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Identification and Comparison of Electrical Tapes Using Instrumental and Statistical Techniques: II. Organic Composition of the Tape Backing and Adhesive*

ABSTRACT: The microtexture and elemental composition of the backing of electrical tapes have been shown to be highly discriminating. In this study, the organic composition of electrical tape was evaluated as a complementary means of distinguishing tape brands. The backing and adhesive of 72 rolls of electrical tape were analyzed via Attenuated Total Reflectance Fourier Transform Infrared Spectroscopy (ATR FTIR) and discriminant analysis was used to classify all samples by brand. Generally, the accuracy for FTIR data (88–99%) was higher than that for elemental data (86–94%). FTIR spectra from the adhesive layer were the most discriminating. In separate studies, two fragments of blast-damaged tape were correctly assigned to their brand of origin and discriminant analysis was used to quantitatively associate or exclude tape samples from two bombing cases.

KEYWORDS: forensic science, explosives, electrical tape, infrared spectroscopy, gas chromatography, mass spectrometry, agglomerative hierarchical clustering, principal components analysis, discriminant analysis

Polyvinyl chloride (PVC) electrical tape is a widespread commercial and household product that is often encountered as physical evidence, particularly in explosives investigations. When questioned and known samples of electrical tape are presented to a forensic scientist, the possibility that the samples share a common origin can be estimated through examining class characteristics. While the probability of a coincidental association between an unknown and known exhibit should be low, the burden is on the forensic scientist to evaluate this risk by demonstrating the diversity of the sample type in question. Given the various analytical techniques that are available for this purpose, a comprehensive study has been undertaken to establish an optimal instrumental methodology for

electrical tape comparisons as well as develop statistical tests to quantitatively assess similarity or dissimilarity (1).

There are two main issues that must be addressed when conducting population studies involving class evidence. The first is establishing that the sample population is inherently diverse. Estimating the diversity of a given sample type requires the following key steps:

- 1 Understand the product population, including manufacturing and distribution
- 2 Obtain a large, representative collection
- 3 Analyze samples using multiple, orthogonal techniques
- 4 Avoid microheterogeneity through appropriate sample sizes
- 5 Assess the diversity of the sample collection with rigorous quantitative methods
- 6 Monitor changes in the population over time

Following these steps, the second issue is demonstrating that laboratory techniques can reliably discern this diversity in both pristine and evidentiary samples. In the case of electrical tape, both intact and blast damaged tape samples must be successfully associated with their nominal brands and one another. Included in this task is appropriate blind testing, to ensure that interpretation of the analytical methods is largely operator-independent and reliable.

This study has made advances on several of the steps beyond that which was previously reported (1). For example, the exemplar collection has been expanded and is now more representative of various electrical tape products. Overall, 36 nominal brands are represented and 79 exemplar rolls of tape have been analyzed, including 12 rolls that have been acquired since completing the prior study.

It has been established that scanning electron microscopy with energy dispersive spectroscopy (SEM-EDS) is able to discriminate

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tape samples based on the surface microtexture and elemental composition of the tape backing, despite relatively small sampling areas. In addition, multivariate statistical techniques were able to discern subtle differences between samples as well as provide quantitative estimates of similarity or dissimilarity (1). However, it is dangerous to presume that any single analytical method will be both necessary and sufficient for a successful analysis of any given type of evidence. For that reason, the use of organic analytical methods such as infrared spectroscopy and gas chromatography–mass spectrometry has been explored and the results compared with previous inorganic data.

Infrared spectroscopy has already been demonstrated as a suitable technique for tape analysis (2–4). The use of multivariate techniques to analyze Fourier Transform Infrared Spectroscopy (FTIR) data has also been documented (5,6). However, these statistical techniques have not yet been applied to the analysis of electrical tape via FTIR. While pyrolysis gas chromatography–mass spectrometry (Py GC–MS) has been utilized to compare electrical tape samples (7), experiments conducted in the ATF Laboratory exhibited unsatisfactory repeatability. This lack of repeatability was due in part to the large plasticizer content of electrical tape. A new technique introduced for this study, high-temperature gas chromatography–mass spectrometry (HT GC–MS), was used to profile the plasticizer content of tape samples without relying upon pyrolysis of the material. HT GC–MS has been applied to a number of samples of forensic interest, such as motor oils, waxes, and the process oil in U.S. Military Composition C-4 (8–10).

Most importantly, the degree to which an explosive event perturbs the composition of electrical tape and the extent to which postblast tape debris can be associated with its roll of origin has also been investigated. Using a single-blind study, case examples as well as a structured postblast experiment, the reliability of this particular type of class evidence is demonstrated.

Materials and Methods

Tape Exemplars

The additional tape exemplars that have been acquired are listed in Table 1 along with the manufacturing information that was present on the product packaging. The state from and year in which the exemplar was acquired are listed in the “Source” column. For the two rolls of Scotch Super 33+, the year of manufacture is reported as it is printed on the tape roll core.

Instrumental Analysis

All samples were handled with gloves to avoid any contamination. In addition, a length of tape encompassing the outer circumference of the roll was removed and discarded prior to sampling to ensure a pristine surface for analysis. All tape samples were also

immediately categorized according to the color of their adhesive (i.e., black or clear). It is known that 3M is the only manufacturer that uses carbon black filler in its adhesive layer; therefore, a careful visual examination of the adhesive color immediately discriminates 3M tapes from other brands.

The conditions used for SEM–EDS have been described previously (1). For FTIR, three separate locations on the tape backing and adhesive were analyzed using a TravelIR FTIR spectrometer (Smiths Detection, Danbury, CT). This instrument compresses the sample against a single-bounce Diamond/ZnSe crystal and utilizes Attenuated Total Reflectance (ATR) to gather spectra from the surface of the sample. In this study, all tape samples were compressed to the same nominal extent using the pressure transducer reading on the TravelIR instrument. Spectral resolution was set to 4 cm⁻¹ and a total of 16 background scans and 16 sample scans were acquired for each sample. Generally, ATR is considered a surface technique with depths of penetration into the sample on the order of microns. However, a concern when compressing the adhesive side of electrical tapes against the ATR crystal is that the adjacent layer of tape backing may produce spectral interferences. This possibility was ruled out by analyzing samples of adhesive physically removed from the backing layer and verifying that the spectra obtained were consistent with spectra obtained from the adhesive side of the intact tape. Various levels of compression were also evaluated ranging from applying the tape to the crystal without applied pressure to very high degrees of compression. No significant differences in the spectra were observed.

For GC–MS analysis, three samples of tape measuring approximately 3/4-inches square were excised from each roll using a scalpel and each was extracted with approximately 4 mL of pentane (Burdick and Jackson, Morristown, NJ). The extracts were filtered through 0.45 µm Teflon membranes (Whatman, Florham Park, NJ). The extracts were then analyzed using a Perkin Elmer Clarus 500 gas chromatograph (Perkin Elmer, Waltham, MA) with an HT5 (5% phenyl equivalent polycarborane-siloxane) aluminum-clad column (25 m × 0.22 mm i.d. × 0.1 µm film). The temperature program used an initial temperature of 100°C held for 1 min, ramped at 15°C/min to 400°C, then held for 5 min. The injector was a programmable temperature vaporizer that was ballistically heated from 50°C to 480°C, held for 2 min, reduced to 400°C, and held for 3 min. The split ratio was 30:1 and the carrier gas was hydrogen at 1 mL/min. The mass spectrometer was a Perkin Elmer Clarus 500 MS with a quadrupole analyzer and electron impact (EI) ionization. The detector was scanned from m/z 40–m/z 450 in 0.15 sec. The inlet temperature was 300°C, source temperature was 300°C, and electron energy was 70 eV. The software used was Turbomass 5.0.

