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PAPER

CRIMINALISTICS

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Analysis and Discrimination of Electrical Tapes: Part I. Adhesives*,†

ABSTRACT: This study involved the comparative analysis and discrimination of 90 electrical tape adhesives. The objectives included the evaluation of the ability of individual techniques to discriminate samples and the assessment of the ability of the techniques combined to distinguish samples. The techniques utilized were stereomicroscopy, Fourier transform infrared spectroscopy (FTIR), pyrolysis-gas chromatography/mass spectrometry (Py-GC/MS), and scanning electron microscopy/energy dispersive spectroscopy (SEM/EDS). Stereomicroscopy, to assess adhesive colors of black, clear/colorless, and clear adhesives with brown tint, resulted in a discrimination of 53%. FTIR analysis yielded eight distinct groups with a discrimination of 67%. Py-GC/MS analysis resulted in 16 groups with a discrimination of 83%. These analyses confirmed and further subdivided the FTIR groups. SEM/EDS resulted in five separate groups at 17% discrimination, increasing the overall discrimination to above 85%.

KEYWORDS: forensic science, trace evidence, electrical tape, adhesive, discrimination, stereomicroscopy, Fourier transform infrared spectroscopy, pyrolysis-gas chromatography/mass spectrometry, scanning electron microscopy/energy dispersive spectroscopy

Electrical tapes are often submitted to crime laboratories as evidence associated with improvised explosive devices or other violent criminal activities. Comparative electrical tape examinations are performed to explore the possibility of an evidentiary link between a suspect and a crime or between different items or scenes.

An electrical tape sample consists of two basic components: the backing and the pressure sensitive adhesive. Although electrical tapes in the United States are available in a wide variety of backing colors, black is by far the most common (1). Most backings consist of polyvinyl chloride (PVC), but polyethylene and polypropylene can also be used. In PVC-backed tapes, aromatic or aliphatic plasticizers are also added to impart flexibility to the tape. Stabilizers, fillers, and flame retardants may also be present in a plasticized PVC backing. Adhesives are mixtures of multiple components: an elastomer base, tackifying resins, aromatic and/or aliphatic plasticizers, antioxidants, flame retardants, and sometimes fillers. The elastomer frequently consists of polyisoprene, polyisobutylene, a styrene/isoprene copolymer, a styrene/butadiene copolymer, or an acrylic.

Within the FBI Laboratory, submitted samples are first evaluated by visual and microscopical means to assess physical characteristics, such as adhesive color; backing color, degree of gloss, and surface texture and features; width; and thickness.

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The free ends of physically indistinguishable tape pieces are then evaluated for possible reconstructions. In 1986, Agron and Schecter (2) demonstrated that physical matches can be used to reconstruct torn pieces of electrical tape. However, due in part to the amorphous nature of such an easily deformable material, there is a possibility of incorrectly matching two free ends of tape (3). Chemical analysis, on the other hand, provides an independent means of discrimination regardless of the existence of an end match. Further, end matching of an amorphous material in the absence of an orthogonal means of comparing samples may be subject to challenge in court. Therefore, the FBI Laboratory protocol dictates that chemical analysis be performed regardless of whether tape pieces can be reconstructed. It should be noted that FBI protocols do not prohibit an examiner from reporting end matches of solid, rigid materials (e.g., metal, glass, paint) without subsequent chemical analysis.

Regardless of the presence or absence of an end match, when tape pieces are determined to be indistinguishable following visual and microscopical examinations, chemical composition of the tape adhesive and backing is evaluated. Current FBI protocol calls first for chemical analysis via Fourier transform infrared spectroscopy (FTIR) with a microscope attachment, followed by scanning electron microscopy/energy dispersive spectroscopy (SEM/EDS) and pyrolysis-gas chromatography/mass spectrometry (Py-GC/MS).

According to Johnston and Serra (1), infrared analysis of a vinyl electrical tape adhesive is the easiest way to identify the supplier. FTIR analysis can provide information regarding the elastomer and polymeric materials used to formulate both a tape's adhesive and backing, as well as some information about the plasticizers, flame retardants, and fillers that are present. Multi-component adhesives, those consisting of natural or synthetic rubber and resin, are manufactured on-site by each manufacturer using a range of chemicals in differing ratios. This variability results in infrared spectra that are easily differentiated. Acrylic adhesives may be produced on-site but are often purchased from commercial sources. Although distinct spectra may result, FTIR analysis of acrylic adhesives cannot solely be used to determine the manufacturer. Due to the many possible combinations of components in an adhesive, more discrimination is expected from FTIR analysis of the adhesive as compared to the backing (4). Goodpaster et al. (5) confirmed this expectation for tapes with black adhesives when it was found that the overall discrimination of adhesives by FTIR analysis was more reliable than FTIR or SEM/EDS analysis of the backings. For tapes with clear adhesives, the discriminating power of FTIR analysis was about the same for adhesives as for backings.

Experience at the authors' laboratory has shown that because of the multiple components within adhesives, a significant amount of FTIR peak overlap can occur, making spectral interpretation difficult. Therefore, in most instances the individual chemical constituents are categorized into general classes rather than identified.

For samples that cannot be differentiated by FTIR examination, SEM/EDS is then performed to compare elemental composition. Recently, Goodpaster et al. (6) evaluated SEM/EDS for the analysis of vinyl electrical tape backings and found that the relative elemental compositions varied greatly between brands and sometimes could narrow the range of the years of manufacture. An exception occurred when two premium brands of 3M tape (Super 88 and Super 33+) had indistinguishable elemental compositions. While a single-blind study resulted in a correct association of questioned samples to known exemplars, the authors did not analyze the adhesives within their sample set. The authors noted that 3M was the only manufacturer using black filler in its electrical tape adhesives.

As a final means of elucidating differences between electrical tape samples, the FBI Laboratory performs Py-GC/MS on both adhesive and backing components. This technique breaks the organic components down, separates them, and provides more conclusive information as to the identity of the chemical constituents. As a result, Py-GC/MS is particularly useful in identifying the elastomer(s), the type(s) of plasticizers, and any other organic additives present. Williams and Munson (7) analyzed 30 electrical tapes (adhesive and backing together) by Py-GC and reported both within-roll homogeneity and between-roll discrimination. Twenty-six of the pyrograms were unique based on the presence or absence of certain peaks. Of the remaining four samples, one indistinguishable pair reportedly came from the same manufacturer. The other indistinguishable pair consisted of tapes with different brand names. Maynard et al. (8) analyzed packaging tapes and office-type tapes by FTIR and Py-GC/MS, among other techniques, and concluded that Py-GC/MS was slightly more discriminating than FTIR. However, the authors in that study cautioned that because of the destruction of the sample and relative length of analysis time, Py-GC/MS should be considered a final step in the analytical scheme for adhesive tapes.

Keto (9) analyzed six brands of black electrical tape and found less intra-roll variations than inter-roll variations. Furthermore, each brand was differentiated from the others based on any one of three techniques (microscopy, infrared analysis, and X-ray fluorescence). The author concluded that a combination of multiple techniques could lead to a very high degree of discrimination between tapes.

This publication is the first part of a FBI Laboratory study on the analysis and discrimination of electrical tape samples. The focus of this work involved the adhesive component of electrical tapes. While numerous studies have been performed and referenced, none of them have investigated the relative discrimination potential of analytical techniques on the adhesive component of an electrical tape sample set of this size. Further, with the exception of Maynard et al. (8), recent publications on pressure-sensitive tape adhesives do not address the discrimination power of Py-GC/MS.

In this study, the ability of the individual techniques to discriminate adhesive samples is evaluated as well as the ability of the techniques combined. Forthcoming work will describe the analysis and discrimination of the backings, including an assessment of the cumulative discrimination of tape samples.

Materials and Methods

Tape Collection

This study involved the analysis of 90 black electrical tape samples utilizing current FBI Laboratory protocols. Most of the tapes were purchased by FBI personnel at discount stores or home-improvement retailers, were marketed as general purpose or economy grade, and originated from Taiwan, China, or the United States. Therefore, the sample set represented tapes that could be easily obtained by consumers and would be comparable to casework evidence submitted to forensic laboratories. Table 1 is a summary of the products that were evaluated in this work. For a number of samples, the manufacturer was not listed on the packaging. Further, it is common practice for tapes to be purchased from a manufacturer and distributed under one or more private labels, such as Ace Hardware, Duck®, and Frost King® (5,6). While Underwriters Laboratories (UL) numbers can be used to determine a tape manufacturer, they were not available for all tapes in the collection. As manufacturer information was not available for all samples nor was the sample set selected to be representative of all manufacturers, the sample set is not intended to be used to provide sourcing information regarding a tape sample of unknown origin.

