fibers. For these types of tapes, the fiber reinforcement is evaluated by a fiber or geology forensic examiner. Other features of note are tape width and the nature of the film with respect to its tearability. Generally speaking, hand torn tapes are considered to be comprised of mono-oriented polypropylene (MOPP construction) and those that cannot be hand torn are recorded as bi-axially oriented PP (BOPP).

The film construction can be further investigated and documented using polarized light microscopy (PLM). Adjacent films are aligned along their respective machine edges while viewed under polarized light and rotated in order to observe any differences in film orientation/construction (17). In practice, this step is conducted by placing a small square of the film onto a glass microscope slide using its own adhesive and then placing a comparative piece of film right next to the first so that there is a small gap between them and they are aligned machine edge to machine edge.

In Figure 2, clear, colorless packaging tape films are oriented with adjacent machine edges and viewed under crossed poles (left side of figure). The difference in color is indicative of a difference in film thicknesses resulting in refractive index differences. When a full wave plate is inserted into the field of view within the PLM (right side of figure), the color differences are equally dramatic. This color difference demonstrates that although the films are constructed from the same polymer class (i.e., polypropylene), even slight differences in film thickness can significantly change the birefringence for each film.

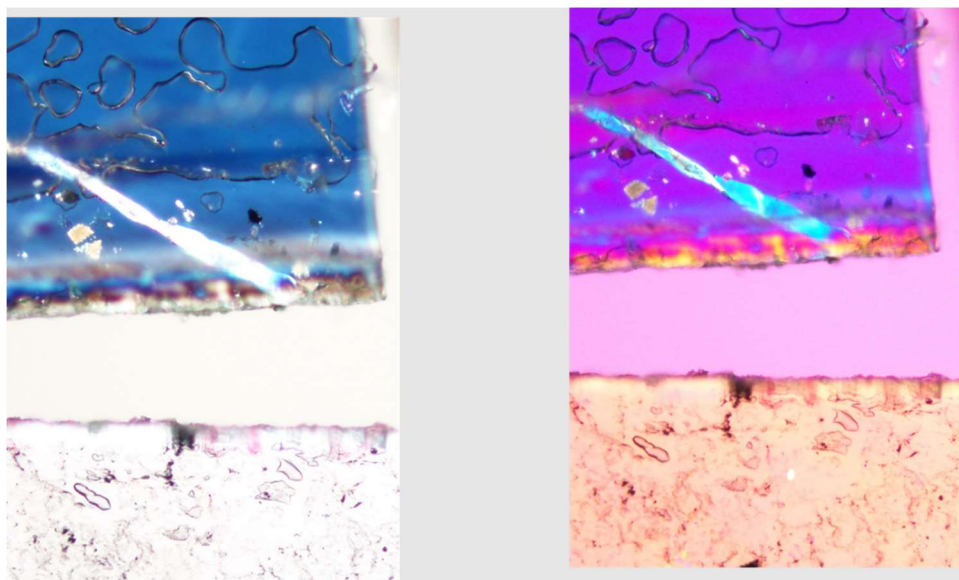


Figure 2. Polarized light microscopy images of two clear, colorless packaging tapes observed adjacent to one another. The left image depicts the films under crossed poles and the right image shows the film color changes when viewed with a full wave plate inserted into the field of view. Magnification is $\sim 50\times$.

Examples of instrumental data

Now that the general analytical scheme has been described, several examples of the utility of the selected instrumental techniques used for the commonly analyzed tape types can be provided for further elucidation of the inherent differences that are exploited in a comparison examination.

FTIR analyses

Interpretation of FTIR data for duct tape adhesives focuses on identifying peaks associated with commonly observed rubber formulations and pigments and/or filler components. As a result, peaks associated with talc (3676 , 1019 , 669 cm^{-1}), styrene (1601 , 759 , 699 , and the region above 3000 cm^{-1}), calcite and/or dolomite (1452 , 1376 , 879 cm^{-1}), isoprene (1452 , 1376 , 839 cm^{-1}), and butadiene (967 cm^{-1}), are commonly observed. Confirmation of these peak assignments require an orthogonal technique such as SEM/EDS or PyGC/MS, the latter being particularly helpful in identifying the presence of butadiene vs a tackifying resin at 967 cm^{-1} in an IR spectrum.

