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Duct Tape Sourcing Examinations: Developing Investigative Leads using Multiple Resources

ABSTRACT

The case example presented describes how the FBI Laboratory was able to develop lead information in a homicide investigation using resources such as the National Forensic Tape File (NFTF) database, industry contacts, and the internet. This case highlights the utility of employing a variety of analytical and reference-based resources to source manmade, mass-produced materials. It is also unusual in that the chemical analyses proved to be more discriminating than the totality of the physical characteristics of the tape. The adhesive formulation contained styrene-butadiene-styrene (SBS) as well as filler materials with impurities not previously identified in the authors' laboratory. Based on these characteristics of the tape, compelling investigative lead information was reported to the contributor using the described resources, most of which are readily available or can be developed by any laboratory system interested in conducting duct tape sourcing examinations.

Keywords: Duct Tapes, Sourcing, FTIR, SEM/EDS, XRD, Py-GC/MS, Reference Collections

INTRODUCTION

In early 2010, the FBI Laboratory received a request for assistance in a robbery/homicide investigation in which duct tape bindings were recovered. When latent print and DNA examinations did not further the investigation, the Chemistry Unit received the duct tape for analysis. The contributor requested the following information about the tape: its composition, overall length, the manner in which the pieces were separated (e.g., cut

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This is the FBI Laboratory Division's publication number 12-12. Names of commercial manufacturers are provided for identification only, and inclusion does not imply endorsement of the manufacturer, or its products or services, by the FBI. The views expressed are those of the authors and do not necessarily reflect the official policy or position of the FBI or the U.S. Government.

or torn) as well as manufacturer/product information.

Physical examinations of duct tape in the FBI Laboratory include assessment and documentation of features associated with all three components of the tape: the polymeric backing, the adhesive, and the fabric reinforcement. Characteristics such as the backing color, appearance, and layer structure; adhesive color; width and thickness; scrim /yarn count per inch in the warp (length) and fill (width) directions; fabric weave type (plain or weft insertion); and whether the scrim fluoresces under long wavelength UV light (~366 nm) are useful for classification or discrimination (1–4). These examinations may be conducted prior to or concurrent with end matching assessments.

Once physical examinations are completed, chemical analysis is conducted on the adhesive and backing layers using Fourier transform infrared spectroscopy (FTIR) (5,6) and scanning electron microscopy with energy dispersive X-ray spectroscopy (SEM/EDS) (7,8). X-ray diffractometry (XRD) is conducted on the intact specimen (1,2). On occasion, pyrolysis gas chromatography with mass spectral detection (Py-GC/MS) may also be used to characterize the adhesive portion of a duct tape. In a comparison examination in which samples remain undifferentiated at the conclusion of this sequence of analyses, the tapes are transferred to the Trace Evidence Unit for additional fabric examinations. Those examinations will not be discussed further as they are not necessary in a sourcing request where the objective is to associate the unknown sample to a type of tape or manufacturer/distributor.

For this case, instrumental analysis by XRD provided valuable information when previously unknown contaminants were observed and identified in the adhesive layer. Pyrolysis gas chromatography with mass spectral detection (Py-GC/MS) was also used to complement the FTIR data obtained for the rubber component of the adhesive (9, 10). Industry contacts were consulted to gather information on the distribution of the tape product that best correlated to the tape evidence in order to provide this information to the investigators.

MATERIALS AND METHODS

Visual and microscopical examinations

The following parameters were examined and documented: the backing and adhesive colors; the backing surface features, layer structure, and thickness; the tape width; and, the fabric characteristics of weave/knit pattern, yarn description (e.g., twisted), yarn composition (e.g., synthetic or cotton), yarn fluorescence, and scrim count.

For the backing and adhesive color, the observations were conducted with the unaided eye. Surface features of the backings were also observed unaided as well as with a stereomicroscope. To determine the layer structure of the backings, thin cross-sections were prepared and viewed with transmission light microscopy (5).

Width measurements were taken with a ruler to the nearest 0.5 mm. To measure the backing thickness, a portion of the adhesive was removed with hexane, the scrim was cut away with a scalpel, and the exposed backing layer was placed between the two faces of a digital micrometer (Mitutoyo America Corporation, Aurora, IL). A minimum of five areas were measured. The values were recorded to the nearest 0.05 mil (1 mil = 1/1000 inch), and then averaged.

To best visualize the fabric, the adhesive was removed with hexane. The weave/knit pattern and general yarn description were both observed by stereomicroscopy. Yarn fluorescence was observed under long wave UV light ($\lambda = 366$ nm).