Microscopical Examinations

The color of the adhesive was evaluated using a stereomicroscope following manual separation of the adhesive from its backing.

FTIR

All samples were smeared on a KBr disc or a diamond window of a compression cell and analyzed using a Continuum microscope attached to a Nicolet Nexus 670 FTIR E.S.P. spectrometer (a division of Thermo Nicolet, Madison, WI). An MCT/A (Mercury Cadmium Tellurium/Type A) detector was used (4000–650 cm^{-1}) at a resolution of 4 cm^{-1}. The number of sample and background scans was 128 each. Although many samples were analyzed once, replicate analyses were conducted on numerous samples to confirm reproducibility of the data.

Py-GC/MS

All samples were placed in a quartz pyrolysis tube in approximately the same position (*c.* 15 mm from the open end) and pyrolyzed using a CDS Analytical AS-2500 pyrolysis autosampler (Oxford, PA). The initial temperature was set at 300°C for 1 sec, ramped at a rate of 20°C/ms to 880°C, and then held at that temperature for 10 sec. The temperature of the interface was 321°C. The pyrolysis unit was coupled to an Agilent 6890 Gas Chromatograph (Agilent Technologies, Wilmington, DE). The GC column was a DB5-MS, 30 m, 0.25 mm i.d., with a 0.25 μm film thickness. The carrier gas was helium and had a purity of 99.99%. The GC was operated at an initial temperature of 50°C for 2 min, ramped at a rate of 13°C/min to 325°C, and held for 5 min. The GC inlet was also operated in 50:1 split mode at 300°C, with a split flow of 34.9 mL/min. The mass spectrometer was a single quadrupole

TABLE 1—*Product information for sample set.*

Sample Roll	Brand Name	Product	UL	CSA Reference	Country of Origin
1	Marcy Enterprises, Inc.	MA 750	111K		Taiwan
2	Advance®	AT7, BS3924, 31/90Tp			England
3	Work Saver™, a Royal Tools brand	Stock no. 55, 5 color P.V.C Tape Assortment			China
4	tesa tape, inc.	40201, No. 111 E52811A	362K		Taiwan
5	Tape It, Inc.	E-60	119K		Taiwan
6	Qualpack®	1346, 6-Color			China
7	Marcy Enterprises, Inc.	MA 750	111K		Taiwan
8	Manco®	200 MPH, AE-66	590J	LR31971	Taiwan
9	Archer® (Packaged for Radio Shack)	64-2349	590J		Taiwan
10	3M Scotch™	Super 88, 054007-06143	539H	LR48769	U.S.A.
11	3M Scotch™	Super 33+, 10414 NA	539H	LR48769	U.S.A.
12	3M Scotch™	Super 33+, 10455 NA	539H	LR48769	U.S.A.
13	3M Scotch™	Super 33+	539H	LR48769	U.S.A.
14	Frost King®	ET60	206T		Taiwan
15	3M Scotch™	Super 33+, 10455 NA	539H	LR48769	U.S.A.
16	3M	Tartan™ 1710, part no. 054007 49656	539H	LR48769	U.S.A.
17	3M Scotch™	Super 88 054007-06143	539H	LR48769	U.S.A.
18	3M Scotch™	Super 33+, Cat. 195NA	539H	LR48769	U.S.A.
19	3M Scotch™	Super 33+, Cat. 194NA	539H	LR48769	U.S.A.
20	3M Scotch™	Super 33+, 10414 NA	539H	LR48769	U.S.A.
21	Manco®	P-66	590J	LR31971	Taiwan
22	Manco®	667 Pro Series™	590J	LR31971	Taiwan
23	3M Scotch™	Super 88, 054007-06143	539H	LR48769	U.S.A.
24	3M Scotch™	Super 88, 054007-06143	539H	LR48769	U.S.A.
25	3M Scotch™	Super 33+, 054007-06132	539H	LR48769	U.S.A.
26	3M Scotch™	Super 33+, 054007-06132	539H	LR48769	U.S.A.
27	3M	Tartan™ 1710, part no. 054007 49656	539H	LR48769	U.S.A.
28	3M	Tartan™ 1710, part no. 054007 49656	539H	LR48769	U.S.A.
29	3M	Temflex™,1700, 54007-69764	539H	LR48769	U.S.A.
30	3M	Temflex™,1700, 54007-69764	539H	LR48769	U.S.A.
31	Regal®	Model ET-6			Taiwan
32	GE	GE2472-3DD	206T		Taiwan
33	3M Scotch™	Cat. 190			U.S.A.
34	3M	Tartan™ 1710, part no. 54007-49656	539H	LR48769	U.S.A.
35	Frost King®	ET60	206T		Taiwan
36	3M	Tartan™ 1710, part no. 49656	539H		U.S.A.
37	National	All-Purpose Grade	206T		Taiwan
38	Manco®	P-660	590J	LR31971	Taiwan
39	3M Scotch™	Super 33+, 3744NA	539H	LR48769	U.S.A.
40	3M	Tartan™ 1710, part no. 054007 49656	539H	LR48769	U.S.A.
41	3M Scotch™	Super 33+, 200NA	539H	LR48769	U.S.A.
42	National	All-Purpose	362K		Taiwan
43	3M	Tartan™ 1710, part no. 054007 49656	539H	LR48769	U.S.A.
44	3M	Tartan™ 1710, part no. 054007 49656	539H	LR48769	U.S.A.
45	Calterm®	49605	590J		Taiwan
46	Manco®	P-20	590J	LR31971	Taiwan
47	3M	Tartan™ 1710, part no. 054007 49656	539H	LR48769	U.S.A.
48	Tape-It	36-T			U.S.A.
49	Tape-It	36-T			U.S.A.
50	General Electric	GE2472-31D	206T		Taiwan
51	National	No. 101, E52811A	362K	LR32044	Taiwan
52	Frost King®	ET60FR	906B		U.S.A.
53	National	No. 101, E52811A	362K	LR32044	Taiwan
54	3M Scotch™	Super 33+ on core, 03404NA on packaging	539H/5364	LR48769	U.S.A.
55	Manco®	1219-60	590J	LR31971	Taiwan
56	Victor Automotive Products, Thermoflex	33-UL60, No. 101 E52811A	362K		Taiwan
57	United Tape Company	UT-602	114K	E34833	Taiwan
58	Frost King®	ET60	590J		Taiwan
59	Tuff™ Hand Tools				China
60	Tuff™ Hand Tools				China
61	3M Scotch™	88T			U.S.A.
62	Nitto Denko	No. 228	101K/E34833		Taiwan
63	3M Scotch™	Super 88, 054007-06143	539H	LR48769	U.S.A.
64	3M Scotch™	Super 33+, 10455NA	539H	LR48769	U.S.A.
65	3M Scotch™	700 Commercial Grade, 054007-04218	539H		U.S.A.
66	L.G. Sourcing, Inc	19453	206T	E62265	Taiwan
67	Manco	P-66	590J	LR31971	Taiwan

TABLE 1—(*Continued*)

Sample Roll	Brand Name	Product	UL	CSA Reference	Country of Origin
68	3M Scotch™	Super 33+	539H	LR48769	U.S.A.
69	3M	Tartan™ 1710, part no. 054007-49656	9Z53		Taiwan
70	Tyco Adhesives National Tape Products	No. 101, E52811A	362K	LR32044	Taiwan
71	Qualpack®	1346, 6-Color			China
72	Nitto Denko	Nitto® No. 228	101K/E34833		Taiwan
73	Frost King®, Thermwell Products Co., Inc.	ET60FR	57RJ		China
74	3M Scotch®	700 Commercial Grade, 054007-04218	539H	LR48769	U.S.A.
75	3M Scotch™	Linerless Electrical Rubber Splicing Tape, 2242, 06165			U.S.A.
76	3M Scotch®	Super 33+, Cold Weather Electrical Tape, 16736NA	539H		U.S.A.
77	3M Scotch®	Super 33+, 054007-06132	539H		U.S.A.
78	3M	Tartan™1710 General Use, 054007-49656	539H/9Z53	LR48769/LR702174	Taiwan
79	3M Scotch®	700 Commercial Grade, 054007-04218	539H	LR48769	U.S.A.
80	3M Scotch®	Super 88, 054007-06143	539H		U.S.A.
81	Ace (Imported for Henkel Capital)	All Weather	362K/E49341	LR32044	Taiwan
82	Ace (Imported for Henkel Capital)	Weather Resistant	362K/E49341	LR32044	Taiwan
83	3M Scotch®	Super 33+, 10414NA	539H		U.S.A.
84	3M	Tartan™ 1710, General Use, 054007-49656	9Z53	LR702174	Taiwan
85	Frost King®, Thermwell Products Co., Inc.	ET60FR	57RJ		China
86	Duck, Henkel Consumer Adhesives	Vinyl Electrical Tape	362K/E49341	LR32044	Taiwan
87	Nitto Denko	No. 21E			China
88	Frost King®, Thermwell Products Co., Inc.	ET60FR	906B		China
89	Power Pro Craft	ETF	VT18/4K71/E220411		China
90	Duck, Henkel Consumer Adhesives	Extra wide electrical tape	74HK/E49341/ ATC-F100	232957	China

Agilent 5973 Mass Selective Detector with a dedicated electron impact ionization source. The source temperature was 230°C. Full scan mode was employed with a scan range of 34–650 m/z. Although many samples were analyzed once, replicate analyses were conducted on numerous samples to confirm reproducibility of the data.