Data Analysis

Each of the FTIR spectra obtained was imported into a spectral library for future comparisons. For the purposes of statistical

TABLE 1—Product information for 12 additional electrical tape exemplars.

Brand	Roll	Source	Origin	UL	CSA	Listed Manufacturer
Duck	C	PA (06)	China	74HK (E49341)	202432	ACHEM Technology
Duck (Auto)	–	PA (06)	China	74HK (E49341)	202432	ACHEM Technology
Duck (Flame Retardant)	A, B, C	PA (06)	Taiwan	362K (E49341)	32044	ACHEM Technology
Marcy (MA 750)	–	VA (98)	Taiwan	111K	–	–
Power First	D	IL (05)	China	98LJ (E174965)	–	Hebei Huaxia Enterprise
Scotch Super 33+	PB1, PB2	IL (05)	USA	539H	48769	3M
Tartan 1710	C	–	Taiwan	9Z53	702174	3M
Tape-It (E-60)	–	VA (98)	Taiwan	119K	–	–
Tesa	–	VA (98)	Taiwan	362K (E52811, #101)	–	ACHEM Technology

analysis, a constant background was subtracted from each spectrum based on its measured absorbance at 4000 cm^{-1} . Each spectrum was then normalized by calculating the sum of the squares of all absorbance values between 4000 cm^{-1} and 650 cm^{-1} . Then, each absorbance value was divided by the square root of this sum (11,12). This procedure eliminated any variability in the data due to sample amount and instrument response. Statistical evaluation of the data was completed as previously described using Microsoft Excel and a separately available add-in, XLSTAT 2008 (Addinsoft SARL, Paris, France) (1). Three stages of statistical analysis were utilized: (i) Agglomerative Hierarchical Clustering (AHC), (ii) Principal Components Analysis (PCA), and (iii) Discriminant Analysis (DA).

For AHC, the Euclidean distance was calculated between each spectrum and Ward's algorithm was used to determine clustering of the data. A truncation point was determined automatically based on the histogram of node positions for each dendrogram and served as a consistent means of indicating the major groups within each population. PCA was utilized as a means to reduce the dimensionality of the data as well as filter noise. As discussed below, PCA allows for less informative variables (wavelengths) to be effectively filtered out. For example, preliminary calculations showed that deliberately eliminating regions of the spectra dominated by diamond absorption (e.g., $1850\text{--}2250\text{ cm}^{-1}$) saved computational time but did not offer any improvement in overall classification. As PCA was utilized as a preprocessing step for DA, the PCA algorithm had already diminished the importance of those wavelengths based on their contribution to the variance in the dataset.

Method Validation

Validation of statistical models such as DA is crucial to their proper interpretation, and both validation of the estimation sample (learning set) and single-blind studies were utilized previously. This paper has adopted two additional statistical methods.

First, the nature of principal components (PCs) dictates that the initial PCs contain more information than later PCs, which consist largely of noise. This is advantageous because initial PCs can be retained for subsequent calculations, higher PCs can be disregarded, and therefore noise removed from the dataset. Deciding how many PCs to retain, on the other hand, requires some sort of criterion. One approach is to specify the amount of cumulative variance that should be encapsulated by the initial PCs and only retain enough PCs that are necessary to accomplish this (as was done previously [1]). A more objective method that was adopted for this paper is to examine the eigenvalues which are associated with each PC, as the eigenvalue for each PC directly reflects its information content. In this case, the PCs were prescreened using the *F*-test for reduced eigenvalues at a 95% confidence level (11) such that only those PCs whose eigenvalues were larger than all those that followed were used to construct the canonical variates (CVs) in subsequent DA calculations.

Second, prior validation of the DA results involved checking for any reclassification of the original observations following construction of the model. A more stringent method utilized in this paper involves a "leave one out" approach whereby single observations are systematically excluded from the model prior to its construction and then treated as unknowns. As was reported previously using SEM-EDS, a single-blind study was also conducted whereby small sections of previously analyzed tape were presented as unknowns to a co-author (KLA) and analyzed by FTIR. DA was then conducted whereby the unknown sample was treated as supplemental data for the pattern recognition algorithm and a group membership assigned.

Postblast Study

A pipe bomb was constructed by personnel from the ATF Explosives Training Branch in Fort A.P. Hill, VA using 1.5×12 inches galvanized steel pipe (Southland Products, Laredo, TX), $\frac{1}{4}$ -inches galvanized steel end caps (Mueller Industries Inc., Memphis, TN), the propellant Pyrodex (Hodgon, Shawnee Mission, KS), electric matches (Daveyfire, Walnut Creek, CA), and a popular general purpose brand of electrical tape (Power First, Roll D). The Pyrodex was placed into small plastic bags and wrapped with a second brand of electrical tape (Tartan 1710, Roll C). The device was buried in sand in order to contain the detonated fragments. One five-layered tape sample and one two-layered tape sample were recovered from the debris field. The multilayered tapes were able to be separated using forceps, leaving a near pristine backing and adhesive layer that were easily analyzed.

Results and Discussion

Between-Brand Discrimination (Black Adhesives)

While the focus of this paper is on organic analytical methods, the acquisition and analysis of additional exemplars will necessarily change the statistical models for any previous methods (i.e., SEM-EDS). Hence, all additional exemplars were also analyzed using SEM-EDS, and Table 2 contains their elemental compositions expressed as normalized peak areas.

Excluding the newer rolls of Scotch Super 33+ (D, E, F, PB1 and PB2), 24 rolls of tape with black adhesive have been analyzed in triplicate by SEM-EDS (backing side) as well as FTIR (backing and adhesive sides). These 24 rolls of tape, comprising 72 observations, will form the basis for direct comparison of EDS and FTIR. The remaining exemplars, duplicates, and/or single-blind samples will serve as validation samples.

The first stage of analysis for the FTIR spectra acquired from the backing and adhesive sides of the 3M tapes was AHC. The dendrogram for FTIR spectra acquired from the tape backing is shown in Fig. 1. Three major classes are seen, which incorporate Scotch 33 (Cluster A), Temflex 1700 (Cluster B), and a mixture of mid-grade and premium brands (Cluster C). The grouping of samples within Cluster C indicates that brand discrimination may be possible between spectra that are relatively similar. For example, all samples of Commercial 700 group together as do the older rolls of Super 88 and Super 33+ (rolls A2 and B2 for each brand), followed by the newer rolls of Super 88 and Super 33+. Interestingly, this organization is similar to that obtained from EDS data and may indicate a correlation between the inorganic and organic components that result in discrimination of samples.

When an AHC dendrogram is created, it is possible to calculate the "central objects" of each class. A central object represents the sample that lies closest in Euclidean distance to the centroid of the class as defined by AHC. These spectra, therefore, will be presented to the reader as statistically determined exemplars that illustrate the oftentimes subtle differences between classes. As is the case with all FTIR spectra of electrical tapes, the plasticizer (typically an aromatic and/or aliphatic ester) tends to dominate the spectrum with a large carbonyl absorption at 1730 cm^{-1} and C–O stretching evident in the fingerprint region. However, differences in plasticizer type and content as well as additional components of the tape backing and adhesive allow for differentiation.