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

FTIR

FTIR analysis of the adhesive was conducted by smearing a thin film of the adhesive on a single diamond window using a Continuum microscope equipped with a MCT/A detector (4000–650 cm^{-1}), coupled to a Nicolet 6700 Nexus FTIR E.S.P. spectrometer (Thermo Nicolet, Madison, WI). The spectra were collected using an approximate aperture size of 100 x 100 microns and 128 scans at 4 cm^{-1} resolution. Data collection and processing were conducted using OMNIC software (version 6.1, Thermo Nicolet).

Analysis of the backing layer was conducted by removing the adhesive with hexane and manually cutting away the scrim using a scalpel and tweezers. Then, each side of the backing was analyzed using a SmartOrbit diamond ATR accessory (Thermo Scientific, Madison, WI) attached to the same Nicolet 6700 Nexus FTIR E.S.P. spectrometer described previously. The spectra were collected using 32 scans at 4 cm^{-1} resolution.

SEM/EDS

The adhesive was sampled using a solid metal probe to tease up a portion of the adhesive into a ball that was smoothed into a thin film on a pyrolytic carbon planchet. A

small sample (~5mm) of the tape was cut with a No. 11 scalpel blade and affixed to the same carbon planchet using its own adhesive. A thin layer of carbon paint was then applied to the edges of the backing and the planchet was carbon coated by vacuum evaporation. Elemental analysis was conducted using a JEOL-6510 scanning electron microscope under high vacuum with a tungsten filament as the source (Peabody, MA). The EDAX energy dispersive spectrometer (Mahwah, NJ) was operated with a dead time of approximately 30% and live count time of 100 s. The working distance was approximately 12 mm, the take-off angle was approximately 30°, and the accelerating voltage was 25 kV. Spectral Library Identification and Classification Explorer (SLICE) software (Horiba Scientific, Edison, NJ) was used to process the data and conduct spectral searches of the tapes in the NFTF.

XRD

A portion of the intact tape was cut with a No. 11 scalpel blade to roughly the size of the silicon wafer (~ 1" diameter) used to mount it into a circular low background sample holder that was placed onto a rotating stage within the XRD. A PANalytical X'Pert Pro MPD X-ray diffractometer (Westborough, MA) was employed using Cu K α radiation, operated at 45kV and 40 mA, scanning continuously between 8 and 80° 2 θ with a step size of 0.0170° 2 θ , and a 10 mm beam mask. Total analysis time was approximately 8 minutes.

Py-GC/MS

The adhesive sample size was approximately 50 micrograms as needed to ensure an adequate signal response. Samples were placed in a quartz pyrolysis tube using quartz wool as a support medium to position each sample approximately 15 mm from the top of the tube. Pyrolysis was conducted using a CDS AS-5250 autosampler (CDS Analytical, Oxford, PA) coupled to an Agilent 6890 Gas Chromatograph (Agilent Technologies, San Jose, CA). Working conditions for the pyrolysis unit included a ramping step in which the initial temperature was set at 300 °C for 1 s, ramped at a rate of 20 °C/ms to 880 °C, and held at that temperature for 10 s, with an interface temperature of 321 °C. The column was a DB5-MS (30 m X 0.25 mm X 0.25 μ m). The carrier gas was helium at a purity of 99.99% and a flow rate of 0.7 mL/min. The GC was 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 5 minutes. The inlet was operated at 300 °C in 50:1 split mode and the transfer line temperature was set to 300 °C. Analytes were detected on a single quadrupole Agilent

5973 Mass Selective Detector with a dedicated electron impact ionization source run in full scan mode at a scan range of 34–650 m/z. The source temperature was 230 °C and the quadrupole temperature was 150 °C.

RESULTS AND DISCUSSION

The submitted tape pieces included two long strips of nominally 2” (50.8 mm) wide silver duct tape reportedly recovered from the ankle area. The pieces were approximately 34.5 inches and 67.5 inches in length, respectively, comprising about 8.5 feet of duct tape. The free ends consisted of both cut and torn edges and were of sufficient quality that they could be physically aligned to form one continuous piece of duct tape. Eight additional strips of physically consistent duct tape were submitted as tape from the head and wrists. These pieces varied in length from four to 28 inches, and also exhibited both torn and cut ends. The manner in which the cut tape pieces had been separated from the roll (e.g., knife, key, scissors) could not, however, be determined based on the appearance of the free ends.

When all ten pieces were assessed for possible end matches, three continuous pieces of duct tape resulted. The longest piece was slightly less than 12 feet and included the two pieces from the ankle surrounded by pieces from the head and/or wrists. The other two pieces measured approximately four feet and one foot, respectively.

The physical attributes of the tape are listed in Table 1.

Table 1. Recorded observations and measurements for the submitted duct tape.