SEM/EDS

All samples were smeared onto a pyrolytic carbon planchet and carbon coated by vacuum evaporation. Analysis was performed on a JEOL JSM-6300 SEM (Peabody, MA) with a tungsten filament as the source. The working distance was *c.* 15 mm, the take-off angle was *c.* 30°, and the accelerating voltage was 25 keV. The 4pi Analytical energy dispersive spectrometer (4pi Analysis, Inc., Durham, NC) was operated with a dead time of *c.* 30% and counting time of 200 sec. Although many samples were analyzed once, replicate analyses were conducted on numerous samples to confirm reproducibility of the data.

Evaluation of Discrimination

For each technique, two examiners independently reviewed the data and grouped samples according to similar characteristics/composition. The examiners then compared their groupings and discussed any differences of opinion. If agreement could not be reached, a third examiner was consulted and/or the more conservative opinion was taken. For this study, the more conservative opinion was considered to be the one that resulted in less discrimination, which is in direct contrast to the conservative approach that would be taken in casework. In casework, the conservative approach would be to err on the side of discrimination. However, to do so for this study could incorrectly inflate the discrimination ability. Therefore, when a sample was considered to belong to two separate groups, it was appropriate to merge the two groups.

The total number of comparison pairs possible from a population of 90 samples was 4005, calculated with the formula $\frac{n(n-1)}{2}$, derived from the binomial coefficient, where n is the number of samples (10). To calculate the number of indistinguishable pairs resulting from each technique, the number of comparison pairs was calculated for each group. The results were then summed across the groups and the percentage relative to 4005 was calculated. This value was subtracted from 100% to provide the number of distinguishable pairs, which was equivalent to the discrimination ability of the technique. An example follows for the results of the microscopical examinations. These calculated values were used to compare the relative discrimination of each technique. The discrimination value for the techniques combined was likewise calculated using the indistinguishable sample sets following analysis and comparison of all samples by all techniques.

Results and Discussion

Microscopical Examinations

The 90 tape samples were initially grouped according to adhesive color. Three groups resulted: tapes with clear, colorless

TABLE 2—*Tape groups as determined by adhesive color.*

Clear, Colorless	Clear, with Brown Tint	Black
1, 2, 3, 4, 5, 6, 7, 8, 9, 14, 21, 22, 31, 32, 35, 37, 38, 42, 45, 46, 48, 49, 50, 51, 53, 55, 56, 57, 58, 59, 60, 66, 67, 69, 70, 71, 72, 73, 75, 81, 82, 84, 85, 86, 87, 88, 89, 90	33, 52, 62	10, 11, 12, 13, 15, 16, 17, 18, 19, 20, 23, 24, 25, 26, 27, 28, 29, 30, 34, 36, 39, 40, 41, 43, 44, 47, 54, 61, 63, 64, 65, 68, 74, 76, 77, 78, 79, 80, 83

adhesives; clear adhesives with a brown tint; and black adhesives. These groups contained 48, 3, and 39 tapes, respectively, resulting in a discrimination of 53%:

$$\left[100\% - \frac{\left(\frac{48(48-1)}{2} + \frac{3(3-1)}{2} + \frac{39(39-1)}{2}\right)}{4005} \times 100\%\right]$$

Table 2 depicts the samples represented in each group.

FTIR

Not surprisingly, the colors of the adhesives could be inferred based on the slope of the baseline in the infrared spectra. The clear adhesives' spectra had a relatively flat baseline, while the black adhesives' spectra had a sloping baseline. This phenomenon is expected from a black adhesive that scatters the infrared radiation. Therefore, the spectra of the adhesives were readily distinguished initially by the slope of the baseline. Figure 1 compares the spectrum from a typical clear adhesive to that of a typical black adhesive. Tape samples were further grouped according to the presence/absence of peaks as well as by ratios of the peaks. Grouping of the samples according to similar FTIR spectra resulted in eight groups (Groups A–H). Six groups contained only clear (colorless and brown-tinted) adhesives and two contained only black adhesives. Table 3 depicts the samples represented in each group. The resulting discrimination was 67%.

Not only were the groups defined by their spectral patterns, but the chemical compositions of the adhesives were also evaluated from the resulting spectra. Figure 2 displays a typical isoprene-based adhesive spectrum overlaid with a typical acrylic-based adhesive spectrum. The rubber matrix was the simplest component to classify. The presence of some other components within the adhesives was more difficult to assess due to similarities in chemical structure (e.g., butadiene vs. a tackifying resin, styrene vs. aromatic plasticizers). Owing to these difficulties, the rubber matrix was the only component evaluated at this stage.

Once the data analysis was completed for the remainder of the analytical techniques, the information obtained via those techniques was used to confirm the infrared classifications. During this process, it was noted that the single sample in Group A (Sample 33) had been misclassified during the FTIR data analysis as an isoprene-based rubber. Figure 3 shows this sample spectrum overlaid with the spectrum for isoprene: of interest are the 1300–1500 and 875 cm^{-1} regions. These peaks are from the presence of calcium carbonate in the adhesive. The rubber compositions of each of the groups are listed below:

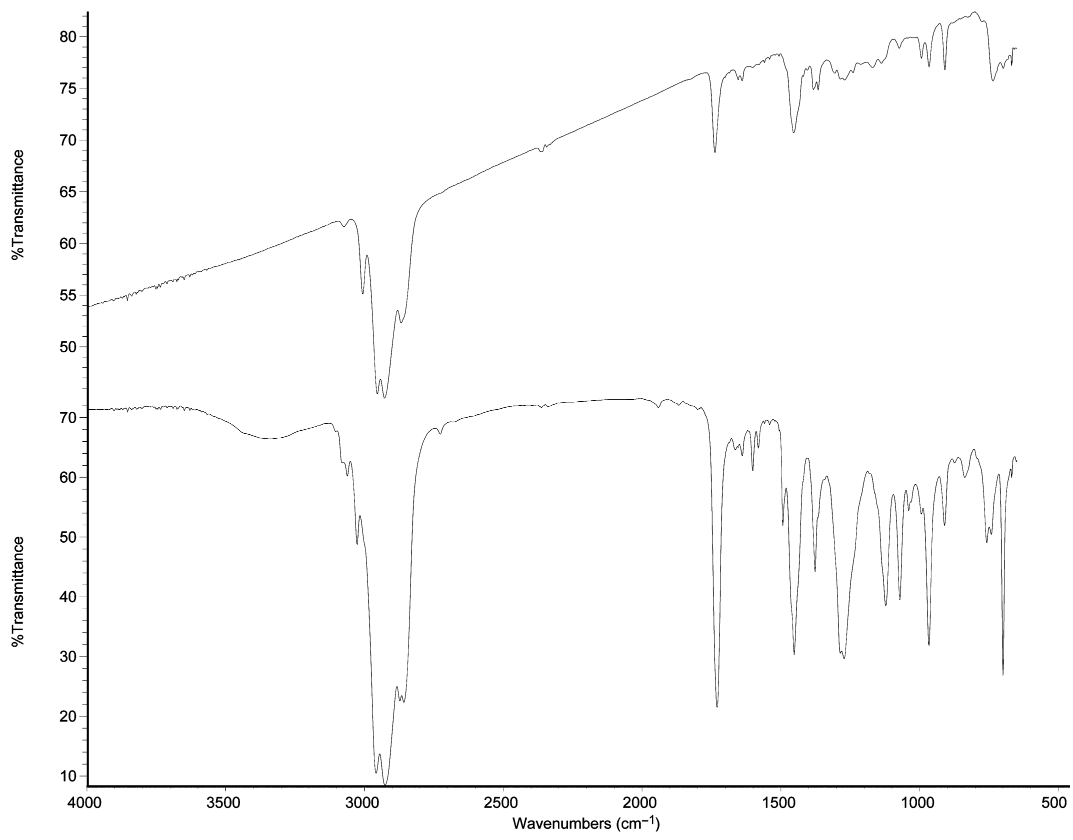

FIG. 1—*FTIR spectra of typical black (Sample 10, top) and clear (Sample 1, bottom) adhesives.*