Figure 2 contains three FTIR spectra of the backing surfaces from Scotch 33 (D), Temflex 1700 (A), and Super 33+ (A), which are the central objects for Clusters A, B, and C, respectively. As

TABLE 2—Elemental composition of the 12 additional tape exemplars.

Brand (Rolls)	n		Normalized Peak Area ($\times 100$)						
			Mg	Al	Si	S + Pb	Cl	Sb	Ca
Duck (C)	3	Mean	—	—	—	1.00	99.65	—	8.26
		SD	—	—	—	0.04	0.01	—	0.17
		% RSD	—	—	—	3.66	0.01	—	2.08
Duck Auto	3	Mean	—	—	—	1.03	99.46	—	10.29
		SD	—	—	—	0.01	0.02	—	0.17
		% RSD	—	—	—	0.75	0.02	—	1.65
Duck Flame Retardant (A–C)	9	Mean	—	3.48	4.06	1.38	99.81	0.78	2.64
		SD	—	0.11	0.09	0.08	0.01	0.04	0.11
		% RSD	—	3.03	2.10	5.62	0.01	5.07	4.27
Marcy (MA 750)	3	Mean	0.39	0.36	—	—	99.97	0.86	2.12
		SD	0.05	0.03	—	—	0.001	0.05	0.07
		% RSD	12.7	8.25	—	—	0.001	5.58	3.11
Power First (D*)	6	Mean	—	—	—	—	98.63	1.01	16.44
		SD	—	—	—	—	0.04	0.16	0.26
		% RSD	—	—	—	—	0.05	15.4	1.59
Scotch Super 33+ (PB1, PB2)	6	Mean	0.51	0.58	—	—	99.99	1.03	0.82
		SD	0.03	0.03	—	—	0.001	0.02	0.05
		% RSD	6.79	4.84	—	—	0.001	2.27	5.69
Tartan 1710 (C)	3	Mean	—	—	—	—	99.98	1.08	1.57
		SD	—	—	—	—	0.0003	0.02	0.03
		% RSD	—	—	—	—	0.0003	1.87	1.93
Tape-It (E-60)	3	Mean	0.42	0.39	—	—	99.96	0.86	2.69
		SD	0.04	0.03	—	—	0.001	0.07	0.08
		% RSD	10.1	7.54	—	—	0.001	7.91	2.80
Tesa	3	Mean	—	3.06	3.68	1.49	99.85	—	2.10
		SD	—	0.06	0.02	0.03	0.003	—	0.01
		% RSD	—	1.99	0.52	2.08	0.003	—	0.66

*This roll was sampled in triplicate on two separate occasions. RSD, relative standard deviation.

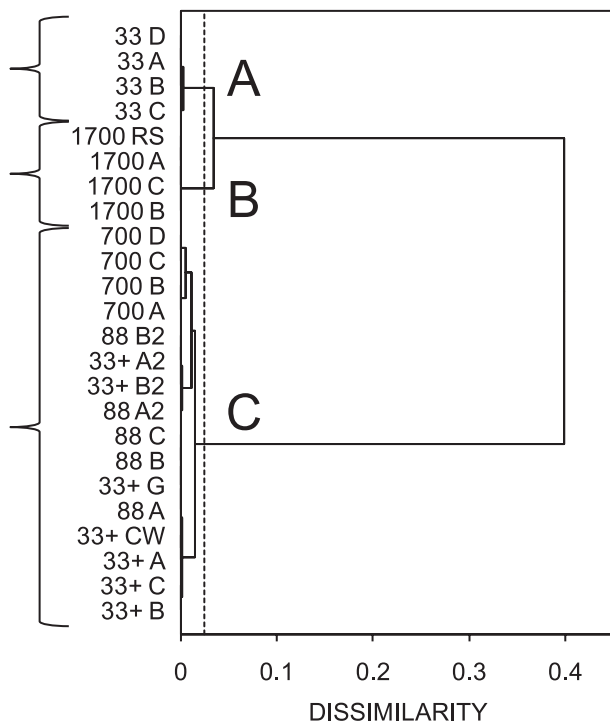


FIG. 1—Dendrogram resulting from AHC of FTIR spectra acquired from the backing side of 3M tapes. The dashed line represents an automatic truncation of the dendrogram.

would be expected based on the dendrogram, Scotch 33 (D) and Temflex 1700 (A) are highly similar with only very small differences between 1000–1200 cm^{-1} . Super 33+ (A) is more easily

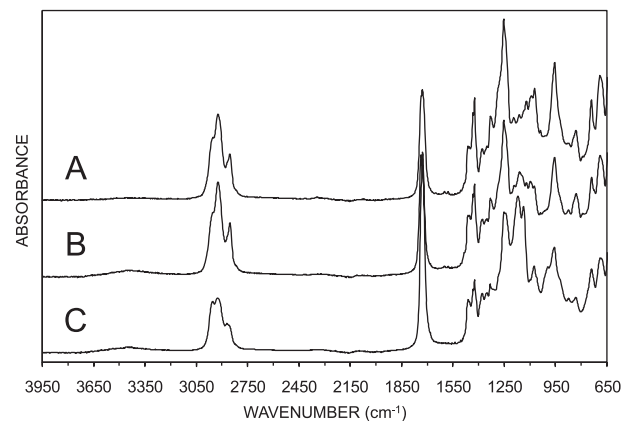


FIG. 2—FTIR spectra of the central objects as identified by AHC in Fig. 1. Class A, Scotch 33 (D); Class B, Temflex 1700 (A); and Class C, Super 33+ (A).

discernible, particularly in the C–H stretching region ($c. 2900 \text{ cm}^{-1}$) and in the fingerprint region ($950\text{--}1250 \text{ cm}^{-1}$).

In comparison, Fig. 3 contains the AHC results for FTIR spectra acquired from the adhesive side of 3M tapes. As was seen with spectra from the backing side, the organization of the classes closely follows that of the brands themselves; Cluster A contains the premium brands (Scotch 88 and Scotch 33+) while the closely related Cluster B contains mid-grade and general-use brands (Commercial 700 and Temflex 1700). Cluster C contains spectra from Scotch 33, which are more dissimilar to the other brands. The branching of the dendrogram beyond Cluster A indicates that the older rolls of Super 88 and Super 33+ are similar to one another,

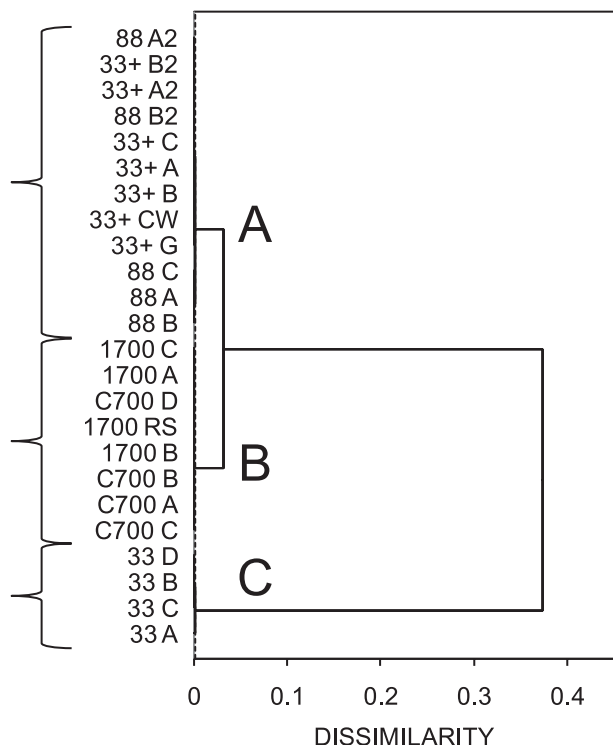


FIG. 3—Dendrogram resulting from AHC of FTIR spectra acquired from the adhesive side of 3M tapes.

while the newer rolls of these two brands cluster separately. However, these divisions are extremely compressed and are relatively minor in comparison to the initial bifurcation.