Specimen	Backing appearance	Backing construction	Adhesive color	Weave pattern	Scrim count (per in. ²)	Yarn fluorescence (~366 nm)
Tape from ankle	silver film; smooth surface with scrim imprint on adh side	single layer	dark gray	simple/plain	w-19 f-7	w-no f-no
Tape from head/wrist	silver film; smooth surface with scrim imprint on adh side	single layer	dark gray	simple/plain	w-19 f-6	w-no f-no

Based on the results obtained for the samples listed in Table 1, one strip of the tape removed from the head and wrists was taken as representative of the bindings and further analyzed.

FTIR analyses

Interpretation of the FTIR data obtained for the adhesive included identification of 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}), which can be observed in the top half of Figure 1. The peaks at 1452, 1376, and 967 cm^{-1} can also be indicative of a tackifying resin. Confirmation of the peak assignments required an orthogonal technique as described in a subsequent section.

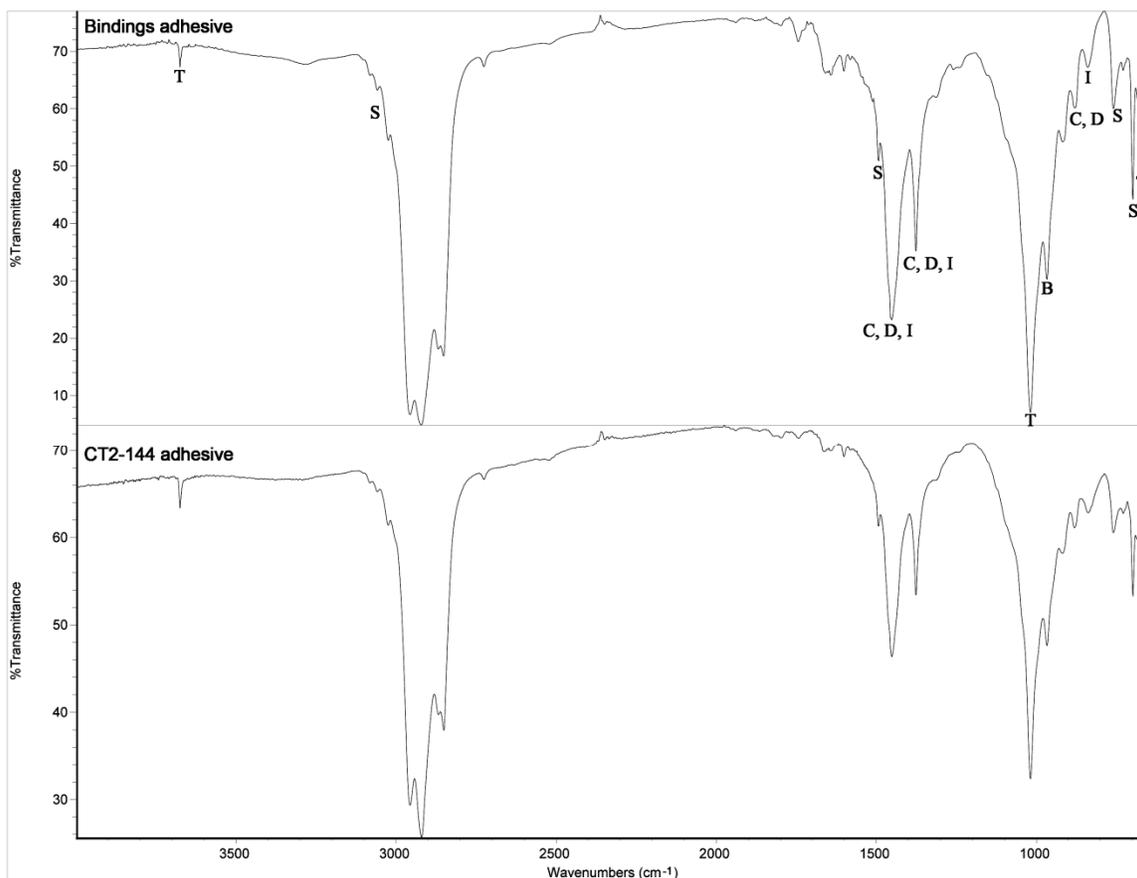


Figure 1. FTIR spectra depicting the adhesive formulations for the duct tape binding (top) and CT2-144 (bottom) with the peaks labeled for butadiene (B), calcite (C), dolomite (D), isoprene (I), styrene (S), and talc (T).

The backing spectra revealed that polyethylene was present on both sides of the film. The upper half of Figure 2 is the spectral overlay of the top (surface) and adhesive sides of the backing film for the bindings as analyzed by ATR.