TABLE 3—*Tape groups as determined by FTIR analysis.*

Clear Adhesives						Black Adhesives	
A	B	C	D	E	F	G	H
33	52	75	6, 71	59, 60	1, 2, 3, 4, 5, 7, 8, 9, 14, 21, 22, 31, 32, 35, 37, 38, 42, 45, 46, 48, 49, 50, 51, 53, 55, 56, 57, 58, 62, 66, 67, 69, 70, 72, 73, 81, 82, 84, 85, 86, 87, 88, 89, 90	16, 27, 28, 29, 30, 34, 36, 40, 43, 44, 47, 65, 74, 78, 79	10, 11, 12, 13, 15, 17, 18, 19, 20, 23, 24, 25, 26, 39, 41, 54, 61, 63, 64, 68, 76, 77, 80, 83

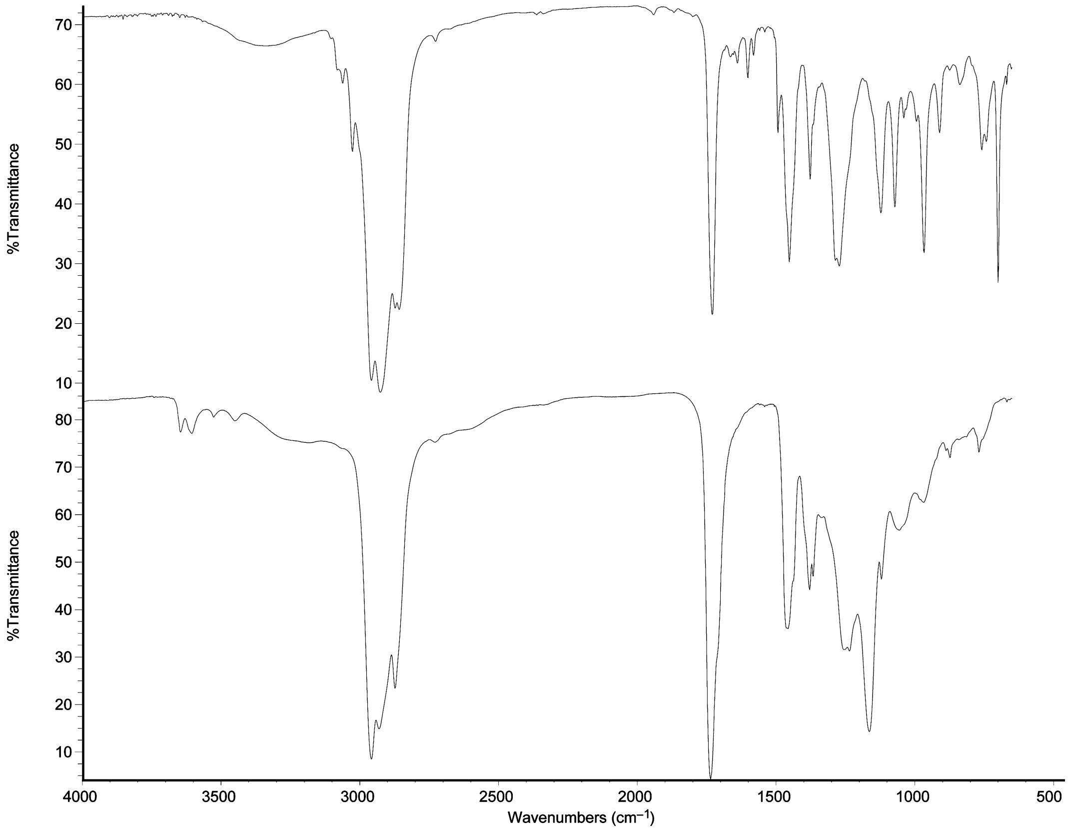

FIG. 2—*FTIR spectra of typical butadiene/isoprene/styrene (Sample 1, top) and acrylic (Sample 75, bottom) adhesives.*

A: butadiene
B: isoprene
C: acrylic
D: acrylic
E: acrylic
F: isoprene
G: isoprene
H: not easily interpreted

Py-GC/MS

Evaluation of the Py-GC/MS data separated the 90 tapes into 16 groups, with a resulting discrimination ability of 83%. The Py-GC/MS data confirmed the FTIR groups and was also able to discriminate them further. As a result, the Py-GC/MS groups were labeled using the same notations as the FTIR groups, but with additional subdivisions. Table 4 shows the breakdown of these groups. Samples 1 and 84 were indistinguishable by FTIR but could be differentiated by Py-GC/MS. Their resulting spectra and pyrograms are depicted in Figs 4 and 5. Figure 6 demonstrates the typical reproducibility of Py-GC/MS, using Sample 1 as an example.

As with the FTIR data, the Py-GC/MS data was used to evaluate the range of chemical compositions found in the adhesives of electrical tapes. However, much more information was able to be elucidated from Py-GC/MS. This technique separates and facilitates identification of the organic components of the tape adhesive. The difficulties inherent in analyzing a mixture by FTIR, which result in spectral overlap, are eliminated with Py-GC/MS. The chemical compositions determined by the Py-GC/MS analysis are as follows:

A: butadiene (B), styrene (S), mixture of phthalates
B: B, isoprene (I), S, fatty acids, adipates, benzenamine, others
C: acrylic
D: acrylic
E: acrylic
F1: B, I, S, single phthalate, trimellitate
F2: B, I, S, single phthalate, mixture of phthalates
F3: B, I, S, single phthalate, single adipate, mixture of phthalates

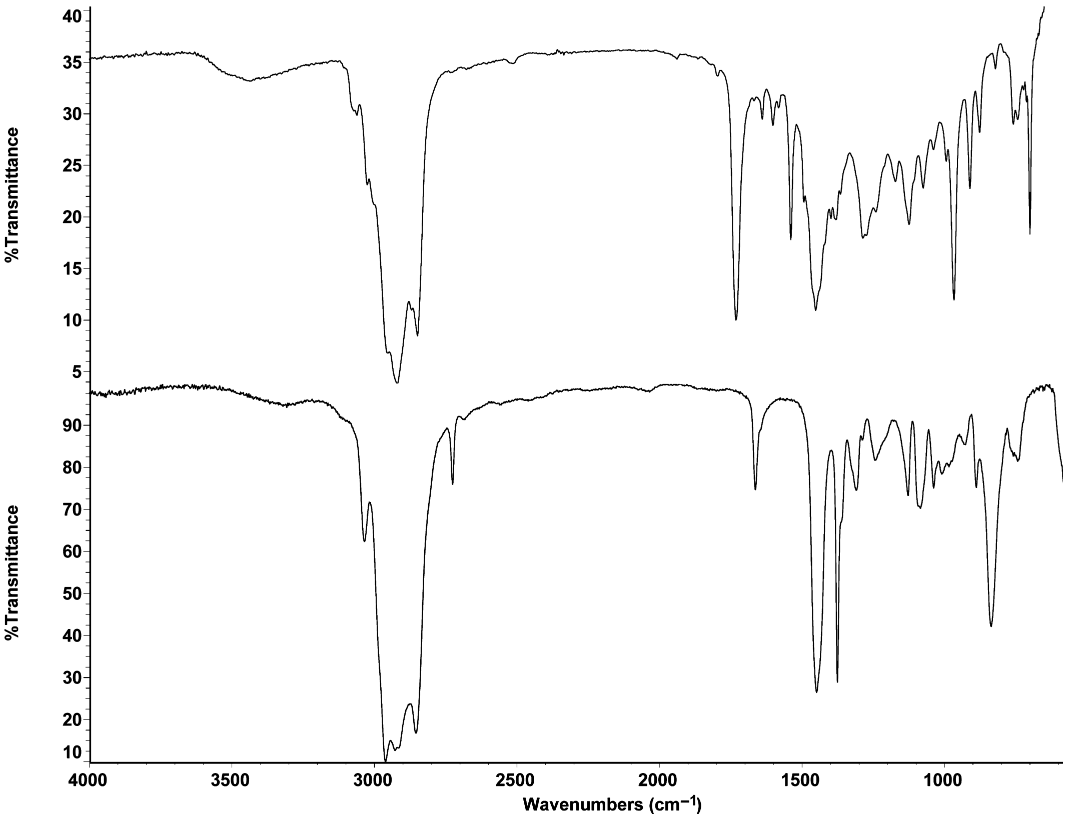

FIG. 3—*Sample 33 (top) was originally misclassified as an isoprene (bottom)-based adhesive.*