Figure 3 depicts the IR data for adhesives analyzed in two black-colored duct tapes in which the adhesives were gray (green and purple spectra) and black (blue and red spectra), respectively. The gray adhesive was typical of what is commonly observed in forensic casework, while the black adhesive was new to the FBI Laboratory.

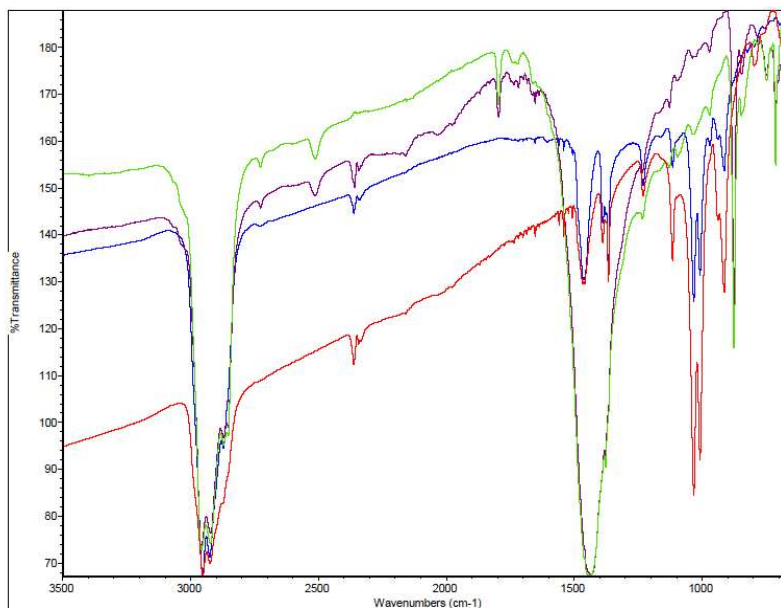


Figure 3. μ FTIR spectra depicting the adhesive formulations for black-film duct tapes: with black adhesives (blue and red spectra); and, with the more commonly submitted gray adhesive (green and purple spectra). Note that calcite peaks at ~ 1450 , ~ 875 , and ~ 712 cm^{-1} dominate the gray adhesive spectra.

Conversely, as seen in Figure 4, the spectra for the black-colored films of the same tapes depicted in Figure 3 revealed no differences between the films associated with the gray and black adhesives. As expected, polyethylene was observed on both sides of the film. The spectra also indicate the presence of an acrylic-based tie layer on the underside of the films, with peaks at ~ 1730 cm^{-1} (C=O) and $\sim 1150/1260$ cm^{-1} that are not present in the surface film (PE). These data confirm manual cross sectioning of the film that indicated a multi-layered film backing.