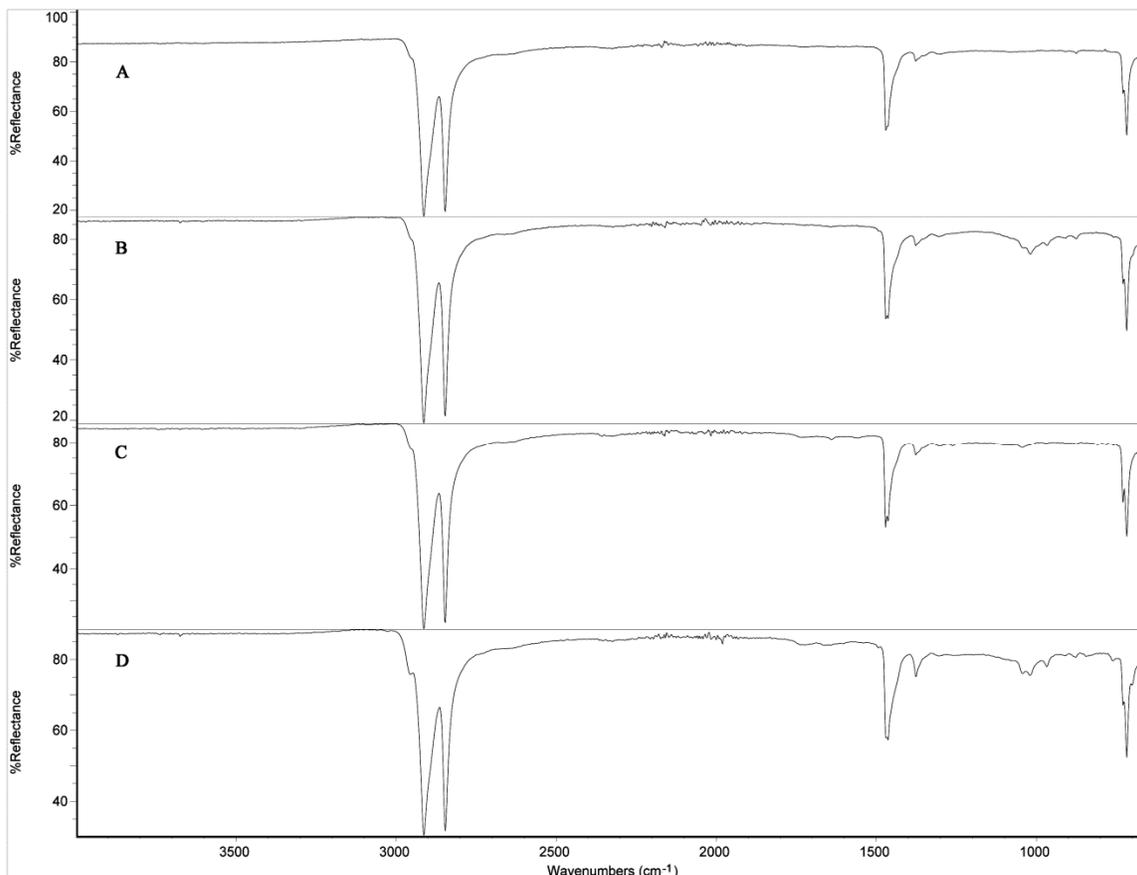


Figure 2. ATR spectra of both sides of the duct tape binding backing, where A is the surface side and B is the adhesive side of the CT2-144 backing, and where C is the surface side and D is the adhesive side of the bindings.

SEM/EDS analyses

Magnesium, aluminum, silicon, calcium, and titanium were observed in the adhesive layer with potassium and iron also possible. Aluminum was observed in the backing layer with silicon and calcium also likely, though not confirmed. The upper spectra in Figures 3 and 4, respectively, depict these results.

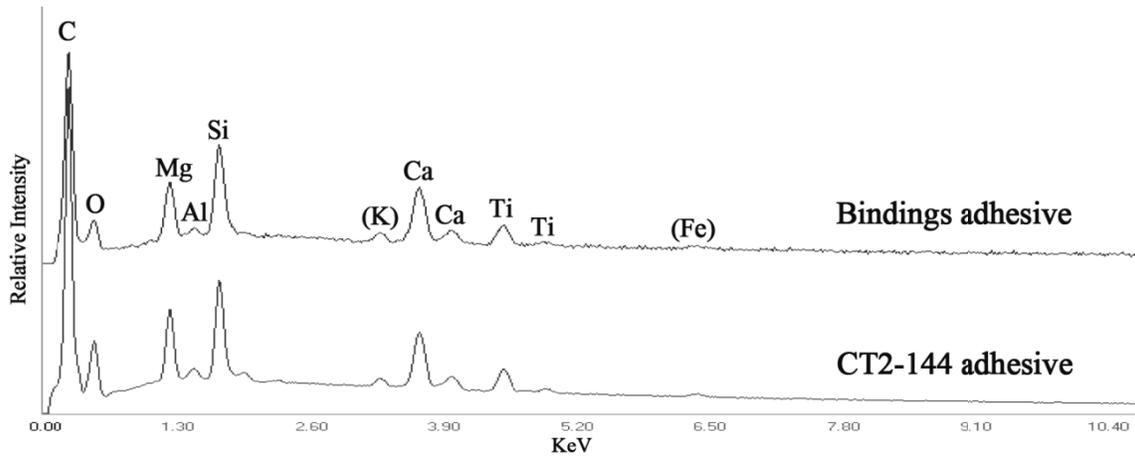


Figure 3. SEM/EDS spectra of the adhesive layer for the duct tape binding (top) and CT2-144 (bottom), displayed using a square root scale and scaled on the background.