TABLE 4—*Tape groups as determined by Py-GC/MS analysis. As Py-GC/MS confirmed the FTIR groups but further subdivided them, the groups below are displayed as subdivisions of the original FTIR groups.*

A	B	C	D	E	F							G		H	
					F1	F2	F3	F4	F5	F6	F7	G1	G2	H1	H2
33	52	75	6, 71	59, 60	1, 5, 7, 48, 49, 57, 62, 69, 72	2, 8, 9, 21, 22, 31, 38, 42, 45, 46, 51, 53, 55, 56, 58, 66, 67, 70, 73, 81, 82, 85, 86, 88, 89	14, 35, 37, 50	4, 87, 90	3	32	84	16, 27, 28, 29, 30, 34, 36, 40, 43, 44, 47, 78	65, 74, 79	10, 11, 12, 13, 15, 18, 19, 20, 23, 24, 25, 26, 39, 41, 54, 61, 63, 64, 68, 76, 77, 80, 83	17

F4: B, I, S, single phthalate, methyl methacrylate
F5: like F2, but also including an isoprene dimer
F6: like F2, but also including a mixture of adipates
F7: B, I, S, significant amounts of a single adipate and a mixture of phthalates
G1: B, I, mixture of phthalates
G2: B, I, single adipate, mixture of phthalates, sebacate
H1: B, I, S, single adipate, mixture of phthalates, sebacate
H2: B, I, S, single adipate, mixture of phthalates, trimellitate

Although no such tapes were present in this study, it is feasible that FTIR could have discriminated two tapes that Py-GC/MS could not if one of those tapes contained an inorganic component.

SEM/EDS

Discrimination of the adhesives via the SEM/EDS data proved difficult because of the low levels of some elements present in the adhesive. While inorganic crystalline components historically are rarely noted in electrical tape adhesives, SEM/EDS data was still able to provide some discrimination. The samples were divided into five groups with a resulting discrimination of nearly 17%. Table 5 outlines the various groups noted. As a result of the low levels of the elements present in the adhesives, all of the samples in Groups II–V and a fraction of those in Group I were prepared again and analyzed to confirm reproducibility of the spectra as well as placement of the adhesives into each group. Reproducibility was confirmed in each instance. Figure 7 shows representative spectra from each of those groups, and Fig. 8 is an overlay of the spectra from all samples of a single group to demonstrate intra-group similarity between spectra.

Group I (Sample 1) contained no significant level of inorganic elements. Since many electrical tape adhesives are not expected to have inorganic additives, it is not surprising that this group had the largest number of samples. Group II (Sample 3), Group III (Sample 33), and Group IV (Sample 42) all contained calcium. Group II also contained small amounts of sulfur, lead,

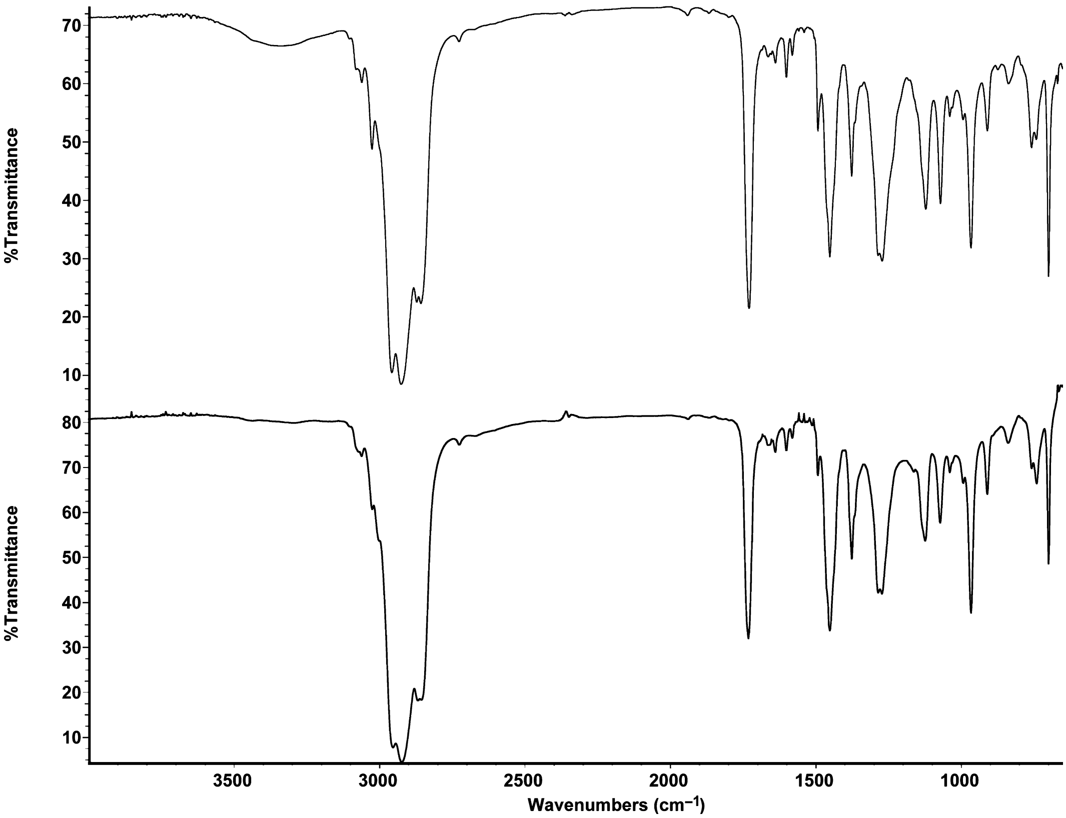

FIG. 4—*FTIR spectra for Samples 1 (top) and 84 (bottom). Determined to be indistinguishable.*