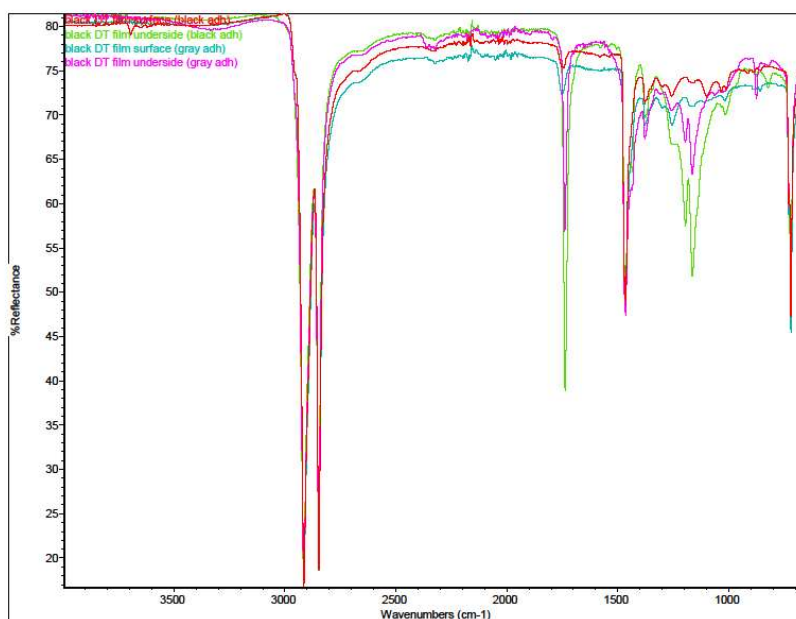


Figure 4. ATR spectra of both sides of the black-colored duct tape films, where the red and teal spectra constitute the surface side of the black and gray-colored adhesives, respectively, and the green and pink spectra represent the adhesive side of these same films.

Another examination that has always been part of the evaluation and comparison of duct tape has been examination of the scrim fibers in both the warp and weft directions. This examination was previously performed by forensic fiber examiners, but more detailed discussions with industry contacts indicated that the majority of duct tape scrim is comprised of cotton, polyester, or a cotton/poly blend, which makes incorporation into the totality of the duct tape examination process a logical progression. Therefore, this examination is now conducted concurrently with the other physical and IR examinations conducted on the various duct tape components.

Figure 5 illustrates the spectra collected for warp and weft fiber yarns on two black-colored duct tapes received in a recent case, in which the adhesive was also black. This data aligned with that collected for the black film/gray adhesive tapes also examined in the same case. Library searches against an internal FBI Laboratory fiber library indicate polyethylene terephthalate (PET) construction for both the warp and weft yarns. The weft yarns also contained cotton, but that is only examined and compared microscopically.

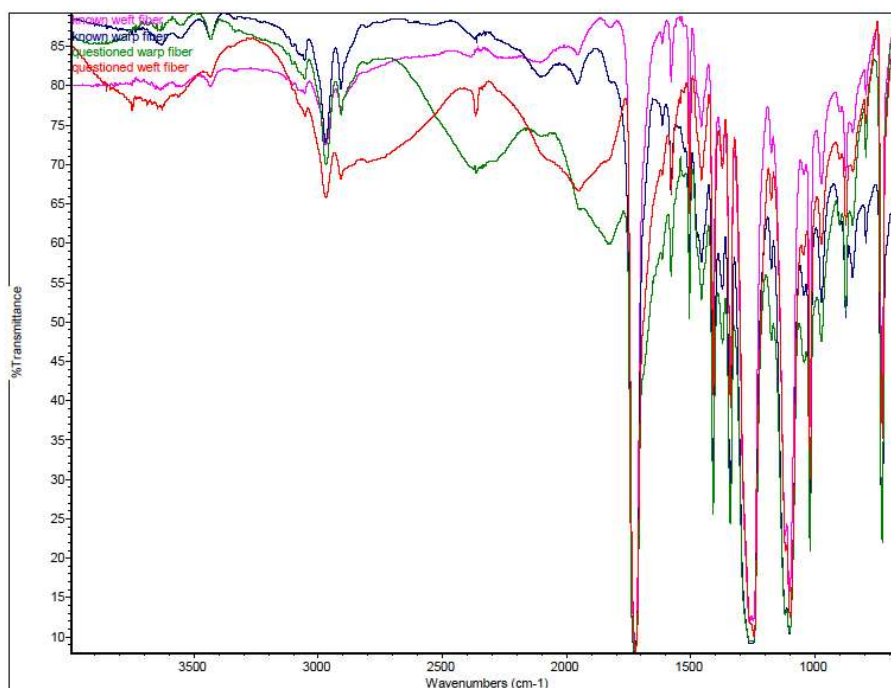


Figure 5. μ FTIR spectra depicting the warp and weft fiber compositions for two black duct tape samples: with warp yarn fibers (blue and green spectra) revealing slight differences from the weft yarn fibers (pink and red spectra) at $\sim 1250\text{ cm}^{-1}$ and $\sim 1050\text{ cm}^{-1}$.