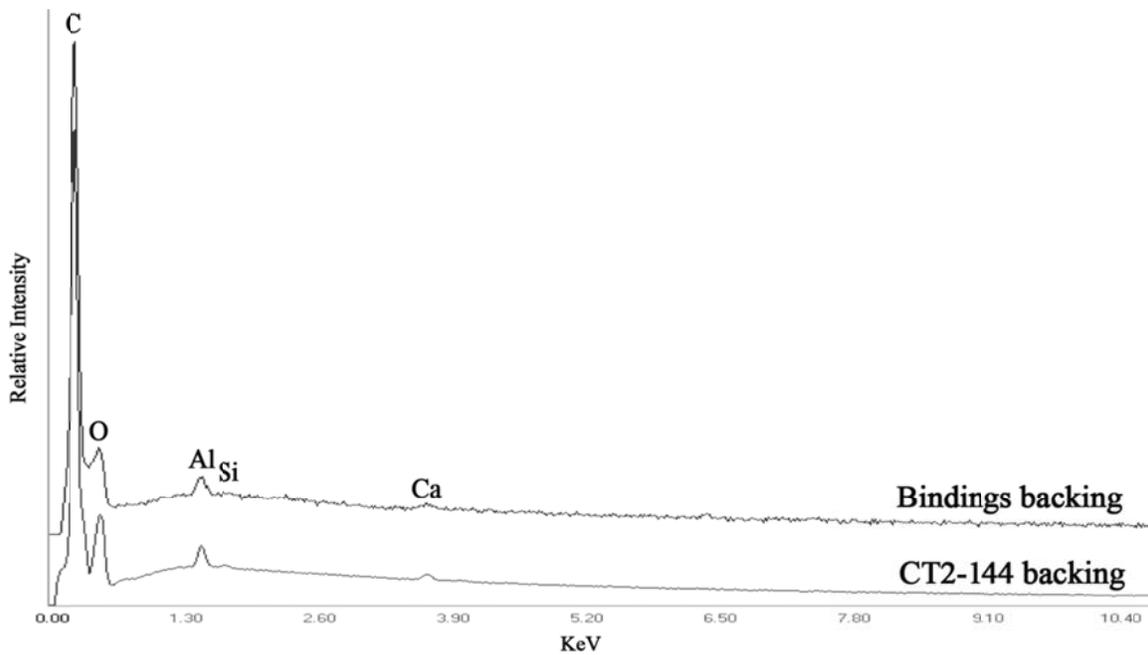


Figure 4. SEM/EDS spectra of the backing layer for the duct tape binding (top) and CT2-144 (bottom), displayed in square root scale and scaled on the background.

XRD analyses

XRD results supported the SEM/EDS data as shown in the upper half of Figure 5. Talc (magnesium silicate), anatase (titanium dioxide), calcite (calcium carbonate), and

dolomite (calcium magnesium carbonate) were all detected. These fillers are all commonly observed in duct tape adhesives. However, lesser known inorganic components were also observed.