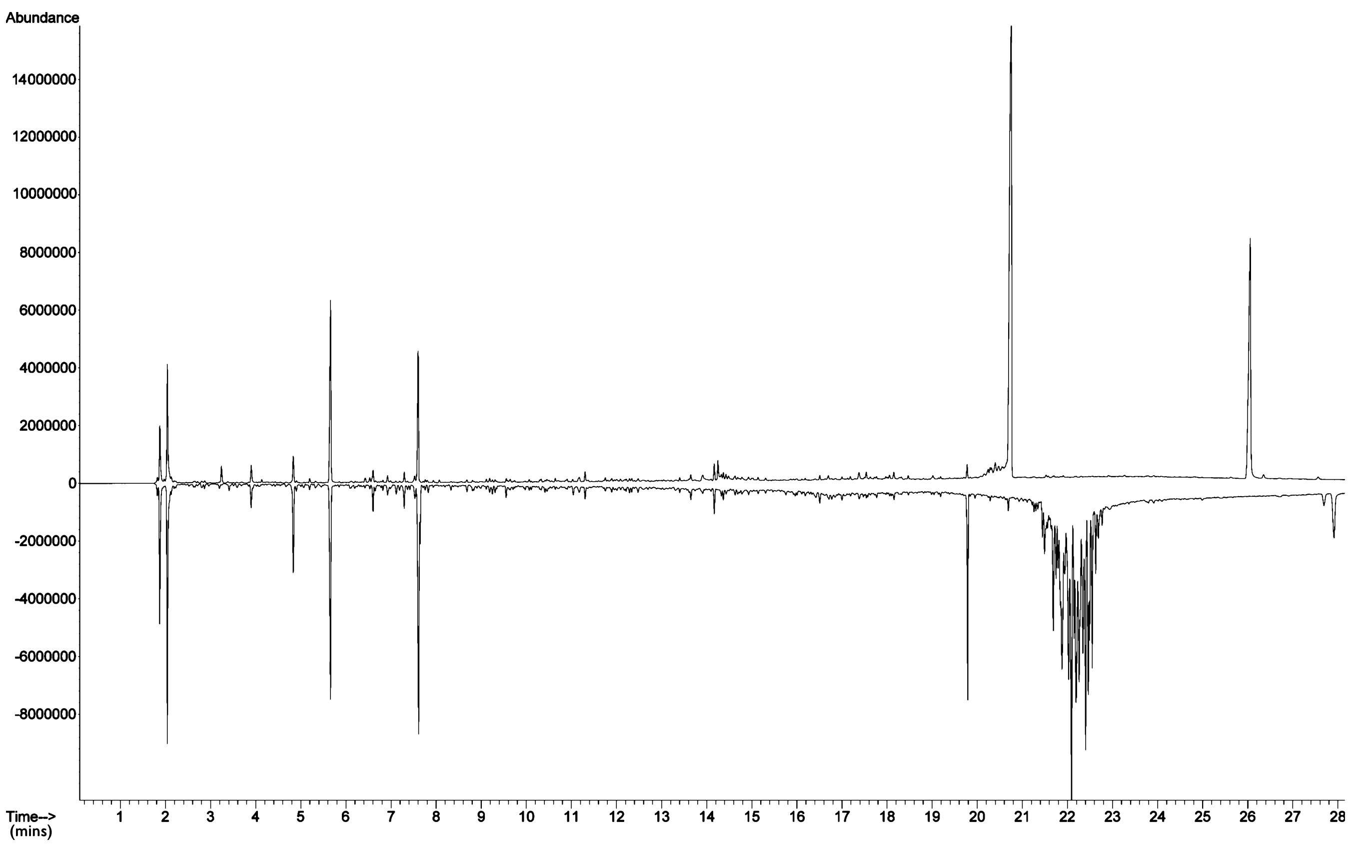

FIG. 5—*Py-GC/MS pyrograms for Samples 1 (top) and 84 (inverted). These samples have different plasticizer components.*

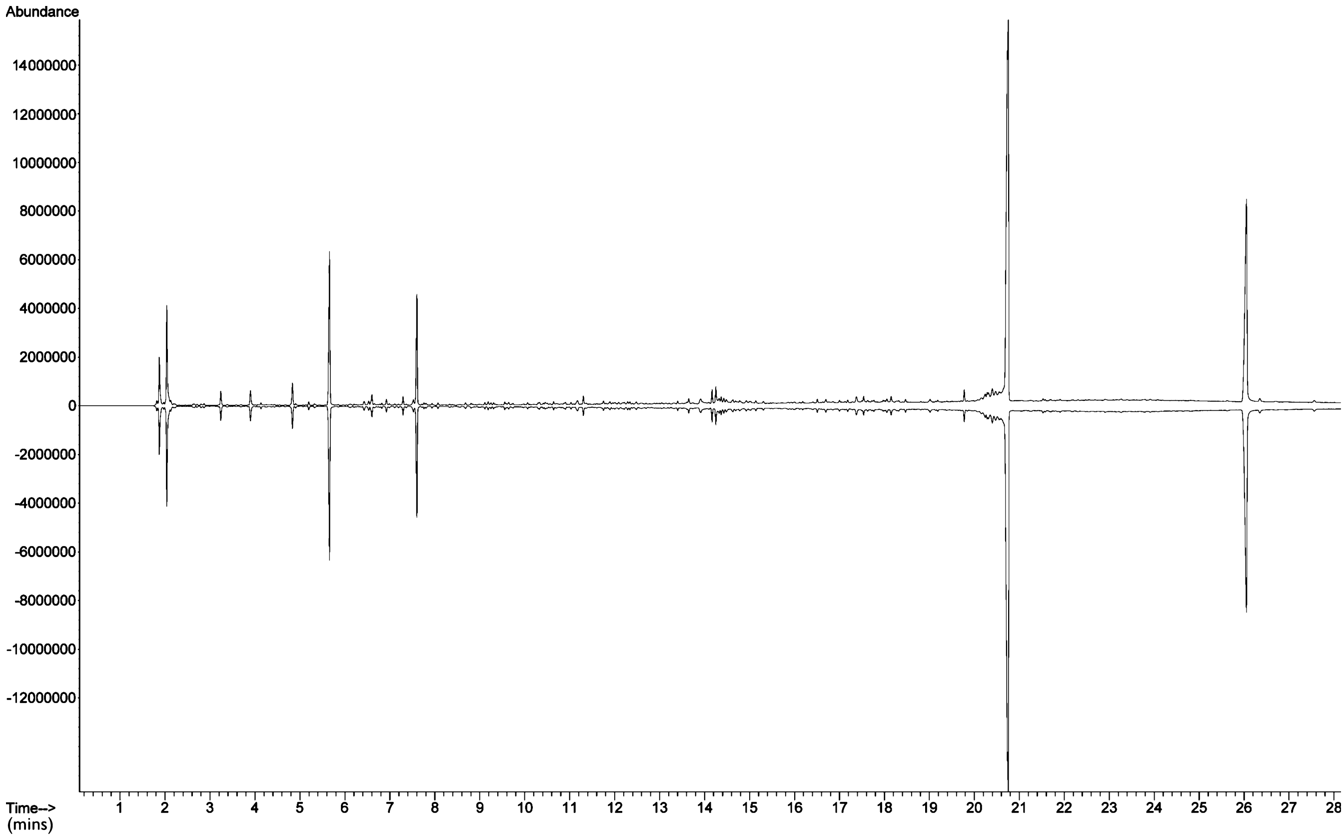

FIG. 6—*Py-GC/MS pyrograms for replicate analyses of Sample 1, demonstrating reproducibility.*

TABLE 5—*Tape groups as determined by SEM/EDS analysis.*

I	II	III	IV	V
1, 2, 4, 5, 6, 7, 8, 9, 10, 11, 12, 13, 14, 15, 16, 17, 18, 19, 20, 21, 22, 23, 24, 25, 26, 27, 28, 29, 30, 31, 32, 34, 35, 36, 37, 38, 39, 40, 41, 43, 44, 45, 46, 47, 48, 49, 50, 54, 55, 57, 58, 59, 60, 61, 62, 63, 64, 65, 66, 67, 68, 69, 70, 72, 73, 74, 75, 76, 77, 78, 79, 80, 81, 82, 83, 84, 85, 86, 87, 88, 89, 90	3	33, 52	42, 51, 53, 56	71

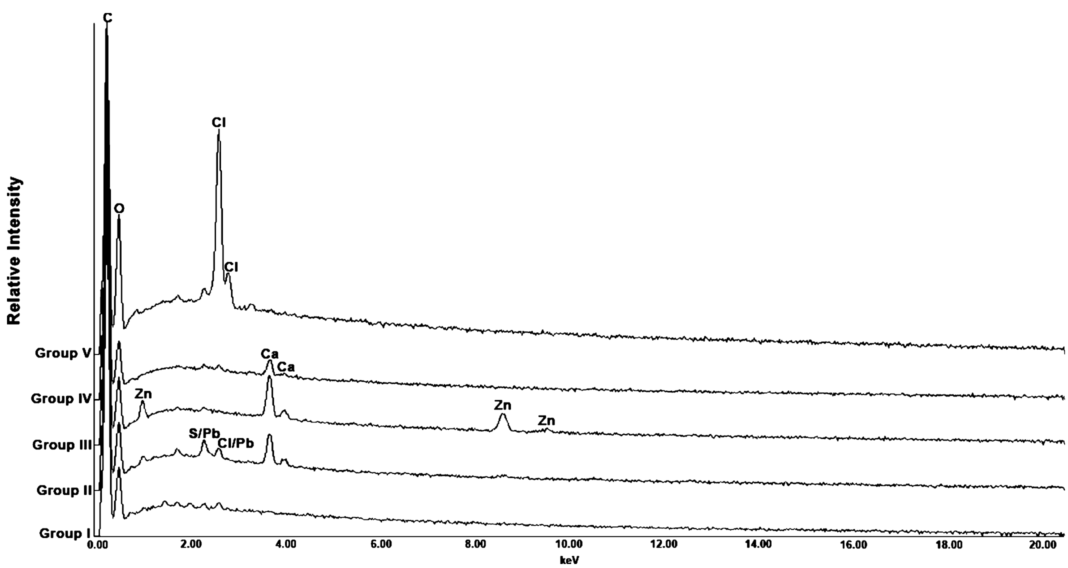

FIG. 7—*EDS spectra of representative samples from each EDS grouping, displayed in square root scale. The groups are represented by the following samples: Group I – Sample 1, Group II – Sample 3, Group III – Sample 33, Group IV – Sample 42, and Group V – Sample 71.*