Py-GC/MS

As mentioned previously, this technique is not routinely used in the FBI Laboratory for duct tape examinations. However, when FTIR analysis of an adhesive does not clearly elucidate the formulation as a natural rubber, SIS (styrene isoprene styrene), or SBS (styrene butadiene styrene) copolymer, an orthogonal technique is warranted. In one case, this question was more critical because there was no roll for comparison and therefore, bindings from a victim were being analyzed to generate investigative lead

information as to a potential manufacturer and/or product line. The absence of butadiene in the bindings would have called into question the apparent correlation observed between the suspected manufacturer's products within the FBI's internal duct tape reference collection and the information relayed by the manufacturer about their formulation. Py-GC/MS readily resolved the issue.

Figure 6 is an example of the pyrogram obtained for the duct tape adhesive depicted in Figure 3 for the black colored adhesive. Use of this technique confirmed the absence of common components observed in FTIR spectra for duct tape adhesives, namely isoprene, styrene, and/or butadiene. Instead, this formulation was revealed to contain several types of unsaturated compounds along with phenols and a sulfur-containing long chain ester. From a practical standpoint, the adhesion of this tape to a polymeric substrate was not noticeably different from that observed for the more commonly examined gray adhesives used by this same manufacturer; therefore, the physical and chemical differences in the adhesive formulations were striking from a forensic standpoint.

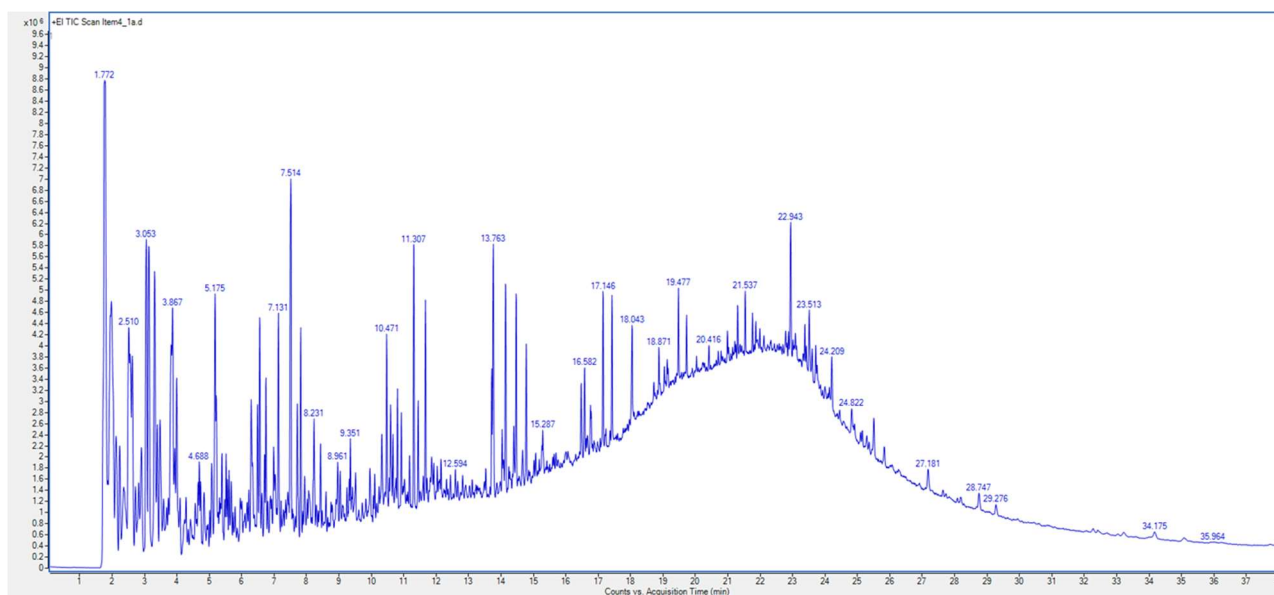


Figure 6. Py-GC/MS chromatogram of a black-colored duct tape adhesive featuring prominent peaks for butene (1.77 mins), heptene (7.51 mins), and amino (22.54 mins) compounds in striking contrast to what is typically observed for duct tape adhesive formulations received in forensic casework.