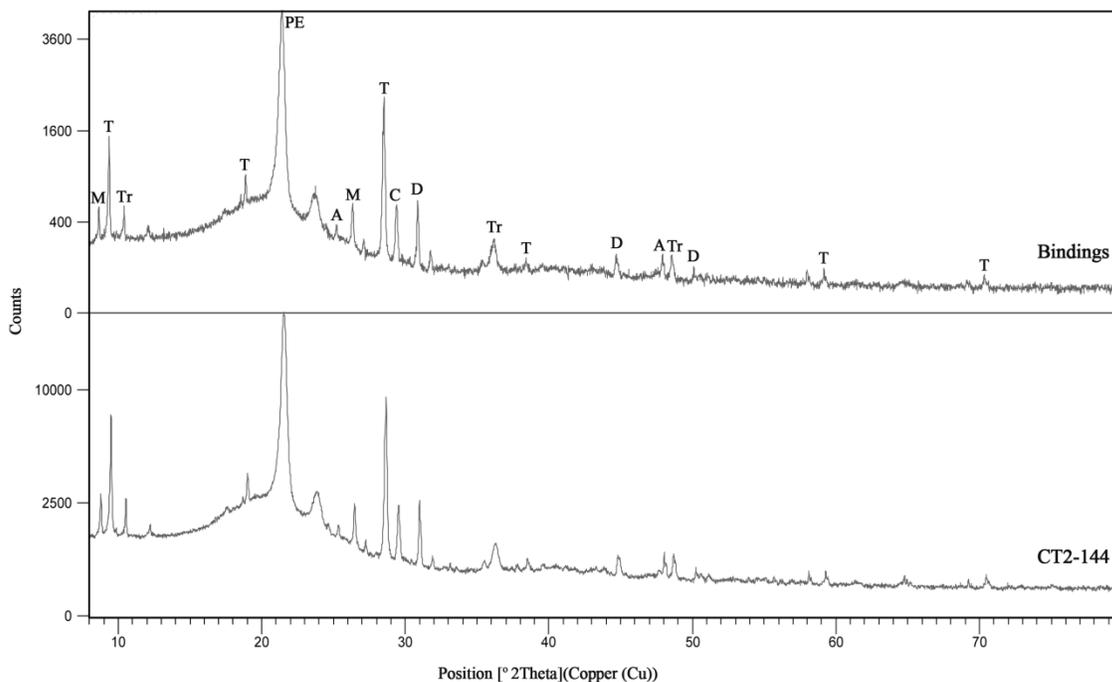


Figure 5. Diffraction patterns obtained for the duct tape binding (top) and CT2-144 (bottom), where, peak labels are as follow: mica (M), talc (T), tremolite (Tr), polyethylene (PE), anatase (A), calcite (C), and dolomite (D).

Spectral searches using SLICE

The FBI Laboratory maintains a collection of duct tape products known as the National Forensic Tape File (NFTF), which contains approximately 400 rolls purchased from local retailers, acquired from tours of manufacturing plants or via requests to specific companies when deficiencies within the collection are identified. One of the objectives in the creation and maintenance of the NFTF is to compile representative rolls of duct tape that are commonly available to the general public via retail outlets that market to contractors, individual consumers, industry-specific product needs (e.g., gaffer's tape), or small businesses. The NFTF is not a population database in that not every product or grade of tape produced by each manufacturer has been acquired. However, it does contain a majority of the types and grades of duct tapes that manufacturers produce or distribute within North America. Physical features such as the adhesive and backing

colors, images of the backing appearance and scrim pattern, and the scrim and yarn characteristics are compiled on these tapes and archived in SLICE.

Therefore, within SLICE, possible manufacturing sources for the duct tape binding were developed by conducting independent spectral searches for both the adhesive and backing layers using the acquired EDS data and the following physical characteristics with a “best fit” search criterion:

weave pattern: plain (basket)

warp yarn count: 19 ± 1

fill yarn count: 6 ± 1

For the backing search, six possible candidates were identified (3M, Henkel, Manco, Tyco, Tyco Polyken, and one with an unbranded core). All of these tapes were rejected as suitable candidates based on differences observed in film type (e.g., calendared versus blown/smooth), adhesive color, or yarn fluorescence within the scrim.

The adhesive search yielded the same six possible candidates that had been eliminated in the backing search.

Industry contact information

Requests for assistance were sent to several industry contacts at tape manufacturers in the United States as well as in Canada in an attempt to concentrate efforts on possible manufacturers. However, no specific leads were developed from the supplied information.

While this case was being worked, a set of six duct tapes recently received from a manufacturer specifically for inclusion in the NFTF collection had been analyzed, but not yet entered into SLICE. One of the authors (AM) manages the NFTF. During discussions of the data collected for the tape bindings, she recognized that many of the parameters were in general agreement with one of these recently acquired tapes (labeled in the NFTF as CT2-144).

The tape had been acquired from a Canadian manufacturer. This company had been included in the initial correspondence with industry contacts even though it was not identified as a possible source based on the collected analytical data. A company representative had indicated that the binding adhesive was similar to their products in that a styrene-butadiene-styrene (SBS) block copolymer was used. However, the

company also stated they did not add dolomite to their adhesives, which was suspected to be a component in the bindings after FTIR and SEM/EDS analyses. Ultimately, dolomite was confirmed via XRD, both in the bindings as well as in a product from this company, CT2-144.

All of the physical characteristics listed in Table 1 for the bindings were comparable to CT2-144. Thickness measurements and backing layer construction were also consistent. As depicted in Figures 1 through 5, the chemical analyses were also in good agreement such that the Canadian manufacturer and more specifically, the CT2-144 product type was viable as a possible source of the tape bindings.

Having identified CT2-144 as a possible candidate, a spectral library inclusive of all of the tapes in the NTF, to include those recently received from the Canadian manufacturer, was created in Omnic and used to search for adhesive formulations comparable to the duct tape bindings. The top five hits were all recently acquired products by the manufacturer of CT2-144. Figure 1 displays the agreement observed for the adhesive formulations of the duct tape bindings compared to CT2-144, while Figure 2 displays the agreement for the backing formulations.