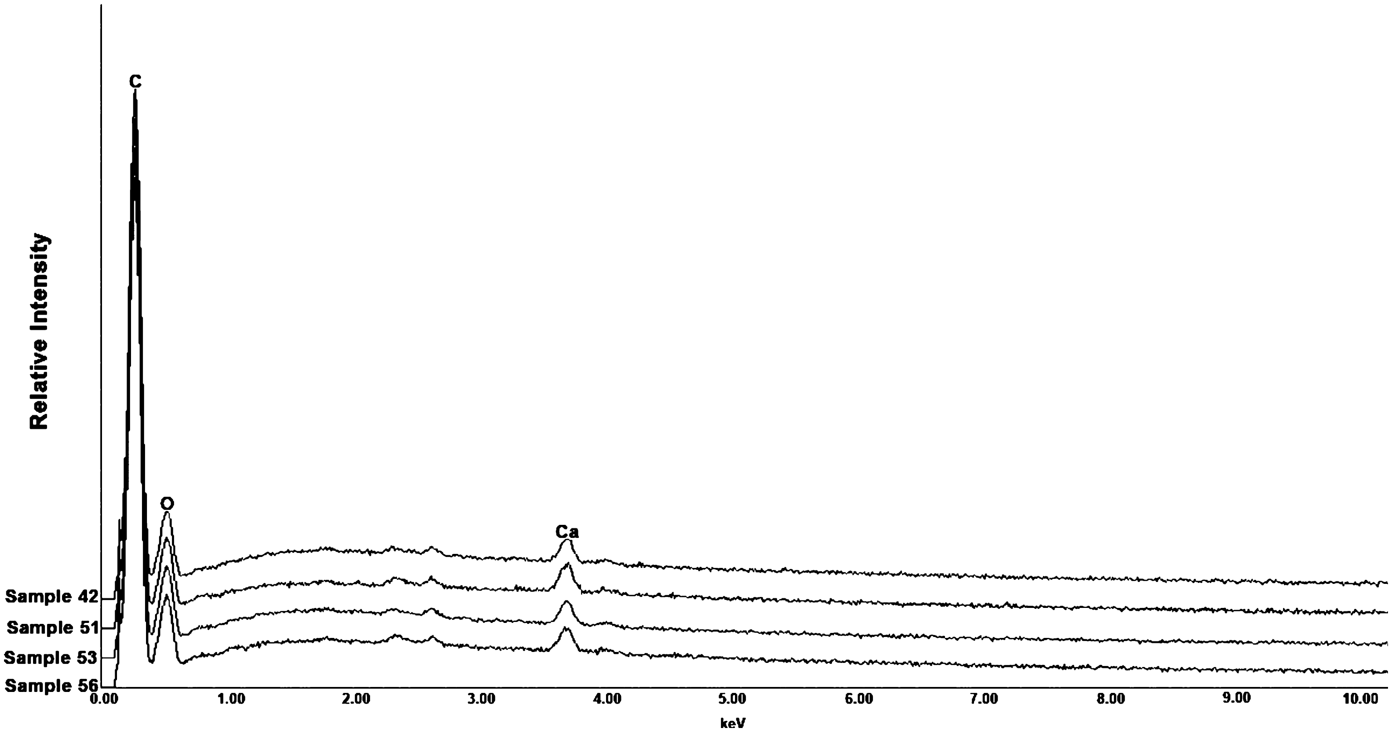

FIG. 8—*EDS spectra of all samples from EDS Group IV, displayed in square root scale.*

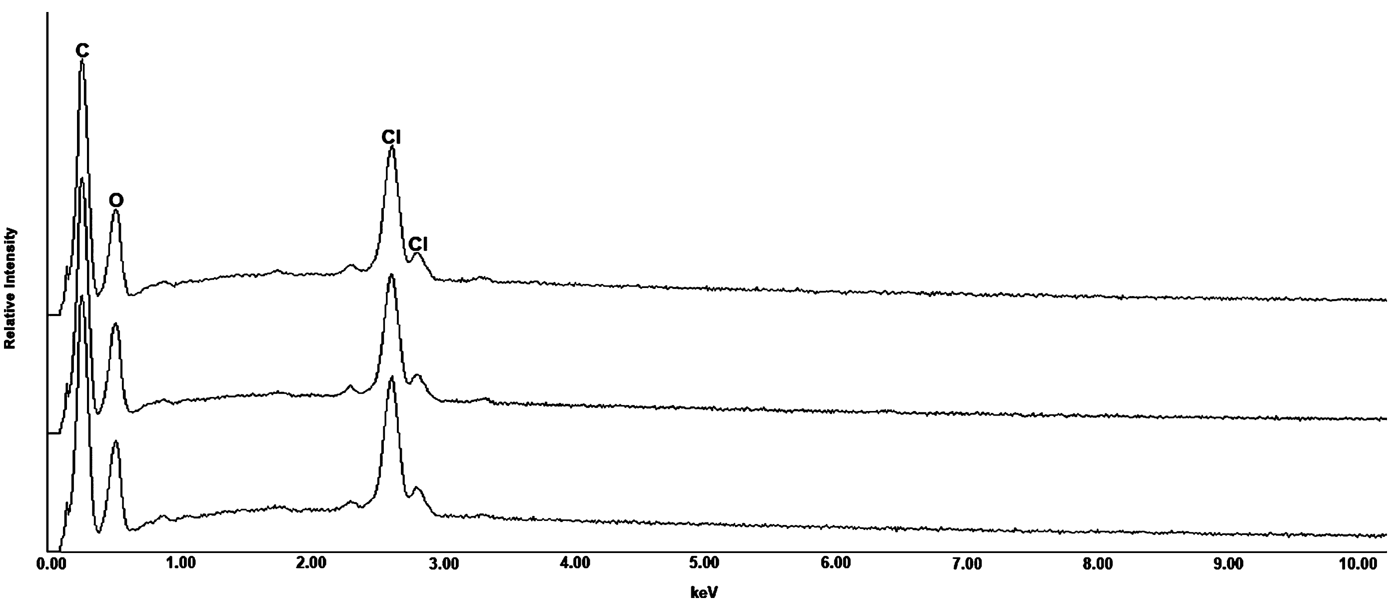

FIG. 9—*EDS spectra of replicate analyses of Sample 71, displayed in square root scale.*

and/or chlorine; however, because of the levels with which they were present and the overlap that occurs in this spectral region, specific elemental labels could not be definitively assigned. Group III differed from the others based on its level of zinc. Finally, Group V (Sample 71) had a large amount of chlorine relative to any other sample in the collection. The reproducibility of this sample is demonstrated in Fig. 9, indicating that the chlorine is not the result of contamination from the backing during sample preparation.

Techniques Combined

Following the evaluation of the individual techniques, the discrimination of the techniques combined was assessed. The result was that 85.4% of the tapes could be discriminated following adhesive analysis via the full analytical protocol carried out in this study. Table 6 displays the sample groupings according to indistinguishable adhesive physical characteristics and chemical compositions, along with the available product information.