XRD

Most duct tapes observed in routine casework contain some combination of inorganic components used as colorants and/or fillers (e.g., kaolin, talc, titanium dioxide, calcite). Confirmation of one or more of these components is readily achieved by XRD and critical in the discrimination of otherwise undifferentiated duct tape samples.

When tape bindings in a particular case were analyzed by XRD, peaks at approximately $8.68^{\circ}2\theta$ (10.19 Å, d-spacing) and $10.40^{\circ}2\theta$ (8.51 Å, d-spacing) were observed on either side of the first talc peak in the diffraction pattern (see Figure 7). These peaks were clearly associated with a component other than kaolin or talc but attempts to identify them via searches of known diffraction cards were hampered by a lack of experiential knowledge in mineralogy. Discussions with several geologists and further investigation determined that these minerals could have been inclusions in the filler materials used by the tape manufacturer in the adhesive formulation. The identified minerals were tremolite and a form of

mica, both of which are common to North American mines where the inorganic raw materials (i.e., talc) likely originated (11).

Tremolite, $\text{Ca}_2\text{Mg}_5\text{Si}_8\text{O}_{22}(\text{OH})_2$, is one of six fibrous minerals in the asbestos family and is commonly found as a normal contaminant in industrial talc [$\text{Mg}_3\text{Si}_4\text{O}_{10}(\text{OH})_2$] ores (11,12). Neither the presence of the mica or tremolite affects quality control in the manufacture of the adhesive, and moreover, could be present in many adhesive formulations that utilize the fillers that contained these minerals. In fact, when the tape manufacturer was contacted to request additional information as to the source of their raw materials, the Material Data Sheet for their talc source listed tremolite as an active ingredient that could occur between 15-40 percent by weight.

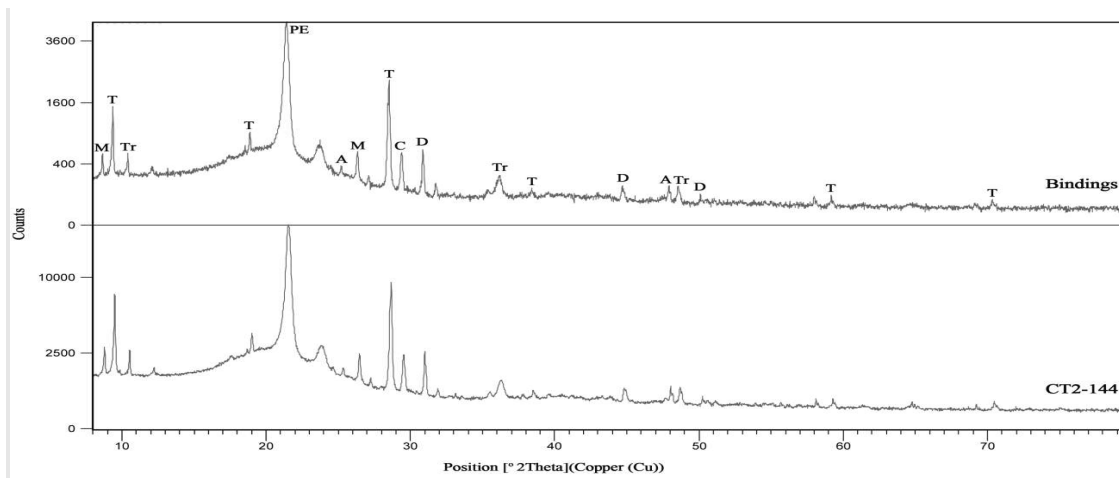


Figure 7. Diffraction patterns obtained for the duct tape binding (top) and CT2-144 (bottom), where peak labels are as follow: mica (M), talc (T), tremolite (Tr), polyethylene (PE), anatase (A), calcite (C), and dolomite (D).