Spectral overlays of the SEM/EDS data for the respective adhesive and backing layers indicated similar elemental profiles for the two tapes (Figures 3 and 4). A shorter count time for the case sample in comparison to the reference sample explains the difference in the background signal.

Py-GC/MS

This technique is not routinely used in the authors' laboratory for duct tape examinations. However, FTIR analysis of the adhesive did not clearly elucidate the formulation as a SIS (styrene isoprene styrene) or SBS (styrene butadiene styrene) copolymer, a question made more critical by the Canadian duct tape manufacturer's named use of the latter in their product line. The absence of butadiene in the bindings would have called into question the apparent correlation observed between the manufacturer's products or the information relayed to the authors about their formulation. Py-GC/MS readily resolved the issue.

Figure 6 is an overlay of the pyrograms obtained for the adhesives of the tape bindings as well as CT2-144. Use of this technique identified the presence of butadiene, isoprene, and styrene in the formulation and strengthened support for the producer of

CT2-144 as the manufacturer for the bindings as well. Butadiene was detected in the form of the monomer (in order of abundance: masses 39 and 54 amu) and dimer, 4-ethenyl cyclohexene (54, 79, 66, 39, 93, 108 amu). Isoprene was observed as the monomer (67, 53, 39 amu) and dimer, D-limonene (68, 93, 79, 121, 136 amu). Styrene was confirmed by the presence of the monomer (104, 78 amu), alpha-methylstyrene (118, 103, 78 amu), and the trimer (91, 117, 207, 312 amu).

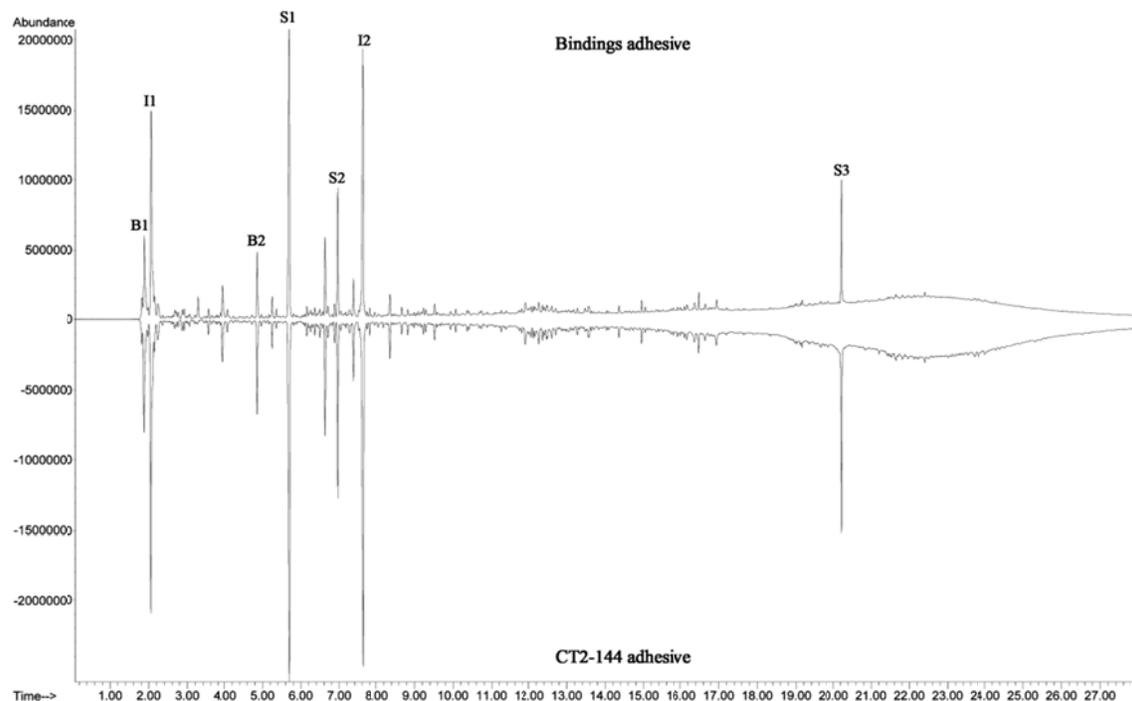


Figure 6. Py-GC/MS chromatograms of the duct tape binding adhesive (top) in comparison to the CT2-144 adhesive (inverted). Peak labels are as follow: 1,3 butadiene (B1), isoprene monomer (I1), 4-ethenyl cyclohexene/butadiene dimer (B2), styrene monomer (S1), alpha methyl styrene (S2), D-limonene/isoprene dimer (I2), and styrene trimer (S3).