Figure 4 contains the FTIR spectra of the backing surfaces from Super 33+ (C), Commercial 700 (D), and Scotch 33 (D), which are the central objects for Clusters A, B, and C, respectively. In this case, the close similarity of the Super 33+ and Commercial 700 samples is obvious. Both feature a strong contribution from carbon black in their adhesive in the form of a sloping baseline and dispersion effects in the region of diamond absorption ($1850\text{--}2300\text{ cm}^{-1}$). However, subtle differences can be seen between 800 and 1400 cm^{-1} . The FTIR spectrum of the adhesive from Scotch 33, although it contains carbon black, is not as severely perturbed.

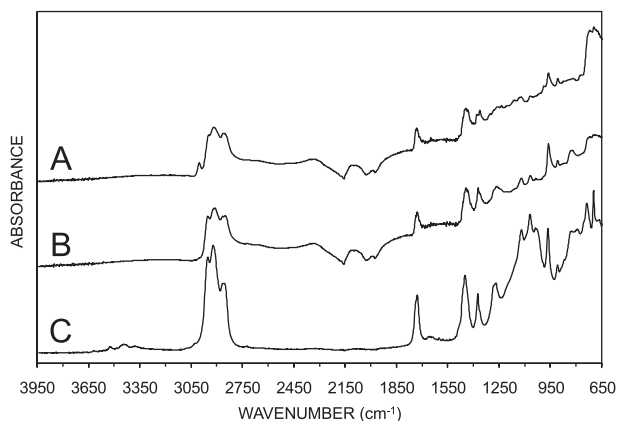


FIG. 4—FTIR spectra of the central objects as identified by AHC in Fig. 3. Class A, Super 33+ (C); Class B, Commercial 700 (D); and Class C, Scotch 33 (D).

The adhesive layer of Scotch 33, unlike any other tape studied, also exhibits absorptions at relatively high frequencies ($3350\text{--}3650\text{ cm}^{-1}$).

In total, there are then three datasets that describe the properties of tapes with black adhesive: SEM analysis of the backing, FTIR analysis of the backing, and FTIR analysis of the adhesive. When these datasets are treated with PCA and DA, significant brand discrimination is realized. The results of this analysis are summarized in Table 3, which lists the number of significant PCs, the amount of variance they captured, and the results of a DA based on this prefiltered data. Both the accuracy of the classification following cross-validation as well as the brands that exhibited any misclassifications is listed.

Interestingly, the brand discrimination achieved through analysis of the tape backing (via EDS and FTIR) appears very similar. For both techniques, samples of Super 33+ and Super 88 manufactured in the same time frame are occasionally confused, thereby reducing the overall accuracy. However, the same four brands (Scotch 33, Temflex, and old and new rolls of Commercial 700) were identified without error by both techniques. In essence, not much is gained through analysis of the tape backing by FTIR than was already apparent using EDS.

The analysis of the tape adhesive via FTIR resulted in a degree of discrimination that is extraordinarily high for all brands considered. The accuracy rate was above 97%, with only occasional confusion between older rolls of Super 88 and various older and newer rolls of Super 33+. While discrimination of older and newer rolls of the other brands was successful, it appears that the dramatic and easily discernible change in formulation for 3M tapes in 2002 was largely limited to the chemical composition of the backing (1). The success of classification based on FTIR analysis of the adhesive relies in part on differences between the classes in higher dimensions (e.g., CV3). For example, recently manufactured rolls of Super 88 have a negative score along CV3 and there is a difference along CV3 between older and newer rolls of Commercial 700. The fact that FTIR analysis of the tape adhesive appears to be so strikingly accurate is a significant finding that merits careful validation (see below).

Several rolls of tape that were not included in the comparison sample discussed above were also analyzed using both EDS and FTIR. These observations (e.g., temporal replicates, single-blind samples, and any additional exemplars) serve as an external validation sample and test the extent to which brands are truly discriminated. Table 4 summarizes the findings for four different brands and lists the accuracy for each type of analysis (EDS—backing, FTIR—backing, and FTIR—adhesive). In general, the results echo previous findings, in that validation samples from brands that are highly differentiable (e.g., Temflex) were classified with 100% accuracy. Errors are evident among validation samples from the premium brands, however, and these conform to the trends discussed above and in Table 3. Furthermore, the overall accuracy for these validation samples shows that FTIR analysis of the adhesive layer is the most reliable (87%) compared with analysis of the backing by either FTIR (80%) or EDS (80%).

Between-Brand Discrimination (Clear Adhesives)

For accurate comparison to subsequent FTIR results, the EDS data from the backing side of tapes with clear adhesive was modeled as one set rather than segregating brands based on the presence of aluminosilicate filler as had been done previously (1). Excluding three rolls of Power First (A, B, C), 47 rolls of tape with clear adhesive were analyzed by EDS and FTIR, resulting in a total

TABLE 3—Summary of PCA and DA results for 3M electrical tape.

	Backing		Adhesive	
	EDS	FTIR	FTIR	
No. of PCs	3	6	8	
Variance	99%	89%	97%	
Accuracy	86%	88%	97%	
Confused brands*	33+ (old) ↔ 88 (old) 33+ (new) ↔ 88 (new)	33+ (old) ↔ 88 (old) 33+ (new) ↔ 88 (new)	88 (old) ↔ 33+ (new) 88 (old) → 33+ (old)	

*Based on cross-validation results and prediction samples (see Table 4).

TABLE 4—Number of observations for which class membership was correctly predicted for validation samples with black adhesive based on EDS and FTIR data.

Brand	Backing		Adhesive
	EDS	FTIR	FTIR
Super 33+ (2003–2005)	22/24 (92%)	6/6 (100%)	17/18 (94%)
Super 88 (2000–2001)	3/3 (100%)	0/3 (0%)	0/3 (100%)
Super 88 (2003)	0/6 (0%)	3/3 (100%)	6/6 (100%)
Temflex 1700 (2003–2005)	3/3 (100%)	3/3 (100%)	3/3 (100%)
Overall	31/39 (80%)	12/15 (80%)	26/30 (87%)

EDS, energy dispersive spectroscopy; FTIR, Fourier Transform Infrared Spectroscopy.

of 141 observations. Rolls of tape purchased in the same location and timeframe (e.g., Bengal A/B/C, Duck Flame A/B/C, Vanguard A/B, Champion A/B/C/D, and Frost King C/D) were treated as homogenous classes for subsequent analysis by PCA and DA. Some rolls of the same brand do appear to be differentiable based on elemental composition and/or the FTIR data to be discussed below. These rolls will be treated as separate (but potentially intermingled) classes, such as Tartan 1710 A/B/C, Frost King A/B/E, and Duck Vinyl A/B/C.