This case was worked over a decade ago, and tremolite has not been observed in any duct tapes analyzed since then. It is recognized that it may not even be found in today's tapes produced by this same manufacturer. Further, it is quite realistic to assume that not even a small fraction of the types of tapes produced each day end up as forensic evidence in a case where sourcing or comparison is requested. Hence, the ability to keep current with manufacturing trends and tape producers is crucial to interpretation and context when forensic examiners write reports and testify in court.

SEM/EDS analyses

As stated previously, SEM/EDS was found to provide 87% discrimination of electrical tape film backings in a 2011 study by the FBI Laboratory (14). A separate study reported only 17% discrimination for adhesives (13). That is not to say that adhesive analysis is not of value (see Figure 8), where obvious elemental differences are displayed for a series of electrical tapes. However, these results were collected on newly purchased rolls of tape and do not represent the condition often encountered in casework adhesive samples.

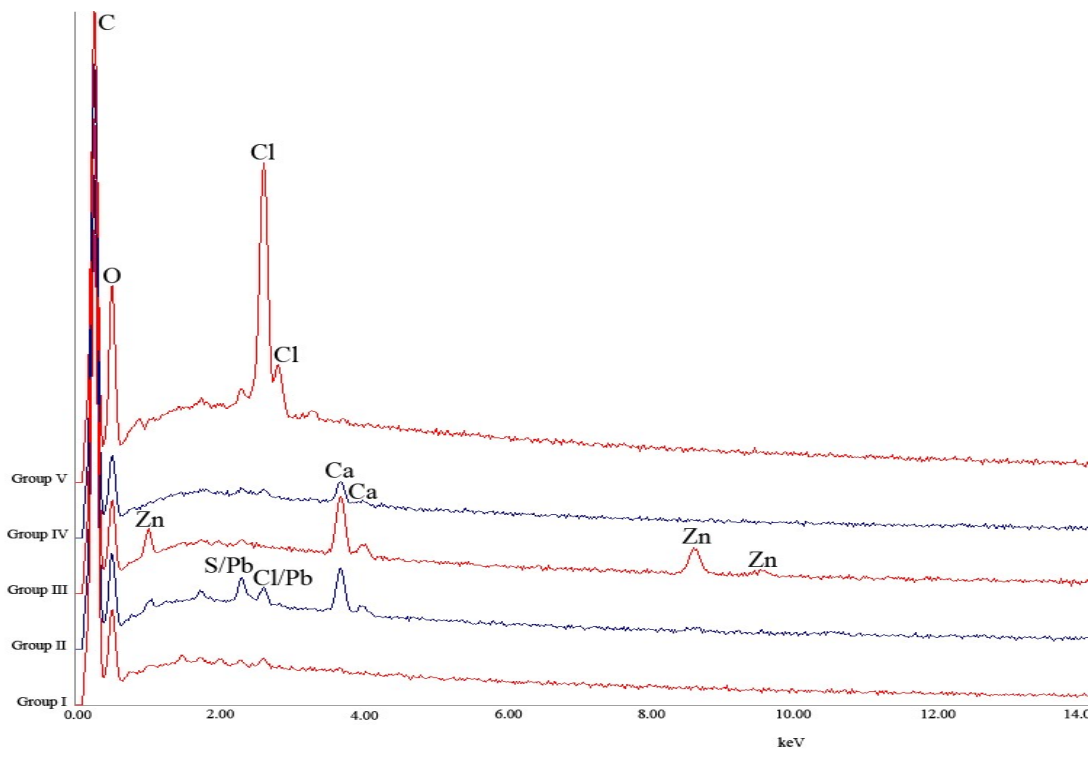


Figure 8. SEM/EDS spectra of the adhesive layer for a series of black electrical tapes, displayed using a square root scale and scaled on the background, to indicate differences that are be elucidated with this technique.