XRD

Most duct tapes contain some combination of routinely observed 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 also critical in the discrimination of otherwise undifferentiated duct tape samples.

When the bindings in this case were analyzed by XRD, peaks that are apparent in Figure 5 at approximately $8.68^{\circ}2\theta$ (10.19 \AA , d-spacing) and $10.40^{\circ}2\theta$ (8.51 \AA , d-spacing)

were observed on either side of the first talc peak in the diffraction pattern. These peaks were clearly associated with a component other than kaolin or talc, but attempts to identify them via searches of known diffraction cards was 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).

Mica is a broad characterization of a group of aluminosilicates with a sheet or plate-like structure. The specific form of mica could be muscovite, $KAl_2(Si_3Al)O_{10}(OH)_2$, or phlogopite, $KMg_3(Si_3Al)O_{10}(OH)_2$, both of which are commercially important in a variety of industries. In its dry-ground form at a fine particle size, mica is commonly used in the paint industry as an extender pigment to facilitate suspension and adhesion to the substrate, reduce chalking, increase resistance to water penetration and weathering, and prevent fouling (11).

Tremolite, $Ca_2Mg_5Si_8O_{22}(OH)_2$, is one of six fibrous minerals in the asbestos family and is commonly found as a normal contaminant in industrial talc [$Mg_3Si_4O_{10}(OH)_2$] 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 MSDS for their talc source listed tremolite as an active ingredient that could occur between 15–40 percent by weight.

Thus, the peaks observed in the XRD data for mica and tremolite are noteworthy as additional chemical markers that could aid in discrimination in future casework. In this case, they served to further associate the tape bindings with this manufacturer's product line.

Distribution of CT2-144

Given the correspondence between the physical and chemical characteristics of the duct tape bindings with CT2-144, the manufacturer was considered to have been identified. CT2-144 is marketed as a general purpose grade tape at 8.5 mils, which was in good agreement with the average measured value obtained for CT2-144 in the authors'

laboratory, 8.6 mils (n=5). Film thickness measurements for CT2-144 and the bindings were also measured five times each and the averages were 5.0 and 5.1, respectively. A chemically indistinguishable product by the same manufacturer (CT2-143) was also evaluated. This tape was marketed as an economy grade product (i.e., 7.0 mils overall thickness). The film thickness was measured at 3.9 mils, much thinner (i.e., of cheaper construction) and therefore not comparable to the bindings. This parameter was the only difference observed between the two rolls obtained from the manufacturer (CT2-143 and CT2-144), and the only difference cited in their product literature. However, this measurement difference was sufficient to target the general purpose grade tape, CT2-144, as the appropriate product designation from this manufacturer for the bindings.

The next question to be addressed was distribution of this particular product, and for that purpose, the region of the country where the crime occurred was provided to the manufacturer. In a two state radius, the number of customers for the CT2-144 product was one, a company specializing in the supply of an array of products to manufacturers in a variety of industries. This intermediate supplier was contacted and subsequently provided a list of eight companies that they supplied with the CT2-144 product. The list included a cabinetry maker, an adhesives manufacturer for the wood working industry, a manufacturer of storage tanks, a non-profit organization that manufactured office supplies, a large scale multi-disciplinary consumable products company, a funeral home chain, a food manufacturer, and a consumer product company. Several of these customers used the tape product at multiple plants or sites within their companies, but all were located within a specific area of a state adjacent to the one in which the crime occurred (approximately two hours drive from the crime scene).

As of this writing, local law enforcement was seeking the assistance of the companies' respective human resources divisions to attempt to locate a nexus between their personnel and the crime.

CONCLUSIONS

This study demonstrates that duct tape sourcing examinations can yield descriptive investigative lead information due to the variety of characteristics inherent within the manufacturing process, even for multiple products from a single manufacturer. While it would be impractical for every forensic laboratory to invest in the establishment of a physical duct tape reference collection, even a small searchable library of reference

chemical data and physical characteristics could be of great benefit. Additionally, although the NFTF cannot be extended to other forensic laboratories because it is a physical collection of tape rolls, the FBI Laboratory has the capability to provide similar investigative lead information to forensic tape examiners in the law enforcement community if the relevant physical and chemical parameters are shared.

Increasingly, duct tape manufacturers are consolidating multiple brand names under a single manufacturer or distributor. Searches of company marketing media and website information have also proven useful in gaining information and can be used to identify a technical representative who might provide more detailed insight into manufacturing and distribution processes.

ACKNOWLEDGEMENTS

The authors thank the industry contacts that graciously provided technical information as well as Ms. Maureen Bottrell, Dr. Philip Candela, and Dr. Bruce Rothman for their willingness to provide insight into the mineralogical information presented in the text. Sincere gratitude is also extended to the reviewers who provided helpful feedback to improve the content of this manuscript.

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