Unlike 3M tapes, analysis of the backing side of these samples by FTIR resulted in a classification structure that differed from EDS. The AHC dendrogram for the FTIR spectra obtained from the backing side of these tapes is shown in Fig. 5. In this case, three clusters are evident, although additional clustering beyond these broad classes is also visible. Furthermore, the clustering of this data supports the presumed homogeneity or heterogeneity of the brands discussed above. There are some interesting similarities between the structure of this dataset and that of the EDS data, although the elemental data was analyzed as two datasets (1). For example, tapes in Cluster C all exhibit high levels of calcium in their backing from calcium carbonate, which in turn can be identified in the fingerprint region of the IR as a broad absorption around 1430 cm^{-1} and a sharp absorption at 872 cm^{-1} . These features can be seen in the Cluster C central object spectrum for Duck (Auto) in Fig. 6. As expected, the central object spectra for Clusters A (Michigan Industrial Tools) and B (Globe) are similar, yet visually differentiable. All of the spectra exhibit small but characteristic absorptions due to phthalates around $1580\text{--}1600\text{ cm}^{-1}$.

The final dendrogram composed of FTIR spectra of clear adhesives is presented in Fig. 7. Analysis of the FTIR spectra of the adhesive sides resulted in a distinctly different classification scheme. Although the truncation of the dendrogram results in six clusters, they are organized broadly into two closely related groups (Clusters A/B/C and Cluster D) and one dissimilar group (Clusters E and F). There appears to be little to no correspondence between how the FTIR spectra from the backing and adhesive sides of these

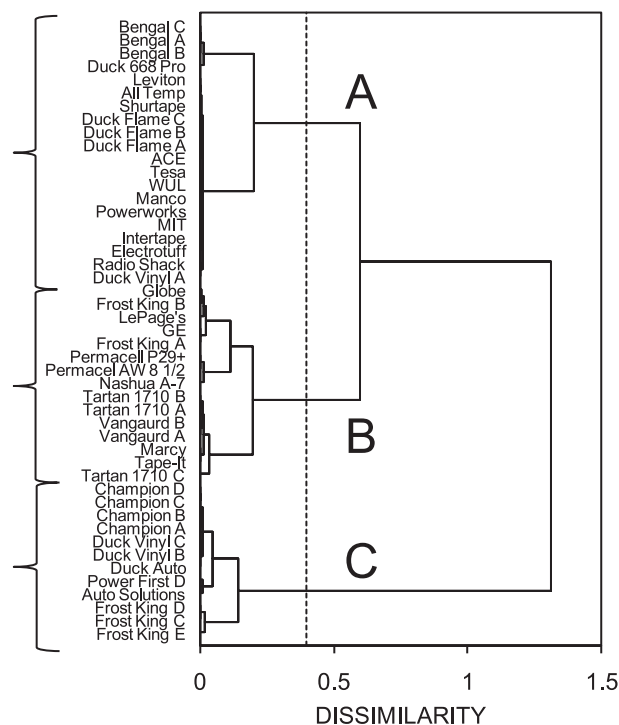


FIG. 5—Dendrogram resulting from AHC of FTIR spectra acquired from the backing side of tapes with clear adhesive.

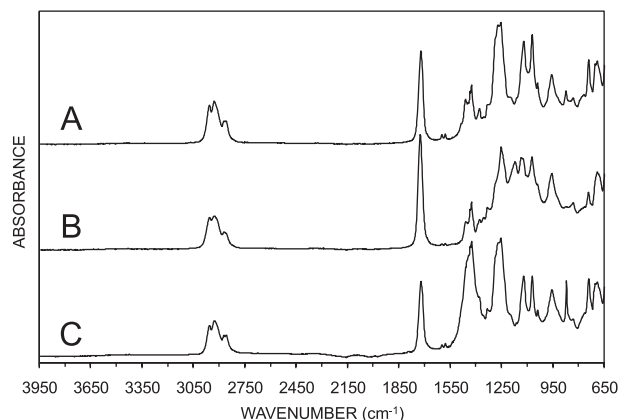


FIG. 6—FTIR spectra of the central objects as identified by AHC in Fig. 5. Class A, Michigan Industrial Tools; Class B, Globe; and Class C, Duck (Auto).

tapes are organized, save for conservation of homogenous classes such as Bengal A/B/C, Duck Flame A/B/C, Vanguard A/B, Champion A/B/C/D, Frost King C/D.

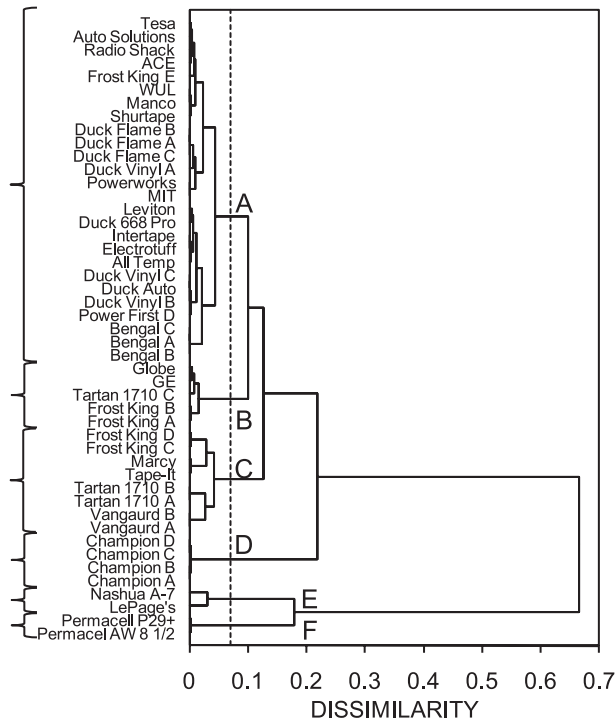


FIG. 7—Dendrogram resulting from AHC of FTIR spectra acquired from the adhesive side of tapes with clear adhesive.

The central object for each class is presented in Fig. 8, and as indicated by the dendrogram, the central objects from Clusters A (Duck Flame Retardant [C]), B (GE), and C (Vanguard [B]) are strikingly similar, whereas the central object from Cluster D (Champion [B]) is more differentiable, in part due to a relatively low level of styrene (as indicated by the sharp absorption at $c. 700 \text{ cm}^{-1}$). The central objects from Clusters E (Nashua A-7) and F (Permacell P-29+) are more distinct in part due to the relatively minor contribution of the carbonyl absorption at $c. 1730 \text{ cm}^{-1}$ (from an aliphatic or aromatic plasticizer).