TABLE 6—*Sample groupings following all adhesive examinations.*

Sample Roll	Brand Name	Product	UL	CSA Reference	Country of Origin
33	3M Scotch™	Cat. 190			USA
52	Frost King®	ET60FR	906B		USA
75	3M Scotch™	Electrical Rubber Splicing Tape			USA
6	Qualpack®	1346, 6-Color			China
71	Qualpack®	1346, 6-Color			China
59	Tuff™ Hand Tools				China
60	Tuff™ Hand Tools				China
1	Marcy Enterprises, Inc.	MA 750	111K		Taiwan
5	Tape It, Inc.	E-60	119K		Taiwan
7	Marcy Enterprises, Inc.	MA 750	111K		Taiwan
48	Tape-It	36-T			USA
49	Tape-It	36-T			USA
57	United Tape Company	UT-602	114K/E34833		Taiwan
62	Nitto Denko	No. 228	101K/E34833		Taiwan
69	3M	Tartan™ 1710, part no. 054007-49656	9Z53		Taiwan
72	Nitto Denko	Nitto® No. 228	101K/E34833		Taiwan
2	Advance®	AT7, BS3924, 31/90Tp			England
8	Manco®	200 MPH, AE-66	590J	LR31971	Taiwan
9	Archer® (Packaged for Radio Shack)	64-2349	590J		Taiwan
21	Manco®	P-66	590J	LR31971	Taiwan
22	Manco®	667 Pro Series™	590J	LR31971	Taiwan
31	Regal®	Model ET-6			Taiwan
38	Manco®	P-660	590J	LR31971	Taiwan
45	Calterm®	49605	590J		Taiwan
46	Manco®	P-20	590J	LR31971	Taiwan
55	Manco®	1219-60	590J	LR31971	Taiwan
58	Frost King®	ET60	590J		Taiwan
66	L.G. Sourcing, Inc	19453	206T/E62265		Taiwan
67	Manco®	P-66	590J	LR31971	Taiwan
70	Tyco Adhesives	No. 101, E52811A	362K	LR32044	Taiwan
73	Frost King®	ET60FR	57RJ		China
81	Ace (Imported for Henkel Capital)	All Weather	362K/E49341	LR32044	Taiwan
82	Ace (Imported for Henkel Capital)	Weather Resistant	362K/E49341	LR32044	Taiwan
85	Frost King®	ET60FR	57RJ		China
86	Duck, Henkel Consumer Adhesives	Vinyl Electrical Tape	362K/E49341	LR32044	Taiwan
88	Frost King®	ET60FR	906B		China
89	Power Pro Craft	ETF	VT18/4K71/E220411		China
42	National	All-Purpose	362K		Taiwan
51	National	No. 101, E52811A	362K	LR32044	Taiwan
53	National	No. 101, E52811A	362K	LR32044	Taiwan
56	Victor Automotive Products	33-UL60, No. 101 E52811A	362K		Taiwan
14	Frost King®	ET60	206T		Taiwan
35	Frost King®	ET60	206T		Taiwan
37	National	All-Purpose Grade	206T		Taiwan
50	General Electric	GE2472-31D	206T		Taiwan
4	tesa tape, inc.	40201, No. 111 E52811A	362K		Taiwan
87	Nitto Denko	No. 21E			China
90	Duck, Henkel Consumer Adhesives	Extra wide electrical tape	74HK/E49341	232957	China
3	Work Saver™, a Royal Tools brand	Stock no. 55, 5 color P.V.C Tape Assortment			China
32	GE	GE2472-3DD	206T		Taiwan
84	3M	Tartan™ 1710, General Use, 054007 49656	9Z53	LR702174	Taiwan
16	3M	Tartan™ 1710, part no. 054007 49656	539H	LR48769	USA
27	3M	Tartan™ 1710, part no. 054007 49656	539H	LR48769	USA
28	3M	Tartan™ 1710, part no. 054007 49656	539H	LR48769	USA
29	3M	Temflex™, 1700, 54007-69764	539H	LR48769	USA
30	3M	Temflex™, 1700, 54007-69764	539H	LR48769	USA
34	3M	Tartan™ 1710, part no. 54007-49656	539H	LR48769	USA
36	3M	Tartan™ 1710, part no. 49656	539H		USA
40	3M	Tartan™ 1710, part no. 054007 49656	539H	LR48769	USA
43	3M	Tartan™ 1710, part no. 054007 49656	539H	LR48769	USA
44	3M	Tartan™ 1710, part no. 054007 49656	539H	LR48769	USA
47	3M	Tartan™ 1710, part no. 054007 49656	539H	LR48769	USA
78	3M	Tartan™ 1710, part no. 054007 49656	539H/9Z53	LR48769	Taiwan
65	3M Scotch™	700 Commercial Grade, 054007-04218	539H		USA

TABLE 6—*Continued.*

Sample Roll	Brand Name	Product	UL	CSA Reference	Country of Origin
74	3M Scotch®	700 Commercial Grade, 054007-04218	539H	LR48769	USA
79	3M Scotch®	700 Commercial Grade, 054007-04218	539H	LR48769	USA
10	3M Scotch™	Super 88, 054007-06143	539H	LR48769	USA
11	3M Scotch™	Super 33+, 10414 NA	539H	LR48769	USA
12	3M Scotch™	Super 33+, 10455 NA	539H	LR48769	USA
13	3M Scotch™	Super 33+	539H	LR48769	USA
15	3M Scotch™	Super 33+, 10455 NA	539H	LR48769	USA
18	3M Scotch™	Super 33+, Cat. 195NA	539H	LR48769	USA
19	3M Scotch™	Super 33+, Cat. 194NA	539H	LR48769	USA
20	3M Scotch™	Super 33+, 10414 NA	539H	LR48769	USA
23	3M Scotch™	Super 88, 054007-06143	539H	LR48769	USA
24	3M Scotch™	Super 88, 054007-06143	539H	LR48769	USA
25	3M Scotch™	Super 33+, 054007-06132	539H	LR48769	USA
26	3M Scotch™	Super 33+, 054007-06132	539H	LR48769	USA
39	3M Scotch™	Super 33+, 3744NA	539H	LR48769	USA
41	3M Scotch™	Super 33+, 200NA	539H	LR48769	USA
54	3M Scotch™	Super 33+, 03404NA	539H/5364	LR48769	USA
61	3M Scotch™	88T			USA
63	3M Scotch™	Super 88, 054007-06143	539H	LR48769	USA
64	3M Scotch™	Super 33+, 10455NA	539H	LR48769	USA
68	3M Scotch™	Super 33+	539H	LR48769	USA
76	3M Scotch®	Super 33+, 16736NA	539H		USA
77	3M Scotch®	Super 33+, 054007-06132	539H		USA
80	3M Scotch®	Super 88, 054007-06143	539H		USA
83	3M Scotch®	Super 33+, 10414NA	539H		USA
17	3M Scotch™	Super 88 054007-06143	539H	LR48769	USA

Py-GC/MS provided the most discrimination of these tape adhesives. As stated earlier, this technique correlated with the groupings established by FTIR and further subdivided them. Differences between groups most frequently resulted from identification of the plasticizer(s) within the adhesive.

Limited additional discrimination occurred when SEM/EDS data was incorporated into overall discrimination assessments. This was not surprising as electrical tape adhesives are not expected to contain inorganic components. In fact, for those tapes in the study that had black adhesives, no additional discrimination was obtained. However, there are examples in which elements that were present as determined by SEM/EDS analysis lead to an increase in overall discrimination. For instance, four samples (comprising SEM Group IV) were further subdivided from Group F2 following incorporation of the SEM/EDS results.

Protocol Recommendations

As a result of this study, the FBI Laboratory does not plan to modify its standard procedure for electrical tape adhesive analysis. While FTIR was unable to add any additional discrimination over Py-GC/MS, it was an appropriate screening technique for subsequent Py-GC/MS analysis. Furthermore, unlike Py-GC/MS, FTIR is a technique that is widely available in forensic science laboratories. Although Py-GC/MS was the most discriminating technique for the analysis of the electrical tape adhesives in this collection, it is destructive to the sample and time consuming in terms of sample preparation and analysis. Therefore, Py-GC/MS should be one of the final techniques to be used. However, given the low level of discrimination obtained through SEM/EDS analysis for most of the analyzed samples, the order of SEM/EDS and Py-GC/MS examinations should be case dependent and remain at the examiner's discretion.

The FBI Laboratory does not include sourcing of electrical tapes of unknown origin in its protocol nor does it plan to as a result of this study. As previously mentioned, the sample set was not meant to be used for sourcing purposes, although there does appear to be some limited correlation of the groupings to certain products. However, caution is advised in making assumptions based on any apparent correlations.

As a demonstration, numerous 3M Tartan 1710 products are included in the sample set. Most have black adhesives that are indistinguishable from each other as well as packaging markings indicating they were manufactured in the United States. However, there are exceptions. For instance, Sample 84 is a 3M Tartan 1710 product with a clear adhesive, and Sample 78 is a 3M Tartan 1710 product that is labeled as having been made in Taiwan. Therefore, the information provided on the labels is not necessarily an accurate correlation back to a manufacturer or country of manufacture.

On the other hand, the manufacturing information might explain why some samples were not discriminated. For example, Group H1 contained 23 samples of either the Super 33+ or the Super 88 tape product. Based on manufacturer information reported by Goodpaster et al. (6), the primary difference between those products is tape thickness. Therefore, it was not surprising that Super 33+ and Super 88 adhesives were not further discriminated by chemical means.

Final Note

A similar study was undertaken by the authors with regard to discrimination of electrical tape backings. The results of that study have been evaluated and compared to the results of this study in order to assess discrimination capabilities of the analytical techniques for an intact electrical tape sample. As expected, further discrimination of the samples was achieved and will be the subject of a subsequent publication.

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