XRF analyses

After the 2011 studies were published, colleagues at West Virginia University requested access to the 90 electrical tapes used in the study in order to investigate whether they could validate a method using XRF for electrical tape backings that would improve the discrimination capability achieved by SEM/EDS. Several publications resulted from that work, with the first of the series demonstrating that XRF was worth exploring for casework (16). As a result of this work, XRF has been incorporated into the analytical suite of examinations used on electrical tape films.

Generally speaking, the absence or presence of a given element is used to discriminate between tapes. However, concentration differences are also evaluated where the differences are repeatable, sufficiently greater than the background signal-to-noise, and possibly confirmable by a complementary technique (e.g., FTIR, SEM). Figure 9 depicts the discrimination capability achieved for elements such as zinc (Zn) in two electrical tape film backings. Note that zinc was detected in both tapes, but at markedly difference intensities.

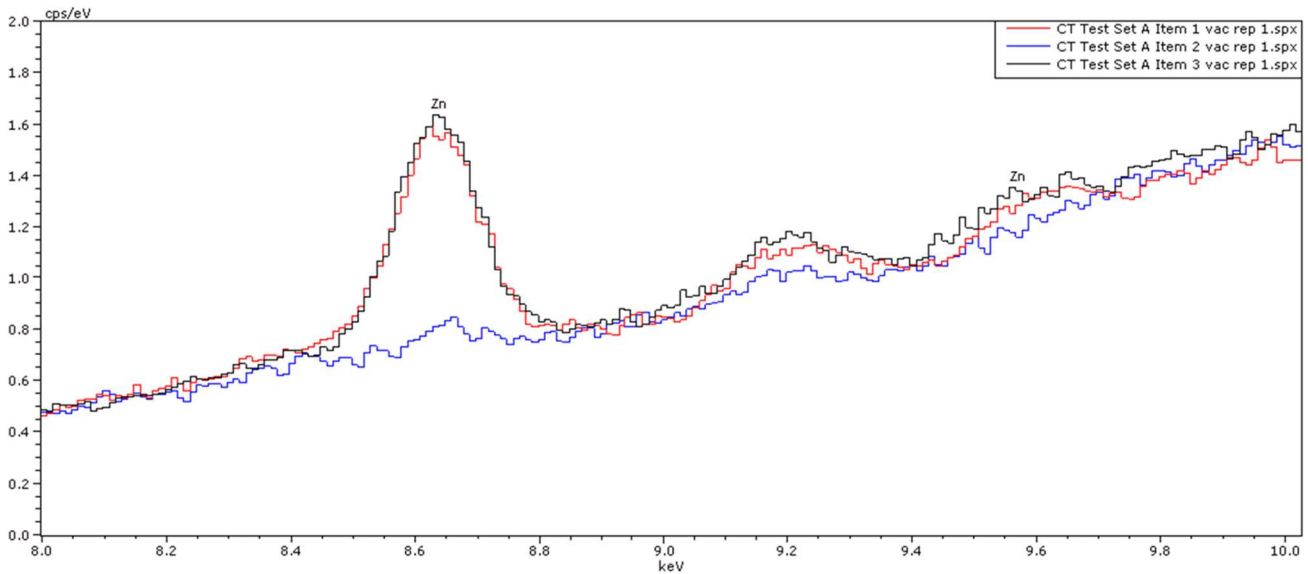


Figure 9. XRF spectra of the backing layer for black electrical tapes demonstrating discrimination capability between samples.

Conclusions

The most definitive approach to discriminating between the commonly encountered PSA tapes received in forensic laboratories is to conduct a comprehensive evaluation of the physical differences observed between tapes of the same type. Understanding the difference between intentional differences between products and incidental ones is a key means of developing an approach to further examination steps and eventually to writing a report and testifying to the opinions rendered.

Therefore, maintaining contact with industry representatives is vital to a forensic laboratory's ability to provide accurate and timely information to investigators as well as writing a final report of examination. The FBI Laboratory takes these relationships one step further with respect to maintaining a reference collection of duct and electrical tape products in order to refer to them as needed to address specific casework questions and to have a population of tapes to use for research purposes as needed.

Acknowledgments

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