The analysis of tapes with clear adhesives by both EDS and FTIR using PCA and DA is summarized in Table 5. In some cases, the confusion between brands was not mutual, and is so

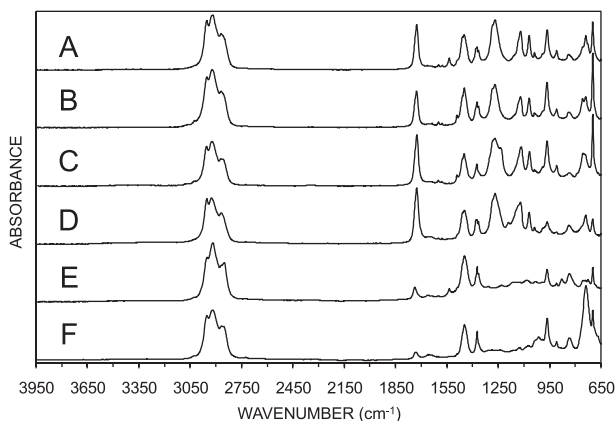


FIG. 8—FTIR spectra of the central objects as identified by AHC in Fig. 7. Class A, Duck Flame Retardant (C); Class B, GE; Class C, Vanguard (B); Class D, Champion B; Class E, Nashua A-7; and Class F, Permacell P29+.

indicated with arrows. In terms of the EDS data, several of these tape brands have already been indicated as samples that are potentially confused with other tapes based on their inorganic composition (e.g., Duck 668 Pro/Tartan 1710 [A], Leviton/All Temp, and Michigan Industrial Tools/Powerworks). The addition of 10 additional rolls of tape with clear adhesive revealed another pair of brands (Marcy/Tape-It) that exhibit confusion. It is possible, however, to differentiate some of these tapes based on their surface microtexture (e.g., Duck 668 Pro/Tartan 1710) (1). Overall, the accuracy with which tapes with clear adhesive were classified based on their EDS data remains high (greater than 94% for 141 observations).

The use of FTIR data from the tape backings to classify samples was significantly more accurate (99%) than EDS. However, some of the same tape brands that exhibited confusion in their EDS data were also confused with other brands based on their FTIR data (e.g., Michigan Industrial Tools and Tartan [A]). FTIR spectra from the tape adhesive side show that only two pairs of brands exhibit any type of confusion, and the overall accuracy (98%) is on par with the FTIR data generated from the backing side.

The results for validation samples are shown in Table 6, including some classification errors that were not previously seen in cross-validation (Michigan Industrial Tools \rightarrow Duck [A] and Frost King [A] \rightarrow GE). These errors have been included in Table 5 to provide a comprehensive view of the reliability of the various techniques. The results from EDS analysis of the tape backing were classified with 100% accuracy. Also, and consistent with data from 3M tapes, FTIR analysis of the adhesive was more accurate than that of the backing. Overall, the classification of external validation samples was highly successful, with all brands except for two (Frost King [A] and Michigan Industrial Tools) classified without error.

Postblast Study

Ultimately, any method for identifying and comparing trace evidence must be put to the test to verify its utility in real situations. In particular, material recovered from an improvised explosive device (IED) as a part of an explosives investigation is typically damaged and degraded, yet it must still be compared with a relatively pristine known sample from a suspect or search warrant. Hence, a postblast study was devised whereby known brands of tape were incorporated into a pipe bomb, one of the most commonly encountered explosive devices. These damaged tape fragments were then compared with their known rolls of origin. One exemplar brand of black electrical tape (Powerfirst [D]) was wrapped at least five times around the outside of the pipe bomb, and a second brand (Tartan [C]) was located inside the device, wrapped twice around plastic bags containing the propellant Pyrodex.

Figure 9 shows the remains of the pipe postblast. In this case, the pipe is exhibiting "banana peel" behavior, with multiple fragments from the end caps present. This is typical damage due to the deflagration of Pyrodex. In the debris field, multiple pieces of electrical tape were found nearby and ranged in size from fairly intact five- and two-layered tape fragments to small shards of extremely damaged single-layer tape. These fragments were collected and mounted on glass slides for visual examination (see Fig. 10). The backing portion was analyzed with SEM-EDS and FTIR, while the adhesive layer was analyzed only with FTIR. The results were then evaluated using multivariate statistical models.

The results of the instrumental and statistical analysis for two fragments that were recovered directly from the device are shown in Table 7. In this case, it is fortuitous that fragments

TABLE 5—Summary of PCA and DA results for electrical tape with clear adhesive.

	Backing		Adhesive
	EDS	FTIR	FTIR
# of PCs	7	11	15
Variance	99%	92%	90%
Accuracy	94%	99%	98%
Confused brands*	Duck (668) ↔ Tartan (A) Leviton → All Temp Marcy ↔ Tape-It M.I.T. → Powerworks Tartan (A) → Vangaurd	M.I.T. → Duck (A) Tartan (B) → Tartan (A) Tesa → Michigan Industrial Tools	Duck (Auto) ↔ Duck (C) Frost King (A) → GE

*Based on cross-validation results and prediction samples (see Table 6).
EDS, energy dispersive spectroscopy; FTIR, Fourier Transform Infrared Spectroscopy.

TABLE 6—Accuracy with which class membership was predicted for validation samples with clear adhesive based on EDS and FTIR data.

Brand (Roll)	Backing		Adhesive
	EDS	FTIR	FTIR
Duck (A)	3/3 (100%)	3/3 (100%)	3/3 (100%)
Frost King (A)	3/3 (100%)	3/3 (100%)	2/3 (66.7%)
Frost King (B)	3/3 (100%)	3/3 (100%)	3/3 (100%)
Michigan Industrial Tools	3/3 (100%)	0/3 (0%)	3/3 (100%)
Power First	6/6 (100%)	6/6 (100%)	6/6 (100%)
	18/18 (100%)	15/18 (83%)	17/18 (94%)

EDS, energy dispersive spectroscopy; FTIR, Fourier Transform Infrared Spectroscopy.

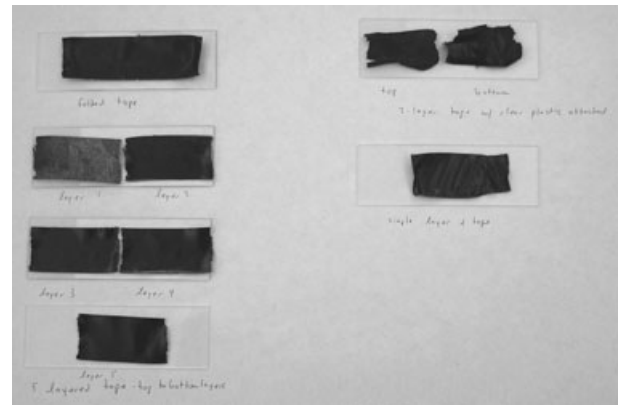


FIG. 10—Tape fragments that were recovered from the device in Fig. 9 and mounted on glass microscope slides.

TABLE 7—Accuracy with which class membership was predicted for postblast samples based on EDS and FTIR data. Tape from a roll of Power First brand was applied to the exterior of the pipe nipple, while tape from a roll of Tartan 1710 was used to secure the secondary packaging that held the explosive powder inside the pipe.

Fragment	Brand	Backing		Adhesive
		EDS	FTIR	FTIR
A	Tartan 1710 (C)	3/3 (100%)	3/3 (100%)	3/3 (100%)
B	Power First (D)	3/3 (100%)	3/3 (100%)	2/3 (67%)*

*One observation was misclassified as Duck Auto ($p = 0.96$).
EDS, energy dispersive spectroscopy; FTIR, Fourier Transform Infrared Spectroscopy.



FIG. 9—The remains of a galvanized steel pipe bomb filled with Pyrodex and initiated with a pyrotechnic fuse. The fragments of this device were fumed in the field with cyanoacrylate and are shown after removal of multiple layers of Power First brand electrical tape that were applied to the exterior of the pipe nipple. Tape from a roll of Tartan 1710 was used to secure the secondary packaging for the explosive filler inside the pipe nipple.

consisting of more than one layer of tape were recovered, as that ensures that a clean backing and adhesive surface are available for analysis. In practice, even single layers of tape folded upon themselves can be pulled apart and a suitably clean surface analyzed.

Fragment A was identified as tape from the interior of the device (it was still adhered to a shard of clear plastic from the interior

packaging). The identification of this tape fragment was very successful, with all techniques agreeing with confidence that the fragment belonged to the Tartan (C) class.

Fragment B consisted of five layers of tape, consistent with tape layers from the exterior of the pipe. The middle layer of this fragment was isolated and analyzed. This fragment was also successfully classified as belonging to the class Powerfirst, although one FTIR spectrum from the tape adhesive was misclassified as Duck (Auto) brand. Although these two brands have not been previously indicated as intermingled, the FTIR data from both backing and adhesive cluster tightly together (see Figs. 3 and 5) and visual comparison of the data confirms this high degree of similarity. These two brands, however, differ markedly in their elemental compositions (see Table 2), which serves to remove any ambiguity.

Overall, this study affirms that electrical tape is a resilient material that can survive a blast (both outside and inside a pipe bomb) in a condition that is still suitable for comparison. Of course, the device described here is admittedly quite far from a “worst-case scenario.” However, it is also fairly typical of a simple device with a widely available, yet not excessively powerful explosive filler.

Case Examples

The application of multivariate techniques to decisions in forensic casework is a powerful idea given that so many trace evidence comparisons rely upon data that is not readily quantified or for which comprehensive databases do not exist. Two case examples have already been discussed in reference to the use of EDS data to either associate or exclude samples as having come from the same source (1). This paper incorporates FTIR data into these comparisons, thereby adding a level of certainty. Table 8 summarizes the findings for the two cases, one an association, the other an exclusion.

In the association case, the tape from a propane cylinder was physically matched to the torn end on a roll of tape found in a suspect's home (1). Hence, there is a clear external confirmation that the chemical composition of the Q and K should be associated, barring some unforeseen contamination or heterogeneity in the tape product itself. The two samples were found to be elementally indistinguishable, and both were associated with the class containing a roll of Frost King (E). Unfortunately, this could not be confirmed since the known tape from the case did not have any identification markings. As can be seen in Table 8, the FTIR data served to further confirm this finding with spectra from both the backing and adhesive that were visually indistinguishable from one another as well as a previously analyzed exemplar of Frost King (E). Ultimately, both the Q and K were assigned to the same class with 100% probability. In many ways this case is a nearly ideal situation and serves as a convenient “positive control,” given the establishment of a physical match between the Q and K and the fact that both Q and K were assigned to a brand of tape that was relatively easy to distinguish elementally. Frost King (E) also did not exhibit any confusion with any other brand during validation of the FTIR data.

In contrast, the exclusion case does not involve a physical match and it features two brands that suffer from significant confusion: Scotch Super 33+ and Scotch Super 88. The initial analysis using SEM/EDS noted that the Q, a small strip of tape removed from a valve stem on a large inner tube, contained an anomalously high

level of silicon relative to the K, a roll of tape from a suspect (1). Technically, this could exclude the Q immediately given that this composition differs from all other known rolls of Super 33+ or Super 88. However, the prospect that trace contamination may be perturbing the tape backing on the Q sample remains a possibility. Again referring to Table 8, one can see that if silicon is not included in the comparison, it was found that both the Q and the K are classified by DA as belonging to the class containing older rolls of Super 33+, albeit with only moderate confidence.

The use of FTIR data can hopefully shed light on this comparison. When the backing surface of the Q and K are analyzed and classified, small but significant differences are noted. Specifically, the Q is placed in the class containing older rolls of Super 88, whereas the K is again placed in the class containing older rolls of Super 33+ (as is appropriate given the markings on the tape roll core). The spectra of the Q and K are shown in Fig. 11, along with spectra that represent the centroids for the classes containing old and new rolls of Super 33+ and Super 88. The extraordinary similarity between the spectra of tape backings from Super 33+ and Super 88 (regardless of year of manufacture) is immediately apparent. However, it should also be clear that the spectrum from the K in this case is largely indistinguishable from the exemplars. The fact that this sample was correctly classified as an older roll of Super 33+ despite the similarity of the alternatives (and the fact that previous validation of these spectra showed reliable differentiation between newer and older rolls) is also impressive.

Examination of the Q sample does show differences between it and the K, particularly around 1000 cm^{-1} . This small perturbation is also unlike anything seen in the exemplar spectra. So, as was the case with the presence of silicon in this tape backing, a unique feature differentiates it from the K sample as well as the database samples. The fact that this tape sample was assigned to the group containing older rolls of Super 88 (again with only moderate confidence) underscores one aspect of DA, in that a visual comparison will often reveal systematic differences that do not de-rail a classification, but make it suspect. Furthermore, inspection of the projection of an unknown into the factor space defined by DA is essential as well to ensure that a classification is not made despite a relatively large separation distance between the unknown data and the class in question.

The “last straw” for this tape comparison using multivariate techniques comes when the adhesive layers are compared using FTIR. In this case, the Q and K are again assigned to different groups (newer rolls of Super 88 vs. newer rolls of Super 33+). The fact that the K is misclassified (as a newer rather than older roll of

TABLE 8—Two comparisons of tape removed from IEDs to tape in a suspect's possession based on EDS and FTIR data (1). Except where noted, membership probabilities are reported as a range for three replicates.

Sample	Backing		Adhesive
	EDS	FTIR	FTIR
K1 (unknown brand)	Frost King (E) $p = 1.0$	Frost King (E) $p = 1.0$	Frost King (E) $p = 1.0$
Q1 (associated)	Frost King (E) $p = 1.0$	Frost King (E) $p = 1.0$	Frost King (E) $p = 1.0$
K2 (Super 33+)	Super 33+ (old) $p = 0.53\text{--}0.69$	Super 33+ (old) $p = 0.64 (n = 1)$	Super 33+ (new) $p = 0.64\text{--}1.0$
Q2 (excluded)	Super 33+ (old)* $p = 0.58\text{--}0.74$	Super 88 (old) $p = 0.67 (n = 1)$	Super 88 (new) $p = 1.0$

*This class prediction disregards the presence of an anomalously high level of silicon on the backing of the “Q2” tape fragment. See text and Ref. (1) for further discussion.

EDS, energy dispersive spectroscopy; FTIR, Fourier Transform Infrared Spectroscopy.

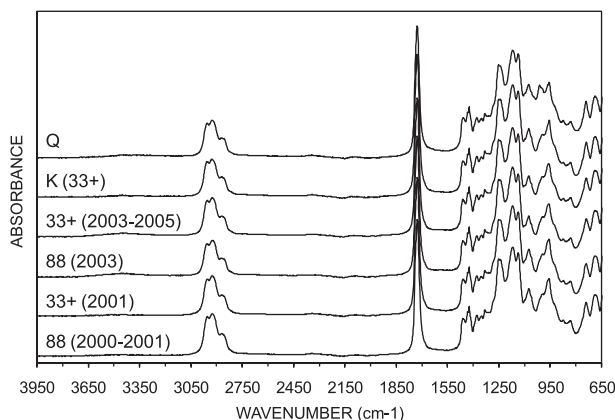


FIG. 11—FTIR spectra of the backing side of a questioned and known sample along with exemplars of Scotch Super 33+ and Super 88.

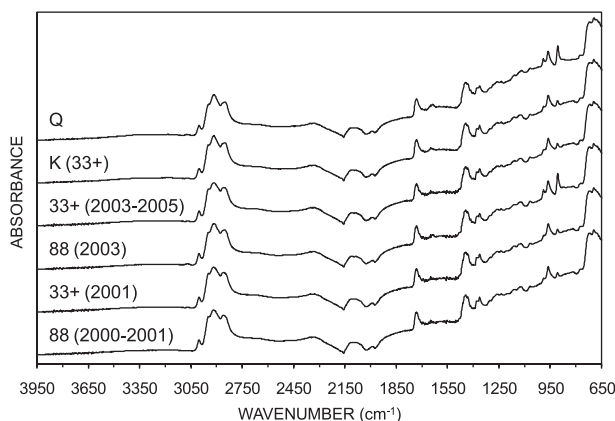


FIG. 12—FTIR spectra of the adhesive side of a questioned and known sample along with exemplars of Scotch Super 33+ and Super 88.

Super 33+) is a concern, although inspection of the extremely similar spectra in Fig. 12 makes this more understandable. Previous validation of FTIR data from the tape adhesive (Table 3) also indicates confusion between new and old rolls of premium 3M tapes, which are more clearly distinguished based on their backings. The critical findings for the adhesive analysis are (i) the certainty with which the Q is classified as a newer roll of Super 88 ($p = 1.0$), (ii) the clear correspondence between the Q spectrum and the Scotch Super 88 (2003) exemplar, (iii) the visible difference between this particular class and all others due to a distinct pattern of absorptions around $900\text{--}1000\text{ cm}^{-1}$, and (iv) the reliability with which newer rolls of Super 88 are differentiated by DA from all other premium brands.

GC-MS Analysis

As mentioned in the introduction, Py GC-MS is a popular technique for the analysis of polymers of all kinds, including those found in tapes and adhesives. However, the highly plasticized nature of electrical tape led to inconsistent results in our laboratory. For example, it was found that plasticizers would thermally desorb from the tape at relatively low temperatures during instrument equilibration and initial pyrolysis. This process was difficult to prevent or control, and hence reproducibility suffered.

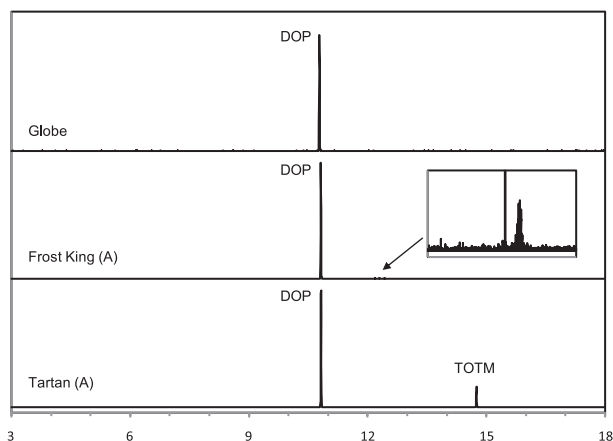


FIG. 13—Extracted ion chromatograms (EICs) of three rolls of electrical tape using HT GC-MS. Each EIC represents the sum of m/z 149 (phthalate) and m/z 305 (mellitate) where DOP, dioctylphthalate and TOTM, trioctyltrimellitate.

HT GC-MS, an analytical method that was in use in the ATF Laboratory for other types of evidence (e.g., oils and waxes), was proposed as a means to profile the types and relative amounts of plasticizer oils that were present in electrical tape. A comprehensive analysis of all tape exemplars by GC-MS is beyond the scope of this paper, but preliminary data suggests that plasticizer content may provide clearer distinctions between some tapes that have similar organic composition, or discriminate brands that are otherwise indistinguishable.

For example, Globe, Frost King (A), and Tartan (A) are all general purpose tapes with clear adhesive. Although the elemental composition of Tartan (A) is distinct (S/Pb, Cl, Sb), the elemental composition of Globe and Frost King are qualitatively the same (Al, Si, S/Pb, Cl, Ca). The FTIR data for these tapes is even more similar, in that the backing surfaces all fall within Cluster B (see Fig. 5) and the adhesive surfaces (see Fig. 7) fall within clusters B and C, which are highly similar to one another.

Figure 13 contains GC-MS data from pentane extracts of these three tapes. The data is presented as extracted ion chromatograms, which represent the sum of m/z 149 and m/z 305. Phthalates are extremely common plasticizers in tape and other polymers, and their characteristic fragment ion at m/z 149 is an obvious target for a discrimination scheme. The most common phthalate found in electrical tape was di(2-ethylhexyl)phthalate (dioctylphthalate). While several tape brands utilized dioctylphthalate (DOP), it was also apparent that mixtures of higher boiling phthalate isomers could be seen, as shown in the EIC for Frost King (A). Such complex mixtures of diisononyl- or diisodecyl phthalates were noted in several general-use tapes, and therefore they may allow for increased discrimination of such brands.

Other high boiling plasticizers were also found, such as trioctyltrimellitate (TOTM) in Tartan (A) (see Fig. 13). TOTM has a molecular weight of 546.79 amu and its mass spectrum features a prominent fragment ion at m/z 305. Unlike DOP, TOTM was rather uncommon and serves to immediately distinguish Tartan 1700 from other tape brands. It is also important to note that under the separation conditions used here, TOTM was retained 14.75 min, which corresponds to an elution temperature of 306°C . It would be difficult or impossible to elute this compound using a conventional GC column with a thicker stationary phase and lower temperature limits.

Conclusions

When confronted with an electrical tape comparison, a forensic scientist has numerous methodologies from which to choose. Regardless of methodology, the results of testing on a questioned and known sample can also be evaluated based on the analyst's training and experience. What is lacking is reliable information as to the true diversity of tape brands, the optimal analytical techniques to use, and the extent to which a questioned and known sample can be conclusively associated. It is the goal of this ongoing project to address these issues and the findings to date are summarized below.

- Analysis of the tape backing (both surface microtexture and elemental composition) has already established that electrical tapes can be differentiated from one another, including rolls of the same nominal brand due to changes in formulation.
- A direct comparison of EDS and FTIR using 24 rolls of 3M tapes with black adhesive showed that FTIR analysis of the tape backing did not offer any significant advantages. The accuracy rates for EDS and FTIR were very similar (86% and 88%) and the same brands were confused when attempting DA (i.e., rolls of Super 88 and Super 33+ manufactured in the same time frame).
- DA based on FTIR analysis of the adhesive was significantly more accurate (97%). In addition, recently manufactured rolls of Super 88 were unambiguously identified based on subtle but systematic features in their adhesive FTIR spectra.
- A direct comparison of EDS and FTIR using 47 rolls of tape with clear adhesive showed that FTIR analysis was generally superior to EDS. The accuracy rates for analysis of the tape backing and adhesive by FTIR were higher (99% and 98%, respectively) and the overall level of confusion between brands was much reduced.
- A postblast study demonstrated that DA can correctly associate blast-damaged tape to its brand of origin, particularly when both EDS and FTIR are used to cross-check such a conclusion.
- The application of DA to actual case samples demonstrates that a conclusive and quantitative association can be made (which was externally validated by a physical match). Exclusion of two samples is also possible via DA, and in this case, FTIR analysis of the tape adhesive layers proved to be the most critical information.
- Analysis of electrical tape via Py GC-MS can be difficult, and HT GC-MS was introduced as a means to profile the plasticizer content of tape. Several general-use tapes were analyzed that contain a very common plasticizer (e.g., DOP), but the presence of other phthalate isomers or a higher boiling plasticizer (e.g., TOTM) immediately discriminates the samples.

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